CHEMICAL HERITAGE FOUNDATION

VINCENT J. COATES

A Work In Analytical Instrument Design
(1925-2003)

Transcript of an Interview
Conducted by

David C. Brock

at

Milpitas, California

on

5 May 2003

(With Subsequent Corrects and Additions)

Vincent J. Coates

CHEMICAL HERITAGE FOUNDATION
Oral History Program
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Chemical Heritage Foundation
Oral History Program
315 Chestnut Street
Philadelphia, Pennsylvania 19106

VINCENT J. COATES

1925	Born in Bridgeport, Connecticut on 19 January

Education

1946	B.S., engineering, Yale University

Professional Experience

1943-1946	United States Naval Reserve
1946-1948	Designer, Chance-Vought Aircraft Corp.
	Perkin-Elmer Corporation
1948-1952	Project Engineer
1952-1954	Sales Engineer
1954-1955	Manager, Special Engineering Sales
1955-1959	Manager, Applied Engineering
1958-1963	Director, Research Internal Operations
1963	Director, Distribution Products Department
1963	Co-Founder, Coates and Welter Instrument Corporation
	Nanometrics Incorporated
1975	Founder
1975-1996	President
1975-1997	Chief Executive Officer
1989	Secretary
1999-present	President, Vincent J. Coates Foundation

Honors

1995	Semiconductor and Equipment and Materials International Award

ABSTRACT

Vincent J. Coates begins the interview with a description of his childhood in Bridgeport, Connecticut. Having been too young to join the military at the start of World War II, Coates got a job filing machine parts and began attending the Bridgeport Engineering Institute. He later applied the knowledge he had gained at the Institute on the Navy's Officer Candidate School exam, earning him the highest score in Connecticut. At the behest of his mother, Coates attended Yale University, majoring in mechanical engineering. After a short tour in the Navy, Coates took a job at Chance-Vought Aircraft. He worked there for two years, but when he learned that the company planned to move to Texas, he decided to seek employment elsewhere. In 1948, he was hired at Perkin-Elmer Corporation; a job that was to have a great impact on his life. He began as a project engineer, but when John U. White left suddenly in 1949, the responsibility for their project, the Model 21, fell completely on Coates's shoulders. He was undaunted, however, and after extensive research of infrared spectroscopy, Coates, with the help of John Atwood, finished the instrument. After the original Model 21 became a proven success, Coates began developing accessories for the instrument, such as the Prism Interchange Unit, to expand its potential market. Eventually, he was moved to California to head their Ultech Company subsidiary. Coates decided to leave Perkin-Elmer after the president decided to shut down Coates's field-emission scanning electron microscopes [FESEM] project at the request of Hitachi. Having realized the potential of FESEMs, Coates and Len Welter started the Coates & Welter Instrument Company to produce the world's first commercial FESEMs. Though they had a good business, they soon ran out of money and were acquired by the American Optical Corporation [AO]. Coates worked for AO briefly, and then he stared his own business, Nanometrics Incorporated, in 1975. At first, Coates attempted to build and sell a Raman spectrophotometer system, but the instruments resolution proved inadequate for measuring Raman lines. He then adapted his instrument for measuring fluorescent-tagged samples. He had assumed the instrument would be useful for biological research, but nobody was interested initially. He finally, and unexpectedly, found a niche for the instrument in the measurement of integrated circuits. His Microspot Film-Thickness-Measurement Systems became essential for the manufacture of advanced microchips, and his company became extremely successful as a result. Currently, Coates shares his successes with the scientific community through the philanthropy of the Vincent J. Coates Foundation.

INTERVIEWERS

David C. Brock is Program Manager for Educational and Historical Services at the Chemical Heritage Foundation in Philadelphia. He is currently a Ph.D. candidate in the History Department, Program in the History of Science at Princeton University. In 1995, Mr. Brock received his M.A. in the History of Science from Princeton University and in 1992, he earned a M.Sc. in the Sociology of Scientific Knowledge from the University of Edinburgh.

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INTERVIEWEE: Vincent J. Coates

INTERVIEWER: David C. Brock

LOCATION: Milpitas, California

DATE: 5 May 2003

BROCK: Please discuss your childhood.

COATES: I was born in Bridgeport, Connecticut, in January of 1925. I went to grammar school and high school in Bridgeport, graduating in 1941. I was only 16 years old then, and World War II had just begun. Since I was too young to be drafted, I got a job. The only job that I could get at that time was at Moore Special Tool Company near my home. I worked on the bench filing the burrs off of machined parts. I would spend an entire day filing one box of parts, only to come in the next day to file another large box of parts. There were a lot of Defense contracts issued at the beginning of the War; one of which they received. We all worked twelve hours a day, seven days a week. I was paid 25 cents per hour. It didn't allow me much time for a social life, but that was my life when I was 16 years old.

One day, while at work, the guy next to me struck up a conversation. He said, "I just signed up with Bridgeport Engineering Institute in downtown Bridgeport. I don't see myself doing this job the rest of my life!" I said, "For God's sake, that's right. I don't see it either. How did you sign up? I think I'll do it too." So he and I, despite our long schedules, would leave work at seven o'clock and take the bus downtown for classes from eight to ten. We did that for about a year-and-a-half. I studied math, physics, chemistry, metallurgy, and so forth. After about a year of taking courses at the Bridgeport Engineering Institute, I was qualified for a much better job. I left my job of filing parts and took a position in the microscope laboratory at the Bridgeport Brass Company. The laboratory tested the brass parts that were made at the factory. We tested many of the brass's properties, including its hardness and the general quality of its grain structures under the microscope.

While I was working at the Brass Company, the military was constantly drafting my bosses and fellow employees. At seventeen years old, I ended up running the lab during the night shift from 11:00 pm until 7:00 am. There were three or four other people working with me at night because the lab was a twenty-four-hour-a-day operation. I learned a lot about how microscopes worked during that time period. Eventually, I became the supervisor.

I learned about the Navy's plan for a new officer training school while I was working at the Bridgeport Brass Company. One of the metallurgists who worked during the day would leave me samples that he wanted me to prepare for his projects. One day, he read an article in the newspaper about a competitive test being conducted at the local high school. He told me about the test, and said that those who passed it could enter the Navy's Officer Candidate School [OCS], or the V-12 Program. So I went to the local high school to take the test. I was the same age as the other guys taking the test, but by that time I had a few more years of experience and education than they did. I took the test and received the highest score in Connecticut.

My mother was overjoyed to hear that I had passed and been invited into the program. We were given a list of universities to select from; though I was considering a university in Maryland, my mother told me, in no uncertain terms, "You're going to Yale [University]!" [laughter] Yet another good decision made by my mother. I was scheduled to go to Yale on July 1, 1943, and was drafted just beforehand. I had just turned eighteen, but I had got into Yale in the knick of time; otherwise, I might have had to go overseas in the Army.

I started in the Navy V-12 Program at Yale University on July 1, 1943. There, I studied mechanical engineering in an accelerated program for 28 months. Our studies went unimpeded; there were few holidays, vacations, and so forth. I was awarded my mechanical engineering degree in February of 1946; by then, the War was over. I received my officer designation as Ensign in the U.S. Navy, and spent the next five or six months in shipboard training.

First, I was sent to Newport, Rhode Island, for some very intense training. Then, I was assigned to the *U.S.S. Cleveland*; a light cruiser that had seen some serious combat in the Pacific [Ocean] during the War. The two to three month trip was very boring. The only bit of excitement was a hurricane that we encountered in the Atlantic Ocean. I remember the waves breaking right over the bridge of the ship; it was incredible. Ninety percent of the people aboard became seasick. I didn't get seasick, fortunately; though, I don't know why. Eventually, we ended up in beautiful Bermuda. The Bermuda Officers' Club was selling bottles of French champagne for a dollar; it was all very nice. So I didn't have a very tough time following World War II.

My Navy tour also took me north to Canada, down the St. Lawrence River to Montreal, and then back down to New York City, where I got off in July 1946. I was assigned to the U.S. Navy Reserve with the knowledge that I could be called back at any time. In August of 1946, I took a job at the Chance-Vought Aircraft Company in Stratford, Connecticut. I was honorably discharged from the Navy Reserve several years later.

BROCK: Would you please talk more about your family?

COATES: Yes. My father, Joseph Coates, was born in Bridgeport, Connecticut. He was a machinist who did a lot of precision work. He always told me about his job when I was young. He was very proud of me and my acceptance to Yale; he called me "pal." My mother came from England with her family in 1912. She never attended high school, but she was very bright, and she always knew what was best for her kids. She was always very optimistic about my career and personal affairs. I had a great family. My father died from Parkinson's disease in 1960. My mother passed away in 1981.

My dad made this model submarine for me in the 1930s. [displaying] It's a brass submarine that he machined in good detail. It's kind of neat. He was very clever and handy.

I have two brothers and a younger sister. My older brother is named Bill and my younger brother is named Joe. They currently live in Connecticut. My younger sister is named Ethel; she lives in Florida. My son, Norman, is a productive farmer and winemaker in Northern California. He has two children, Melonie and Carl. Melonie graduated from UC [University of California at] Berkeley last year. My daughter, Darryl, married Gerry Manning and now lives in Connecticut with my two other grandchildren, Lauren [who is a senior at Yale] and Tommy, in high school. My youngest son is named John, but he likes to call himself Trevor. He has a PhD in German Literature from the University of [California at] Santa Cruz. His wife, Sarah, is also an expert in German language and culture. They both teach at Western Massachusetts College. Of all my children and grandchildren, there is not a single scientist or engineer among them; isn't that unusual?

My spouse, Stella, is my second wife. We have been married for 24 years as of June, 2003; but we've had no children together. Both of us participate in the Vincent J. Coates Foundation, which was established to fund active research in the chemistry of the brain, especially brain diseases, such as Alzheimer's disease, and Parkinson's disease.

BROCK: Why did you graduate high school so young?

COATES: My mother enrolled me in our local Catholic elementary school when I was four years old. There was no kindergarten. In those days, I guess they accepted very young children. I went through the school system and graduated at 16. It took the normal twelve years. Occasionally, the Catholic school accepted young children who wouldn't pass their first year. Instead of being able to move on, they were forced to repeat the grade. Fortunately, I was able to make it through.

BROCK: So you were in a Catholic school system, and then you went into the public school system?

COATES: Yes. I went to Warren Harding High School in Bridgeport. The transition was all right. I was never motivated in high school, but I guess I got through and graduated with B's and C's.

BROCK: Did you have an interest in technology or science while you were in grade school or high school?

COATES: Not that I remember. I was more interested in the English language, writing, and so forth. When I got out of high school, the only job I could get was in a machine shop. I attended the Bridgeport Engineering Institute at night because the guy working with me had decided to enroll. The Institute was where I learned about science and engineering. It was a great institution, and I was lucky to enroll. The knowledge of mathematics, chemistry, and physics that I'd gained at Bridgeport allowed me to keep pace with the courses at Yale.

BROCK: Describe the Navy's V-12 Program.

COATES: It was a rigid program. The Navy wanted guys to study engineering, so I was told I was going to become a mechanical engineer! [laughter] It was a compressed version of Yale's program, with lots of math, chemistry, and physics. Yale was a wonderful place because I couldn't avoid becoming educated. I was able to take lots of other courses, like English literature and language courses. Further, when I had bull sessions with the other guys, they were always about sophisticated topics, like poetry, classical music, and so forth. For instance, after reading T. S. Elliot we had bull sessions about him. Yale was a wonderful place to go to school, and I ended up with a complete education, from one end to the other.

I became very interested in mechanical design while I was at Yale. Mechanical engineering consists of things like thermodynamics, which one might use, and it also includes internal combustion engines. [laughter] For me, the most attractive aspect of mechanical engineering was the prospect of designing products. I spent a lot of time studying and working on mechanical design. After being discharged from the Navy, I wanted to get involved in design. That is why I took the job at Chance-Vought Aircraft in Stratford, Connecticut.

I worked there from August of 1946, until November of 1948. Chance-Vought had a full program with lots of overtime. At first, I worked on the design-drafting board making blueprints, drawings, and learning the proper drafting techniques. I also observed the organization in the design and drafting room. Then, I was assigned to the "mold loft," where I had to make parts that fitted the aeronautically-tested shapes of airplane wings. Next, I was assigned to the hydraulics laboratory. My job was to test a new generation of hydraulic parts that were to be used in the F7U carrier-based jet. I designed portions of the F7U's arresting hook. I worked on the actual parts, like the motors, jacks, and valves used in the hydraulic system. The United States Navy ordered a changeover from a 1,500 psi [pounds per square inch] hydraulic system to a 3,000 psi system, allowing the landing gear, bomb doors, and so forth, to work more rapidly. A problem with the airplane's hydraulic systems was that all the hydraulic fluid was pumped-out if the systems were busted during combat, causing the pilot to lose control and crash. My job in the hydraulic lab was to help solve that problem. I invented a hydraulic-valve fuse that automatically closed down the hydraulic line whenever the pressure dropped on the other side of the fuse. It was the first actual invention that I helped to develop. I tested it from room temperature to minus 60 degrees F [Fahrenheit], and it worked quite well. I don't know whether the Chance-Vought ever applied for a patent on it.

In October 1948, Chance-Vought announced they were going to move the entire company to Texas; Lyndon Johnson may have been involved in that decision. Not wanting to move to Texas, I started looking for another job. I interviewed at a small company in Glenbrook, Connecticut, called the Perkin-Elmer Corporation. They had a smaller building than the present Nanometrics, with maybe one hundred and fifty employees. They had got involved with infrared [IR] spectroscopy during World War II, but Richard [S.] Perkin was more interested in astronomy and advanced optical systems.

Perkin worked on Wall Street and also belonged to an amateur astronomy society in NYC [New York City] that met once a month. The society discussed telescope design, celestial objects, photographs they had taken, and so forth. As a result of the tensions with Germany before World War II, their society could no longer order astronomical telescopes from Zeiss [Carl Zeiss Jena] and Leitz [Ernst Leitz] in Germany. So Richard Perkin and his friend, Charles Elmer, a court stenographer and society member, formed a contracting company to find optical shops in the U.S. [United States] that could make telescoping optics. They eventually found and contracted with a shop in New Jersey, named Mogey & Sons, which made optics quite successfully. At the outbreak of World War II, Perkin-Elmer won a contract to build optics for bombsights, so they contracted with Mogey & Sons to make the parts. Eventually, the two companies decided to merge. They moved to Glenbrook, Connecticut, and produced specialized roof-prisms for the US Air Force during the War.

During that time period, the American Cyanamid Company in nearby Stamford, Connecticut, was designing an IR spectrophotometer; and they requested Perkin-Elmer manufacture the optics for the instrument. American Cyanamid scientists realized that IR spectroscopy was useful for chemical elucidation and analysis, and since no commercial companies were making spectrophotometers during that time period, they needed to build their own. They purchased optics from Perkin-Elmer and built a large, and innovative, IR spectrophotometer.

Many people in Perkin-Elmer's optical department who had worked on American Cyanamid's optics asked Richard Perkin to consider building IR spectrophotometers after the War; something Perkin eventually decided to do. Then, an engineer at Perkin-Elmer, Richard [F.] Kinnaird, proposed the design of an IR system to Perkin-Elmer's optical department that was about one quarter the size of American Cyanamid's system.

Dr. Van Zandt Williams became famous for a review that he and colleagues had written about IR spectroscopy work during the early twentieth century. It's entitled, "Synthetic Rubber: A Spectroscopic Method for Analysis and Control (1)"; we used to give it to our customers. Williams worked at American Cyanamid. Eventually, Richard Perkin invited him to join Perkin-Elmer. Williams accepted, bringing to Perkin-Elmer his knowledge of American Cyanamid's spectrophotometer, which aided in the construction of Perkin-Elmer's first commercial product, the Model 12. It was a table-top instrument that incorporated aspheric optics, Halley Mogey's addition, to make it a more compact instrument. It had paraboloids, ellipsoids, and so forth, instead of strictly spherical optics. The instrument's electronics were rather crude, but the instrument was superior to American Cyanamid's instrument and found early success.

During that time period, Max [D.] Liston, of General Motors' Research Laboratory, had been involved in the design of IR spectrophotometers. General Motor's engineers had built some spectrophotometers for which Max Liston had designed a breaker amplifier [breaker-type DC amplifier] and a powerful, infrared-sensitive vacuum thermocouple. Perkin-Elmer required those instruments for their IR spectrophotometer, so Richard Perkin invited Liston, along with his breaker amplifier, to join Perkin-Elmer. General Motors had used Liston's amplifier as a product line during the War, but they weren't interested in developing IR spectrophotometry afterwards. Amplifier sales were small potatoes for GM.

Having arrived at Perkin-Elmer, Liston applied his breaker amplifier and electronics knowledge to the Model 12, forming a complete system that point-plotted IR spectra. Perkin-Elmer then developed the Model 12B, and later the Model 12C IR spectrophotometer. Soon after, Dr. John U. White was hired to extend the design of the Model 12C. I think he came out of Esso Research, which was what Exxon [Exxon-Mobil Corporation] had been named during that time period. He and Liston added a beam chopper to the Model 12 that was detected by Liston's vacuum thermocouple. The Model 12B had used direct-current [DC] detection that drifted and was very noisy, causing the data from the Model 12C's predecessors to be less useful. The users, who had to plot spectra by hand, were very unhappy because it took weeks to get a spectrum. Conversely, the chopped-beam version of the Model 12C with the fast thermocouple was the first spectrophotometer to give stable, reproducible results using a paper-chart recorder.

I appeared on the scene during that time period. I was interviewed by John White in November of 1948. He and Liston had already started designing a new instrument, the Model 21 which they based on a system built at Dow Chemical [Company]. They realized the Model 12C had the absorption bands of CO_2 [carbon dioxide], water vapor, and anything else in the atmosphere, superimposed on all of their absorption data, decreasing its usefulness. Though it was useful for observing changes at pre-selected wavelengths using quantitative analysis, the Model 12C was inconvenient for the spectral analysis of general organic compounds over a wide range. During that time period, IR spectroscopy was being used in the pharmaceutical industry, the oil industry, et cetera. Liston and White had started to design a spectrophotometer that took the optics from the Model 12C and incorporated them into a double-beam design. A company in Long Island had done the mechanical designing for the instrument, but after White received the plans and constructed the instrument, he found it didn't work. [laughter]

[END OF TAPE, SIDE 1]

COATES: The recording system didn't work, and the optical design was incomplete, and poorly constructed. Consequently, the whole project seemed like a disaster from the start. From what I understand, Richard Perkin had considered canceling the project because John White told him that the Model 21's external design was terrible and valueless. White convinced Perkin to hire a designer; someone who could take White's original ideas and put them into a working design.

I had been looking for a job around that time period, and John White was impressed enough with my interview that he hired me. I was given a drafting board, and a small office, which was the engineering department. I didn't know anything about IR spectroscopy; I didn't even know what either IR or spectroscopy was! I spent the first few months just working with Dr. White and talking about the mechanical design problems. He didn't tell me much about the Model 21's purpose or direction. For instance, he would sit next to me and say, "I want a gear box that has these features, or I need a new design that does this." So, I designed new parts and assemblies as requested.

In 1949, after we'd been working on the Model 21 less than a year, Dr. White came in to my office and told me that he had just quit. He'd had an argument with Richard Perkin and Van Zandt Williams about something, and then came in to my office and said, "Vince, I'm sorry to tell you this, but I'm leaving." After he left, I said to myself, "What do I do now?" [laughter] A few hours later, Van Williams came in and said, "Vince, we'll hire another physicist to take over the project. Do you have some things to work on?" I said, "Yes. I guess I've got some things I could do." After he left, I thought to myself, "I'd better learn something about IR spectroscopy!"

Perkin-Elmer had a small library. There, I found issues of the *Journal of the Optical Society of America* since the 1920s. I spent two months at the Perkin-Elmer library, several hours a day, reading virtually every article I could find on IR spectroscopy. I also studied the *Review of Scientific Instruments*. I learned a lot by the time I was finished; it was a very interesting field. Looking at the design of the Model 21, I realized that it was far from being completed. So, I sat down, at 25, and started to complete the design. [laughter] It was a big opportunity for a young guy like me; and for John Atwood, who designed electronics modules.

Van Williams ignored my redesign work on the Model 21. He knew I was in the engineering department designing things, but he didn't know what I was doing. He never hired the new physicist. A salesman at Perkin-Elmer, Paul A. Wilks, was selling the Model 12C as a quantitative tool. I generally sat in my little room designing, so I didn't have much contact with him. [laughter] After about a year, I finished the new prototype and tested it on a bench in the optical-manufacturing room. That became the finished design for the Model 21 instrument. During the instrument's testing, John White and Max Liston requested test data, photos, and so forth, for papers they were to submit to the *Journal of the Optical Society of America* (2). Both White and Liston had left Perkin-Elmer by the time their articles were published, but they still created a lot of interest.

I had also designed a very versatile recording system for the Model 21. I designed the drum for the recording paper, the drive for the pen, and the pen itself. I designed everything, including the paper holder, the automatic-scanning switches, and the repetitive scans for the kinetic studies of fluctuating-sample properties. As requested by our customers, the Model 21 provided the user with many options. The monochromator's optical parts were taken from the Model 12C as before.

A problem with the Model 21 was the unreliability of its IR source. To fix that problem, I consulted numerous resources on IR, and designed a new, stable mount. The design allowed users to operate for years without needing to replace a burned-out source, which was a very important quality that made the instrument reliable and easy to use.

BROCK: What was that source called?

COATES: It was called a Nernst glower. A problem was that the data became noisy if the hot source ever bent. It was heated to 1800 degrees F. To fix the problem, I designed a source that didn't move around or bend, was optically correct, and was easy to replace. Spectral-data accuracy was essential for users of the instrument because comparative spectra were run on the same paper to accurately show their differences. If the source distorted after being heated, the inaccurate results made the spectra inconclusive.

After testing the prototype in early March, I personally drove it, in the company station wagon, from Connecticut to the 1950 Pittsburgh Conference. For the Model 21's first showing, Perkin-Elmer had rented a small booth in the ballroom at the top floor of the William Penn Hotel. There was no other Perkin-Elmer equipment in the show that year. I placed the Model 21 on the provided bench, which sagged quite a bit. Then, I scanned the spectra of a Polystyrene sample film with superimposed 20 minute scans, thirty or forty times each day. Each time the scan repeated within pen-width. When I returned to Perkin-Elmer with the instrument, Williams was surprised when I told him the booth was constantly packed with people; I never even had time for lunch! He was also surprised that I didn't have any problems with the instrument.

After the instrument's initial success with customers, I began developing accessories, including solid, liquid, and gas cells for the Model 21. I eventually designed some gas cells that were 40 meters long. These were sold to the Los Angeles Air Pollution Control District [APCD] to measure the small concentrations of gases in smog. I folded the path to fit in the beam so that it bounced back and forth, making an effective forty-meter path. When its gas-cell pair, matching the sample and reference beams, was completed, tests showed that the background noise was surprisingly small. As a result, it seemed feasible to measure even smaller quantities of trace components. We had the option to design a gas cell with a longer pathway, but the design was expensive and impractical.

Another innovative concept was to expand the percent-transmittance scale of the Model 21 to magnify the absorption peaks. I designed a manually-controlled, variable gearbox and servomotor system to expand the travel of the optical wedge. The new gearbox permitted the expansion of the transmission-scale recording. For instance, a 10 percent change in the absorption caused the chart's pen to travel full scale, magnifying the recorded absorption. We found that the Model 21, with its big optics, had an all-stops-out signal-to-noise ratio of better than five thousand to one. It was an improvement that we hadn't realized before. It allowed a user to expand the scales up to 25 times while still showing a noise of less than 2 percent on the chart.

When we tested the new Model 21's scale-expansion system, we saw the peak heights of trace samples in atmospheric smog, including ozone, nitrous oxide, and other photosensitive air pollutants, subsequently proving the instrument could detect particle concentrations less than several parts-per-billion. As a result, we used forty-meter cells to produce the equivalent of a four-hundred-meter pathway. The scale-expansion control became an important feature on all Model 21s produced thereafter. It was very useful for operators identifying chemical components at low concentrations in the IR spectra. At the 1957 Pittsburgh Conference, we described the new scale-expansion system as a major breakthrough in infrared.

The Model 21's redesigned slit-width control variable potentiometer allowed a constant signal across the entire wavelength [or wavenumber range] of interest. It allowed the highest spectral resolution of the adjacent peaks, or the best possible signal-to-noise ratios during scanning. I worked with some people at the Sloan Kettering Institute who were very interested in measuring small components, mainly steroids, in biological materials. I designed a micro cell that was half the height and width of the entrance slit, and placed it inside the sample space's cover, near the slit image. It had a small volume, but radiation passed through it efficiently. It offered new capability for trace work.

I worked constantly on those interesting applications for the Model 21 from 1949 to 1953. The market for the Model 21 expanded each time I designed a new application. For instance, I designed a specular-reflectance attachment that allowed engineers to measure the reflection of materials against a known reference material; it was very useful for the tin-can industry. Engineers in that field wanted to know the composition and thickness of a sprayed on polymer coating that kept the tin can from corroding and poisoning its contents. To measure the thickness and chemical composition of the polymer, we put a small piece of the coated can in to the Model 21 and reflected light off of it. The experience gained from measuring thin films on substrates is also valuable for work in my present company.

I worked on another useful design project around 1952. I designed standard charts for storing the pure-spectra recordings used in photography. The standardized recordings were obtained on the Model 21 by running spectra of very pure, carefully-refined compounds in the sample cells. The photographs were then placed in organized files to be used as comparison standards for the identification of new compounds. The process became important for many Model 21 users, and led to the publication of thousands of spectra in a useful, standard format (3). Pure spectra are still used today.

BROCK: Did you work on that project from 1949 to 1956?

COATES: Yes. The work was part of the Model 21's legacy. Another accessory I designed was for use in the reference beam. It was a precise mechanical cell with a sliding seal designed to move two NaCl [sodium chloride] windows apart with a narrow gap. It was called a variable-thickness liquid-absorption cell. When a sample with an insignificant carbon-hydrogen absorption of an unknown solid or liquid, such as carbon tetrachloride or carbon disulfide, was placed in the sample beam, the variable-thickness cell in the reference beam was matched to cancel-out the background absorption of the unwanted solvent. The cell's design incorporated one of the first uses of Teflon as a seal. The Teflon was effective because it didn't react with the solvent. I wrote a description of the cell's design that was published in the November 1951 issue of the *Review of Scientific Instruments* (4).

Paul Wilks and Van Zandt Williams hadn't shown an interest in the Model 21 during that time period. Regardless, order requests for the instrument became so numerous that I personally had to handle some of the calls from customers who were wondering when we could deliver an instrument. During that time period, I was personally assembling and testing about one Model 21 a month. Monthly order requests for the unit amounted to around five or seven units. Eventually, I went directly to Richard Perkin and convinced him to expand the factory for Model 21 production. I was slightly intimidated because it was my first direct meeting with him, but we were overwhelmed with orders and something had to be done. Though I came off as naive, he was attentive to my proposal. When I suggested the possibility of one-hundred orders per year, the managers realized the instrument could become their biggest seller. At the behest of Richard Perkin, who overrode the objections of others, my request was approved, and Perkin-Elmer dominated the IR spectrometer market for years thereafter.

I worked with the manufacturing and purchasing departments to formulate a plan for efficiently making and testing the instrument. I was very involved in the manufacture of the Model 21 during that period. After a few years, Perkin-Elmer's cost analysts realized that the Model 21's selling price was fifteen-thousand dollars, but the building price was less than four-thousand dollars. The large profit margin helped turn Perkin-Elmer in to a major manufacturer of analytical instruments, rivaling Beckman Instruments [Inc.], and other manufacturers.

BROCK: Were you tracking the customer's need to develop new accessories, or were you dreaming up the new accessories and then finding the customers?

COATES: I think that Van Williams received telephone calls from prospective customers. He frequently came to me with customers' requests. He told me what the customer wanted to use the instrument for, and then I designed the accessory as needed. Most designs had been created with the requests of only one customer in mind, but they usually became valuable for other consumers, too. As a 25-year old with such an opportunity, I took advantage of my situation. It was extremely exciting and interesting; and I learned a lot about spectroscopy design and the general management of new products in that field.

There are some other things I might mention. John White incorporated the Model 12's monochromator in his design for the original Model 21. Perkin-Elmer's optical engineers had earlier designed a number of crystal prisms to fit into that monochromator. Each prism had a different, useful wavelength range and performance. The basic prism for most users was the NaCl [sodium chloride] prism. NaCl was hygroscopic: it fogged-up at room temperature because of atmospheric water vapor. I later added a heater to the Model 21's base, to maintain its temperature at 110 degrees F. The heater solved that expensive problem by keeping water from condensing on the prism's surface. It even worked in humid climates. Each prism was stored in a clean, desiccated container. John White had wanted replaceable prisms in the Model 21, but the instrument's complex cam system, and the electronics system's original design made the exchange of IR prisms extremely time-consuming. One would have had to disassemble the complex cam system, and its required mechanisms, and insert new electronic parts to use the IR prisms, all while preserving each prism's standardized, linear-wavelength recording on the paper chart.

I soon realized that the promise White and Liston had made to our customers concerning a convenient prism-exchange system was basically unachievable. In 1951, when I examined the early design of the Model 21, I concluded that the monochromator-prism exchange was flawed and needed to be redesigned. I took the old assembly and replaced it with a separate, interchangeable unit that mounted the prism, the wavelength cam, the optics, the mechanical parts, and the electronic parts on to a single frame. The unit was designed to be plugged in to the base reproducibly. Further, it had a lift-out handle and plug-in connectors for the electronics. Thus, one could simply take the device out, replace it with another one, and be back in operation in a few minutes.

The prism-interchange unit, as it was called, solved the interchange problem that would have greatly limited sales of the original Model 21. With the release of the interchange unit to production, we had finally fulfilled the advertised concept of a versatile system with wide-ranging performance. The prism-interchange unit not only made the Model 21 more convenient, it also expanded the spectral coverage of the Model 21 to include the visible range, out to 50 microns. Though I had held-up the production of the Model 21 until I finished the unit, much to the chagrin of both manufacturers and customers, the wait was much needed, and well worth it.

As a result of those necessary improvements, Perkin-Elmer's sales amounted to about 90 percent of the spectrophotometer market by 1954. There were other companies manufacturing IR instruments, including Beckman Instruments, and Baird Atomic [Inc.] in Boston, but our instruments sold better because we could demonstrate their wide-ranging performance, host of accessories, and better reliability compared to our competitors.

In early 1960, Beckman Instruments announced its plans to show the world the first IR instrument with a diffraction-grating monochromator system for higher resolution and wide-range scanning of spectra. Their advertisement implied that customers were to be left with prism systems that were poor in comparison to their new technology: Beckman Instrument's ads asserted a better resolution of absorption bands, and a wider range than all previously designed instruments.

By that time period [1959-1961], I was the director of research for the division of international operations. Further, I had turned engineering work for the Model 21 over to other engineers. However, Beckman Instrument's diffraction-grating instrument frightened those engineers because they thought a competitive Perkin-Elmer instrument would take two years to complete. Alarmed, the engineers came to me with fears over losing our market.

As I thought about the situation, I realized a possible solution. I remembered when I ran the applications-engineering group [and was head of the special-engineering division], I designed several kinds of special-instrument systems. For example, I made UV [ultraviolet] spectrophotometers, fluorescence-measurement systems, et cetera. One special project involved the replacement of the Model 12C prism with a diffraction grating; a project that led to our acquisition of many low-cost, replica diffraction gratings. Therefore, I went to our lab and bonded one of those diffraction gratings to a specular-reflectance attachment that I had designed years earlier for the Model 21. Then, I measured the reflectivity of that grating at normal incidence, over wavelengths from 2.5 to 15 microns. In the 6 to 15 micron range, the reflectivity approached 100 percent, meaning the diffraction grating could replace the Littrow mirror that worked with the NaCl prism at long wavelengths. Further, the diffraction grating could be repositioned to rotate over a normal grating angle for short wavelengths, permitting it to resolve the high-resolution peaks from 2.5 to 6.5 microns; a region where the NaCl prism alone, from the original Model 21, offered poor spectral separation.

As a result of those tests, the prism-grating-interchange unit was born. The interchange unit could be used with both new and old instruments as a modest-cost upgrade. The modification retained the standard scan for the existing chart paper, while also producing a competitive, high-resolution scan that was acceptable to all users of the Model 21. Alternatively, Beckman Instrument's IR spectrophotometer had a chart-scanning method that was very inconvenient and not standardized.

I had conducted the prism-grating-interchange unit experiment in January, and two months later we introduced the redesigned instrument at the Pittsburgh Conference, to compete with the Beckman Instrument's model shown at the same time.

Perkin-Elmer's Tom Flynn designed the production system that was shipped several months later. I don't think Beckman received many orders for their new design because our system was superior to theirs right out of the box. I was very pleased with the work I had done saving my old friend, the Model 21, and giving all customers a big lift in capability at modest cost.

A year later, Perkin-Elmer introduced the Model 221 spectrophotometer, which had diffraction gratings with an optical-filter system. The instrument didn't require a prism. If I'm not mistaken, Walter Slavin was involved in part of that design.

By 1955, Perkin-Elmer had been studying many applications for its gas chromatography products. I hired Nathaniel Brenner to work in that field. Incidentally, many years later, Brenner became the corporate planning vice president for Beckman Instruments.

BROCK: Why weren't Van Williams or Paul Wilks interested in the Model 21?

COATES: I believe in those days they thought that the future of IR spectrophotometry lay in the quantitative analysis of chemical components. I have kept in contact with Paul Wilks, and he remains interested in products for quantitative analysis with IR. Over the years, he's built a number of different instruments that perform useful measurements of components' concentrations in solution using IR-quantitative analysis.

There was a grand battle at Perkin-Elmer between those of us who thought the Model 21 was the right product at the right time, and those who disagreed. The Model 21 had found a niche market for chemical identification of substances.

In 1953, there was a respected professor at Columbia University named Ralph S. Halford, who had a graduate student named Abe Savitzky. Together, they had designed an attachment to the Model 12C that allowed it to produce percent-transmission spectra without background atmospheric absorption; in essence, making it operate like a double-beam spectrophotometer. They published a paper describing their modifications (5).

Their system required seemingly simple modifications of the Model 12C to incorporate a segmented-beam chopper after the IR-Globar source, and before the entrance slit. First, a 90-degree segment chopped the beam focused on the top half of the monochromator-entrance slit. Then, a second 90-degree segment compared the beam to the bottom of the entrance slit, with alternate blank-cutoffs in the rotating chopper blade. I was asked to duplicate their design, which I did. It depended on a precise, electronic-switching mechanism attached to the chopper's shaft. The mechanism was synchronized at exactly 90 degrees to sense the difference between the sample signal [slit top] and the reference signal [slit bottom]. The ratio of those signals was then plotted as percent-transmittance on a strip-chart recorder as the Littrow mirror was scanned by a motor. The existing liquid and gas cells could not be adapted easily to accept the unusual slit chopping of the beam. Nonetheless, Van Zandt Williams became very interested in the design, and around 1954, he hired Abe Savitzky to design it as a new product.

By 1954, I was still working alone in the engineering department, busily expanding the Model 21's design features. Others hadn't appreciated the numerous new capabilities I had added to the Model 21, but they had already decided that the design as conceived by White and Liston was insufficient. They did not realize that I had attacked the weaknesses of the White-

Liston concept and redesigned it to achieve a useful, new, and quite different Model 21 product with a range of sampling accessories.

They considered a program to adapt the Halford-Savitzky spectrophotometer to the Model 12C. They started the project by asking me to duplicate the Halford-Savitzky spectrophotometer as originally conceived. I accommodated them. It revealed the weakness of that approach in terms of the small sample cell's split design. Undaunted, Perkin-Elmer hired numerous engineers to design the system as a new double-beam-IR spectrophotometer called the Model 13. The model number was prophetic, because they had a lot of bad luck with the instrument. Savitsky's group worked on the instrument for two years before the prototype was completed. They designed a new front-end chopper to accommodate standard sample cells. They added many other features as well, and what had started as a simple upgrade of the Model 12C became an expensive and complex instrument.

Meanwhile, the sales of the Model 21 and its accessories were booming. It was a useful, acceptable system. Ironically, as I supported the Model 21 and the development of its accessories, Van Zandt Williams was pushing the Model 13 because he thought it would soon replace the Model 21. However, testing of the Model 13's basic performance revealed that it didn't perform up to the specifications that many customers needed. It had very complicated electronics, noisy spectral data, few accessories, and an inconvenient, non-linear chart. The Model 21, was very reliable, and therefore was always available for work. A Model 13 prototype was built and shown to customers, but it never went into production. When the tests were completed, Van Zandt Williams cancelled the project.

I became interested in the discontinued Model 13 about a year later. I thought that it might still be a useful tool. I thought of it as a universal, wide-spectral-range UV-to-IR [though, not only IR] spectrophotometer. My redesign of the Model 13 vindicated the excellent, wide-range design of Richard Kinnaird, and others from the 1940s. I used the quartz prism in it to run spectra of the UV-Vis-NIR [Ultraviolet-Visible-Near-infrared] portion of the spectrum; from 220 nanometers out to 2000 nanometers. Then, I substituted a NaCl prism to run a spectrum from 2 microns to 15 microns. Next, I installed a KBr [potassium bromide] prism to plot a spectrum out to 25 microns. As a result, it became the world's only double-beam spectrophotometer that could scan both UV and IR spectra. It took full advantage of the years of work on to the Model 12C's design. I made many particular design modifications, such as designing a way to flip the mirrors that put the UV source, tungsten-lamp visible-range source, and the IR source in-and-out. The various prisms and detectors available, like the photomultiplier, lead-sulfide cell, and standard thermocouple, also flipped in and out. The instrument took advantage of everything available. Later, the Model 85 IR microscope attachment was added to the Model 13, making it a universal system for micro or macro work.

BROCK: Did the instrument have three prisms in it, or did one have to swap them out?

COATES: It was designed to allow the easy removal and installation of all the prisms. It was not a linear wavelength, spectrum-presentation system; rather, it employed an arbitrary, though reproducible, spectral-chart scan, which was one of the biggest drawbacks of the Model 12C and the Model 13. I introduced the Model 13U several years after the Model 13's original development, and it worked out quite well. It didn't have the simple, standardized operation, or the numerous accessories of the Model 21, but the M13U Universal Spectrophotometer was quite useful for scientists working on semiconductors, electronic devices, new light sources, and other physics and optics programs. The Model 21 had been directed to customers doing IR studies within chemistry and chemical structures, so it wasn't used for physics and optics programs very often.

The Model 13U became quite popular for physicists in Europe first. It later became popular in the U.S. as our U.S. sales guys realized its value to particular customers. I think Perkin-Elmer sold over one hundred of the Model 13U spectrophotometers.

So that's the story of my ironic participation in the development of the Model 13U. I gained a lot of experience in UV spectrophotometry by doing work on the Model 13U, and by taking advantage of the work others had done on the unsuccessful Model 13. I learned about sources, detector characteristics, prisms, and all of the interesting work and results that are used in UV technology. I've drawn on much of that experience in my current company, Nanometrics [Inc.].

In 1951, several scientists asked Van Zandt Williams to design an IR microscope as an attachment to the Model 12C. He requested a design from Abe Offner. He was one of the optical designers at Perkin-Elmer who later became famous for his unique design of the Perkin-Elmer Micralign system for accurate, integrated-circuit lithography. Offner studied the IR microscope literature and developed an all-reflecting lens, optical-design attachment. Then, Van Williams informed him that I was assigned to do the mechanical and systems designing of the microscope. After he had finished the optical design, Offner gave me the proposed optical diagram.

When I received Offner's design, I found that it was quite complicated. A user was required to insert a mirror to catch the light coming through the final slit of the monochromator. The light reflected through a large microscope-condenser lens and then through a small sample holder. Next, the light was focused through an objective lens, and directed to some mirrors that sent it back to the original thermocouple detector installed on the Model 12C. Using one detector was intended to reduce manufacturing cost.

I designed my microscope-lens mounts using Abe Offner's original design, but, when I tested the prototype, I couldn't make the instrument offer a sufficient signal to be useful; I could only get about 5 percent of the original signal through the system, which wasn't enough to be used with tiny microscope samples. We were about to junk the project when I came up with another idea. I suggested that we simplify the optics by installing another detector in the microscope. I designed a second, high sensitivity, small-target detector in my prototype system, placed at the magnified image after the objective lens where the beam was focused to visualize the microscopic area. The instrument also had an eyepiece at that location. We had about 50 to 60 percent of the signal, which was useful. The prototype worked well with improved signal, and I recorded many spectra of various micro-samples using the microscope attachment on a Model 12C. This final concept of our joint design was given to George Brueske, who was a designer of astronomical telescopes for Perkin-Elmer. He had designed a number of the world's great telescopes using Perkin-Elmer's optics.

Brueske was asked to do the production design of the microscope system because he had created the original stylish designs of the parts for the Model 12. We knew it pretty well. He sent us some drawings, and then I built the Model 85 IR microscope attachment; the world's first commercially-designed microscope accessory for the IR spectrophotometer. Afterwards, I was hoping that my next job would be to build a microscope accessory for the double-beam Model 21 spectrophotometer, which many thought was impossible.

For the testing of the Model 85 microscope's final version, I worked with a new engineer, named Dr. Horace Seigler. He was an excellent physicist and he knew IR spectroscopy well. He came from Johns Hopkins University.

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COATES: Horace Seigler thought of building a micro-attachment for the Model 21. It would be used for very small samples and would reduce the sample beam to about 1 millimeter in diameter using a pair of ellipsoidal mirrors. That micro-attachment became the standard micro-sampling attachment for the Model 21, and it sold well, again extending its utility. I didn't consider it to be a true microscope because it didn't allow microscopic manipulation or viewing of specific sample areas. So, the Model 21 never had a "true" microscope attachment.

During the 1980s, some 27 years after I worked on the Model 85 IR microscope attachment [which Perkin-Elmer no longer made], I completed a new IR microscope design Its design led to the first complete IR spectrophotometer with diffraction-limited microscope samples. There were no others. It was a stand alone, complete IR microscope called the NanoSpec 20-IR, which Nanometrics sold successfully to both industrial and research labs. The instrument included a series of four matching and all-reflecting microscope lenses. One lens focused an IR source [the Nernst glower like the Model 21] onto a variable aperture, and then up to a matched-condenser lens that focused on the sample. There was also a matched, 15X-objective lens, and a final lens that focused on the tiny target of a LN [liquid nitrogen]-cooled, mercury-cadmium-telluride detector, which was sensitive from 2.5 to 14.5 microns. It was a chopped beam, low-noise system that obtained spectra from true, diffraction-limited samples, to less than 20 microns in diameter at the longest wavelength. The matched lenses conserved the signal, and the Nernst glower source was bright enough illuminate samples in the visible range, permitting a user to see them through a viewer eyepiece. It was an exact imager.

By the end of the 1950s, my redesigned Model 21 was more or less finished, including several of the prism-interchange units that I had designed. We had many accessories built for the Model 21 that were very useful for all areas of organic chemistry. The big market for IR was the identification of chemicals; the recording and comparison of an unknown material's spectra to determine its composition. The Model 21 was also quantitatively accurate in transmittance over a wide range of compositions, and was often used that way. Its main strength was its ability to take a large variety of chemicals and obtain their spectra, and then compare them. Scientists could compare them with spectra of known substances for identification. The filing of spectral catalogs of chemicals built up for a long period of time in laboratories all over the world. The Sadtler Research Labs [Inc.] collected many, reasonably related pure spectra in published books to aid in identification (6).

In the 1950s, a bright young man named Norman Colthup, who worked at American Cyanamid, compared and correlated the absorption spectra of certain chemical structures. For example, he compared the expected IR wavelengths of sulfonated or carbonated chemical groups, and so forth. He then created a special chart, known as the Colthup Chart, to use as a guide for classifying chemical structures. A scientist would then compare the spectral peaks of an unknown with those on the chart to determine what type of compound he was analyzing. The Colthup Chart was one of the important contributions of the 1950s for IR spectroscopists, and expanded versions are in wide use still today.

BROCK: Were you in charge of establishing the production and sales operations for the Model 21?

COATES: To a certain extent, yes. One of the things that bothered me about the Model 21 program was that Van Zandt Williams and Paul Wilks were not interested in it, even though we were receiving numerous requests for descriptive information on the instrument. Later, I got involved in helping sales. I contacted customers in the Eastern United States and agreed to run spectra for them. I also trained salesmen. My work helped push the Model 21 along. Richard Perkin was interested in astronomy, Van Zandt Williams was interested in the Model 12C; which is why he wanted the microscope accessory, and Paul Wilks was his salesman. In 1952, I was the only one in the company that was paying serious attention to the Model 21. We had a meeting with Richard Perkin, during which I said, "I'm not a salesman. I'm just a young, inexperienced engineer. But it seems to me that if we made them, we could sell at least five Model 21's a month, instead of just one. It seems to me the instrument could be very profitable." Perkin said, "Vince, I think you've got the right idea!" Then, he authorized the establishment of a production line, parts, floor space, et cetera, for the Model 21, and he ordered the purchase of necessary parts. Luckily, my work at Chance-Vought had taught me how to organize standardized production.

Next, I helped establish a sales organization that would make my sales estimate a reality. I trained a number of people and went from there. The transition in 1958 to the grating design was a big event that further increased the sales. The Model 21's many new accessories also increased sales to different scientific fields, such as air pollution, biology, biochemistry, general chemistry, pharmaceutical studies, and the petroleum industry. All areas of organic chemistry were of interest. From 1952 to 1990, I think we sold about seven or eight-thousand units. The Perkin-Elmer astronomy and optics division made some money, but they often did not estimate the costs very well. Perkin-Elmer was selling about one Model 12C a month, and in the early 1950's, Paul Wilks was very busy running that part of the business. Paul Wilks and Van Williams continued to market the Model 12C for quantitative measurements. They thought that was going to be the future of IR. To me, they had underestimated the customers' need to identify various chemical species and perform quantitative analyses.

From 1948 thru 1950, John White also asked me to redesign some of the other Perkin-Elmer products that had design flaws. One of the instruments was the M52 flame photometer, which had been sold to clinical labs to measure the sodium and potassium levels in blood. The instrument was not unreliable, so I redesigned its optical mounts to improve its stability. Perkin-Elmer sold the instrument for several years, until Beckman Instruments introduced a more precise and sensitive unit for the Model DU. Perkin-Elmer had introduced the Tiselius Electrophoresis System in 1948. It was a table-top instrument designed by John White that was mechanically unstable. White asked me to redesign the mechanics of that instrument as well. I did, and it seemed to work well afterwards. Those fields seemed to languish because there was no emphasis on customers. Regardless, after John White left, those products for biology were neglected and soon disappeared. Van Williams had no interest in pursuing them.

The work I did in mass spectrometry and NMR [Nuclear Magnetic Resonance] reveals a similar story. Perkin-Elmer designed a mass spectrometer around 1964, which died because it lacked marketing attention. I went to Japan to participate in a joint venture with Hitachi; to sell Hitachi instruments in the United States. From 1962 through 1969, I was heavily involved in that business with Hitachi, which included electron microscopes and mass spectrometers. I also worked with the joint company, Hitachi/Perkin-Elmer Limited [HIPE], and oversaw the design of a diffraction-grating based, UV table-top system made by Hitachi and designed to compete with the Beckman Model DU. The principle designer at HIPE was Dr. Koroku Nakamura, or "Roku-San," who was a close friend of mine and an expert in optics. He was a major designer of numerous optical instruments at Hitachi Naka Works during the 1960's. He and I worked together to apply the first Perkin-Elmer replicated, fine-line diffraction gratings to the HIPE-UV design. Called the Model 139, the very successful unit was first introduced in the United States in 1964. It was sold by Coleman Instruments, which was acquired by Perkin-Elmer as a Chicago subsidiary. Since the 1930's, Coleman Instruments had been a fierce competitor with Beckman Instruments , and the battle over UV-Vis spectrometers was a major one. From 1964 to 1970, Coleman sold over one-thousand Model 139's, considerably disrupting Beckman Instrument's traditional foothold in that market. Perkin-Elmer also found success with its UV-instrument sales in Europe.

The Hitachi mass spectrometer, which I first saw in 1961, was designed by a brilliant engineer at Hitachi named Dr. Tomatsu Noda. During the 1950's, most mass spectrometers in the United States had been manufactured by the Consolidated Electrodynamics Company [CEC], based in Pasadena, California. Their instrument's design had come out of the Manhattan Project. However, they had designed it to quantitatively analyze petroleum samples only. No one had thought much of applying mass spectrometry to the elucidation of chemical structures. After seeing Tom Noda's mass spectrometer, I obtained a written description of the Hitachi system. My job was to find a market for it. I knew that Consolidated Electrodynamics had 100 percent of the U.S. market, so it was going to be a tough road. [laughter]

Shortly thereafter, while studying the mass spectrometer field, I found a paper written by Professor Klaus Biemann, of MIT [Massachusetts Institute of Technology]. I went to visit him with a copy of the report from Hitachi. When I showed him Noda's mass spectrometer design, he nearly fell off his seat. He told me the Hitachi mass spectrometer was exactly what he needed. Further, he said it was superior to CEC's design. He pointed out that nobody in the U.S. was analyzing chemical structures with mass spectrometry. The resolution of CEC's mass spectrometer was blurry because it couldn't separate masses above 200 amu [atomic mass units]. Nobody cared about the resolution because the instrument was designed for the quantitative analysis of low-mass petrochemicals, like natural gas. Alternatively, Tom Noda designed his mass spectrometer to scan the magnetic field using a constant-accelerating voltage, which allowed the user to resolve beyond 1000 amu.

Biemann said, "This is the perfect machine for chemical structure elucidation because I could scan with unit-mass resolution past 1000 amu. When can I get one?" Surprised, I said, "I'll order one and bring it to Perkin-Elmer. Then, you can examine it and tell me if it's any good."

During that time period, I was also in charge of Hitachi's electron microscope program. Hence, I had the task of introducing both Hitachi electron microscopes and mass spectrometers to the U.S. When I brought the Hitachi mass spectrometer in to the U.S., I needed help from someone who had experience in the field. Therefore, I hired Dr. Al Struck, from American Cyanamid, because he had experience working with CEC's mass spectrometers, and he knew the field very well. While I was busy setting up the U.S. sales organization for the Hitachi electron microscopes [we eventually sold mass spectrometers from there], the Hitachi mass spectrometer that I had ordered from Japan arrived. Tom Noda came to Connecticut to set up the instrument, and he ended up staying for six months.

I remember the day Klaus Biemann was to test the instrument. After we had got the instrument running, I said, "It would be interesting to attach a gas chromatograph to the mass spectrometer, and then use it to identify the gases as the chromatograph separated them." They thought that was pretty good idea. Then, someone realized that the scan time of the mass spectrum, as set up by Tom Noda, was about 15 minutes per spectrum. I said, "That's pretty slow. These peaks are coming out of the gas chromatograph in only a few seconds." So we discarded the idea.

Several days later, Al Struck was scanning some standard compounds to identify the parameters of the Hitachi system. We'd bought the latest and greatest Honeywell fast-chart recorder, and I was there watching it work. I noticed the magnetic field would go up, and run quickly back down again, every time we ran a mass spectrum. When it came back down, it was ready to go again. I asked Struck, "How fast can we expand that chart?" He responded, "What do you want to do that for?" I replied, "Let's take a look at what happens when we scan backwards on this thing." Sure enough, we obtained nearly the same mass spectrum scanning backwards, quickly, as we did scanning forward, slowly. Tom Noda, being very conservative, had made the forward scan very slow to increase the instruments accuracy, but it took only two seconds to scan backwards. With that in mind, I hooked the gas chromatograph up to the machine's mass spectrometer and scanned each separated component backwards. As the resulting scan emerged from the gas chromatograph, it showed a mass spectrum of very low sample concentrations. Thus, we had made the world's first combination gas chromatograph/mass spectrometer [GC/MS]. Those instruments are now the standard analytical tools for analyzing complex mixtures and identifying each component. Further, the assembly of multiple in-line-separating systems with multiple in-line mass spectrometers is presently a proven method.

BROCK: What year did you do that?

COATES: It was in 1962. In 2003, two scientists won the Nobel Prize for mass spectrometry. One of them was John [B.] Fenn, from Yale [University]. I met him a couple months ago and told him about making the first mass spectrum from a gas chromatograph. He had later done the first mass spectra from liquid chromatographs. By that time, the GC/MS was being used worldwide, so he was interested that I had been the first to make one of those instruments work. I never published a paper or anything. I just showed it to Klaus Biemann when he visited us at the Perkin-Elmer Corporation; he had been very excited. Then we shipped that instrument to MIT. Later, Biemann refined the GC/MS and published some of the definitive papers (7).

Biemann's instrument was a one-off system. He published some papers of his work with the instrument (8), and soon after, Perkin-Elmer started selling GC/MS's. Within a couple years, we had 75 percent of the market. Further, CEC left the business because GC analysis of petroleum fractions was considered better than mass-spectrometer analysis alone. The Hitachi mass spectrometers were beautiful machines. Anything that came from Japan after World War II had been dubbed "Japanese junk," so we had a heck of a time getting people interested. However, by providing good service, we broke through.

During that time period, we brought in some electron microscope experts who were faithful users of Siemens's [AG] and Phillips's [Royal Philips Electronics] electron microscopes. After using Hitachi's instrument, they discovered its resolution of specimens' small features was two or three times superior to Siemens's and Philips's microscopes. Further, they got superior pictures of biological and other materials. Afterward, the experts published papers showing new images that created a great interest in the product. Three or four years later, RCA [Radio Corporation of America] gave up and left the electron-microscope business that they had started in the 1940s. Siemens also stopped selling electron microscopes in the mid 60's because Hitachi's microscope had superior optics that showed finer details. To this day, Hitachi is one of the world's largest producers of electron microscopes. I was told by the president of Hitachi that they invested heavily in scientific-instrument design to enhance their reputation in all areas.

BROCK: How did a deal between Hitachi and Perkin-Elmer materialize in 1961?

COATES: The general manager of Hitachi’s instrument-manufacturing plant, Dr. Isao Makino, went directly from Japan to Perkin-Elmer to visit Richard Perkin. He was a Japanese businessman who spoke practically no English, but liked direct action. Without making an appointment with Perkin, he simply showed up in the lobby one day to make a deal. [laughter] We found out who he was when one of our employees, I think, eventually went down to the lobby to chat with him. Dr. Makino’s appearance was interesting enough for Van Zandt Williams, Perkin-Elmer’s the senior vice president, so he invited Dr. Makino up to his office. They had a meeting, one thing led to another, and about a year later the contract was signed. Dr. Makino is one of the post-war heroes of Hitachi’s industrial growth. Almost single-handedly, Dr. Makino created their scientific-instruments business as a broad effort.

The president of Perkin-Elmer, Bob [Robert E.] Lewis, gave me the assignment of determining whether the Hitachi instrumentation was any good. To my surprise, I was impressed by a lot of things I found in Japan. They had been building copies of the Model 21 IR spectrophotometer that were dead on. They had copies of a number of other U.S. products, but their mass spectrometers and the electron microscopes were unique designs that were done by very clever researchers. Don’t let anybody tell you that the Japanese are not creative, because they are, as we know from their cars and so forth. From 1962 to 1970, I was put in charge of selling the major Japanese products throughout the world in a new organization that ran parallel to the Perkin-Elmer structure. It was an interesting job, and another aspect of my education in the instrument business.

It was around 1964, while I was working with Hitachi, that I first met Alfred [O. C.] Nier at the University of Minnesota. He was a scientist who had contributed greatly to the Manhattan Project during World War II. In 1964, he was researching mass spectrometry at the University of Minnesota. I had gone to visit him because I learned he had built a small, high-performance mass spectrometer that he used for his research. After my visit, I got Perkin-Elmer to license his instrument and hire him a consultant. Then, in 1965, Nier and others started a project to design the Perkin-Elmer small, double-focusing-mass spectrometer [Model 270] for chemical analysis. Though Perkin-Elmer's contract stipulated that they and Hitachi could trade products back and forth, Hitachi was threatened by the Model 270 project. As it turned out, they didn’t want Perkin-Elmer to develop its own mass spectrometers.

Our sales group sold the Hitachi mass spectrometers, and the Model 270 was just an additional product to sell. Interestingly, a Perkin-Elmer engineer working on the Model 270 project chose a vacuum-pump oil for the instrument, which, though efficient, superimposed a tremendous background of interfering peaks that hid the sample mass peaks users were trying to analyze. It was a design flaw that killed the Model 270. We just couldn’t get the engineer to change oils. [laughter] Again, the problem had been caused by internal politics and poor communication.

When I left Perkin-Elmer in 1969, their business relationship with Hitachi was falling apart. I think it finally died around 1972 because nobody at Perkin-Elmer was able to carry the torch for either the mass spectrometers or the electron microscopes. As a result, Hitachi no longer had a U.S. sales organization. Coleman's Model 139 UV Spectrometer sales had also decreased. In the 1980's, Hitachi increased its exports of electron microscopes again. By that time, JEOL [Japan Electron Optics Laboratory] had developed a competitive mass spectrometer, as had Shimadzu [Corporation], so Hitachi lost its U.S. business. Hitachi and Perkin-Elmer had held about 80 percent of the mass-spectrometer business in 1969; but 5 years later they had only about 5 percent. Similarly, they had controlled 75 percent of the electron-microscope business in the United States; whereas, 5 years later Hitachi controlled only about 10 percent. However, Hitachi's business finally came back in later years as they aggressively pursued semiconductor manufacturing and introduced new models.

In 1968, I left Connecticut to run the Hitachi's program and engage in new-product development in Northern California. I moved my family to California, as well. Perkin-Elmer had recently acquired a vacuum-pump company in Mountain View, California, called Ultek. It was thought that products employing vacuum systems could be important. I took the position at Perkin-Elmer-Ultek to concentrate on instrument sales and new designs.

BROCK: So you moved out to California to run the Hitachi relationship with Perkin-Elmer?

COATES: Yes. I was a vice president of HIPE, in charge of the electron-microscope products. My work for Perkin-Elmer in California is an interesting story. While I had been selling electron microscopes in Connecticut in mid-1960s, I visited the University of Chicago to meet Professor Albert Crew; a professor who had developed a new kind of electron microscope. Dr. Crew was a distinguished physicist and the head of the Argonne National Laboratory. He received research funds from the government, so as a side project, he built a new kind of electron microscope, which, as he told me, he had thought of on an airplane trip several years earlier. He thought his microscope could be better than existing electron microscopes in all aspects, and he already demonstrated a much better resolution that the previous scanning-electron microscopes [SEM] of that time period. He named it the field-emission SEM [Field Emission Scanning Electron Microscope, FESEM]. It became the design of choice for highest performance.

Dr. Crew was a charming guy, and we got along very well. He permitted Perkin-Elmer to license his design. I informed Chester [W.] Nimitz, Jr., president of Perkin-Elmer, of the new electron-microscope concept. He thought it was an interesting direction for the company to take. Hence, a major reason for my moving to California was to start the FESEM project at Ultek, and to finance it with electron-microscope sales.

In California, I hired an engineering team and we began to design a working model. I brought in Al Crew's chief engineer, Len Welter, and he became my chief engineer for the project. In 1969, after a brief development period, we built a working model and produced micrographs with it in record time.

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COATES: When Hitachi heard that we were working on the FESEM project, they notified Chester Nimitz [Jr.] that they would cancel their contract with Perkin-Elmer if we continued to design our own electron microscopes and mass spectrometers. As a result, Horace [G.] McDonell, the senior vice-president of Perkin-Elmer, came out to California and said to me, "Vince, we want you to go back to Norwalk. We're canceling our electron-microscope program and the mass-spectrometer program." I retorted, "You've got to be kidding!" He replied, "No, I'm not. The Hitachi business deal is very profitable, and we're going to stick with it." I said, "But I built the FESEM business up, and it offers new long-term directions for Perkin-Elmer." He responded, "I know, but we can't afford to lose Hitachi." So I said, "I quit." I left Perkin-Elmer at the end of 1969, went to my home in Los Altos, and started thinking about my next step. The HIPE joint venture died in 1972.

Len Welter came to see me and said, "We know how to build the new microscope now, and we know it's a great instrument. So let's start a company and build it." I thought it was a good idea, so that's how we started the Coates & Welter Instrument Corporation [CWIC]. In 1971, we designed and introduced the world's first commercial FESEM, operating with many new features. In addition, we patented several ideas that are now standard for all SEMs. One of the features, called the low-voltage SEM, allows users to take pictures without damaging the specimen by using very low accelerating voltage electrons. Nevertheless, the SEM takes excellent, high-magnification pictures. Specimens weren't damaged by such low voltages, so a user could measure structures and take inspection-pictures of integrated-circuits without damaging them. It became very useful in biology. Actually, I won an award from the Semiconductor Manufacturing Association for having invented part of the low-voltage SEM. When Nanometrics acquired CWIC in 1979, we redesigned the low-voltage SEM for semiconductor microscopy.

In any event, CWIC was a nice business but we soon ran short of money! [laughter] Then, the American Optical Corporation [AO] came along with a good offer for our business and bought us out completely. For several more years, I worked at building the business with AO, and when I left, Len Welter became president of the company. American Optical continued the Coates & Welter Instrument Company and its SEM program, which turned out to be quite a unique and special business.

In 1970, to appease Hitachi, Horace McDonell gave them lock, stock and barrel, the engineering design and prototype work that we had done at Ultek. Hitachi then proceeded to build low-voltage FESEMs, which became a billion-dollar business for them during the 1990's.

Nanometrics was too small, without enough engineers or money to compete. A huge company like Hitachi thus ended up making two-hundred-fifty-million dollars a year selling FESEM's to the semiconductor industry. Those microscope systems became one of the most important products for inspecting and measuring integrated circuits. Anyway, I was involved in that project and it was a really terrific experience. I'd got away from IR by that time. [laughter]

After CWIC was bought-out by American Optical, I became disenchanted with working for very conservative people. In 1975, I decided to start another company, which I named Nanometrics, Inc. Working alone at home, I sat down at my drawing board and spent a year designing what became the NanoSpec; a Spectrophotometer attached to a microscope. It has become quite a successful product. We've sold more than five-thousand units, about the same number of sales as the original Perkin-Elmer Model 21. I've had a very successful company, and I am proud that I designed it all from scratch. Along the way, I discovered that the NanoSpec has a lot of applications; such as measuring the film thickness of integrated circuits and flat-panel displays. It has some biological and crime-lab applications, also, but those have not been very big markets.

That's my career up to this point. I am still deeply involved with new concepts, new products, and product design; the lattermost being my greatest love and what I like to do more than anything. I should mention that I also designed an IR-spectrometer microscope at Nanometrics in 1980, called the NanoSpec 20-IR. I did so after I learned that Perkin-Elmer had stopped making my Model 85 and nobody else had one. It was Nanometrics first commercial-IR product. There was a hidden market for a microscope that could identify extremely small samples seen in a microscope. Later, a number of companies added IR-microscope attachments to FT-IR [Fourier Transform-IR]. So that's become kind of a standard product for the chemical industry today. I guess I led the way.

BROCK: You were the manager of Perkin-Elmer's applications-engineering department from 1955 to 1959. How did applications engineering fit into the entire Perkin-Elmer landscape? What was its relationship to marketing and product development?

COATES: Before 1955, I concentrated entirely on the Model 21 program and the development of its accessories and design. Then, when I got involved in helping sales, I had to setup a Model 21 demonstration lab that I could use to demonstrate the performance of our product to prospective customers. Some of our customers' samples were quite unusual, so the demonstration lab evolved in to a new, applications-research lab. Then, in 1953, Harry Hausdorff was hired to participate in the applications work and run the demonstration laboratory as its activity increased.

Harry Hausdorff was a very interesting person. Over many years, he and I became very good friends. I believe he came to us from the General Chemical Company in New Jersey. He was intrigued by one of our Model 21's at General Chemical, so he applied for, and was hired as, a demonstrator for Perkin-Elmer's instrumentation. He was Swiss but spoke quite good

English. In fact, he spoke five or six languages, so he became very useful with respect to our European sales. He increased Perkin-Elmer’s influence in Europe.

Richard Perkin was interested in traveling to Europe, and he invited Harry Hausdorff to go along with him on several of his trips. They made a number of trips to Europe on some of the big ocean-liners. Perkin became interested in acquiring a company in Germany that was making theodolites for testing NATO’s [North American Treaty Organization] rockets. In the United States, Perkin-Elmer had a major business selling telescope optics and related structures. Perkin thought that company was a good fit, and since he wanted to expand into Europe, he acquired the company. It was called Bodenseewerk Perkin-Elmer G. m. b. H. The factory was in southern Germany, in a resort area near Lake Constance [the Bodensee]. Perkin-Elmer’s American production group transferred all the drawings to Bodenseewerk so they could manufacture a duplicate of the Model 21. We had been selling maybe five or six per year in Europe before that time. After Bodenseewerk started manufacturing the Model 21, our sales increased to more than fifty instruments a year, just because it was backed up by Germans in Germany. Their sales effort later extended throughout Europe, with service offices and applications labs in each major country.

Later, Perkin-Elmer got involved with other methods of chemical analysis, including gas chromatography. In 1957, working again with Harry Hausdorff, Perkin decided to establish a manufacturing company in England, later called Perkin-Elmer, Ltd. The company was situated in Beckinsfield, north of London. Perkin decided that Perkin-Elmer, Ltd., should transfer the low cost IR spectrometer, the Model 137; they thought there would be a large market for it in England and in Europe. Harry Hausdorff was a principal person working with Richard Perkin on those projects, and he and his family eventually transferred to Switzerland. Harry became Perkin-Elmer’s European manager, working out of its office in Zurich, Switzerland. He established sales organizations in each country, which influenced researchers in those countries and increased Perkin-Elmer’s prestige. Incidentally, he later performed that same service by helping set up Nanometrics’s European sales offices.

Hausdorff was a good IR spectroscopist with whom I worked closely after he joined Perkin-Elmer. We collaborated on various aspects of IR that we identified as being important, and many turned out to be successful ideas. Often, he had an idea and I did the design. Examples were the variable-temperature kinetic studies, for which we presented joint papers on the completed studies at symposia. They gave the company good publicity. When he went to Europe, there was nobody there to run the applications-engineering lab, so I took it over. As head of the lab, one of my tasks was to design and build unique instruments for particular customer requests. Initially, many of the instruments were “special” engineering jobs, but after the instruments had been developed and other customers learned of them, the instruments became a part of the line of product offered by the Perkin-Elmer.

From 1953 onward, the Perkin-Elmer IR-spectrophotometer program for chemical-component identification was mainly in my hands. I was in charge of applications and special-engineering development, and I continued to design from 1953 until about 1960. One of the interesting things that I mentioned to you earlier was the conversion of the Model 13 prototype to the Model 13U. As a product, that was first a special-engineering job for particular customers. We also made other instruments for UV spectroscopy, and for measuring spectro-fluorescence. We were becoming more and more competitive with Beckman Instruments at that point.

Van Zandt Williams met a researcher named Alan Walsh, an Australian, at a Pittsburgh Conference back in the late 1950s, and he invited him to visit Perkin-Elmer to talk about his new research. The guy came in to give the engineering group a lecture about his work in atomic-absorption spectroscopy [AAS]. Everybody sort of listened and said "Why is that important?" [laughter] Back then, our engineers were doing IR spectroscopy; they weren't interested in measuring the chemical-emission spectroscopy of the elements, or any of that kind of stuff. I was very impressed by what he told us about measuring the concentrations of various elements in a flame.

I ran the applications lab and had freedom to do things, so out of curiosity I set up the Model 13 prototype to do AAS in the visible spectrum. It was the first time I had used the Model 13 as a visible-range system. I used two Beckman flame sources; one in place of the IR source, and the other in the sample space. The first beam was chopped and focused on to the second flame. Then, several calibrated water solutions containing particles of the specimen were sucked in to the second flame. I think I used solutions that contained magnesium samples in different concentrations. I saw the variation immediately. It measured the concentration of magnesium, by George! I did that experiment about a year after Alan Walsh had given us his lecture.

I visited with Van Zandt Williams and said, "Please take a look at this. AAS works! This is what Walsh talked about." He said, "I don't know if it's something we're really interested in." It was familiar response. I showed my results to other people in the company, and I wrote a report on it. I believe my encouragement pushed the company to approve a project for building some AAS systems. Walter Slavin was in charge of designing those. Within five years of its inception, Perkin-Elmer's AAS products were earning the company several-hundred-million dollars annually. Perkin-Elmer didn't pay much attention to AAS at first, but by showing that the technology was useful, I think I lit the flame, so to speak. It was something that I was proud of then, and now.

BROCK: Was Walter working for you at that time?

COATES: No. He had been working under the vice president of engineering. Before that job, he designed a diffraction-grating version of the Model 137, low-cost-IR spectrometer; it was another successful effort of his. He got involved with AAS afterwards, but you would have to talk to him about all those details; it's a very complicated story. He worked with Herb [Herbert] Kahn; an excellent engineer who was very hand-in-glove on many of the potential applications of atomic absorption. In the early 1960s, there was an enormous amount of work done in AAS by Walter and Kahn. They were excellent leaders and contributed to the growth of Perkin-Elmer during the 1960's and 1970's. They deserve a lot of credit.

BROCK: At this time, was there a distinct R&D [research and development] or new-products department?

COATES: The engineering department did the production design for new instruments; like the Model 137, AAS, and so forth. While in the applications-engineering department, I researched specific topics and then figured out the specifications for improved instruments that could fulfill the needs of particular customers.

BROCK: Describe your transition from the applications-engineering department to your next project.

COATES: After having worked in the applications-engineering department, I became the director of R&D [international operations division] for the European subsidiaries that Richard Perkin had established in the United Kingdom and Germany. I had to travel to Europe frequently for my new position, which was delightful. Once there, I assisted the engineers at our German and English factories with their preparations to manufacture the Model 21 and the Model 137. I also helped steer their efforts towards future involvement in gas chromatography. From 1959 to 1961, I traveled frequently to both Perkin-Elmer, Ltd., and to Bodenseewerk-Perkin-Elmer to work on those instruments; until those subsidiaries began making their own instruments. The position tapped my 12 years of experience in instrument design and planning.

As R&D director, I was asked to visit a company in Paris, France, called Jobin Yvon. They had noticed our activities in Germany and England, and were interested in having Perkin-Elmer as an investor in France [which Perkin-Elmer did eventually]. One of Perkin-Elmer's IR consultants in Paris, Professor M. LeCompte, informed me of new research in IR interferometry being conducted by several French researchers. Over the next few months, I visited people who were doing early FT-IR work, including [Pierre] Jacquinot, [Peter B.] Fellgett, in England, and others. Fellgett had calculated that FT-IR gave a major boost in performance because the interferometer scanned every wavelength, all the time, as opposed to slit-dispersive systems that examined one wavelength at a time. As a result, the signal to noise ratio was 60 times better in FT-IR instruments, which was unheard of in infrared at that time. However, the advantages of FT-IR weren't realized until the development of small, fast computers.

Although I tried as hard as I could, I couldn't get the engineers at Perkin-Elmer interested in the FT-IR work done by Fellgett and others. They had too much tied up in the design of dispersive products, which were Perkin-Elmer's bread and butter. They put their heads in the sand and didn't look up again until they had almost lost their IR business. Consequently, Perkin-Elmer let its chance to develop the first commercial-FT-IR instrument slip by. I had a good job as the R&D director of the international operations division, but nobody would listen to my ideas. It was around that time that I was sent to Japan.

In 1961, going to Japan was the last thing I had wanted to do. I had no interest in Japan. Once I got there, I was fascinated by their high-quality engineering projects. What an interesting country! [laughter] My involvement with Hitachi's program signaled an end to my work with the European programs; specifically, Bodenseewerk and Perkin-Elmer, Ltd. Their products had been transferred and put in production, they had good people in place, and they were busy with their own work.

BROCK: In coming out to California initially, still working with Perkin-Elmer, and then starting a company of your own; it seems like you were moving from a quintessentially East Coast company, in terms of corporate culture.
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COATES: By that time period, Perkin-Elmer had become a large and complex corporation with numerous divisions of people. They had started a large semiconductor division, in addition to their various telescopes and optical products, their government contracts, and their analytical-instrument division. Also, Chester Nimitz had just appointed Horace McDonell as the senior vice-president of Perkin-Elmer; so a lot of things were happening at the company. In fact, the company became so large that it was hard to keep track of what was happening in the other divisions. Basically, each manager focused on the little area he was working in.

BROCK: Describe the difference between the business climate in Connecticut and the business climate in California.

COATES: Perkin-Elmer had become very large with headquarters in Connecticut. They had numerous sales offices throughout North America, and other parts of the world, including a good business relationship with Hitachi. Further, they had their Bodenseewerk and Perkin Elmer, Ltd. factories, and the European sales organizations established by Harry Hausdorff. Furthermore, they were selling over four-hundred-million dollars a year in products. I was still involved with other products, but I had a lot more to pay attention to once I got involved in the Hitachi program. I had already been to California many times, and I liked the idea of moving there because it seemed like a place where I'd like to make my home. So that's what I did. When they acquired the Ultek Company in Mountain View, which was a small company, it sounded like a nice atmosphere; a small but expanding company where I could get involved with electron microscopes and other new and exciting areas.

BROCK: What was Ultek producing at that time?

COATES: They were producing high-vacuum ion pumps; a growing business. Ion pumps can actually produce a vacuum way below the norm; the ultimate vacuum, 10^{-11} torr, which made them very special for new research. So their principal work was selling those pumps. They did not have an analytical instrument business. I brought the idea of an instrument business with me when I moved to California. I figured Ultek could sell electron microscopes, and perhaps design other instruments. I met Al Crew and got involved with the FESEM around that time. The place to design and build it was at Ultek because they had the high-vacuum pumps that were needed for those products.

BROCK: When you were starting your new company, what lessons had you learned at Perkin-Elmer that you wanted to repeat or avoid in your new venture?

COATES: Perkin-Elmer had become so complicated that it was a relief to have a single product with only a few people working on it; to concentrate on making that one product successful. That's basically it. As you know, I was an engineer who had designed and been involved with special engineering and instrument designing. I had been involved with numerous customers and participated in conferences, like the Pittsburgh Conference. Further, I had traveled and worked with sales people in the United States and Europe while I was in the international operations division. Hence, all of that previous experience made it easier for me to start a company and operate it successfully.

BROCK: What was your financial strategy for setting up your business?

COATES: I had saved some money from working at Perkin-Elmer, as well as some stock options that I cashed in, so I financed the early business myself. Len Welter did not have any money, but he had learned all about field-emission electron microscopy at the University of Chicago. As a result, we worked very well together in developing the field-emission gun itself. I loved the time I spent working with Welter. Designing on the draft board when I was younger gave me a valuable level of engineering skill for work with Welter, and made it fun to contribute my own ideas. I especially enjoyed designing the specimen chamber and the overall systems assembly.

We sold our first product to a company, called Material Analysis Corporation [MAC]. They had developed an SEM system and wanted to add our field-emission gun to the instrument. It was an order of ten to fifteen guns, which helped finance our company for the first year. When we got their order, Welter and I adapted our field-emission gun's design to complete an entire SEM system of our own, which resulted in a unique product. By the time we were finished, we were out of money, and looking for orders and financial backers. Then, American Optical saw a demonstration of our product at a show and became extremely interested. They had been selling optical microscopes for years and considered involvement in SEM sales as a new direction for their company. They bought us out and gave us decent royalties. In addition, they gave us funds to proceed with the design, introduce the product, and so forth. We expected them to back our future endeavors. Unfortunately, they had problems with that because of internal problems with their other businesses.

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COATES: At Perkin-Elmer, and later at American Optical, I recognized that their management styles were arch-conservative. They were literally afraid to try anything new. It was a miracle that they had convinced themselves to buy CWIC. We had complicated electronics, a super-vacuum system that was technologically advanced, and complex product; it certainly was much more complicated than making eyeglasses, optical microscopes, and so forth. So they fell back. As did their optical microscope business.

American Optical showed its conservative nature whenever I proposed undertaking innovative, advanced projects. They almost always turned my ideas down, especially if they involved any serious investment. Eastern business' conservatism, during those days, was deadly. It was so prevalent, even with the emergence of Silicon Valley and all of the hot ideas. Those wonderful new ideas! It was such a revelation to me, because that was how Perkin-Elmer had originally run their business. I suddenly realized that California, the place were most of the new technology was growing, was the place to make an investment. Nanometrics is a good example of such an investment. My company could never have been successful back East.

BROCK: Why not?

COATES: When I first started Nanometrics and designed the first NanoSpecs, I invited American Optical to help sell the products for me. They sent their sales manager, a friend of mine, from Buffalo, New York, to inspect my design. He said, "Vince, what did you design that for? There's no market for that!" I said, "What do you mean there's no market? It's original and unique; therefore you have to develop a market." He said, "I see. We're busy selling optical microscopes and we can't be bothered with the development of a new market." His response was typical of most eastern businesses. Of course, Bell Labs, IBM [International Business Machines], and some other companies are exceptions. Much of the electronics innovation and investment has been done in the West by companies like Intel [Corporation], National Semiconductor, and the rest. Lately, most of the contemporary-electronics innovation and investment has moved from California to Asia, though the recent SARS [Severe Acute Respiratory Syndrome] epidemic may slow that transition. The technological effort has taken root in China, but whether it'll continue to grow is still up in the air; there's so much going on. There are so many challenges that I wonder what's going to happen to the electronics industry in Asia. The United States seems to be less innovative than it once was. Even California seems to have become wary and slow down.

BROCK: How did the semiconductor-metrology market emerge as your focus?

COATES: After leaving the CWIC in mid-1974, and before starting Nanometrics, Inc., I spent November and December of 1974 reviewing the scientific literature on the latest news in optics and scientific technology. Each day I studied a number of journals available at Stanford University's library. Lasers had been introduced during that time, as well as small, compact spectrophotometers using replica diffraction-gratings to cover the UV and visible-spectral regions. The combination of lasers and spectrometry were reminiscent of work I had done in the late 1950s at Perkin-Elmer, when we built what I believe was the first Raman spectrometer with a paper-chart recorder.

In 1955, that instrument was a new design to replace the photographic method of Raman spectra. Perkin-Elmer's Raman system had been purchased for a university research laboratory as an advanced capability that was unique to the field. We used the Perkin-Elmer Model 12C platform as the basic monochromator system. It was modified with a quartz prism, high-sensitivity, light-gathering technology, and high spectral resolution. We equipped it with the latest, highly-sensitive photomultiplier detector for detection of the Raman-scattered light. A cylindrical-glass coil, high-intensity mercury lamp surrounded the sample cuvette, which was contained in a 10 cc [cubic centimeter] glass test tube. A cylindrical-glass filter placed inside the lamp coil and before the sample tube removed the interference from the UV and blue light caused by the mercury lamp's emissions. It allowed the green line of mercury, at 546 nanometers, to be transmitted to the sample as the excitation line. The green excitation line then directed the sample's scattered Raman light to a focusing mirror. Next, the Raman light was focused on the slit of the monochromator, which was equipped with a large, 60-degree-quartz prism. The radiation was scanned over the wavelength range by a motor-driven Littrow mirror, focused by an off-axis parabolic lens, and passed through the exit slit. Finally, the monochromator light was focused to fill the PMT [position mode tracking] target, generating a signal that was amplified and recorded as a spectrum on a chart-paper recorder. The spectrum was recorded as wavelength position versus Raman intensity.

My fellow workers and I were excited when we saw the first Raman spectrum recorded on paper. We clearly resolved peaks located at the known Raman frequencies from a carbon tetrachloride test sample. With other experiments, we learned that samples fluoresced, especially water solutions. Their fluorescence completely obscured the Raman lines. Fluorescence was a major limitation of the method. Only liquids in solution were sampled. Solid samples could not be analyzed with this system and gas samples could not be measured because the lines they produced were too weak. Nevertheless, we sold a small number of those early Raman-recording systems for Perkin-Elmer. Five years after our work in Raman, Howard Cary of the Cary Instrument Corporation designed and built an excellent, high-performance-Raman spectrophotometer that used laser excitation of Raman lines. I admired his well-executed design after the marginal performance of Perkin-Elmer's early Raman system.

While studying advances in optics at the Stanford library in 1974, it occurred to me that biologists might need to have a microscope-based, Raman-spectrometer system. I thought Raman spectra from microscope-defined areas might permit the identification of unknown chemical substances. Long wavelength, IR microspectrophotometers, such as the Model 85, could never measure small samples. I thought it would be possible to reflect scattered Raman light from the specimen back up the microscope tube. Exciting the tiny samples in the microscope might be done with an intense beam from a helium-neon 632 nm [nanometer] laser. A high-resolution monochromator could focus the sample at high magnification, and a sensitive-photomultiplier detector could measure the Raman-emission lines.

In January of 1975, I established Nanometrics as a corporation and decided to start the design of the microscope-based, Raman-spectrophotometer system. Working alone, it took me six months to complete the instrument's design. I designed the spectrophotometer head to plug in to the camera-port of a standard optical microscope. The instrument employed state-of-the-art lasers, diffraction gratings, red-sensitive photomultipliers, which were new at that time, and highly accurate, digital electronics. I decided to attach my compact-spectrophotometer head to an American Optical Model 10 microscope, which seemed well suited for this work. I built four of those units, and called it the NanoSpec 10 microscope attachment. I purchased the helium-neon laser and the AO Model 10 microscope commercially, and then I put together a completed system with my spectrophotometer head.

It was exciting to place the first test sample on the microscope stage to record my first micro-Raman spectra. However, after several frustrating weeks of testing, I couldn't get any Raman bands to appear. I discovered later that the resolution of the grating monochromator I designed was inadequate to resolve the narrow Raman bands. Apparently, they were being lost in the noise of the system, if they were there at all. This illustrates that good intentions often end in failure. It was one of my many failures.

After that disappointment, it was necessary to take Nanometrics in a new direction. It occurred to me that the high-performance microscope-spectrophotometer system I had designed might be ideal for the measurement of weak fluorescence from microscope samples. Therefore, I changed my goal to the fluorescence-measuring mode without any need to change the existing design. I had made it sensitive enough to easily measure the fluorescence spectra radiating from biological samples at specific wavelengths. The system could also measure transmitted-light absorption from UV and near-IR samples at various wavelengths. This design included a small, versatile, personal computer [PC]. It was an early personal computer based on the early, Intel 8080 chip. It stored data as a background reference in its memory, which could be subtracted by the computer to reveal clean spectra over the wavelength range, from 200 to 1200 nm.

Prof. Michael Bernes, a cell-biology researcher at U. C. Irvine [University of California at Irvine], became interested in my fluorescence-spectra measurement instrument soon after its development. He wanted to measure the changing fluorescence of cancerous-tissue cells undergoing mitosis, as seen at high magnification under the microscope. He prepared stained sections of tumor cells, which could be studied using my instrument. We recorded fluorescence spectra that compared both cancer and normal cells. We published the results, which showed remarkable differences, in 1976 (9).

I designed the product to measure biological samples that had been tagged with fluorescence dyes to identify cancer. I thought that my spectrophotometer would be useful in measuring the emissions from those samples because it enabled a user to identify the composition of samples that he or she saw through the microscope.

I rented a booth to show my new product at a meeting of about five to ten-thousand cell biologists in Boston. My salesman for the Eastern United States met me in Boston, and we set up the NanoSpec 10 microscope-spectrometer in the booth. We waited, and people kept walking by, but nobody ever came in. I actually tried to grab people and bring them in, but there was nobody who seemed interested in our fluorescence system. Twenty-five years later, fluorescence is an important process for genomic studies and many other areas of biological research.

As we were sitting, discouraged, in the sales booth, my New Jersey salesman said, “I was talking about your product to a guy down at Bell Labs and he told me it sounded interesting.” I said, “Really? I didn’t know that Bell Labs was doing a lot of biological stuff.” He said, “No. He’s working on something called integrated circuits, and he thought it could be used to measure the film thickness of tiny areas on them.” I said, “We can do that!” I immediately packed our equipment back in its boxes and left the show. Two hours later, we drove down to Bell Labs in New Jersey.

As I was setting up our equipment in his lab, my sales guy went and found our interested customer. He put a patterned-semiconductor wafer on the stage. I focused on an area, pushed the scan button, and recorded some spectra for him right on the spot. That’s the story. I had never intended to move Nanometrics in to that line of work. We ended up measuring integrated-circuit films as our main business, purely by happenstance. The first rule: Recognize an opportunity when it comes knocking at your door!

BROCK: Had you become acquainted with cell biology through your electron-microscopy work?

COATES: To a certain extent. I thought there would be future interest in it, and there certainly is today. The current focus in cell biology is on tagging cells with fluorescence. For instance, a researcher who just identified SARS, Joe [Joseph] DeRisi, at University of California-San Francisco, used that technology. He put 12,000 little viruses in an array, each in its slot on a microscope slide, and then he put a SARS virus coating on the slide. A little fluorescent spot lit-up at a certain position in his microscope’s field of view, identifying the SARS virus. Today, he’s like a rock star in biology. He has got a lot of publicity. [laughter] I was only 25 years ahead with my idea for microscope spectroscopy of biological materials. [laughter] It has turned out to be a good idea!

BROCK: How did the scientist at Bell Labs help to change your company's direction?

COATES: He described his need to measure film thickness in a small area in production. He recommended that we modify our computer so it would print the answers. So, I went back to my shop in California and modified the design. Before I got that instrument to him, Intel wanted one, and then National Semiconductor. Before I knew it, I had fifteen to twenty orders for the NanoSpec Microspot Film-Thickness Measurement Systems. I suddenly and almost accidentally had a successful business. So my advice to all startups is: Follow your nose, but keep your eyes and ears open, too.

BROCK: How did other integrated-circuit manufacturers learn of you product?

COATES: Once we had learned of the instrument's need, we went around to the microchip manufacturers in Silicon Valley and told them of our product. By then, we knew that there was a common application for it in the semiconductor industry. Therefore, we simply asked manufacturers if they were interested in measuring films in very small areas. They would say, "Of course we are. Do you know of a way of doing it?" The industry had been using a gross, large-spot test, but they couldn't find a small test area on a chip, and measure that test spot repetitively during production. Such measurements became essential for quality control in the semiconductor industry, and with a big payoff. That was the start of our thirty years of continued success in microchip measurement, which has extended to flat-panel displays, and other nanotechnology devices.

BROCK: And how did you acquire the technology to combine the hardware and software with the instruments you were producing at Nanometrics?

COATES: In 1975, when I first designed that product, the personal computer did not yet exist. Next door to Nanometrics, in Sunnyvale, where I was building my products, there was a company that was building some of the first compact computers. I happened to talk to him and went to his small shop, and he showed me his interesting design. I consulted with a software engineer, Dr. Dennis Paul, who worked at Cary Instruments for a time after graduating from Caltech, and together using that computer, we did the software programming for films. Our customers wanted to have their data printed-out, and we gave operating directions to each user for each measurement. We had to write clever software to make our instrument work. The guy next door built the early computer boxes for our instruments, and we supplied the programs that contained the real know-how. It was very early computer work with a PC-type system.

Several years later, of course, we could buy general-purpose PCs from many companies. In fact, Intel perfected the programmable-memory chips we had been using in our computers during that time. Later, we designed a number of other measurement tools for semiconductor process-control, including tools for measuring the gate widths of transistors, and measuring the overlay registration of superimposed layers on a chip.

BROCK: Over time, how closely connected was your emerging semiconductor-metrology business with the fate of the U.S. semiconductor industry? Did your business match their success cycles?

COATES: Yes, almost exactly. We grew like mad as the semiconductor business grew, and became a large, profitable company, with many customers worldwide. We've sold about five thousand Microspot Film-Thickness-Measurement Systems in 30 years, in addition to other products that we added along the way. It should be realized that our film thickness measurements on silicon wafers were based on a combination of isolating a Microspot, reflection interferometry, scanning UV-Visible spectroscopy, and computer calculations.

In 1979, the CWIC, the subsidiary of American Optical Company started by Len Welter and me, was losing business as a result of heavy Japanese competition, and weakened support from American Optical. AO was also losing business to competitors in other key aspects of their business. Their microscope business, which had its origins in Buffalo, New York, since 1850, was succumbing to Japanese microscope makers as well.

Because of Nanometrics' growth in semiconductor metrology, it seemed logical for me to acquire the complete assets of CWIC, and add their SEM knowledge to our products. The high-magnification images that their SEM's produced were ideal for the measurement of microcircuit features designed by semiconductor manufacturers. No one had yet recognized this need.

The CWIC's FESEMs were based on Len Welter's and my patents, some of which related to a unique ability to produce high-magnification images at low-electron voltages. I had worked with the U.S. Defense Department in the early 1970s at CWIC to determine if this low-voltage operation could be used to inspect integrated circuits without damaging them. The older SEM's used destructive, high-voltage electrons with energies above 20,000 volts. We found that FESEM electrons below 1,000 volts would not damage the circuits; it was a unique capability.

In the early 1980s, after we acquired the assets of CWIC from AO, we started the design of a low-voltage-electron microscope, which, for the first time, would be used to measure and inspect the smallest areas on integrated circuits without damaging them. In 1984 and 1985, we completed several models of the FESEM's for use in laboratories.

In 1986, Nanometrics' new product was the CD-SEM [critical dimension scanning electron microscope]. It was an SEM designed for automated measurement and inspection of the latest integrated circuits. The instrument was to measure silicon wafers at low voltages during manufacture. During the next few years, we sold and installed about fifty CD-SEMs to semiconductor companies around the world. Those companies included IBM, Intel, AT&T-Bell Laboratories, and various Japanese and Korean semiconductor companies. The ability of our CD-SEM to measure and inspect large-diameter silicon wafers non-destructively was a major breakthrough in semiconductor metrology.

The CD-SEM was the most complicated product that we had designed during that time period, and the development of the instrument pushed our engineering and manufacturing skills to the limit. In 1988, Hitachi, Ltd., of Japan, with whom I had worked in the 1960s, followed our lead, introducing a competitive CD-SEM. Incidentally, Hitachi's CD-SEM was based on information regarding field-emission technology that Len Welter and I had developed, which Perkin-Elmer had given to Hitachi for free. By 1986, Hitachi had employed a much larger, more experienced engineering group than ours, but their instrument was basically a copy of our CD-SEM. We eventually sued them, and later we sold them a license. By 1990, however, our CD-SEM business was overwhelmed by Hitachi's size and money.

Over the years, Nanometrics continued to develop a variety of advanced optical, microscope-based measurement and inspection systems. Today, our latest measuring systems are designed to be built right in to integrated-circuit manufacturing equipment for continuous quality control of lithography, etching of circuit elements, coating of films, and so forth. Our sales continue to grow as our engineers develop and design new products. I continue to participate, but our new generation of designers has taken over the task of designing, measuring, and inspecting systems for the more compact and highly-integrated circuits that are produced in all parts of the world.

As I mentioned, Silicon Valley has become quieter these days. There aren't very many new companies coming in and making integrated circuits. Taiwan, Singapore, Japan, China, and Korea are all experiencing growth. In fact, as you drove in to our parking lot, you may have noticed there are a number of empty buildings around us. That's just happened in the last couple of years. I don't know where they've gone to, but they're gone. [laughter] So it's an interesting illustration of what‘s happening to industry in general, and to our industry in particular. Our nation seems to have become service-oriented; importing products from China and other places, and then having people sell them in the United States. Nanometrics has a unique business selling our products in Japan, Korea, Taiwan, Singapore, and China. The largest part of Nanometrics' present business is in Asia.

BROCK: Are you planning to establish a foreign subsidiary when your Chinese markets grow large enough?

COATES: We will establish a service company in China with service engineers, and parts available, so that we could quickly service our products when we deliver them to that country. Then, as time passes, we may find that our profits would increase if we transferred our manufacturing facilities. For instance, our Korean customers want to have a local manufacturer, which would help our business as well. We've already built manufacturing facilities in Japan and Korea. We haven't built them in Taiwan yet, though we have a very sizable service and sales organization there.

BROCK: Do you have a subsidiary in Europe?

COATES: The European semiconductor market has been modest. Some of the larger companies such as Siemens's and Phillips have participated in it. There haven't been an awful lot of other companies that have expanded there. Though the European market is somewhat weak, Europeans are very brand loyal, choosing to buy from Siemen's and Phillips to preserve jobs in the European market.

BROCK: Do you enjoy working for yourself at Nanometrics more than working for others?

COATES: Yes. I enjoy making decisions and directing my own company. At Perkin-Elmer, I had been able to make my own decisions because nobody was paying attention to my work. For my first four or five years at Perkin-Elmer, I made all of my own decisions. For instance, I taught the manufacturing department how to produce my product, I ordered my own parts, and I worked with the sales people occasionally. Then, as the business grew and became very profitable, many executives became interested in my work and I wasn't allowed to make as many decisions. It was very difficult to go from being in charge to needing someone else's approval to make a business decision. Hence, the loss of the fast action to make my own decisions became a contentious point between the company and me. At Nanometrics, I've tried to avoid taking away my employees' freedom of action. They have the freedom to do stuff on their own, without having others to second-guess them. If they have a good idea, they're permitted to make that idea a reality. I think that style of business has been very successful for us. It is characteristic of Silicon Valley.

BROCK: Did you lose some of your decision-making freedom when you incorporated your business?

COATES: No. It had no effect at all. People who've bought our public stock have never bothered us very much; they've just enjoyed the profits! We received a lot of money from common stock sales. For a long time, I retained majority ownership, so I had the ability to make decisions. [laughter] As long as the company was making money they were happy!

Much of the analytical-instrument business seems to have become rolled into the bio-instrument business. There are many new methods that have been developed for that field of instrumentation. I don't even know what the market is for IR spectrometers anymore, or how many chemical companies buy them. It seems that many FT-IR companies have recently faded from the scene. I think the original Perkin-Elmer group is still doing quite well. I don't know what's going to happen, but it looks like its getting folded in with a lot of other stuff. It's no longer a business unto itself, with ideas of its own. The gas chromatography thing came along, UV spectroscopy, IR spectroscopy, mass spectrometry, and liquid chromatography. Those techniques have all been plugged in to other total systems. For instance, Applied BioSystems builds omnibus products that are valuable for the biological field.

Over the years, Paul Wilks has done a wonderful job of finding niches for his innovative designs. I've always admired what he's done with his companies. He's done some remarkable new things with IR in particular, and he's found some sizeable markets, such as measuring carbon dioxide in Coke. [laughter] He's been very successful.

BROCK: How has the rapid evolution of the semiconductor industry affected your product development in the metrology sector?

COATES: Our pace has quickened. Our designs have become increasingly complex to measure the many new materials and complex circuit designs. The semiconductor industry has continued to evolve into high-performance products that have increased in complexity while decreasing in size. As a result, the ability to make precise measurements has become a greater necessity for the semiconductor industry. By continuing to push forward, advance, and introduce high-tech products, Nanometrics has continued to prosper.

BROCK: Please describe your life outside your business.

COATES: For the last four or five years, I have become more interested in the way the human brain works. I have provided some philanthropic investments to researchers who are doing important work in that field; people who do research in Alzheimer's disease, Parkinson's disease, and so forth. I've always been very interested in the chemical nature of the brain. My curiosity about the brain has grown out of my work with analytical instruments. To satisfy that interest, my Foundation has donated some sizeable research funds to laboratories at Stanford, UC Berkeley [University of California at Berkeley], University of California at San Francisco, Yale, Harvard [University], UCLA [University of California Los Angeles], the Salk Institute, and others. Further, I have funded mass-spectrometer labs at Stanford, the University of California-San Francisco, and the Salk Institute. I've also provided financial assistance to a number of individuals working in those fields.

I search for researchers who need funds to do their work, and I usually like to find guys that are in their late twenties or early thirties because I believe that's where the breakthroughs come from. I provide them with funds to do research, buy equipment, travel to expand their knowledge, whatever. There's a Professor at Yale University named Steve Strittmatter, who is researching why nerve damage in the central nervous system causes people to become paraplegic. He's done research on why nerves don't grow back after being damaged. He's on the verge of announcing a vaccine that will regenerate those damaged nerves. It could save millions of poor souls. I consider those donations essential.

There's a researcher at Harvard that I've funded as a full professor named Dennis Selkoe. He is developing a vaccine for Alzheimer's, and I've financed some of his work for the last few years. As we get into our sixties and seventies, Alzheimer's becomes a major thing. It's going to become about five times more serious a problem for the world than it is at the moment, even though it's very serious right now. Around the year 2010 and thereafter, there will not be enough medical people to take care of the people who have Alzheimer's. Unless a vaccine is developed, that is going to be a major economic catastrophe for the world, just as AIDS [Acquired Immune Deficiency Syndrome] is. This is why I'm interested funding the right researchers.

BROCK: Do these researchers have a great demand for equipment, like analytical instruments?

COATES: Yes. They usually request fancy optical microscopes to enhance their specimen viewing. Some researchers request mass spectrometers to measure protein chemistry and determine its composition and structure.

I have a recent report that came in from Prof. Joe DeRisi at the University of California-San Francisco. It includes a picture of the microscope-slide array he's built in his lab. I went down and visited him last week. He's only 30-years old and he's come up with a blockbuster. His simple device can identify thousands of different types of viruses. They call him the "rock star of Biology." [laughter] So that's good stuff. Those are the things that I'm working on, besides continuing to participate in Nanometrics. If I can take some of my financial gains and put them into areas that prove useful to humankind, that's what I like to do. My business life at Nanometrics is very fulfilling.

As far as philanthropy is concerned, I 'm not interested in bricks and mortar. I'm interested in people working at the leading edge of their research; people who might be able to make advances in research if they had some more funding. There are young kids, undergrads, who need just five-thousand dollars to present a paper at a meeting. Much of my philanthropy goes to those kinds of people. There's a lot going on in bricks and mortar. There are a lot of philanthropists in the Bay Area who donate money for buildings that house important research; but they don't know what's going on inside those buildings. They have no idea. I'm interested in the guy in the lab who's looking through the microscope and seeing something new. [laughter]

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RICHARD ULRYCH: Do you think Nimitz was a positive influence as president of Perkin-Elmer?

COATES: Yes. He made things happen. He worked well with the engineers and responded well to good ideas. In my experience, Horace McDonell was not that way, but the president before Chester, Bob [Robert E.] Lewis, was never afraid of tackling new projects. Though Richard Perkin was not very interested in the analytical business, he contributed to its growth. He was consistently interested in astronomical telescopes, and government business as well. He focused mainly on special optics, bombsights, and so forth, but he also supported the analytical business. In Perkin-Elmer's early days, Perkin allowed Van Zandt Williams to basically run the business; and Williams had a great staff that helped to advance the company.

I believe Williams resigned in the early 1960s. He took a job as the president of the American Physical Society, which he enjoyed because he was in the company of his fellow physicists. He traveled around the world, and had many contacts in England and Europe. He was a charming guy, had a great sense of humor, and he was well thought of by everybody who knew him. He contributed greatly to the IR field, and was instrumental in associating it with the chemical industry. The more he talked, the more attention the researchers paid to his advice, and it became pretty usual for them to set up infrared-spectroscopy labs based on his advice. He died suddenly in London, in 1962.

During our initial discussion in meetings about small IR instruments, Paul Wilks had proposed, I suppose from his point of view, the design of a low cost version of the single-beam Model 12. I said such an instrument would never have been of much interest to a bench chemist. I did a cost study of a small IR version of the Model 21, which had long been proven reliable, and I proposed a cost reduced, miniature version of the Model 21. I suggested a simple design; it had a cam drive on the same shaft as a vertical-chart recorder, similar to the Model 21. I believe my concept for that instrument, which became the Model 137 Infracord, was the low-cost breakthrough. Perkin-Elmer sold thousands of those instruments after they were introduced in 1962.

It has been often said that "failure is an orphan, but success has a thousand fathers." Many people enthusiastically contributed good ideas to the Infracord. Dr. Hamilton Marshall was the key guy as project engineer, with very imaginative ideas for a new kind of lower-cost manufacturing that affected Perkin-Elmer's design for many years, and produced profit.

There weren't that many chemists who wanted to do their own infrared analysis. The same problem has arisen in mass spectrometry right now. There are many mass-spectrometer labs, I financed the one at Stanford, with brilliant mass spectrometrists, but there are several million proteins that have yet to be identified in the human brain. There's no way that biologists can devote their time to learn how to run mass spectrometers and do their main work. What the field needs is a cost-effective, fast, mass-spectrometer system that analyzes the protein for a researcher, and quickly prints out a possible structure in a form that it easy to understand. Fast but precise mass spectrometers; fast computers; that is the direction.

There's a research Professor named Al Burlingame at University of California-San Francisco who's building a high-speed computer system for mass-spectrometer analysis. I've financed some of his work in that area. The same thing is true of the mass-spectrometer lab at Stanford. Researchers are just becoming aware of how important it is to identify the proteins. There are millions of proteins in the brain that have yet to be identified in function and form. The human genome project is interesting, but only a beginning. It is important to identify the proteins and figure out what they do. A good example is Mad Cow disease. Mad Cow disease is caused by a prion; a mutated protein that causes the other proteins in the brain to mimic its shape, rendering them useless. It's important to realize that viruses, bacteria, and parasites aren't the only dangers to humans; some proteins are dangerous too, and they must be studied.

Mass spectrometry may be the only technique that is sensitive enough to structurally-analyze proteins. Much of Klaus Biemann's early work plays in to that field. It involved the interpretation of mass spectra and the concept of deciphering a definite structure that may only result from a particular group of masses. His concepts have advanced rapidly since their inception, and now the field of proteomics relies heavily on this work. I assume that some of the employees of Perkin-Elmer, who are now in Foster City, must have some thoughts about the growth and advancement of the mass-spectrometry field.

BROCK: Please discuss the course of gas chromatography at Perkin Elmer while you were employed with that company.

COATES: The story, as I understand it, is that Perkin-Elmer's consultant at Oxford, England, Professor Tommy Thompson, told Van Zandt Williams that there was a new technique called gas chromatography and that Perkin-Elmer should look into it. The process had been developed in England, so the next time that Van Zandt Williams went to England, he and Harry Hausdorff visited Thompson to discuss that technology. They collected some publications, and visited somebody at Oxford who was doing work in gas chromatography. I didn't know anything about it until they came back from England and Harry started making a lot of noise about the potential of gas chromatography. He figured it was a simple system that Perkin-Elmer could replicate as a product easily and quickly. Van Zandt Williams supported the development project, and so Harry and an engineer, Emmett Watson, put a team together. Harry then began giving reports on their findings, and soon the project landed in my applications-engineering department. When Harry went back to Europe, there was no one left working on the gas-chromatography applications, of which there were many. Therefore, I hired Nathaniel Brenner to take over the work. It was a new field for us and he did a wonderful job. He ran customer samples, wrote research papers, and gave talks on his research, which was necessary because it was a new field. Together we spread the word.

In 1958, I had been in very close contact with Dr. Marcel [J. E.] Golay, a famous scientist who was also a Perkin-Elmer consultant. He visited the company about once a week to consult with us. We had a session where we talked about gas chromatography on a theoretical basis. He didn't know anything about it, nor was he very interested in it, but as we continued to talk about its uses and the fundamental principles behind it, he came up with an idea. Having done some research on the subject, Golay returned to Perkin-Elmer the following week and asserted, "Gas chromatography is nothing more than a telephone system!" [laughter] He said that as the molecules pass from one thing to another, it's like a telephone system. [laughter] He'd done all the equations and later published a paper proving that assertion and predicting major improvements (10).

In 1957, I was asked to organize the ISA's [Instrument Society of America] scientific program for the first gas-chromatography symposium at Michigan State University (11). I had to find active researchers to give papers on research at the symposium. Since Perkin-Elmer had effectively pioneered gas chromatography in the U.S., I asked several Perkin-Elmer guys and a few other people to give speeches. I also invited the guy who had literally invented gas chromatography, A. J. P. [Archer John Porter] Martin; and paid his expenses to fly from England and give a talk on his work. This marvelous talk is presented in the book I edited about that first gas-chromatography symposium in the U. S. (12). His predictions were right-on and extremely useful.

Further, I asked Marcel Golay to give a talk on his theory of how gas chromatography really works. [laughter] By that time, he'd concluded that the way it was designed by the guys in England was completely wrong. He thought the separations shouldn't be done with a big packed column, but rather, for the best separations, it should be done with a very small diameter, empty capillary tube with a liquid medium on the walls. His idea turned out to be correct. He then had some of my lab guys make some runs for him on crude capillaries, and he showed that he had about ten-times-higher resolution with his system than anyone had ever shown. In any case, he read his paper at the ISA meeting, entitled, "Theory and Practice of Gas-Liquid Partition Chromatography with Coated Capillaries (13)." I don't think anybody really understood it until much later. Marcel Golay was a brilliant guy. Anyway, that is an interesting story about the early history of gas chromatography. I personally did a lot of work and published papers with various colleagues in gas chromatography until about 1959, when I transferred to the international operations division.

Perkin-Elmer built many kinds of gas chromatographs using the first Golay "columns." They built high-performance gas chromatographs based on his design, which really pushed Perkin-Elmer way out in front. They had about 85 percent of the gas-chromatography business by the end of the 1950s. I was in Japan during that time period, so I wasn't involved with that research. However, Nathaniel Brenner was still involved with the program.

Eventually, Perkin-Elmer made a strategic design error. An engineer at DuPont built a gas chromatograph that he programmed to continuously adjust the column temperature over a range, allowing certain separated components to come out well-separated, but faster. For some reason, the guys at Perkin-Elmer decided that DuPont's modification was not useful because the loss of resolution caused by temperature programming made the process ineffective. The F&M Scientific Corporation built the first temperature-programmed gas chromatograph, and one year later, Perkin-Elmer's 85 percent share of the market dropped to 25 percent, F&M having grabbed 60 percent. Then, HP bought F&M, and HP has been dominant in that business ever since. Perkin-Elmer eventually built temperature-programmed gas chromatographs, but it was too late. I don't know what's happened to Perkin Elmer's gas-chromatograph business.

BROCK: Where does the triple-stage gas chromatograph fit in to that story?

COATES: As Perkin-Elmer was developing the gas chromatograph, it occurred to me that the design should have multiple columns. A second column could take the output from one column and send it into another, different column that would allow other components to separate. Then, they could even add a third column to analyze a complete mixture. An example is a gasoline sample, where there might be a thousand components recorded on three different charts; an operator could separate them and then identify the components. I thought that gas chromatography might go in that direction, so I designed a triple-stage unit by combining three standard ovens. There were a number of those sold, but it was done with the same people who didn't understand temperature programming! [laughter]

BROCK: Who were the key decision makers for gas-chromatography development?

COATES: Many of the employees at Perkin-Elmer were sales oriented. They had numerous ideas for selling the instrument, but they didn't want to risk changing or improving its design and make the old design obsolete. I think that was basically it. I think they were more like conservative salesmen than scientists.

BROCK: Discuss your involvement with UV spectrophotometry at Perkin-Elmer.

COATES: I had designed the Model 12C with a quartz prism that could be plugged in to the instrument to make measurements in the UV-spectral range. Very few people at Perkin-Elmer endorsed that design because UV was mysterious. They had designed the Model 12C with all prisms in mind, and since the UV prism existed during the unit's development, I made an interchange unit for it. I also installed hydrogen and tungsten-lamp sources, a photomultiplier detector, mirror flippers to select those, and a special cam to scan linear-wavelength spectra in the ultraviolet and visible regions.

During that time period, Howard Cary had just introduced his double-beam system, and we considered selling the seemingly competitive, Perkin-Elmer Model 21UV-VIS Spectrophotometer. I made it work quite well, and got superior spectra compared to published data (14). I think we published a paper on it (15). I wanted to introduce the instrument as a serious product. Van Zandt Williams said, "Perkin-Elmer doesn't know anything about UV spectroscopy. We're not going to touch it." I replied, "Don't you think we can learn? I think we can learn. I've looked at other people's results, and I think our huge prism will give us four or five times the resolution and accuracy that has been achieved by others." Despite my protests, and the existence of a completed, tested prototype, the project was discontinued. Van Zandt Williams was not interested in UV, and he was the boss.

About two years later, Williams ran into a man named Chuck Warren at the Pittsburgh Conference. Warren's company had built a double-beam-UV system, called the Ultracord, as an attachment to the Beckman Model DU. Williams thought Warren's design was interesting, so Perkin-Elmer bought his company and gave Chuck Warren some engineers to produce his instrument. As far as I know it never worked. They introduced it, but it couldn't compete with the other UV systems. I had nothing to say about it. Conversely, the Model 21UV had a lot going for it because it had higher resolution, a huge prism, chart paper, the ability to scan linearized spectra, and it was built into the Model 21's manufacturing structure. Nevertheless, we built an interchange unit with a quartz prism and the ability to operate in UV. I was heavily involved in that project. But that's the end of my story! [laughter] I got really involved with UV and visible light again when I started Nanometrics.

[END OF TAPE, SIDE 6]

COATES: Somewhere around 1955, Van Zandt Williams became interested in the application of IR-to-automatic measurements on chemical process-streams. He thought that would be an application for the next generation. He was always interested in quantitative analysis, as I've mentioned, so he started a project to design IR instruments that were rugged enough to work in a process-chemical-plant's environment; where they would sample some of the gas or liquid, send it through a cell, and then back into the stream, while continuously monitoring a particular component at a wavelength of interest. There were two products developed, one was called the Trinon [Analyzer] because it was a triple beam, non-dispersive IR instrument, and the other one was called the Bi-Chromator Analyzer; a dispersed, spectral-analysis system.

[INTERRUPTION]

COATES: The chief engineer of the Trinon Analyzer was Elliot Woodall. The Bi-Chromatic Analyzer was designed by Abe Savitsky. It was a very rugged IR spectrometer that had a split mirror to simultaneously measure and ratio two wavelengths; which was a pretty good idea.

Perkin-Elmer invested more money in those two products than they had ever invested in anything before. When the units were finished, their introduction on the market interested many in the chemical field. Then, consumers discovered a very interesting problem with the instrument. Chemical companies did not know the IR characteristics of the “other” impurities and components that were in the streams. To sell them a system, we had to identify the exact contents of the streams. If we didn’t, an entire plant could shut down, or something could explode, because an unidentified substance went through the stream and gave a user a false reading.

For instance, I remember a group from Eastman Kodak [Company] who needed to examine factory air pollution in the film business. The company formed a group to set up the instrument, but the Trinon’s measurements of customers’ samples never made any sense. As a result, Perkin-Elmer spent about one-million dollars in R&D, but had very few sales to show from it. The instrument was a disaster and it was soon discontinued. Williams never talked much about it again, nor did any of the people who worked on it. The engineers were assigned to other projects, and the instruments just sat there in a big room in the back.

In 1958, I received a call from Van Zandt Williams, who said, “Vince, I want you to take over the Trinon and Bi-Chromatic Analyzers’ developments as part of the applications-engineering department.” I replied, “I don’t know anything about those machines.” He countered, “They’re being discontinued, so there’s not much to be done. Just take it over.” About a month later, I got a call from one of our sales guys who said General Motors had just introduced pneumatic springs for their cars. They were planning on installing the springs on many of their cars the following year, but first they required machines that could measure for leaks. An engineer from GM asked if we had an instrument that could measure the leakage. I said, “If you can put nitrous-oxide in to the pneumatic tube, we could use that gas as a tag to detect any possible leaks. He said, “We can do that.” So, I replied, “We have about fifty machines in stock that can measure all that stuff perfectly.” [laughter] As a result, we sold all of our Trinons to General Motors and made money on our investment. Soon after, we had to put the Trinon back in to production because Ford [Motor Company], who was in strong competition with General Motors, had introduced similar pneumatic springs. I guess it was smart of Van to finally turn the product over to the applications-engineering department!

We managed to sell our entire Trinon inventory. During that time period, Max [D.] Liston developed a similar IR-measuring device. It was a very successful system because he had done such a good job designing it. It didn’t require the very complex tuning-up that the original Trinon had required. The Trinon was called a closed-loop-measuring system. We had a saying in those days: “Maxie Liston, he’s no dope, he did it all with an open loop!” [laughter]

BROCK: Did Perkin-Elmer ever really get into process control?

COATES: Yes. They later designed a process-control gas chromatograph that sold for several years. However, once their Trinon inventory was exhausted, they weren't about to get back in to that business. The instrument required a very complex, tuning-up process. Some other companies had made IR analyzers that were used in coal mines to detect explosive gas in the atmosphere. For example, Mine Safety Appliances built an IR system that they were very knowledgeable about because they had already sold products to coal mines and other industrial workplaces. I would like to ask Max Liston who his present customers are; he must have an interesting business.

Since we're telling stories about process monitoring, I have another one. There was a different company that had gone in to that business and taken a piece of our Trinon market. We had come very close to getting an order from the Dow Chemical Company, but we lost the order to them. Dow had built a multimillion-dollar plant to make a certain chemical component, and they wanted to make sure that their chemical process gave then the optimum results. We didn't get the Dow Chemical order because they bought a better IR analyzer from our competitor. After our competitor installed the instrument, they continuously monitored and tuned the temperatures and adjustments. As the desired composition went higher, the company increased its profits. At 3:00 am one morning, the composition got to the point where the entire process in the pipe turned into a solid! [laughter] Here was a multi-million-dollar plant using a process-stream-IR analyzer. There was no way to clean the solid out of the system; all they could do was dump it! Dow Chemical lost its entire investment.

The IR analyzer was thought to be the culprit. Our competitor blamed the failure on the engineers who had designed it, saying that they weren't informed of the chemical properties and were only supposed to measure the efficiency. In chemistry, a substance sometimes has two general isomer forms: cis and trans. The company had wanted more trans than cis isomers. But anyway, that's a famous story of infrared process control. [laughter] Perkin-Elmer's management was slightly scared by that story. It shows things that seem simple on the surface may not be.

BROCK: Thank you very much for the interview.

[END OF TAPE, SIDE 7]

[END OF INTERVIEW]

NOTES

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11. *Gas Chromatography: A Symposium Held Under the Auspices of the Analysis Instrumentation Division of the Instrument Society of America, August, 1957*, ed. Vincent J. Coates, Heney J. Noebels, and Irving S. Fagerson (New York: Academic Press, 1957).

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APPENDIXES

APPENDIXES

I: Publications

II: Patents Issued

III: *The Perkin-Elmer Instrument News* (1960): 2-3.

IV: *Spettroscopia E Chromatografia Di Gas: International Section* (1958): 10-11.

V: Vincent J. Coates, Abe Offner, and E. H. Siegler, Jr., "Design and Performance of an Infrared Microscope Attachment," *Journal of the Optical Society of America* 43, no. 11 (November 1953):984-989.

VI: Vincent J. Coates, "A Variable Thickness Liquid Absorption Cell," *The Review of Scientific Instruments* 22, no. 11 (November 1951): 853-854.

VII: *PEN* 9, no. 10 (November 1961): 1, 8.

VIII: Photograph of Coates at an International Sales Meeting [Date and publication unknown]

IX: Photographs of Coates at Hitachi Perkin-Elmer, Ltd. Meeting held in Japan [Date and publication unknown]

X: Vincent J. Coates, "Differential Knob Device," U.S. Patent # 2,658, 395. Issued 10 November 1953.

XI: Bryce Crawford, Jr., "Chemical Analysis by Infrared," *Scientific American* (October 1953):42-48.

XII: "Comparison of Various Infrared Spectrometric Systems"

XIII: "Laboratory of the Month," *Analytical Chemistry* [Date unknown]

XIV: *Perkin-Elmer Instrument News for Science and Industry* 6, no. 3 (Spring 1955):1, 3-4, 8.

XV: Vincent J. Coates and Nathaniel Brenner, "Fuel Gas Analysis by Chromatography?" *Petroleum Refiner* 35, no. 11 (November 1956): 197-201.

XVI: Vincent J. Coates and Robert Anacreon, "Model 21 Ordinate Scale Expansion System Extends IR Measurement Sensitivity," *Perkin-Elmer Instrument News for Science and Industry* 9, no. 2 (Winter 1958): 1, 9-12.

XVII: *Perkin-Elmer Instrument News for Science and Industry* 4, no. 4 (Summer 1953): 1, 6-8.

XVIII: Vincent J. Coates and Harry Hausdorff, "Interferometric Method of Measuring Spectral Slit Width of Spectrometers," *Journal of the Optical Society of America* 45, no. 6 (June 1955): 425-430.

XIX: Society for Applied Spectroscopy Program, Ninth Annual Meeting, 27-28 May 1954.

XX: Vincent J. Coates, Thomas Miller, and Abraham Savitzky, "The Performance of the Perkin-Elmer Model 21 in the Region 210mu to 2000mu," *Applied Spectroscopy* 9, no. 1 (February 1955): 14-19.

XXI: Nanospec 20IR Infrared Microscope Spectrophotometer, Nanometrics Incorporated, Milpitas, California.

LIST OF VINCENT J. COATES' PUBLICATIONS

TITLE	CO-AUTHOR	PUBLICATION
Variable Thickness Liquid Absorption Cell	--	The Review of Scientific Instruments, Vol.22, #11, 11/51
Design and Performance of an Infrared Microscope Attachment	A. Offner/ E.H.Siegler,Jr.	Journal of the Optical Society of America, Vol.43, #11, 11/53
The Performance of the Perkin Elmer Model 21 in the Region 210mu to 2300mu	T. Miller/ A. Savitzky	Applied Spectroscopy, Vol. 9, #1, Feb. 1955
Interferometric Method of Measuring the Spectral Slit Width of Spectrometers	H.Hausdorff	Journal of the Optical Society of America, Vol.45 #6, June 1955
Fuel Gas Analysis by Chromatography	N. Brenner	Petroleum Refiner, 11/56
Gas Chromatography	H.J.Noebels/ I.S.Fagerson	Instrument Society of America, 8/57 Symposium
Model 21 Ordinate Scale-Expansion System Extends IR Measurement Sensitivity	R.Anacreon	Perkin-Elmer Instrument News, Vol.9, #2, 1958
Molecular Sieves as Subtractors in Gas Chromatographic Analysis	N. Brenner	Nature, Vol. 181, May 17, 1958
Resolution in Scanning Electron Microscopes	N. Brenner	Research/Development 6/73, Vol.24, #6
High Resolution Scanning Electron Microscopy at Low Accelerating Voltages	L. M. Welter	Proceedings of the 7th Annual Scanning Electron Microscope Symposium, Chicago, Apr. 1974
The Raman Microprobe - Theoretical and Practical Considerations	--	Presented at 1975 Pacific Conf.on Chemistry & Spectroscopy, Oct. 1975
A New and Simple Approach to Quantitative Microspectrofluorometry	M.W.Bernes	Presented at the First International Congress on Cell Biology Boston, Sept. 1976
NanoSpec/10 - A Wide Range and General Purpose Computerized Microspectrophometer System	--	The Microscope, Vol. 24, 4th Quarter 1976
Computerized Microspectrophotometer System	--	Presented at Pacific Conf. on Chemistry & Spectroscopy S.F., Sept. 1978

Advances in Microspectrophotometry I. Reflectance, Absorbance & Corrected Fluorescence Measurements in the Visible and Deep Red Regions Using a Computerized Microspectrophotometer	—	Presented at Pittsburgh Conference on Analytical Chemistry & Spectroscopy, 1978.
Near Infra-red Microspectrophotometry of Organic and Inorganic Materials	M.J.Vogel	1978 Royal Microscopical Society Proceedings, Vol.13, Part 4
Computerized Optical System for Precision Linewidth Measurements	—	Presented at the Microelectronics Measurement Techn. Seminar, S.J., 2/7/79
Computerized Optical Systems for Linewidth and Film Thickness Measurements on Microelectronic Circuits	—	SPIE Proceedings, Vol.174, S.J., 4/23/79
Application of Nanospec/10S Microspectrophotometer with SDP-2000 Spectral Data Processor To Differential Color Analysis	—	Nanometrics' Company Publication
Optical Characterization Techniques for Semiconductor Technology	D. Zaring	Proceedings of the Society of Photo-Optical Instrumentation Engineers, S.J.,4/81
Computerized Electron Beam Linewidth Measuring & Inspection - A New Tool	T. Pomposo	Dec. 22, 1981
High Resolution Low Accelerating Voltage Scanning Electron Microscopy of Uncoated, Non-Conducting Surfaces	—	40th Annual Proc. EMSA, Washington DC, 1982
Computerized Microspectrophotometers For Forensic Investigations	H.Hausdorff	Pittsburgh Conf., NJ, 3/8-13/82
Color Matching By Microspectrophotometry, A New Dimension in Document Examination	H. Hausdorff	SPIE, Vol. 411, 1983
Sample Size Limitations in Infrared Analysis	L. Kisken	Presented at 5/84 Pittsburgh Conference
Electron Beam X-Ray, Ion-Beam Techniques for Submicrometer Lithographies	W. Roth III	SPIE, Vol. 471, S.C. 3/15/84
An Infrared Microspectrophotometer Optimized for Microanalysis	W. Telfair H.Hausdorff	Presented at 5/84 Pittsburgh Conference, N.J.
Fab Area Networks for Improved Process Monitoring Using Measurement Data	J.McCall	VLSI Fall '84 Conference, Boston, MA
Measurement & Inspection Cwikscan SEM Using Schottky Gun	M.Ohtsuki/ D.Holmes/L.Denney	Semicon Korea, 3/86

New Automated Inspection & Measurement Field Emission SEM Equipped with Schottky Electron Emitter	M.Ohtsuki/ J.Ikovic/D.Holmes/ A.Abel/L.Denney	ICEM, 8/31/86, Kyoto, Japan
Applications of Low Accelerating Voltage Field Emission Scanning Electron Microscope to Uncoated, Non-conducting Biological Specimen	M.Ohtsuki/ Y.Sugiura/T.Makita	ICEM, Kyoto, 1986
Low Accelerating Voltage Field Emission Scanning Electron Microscopy on Uncoated, Non-conducting Plastic Replica or Blood Vessels of Rat Gastric Mucous Epithelium	M. Ohtsuki/ T. Makita	ICEM, Kyoto, 1986
Automated SEM and Optical Measurement Stations for Manufacturing Process Control of Integrated Circuits	W. Lin	ICSTCT, Beijing, China, 10/86
The Origin & Present State of Analytical Microspectrometry	—	FACSS, Detroit, MI, 10/4/87
Basic Improvements in Submicron Optical Measurement Methods	S.Williams/ W.Howe/R.Ingalls	SPIE Symposium, 1988

LIST OF PATENTS ISSUED - VINCENT J. COATES

UNITED STATES PATENTS

PATENT NO.	PATENT TITLE	DATE	INVENTORS
5,045,704	METHOD FOR DETERMINING ABSOLUTE REFLECTANCE OF A MATERIAL IN THE ULTRAVIOLET RANGE, RE.34,783, 11/8/94	9/3/91	V.J.COATES
RE. 34,783	- SAME AS ABOVE	11/8/94	V.J.COATES
4,884,890	METHOD FOR NORMALIZING THE DETECTION SIGNALS OF MAGNIFIED IMAGES OF FLUORESCING MATERIALS	12/5/89	V.J.COATES
4,849,694	THICKNESS MEASUREMENTS OF THIN CONDUCTING FILMS	7/18/89	V.J.COATES
4,826,321	THIN DIELECTRIC FILM MEASURING SYSTEM	5/2/89	V.J.COATES/W.LI
4,743,757	SECONDARY ELECTRON EMISSION CONTROL IN ELECTRON MICROSCOPES	5/10/88	V. J. COATES
4,596,929	THREE-STAGE SECONDARY EMISSION ELECTRON DETECTION IN ELECTRON MICROSCOPES	6/24/86	V.J.COATES/D.C HOLMES/G. TORO-LIRA
4,521,686	LINEWIDTH MEASURING WITH LINEARITY CALIBRATION OF THE T.V.CAMERA TUBE	6/4/85	V.J.COATES/J.E. GRUND/S.WESTRATE
4,373,817	COMPUTERIZED MICROMEASURING SYSTEM AND METHOD THEREFOR	2/15/83	V. J. COATES
4,308,586	METHOD FOR THE PRECISE DETERMINATION OF PHOTORESIST EXPOSURE TIME	12/29/81	V. J. COATES
4,020,387	FIELD EMISSION ELECTRON GUN	4/26/77	V.J.COATES/L.M. WELTER
3,925,664	FIELD EMISSION ELECTRON GUN	12/9/75	" "
3,931,519	FIELD EMISSION ELECTRON GUN	1/6/76	" "
3,931,517	FIELD EMISSION ELECTRON GUN	1/6/76	" "
3,842,272	SCANNING CHARGED PARTICLE MICROPROBE WITH EXTERNAL SPURIOUS ELECTRIC FIELD EFFECT CORRECTION	10/15/74	" "/ J. J. GOLD
3,784,815	LOW VOLTAGE FIELD EMISSION SCANNING ELECTRON MICROSCOPE	1/8/74	V.J.COATES/L.M WELTER

3,766,427	FIELD EMISSION ELECTRON GUN	10/16/73	V.J.COATES/L. WELTER
3,678,333	FIELD EMISSION ELECTRON GUN UTILIZING MEANS FOR PROTECTING THE FIELD EMISSION TIP FROM HIGH VOLTAGE DISCHARGES	7/18/72	" "
3,767,926	FIELD EMISSION SCANNING MISCROSCOPE DISPLAY	10/23/73	" "
RE. 28,153	" " " "	9/10/74	"

IN ADDITION TO THE FOREGOING U.S. PATENTS, THERE ARE VARIOUS CORRESPONDIN PATENTS IN OTHER COUNTRIES.

OFF THE BEAM

We'll See You at the Chicago and Philadelphia Meetings

Perkin-Elmer will exhibit at the 10th National Chemical Exposition in the International Amphitheater in Chicago, September 9-12 during the 134th national meeting of the American Chemical Society. On exhibit in P-E Booth No. 107 will be the *Model 21 infrared spectrophotometer with the beam condensing system for micro sampling and slave recorder*, the *Model 154-C Vapor Fractometer with the new Perkin-Elmer Printing Integrator*, the *Model 4000-A Spectracord® Ultraviolet Spectrophotometer with the diffuse reflectance attachment*, the *Model 137 NaCl Infracord® spectrophotometer*, and the new *KBr Infracord spectrophotometer*.

The following week, Perkin-Elmer will exhibit at the 13th Annual Instrument-Automation Conference and Exhibit presented by the I.S.A. at Philadelphia Convention Hall, Booth 910-1011. Products to be displayed include: The *Model 21 Infrared Spectrophotometer* with *slave recorder* and *beam condensing system for micro sampling*, the *Model 4000-A Spectracord® Ultraviolet Spectrophotometer* with *diffuse reflectance attachment*, the *Model 137 NaCl Infracord® spectrophotometer*, the *new Model 137 KBr Infracord*, the *Model 154-C Vapor Fractometer* with the new *Printing Integrator*, the *Model 184 Process Vapor Fractometer*, the *Model 188 Wide Range Vapor Fractometer* and the *Model 105 Leak Detector*.

We look forward to seeing you at these two important meetings. Our booth No. 107 at the Chicago exposition is right next to the main registration area. At Philadelphia Convention Hall we're located in the Grand Hall between the 900 and 1000 aisles.

A valuable source of reference for chemists, physicists and clinical analysts is *Infrared Absorption Spectra of Steroids, An Atlas, Vol. II*, by Glyn Roberts and Beatrice S. Gallagher of Sloan-Kettering Institute for Cancer Research and R. Norman Jones of the National Research Council, Ottawa. Volume II provides curves for 362 additional steroids, and contains 452 charts. It lists curves in both volumes alphabetically and by functional groups, contains a table of characteristic frequencies and bibliographies. Published by Interscience Publishers, Inc., 250 Fifth Avenue, New York 1, New York. 875 pages, price $20.00.

Gas Chromatography, edited by Coates, V. J., Noebels, H. J., and Fagerson, I. S., Academic Press, New York, 1958. 323 pages.

This collection of the proceedings of the August 1957 Symposium on Gas Chromatography of the Instrument Society of America is a necessary addition to any library of literature in the gas chromatography field. Included are papers which describe such novel apparatus as the capillary column chromatograph of Golay, the high temperature "glow plug" chromatograph of Felton, Kirkland's preparative apparatus, and several advanced commercial laboratory and process control instruments. Theoretical studies of the fundamental chromatographic process by Young and by Hinkle and Johnson, and an exhaustive survey of thermistor behavior by Cowan and Sterling are also notable contributions. Other valuable discussions relate to special applications of the technique and studies on column construction parameters. A transcript of the stimulating address delivered by A. J. P. Martin is also included. One of the indispensable features of the volume is the 442-entry Bibliography on Gas Chromatography compiled by Langer and Zahn and the Bureau of Mines staff. This author-indexed list covers the years 1952 through 1957 and provides a much needed compilation of gas chromatographic literature.

A Committee on Nomenclature was established in conjunction with the Symposium. The report of its considerations and conclusions has been appended to the proceedings. Included here are accepted definitions of column performance.

Jack Baudean . . .

P-E Southern Office Manager

Jack Baudean, Southern Regional Sales Manager for Perkin-Elmer, was born 33 years ago in the same city where he now holds sway—New Orleans, Louisiana. Known by his colleagues in the Southern office as "The Professor," he combines a thorough academic background in physics and math with considerable administrative ability.


Jack Baudean

Jack has, in fact, been P-E's New Orleans manager since 1955, graduating to that post after two years in the bayous as a Perkin-Elmer Sales and Service Engineer. Before joining us, he was Acting Head of the Physics Department at Xavier University, New Orleans, for four years.

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KNOW YOUR P-E SALESMEN

☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆ ☆

His academic background started at Jesuit High School, from which he progressed to undergraduate studies at Tulane, receiving his B.S. in 1947. Two years later he earned his Master's degree, also at Tulane. His thesis for that degree—"Viscous Fluid Flow Between Two Coaxial Rotating Cylinders" was exposed to the scientific world in the *American Journal of Physics*—Vol. 19, No. 2, Feb. 1951, if you want to look it up.

He and his wife Mary Lou number three children—Suzanne, 6, Lise, 4, and John, 2. Jack claims no discrete hobbies except "waiting at airports, and trying to catch up on sleep when at home."

Perkin-Elmer sales records show that Jack's calorific personality and persuasive Southern ways have borne fruit in an enviable annual volume.

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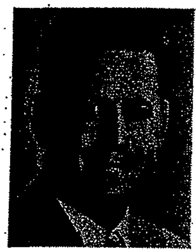
EDITORIALLY SPEAKING

A Second Symposium on Gas Chromatography, organized by the Gas Chromatography Discussion Group under the auspices of the Hydrocarbon Research Group of the Institute of Petroleum of Great Britain and the Koninklijke Nederlandse Chemische Vereniging, was held May 19th through 23rd in Amsterdam, Holland. This meeting, attended by over 400 persons from many parts of the world, indicated quite clearly that the young science of gas chromatography was moving forward in a very vigorous manner.

Twenty-eight papers were contributed and intensively discussed, covering elucidation of theory as well as description of techniques and apparatus. Important information concerning new applications of the method was presented.

Many of the papers disclosed new and important advances in the gas chromatography field. In particular, the rigorous work of J. Behemen and J. H. Purnell relating to improving the performance of packed columns deserves mention. M. J. E. Golay of Perkin-Elmer presented a paper enlarging on his theory of the chromatography of open and coated capillary columns. Experimental work performed by Dr. Golay indicated that efficiencies of the order of 50,000 theoretical plates or better were attainable with capillary columns. McWilliams and Dewar described an interesting new flame ionization detector for gas chromatography capable of analyzing trace compounds with sensitivities exceeding 10^9 mV ml/mg.


Vincent J. Coates

R. P. W. Scott showed chromatograms resulting from improved column construction and operation, which indicated efficiencies of the order of 40,000 theoretical plates. A spirited discussion of the influence of the nature of the solid support on column performance indicated a need for further study and clarification of this area. A series of papers were presented describing the optimal use of programmed column heating as a means of decreasing analysis time in gas chromatography when wide boiling range mixtures are analyzed.

Several interesting papers were presented concerning the applications of gas chromatography to particular analytical problems. Ellis and Iveson described the analysis of some halogenated compounds. Bovijn, Pirotte and Berger discussed the measurement of low concentrations of hydrogen in water, and Liberti and Cartoni presented a most interesting paper on the analysis of essential oils. Bayer discussed the separation of derivatives of amino acids. Ambrose and Purnell gave examples of the method of presenting standard retention data using various solvents based on recommendations made by a Committee of the Gas Chromatography Discussion Group.

The published proceedings of this important symposium will appear shortly. They will be edited by D. H. Desty and published by Butterworth Scientific Publications in London. It is recommended that those interested in gas chromatography should refer to the published proceedings. A wealth of important new information is contained in both the papers and the intensive discussion which follows.

At the Instrument Exhibition held in conjunction with the Symposium, P-E exhibited the Model 188 Triple Stage Vapor Fractometer and the Model 184 Process Vapor Fractometer. Bodenseewerk Perkin-Elmer (Germany) exhibited the Model 116 Fractometer with an automatic gas sampling accessory.

Those attending the symposium were grateful that the Organizing Committee had chosen to hold the symposium during the lovely tulip season prior to Whitsuntide. Also memorable were the side excursions to points of interest, the boat trip on the canals and the reception at the Tropical Institute and the State Museum.

V. J. C.

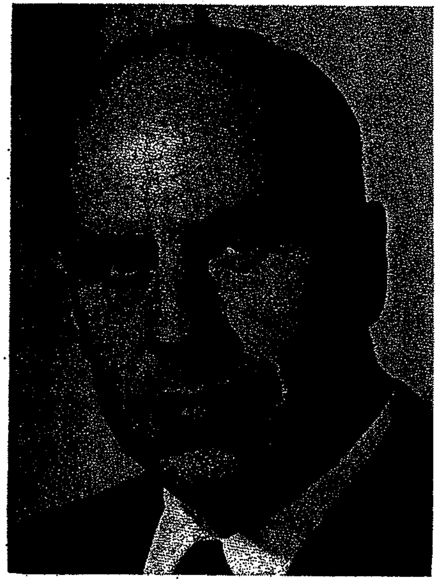

A view of the conferees in the auditorium of the Tropical Institute during one of the Gas Chromatography sessions in Amsterdam.

P-E's Vincent J. Coates, Manager of Application Engineering, served as Chairman for the May 21 session on Techniques and Apparatus at the Amsterdam Symposium. Other P-E people attending the sessions included: M. Golay, H. Hausdorff (Norwalk), G. Zahler, H. Hediger, D. Orr, G. Caroti, W. Quadt (P-E A. G. Zurich), J. Wolff, A. Loew, Dr. Palm (Bodenseewerk Perkin-Elmer).

International Section

BRITISH INFRACORDS® NOW AVAILABLE

A. R. Gilson, Managing Director of P-E's British subsidiary, Perkin-Elmer Ltd., Beaconsfield, Bucks., announces the availability of Model 137 Infracord spectrophotometers completely manufactured in England. Further information may be obtained from your nearest Sales and Service Office.



A. Ralph Gilson

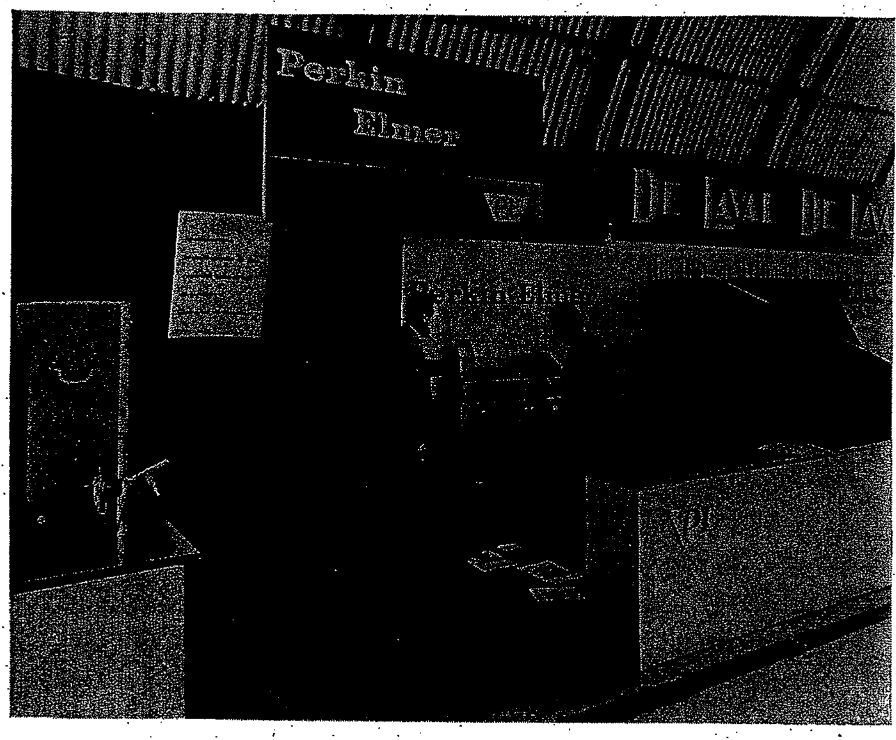
A. R. GILSON HONORED BY BRITISH GOVERNMENT

In recognition of his service to science, A. Ralph Gilson has been named to Her Majesty the Queen's Honour List and awarded as a Member of the Order of the British Empire (M.B.E.).

The Managing Director of our British manufacturing subsidiary, Perkin-Elmer Limited, Mr. Gilson has received this coveted award for his work in building and equipping the new Chemical Laboratories at Cambridge University. These are the largest, and considered to be the finest academic laboratories in the world.

He was formerly Executive Director of the Laboratories for 13 years.

SPETTROSCOPIA E CHROMATOGRAFIA DI GAS



Perkin-Elmer A.G. exhibited at the Milan Fair, April 12-27. Dr. Caroti discusses the Model 21 with a visitor.

PERKIN-ELMER ITALIANA, S.p.A.

On June 18, 1958, P-E A.G. Zurich established an Italian sales company in Milan. Our new European sales subsidiary will handle sales and service of products manufactured in Norwalk and at our British and German plants.

Dr. Gino Caroti

Dr. Gino Caroti, Citizen of Florence, is manager of the Milan office, one of several affiliated companies of P-E's European sales organization. Dr. Caroti, well known in infrared circles, studied at the University of Florence and at Princeton. He was previously in charge of the infrared laboratory of the Stazione Sperimentale per Combustible in Milan.

FREIBURG INFRARED COURSE

Under the direction of Prof. Dr. R. Mecke, an introductory course to practical infrared spectroscopy will be held at the Institute for Physical Chemistry of the University of Freiburg im Breisgau from October 21-28, 1958. The course will consist of both lectures and laboratory work and will cover general theory, instrument design and operation, use of accessories, interpretation of spectra and other related subjects, including Raman and microwave spectroscopy, and the use of gas chromatography in combination with infrared. Those interested in participating are requested to contact Prof. Dr. R. Mecke before September 15, 1958, at Hebelstrasse 38, Freiburg i. Br., Germany. The number of participants is limited.

Dealer Seminar

Perkin-Elmer A.G. held its first dealer seminar on June 10-13 at the Zurich offices. Attending were most of P-E's European dealers and personnel from the Company's sales subsidiaries. Plans were discussed for streamlining customer services and there was a general orientation program on instruments and applications. H. H. Hausdorff and V. J. Coates (Norwalk) assisted in the program.

Bodenseewerk Instrumentation Course

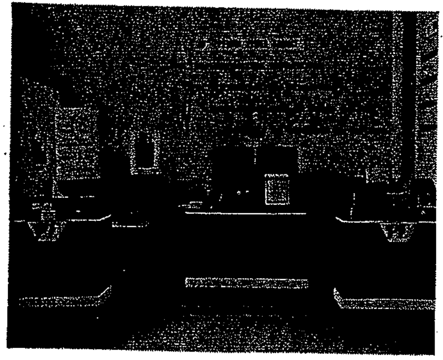
Bodenseewerk Perkin-Elmer G.m.b.H. conducted an instrumentation course the week following the seminar (June 16-19) in Uberlingen. Attendees included twenty-two of our German customers as well as P-E personnel. The program covered the operation of Perkin-Elmer instruments, sampling techniques and general background information on instrument self-service. BSW hopes to make the course an annual affair.

FIFTH OTTAWA SYMPOSIUM ON APPLIED SPECTROSCOPY

The Canadian Association for Applied Spectroscopy will hold its Fifth Symposium on September 15-17, in the Lecture Hall, Victoria Museum, at Metcalfe and MacLeod Streets, Ottawa, Ontario. Registration will begin at 8:30 Monday morning, and the first paper at 9:05. The symposium dinner will be held Tuesday evening at the Quebec Suite of the Chateau Laurier. Inquiries should be addressed to:

Mr. W. J. Wright
Program Committee
Fifth Ottawa Symposium for Applied Spectroscopy
c/o Noranda Copper and Brass, Ltd.
P. O. Box 1238, Place D'Armes
Montreal, Quebec.

Spectrophotometres Infrarouge et Chromatographie en Phase Gazeuse

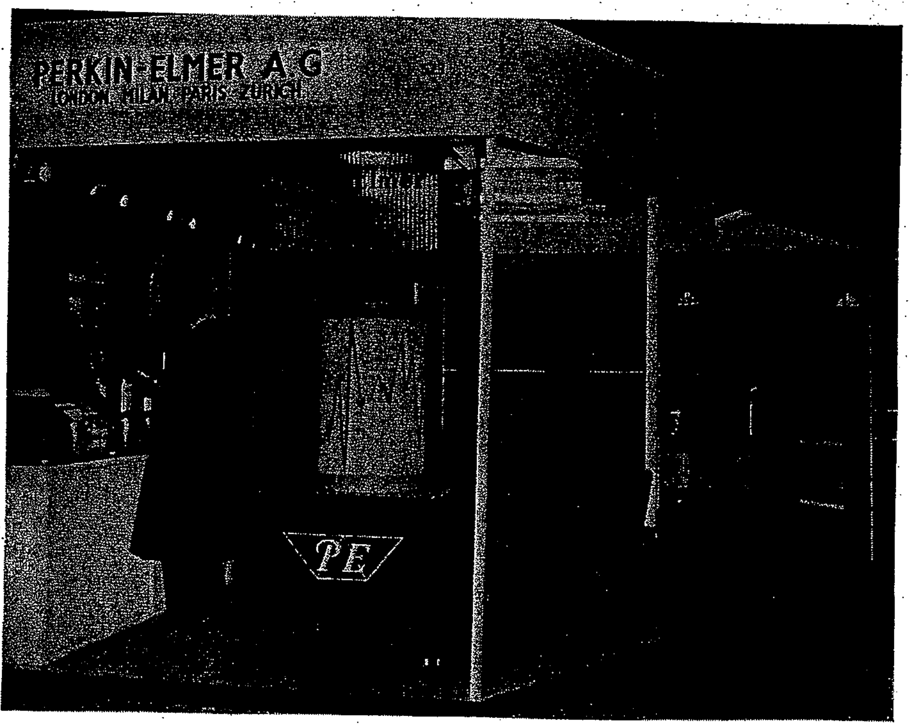


Perkin-Elmer France S.A.R.L., P.E.'s sales and service subsidiary in Paris, exhibited a Model 21 infrared spectrophotometer and Model 154-C Vapor Fractometer manufactured at Perkin-Elmer Bodenseewerk G.m.b.H., and a Model 137 Infracord® spectrophotometer manufactured at Norwalk, at the Paris Physics Exhibition in April. M. Vassy is Sales and Service Manager, and M. Derazey, Office Manager, of the French company.

INTERLUDE AT THE ACHEMA

Amid escargots and wine, J. Wolff, Sales Manager of P-E's Frankfurt Office, Vincent J. Coates, Manager of Application Engineering, Norwalk, Harry Hausdorff, Director of Sales, International Operations, Norwalk and Al Loew, Service Manager, Frankfurt, got together in June at the Brückenkeller in Frankfurt following the arduous day's activities at the show.

P-E at the Olympia



April was a busy month for Perkin-Elmer's foreign subsidiaries and sales offices, with exhibits at Brussels, Milan, London and Paris. Above is shown our booth at the Instruments, Electronics and Automation Exhibit in London on April 16-25.

JOURNAL OF THE OPTICAL SOCIETY OF AMERICA VOLUME 43, NUMBER 11 NOVEMBER, 1953

Design and Performance of an Infrared Microscope Attachment*

VINCENT J. COATES, ABE OFFNER, AND E. H. SIEGLER, JR.
The Perkin-Elmer Corporation, Norwalk, Connecticut

(Received July 3, 1953)

An infrared microscope employing reflecting optics has been designed and constructed as an attachment to Perkin-Elmer single beam spectrometers. It is mounted after the exit slit of the monochromator in the dispersed beam to minimize possible heating and photochemical effects in the sample. The normal macro functions of the spectrometer are not disrupted by the addition of the microscope. Convenient macro-micro conversion is provided.

The condenser and objective pair, which are of the Schwarzschild type, operate at a numerical aperture of 0.75 with a 0.4 obscuration ratio and are designed to provide optimum imagery in the infrared region of the spectrum. The condenser forms an image of the exit slit at the sample space reduced 8.5×, and the objective provides a 25× enlarged image of the sample at an adjustable diaphragm. A viewing and manipulating system is provided to allow accurate positioning of small samples. The maximum field size is 0.650×0.220 mm. About 35 percent of the radiation available from the monochromator is conserved by minimizing the number of reflecting surfaces and by utilizing field mirrors to provide efficient energy transfer. The radiation is brought to a separate detector and preamplifier which are connected to the amplifier and recorder of the spectrometer.

The minimum sample size which can be effectively studied depends on wavelength and is primarily limited by the available energy. Minimum sample size and spectral dilution are discussed.

The infrared absorption spectra of fibers, crystals, and biological tissue sections, as well as solutions in cells, of extremely small volume, have been obtained using this instrument. Performance data are given and a figure of merit determined.

INTRODUCTION

AN infrared microscope attachment to the Perkin-Elmer Models 12, 112, and 13 spectrometers has been constructed. The design aims for this instrument were arrived at through consultation with potential users of such equipment and by careful study of instruments already in use in several laboratories. It was felt that by this means an instrument capable of application to a variety of problems in a relatively new and expanding field would be achieved.

The design requirements of infrared microscopes, or more accurately infrared micro-sampling attachments, have been rather thoroughly explored in recent years and a number of systems have been constructed.[1–12] All of these instruments have the common property of measuring, as a function of wavelength, the infrared absorption of minute samples. The resulting absorption spectra are similar to the infrared spectra of macroscopic samples so that the well known applications of infrared[13]—qualitative analysis, quantitative analysis, and molecular structure investigations—are possible.

Infrared microscopes have unique importance where (a) the amount of sample is small, (b) the dimensions of the sample are small, and (c) the sample is inhomogeneous. As little as 0.1 microgram of sample in the field of the microscope can yield useful spectra.

Samples which have been studied using such equipment include natural and synthetic fibers, single crystals, biological tissue sections, and bacterial cultures. Instruments of this type allow the study of liquid extracts and solutions in cells of extremely small volume. Some work has been done using compounds separated by chromatography. Recently a technique[14,15] for compressing finely ground samples mixed with potassium bromide powder into optically clear pellets of optimum dimensions has created interest and appears to be a useful sampling procedure for infrared microspectrophotometry. Polarization effects permit investigation of molecular orientation in dichroic samples.

DESIGN

The infrared microscope attachment has been designed to yield infrared spectra of minimum size samples. Figure 1 shows the prototype instrument mounted on a spectrometer.

The arrangement of optical parts is indicated in Fig. 2. Energy from the exit slit of the monochromator is incident on a field mirror which directs it upward and forms a reduced image of the pupil of the monochromator (the Littrow mirror) near the convex mirror of the condenser. By this means radiation from the entire useful slit is directed to the condenser. The condenser forms a reduced image of the exit slit at the sample space and at the same time reimages the Littrow mirror near the center of curvature of the objective mirrors. The objective collects the energy which has passed through the sample and forms a magnified image of the sample at an adjustable dia-

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* Presented in part at the Ohio State University Symposium on Molecular Structure and Spectroscopy, 1953.

[1] Barer, Cole, and Thompson, Nature 163, 198 (1949).

[2] R. C. Gore, Science 110, 710 (1949).

[3] Elliott, Ambrose, and Temple, J. Sci. Instr. 27, 21 (1950).

[4] Blout, Bird, and Grey, J. Opt. Soc. Am. 40, 304 (1950).

[5] D. L. Wood, Rev. Sci. Instr. 21, 764 (1950).

[6] R. D. B. Fraser, Discussions Faraday Soc. No. 9, 378 (1950).

[7] E. R. Blout and G. R. Bird, J. Opt. Soc. Am. 41, 547 (1951).

[8] R. M. Badger and R. Newman, Rev. Sci. Instr. 22, 935 (1951).

[9] A. R. H. Cole and R. N. Jones, J. Opt. Soc. Am. 42, 348 (1952).

[10] M. B. Hall (private communication).

[11] G. A. Contos, Ph.D. dissertation, Columbia University.

[12] C. S. Rupert (private communication).

[13] V. Z. Williams, Rev. Sci. Instr. 19, 135 (1948).

[14] U. Schiedt, Z. Naturforsch. 7b, 270 (1952).

[15] M. M. Stimson and M. J. O'Donnell, J. Am. Chem. Soc. 74, 1805 (1952).

phragm. The energy then passes to a second field mirror which forms an image of the Littrow mirror near the center of curvature of the thermocouple condenser. The thermocouple condenser forms a reduced image of the slit on the thermocouple.

The condenser reduction is about 8.5×, the minimum required to fill the aperture of the objective with radiation from the spectrometer. The maximum sample area is 650×220 microns which corresponds to a maximum slit height of 5.4 mm and the maximum slit width of 2.0 mm. The sample image at the adjustable diaphragm is magnified 25×. A viewing system consisting of a transfer lens and a 7× eyepiece can be used to observe the image of the sample at the adjustable diaphragm by moving the viewing mirror into the light path. The diaphragm is adjusted to frame the sample while the sample is being observed at 175× magnification. An image of the exit slit is provided in the sample space by an auxiliary diffuse light source so that the sample may be accurately centered.

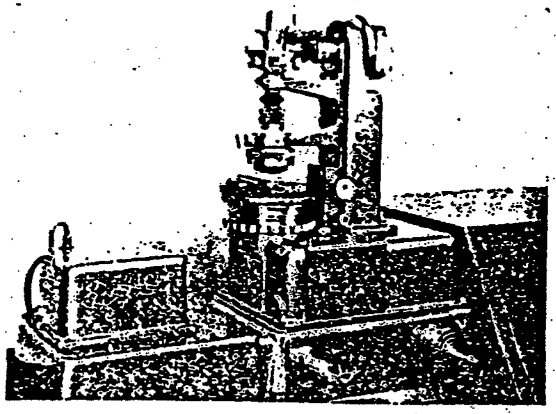

Fig. 1. Infrared microscope attachment prototype mounted on a Perkin-Elmer Model 112 spectrometer.

The system is composed entirely of reflecting elements so it is free of chromatic aberration. In addition, the useful wavelength range is not limited by absorption in refracting elements.

Infrared microscope attachments have been placed in two positions in the spectrometer: 1. Between the source and the entrance slit of the monochromator. 2. Between the exit slit of the monochromator and the detector.

In the first case, the undispersed radiation of the source is condensed onto the sample. This necessitates the use of special techniques[9] for samples which are sensitive to heat or photochemical effects. The present microscope has been placed in the dispersed beam between the exit slit and the detector, thus avoiding this disadvantage.

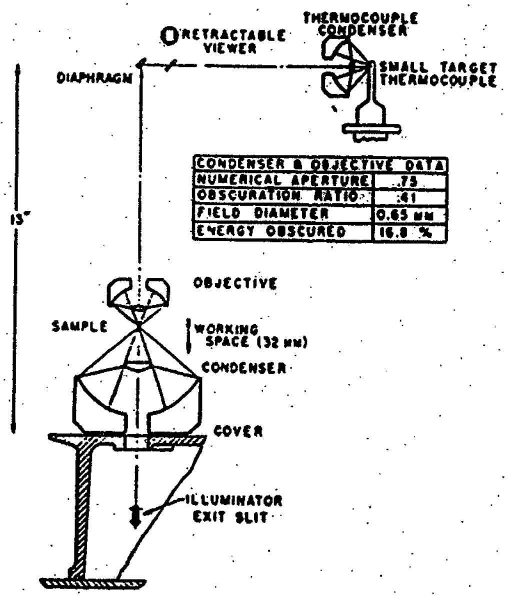

Fig. 2. Schematic optical layout.

The objective and condenser each consist of two spherical mirrors of the Schwarzschild type.[16] When such a system is monocentric and used with its pupil at the common center of curvature of the two mirrors, the image is coma-free and stigmatic. The useful field that can be covered by this form of objective is limited only by the field curvature and vignetting. The obscuration ratio of such systems is relatively high, but it can be shown[17] that the principal effect of this is to throw energy from the Airy disk into the first bright ring of the diffraction pattern. Since increasing the numerical aperture reduces the diameter of the diffraction pattern, high obscuration ratio can be compensated for by large numerical aperture. Thus with an objective of numerical aperture 0.75 and obscuration ratio 0.4, 80 percent of the energy in the entire diffraction pattern is within a circle whose diameter is 2.3λ. With an objective of numerical aperture 0.6 and obscuration ratio 0.2, 80 percent of the energy in the entire diffraction pattern is again within a circle whose diameter is approximately 2.3λ. Despite the larger obscuration ratio, however, the greater numerical aperture of the first objective enables it to collect 40 percent more energy from a given area than the second.

The focal length of the objective was determined by the desired working distance. The numerical aperture was then set as the maximum which would result in a departure of the wave form from sphericity by one-quarter of a wavelength at the short-wavelength end of the infrared region. The numerical aperture of the objective is 0.75, and its obscuration ratio is 0.4. The focal length is 8 mm and the working distance is 14.9 mm. In combination with the large condenser this results in a total available space of 32 mm between the objective and condenser mounts.

The objective has been designed for optimum

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[16] C. R. Burch, Proc. Roy. Soc. (London) 59, 41 (1947).

[17] T. Dunham, Jr. (private communication).

TABLE I. Guide to minimum sample area.

Wave-length	Signal/noise (peak to peak)	Time constant	Spectral slit width	Length	Sample width	Area
2μ	19	2.1 sec	34 cm^{-1}	100μ	6μ	600μ^2
4	16	2.1	20	100	8	800
6	19	2.1	12	100	17	1700
8	19	2.1	6	100	25	2500
10	18	2.1	3.2	100	35	3500
12	16	2.1	3	100	50	5000
14	13	2.1	3	100	100	10000

performance in the infrared region of the spectrum and in this region its performance is limited essentially by diffraction. At a wavelength of 3 microns it will form an image of a point source in which 80 percent of the energy is within a circle whose diameter is 7 microns. At longer wavelengths, the diameter of the circle increases linearly with the wavelength.

In the visible and ultraviolet regions, however, the wavelengths are small compared to the residual spherical aberration of the objective so that this aberration limits the performance. In these regions the objective design indicates a concentration of more than 85 percent of the energy from a point source within a circle whose diameter is 11 microns.

The image of the slit formed by the thermocouple condenser falls at an accessible point in space. Use of this Cassegrain-type condenser permits the substitution of photoconductive cells, photomultiplier tubes, or other types of detectors in place of the thermocouple if they are required.

The optical design allows 5.4 mm of the slit height to be utilized with the microscope. Since this is about half the macroscopic slit height, the height of the thermocouple target was reduced by a factor of two with a resultant gain in the thermocouple detectivity.

The optical elements are mounted on a convenient and stable mechanical system. Focusing and locking adjustments are provided for the objective. Positioning of the sample is accomplished by coarse and fine focusing adjustments, rotation of the stage and x and y translation using a mechanical stage. The illuminator and viewer allow accurate and reproducible positioning of the sample. The adjustable diaphragm, used to restrict the field to the area covered by the sample, is rectangular in shape and its length and width are independently and continuously variable. At this point there is also available a slide which allows insertion of special diaphragms cut as masks for irregularly shaped samples.

The microscope attachment is mounted on the monochromator cover. It is designed so that all the standard macroscopic functions of the spectrometer are preserved and the change from macro-to-micro operation may be made quickly. Access is provided for easy prism interchange. In addition, provision is made for flushing the optical path with dry nitrogen to reduce CO_2 and water vapor absorption.

SAMPLE SIZE AND SPECTRAL DILUTION

There are two fundamental points which should be mentioned in discussing the operation of a microspectrometer. The first involves the size of sample necessary for obtaining useful spectral data, while the second concerns the purity of the radiation striking the microspectrometer detector.

In practice, sample size is restricted by limitations on both thickness and cross sectional area. The thickness of a particular sample is restricted to a certain range in order to obtain the proper strength of infrared absorption. The required thickness is almost the same as for macroscopic work, very frequently being about 25 microns, a convenient value for infrared microspectrophotometry. The minimum sample area is that required to provide sufficient energy for satisfactory detection. A variety of factors affect this energy, some determined by the instrument and others set by the operator. The instrument fixed parameters are (1) source brightness, (2) optical geometry, (3) transmission efficiency of the optical system, and (4) detector detectivity.

It can be shown that for a well designed system and a fixed set of operating conditions, the minimum sample area is inversely proportional to the source brightness, the transmission efficiency, the detector detectivity, and the square of the effective numerical aperture of the microscope objective. These relationships show the importance of using as large a numerical aperture as possible, of increasing detector detectivity, and of optimizing transmission efficiency, all of which have been done in the present instrument. A significant decrease in minimum sample area can be made by using a high intensity infrared source (carbon arc,[18] zirconium arc,[19] or tungsten glower[20]) although as yet this has not been incorporated in this instrument. Additional reduction in sample size may be realized by employing photoconductive detectors in their limited spectral ranges.

The operator must strike a compromise between (1) cross-sectional area, (2) amplifier band pass and thus scanning rate, (3) signal-to-noise ratio, and (4) spectral resolution.

For any instrument, the minimum sample area is directly proportional to the square root of the amplifier band pass, directly proportional to the signal-to-noise ratio and inversely proportional to the spectral band width. The ability to adjust these quantities enables the operating conditions to be chosen so they are best for each particular sample.

Because of the variety of factors effecting energy discussed above and their variation with wavelength, no single minimum sample area can be stated as the

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[18] C. S. Rupert and J. Strong, J. Opt. Soc. Am. 40, 455 (1950)

[19] M. B. Hall and R. G. Nester, J. Opt. Soc. Am. 42, 257 (1952)

[20] Taylor, Rupert, and Strong, J. Opt. Soc. Am. 41, 626 (1951)

limit even for a single instrument. Nevertheless, for all infrared microspectrometers so far constructed, it is possible to choose a set of reasonable operating conditions and find that the sample area required to pass an adequate amount of radiation throughout the infrared range is considerably larger than the ultimate limit on sample area which is imposed by diffraction.

It is possible therefore to assume a particular set of operating conditions for the present instrument and to state the area limit for these conditions. This has been done here in order to provide a *guide* to minimum sample area. Obviously other "limits" would obtain with a different choice of operating conditions. It should also be emphasized that these values pertain to the present experimental setup and could be improved as mentioned above. The data which neglect diffraction effects are given in Table I.

If the energy reaching the detector is reduced by scattering at the sample, reflection losses at the surfaces of a sample mounting slide, introduction of a polarizer, or by any other cause, adjustment of operating conditions is required to offset the loss.

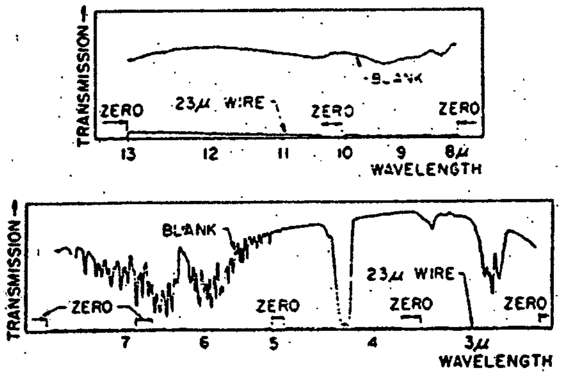

FIG. 3. Impurity radiation. No sample was used for the background run while a 23μ diameter wire was placed at the sample position for the dilution test. The diaphragm was set for an opening 15×650μ.

The question of the purity of the radiation reaching the detector will now be discussed. If the sample does not cover the entire field of the microscope, some radiation will reach the detector without being subjected to absorption by the sample. This radiation "dilutes" the spectrum and may be termed impurity radiation. Dilution of the spectra can result not only from sample misalignment, but also from the diffraction and aberrations introduced by the objective.

Consider a point source at the sample plane. At the plane of the diaphragm the energy from the source will not be concentrated at one point, but rather will be spread over an area as a result of the diffraction and the aberrations of the objective. If, now, the point source is situated just beyond the edge of a sample and if the diaphragm is closed down so that the sample is just framed, some radiation from the point source will pass through the diaphragm opening and will constitute dilution.

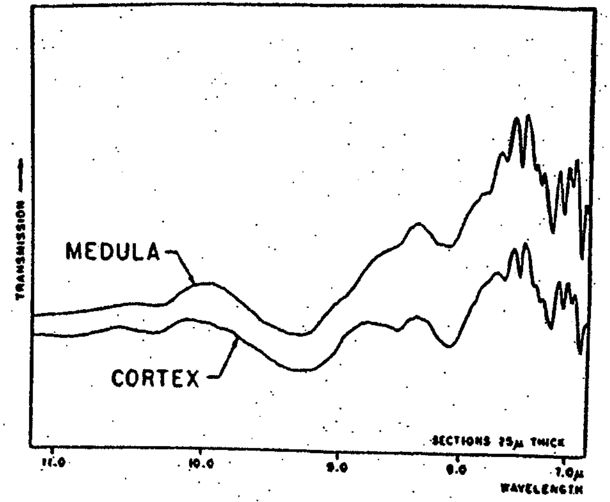

FIG. 4. Tissue spectra. Regions of the cortex and the medula of the normal rat adrenal section mounted on an AgCl slide served as samples.

The reduction of such impurity radiation is especially important for quantitative work involving long, narrow samples such as fibers. This infrared microscope provides for such reduction in several ways. The viewer system allows precise alignment of the slit image, sample, and diaphragm opening. The diaphragm is adjustable so that it can be made slightly smaller than the sample image. The large numerical aperture of the objective reduces the size of the diffraction pattern. The objective is free from coma, astigmatism, and chromatic aberration. The spherical aberration is ¼ wave at the short-wavelength end of the infrared spectrum and becomes negligible at long wavelengths.

The dilution for the case of a long, narrow sample was measured using a wire 23μ in diameter at the sample space. The diaphragm was set to an opening 15μ wide and 650μ long. Figure 3 shows the background energy and the dilution which occurred with the wire in place. The dilution increased with wavelength as is to be expected from the enlargement of the diffraction pattern and from the increase in the width of the illuminated field as the slits were progressively opened. In this extreme case, the impurity radiation was 1 percent of the incident energy at a wavelength of 2, 4, and 6μ, 2 percent at 8μ, 3 percent at 10μ, and 5 percent at 12μ. With wider samples the dilution becomes negligible.

PERFORMANCE

The work reported here was performed with the infrared microscope mounted on a Perkin-Elmer Model 112, single-beam, double-pass spectrometer. The standard globar source, a 60° NaCl prism, and the usual amplifying and recording system were used. The detector was a Perkin-Elmer small target (1×0.2 mm) thermocouple designed for use with the microscope. Using the full field of the microscope and identical slit spectrum and the recording time was again 15 minutes. It is shown by the complete CS_2 absorption at 6.5μ that no observable dilution occurs with this size sample.

Crystal Spectrum

Meta-nitrochlorobenzene was used to obtain a crystal spectrum primarily to illustrate the use of a silver chloride polarizer in conjunction with the microscope. The standard six sheet polarizer was inserted between the source and the monochromator. The sample was rotated 90° between the running of each of the spectra shown. Marked dichroism indicating bond orientation may be observed in the figure. While the crystal filled the field of the microscope, a diaphragm of $150\times650\mu$ was used.

ACKNOWLEDGMENT

We would like to thank Dr. H. P. Schwarz of the Philadelphia General Hospital who provided the tissue samples and Dr. E. R. Blout of the Polaroid Corporation and the Children's Medical Center who furnished the silver chloride capillary tubing. We would also like to thank Mr. George Brueske of the Perkin-Elmer Corporation who designed the mechanical system and Mr. Herman Ecker of The Perkin-Elmer Corporation who constructed the instrument.

JOURNAL OF THE OPTICAL SOCIETY OF AMERICA VOLUME 43, NUMBER 11 NOVEMBER, 1953

A Variable Thickness Liquid Absorption Cell

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(Received July 27, 1951)

A VARIABLE thickness liquid absorption cell of the piston and cylinder type driven by a differential nut was recently described by White.[1] The performance of this cell is satisfactory in all respects, but certain difficulties arise in its construction. The fitting of the parts is a time-consuming and tedious process; the assembly is sensitive to foreign matter which causes the cell to bind. A new design which retains the desirable performance characteristics but of simpler construction than the previous cell has therefore been attempted.

Three approaches were considered. A bellows type was designed and constructed. Satisfactory operation was obtained from this cell, but cleaning the cell after use proved to be difficult because the liquid became trapped in the bellows corrugations. Furthermore, large amounts of sample were required.

A glass syringe type has been considered and has reached the design stage. Unavailability of accurately ground syringes of the proper dimensions has temporarily held up further investigation of this design.

The cell (Fig. 1) represents the most successful design we have achieved up to this time. The windows, W_1 and W_2, are mounted in the manner originally discussed by White.[1] The large window, W_1, is pressed against the lapped face of the cylinder while the smaller window, W_2, is ground to mate exactly with the spherical metal seat. This method allows simple assembly and adjustment of the window for parallelism while providing an excellent seal. A ring of Teflon, T, prevents leakage between the stainless steel piston and cylinder. This ring is machined to fit the cylinder with a clearance of the order of 0.0005 of an inch. Because Teflon has good bearing properties, the piston runs smoothly with respect to the cylinder. If necessary, replacement of the Teflon ring can be accomplished quickly. The land, L, acts as a guide to prevent wobble in the assembly. A Woodruff key, K, prevents rotation of the piston with respect to the cylinder. The spring, S_1, minimizes backlash as the piston is pushed or pulled back and forth. It also acts to prevent excessive pressures if the two windows are brought together. The spring, S_2, removes backlash in the threads. The screw thread has a pitch of 1 mm and was cut on a lathe with normal care. The bronze nut, N, was cut on the same lathe and fitted for minimum play. The nut is long enough to average out systematic errors in the threads.

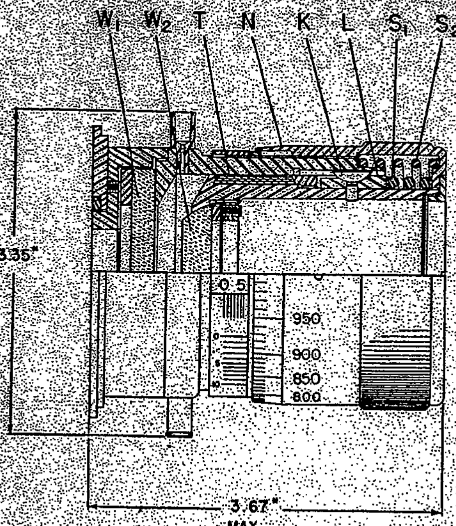

FIG. 1. Variable thickness liquid absorption cell. W_1, fixed window; T, Teflon piston seal; K, Woodruff key; S_1, piston anti-backlash spring; W_2, adjustable window; N, micrometer nut; L, guide land; S_2, micrometer screw anti-backlash spring.

The outside circumference of the nut is divided into 100 divisions, each division representing 0.010 mm. The adjustable micrometer scale reads 6 mm maximum opening and is provided with a vernier which allows the scale to be read to 0.001 mm. The cell may be disassembled for cleaning by unscrewing the nut, N. The cell then separates into two parts revealing the cell chamber. This process does not change the calibration index if the parts are handled carefully.

The maximum cell dimensions are 3.35 in.×3.67 in., as shown. Thus the cell is capable of fitting into relatively small spaces. The maximum clear aperture for a collimated beam is 25 mm.

In order to test the accuracy of the cell, visible and infrared interference fringes were used. The windows were adjusted to parallelism while observing the reflected fringes under a mercury arc. Accurate thickness measurements[2] were made in the infrared by recording the fringe pattern. Measurements of several repeated settings in the opening direction for six different thicknesses between 0.020 mm and 2.0 mm indicated a maximum departure from the mean of 0.002 mm. Approached from the opposite direction a maximum error of 0.010 mm was observed. Reproducibility after rough handling was within 0.002 mm after returning to zero and opening to the same thickness. A plot of absorbance *versus* sample thickness showed a maximum departure of 0.004 absorbance unit in the range where Lambert's law holds. Ten hours after filling the cell with chloroform and placing stoppers in the hypodermic shanks, the meniscus had not appeared in the window space, indicating negligible leakage. The cell performed satisfactorily as a solvent compensating device on a double beam infrared spectrophotometer and it is anticipated that the cell will prove to be the most useful for this purpose.

It is felt that the present design retains all the excellent performance characteristics of the previous cell while minimizing many of the construction problems.

I wish to acknowledge the assistance rendered by Mr. J. Gregory in the design of this cell. I wish also to thank Mr. J. Ferguson whose practical suggestions proved of great value.

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[1] J. U. White, Rev. Sci. Instr. **21**, 7 (1950).
[2] D. C. Smith and E. C. Miller, J. Opt. Soc. Am. **34**, 130 (1944).

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HALLEY MOGEY INVENTION GAINED EARLY RECOGNITION

Historical note on Perkin-Elmer

By SAMUEL S. CROSS, JR.
Secretary and General Counsel

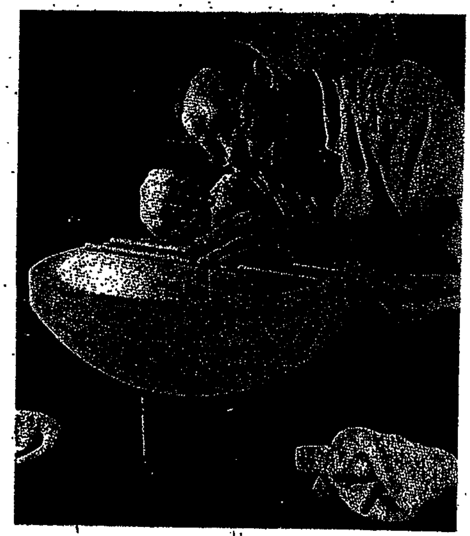
In an unassuming way, November 28, 1961, marked a turning point of an era for Perkin-Elmer. On that day United States Patent No. 2,363,544 expired, thus placing in the public domain Halley Mogey's invention of a block for finishing roof prisms.

Roof prisms occupy a special place in the history of Perkin-Elmer. It was through them that accounted for the Company's first recognition in military circles. The roof prism was designed in the 19th century by the Italian astronomer Amici and has the quality of inverting an image without reversing it. At the same time, it will deviate the line of sight through a desired angle. The critical dimension is the 90° "roof" angle.

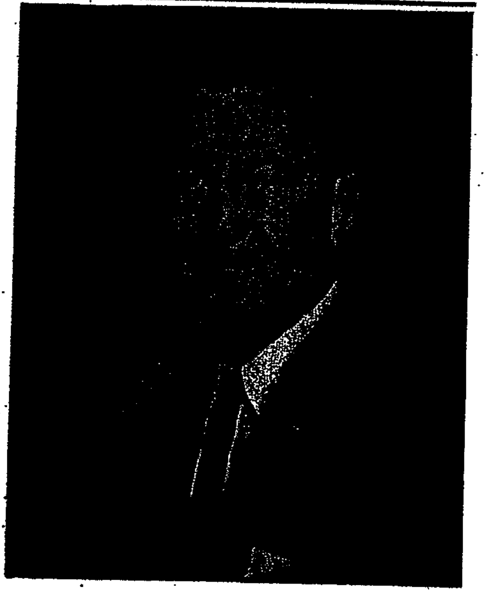
As the shadows of war approached in 1940 the Army began to order such elements in quantity for use in artillery optical instruments. Procurement officers, learning of the Company's achievement, encouraged a move in the fall of 1941 from its humble cellar headquarters in Hoboken, New Jersey to a new plant in Glenbrook, Connecticut, built by the Company.

In October, 1941 thirty persons were employed in the Glenbrook plant. In the "front office" were Mr. Perkin, just turned 35, his elderly "partner" Mr. Elmer, and Evvie Connors, now Mrs. Graham Wallace. Lloyd McCarthy, age 30, and Dick Kinnaird, age 29, were the optical designers. Ted LaLime, then also 29 (now on leave of absence), was foreman of the

(*Continued on Page 8*)



Halley Mogey, examining an 18-inch shell for a Super-Schmidt Meteor Camera.



R. A. Deevey Appointed Director of Industrial Relations

The appointment of Robert A. Deevey as Director of Industrial Relations was announced in November by T. F. Talmage, Administrative Vice President.

Mr. Deevey joins P-E from Continental Can Company where he was Manager of Industrial Relations for the Eastern Metal Division. Before joining Continental Can in 1945 he was with Curtis Wright Corporation.

Mr. Deevey was educated in New England at the Mount Hermon School in Massachusetts and Wesleyan University in Connecticut. He is married and has three children.

Reminder —

Keep Saturday, December 16th open. Plan to take your children to the EAA Children's Christmas party. Details on page 2.

Construction Underway For Atomium Plant

Construction is underway in Billerica, Massachusetts for a 12,000 square foot plant for our affiliate company, the Atomium Corporation.

P-E recently purchased a seven and one-half acre site in Billercia. The Atomium plant is being built on two acres, while the balance of the property will be used for future company needs.

The new Atomium plant is scheduled for occupancy in April, 1962. The company currently occupies a plant in Waltham, Massachusetts for the design, development and manufacture of nuclear instruments.

First of Three Company Yule Parties a Success

Two-hundred seventy five couples attended the first of the three company Christmas parties at the Longshore Country Club on Friday evening, December 1st. Many hailed the party as the most successful since P-E began sponsoring the evening several years ago. Music for dancing for the parties is being provided by Guy Masella's Orchestra.

The second party will be held on Saturday evening December 9th for E-O Engineering and Research, International Operations, and the Legal, Public Relations, Market Research, Industrial Relations and Purchasing Departments. The final party will be held on Friday evening, December 15th for the Instrument Division and Plant Engineering.

According to Jack Dietter of the Industrial Relations Department, who is coordinating the parties, reservations are still being taken for the December 15th party for employees who were unable to attend on the evenings scheduled for their groups.

Werner Woschetzky Heads Vernistat Design-Drafting

Werner Woschetzky was recently appointed Supervisor of the Design-Drafting Section of the Vernistat Division's Engineering Department. Before joining P-E, Werner had been Chief Engineer and later Assistant General Manager of American Powered Metals Company. He holds a degree in Mechanical Engineering.

Historical Note On Perkin-Elmer

(*Continued from Page* 1)

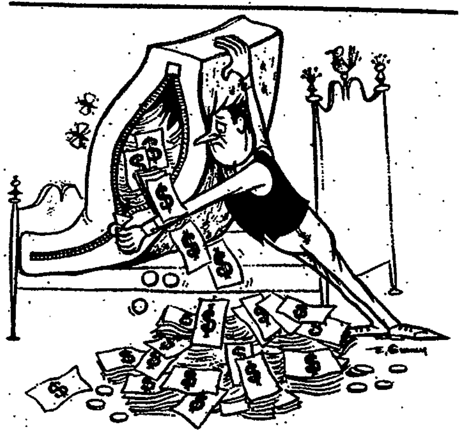
Grinding Room with six grinders including Art Harman. Halley Mogey, age 33, was foreman of the Polishing Room with six polishers, including Jack Roos, Ed Best, Al Van Sickle and Johnny Esposito. Ted Talmage, then 24, was also with the firm, engaged in accounting work. Within two years the ranks had swollen to 140 persons.

Shortly after moving to Glenbrook, a patent application on Halley's invention was filed and assigned to Perkin-Elmer. It covered use of a triangular block instead of a rectangular one for grinding and polishing roof prisms. The block was thin to provide a low center of gravity to permit machine fine-grinding and polishing. One side of a prism roof was optically contacted with a polished side of the block and auto-collimated light from the top face of the block was used for testing angular accuracy.

Does this sound old hat? If so, put yourself back twenty years in the art and take another look. The invention and allied techniques represented a major break-through in the domestic industry and permitted the manufacture *in production quantities* of optical elements equal to or better than those then available only in Germany. Perkin-Elmer pursued a liberal licensing program, which has marked its policy to present day. It granted to Bausch & Lomb and to Keuffel & Esser royalty-free licenses under its techniques for the duration of the national emergency.

The roof prism finishing block was not the first Perkin-Elmer invention to be patented. Nearly two years earlier (and only eight months after incorporation of Perkin-Elmer) an application was filed on Lloyd McCarthy's design of a lens objective for periscopes, range finders, telescopes and similar purposes. That patent expired three years ago.

In May, 1942 George Brueske (now an E-O consultant) joined Perkin-Elmer as an optical instrument designer. He soon designed a range finder which became the subject of two patents which will expire next June. Two years later, Jim Baker (now an occasional consultant) began to design lens systems for the Company. These two men were responsible for introducing Perkin-Elmer's second era of growth into systems and instruments.



Our friend above, 'though in solid financial shape for Christmas, has resorted to an old fashioned means of saving. A less risky and more convenient way to save for Christmas shopping is through the Credit Union Christmas Club Plan. Stop by the CU office to find out how you can join.

Youngsters Have Their Say . . .

While touring the plant with members of his Cub Scout Den, a nine-year-old was asked by a tour guide if he could explain what a prism is.

His reply: "It's a place where they keep bad men."

* * *

An E-O Division engineer was recently invited by an area junior high school to speak to a class on the Solar System. Dressed in a charcoal gray suit and weighted down with a slide projector, attaché case and other material for his talk, the P-E speaker entered through a rear door of the classroom. Before he could step to the front of the room one of the students, in a loud whisper, asked him if he was the speaker. The P-E engineer replied in the affirmative and added that his subject was the sun. Satisfied with the answer, the student turned to the boy seated next to him and explained, "See! I told you he wasn't a magician."

Heired

Bob Prescott, E-O Accounting, a son, Edward Stephen, November 2nd.

Gerhard Koch, E-O Polishing, a son, Gerhard, October 11th.

Ed Larson, ID Manufacturing, a daughter, Diane Karen, October 29th.

Walt Ducharme, ID Final Test, a son, John Arthur, October 26th.

Tony Patricelli, Shipping, a daughter, Jacquelyn Lynn, October 17th.

Ed Orleman, E-O Machine Shop, a daughter, Lori, November 11th.

Stan Szamatulski, Vernistat Division, a son, David Michael, November 4th.

Paired

Connie Green, ID Marketing, to Dom Pasquale, E-O Projector, November 25th.

International Sales Meeting Held at Perkin-Elmer Ltd.

Representatives from European sales offices and manufacturing sites and International Operations in Norwalk attended an International Sales meeting at Perkin-Elmer Limited, Beaconsfield, England from October 25th through the 27th. Dr. Desmond Orr, Sales Manager of P-E Limited conducted the meetings.

Much of the discussion concerned the new analytical instruments introduced by Perkin-Elmer in 1961. Prior to the meeting, a training course was presented to the salesmen on the new Nuclear Magnetic Resonance Spectrometer, which is being produced at Beaconsfield. The course was conducted by Dr. John Leane, one of P-E Limited's NMR experts.

—Don Lawrence, P-E Ltd.

International Sales meeting in progress: from bottom left and continuing clockwise—Vince Coates, IO Norwalk; Gino Caroti, Milan Sales Manager; Dr. Guenter Raupp, Frankfurt Applications Engineer; Ralf Jutvik, Sales Manager, Sweden Office; not identified; A. Ralph Gilson, Managing Director, P-E Limited; Paul Strauss, IO Norwalk; Dr. Peter Zoller, Sales Manager, Zurich Office; Guenter Schalkhammer, Zurich; Jean Cayron, Order Supervisor, Paris Office; Igor P. Vassilieff, Paris Office Manager; Mr. Ritter, Director of Production, Bodenseewerk; Alfred Loew, Export Manager, Bodenseewerk; August Heinzle, Managing Director, Bodenseewerk; Franz Nies, Bodenseewerk Sales Administrator; Dr. Dietrich Jentzsch, Gas Chromatography Development, Bodenseewerk.

Projector Leads P-E Men's Bowling League

Projector maintains a narrow lead in the P-E Men's Bowling League with a 32-12 record thus far. ID Engineering holds second place with 29 wins and 15 defeats. Below is a re-cap of league standings to date and individual leaders, prepared by Dick Ward, league secretary.

Projector	32	12	.727
I.D. Engineering	29	15	.659
Post Road No. 1	27	17	.614
Polishing	22	22	.500
Electronic Assembly	22	22	.500
Sheet Metal	20	24	.455
E-O Model Shop	19	25	.432
Post Road No. 2	18	26	.409
Westport Ave.	17	27	.386
E-O Assembly	14	30	.318

High single games: Ben Troidle, Projector, 227; Ronald Kupan, Projector, 213, and Ted Taylor, E-O Model Shop, 207. High three: Ben Troidle, Projector, Bob Dulin, I.D. Engineering, 580; John Hayes, Westport Ave., 553; Ted Taylor, E-0 Model Shop, Ronald Kupan, 525.

Thanksgiving Dance Draws Capacity Crowd

Laddins Terrace in Stamford was filled to capacity on Friday evening, November 17th, as employees, their husbands and wives and friends attended the Thanksgiving Dance sponsored by the Employees Activities Association. The successful affair, one of two dances sponsored by the EAA during the year, was arranged by EAA President Len Patricelli and the EAA General Committee.

E-O Division Groups Move To Wilton Plant

Employees of the Electro-Optical Division had an opportunity on Thursday, December 7th to tour the new South Wilton plant, which is nearing completion.

Some Division groups have already occupied portions of the second floor of the Engineering building. Groups were moved to the new modern facility during the previous weekend.

P-E Represented at Inter-American Meeting

Harry Hausdorff, Director of Marketing of International Operations, and George Sigmund, IO Sales Correspondent, were in San Juan, Puerto Rico during the latter part of October for the First Inter-American Conference of Chemical Engineers.

In addition to presenting technical papers at the Conference, Harry and George manned the Perkin-Elmer booth (see photo). On display were a Model 154-D Vapor Fractometer and a Model 237 spectrophotometer. Harry read a technical paper entitled "Quantitative Aspects of Gas Chromatography," which was written by Leslie Ettre, ID Applications Engineering. George's paper concerned, "Micro-Sampling in the Ultra Violet Region," authored by Tom Porro, ID Applications Engineer.

Conference attendees were present from Latin America, South America, Puerto Rico and the United States.

Bill Snyder Assists with School Weather Project

Some sixty eighth grade students at Benjamin Franklin Junior High School in South Norwalk recently conducted a project to learn how weather balloons are launched, tracked and used by meteorologists to determine wind velocity and direction. Bill Snyder, of E-O Engineering, provided technical assistance on the project, called "Operation Weatherman", and operated a P-E alignment theodolite.

Four helium-filled pilot weather balloons were launched. With data taken at precise one-minute intervals, the angles of elevation and azimuth will be used by the students to compute the distance traveled by each balloon, wind velocity and direction at various altitudes, and shifts in wind during each flight.

INTERNATIONAL OPERATIONS

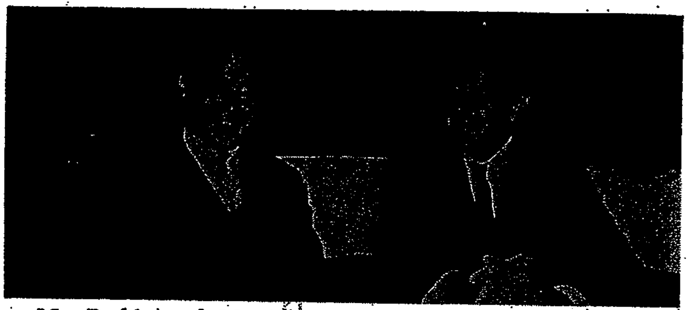
Hitachi Perkin-Elmer, Ltd. Meeings Held in Japan

In October, an official reception was held in Japan to recognize the formation of Hitachi Perkin-Elmer Limited (HIPE), a new company jointly owned by Perkin-Elmer and Hitachi, Perkin-Elmer, Limited, of Tokyo, Japan. Hitachi ranks as Japan's largest industrial company, and manufactures among other products, a broad line of electrical and electronic products including laboratory instruments for chemical analysis.

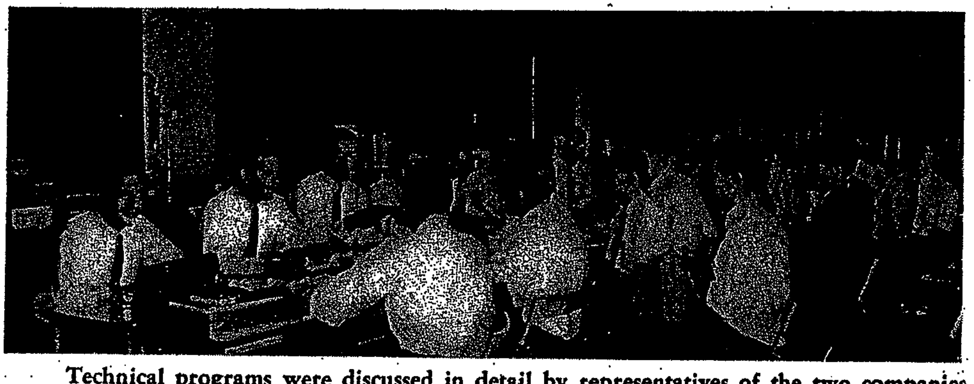
Under the terms of the HIPE agreement, Perkin-Elmer and Hitachi are cooperating on the research, development, manufacture and sale of analytical instruments throughout the Free World. Hitachi and Perkin-Elmer and their affiliated companies may cross-license one another for manufacture of scientific instruments in their respective countries and for sale of such instruments throughout the world.

Photographs below depict activities during the HIPE reception and technical conferences held in Tokyo in October.


At the reception at the Hitachi Mejiro Club, guests were greeted by, from left to right, Aki Hotta, Director and Vice President of HIPE and Director of Japanese Operations for International Operations, Mr. Kurata, Hitachi President, Mr. and Mrs. Perkin, and Mr. Kaiwa, who is Chairman of the Board and President of HIPE, and Assistant General Manager of the Hitachi Instrument Division.


Mr. Perkin and Mr. Kurata, President of Hitachi, Limited, are photographed together during the HIPE reception. The reception gave Mr. Perkin his first opportunity to meet officials of Hitachi as well as industrial, civic and commercial leaders of Japan.


On the lighter side, anglers tried their luck to the rear of the Hitachi Central Research Laboratory. The fish happened to be biting on that particular day as each fisherman pulled in a trout. Baiting hooks or wating for a nibble are Vince Coates, Dr. Tadano, a Director of HIPE, Mr. Kaiwa, Hank Brockschmidt and Mr. Perkin.


Technical programs were discussed in detail by representatives of the two companies to establish a firm working relationship. The photo above was taken during a technical conference on instrumentation at Hitachi's Naka Works. At the left from Norwalk are Nate Brenner, Vince Coates, and Heinie Kessler. In the right foreground at the table are Aki Hotta and Dr. Makino, a Director of HIPE and Director of Hitachi's Scientific Instrument Group at Naka Works.

Nov. 10, 1953 V. J. COATES **2,658,395**

DIFFERENTIAL KNOB DEVICE

Filed Nov. 15, 1951

Fig. 1,

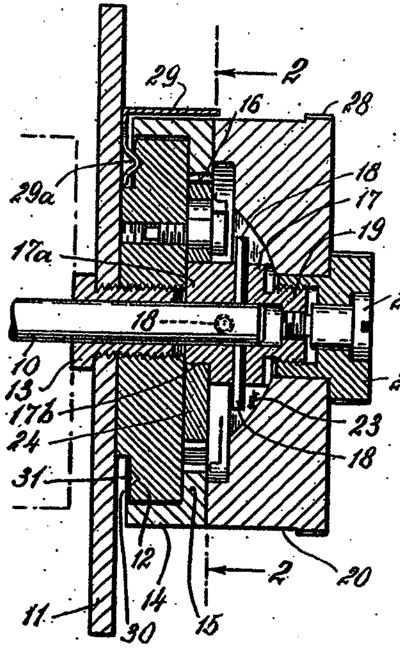


Fig. 2,

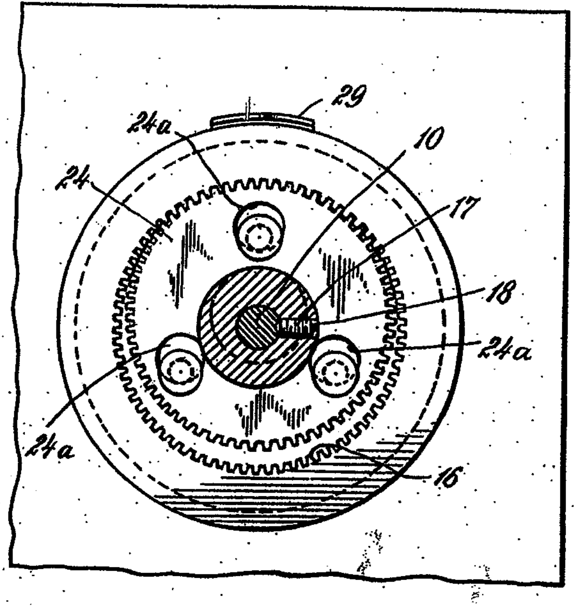


Fig. 3,

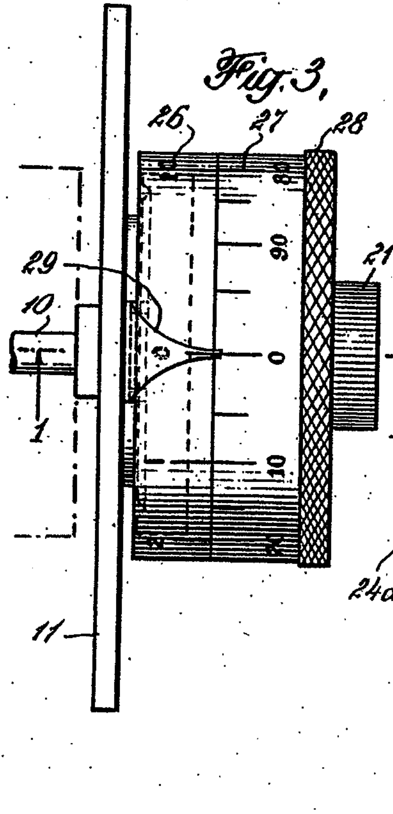
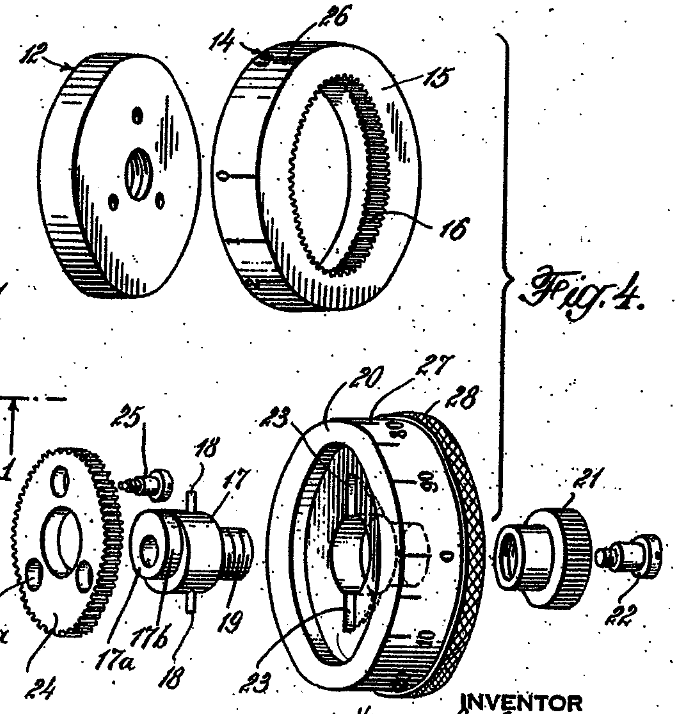


Fig. 4.



INVENTOR
Vincent J. Coates
BY
Pennie Edmonds Morton Barrows & Taylor
ATTORNEYS

Patented Nov. 10, 1953 2,658,395

UNITED STATES PATENT OFFICE

2,658,395

DIFFERENTIAL KNOB DEVICE

Vincent J. Coates, South Norwalk, Conn., assignor
to The Perkin-Elmer Corporation, Norwalk,
Conn., a corporation of New York

Application November 15, 1951, Serial No. 256,509

7 Claims. (Cl. 74—10.52)

This invention relates to differential knob devices used for rotating an element, such as a shaft, and indicating the full turns and fractional parts of a turn, through which the element is moved. More particularly, the invention relates to a novel differential knob device, which is made of a relatively few simple parts and can be readily assembled and taken apart. The new device may be advantageously employed in connection with an instrument having an adjusting shaft, which is rotated in the use of the instrument and the position of which must be exactly set and known, and a form of the device of the invention for use in such an instrument is shown in the accompanying drawing.

In the drawing,

Fig. 1 is a view, partly in elevation and partly in section on the line 1—1 of Fig. 3, of the new device;

Fig. 2 is a sectional view on the line 2—2 of Fig. 1;

Fig. 3 is a top plan view of the device; and

Fig. 4 is an exploded perspective view of the parts of the device.

The differential knob device illustrated in the drawing is for use in operating the shaft 10 of an instrument having a casing provided with a front panel 11. The shaft 10 extends through an opening in the panel and the device is mounted on the exposed end of the shaft. The device includes a base 12, which is of circular outline and has a central opening for the shaft. The base is attached to the outer face of panel 11 in any convenient manner and, in the construction illustrated, the openings through the panel and through the base are internally threaded and an externally threaded headed bushing 13 is turned through the panel opening and into the opening in the base, the tightening of the bushing causing the panel to be clamped between the base and the head of the bushing.

A coarse dial member 14 has a cylindrical section encircling the base and an internal flange 15 overlying the end of the base remote from the panel and provided with internal gear teeth 16. In the construction shown, the flange and the cylindrical section of member 14 are integral, but, if desired, the dial member may be made of a cylindrical section and an internal gear taking the place of the flange and tightly fitting within the cylindrical section.

The shaft projects through the coarse dial member and a bearing bushing 17 is mounted on the exposed end of the shaft and held in place by a set screw 18. The bushing has a section

17a having a peripheral bearing surface 17b, which is eccentric to the central bore through the bushing. The bushing is also provided with a pair of radial pins 18 and with an externally threaded end section 19 projecting beyond the end of the shaft.

A fine dial member 20 is mounted to abut the outer face of the coarse dial member 15 and the member 20 has a central opening, into which the threaded end 19 of the bearing bushing extends. The member 20 is engaged by a headed clamp screw 21 extending through an opening in the member and threaded on the end section 19 of the bearing bushing. The clamp screw holds the member 20 tightly against member 14 and holds member 14 against the base 12. The end section 19 of the bearing bushing is internally threaded and a stop screw 22, passing through a central bore in clamp screw 21 and threaded into end section 19, locks the clamp screw in place. The member 20 is provided with internal radial slots 23 receiving the pins 18 on the bearing bushing and, when member 20 is rotated manually, it causes the member 14, the bearing bushing, and the shaft to rotate with it.

An external ring gear 24 has an axial opening receiving the eccentric section 17a of the bearing bushing and gear 24 has less teeth than there are internal teeth on the flange 15 of the coarse dial member 14. The eccentricity of section 17a of the bearing bushing is such that certain of the teeth of gear 24 are always in mesh with teeth 16. The gear 24 is provided with one or more eccentric openings 24a and headed studs 25, one for each opening, are passed through the openings and threaded into bores in base 12. The portions of the studs lying within the openings in the gear are of substantially less diameter than the openings, and the heads on the studs lie outside the openings. The studs thus permit the gear to be given an eccentric motion, as the shaft is rotated, and hold the gear against axial movement.

The coarse dial member 14 and the fine dial member 20 are provided with peripheral scales 26, 27, respectively, and the fine dial member 20 may be provided with a knurled peripheral section 28, so that the member can be readily grasped and turned. A pointer 29 overlies the scales and may be mounted in place in an convenient manner. In the construction illustrated, the base is formed with a channel 30 between its inner face and panel 11 and the pointer has an end turned at an angle and extending into the channel. This end of the pointer is provided with an offset portion 29*a*, which snaps into an annular recess 31 in the radial surface of the base defining channel 30.

Since the gear 24 has less teeth than there are teeth 16 on the flange of the coarse dial member 14, the member 14 is rotated through a relatively small angle for each complete rotation of the bearing bushing 17 with the shaft. For example, if there are 72 teeth on the flange and 66 teeth on the gear, the number of revolutions of the shaft required to produce a single revolution of member 14 is equal to the quotient of 72 divided by the difference between the number of teeth on the flange and on the gear, that is, is equal to 12. Each complete rotation of the fine dial member 20 thus results in $\frac{1}{12}$ revolution of the coarse dial member 14. The scale 26 is, accordingly, selected in view of the number of gear teeth on the coarse dial member flange and on gear 24.

In the new device, the rotation of the shaft by the fine dial member causes gear 24 to move with a wobble motion, and the gear is always held in place by its contacts with the eccentric bearing surface 17*b* on bearing bushing 17 and with the three studs 25. As the fine dial member is rotated, gear 24 moves smoothly and continuously with little or no backlash and the coarse dial member 14 is turned without retrogression at any point in its rotation.

I claim:

1. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member encircling and mounted for rotation on the base and having an internal flange engaging one end of the base, the inner edge of the flange being provided with internal gear teeth, a fine dial member engaging the outer surface of the coarse dial member and having a central passage through it, a bushing within said passage having a central bore for the shaft and an external bearing surface eccentric with the bore, an external ring gear of less teeth than the number of teeth on said flange having an axial opening receiving the eccentric bearing surface with part of the teeth of the gear engaging teeth on the flange, the ring gear having an eccentric opening parallel to its axis, a projection on the base entering the eccentric opening in the gear, the projection being smaller than said opening, and means attached to the bushing and clamping said dial members together and the coarse dial member against the base.

2. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member encircling and mounted for rotation on the base and having an internal flange engaging one end of the base, the inner edge of the flange being provided with internal gear teeth, a fine dial member engaging the outer surface of the coarse dial member and having a central passage through it, a bushing within said passage having a central bore for the shaft and an external bearing surface eccentric with the bore, an external ring gear of less teeth than the number of teeth on said flange having an axial opening receiving the eccentric bearing surface with part of the teeth of the gear engaging teeth on the flange, the ring gear having an eccentric parallel to its axis, a projection on the base entering the eccentric opening in the gear, the projection being smaller than said opening, means on the bushing engaging the fine dial member and connecting the bushing and member for rotation in unison, and means attached to the bushing and clamping the dial members together and the coarse dial member against the base.

3. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member encircling and mounted for rotation on the base and having an internal flange engaging one end of the base, the inner edge of the flange being provided with internal gear teeth, a fine dial member engaging the outer surface of the coarse dial member and having a central passage through it, a bushing within said passage having a central bore for the shaft and an external bearing surface eccentric with the bore, an external ring gear of less teeth than the number of teeth on said flange having an axial opening receiving the eccentric bearing surface with part of the teeth of the gear engaging teeth on the flange, the ring gear having an eccentric opening parallel to its axis, a projection on the base entering the eccentric opening in the gear, the projection being smaller than said opening, and a clamping element mounted on the bushing and clamping the dial members together and the coarse dial member against the base.

4. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member encircling and mounted for rotation on the base and having an internal flange engaging one end of the base, the inner edge of the flange being provided with internal gear teeth, a fine dial member engaging the outer surface of the coarse dial member and having a central passage through it, a bushing within said passage having a central bore for the shaft and an external bearing surface eccentric with the bore, an external ring gear of less teeth than the number of teeth on said flange having an axial opening receiving the eccentric bearing surface with part of the teeth of the gear engaging teeth on the flange, the ring gear having an eccentric opening parallel to its axis, a projection on the base entering the eccentric opening in the gear, the projection being smaller than said opening, a clamping element mounted on the bushing and clamping the dial members together and the coarse dial member against the base, and means engaging the bushing and the element for retaining the element in fixed relation to the bushing.

5. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member encircling and mounted for rotation on the base and having an internal flange engaging one end of the base, the inner edge of the flange being provided with internal gear teeth, a fine dial member engaging the outer surface of the coarse dial member and having a central passage through it, a bushing within said passage having a central bore for the shaft and an external bearing surface eccentric with the bore, an external ring gear of less teeth than the number of teeth on said flange having an axial opening receiving the eccentric bearing surface with part of the teeth of the gear engaging teeth on the flange, the ring gear having an eccentric

opening parallel to its axis, a projection on the base entering the eccentric opening in the gear, the projection being smaller than said opening, means attached to the bushing and clamping said dial members together and the coarse dial member against the base, the dial members having peripheral scales, and a pointer attached to the base and overlying the scales.

6. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member encircling and mounted for rotation on the base and having an internal flange engaging one end of the base, the inner edge of the flange being provided with internal gear teeth, a fine dial member engaging the outer surface of the coarse dial member and having a central passage through it, a bushing within said passage in contact with the base, the bushing having a central bore for the shaft and an external bearing surface eccentric with the bore, means on the bushing for connecting the bushing and fine dial member for rotation in unison, an external ring gear of less teeth than the number of teeth on said flange having an axial opening receiving the eccentric bearing surface on the bushing with part of the teeth of the gear engaging teeth on the flange, the ring gear having an eccentric opening parallel to its axis, a stud on the base entering the eccentric opening in the gear, the stud being substantially smaller than the opening, and means mounted on the bushing and clamping the dial members together and the coarse dial member against the base.

7. A differential knob device for rotating a shaft, which comprises a base having an external bearing surface and a central opening for the passage of the shaft, a coarse dial member mounted for rotation on the external bearing surface on the base and having an internal flange at the outer end of the base, the flange having internal gear teeth, a fine dial member engaging the outer end of the coarse dial member and having a central passage through it, a bushing within the passage having a central bore for the shaft and an external bearing surface outside of and eccentric with the bore, an external ring gear of less teeth than the number of teeth on the flange having an axial opening receiving the eccentric bearing surface on the bushing with part of the teeth on the gear engaging teeth on the flange, the ring gear having a plurality of equiangularly spaced eccentric openings parallel to the axis of the gear, a projection on the base entering each eccentric opening in the gear, the projection being of less diameter than its opening, means for connecting the bushing, the shaft, and the fine dial member for rotation in unison, and means on the bushing for holding the dial members together and the coarse dial member in fixed relation to the base.

VINCENT J. COATES.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
2,167,677	Petersen	Aug. 1, 1939

Appendix: XI

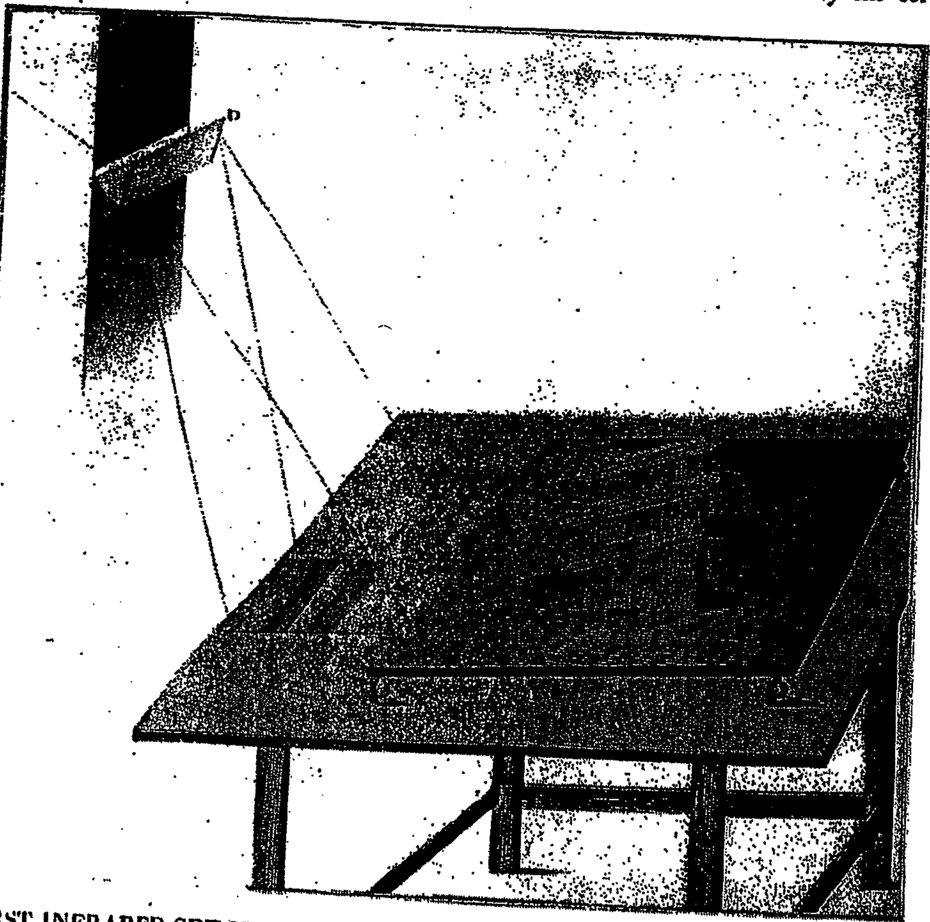
Chemical Analysis by Infrared

Electromagnetic waves make the atoms in a compound vibrate. The frequencies to which a substance responds can tell much about its structure and about the nature of chemical bonds

by Bryce Crawford, Jr.

Infrared spectroscopy has grown like a mushroom in the past 10 years. Before the war it was employed by only a few chemists and physicists, using home-built or custom-built infrared spectrometers. Now the instrument is a standard commercial item supplied by a competitive industry to chemical and medical researchers all over the country. More than 1,300 commercial infrared spectrometers, each representing an investment of two to six Cadillacs, are earning their way in scientific laboratories and industrial plants.

To an old-timer in the field—"old-timer" by virtue of having studied infrared as long as, say, 15 years—this burgeoning use of infrared by his colleagues is most gratifying, but hardly surprising. The power of infrared as a tool for chemical characterization and analysis has been known for some 20 years. The infrared spectrum of a substance is related to its chemical structure in a uniquely convenient way, and organic chemists have been heard to say that the infrared spectrum of an organic compound is its most important physical property.



FIRST INFRARED SPECTROMETER, improvised by Sir William Herschel in 1800, was the means by which he discovered infrared radiation. Thermometers placed in successive bands of solar spectrum registered more heat outside and beyond red region than within.

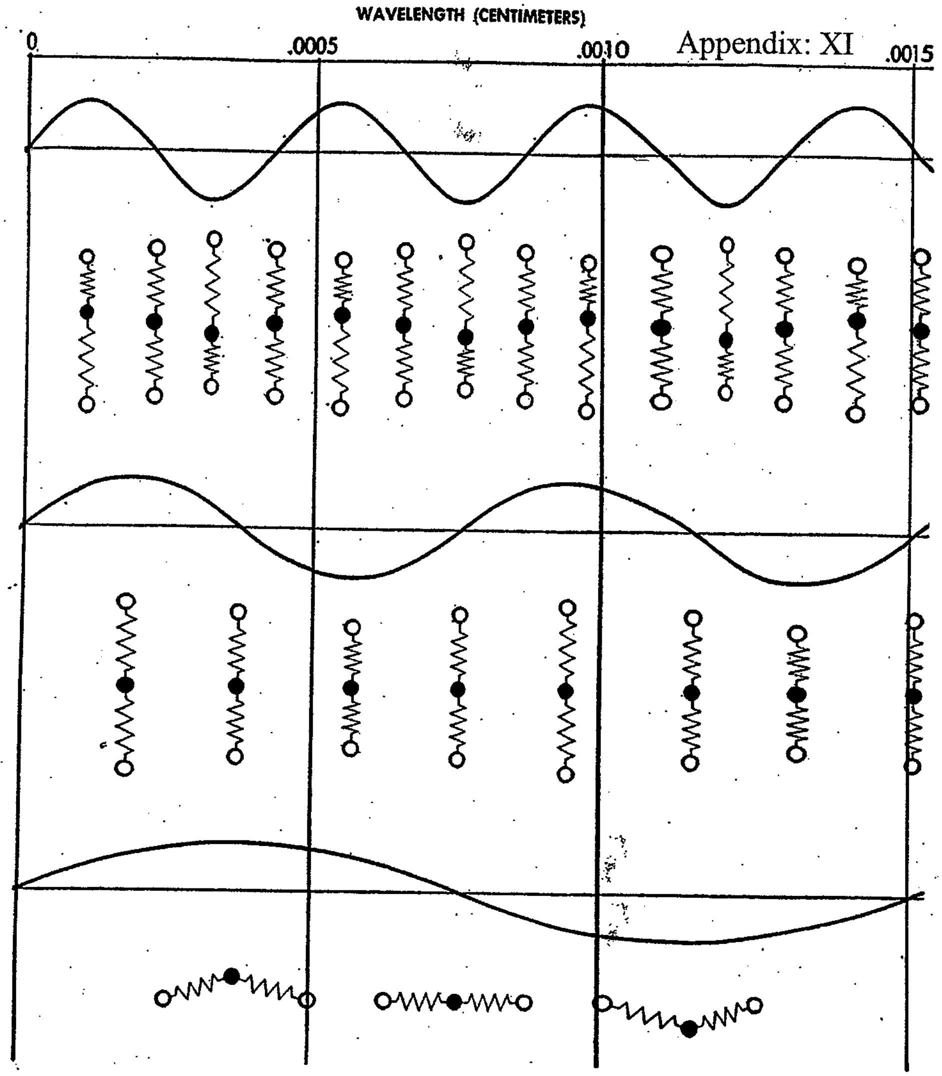
Infrared radiation itself was discovered more than 150 years ago, long before scientists had any clear understanding of radiation. Sir William Herschel, who started life as an organist at Bath and became an astronomer, made the discovery. In 1800 he reported to the Royal Society certain experiments in heat radiation. He resolved sunlight into its spectrum with a glass prism and placed a thermometer at successive positions in the spectrum. He found heat radiation not only in the visible spectrum but in the longer wavelengths beyond the red. Herschel even crudely measured the absorption of this radiation by various substances, including tap water, distilled water, sea water, gin and brandy. He could not know, could not even suspect, how revealing the absorption could be about chemical structure. Before anyone could appreciate the significance of infrared absorption, light radiation itself had to be understood. A century passed before the theory of the nature of light was worked out and the necessary techniques and instruments for infrared analysis were developed.

Sir William had found that the most intense heat radiations were outside the visible part of the spectrum. Consequently for many years it was believed that heat and light were two quite different radiations. But some 35 years later Sir William's son, Sir John Herschel, and other investigators advanced the idea that heat and light might merely be different manifestations of the same radiation—that is, light waves of different wavelengths. Before the end of the century James Clerk Maxwell had shown theoretically and Heinrich Hertz had proved experimentally the essential identity of light, heat and other electromagnetic radiation.

The infrared region, as spectroscopists usually define it, lies between the visible and the radio portions of the spectrum—that is, the wavelengths between one thousandth of a millimeter and one millimeter. (Infrared of course is not synonymous with the common meaning of the word "heat," for, as we have seen, there are heat radiations in the visible part of the spectrum.) The region most useful to the chemist is the range of wavelengths from 2 to 20 microns (thousandths of a millimeter). This is sometimes called the vibrational region.

We shall confine ourselves to this region and to considering how absorption spectra in it give information about matter. The other infrared regions, and the physics of the radiation itself, are just as interesting: it was in the near infrared that Max Planck, by instinct and gentle nature one of the most classical of physicists, was led to the disturbing idea of the quantum, which upset the whole beautiful pattern of 19th-century physics. But we cannot cover the whole subject of infrared in one article.

The chemically useful study of infrared absorption spectra was really begun in 1903 at Cornell University. A graduate student named William W. Coblentz, under the physics professor Edward Nichols, had undertaken research on infrared absorption. After improving the available experimental techniques, he received an appointment as a research associate of the Carnegie Institution of Washington, which enabled him to set about measuring absorption spectra of pure substances. He mapped spectra for two years and in 1905 published a collection of accurate infrared absorption spectra for 131 substances. Even today, after 48 years of improvements in infrared technique (to which Coblentz himself contributed much until his retirement from the National Bureau of Standards in 1945) the monumental work that he did at Cornell still stands worthy of study. Subsequent studies have confirmed more than they have corrected Coblentz's observations.

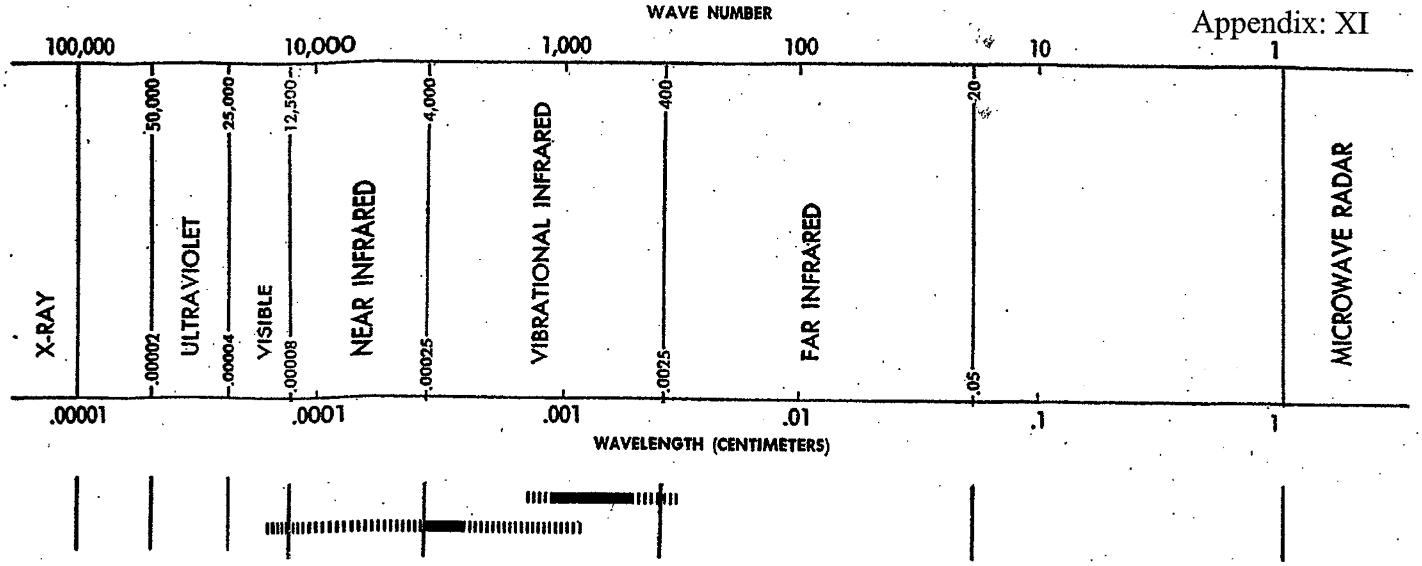


VIBRATION OF MOLECULAR BONDS accounts for selective absorption of infrared energy by various compounds. Each of three modes of vibration of carbon dioxide bonds shown here is resonant to infrared at wavelength indicated. Energy is absorbed, however, only by modes at top and bottom, which disturb geometric and electric symmetry of atom.

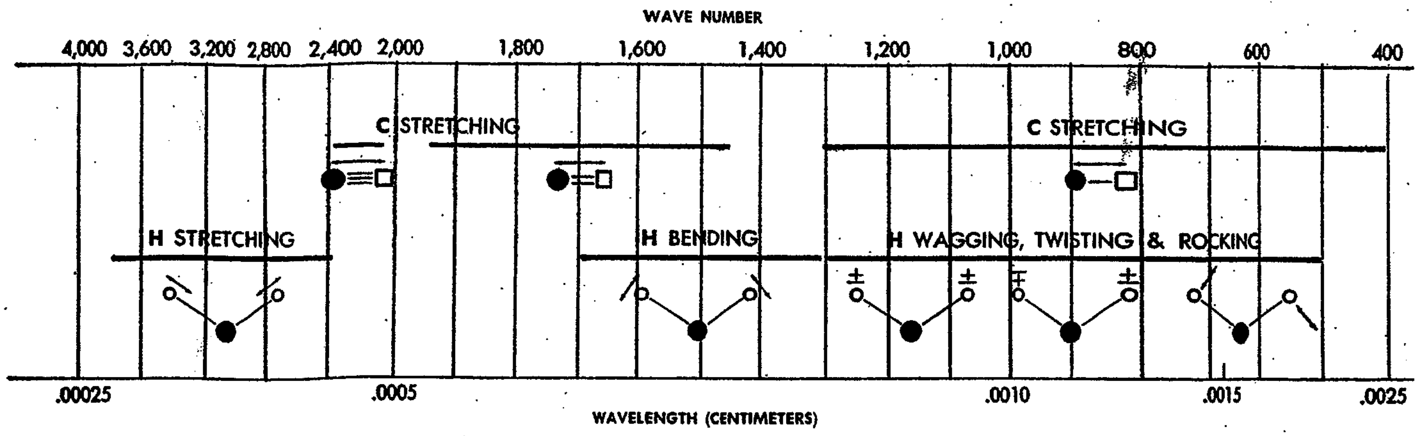
When sunlight falls upon a green leaf of a tree, the leaf absorbs the red wavelengths of the light and reflects the green wavelengths: this is what gives the leaf its color. The absorption tells something about the leaf's molecular composition. Essentially the same principle is involved in studying infrared absorption, except that the radiation is not visible; if our eyes were attuned to the infrared, we could recognize a compound by its characteristic color. Instead we measure its absorption of heat. Infrared radiation is passed through a solution of a compound, and the compound characterizes itself by the wavelengths it absorbs and those it transmits. Each compound has its own absorption spectrum.

The absorption of infrared is due to some disturbance within the molecule; this Coblentz established by observing that he got different spectra from isomers—molecules which are composed of the same atoms but in different arrangements. Coblentz found further that certain subgroupings of atoms within molecules identified themselves by absorbing characteristic wavelengths: for example, phenyl compounds, containing the benzene ring, absorbed at 3.25 and 6.75 microns, while mustard oils, containing the thiocyanate group, absorbed at 4.78 microns. These absorptions were additive: in phenyl mustard oil Coblentz found "the characteristic vibration of the mustard oils superposed upon the vibration of the benzene nucleus." He concluded that "there is a something—call it 'particle,' 'group of atoms,' 'ion' or 'nucleus'—in common, with many of the compounds studied, which causes absorption bands that are characteristic of the great groups of organic compounds, but we do not know what that 'something' is."

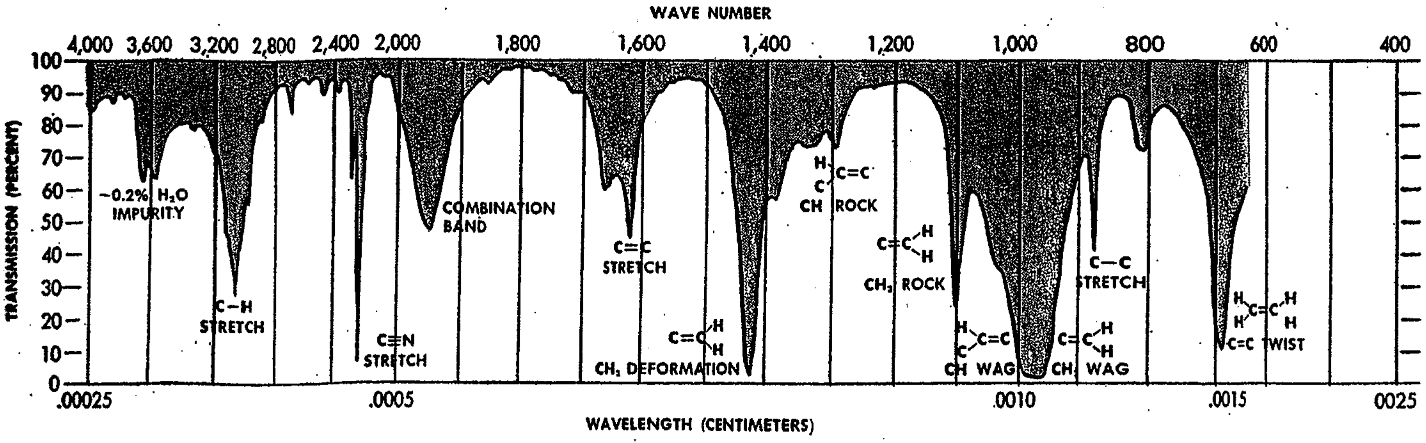
With half a century's progress since Coblentz, we now have a clear idea of the "something." It lies in the bonds

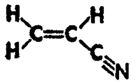


ELECTROMAGNETIC SPECTRUM is diagrammed above. Wave number means the number of wavelengths per centimeter and is proportional to frequency. Wave numbers from 400 to 4,000 have the right frequency to excite molecular vibrations. Bars below locate concentrations of heat radiation from a black body at 80 degrees F. (*upper bar*) and 1,800 degrees (*lower*). In each bar 50 per cent of the total heat energy is in the black sections, 40 per cent in the broken section, and the rest tapers off from the ends.



TYPES OF VIBRATION that cause most common absorption bands are here located in spectrum. Carbon atoms are represented by solid black circles, hydrogen atoms by small open circles, other atoms by squares. Carbon bonds stretch at the indicated frequencies whether running between two carbon atoms or between carbon and a different atom. Hydrogen wagging, twisting and rocking cover the 500 to 1,300 region jointly. Plus and minus signs for wagging and twisting indicate motion in and out of plane of page.



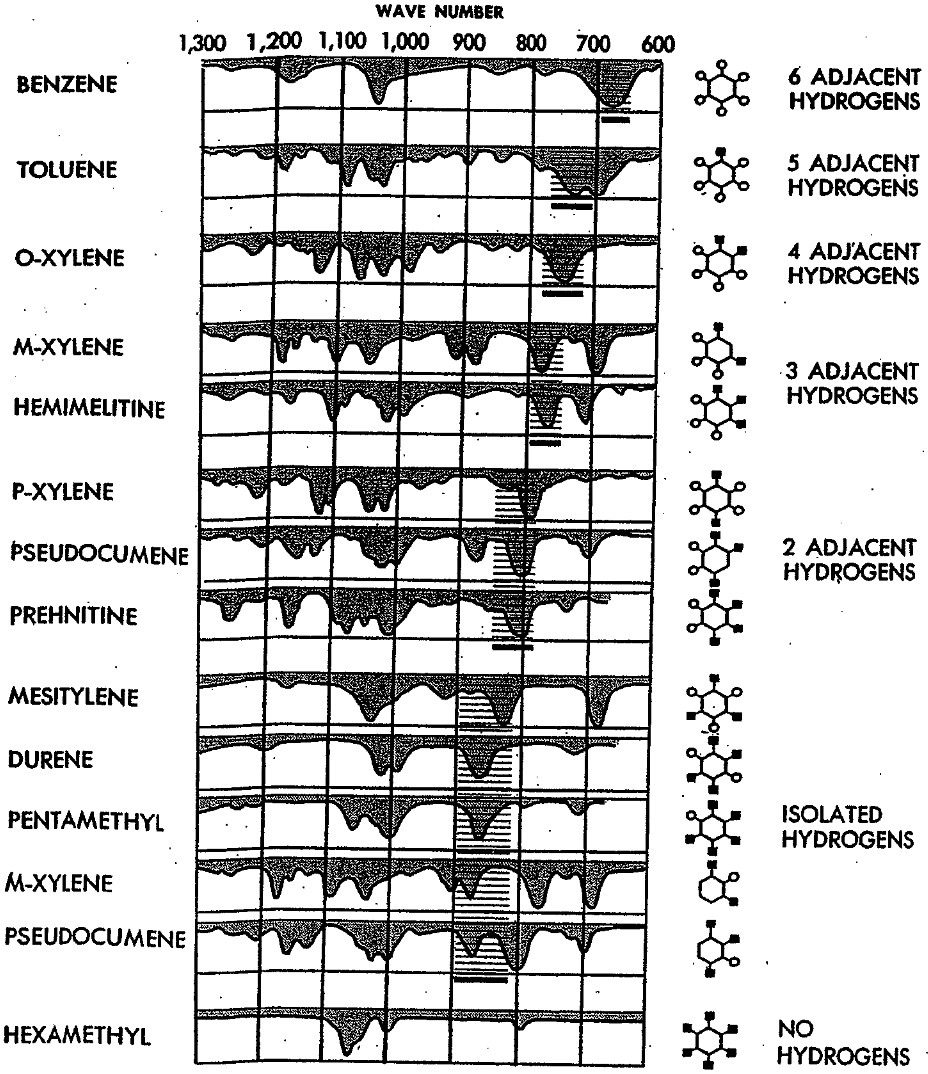


ACRYLONITRILE, whose molecular formula appears at left, gives the infrared spectrum shown above. The curve represents the amount of energy transmitted by a sample of the material as the wavelength increases (*from left to right*). Dips indicate frequencies at which energy is strongly absorbed, showing that a natural frequency of molecular vibration has been reached. Vibrations causing dips are indicated for comparison with chart above.

between the atoms in a molecule. These bonds are written in chemical formulas as single, double and triple lines connecting the atoms. As the Danish chemist Niels Bjerrum once said, they "summarize, in a very compact form, chemistry's knowledge of the creation and destruction of compounds. Nowhere in science has a shorthand notation been developed which summarizes such an abundance of exact knowledge in so small a space." In 1914 Bjerrum showed that, if we think of the atoms as small masses and the bonds as springs holding the atoms together, we can account correctly for the vibrational behavior of molecules, as observed in their infrared spectra and in their heat capacities.

The bonds hold the atoms in position fairly tightly, but not rigidly. The response of the atoms to a light wave is much like the response of cork balls, floating on a lake, to waves on the lake. As waves move past the ball, they push the ball alternately up and down. So a light wave moving past an atom sweeps an oscillating electric field over it, and if the atom carries an electrical charge it will be pushed first one way and then the other. Atoms in general do carry a charge, greater or lesser according to the molecule; thus in the hydrogen chloride molecule the hydrogen atom carries a small positive charge, the chlorine atom a corresponding negative one. Because of these opposite charges, the electric field of the light wave will push the two atoms in opposite directions, and will tend to set them into vibration, stretching and compressing the H-Cl bond alternately.

The bond has a natural frequency of vibration, determined by the masses of the two atoms and the restoring force of the bonds. A light wave with this frequency of oscillation will have most effect on the bond: its energy will greatly increase the natural vibrations of the atoms. The molecule will absorb part of the energy of the light at this resonant frequency, and an absorption detector will show an absorption peak for that wavelength.



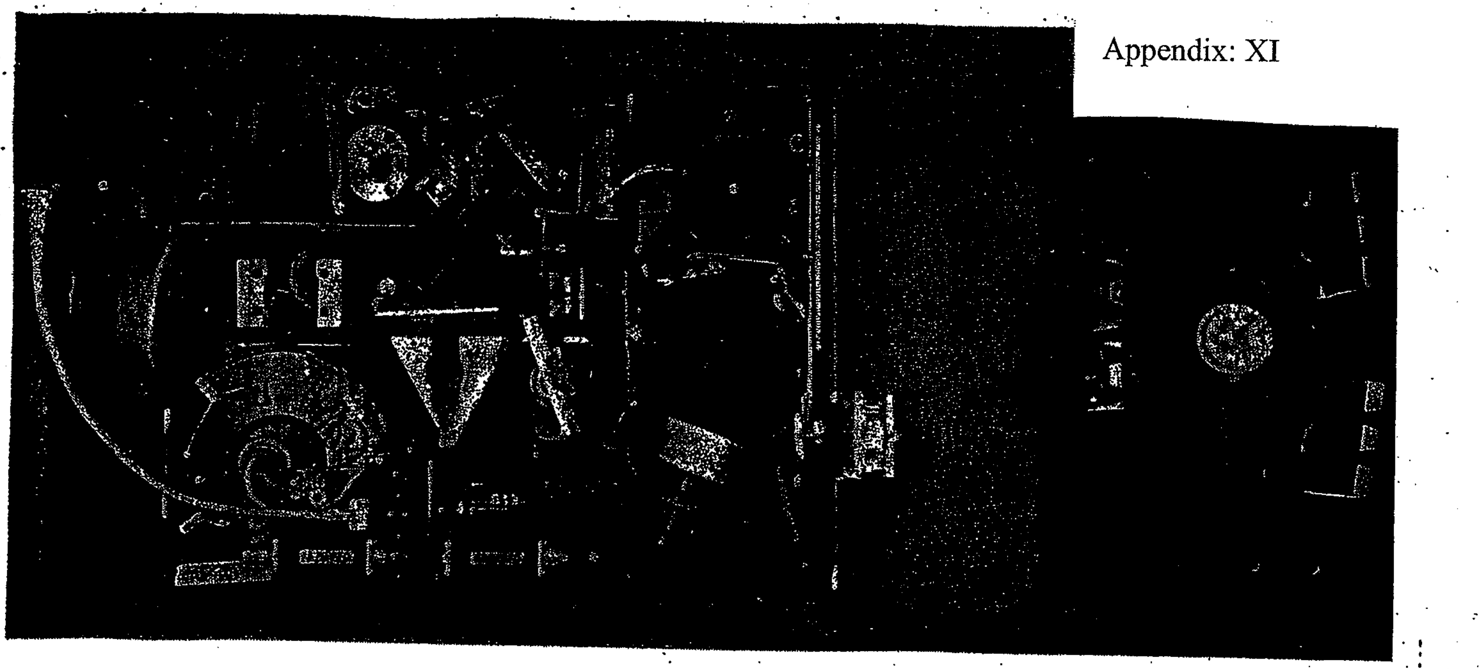
RELATED FAMILY of compounds, the methyl benzenes, exhibit both an over-all similarity and distinctive differences in their infrared spectra. The absorption dips which are marked in red are due to hydrogen wagging (see middle diagram on the facing page). The hydrogen atoms are vibrating in and out of, in a direction perpendicular to, the page surface.

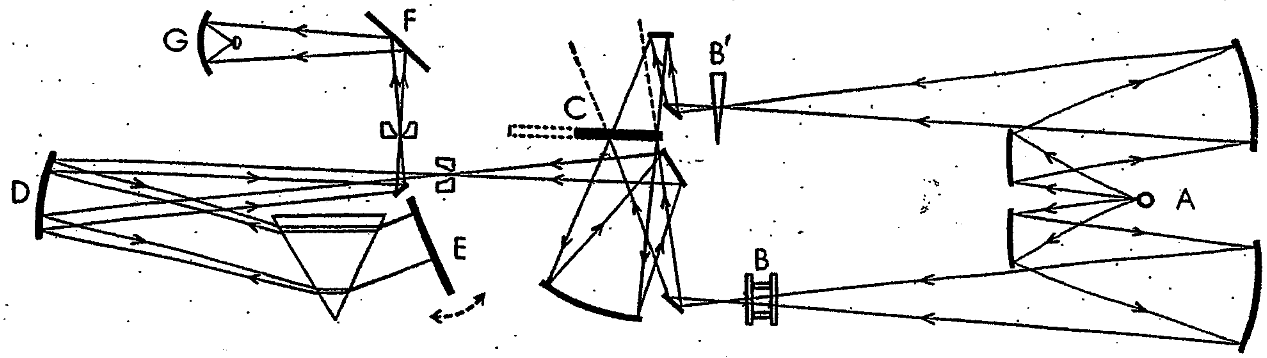
To obtain the infrared spectrum of a sample, we illuminate the sample with infrared radiation of successive wavelengths from 2.5 to 25 microns and measure with the spectrometer the amount of light transmitted by the sample at each wavelength. A modern spectrometer automatically computes the percentage of the light transmitted at each wavelength and in 10 or 15 minutes produces a curve of transmittance against wavelength, or, more commonly nowadays, transmittance against frequency. Frequency, meaning the number of waves that sweep past per second, is easily computed from the wavelength and the speed of light. The measure of frequency is called the "wave number"—it is actually the number of waves in one centimeter of light beam. In the range of infrared that we are considering the wave numbers run from 400 to 4,000 per centimeter.

Modern atomic theory at once shows the chemical importance of the frequency of absorption. Atoms and molecules absorb light in quanta, the energy in each quantum being proportional to the frequency of the light. The absorbed energy is in fact Planck's constant *h* times the frequency, or *hc* times the wave number. Einstein enunciated the principle that, when molecules absorb light, each quantum is wholly absorbed by one molecule.

Now a molecule can safely absorb only so much energy: chemical bonds are not unbreakable—and a good thing, or there'd be no chemistry! The energy required to break the bonds is known fairly accurately; it is comparable to the energy of a quantum of ultraviolet or visible light. When a molecule absorbs such light, the bonds are either broken or profoundly altered, and the "excited" molecule is a very different entity from the original. The deduction of atomic structure from ultraviolet spectra has been compared to deducing the structure of a piano from the sounds emitted as it falls down a flight of stairs.

The absorption of a quantum in the infrared, on the other hand, is not so rough a process. At these wavelengths a quantum of energy is only about one twentieth that in the ultraviolet. Hence the radiation merely sets the bond into vibration. In infrared spectroscopy we don't push the piano down the stairs,





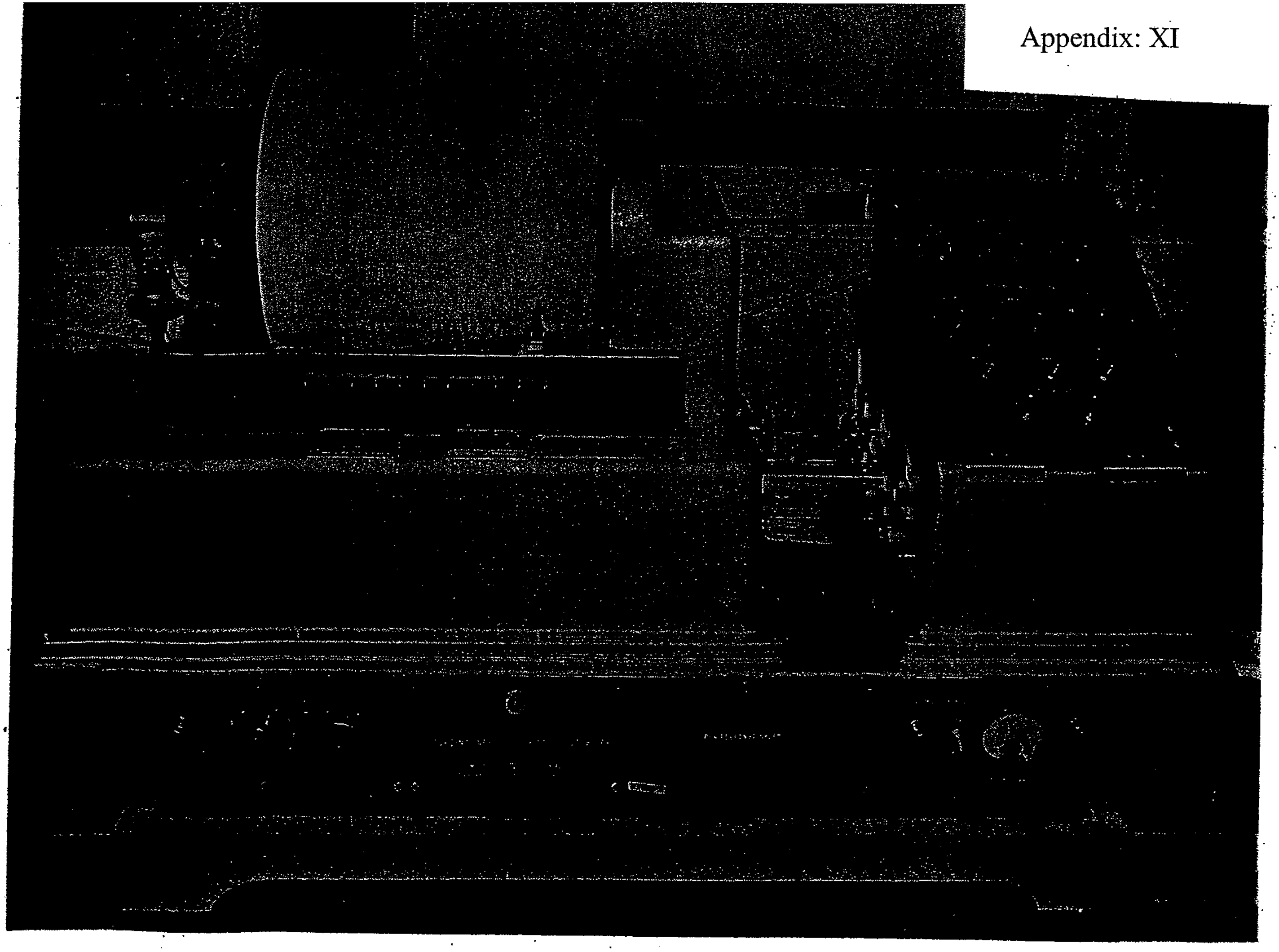
INFRARED SPECTROPHOTOMETER made by the Perkin-Elmer Corporation is shown on opposite page. Above are a photograph and diagram of its optical system. Infrared rays from a heated carbide rod (A) are split into two beams, one passing through the sample to be analyzed (B) and the other through a wedge whose transparency can be adjusted. A rotating mirror and diaphragm (C) alternately passes the two beams, by way of a pair of mirrors, to the curved mirror (D). This reflects the beams through a prism

we just plunk the keyboard a bit, and the sounds are a bit easier to relate to the piano.

We can carry this image further. The infrared spectrum of a two-atom molecule such as HCl shows absorption not only at the single natural frequency of the bond, but also at the overtones of that frequency. A molecule containing several atoms has several bonds and several natural frequencies. Here we cannot match up the frequencies with the individual bonds. We may think of such a molecule as a mechanical system of several masses connected by springs, and the resonant frequencies will be those of such a set of coupled oscillators. When one bond is set into vibration, the rest of the molecule also is involved through the interconnecting bonds. Hence the resonant frequencies are characteristic of the *whole molecule*. They are determined by (1) the masses, which means the specific atoms involved, (2) the spring forces, which means the bonds in the molecule, and (3) the way in which these are coupled, which means the specific geometrical arrangement of the bonds. If any of these changes, the set of resonant frequencies will change.

The infrared spectrum is simply a display of the resonant frequencies of the sample. Actually not all resonant frequencies give rise to absorption. Only those which, like the simple H-Cl vibration, cause some net change in the separation between positive and negative charges will interact with the oscillating electric field. Thus a molecule like Cl_2, which can have no charge separation no matter how the bond length changes (one Cl is like another!), does not absorb anywhere in the infrared. One of the resonant frequencies of CO_2, in which the two oxygen atoms move symmetrically, causes no charge displacement and hence does not appear in the infrared spectrum. But in general there are enough active resonant frequencies in a molecule so that the infrared spectrum is characteristic of the atoms, the bonds and the geometrical arrangement.

And here we see one reason for the great value of infrared: its *specificity.* The infrared spectrum is the most nearly unique property of a substance. Even geometrical isomers, which have the same atoms and the same bonds but differ in arrangement, can be distinguished by infrared. Such isomers are very hard to distinguish by ordinary chemical methods, yet their "slight" structural differences can give rise to profound differences in biological activity. Many, perhaps most, biological phenomena are highly sensitive to such differences in compounds. Consequently medical investigators need a sensitive analytical method. The infrared spectrometer was a key tool for the late cancer researcher Konrad Dobriner at Memorial Hospital in New York City in his



which separates their wavelengths. A rocking mirror (E) sweeps the dispersed beams across the detector, by way of (D), the small plane mirror, (F), and (G). The strengths of the alternately received beams are compared at successive wavelengths. When the detector senses a difference between the sample and the reference beams, an alternating current is generated which adjusts the movable wedge to equalize the signals. The motion of the wedge in turn is transmitted to pen that traces the absorption curve on drum.

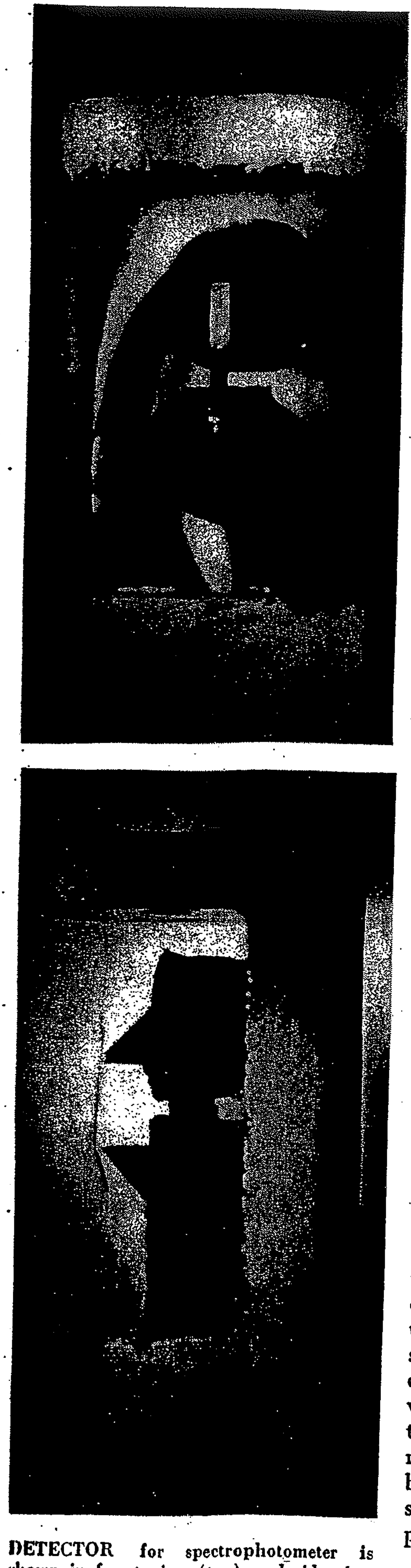
work of unraveling the metabolism of steroids in the body.

Inanimate objects—if internal-combustion engines can be so classified—also are sometimes sensitive to isomeric differences. The difference between "knock" and "anti-knock" gasoline components is a problem nicely suited to the infrared spectrometer. Indeed, the need in the petroleum industry for a fast, reliable and convenient method of distinguishing isomeric hydrocarbons and analyzing mixtures of them has played a large part in the recent upsurge of infrared. The war gave rise to pressing demands for high-test gasoline and for synthetic-rubber intermediates; both of these involved analysis of isomeric hydrocarbons. Infrared had been used in a few industrial laboratories in the late 1930s. Under the wartime challenge it was soon shown that infrared spectra offered the rapid and accurate analytical method so badly needed. And to meet the need for instruments the first commercial infrared spectrometer appeared in 1943.

The infrared also has unusual sensitivity to atomic mass; it can distinguish not only isomers but also isotopes. Practically the only methods for analyzing the stable isotopes used in tracer work are infrared and mass spectroscopy. Which is the better method in a given case will depend on many factors; in cases calling for a fast, nondestructive analytical method applicable *in situ*, the specificity of infrared can be very useful.

Yet all this is not the whole story. The greatest advantage of infrared as a research tool is that an infrared spectrum can be interpreted in terms of the same concepts chemists use in studying chemical properties—bonds and bond groupings. In classifying compounds and thinking about them and working with them, chemists have long spoken of "functional groups"—of olefins, of acid chlorides and so on. The functional groups provide a broad chemical description and a clue to the specific chemical properties of a compound—and they can be related to its infrared spectrum.

Let us consider for a moment the mechanical model. When two or more high-frequency springs are coupled together tightly, the vibrations of the resulting system form a new pattern in general quite distinct from that of the uncoupled springs; but when two high-frequency springs are coupled by means of a low-frequency spring, the resulting vibration pattern will include the frequencies of the high-frequency springs with only small shifts. Now chemical bonds fall approximately into two classes: (1) "high-frequency," which includes all multiple bonds and the single bonds involving hydrogen, and (2) "low-frequency," which includes all other single bonds, such as C-C, C-O, C-N. In



DETECTOR for spectrophotometer is shown in front view (*top*) and side view (*bottom*) about 16 times actual size. Beam of radiation is focused on thermocouple, the

studying infrared spectra we may therefore try out the idea of a "vibrational functional group"—any set of high-frequency bonds directly connected. According to this, all molecules with the group —N=C=S, for example, should have some frequencies in common, but they should differ from those with the —N=C— or C=S groups. Here indeed we find the "something" for which Coblentz was groping!

Now we find a piece of good fortune—one of the rare presents Nature bestows on investigators. Chemical functional groups and vibrational functional groups run parallel. Therefore certain characteristic absorption bands in an infrared spectrum give direct and strong evidence on the chemical nature of the sample. It is not really conclusive evidence, for the functional group idea is an approximation, and the parallelism of chemical and vibrational functional groups is not too strict. The infrared spectrum is not a magic crystal ball in which one reads the structural formula of an unknown sample. But its clues, when wisely used, can shorten by weeks the time required to complete the job. And the direct applicability of the chemical-bond concept and the functional-group concept means that the chemist can understand infrared spectra without having to learn a new language. He *does* have to learn a new dialect of his chemical language—and incautious chemists who have overlooked this have made some serious blunders. But the dialect can be picked up relatively quickly.

The knowledge of molecular structure required to make use of infrared spectroscopy had been achieved by about 1930 or 1935. Why, then, did the blossoming of chemical infrared start only in 1943? Some pioneer applications of infrared *had* been made in the chemical industry as early as 1936. But the technique is difficult. The cost of the modern commercial instruments and their need of maintenance still remind the user that the infrared spectrometer is doing a basically harder job than its ultraviolet counterpart. It has been well said that "no other spectrometric region of the electromagnetic range is so beset with experimental difficulties." Basically these arise from the very fact that infrared radiation is resonant with atomic vibrations. We have seen the advantages stemming from this; let us glance at the price we must pay.

As a source of infrared, we must use vibrating atoms, and the only practical way to set atoms vibrating is through

infrared, we must heat our source to a high temperature; a common source is an electrically heated rod of carborundum at 2,200 degrees Fahrenheit. Refractory materials able to withstand such temperatures are brittle, which can be a nuisance! Moreover, the laws of physics worked out by Planck show that most of the energy goes into the near-infrared and even the visible region, wasting our power and filling our instruments with a lot of unwanted wavelengths which must be filtered out or shunted off.

The same basic trouble arises in the measurement of infrared intensity—detection, as it is appropriately called. The infrared quanta do not disturb molecules enough even to affect a photographic plate! All we can measure is a slight rise in the temperature of the absorber, and we measure it today as Herschel did—by the effect on a blackened thermometer. The "thermometer," however, is a very sensitive thermocouple.

We must find prisms to disperse the infrared spectrum and optics to focus it, windows to let the radiation through the cells holding our samples and solvents in which to dissolve materials we want to study. Since all atoms can vibrate, it is not easy to find substances transparent to the infrared. Glass and water are completely opaque to it; ordinary organic solvents have so many absorption bands themselves that they obscure the spectrum of the substance dissolved in them. So we use mirrors instead of lenses, make our prisms and cell windows of rock salt, and think hard about the choice of solvents.

For infrared, the wartime need was a blessing in disguise—a very perfect disguise, to quote Mark Twain—because urgent necessity stimulated great improvements in instrumentation. The availability of better instruments today is a great blessing not only to analytical chemists and workers in the chemical industry but also to infrared spectroscopists interested in fundamental chemistry. Infrared spectroscopy has helped us win our present understanding of molecular structure, notably the geometry and dynamics summarized in the ball-and-spring model. Nowadays more people than ever before are at work on this fundamental use of infrared, studying the nature of those springs and the distribution of electronic charges in the bonds. Without depreciating the more widespread use of infrared for the analysis of compounds, we may well feel that in the long run the fundamental use will be more exciting. For the nature of the chemical bond is the problem at the

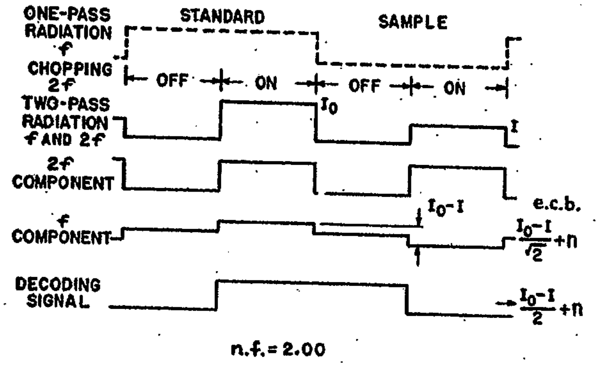


FIG. 4. Rochester system.

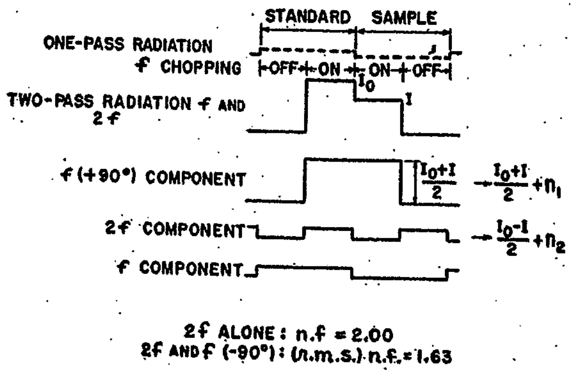


FIG. 6. Coates-Scott system and modified Coates-Scott system.

The two pass radiation signal seen by the detector is made up of a $2f$ sum component and of an f component which has the equivalent chopped beam amplitude $(I_0-I)/\sqrt{2}$. The f component is detected with a decoding signal which must be in quadrature with the one pass radiation also seen by the detector, and is therefore 45° out of phase with this f component. This causes another $\sqrt{2}$ decreases in the detected signal, and the 2 in the denominator of $(I_0-I)/2$ is the noise factor of this system.

The essential weakness of this system is the necessity for exact quadrature between the decoding signal and the one pass radiation signal, and any departure from this condition causes errors which vary linearly with it.

In the system illustrated by Fig. 5, which was suggested by Daly[3] of Unicam, radiation chopping at the intermediate image takes place at half the beam switching rate. When the two pass radiation signal seen by the radiation detector is decomposed into its various components, a component of frequency f is found, which is proportional to the difference signal, and the 2 in the denominator of $(I_0-I)/2$ is the noise factor of this system.

The difference signal requires for its detection a decoding signal of the same form which must be in exact quadrature with the sum signal, and any departure from this quadrature will cause errors which vary linearly with it. Furthermore, any imperfection in the chopping action, which takes place at the detecting frequency, for instance smudges on the chopping shutter, will also cause errors.

The system illustrated by Fig. 6 was developed by Coates and Scott[4] of The Perkin-Elmer Corporation. Beam switching and beam chopping are done at the same frequency, and in quadrature with each other, and signal decoding is done at twice that frequency. The two pass radiation signal seen by the detector is made up of the three components shown, and the magnitude of the decoded $2f$ component indicates that here again the noise factor is 2.

Since decoding takes place at twice the switching and chopping frequency, we do not have the strict quadrature requirement of the Rochester and Daly systems, but the presence of smudges on the chopper could produce a $2f$ component which would cause errors.

In the Coates-Scott system, it would be theoretically possible to make use of the sum signal and detect it in quadrature with the switching operation. The two signals thus obtained could then be combined to give independent values for I_0 and for I, just as in the one pass Hornig system, and this could serve to eliminate the beam weakener and to obtain a better rms noise factor of 1.63. But this arrangement would be open to the same criticism as the Rochester system; it would require exact quadrature between the decoding signal and the one pass radiation signal.

The next two systems are not open to any of the criticisms which can be made about the three preceding systems. In the system illustrated by Fig. 7 beam switching takes place at a frequency $3f$, and beam

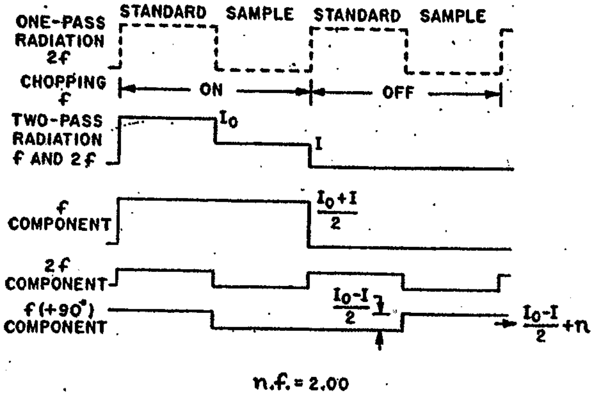


FIG. 5. Daly system.

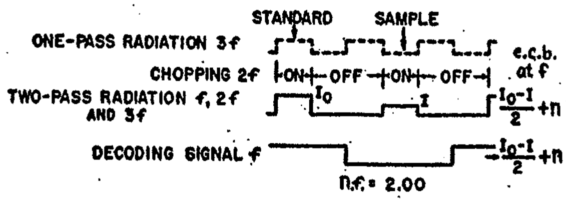


FIG. 7. $3f$ switching, $2f$ chopping, f decoding system.

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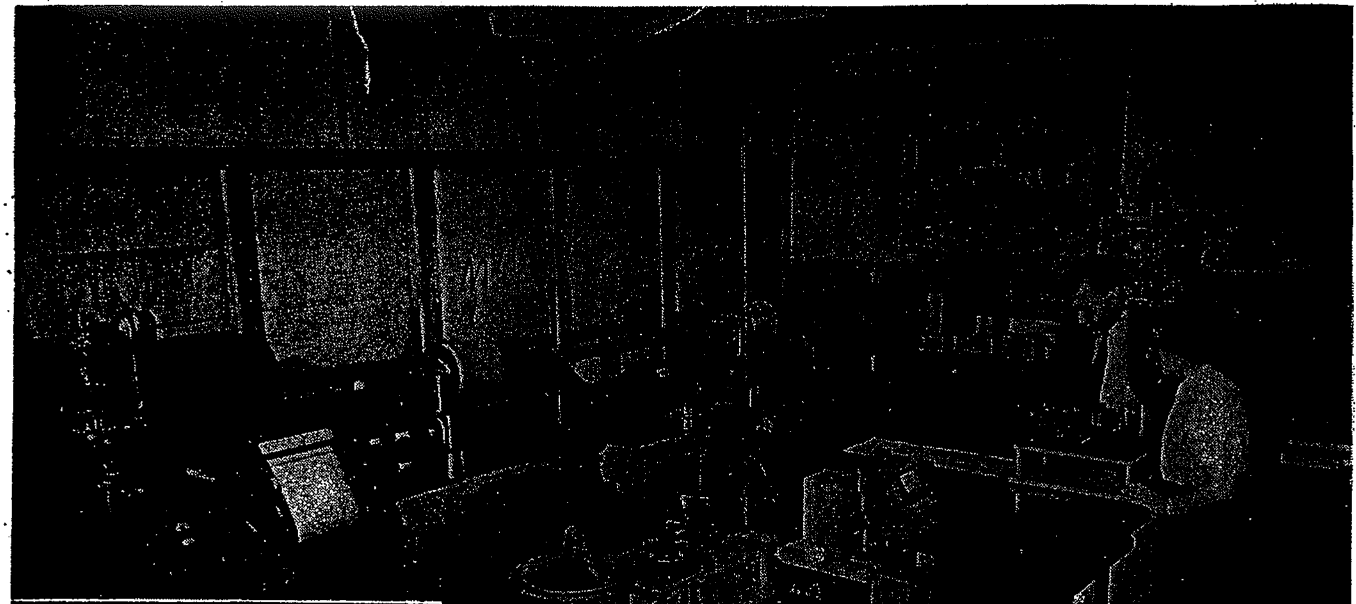
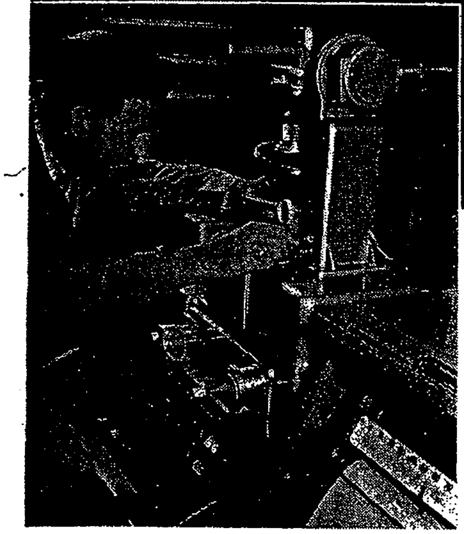
[3] Daly, Nature 171, 560 (1953).

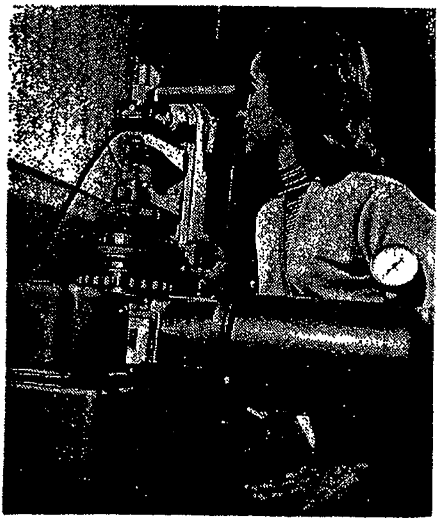
[4] Private communication to the writer.



Over-all view of Perkin-Elmer laboratory. Tables in foreground are used as work surfaces for sample preparation, changing prisms, checking spectra



Infrared spectra of single fibers and crystals can be run with aid of hood on infrared microscope being adjusted by Vincent J. Coates.



Infrared microscope for fiber and crystal studies, and beneath it, a 10-meter gas cell for making analyses of trace components. Martha Lester is the operator

LABORATORY OF THE MONTH

• *Laboratory setup enables prospective user to evaluate use of infrared instrumentation*

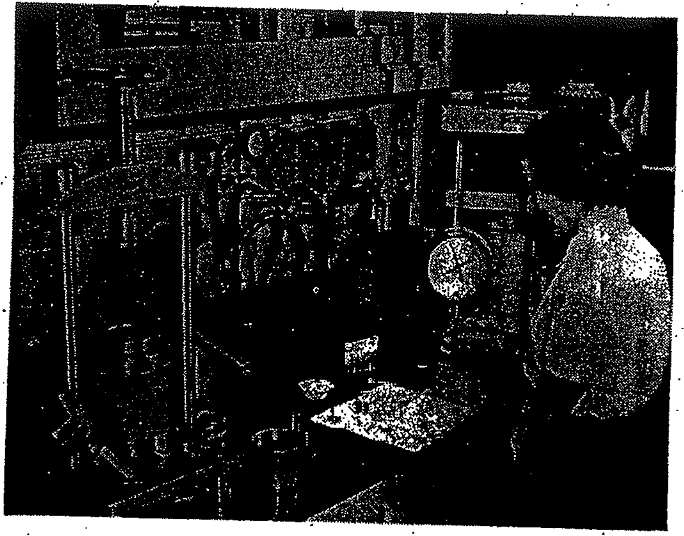
In this era of new instruments and methods, instrument manufacturers are finding that the customer must be shown the value of expensive equipment before he buys. Before a company or research laboratory is willing to invest a sizable sum of money in new instruments, it wants to be convinced that the instrument will prove a real asset to its analytical or quality control laboratory.

About two years ago, Perkin-Elmer Corp. set up a complete infrared laboratory to demonstrate to prospective customers how infrared instrumentation could be used to solve their particular problems. Since virtually every prospect for an infrared spectrometer is found to have some specific problem in mind for the instrument, it is the primary purpose of the laboratory to investigate the application of infrared analysis to prospective customer problems. Occasionally there are requests for flame photometer or electrophoretic analyses. Considerable time is also spent in developing new infrared analytical techniques and instruments, investigating improvements in existing instruments or accessories, and preparing data for papers delivered at technical meetings by Perkin-Elmer staff members.

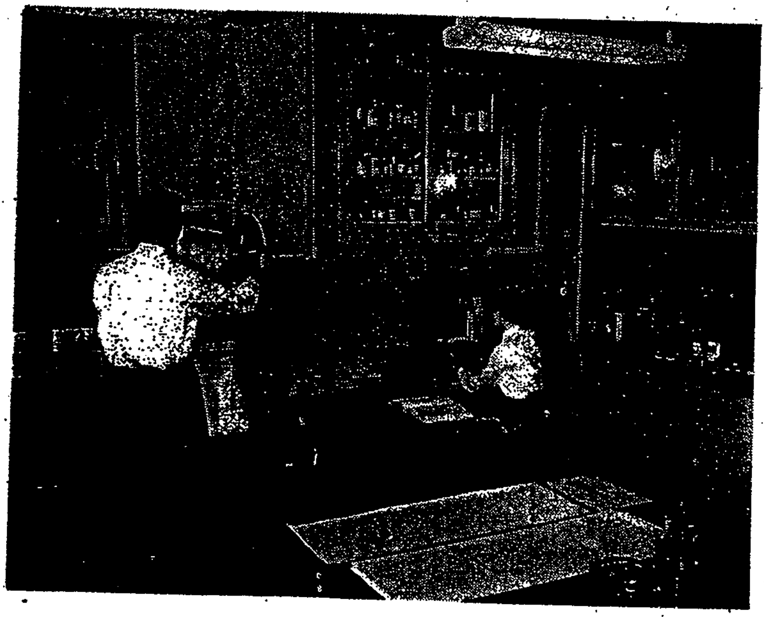
At latest count, the laboratory submitted over 600 problem analysis reports last year. These run the gamut of practically all industries concerned with organics—food and drugs, through plastics, paints, explosives, and petroleum. About 60% of the analyses are conducted on solids, 35% on liquids, and 5% on gases. Over 25% of the requests are for quantitative analyses.

Harry Hausdorff (left), director of the laboratory, explains the intricacies of a specific analysis to visiting chemist

◀



▲ Recent advance in infrared solids analysis is the potassium bromide press. Helene Sternglanz pours a mixture of sample and potassium bromide into the die, which is subsequently evacuated and pressed to form an infrared-transparent pellet



Equipment includes a small oven for drying specimens, a balance, and an exhaust hood. In the foreground is a glass-topped table for viewing and matching spectra

◀

Pure compounds, solvents, and samples are stored in the cabinet to the left. Crystal prisms for different regions of the infrared spectrum are in the center cabinet. To the right is a file of reference spectra including over 5000 spectra run off in the laboratory and 2000 API spectra

▼

THE PERKIN-ELMER
INSTRUMENT NEWS
FOR SCIENCE AND INDUSTRY

Published to further research, material analysis and production through electro-optical instrumentation.

Norwalk, Connecticut SPRING, 1955 Vol. 6, No. 3

Flexibility, Lower Prices Mark TRI-NON Advances

The last issue of INSTRUMENT NEWS explained the Perkin-Elmer philosophy of design for continuous infrared analyzers. Initially, we built the finest instruments possible that could solve the most complex problems in chemical processing. After these instruments had proven themselves in extensive field tests and our engineers had gained complete familiarity with plant processing problems, we began to think about a simplified model of the TRI-NON* Analyzer. Many plant processing problems do not require the complexity and problem-handling capacity designed into the original TRI-NON series.

We decided then to tailor the TRI-NON's complexity to the specfiic customer problem. Recognizing the advantages of the optical null, or self-balancing system, we decided to maintain this principle in all combinations. Hence the present four series of TRI-NON Analyzers – each varying in complexity, each designed to handle a different class of processing problem – and each differing in price. Perkin-Elmer application engineers can advise which series is best adapted to handle a specific processing problem. And so the net result to the user is an analyzer custom-tailored to his processing requirements at a most economical price.

A description of each series follows, plus a table giving characteristics and performance for each, (*page 8*).

Series A is the de luxe series with maximum performance in all respects: high in sensitivity, stability and discrimination. It operates under extreme ambient conditions; it is completely explosion-proof. The pick-up section of this series produces a shaft rotation which is proportional to the concentration of the gas under analysis. This rotation may appear as an indicator at the instrument. Normally, it is transmitted through a slide wire potentiometer bridge circuit to the recorder by a 3-wire cable. This system imposes no restrictions on the separation between the recorder and analyzer. Response time can be easily tailored to any analytical problem since the balancing time of the analyzer is completely independent of the recorder or controller.

Series B has an explosion-proof pick-up section with non-explosion-proof

Continued on page 8

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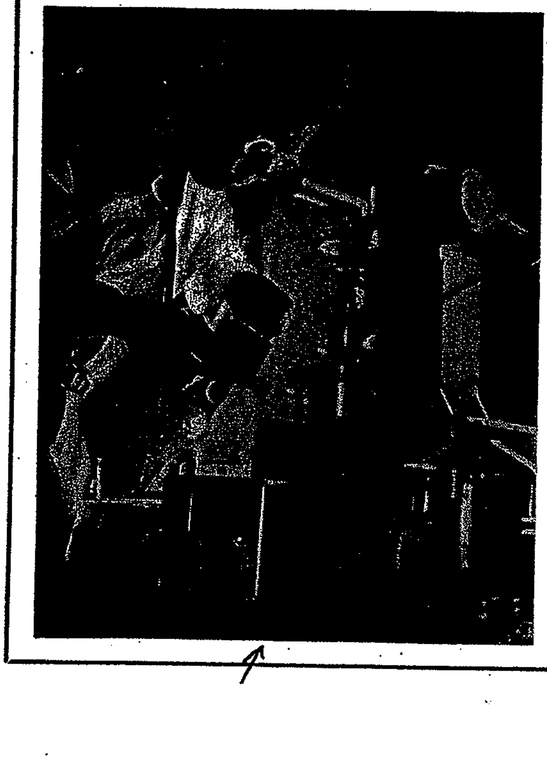
*TM The Perkin-Elmer Corporation.

Spectrometers Get Bigger and Better



Infrared de luxe. *This Model 13 Ratio-Recording Spectrophotometer is one of the most elaborate infrared instruments ever shipped by Perkin-Elmer. It is an outstanding example of the Building Block principle for it incorporates a microscope attachment, double pass monochromator, interchangeable prisms, and a variety of infrared detectors for macro and micro work. This instrument is shown at the General Electric Company's Research Laboratories where it is used as an all-purpose instrument for advanced research problems. Several other Model 13s—with ultraviolet attachments – have been shipped to other large research centers.*

New Approach to Solids Study:

INFRARED SPECTROSCOPY BY REFLECTION

By I. Simon

Most of the work in infrared spectroscopy is being done on samples which are solid films, liquids or gases. It is then perfectly straightforward to obtain significant information by measuring absorption as a function of wavelength. In this case, the techniques are relatively simple since the samples could be introduced in suitable cells with well defined dimensions. The solid materials are somewhat unwieldy since they must be disintegrated into fine powders and mulled, or compressed in pellets, or else fabricated into thin slides. The experimental difficulties become greater when the solids are highly absorbing or opaque in the wavelength range considered.

- **Absorption Difficulties**—We encountered this problem when applying infrared spectroscopy to the study of the structure of crystalline and vitreous forms of silica and other silicates, all of which have a well-known, strong absorption band around 9μ. For instance, quartz is so highly absorbing in this wavelength region that a slide 1μ thick transmits

Continued on page 7

See On-Stream TRI-NON Report, Pp. 4-5

OFF THE BEAM

Very soon the 15 million readers of ***Reader's Digest*** are going to be let in on a secret that you 15 thousand readers of INSTRUMENT NEWS have known for some time—that the infrared spectrometer is a highly potent tool in the hands of a chemist. Mr. Harland Manchester, technical editor of the DIGEST, has written a highly interesting article on the more glamourous side of infrared spectroscopy. Even your editor who has read and written as much on the subject as the next person found much that was new and entertaining in it. Watch for it in an early DIGEST issue.

By now ***Fisk University's*** Annual Infrared Spectroscopy Institutes are a regular feature of the summer infrared scene. This year from August 29-September 2, they are holding their Sixth Annual Institute, planned primarily for introducing newcomers to the advantages of infrared spectroscopy. Further information may be obtained from Nelson Fuson, Fisk University, Nashville.

Another ***infrared course*** is making its debut this summer. It will be held at the Polytechnic Institute of Brooklyn, August 15-19. The course will cover both theory and practice with emphasis on the latter. Interested parties should contact Dr. Robert Bauman, Polytechnic Institute of Brooklyn, 99 Livingston Street, Brooklyn, New York.

Harry Hausdorff and Helene Sternglanz of our laboratory have given a considerable amount of study to ***differential analysis*** in the infrared. Their paper on the subject was received with interest at Pittsburgh. The paper was originally published in the Italian Journal *Nuovo Cimento*. We have obtained reprints and will be pleased to send them on request.

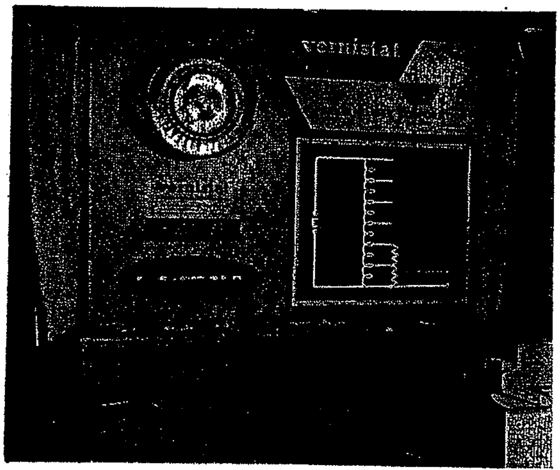
We have learned that infrared spectra of a large number of ***carcinogenic materials*** are badly needed by the ASTM Committee on Standard Data. Anyone having such spectra and not planning to publish them should contact E. Carroll Creitz at the National Bureau of Standards, Washington, D. C. He can supply lists of the wanted spectra.

Those of you who read the article on ***integrated absorption techniques*** must have been as mystified as we were with the X's and Y's in the last paragraph. The article, of course, had been written before all data was available. When the NEWS appeared, we were reminded that it had been sent to us in time for inclusion—sure enough, we found it under a pile of unanswered correspondence on our desk. So here is the last paragraph printed as it should have been in the first place:

"Multiplying the counter reading by a predetermined integration constant provides intensity values in terms of absorbance –cm.$^{-1}$ or absorbance-microns.

"The reproducibility of the device is excellent. For example, on a single band where the peak reading, Dy, equalled 0.9 absorbance, successive readings agreed to 1 part in 500. Over a two-day period, the maximum spread was 1 part in 360."

For those of you who would like to use ***standard UV cells*** for your work on Perkin-Elmer spectrometers in the UV and near infrared regions we have developed a cell adaptor. Its part number is 013-0347 and price $75. It is designed to hold most standard UV cells.



vernistat™ on Display

Featured in the VERNISTAT *display booth, shown here at the New York IRE Show, are a large transparent working model illustrating action of the switch commutator and wiper, and an illuminated schematic of the circuit principle. The* VERNISTAT *will be seen on the West Coast August 24-26 at the Civic Auditorium in San Francisco during the Westcon IRE Show and September 12-16 in Los Angeles (Shrine Exposition Hall) during the ISA Show.*

Dr. Van Zandt Williams

Vice-President,
Sales and Research

If the University of Michigan can be termed the "Cradle of Infrared Spectroscopy" then Princeton University may lay claim to the title "Father of Commercial Infrared Instrumentation." One of its instructors, Dr. R. Bowling Barnes, and two of its pupils have had a profound effect on the instruments now being manufactured by two of the leading manufacturers of infrared equipment. One of the pupils, Dr. Robert Brattain, went to the Shell Development Company where he worked closely with Beckman in its early instrument programs, while the other, Dr. Van Zandt Williams, continued his infrared work at American Cyanamid with Dr. Barnes.

After seven years at Cyanamid, Van Williams moved to Perkin-Elmer where he could spend full time in continuing instrument development and in expanding the uses for infrared spectroscopy. In 1951 he became a vice-president and director of The Perkin-Elmer Corporation and Director of Sales and Research.

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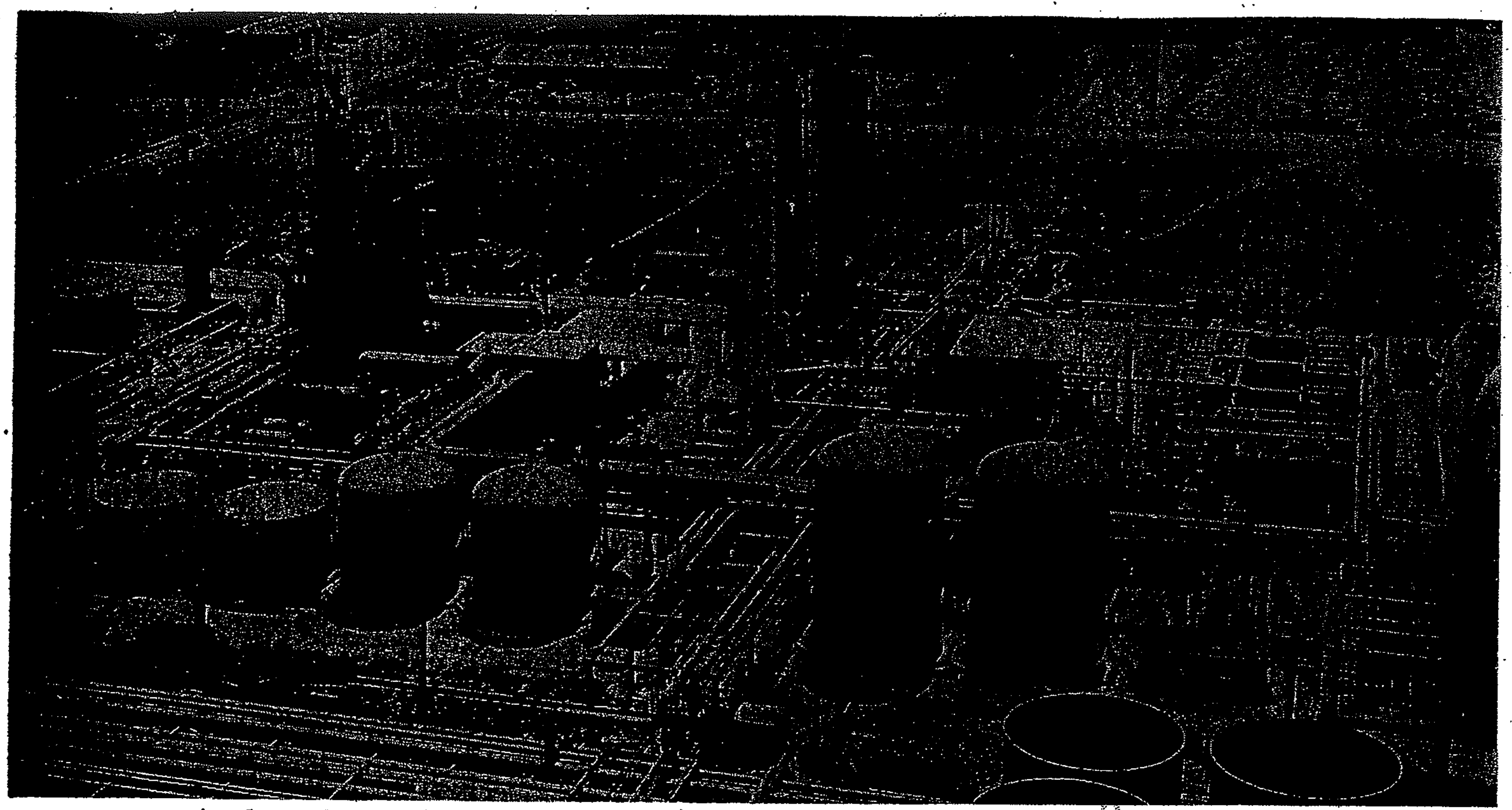
PERKIN-ELMER WHO'S WHO

★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★

Van is well known by all who have worked in infrared for his many publications and for his active participation in meetings and societies where infrared is discussed. He is somewhat of a crusader both inside and outside of Perkin-Elmer for better instruments and better instrument techniques. His most recent preoccupations have been with Integrated Absorption Measurements on the one hand and the Coblentz Society on the other.

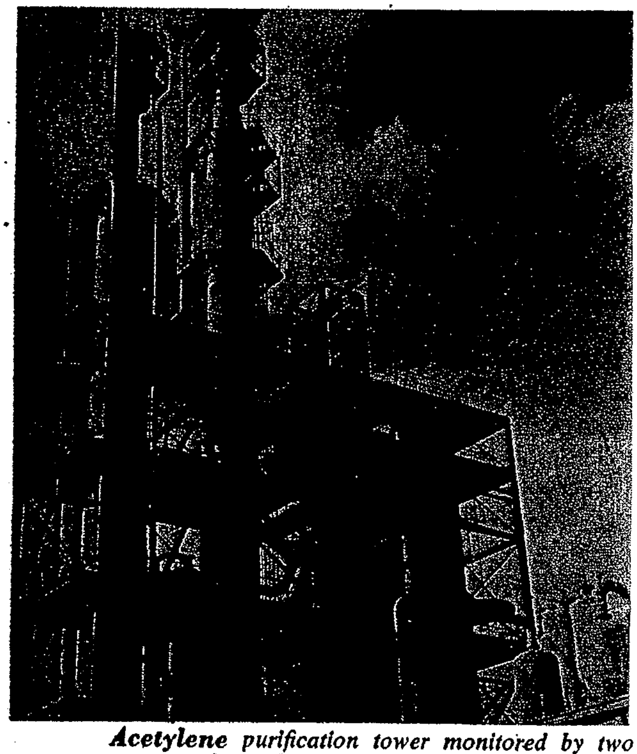
Born in Providence, Rhode Island in 1916, he obtained his B.S. at Brown and his Ph.D. at Princeton. He is a member of Phi Beta Kappa, Sigma XI, the American Chemical Society, the Optical Society of America, the Society for Applied Spectroscopy and—naturally—the Coblentz Society.

Between his many trips, he occasionally is able to spend some time at his home in Riverside, Connecticut with his wife, the former Mary Bridgeman, and their two boys.



Acrylonitrile unit *produces Cyanamid's principal product at Fortier; takes up small section of 600-acre site.*

TRI-NON ANALYZERS MONITOR PROCESS STREAMS AT AMERICAN CYANAMID'S NEW FORTIER PLANT



Acetylene *purification tower monitored by two* TRI-NONS *located near top of steel framework.*

About 20 miles out of New Orleans on the west bank of the Mississippi River stands American Cyanamid Company's new Fortier Petrochemical Plant. Built by Chemical Construction Corporation at a cost of over $50,000,000, its principal product is acrylonitrile, a chemical building block used in the manufacture of synthetic rubber and fibers, plastics and adhesives, insecticides, and pharmaceuticals. An important by-product is anhydrous ammonia.

Nearly a dozen Perkin-Elmer TRI-NON analyzers have been installed to provide continuous analytical information to the operators of the various units. In the ammonia plant, for example, four low range (0-50 ppm) TRI-NON Analyzers monitor CO and CO_2 in the feed streams to the reactor to warn against the possibility of catalyst poisoning. Several other instruments keep tabs on various phases of acetylene production and purification.

After several months of operation, the Cyanamid operating officials have found that the analyzers have proved to be of valuable assistance in controlling the processes in several different ways. The practically instantaneous information concerning stream composition supplied by the analyzers has made it easier for the unit operators to keep them operating at top efficiency at all times. In addition, ample warning of dangerous situations developing are given by the analyzers so that preventive measures can be taken in time to avoid accidents. Finally, the analyzers have helped reduce the number of men needed to run the plant. Such reductions represent considerable savings in operating costs.

Cyanamid's investment in continuous analytical instrumentation at the Fortier Plant has proved to be a wise one.

NEW TRI-NON SERIES

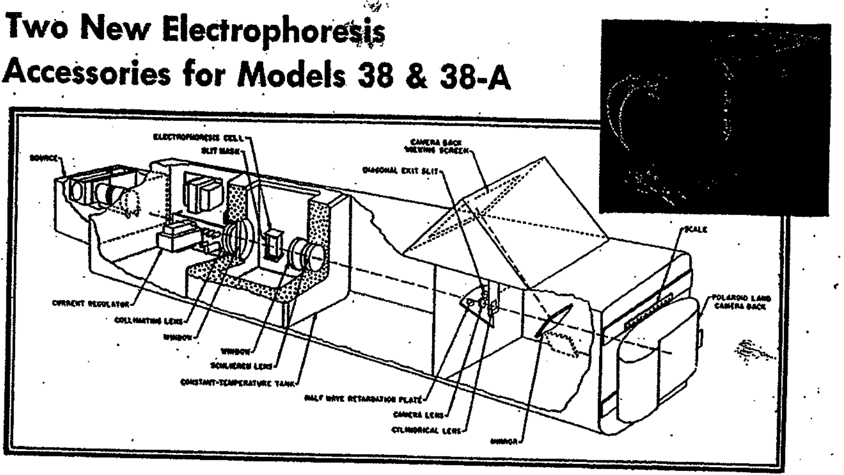
Continued from page 1

electronics and recorder capable of being remotely located up to a maximum of 500 feet. A multi-wire cable connects the two units. Where interference is not as great, performance is the equivalent of a Series A instrument. This model usually incorporates a single servo loop incorporating the recorder. This series provides maximum serviceability since the electronic section is available in the control house.

Series C is non-explosion-proofed. As in Series B, the pick-up section is connected by a multi-wire cable over a maximum of 500 feet to the electronics section, which may be located wherever desired. This model also involves a single servo loop incorporating the recorder, which may also be remotely located. Performance is same as Series B. These models are highly stable and completely adequate for many process streams.

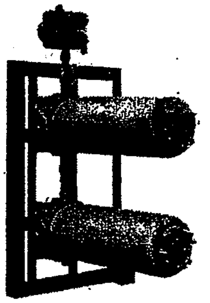
Series D is also non-explosion-proofed. All components: pick-up section, electronics and recorder, are mounted in a cabinet with the pick-up section mechanically coupled to the recorder. Performance characteristics are the same as Series C. This instrument is especially useful in monitoring for toxic or explosive vapors in the atmosphere.

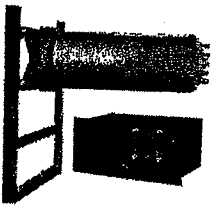
Two New Electrophoresis Accessories for Models 38 & 38-A



A new Rayleigh fringe accessory makes possible fast and accurate index determinations. The unit consists of a high intensity mercury arc lamp, a narrow band interference filter, a horizontal entrance slit superimposed upon a 500-line per inch vertical grid, a mechanical shutter, a special 2 cc sample cell with slit mask, and a half-wave retardation plate. In one of the four modes of operation, the dn/dh curve is superimposed against a background of sharp, well-defined fringes so that the area under the curve may be integrated simply by counting the fringes displaced.

A compact, efficient refrigeration unit measuring 15x12x32 inches (right) which will maintain water bath temperature within ±0.02°C. is now available. Striations or distortions caused by ice bath temperature fluctuations are eliminated when the refrigeration unit is used as a cooling agent.





TRI-NON ANALYZER—CHARACTERISTICS AND PERFORMANCE

SERIES	A	B	C	D
GENERAL APPLICATIONS	Complex streams, trace component analysis, other problems where special instrument characteristics are important.	Complex stream analysis, determination of principle component, partially explosionproof.	Less complex streams Nonhazardous locations	
NOMINAL PERFORMANCE				
Accuracy	± 1% of scale	± 1% of scale	± 1% of scale	± 1% of scale
Stability	± 2% of scale	± 2% of scale	± 2% of scale	± 2% of scale
Response time	As low as 5 seconds	As low as 15 seconds	As low as 15 seconds	As low as 15 seconds
OPERATING PRINCIPLES	**Null balance system.** Servo driven attenuator comb in a chopped system produces true radiation null at detector. **Detector.** Single receiver, dual-condenser microphone type, rendered insensitive to vibration through use of a unique symmetrical construction. Factory adjusted on vibration table at operating frequency. **Source.** Nichrome coil operates at approx. 800°C. Sealed in dry inert gas.			
EXPLOSIONPROOFING				
Pick-up unit	Class I, Group D, Div. I	Class I, Group D, Div. I	none	none
Electronic unit	Class I, Group D, Div. I	none	none	none
SAMPLE CELLS				
Cell material	Stainless steel (Std.)	Stainless steel (Std.)	Aluminum (standard) Others available	
Cell length	1/8", 1/2", 2", 8"	From 1/8" to 12"	From 1/8" to 12" (longer on special order)	
	Complete Sample System provided to Specification at extra cost.			
MAINTENANCE AND CHECKING	**Test circuit.** Built-in test box by which the functional subassemblies may be checked. Optional on all instruments. **Serviceability.** Unitized construction using functional subassemblies which are readily removable for ease of servicing.			
DIMENSION Height	67.5"	12"	66"	66"
Width	25"	12"	22"	22"
Depth	33"	33"	21"	21"
WEIGHT Pounds	370	300	250	250
PRICE RANGE (Excluding recorder)	$5450 and up	$3500 to $4500	$3000 to $3500	$2800 to $3000

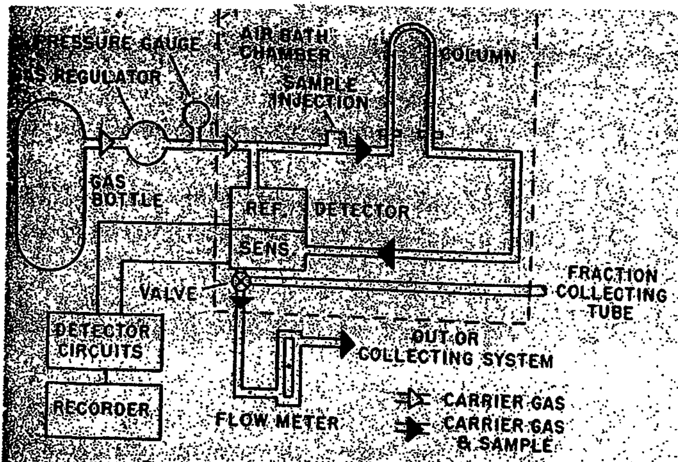


FIGURE 1—Flow scheme for gas chromatography instrument: Accuracy of a mass spec at a lower investment.

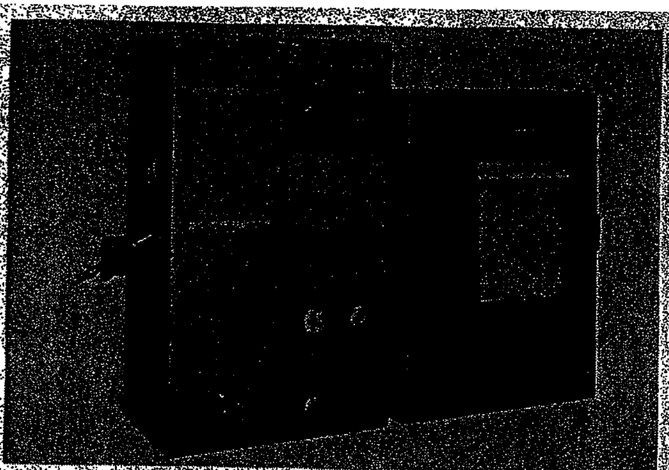


FIGURE 2—Instrument used for the gas analysis. Precision gas sampling valve is mounted on left side.

Fuel Gas Analysis by Chromatography?

It's a more accurate method than Orsat and cheaper than mass spectrometry.

incent J. Coates
athaniel Brenner
he Perkin-Elmer Corporation
orwalk, Conn.

FIRST REPORT on fuel gas analysis by chromatography shows favorable promise. Most common method of analysis in use today for gases is the Orsat apparatus. Instruments of this type, although inexpensive, are not very sensitive and are capable of providing analysis of only a few components in a mixture.

Mass spectrometers are capable of excellent separation, providing accurate analysis of as many as 20 components, but are quite expensive and require skilled operators if consistent results are to be obtained.

Gas chromatography instrumentation is capable of equalling, or exceeding the analytical capabilities of mass spectrometers while requiring an investment not far in excess of that required for an Orsat apparatus.

Fuel gases contain components which boil over a wide range. For the analysis of hydrogen, nitrogen, carbon monoxide, methane, ethane, carbon dioxide, and propane, a two meter, stainless steel column packed with silica gel was chosen (Figure 2). Components heavier than propane have very long retention times and emerge from this column as very broad bands with very weak peak heights in the concentrations encountered here.

Very heavy components present in trace quantities may actually remain in the column during room temperature operation but can be chased out by raising the column temperature for a short while. Using this method of regeneration every 100 analyses or so, the column life can be maintained practically indefinitely and the column can be used with reproducible results for months or even years.

For the analysis of the heavier ends (Figure 3), a liquid partition column packed with 25 percent by weight tetraisobutylene (B.P. 200 C.) coated on amorphous silica (acid washed and sieved 60-100 mesh) was chosen. Two 2-meter stainless steel columns were used in series for best separation. The partition method, as distinct from the adsorption method, tends to produce more symmetrical band shapes for the higher boiling hydrocarbons.

The change from adsorption to partition columns requires only a few minutes. It is estimated that the complete separation of a mixture of fuel gases, hydrogen through pentanes, should require not more than 60 minutes. The separation of hydrogen through propane required about 30 minutes.

Analytical Procedure—Both band area and peak height are closely linear functions of concentration and can be used as a means of measuring

Fuel Gas Analysis by Chromatography? . . .

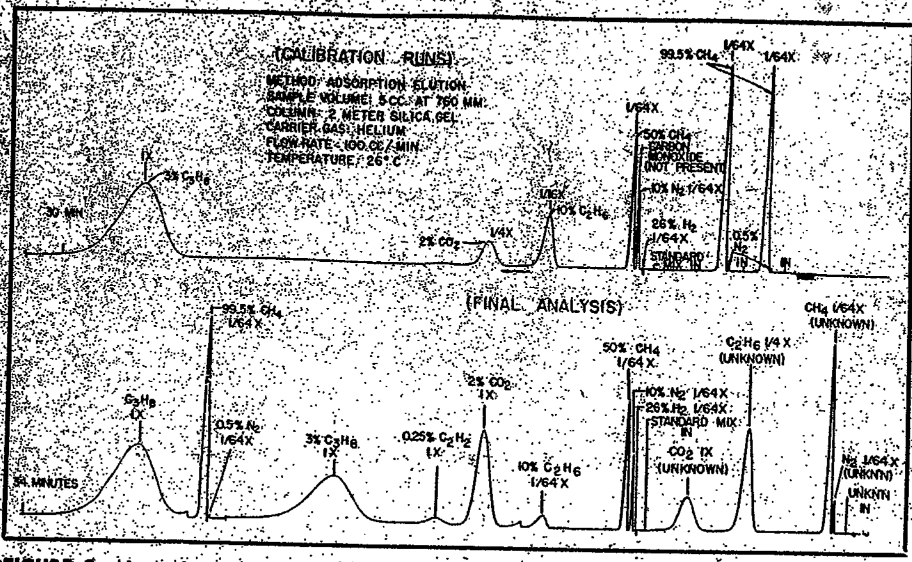


FIGURE 3—Natural gas analysis of light ends: Components heavier than propane have very long retention times.

component concentration. Several methods of utilizing these measurements to obtain analyses of mixtures are employed. The method chosen for this problem was the "calibration using known standards" method.

From known mixtures under reproducible conditions, calibration curves of peak height versus concentration are plotted for each component. By obtaining a chromatogram of the unknown mixture under the same instrumental conditions and measuring peak heights, concentrations are read directly from the calibration curves.

Taking all possible sources of instrument error into account, it can be conservatively estimated that a measurement precision of 1 percent of the amount present can be realized for major components and 4 percent for trace components. This accuracy can be attained directly without complicated and expensive control of ambient conditions. This performance, however does not take into consideration errors in the original calibration mixtures or operator error.

For routine repetitive analyses, where time can be profitably spent in setting up initial conditions and obtaining calibration data, the calibration using known standards method should be used. It has the overriding advantages of speed, simplicity, and accuracy. It seems ideally suited for fuel gas analysis.

Natural Gas Analysis—Natural gas contained under pressure in a cylinder was analyzed using gas chromatography. In order to set up the analysis, separation of all components was accomplished by a combination of adsorption and liquid partition principles. Chromatograms were obtained using a two meter stainless steel column filled with silica gel adsorbent (Figure 2) and two 2 meter liquid partition columns filled with tetraisobutylene on amorphous silica as the stationary phase (Figure 3).

HOW THE INSTRUMENT WORKS

A schematic flow diagram for this instrument is shown in Figure 1. Carrier gas is supplied to the system from an external gas cylinder equipped with a pressure regulator. Helium is normally used because of its high thermal conductivity and inertness. Nitrogen or dry air may be used where high sensitivity is not required.

The carrier gas flows through a second pressure regulator to the reference side of the thermal conductivity detector and from there to the sample injection systems. Two such systems are provided. One, a precision gas sampling valve, and the other, a liquid sampling device where samples are injected into the system by means of a hypodermic syringe. The sample is then carried into the column where the separation of components occurs. As each component arrives at the column exit, it flows through the sensing side of the thermal conductivity detector. The difference in thermal conductivity between the sensing and reference sides (which are part of a bridge circuit) produces an unbalanced voltage which is measured by a standard recorder.

The time required after injection for a given component to appear at the column exit is called retention time. By maintaining a constant flow rate, the retention time for a given component can be exactly reproduced on the recording paper.

After the detector, the flow passes through a three-way valve which in one position allows collection of each component, if desired, and in the other position provids measurement by a flow meter. With slow flow rates used (30-200 cc./min.) infrequent renewal of the carrier gas cylinder is required.

For detector construction, glass-coated thermistor beads are used. The carrier gas is supplied to the reference side by diffusion. Straight-through flow in the sensing side provides speed of measurement which is important for light gas analysis. The volume in the sensing side is minimized (approximately 0.25 cc) to provide maximum resolution of components. In the detector bridge circuit, a steady voltage, provided by dry cell batteries, is adjusted by a rheostat and measured by a voltmeter. A zero adjustment control and a precision step-wise range attenuation control are provided.

The precision gas sampling valve is mounted on the left-hand side of the cabinet (see Figure 2). Access to the column and detector chamber, which incorporates an air bath temperature controlled oven, is provided through a door on the left front. To the right front of the instrument are located an operating control. At the top is the temperature control system (20-225 C.), in the center the flowmeter, and the lower controls comprise the pressure regulator and gage, detector voltage control and voltmeter, recorder zero control, and precision stepwise recorder range control. This important control provides range adjustment in steps of ½ from 1 to 1/128. The recorder is a strip chart recorder, 0-5 mv, with one second full scale response.

The silica gel column completely separated nitrogen, methane, ethane, carbon dioxide and propane in that order. The partition column produced single peaks for nitrogen-methane, ethane, carbon dioxide, propane, isobutane, normal butane, isopentane, and normal pentane respectively.

A 5 cc. sample was used to obtain sufficient peak height of the minor components for accurate measurement, and the range attenuator was used to keep the major components on scale.

A rough check, using the internal normalization method, provided an approximation of the composition of the mixture. From this data a synthetic mixture of known composition was run under similar conditions to obtain data for the plotting of the calibration curves shown in Figure 4. As an additional calibration, pure methane was run to provide a second calibration point for the methane analysis, as can be seen in Figure 2. To complete the analysis in the fastest possible time, the chromatograms for the unknown natural gas sample, the synthetic mix and the pure methane sample were intermixed in such a way as to avoid overlap of components in the silica gel column. This complete run took only 34 minutes. It should be noted that such intermixing does not affect the retention times or the

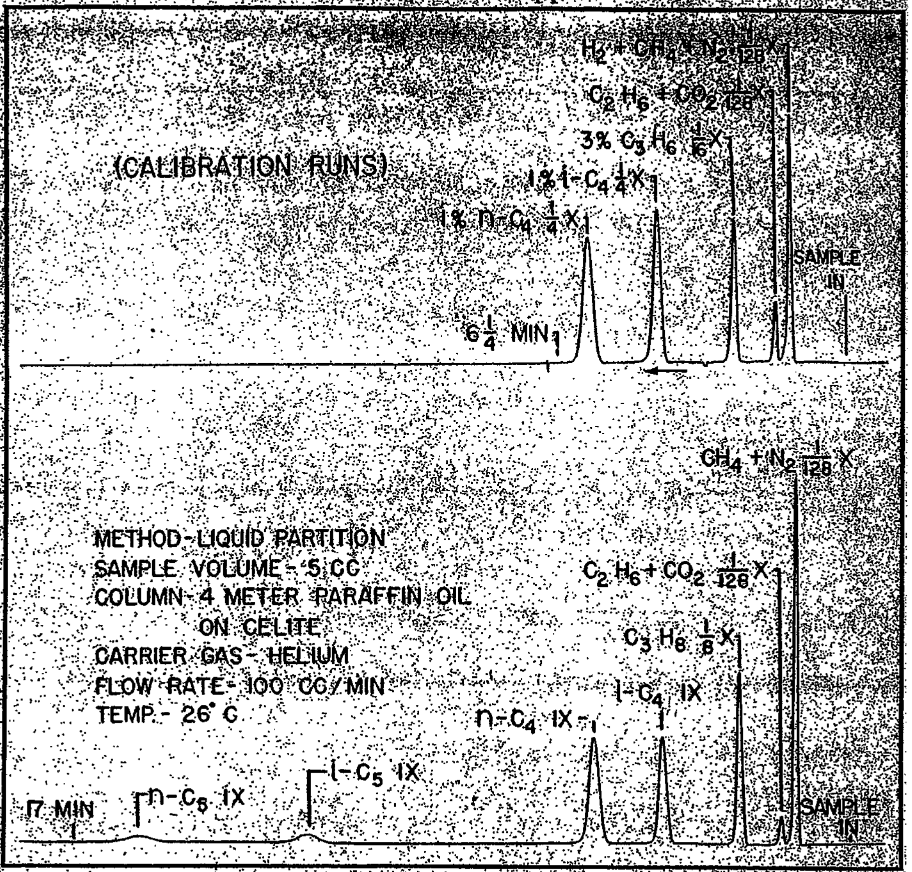


FIGURE 4—Natural gas analysis of heavy ends: Liquid partition column is used.

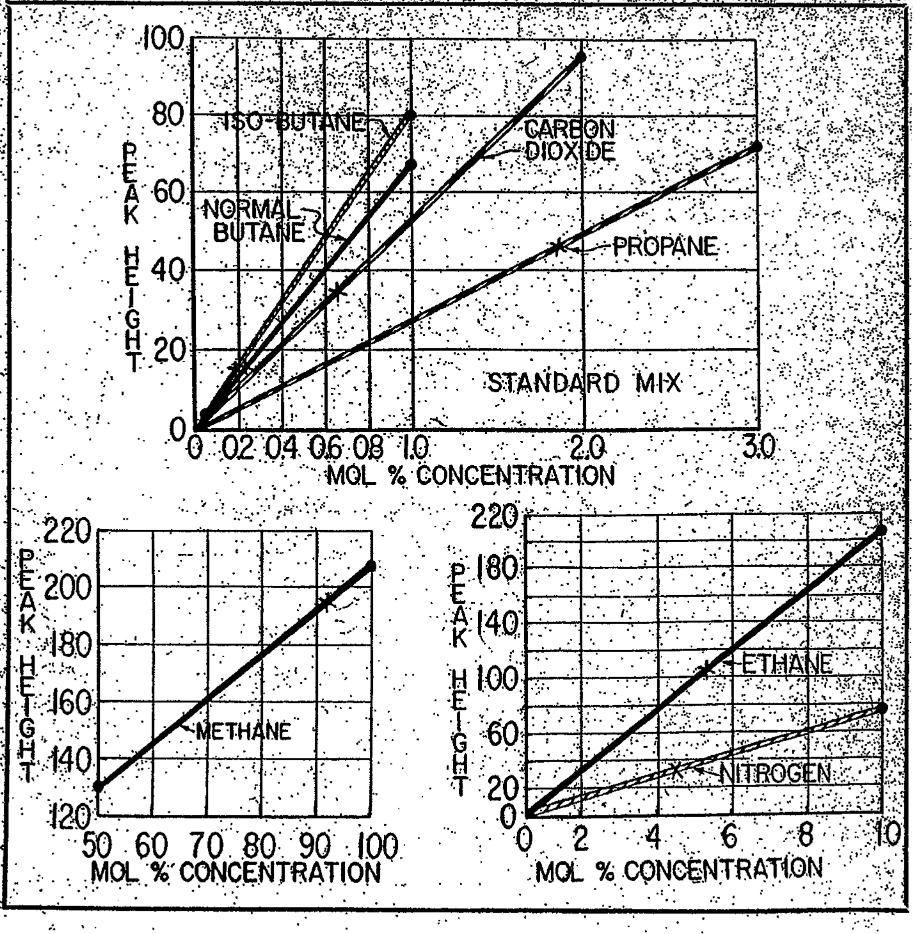


FIGURE 5—Calibration plots for natural gas analysis.

TABLE 1
Natural Gas Analysis

Component	MOL % Concentration By Mass Spectrometer	MOL % Concentration By Vapor Fractometry
Nitrogen	3.4	4.13
Helium	0.09	...
Carbon Dioxide	0.63	0.66
Methane	88.0	88.3
Ethane	4.8	4.7
Propane	1.7	1.76
n-Butane	0.27	0.21
i-Butane	0.21	0.17
n-Pentane	0.07	.04
i-Pentane		.03
Total	100.0	100.0

TABLE 2

Component	% Composition
N_2	21
CH_4	64
CO_2	2.5
C_2H_6	2.2
C_3H_8	0.5
CO	3.0
C_2H_4	0.2
H_2	Balance (6.6)

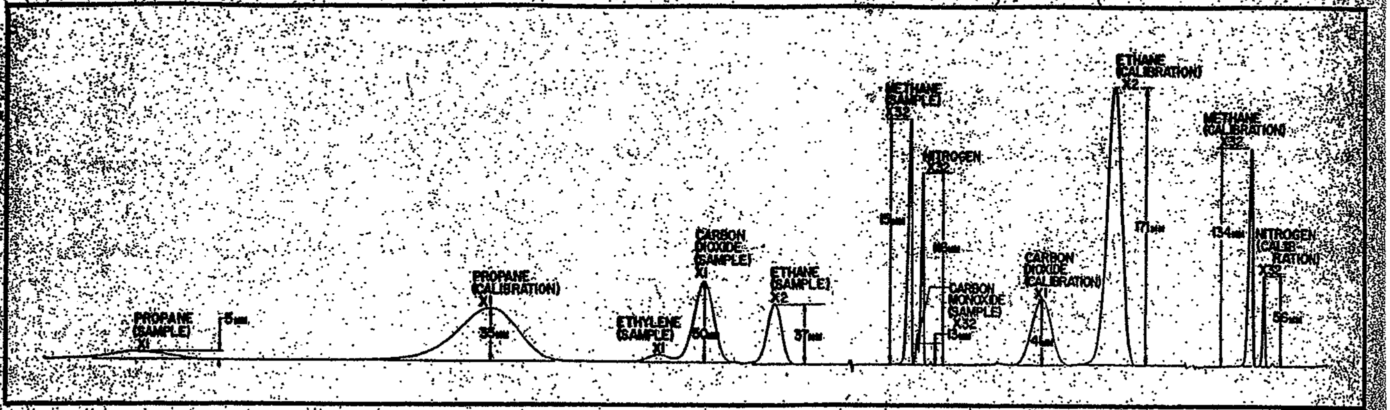


FIGURE 6—Manufactured gas sample: Treated in much the same manner as natural gases (25 C., 1cc sample, 2 meter silica gel column 100 cc/min flow).

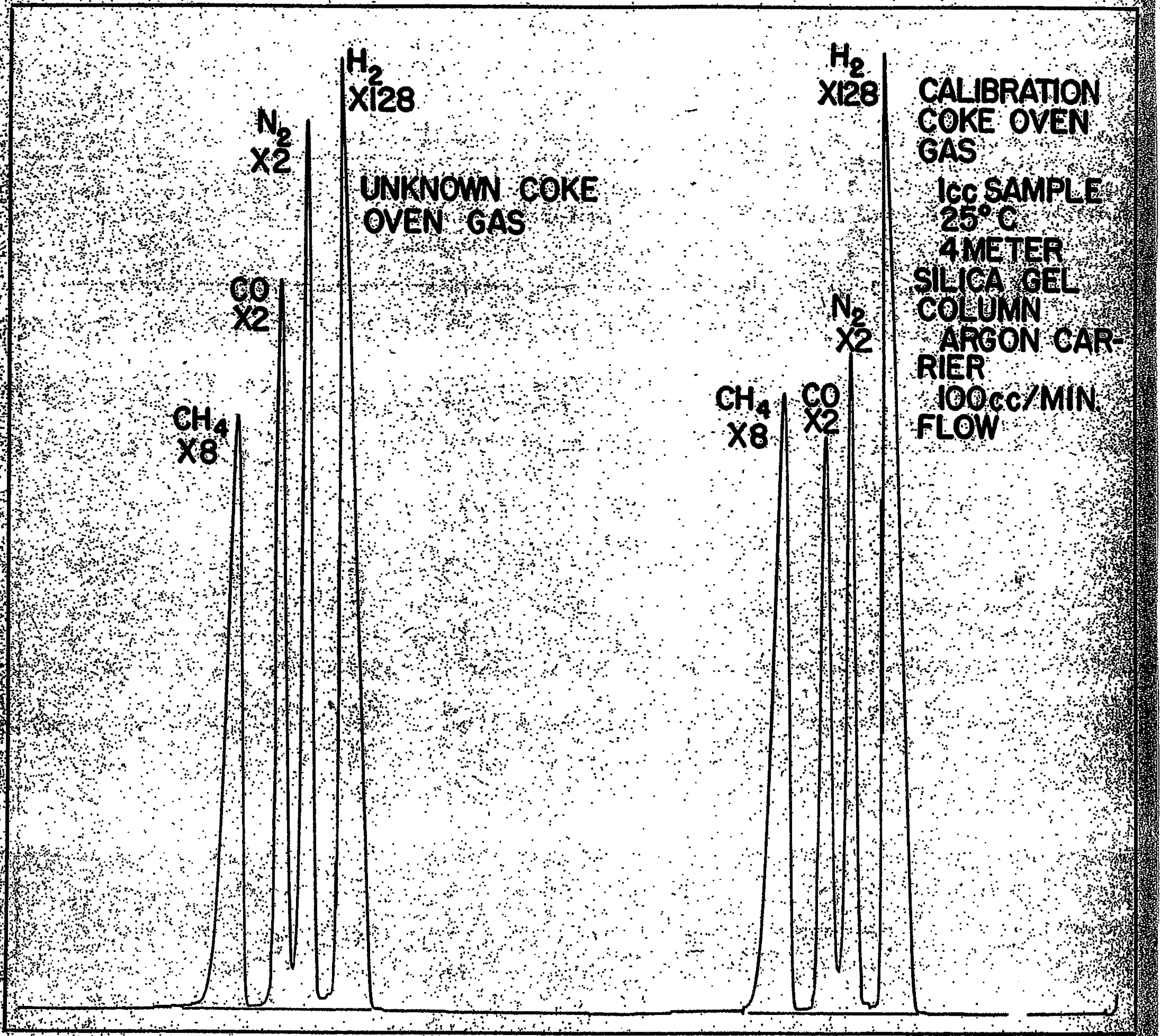


FIGURE 7—Coke oven gas sample: Run clearly shows the major components.

peak heights observed. Separate runs were made on the partition column of the natural gas sample, synthetic blend and pure methane. The analysis is given in Table 1. The pentanes were measured by correcting for half-band width then, using the same calibration curves plotted for the butanes.

It should also be noted that hydrogen, whose peak is in the downscale direction because its thermal conductivity is higher than helium, was included in the synthetic blend. Carbon monoxide was not included, although it is an important component in many fuel gases. Its position is shown by the dotted lines in Figure 2 (calibration runs). For satisfactory separation of this component, probably two 2-meter silica gel adsorbent columns should be used. Silica gel does not provide separation of oxygen and nitrogen; but adsorbents are available that will.

Manufactured Gases—Manufactured gases may be treated in much the same manner as natural gases. In fact some of these gases contain no components heavier than propane, thereby allowing the analysis to be made with a single determination on a silica gel column. Figure 5 shows a chromatogram of a sample of a manufactured gas supplied by a large Eastern distributor. The same calibrated mix used for the natural gas analysis has once again been intermixed during the run so that the bands of N_2, CH_4, C_2H_6, and CO_2 of the standard are followed by the corresponding bands of the sample. The bands of standard and sample propane appear at the end of the run as expected. The peak height ratios were used to determine concentration of these sample components.

It should be noted that two bands of the sample gas representing CO and C_2H_4 respectively, are not contained in the calibration mix. In standard practice the calibration mix for samples of this type would have been formulated to include these gases. In this case a separate run of a gas available in the laboratory and containing a known concentration of CO was run following the above determination, and the peak height ratio of the CO peak in this gas to that of the manufactured gas was used to determine the CO concentration of the manufactured gas. The small amount of ethylene was calculated by area comparison with area under the propane peak in the calibration mix. The final quantitative analysis of the manufactured gas based upon this method of analysis is shown in Table 2.

Coke Oven Gases—Coke oven gas represents a special case in that hydrogen is a major constituent. Since, unlike all other gases, hyrdogen has a higher thermal conductivity than helium, it will cause a negative peak response on the recorder when it appears in a mixture run with helium as a carrier gas. However, if argon is substituted for helium, the hydrogen peak as well as the peaks of all other gases will be in the same direction.

Figure 6 shows a chromatogram of a calibration mix followed by an actual coke oven gas, using argon as a carrier gas and silica gel as a column material. The run clearly shows the major components, hydrogen, nitrogen, carbon monoxide, and methane.

TABLE 3

H_2	56.8%
N_2	5.4
CO	6.2
CH_4	28.1
Other	3.5

The analysis of these components is shown in Table 3 in order. The minor ingredients of the gas, such as propylene, acetylene, and butanes, amounting to less than 4 percent of the mixture, are not measurable on this column, but could have been detected on a partition column with helium carrier gas as previously demonstrated in Figure 3.

This presentation was condensed from the original paper delivered before the American Gas Association, Philadelphia, May 18, 1956.

What is Gas Chromatography?

It's a method that consists of carrying a mixture through a column by means of a carrier gas. The different affinities of the components for the column packing (called the stationary phase) cause them to travel at different rates through the column. They arrive at the column exit separately where they are measured by a suitable detector. Recordings obtained by measuring the detector output result in a series of peaks in which the peak location or characteristic component speed provides the qualitative information. The peak heights or areas can be used for determining percent concentration.

Meet the Authors


Coates


Brenner

VINCENT J. COATES is experienced as a project engineer in design and development of infrared equipment, and as a supervisor for the design and development of laboratory instruments and process applications of Perkin-Elmer Corporation, Norwalk, Conn. Presently he is manager of application engineering with duties as supervisor of special engineering sales and application for the same company. Coates received a B.S. degree from Yale University in 1946.

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THE PERKIN-ELMER

INSTRUMENT NEWS

FOR SCIENCE AND INDUSTRY

Published to further research, material analysis and production through electro-optical instrumentation.

Norwalk, Connecticut WINTER 1958 Vol. 9, No. 2

Model 21 Ordinate Scale Expansion System Extends IR Measurement Sensitivity

by

Vincent J. Coates and Robert Anacreon
The Perkin-Elmer Corporation*

Recent studies and publications have brought recognition of the need for a fresh consideration of a basically important area—trace analysis. In 1955, Stephens, Scott and co-workers at The Franklin Institute published results of what has been a very fruitful study of the organic chemistry of the atmosphere using infrared spectroscopy.[1, 2] The long-path gas cell which they designed also served as a reaction vessel. Photolysis of synthetic mixtures with ultraviolet radiation caused the formation of new compounds characterized and measured by infrared. These compounds occurred in the part per million range, and their accurate measureurement required path lengths of several hundred meters.

A cooperative program, initiated with workers at the Memorial Hospital in New York, led to the development of the infrared microcell which allowed the measurement in solution of as little as 25 micrograms of steroid separated from urine.[3] In order to obtain even this much sample, however, it is necessary to collect samples under controlled conditions over several days. In clinical work much more could be done with infrared if the sensitivity of the method for trace analysis could be extended.

continued on page 10

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[*] From "Improving the Measurement of Weak Infrared Absorption Bands Using Expanded-Scale, Double-Beam Spectrophotometry"—a paper presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March, 1957.

[1] Stephens, E. R.; Haust, P. L.; Doerr, R. C.; Cott, W. E.; Ind. and Eng. Chem. 48, 1948 (1956).

[2] Stephens, E. R.; Scott, W. E.; Haust, P. L.; Doerr, R. C.; Paper presented at the midyear meeting of American Petroleum Institute, Division of Refining, Montreal, Canada, May 15, 1956.

[3] Jones, R. N.; Williams, V. Z.; Whalen, M. J.; and Dobriner, K.; J. Am. Chem. Soc. 70 2024 (1948).

An Examination of Filter Radiometry

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Newport, Rhode Island

The separation of radiant energy emitted by a source into defined spectral components may be attempted either with the aid of a spectroradiometer or, sometimes, more conveniently by employing a filtering technique. Such sources with which we are concerned are generally natural (e.g., the sun and the terrestrial atmosphere) or laboratory (e.g., a tungsten-filament lamp and other artificial radiators). The use of filters, on the one hand, is attractive by virtue of the relative simplicity of the measurements and the low intensities of radiation flux which can be detected; but, on the other hand, where precise results are demanded, careful calibration of filters is essential.

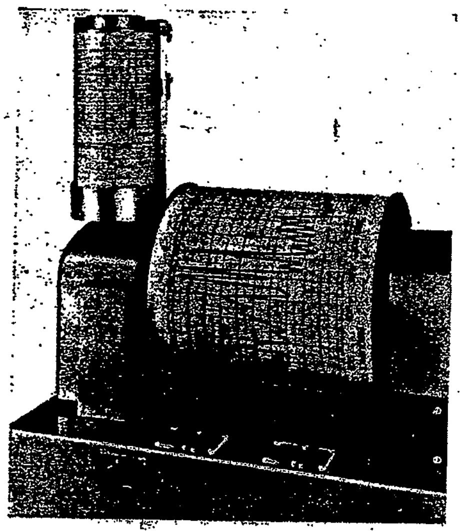
A. J. Drummond

In radiation meteorology, there are numerous applications of spectral data derived from filter measurements. For example, a knowledge of the distribution, with wavelength, of the energy of sunlight is readily acquired by placing a series of colored glass filters, in turn, in front of the receiver of a pyrheliometer which is directed at the sun. These absorption-type filters should be so selected to have well-defined cut-off characteristics; in practice, it has been found easier to produce a sharp spectral cut-off at the lower rather than the higher end of the energy band to be investigated. After correction of the observed intensity for losses through reflection (and, to a much lesser extent, absorption), in the transmitted wavelength band, simple subtraction of one set of measurements from the adjacent set with lower cut-off nearer the ultra-violet end of the spectrum—and so on—yields the desired value for the wavelength band defined by the centers of gravity of the lower cut-offs of the respective pair of filters.

Another equally important aspect

continued on page 6

Model 21 Slave Recorder



Model 21 Slave Recorder (to be shown at Pittsburgh) exactly reduces standard Model 21 spectra to more convenient size (8½" x 11") for handling, mailing, filing. See details on pages 4 and 5, this issue.

VISIT OUR DISPLAY AREAS AT THE PITTSBURGH CONFERENCE, MARCH 3-7, PENN-SHERATON HOTEL. LOOK FOR US IN BOOTHS 7-8, 67, AND IN ROOM 690. SEE YOU THERE!

dependence of filter transmittance ɪn the thickness of the material; com- ison with theoretically derived ues, from a standard set of meas- ments for one thickness (usually 1.0 ı.), would be a good indication of homogeneity of the material with ard to thickness. In Fig. 4 (p. 8), spectral bands resulting from the of a 1 mm. and a 4 mm. thick filter ., measurements treated differenti-) are computed.

he spectral characteristics of two rs are shown, in Fig. 3, (p. 7), for the r infrared; these define three useful ions with boundaries at 700 and ut 1000 mμ.

New Laboratories

he Eppley Laboratory has just com- ed a new group of laboratories and eophysical observatory (for solar lies), where the research and de- pment program will embrace fund- ntal aspects of radiometry and pho- etry, as well as the more routine bration of radiometers and filters.

A special effort will be made to ly the effect of weathering on the smission properties of filters in- led for exposure out of doors. The cation is that the Spectracord will plement excellently the spectro- tometric equipment already avail- at the Laboratory.

STANDARD INFRARED EMISSION LINES

In 1950 Plyler and Peters (Journal of esearch of the National Bureau of Stand- rds, vol. 45, no. 6, 462-467) provided andard emission spectra, in the region om 0.5 to 2.4μ, as obtained from a Gen- al Electric AH-4 mercury lamp. These ata are of prime importance as reference avelengths in spectrographic calibra- ons.

Within limitations of ± 1 mμ, the results f Plyler and Peters were confirmed re- ntly at the Eppley Laboratory, using e new Perkin-Elmer Spectracord® 4000 pectrophotometer with resolution of 1 μ at a wavelength of 0.5 μ, decreasing values of between 3 and 5 mμ in the frared region examined.

The Eppley Laboratory, Inc. has asked erkin-Elmer to make known the follow- g errors in mercury emission line data, pearing in authoritative handbooks of ysical tables.

	Printed as	*Should read*
Handbook of Chemistry and Physics." (39th Edition, 1957-1958, page 2695)	18333Å	18131Å
American Institute of Physics Handbook." (1st Edition, 1957, section 6, page 88)	1.52452μ	1.52952μ
nternational Critical Tables." (1st Edition, vol. 5, 1929, page 299)	18333Å	18131Å

will be noticed that the lower wave- gth has been published 5 mμ in error d the higher wavelength 20 mμ in ror. Previous editions of the "Hand- ok of Physics and Chemistry" contain e important latter one for the last years at least.

The Performance of the P-E Model 21 In the Cesium Bromide Region (15-38μ)

by

Robert E. Anacreon
Supervisor, Sales Laboratory
The Perkin-Elmer Corporation

We are constantly trying to increase our knowledge of the molecule by studying its characteristic infrared vibrations. Considerable information, not clearly developed, however, within fundamental infrared wavelengths (2.5-15μ), is fortunately more clearly provided in the *far* infrared (15-38μ). Slight changes in structure may produce changes in the far infrared spectrum. Far infrared spectra are more sensitive to crystal structure, and molecules that exhibit polymorphism are distinguished by their spectra in the solid state. Characteristic ring vibrations for substituted aromatic compounds occur here. The fundamental vibrations of inorganic crystals such as metal halides and the bond-stretching and skeletal bending vibrations for heavy molecules such as chlorine, bromine, iodine, sulphur and silicon, appear in the far infrared spectrum.

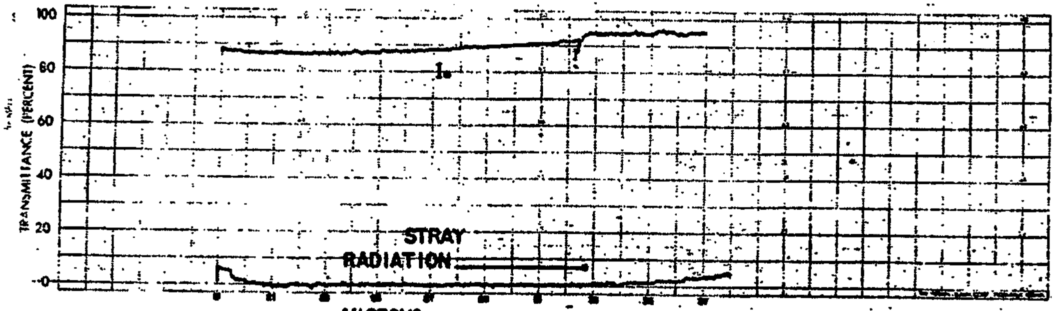
When spectra are to be recorded in the far infrared or cesium bromide region, two factors can affect accuracy of results: lowered energy and stray radiation. Actually these two factors are inter-related. In the cesium bromide spectral region (15-38 microns) following the black body curve, the infrared source energy is low. There are also very strong interfering atmospheric bands in the 15-38μ region. In order, therefore, to obtain enough energy for good response of the instrument, it is necessary to widen the slits of the monochromator. Stray radiation then becomes a problem, since it varies as a direct function of the slit width.

Stray Light with CsBr Prism

An instrument equipped with the cesium bromide prism should, therefore, incorporate some means for reducing stray light. Double-pass or double-monochromator instruments succeed in reducing stray radiation to very nearly zero, but at the further expense of already low energy and wavelength range. The reasons for this loss in energy are as follows:

1. There are losses in the photometry of a double-pass system because of the introduction of a second chopper.
2. Radiation is being passed through the prism twice, causing greater energy loss through prism absorption and reflection, especially at the long wavelength limit.
3. There are an increased number of mirror reflections, causing further energy reduction.

Figure 1



Limited Instrument Range

As an example of the limited range of a double-pass or double-monochromator instrument, we find it is only possible to record to about 35 microns, with a CsBr prism, even when the instrument is flushed with dry air to reduce water interference.

Using the standard single-pass Model 21 equipped with the cesium bromide prism, we can record to 38 microns and still maintain good pen response. All that is needed is some means for reducing the stray radiation (which normally is about 4% at 30 microns and rises to over 40% at 38 microns) to a value which would not seriously interefere with accurate measurements in this region.

James Stewart of the National Bureau

The Model 21 in the CsBr Region

(continued from page 9)

of Standards in Washington solved this problem by mounting two Reststrahlen mirrors in back of the exit slit of the Model 21 monochromator. One is lithium fluoride, and the other is calcium fluoride. We investigated these materials and found that lithium fluoride was very effective in reducing scattered light, up to 30 microns. This is just as efficient as standard polyethylene and Eastman Kodak silver chloride filters which are normally used in a Model 21 equipped with a cesium bromide prism. In the region between 30 and 38 microns the use of a calcium fluoride plate was extremely effective in reducing stray radiation. Furthermore, its reflection is greater than 80% through most of this range, thus maintaining energy.

Fig. 1 (p. 9) shows the scattered light measurement out to 38 microns (a sodium chloride window was used in the sample beam). It is not measurable out to 33 microns. At 37 microns it is only 4%. This is a considerable improvement and is the amount of scattered light that one normally can easily correct (based on detector noise at these long wavelengths). In addition, a stray radiation measurement between 15 and 30 microns using the standard Eastman Kodak silver chloride filter and the aluminized mirror in back of the exit slit (a calcium fluoride window was used in the sample beam), showed no measurable stray radiation in the region 15 to 30 microns.

Figure 2 shows a spectrum of ammonia recorded out to 38 microns. The region between 33 and 38 microns was run twice to show reproducibility. The transients along the base line indicate where the beam was deflected and the pen allowed to return to the base line. There was good pen response and good reproducibility in this region. Extreme difficulty would be encountered in recording these bands on a double-pass or double-monochromator instrument.

Bromo-Benzene Spectrum

Figure 3 shows the spectrum of bromobenzene in a .25 millimeter cell. Note that the totally absorbing band at 22 microns measures zero as does the band at 32 microns.

It is important to note that when spectra are recorded in the cesium bromide region, special care must be taken to choose optimum instrument operating conditions. The reduced energy in this region (as compared to the rock salt region) makes the use of slower scanning speeds and adequate filtering essentials for good spectral recording.

Figure 2

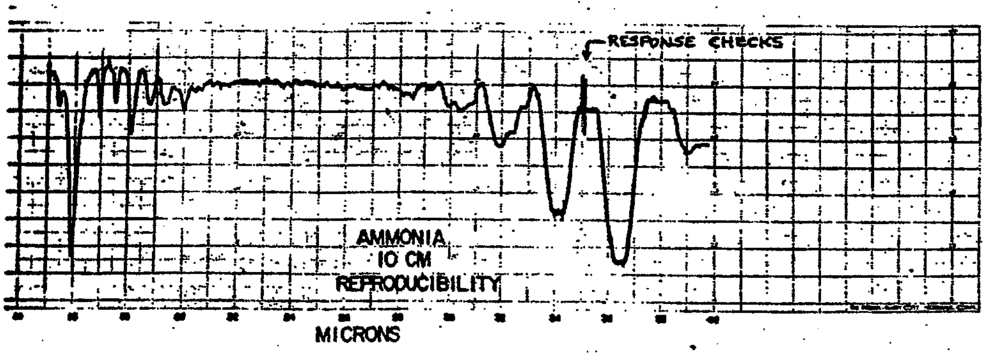
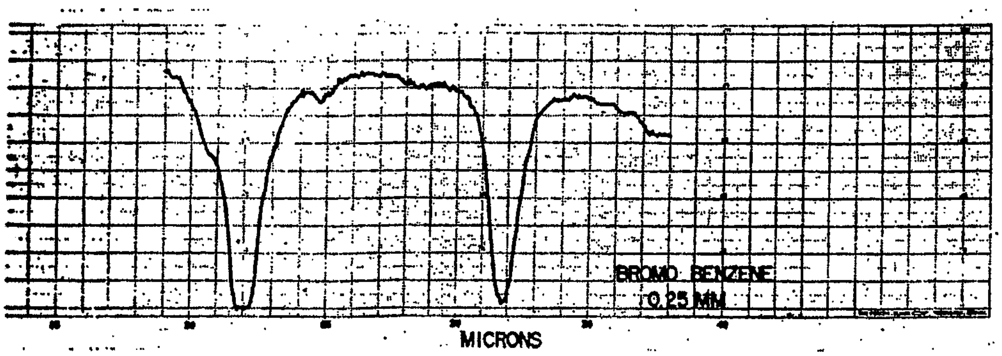


Figure 3



Model 21 Scale Expansion

continued from page 1

Surface Chemistry Studies

About two years ago discussions were held with spectroscopists of a chemical company interested in the use of infrared for surface chemistry studies. Monomolecular films produce such weak absorption bands that existing instruments were incapable of recording them. Multiple traversals through the film increase absorption and reveal much useful information, but large energy losses are encountered.

It should be noted that the performance which has been sought in the cases cited above required careful design of the sampling system. Perhaps as expected, sampling arrangements for trace analysis are usually pushed to the limits of optimum performance and convenience.

Further steps are being taken in sampling system design. However, the fast-moving fields of air pollution control, clinical analysis, and surface chemistry, mentioned above, and others such as the low temperature study of free radicals, and the identification of small quantities of materials separated by gas chromatography call for large factors of performance improvement.

Examination of Performance Criteria

The amount of infrared radiation absorbed at a given wavelength by a sample placed in the beam of an infrared spectrophotometer can be determined from the Lambert-Beers Law which reduces to the familiar expression,

$A_\lambda = \alpha_\lambda cl$ where A_λ = absorbance
α_λ = absorptivity
c = concentration
l = path length

The concentration and path length define the number of molecules in the beam, and the absorptivity defines the ability of the molecule to absorb radiation. For many samples, it is possible by adjusting concentration and path length to measure absorbance quite accurately and to obtain an infrared spectrum which can serve for qualitative identification or quantitative analysis over wide concentration ranges. It has been shown that the maximum change in transmittance is observed for a given change in concentration when the absorbance equals e^{-1} or approximately 0.4 of an absorbance unit. Thus on a linear transmittance instrument such as a double-beam spectrophotometer, maximum accuracy is observed at this point.

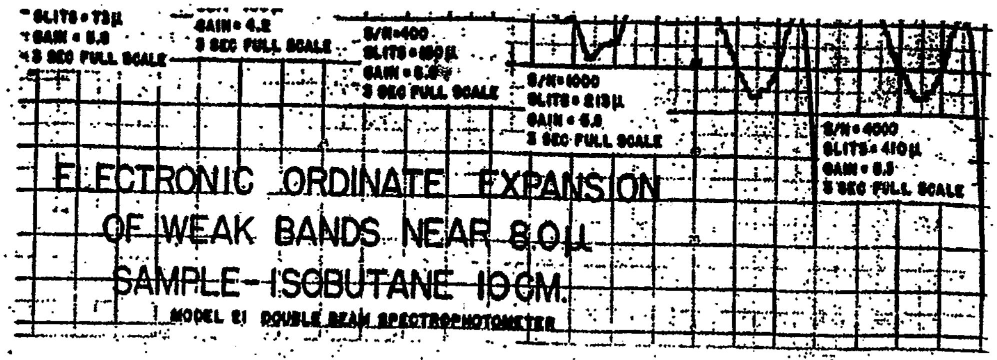


Figure 2

in solution, the background absorption of the solvent at the sample wavelength can affect the accuracy of measurement.

By the use of double-beam differential analysis, which involves placing a matched cell containing the solvent in the reference beam of the spectrophotometer, the solvent absorption interference can be cancelled.[1] This reduces the reference beam signal, which in turn lowers the signal-to-noise ratio depending on the amount of radiation absorbed by the solvent. If measurement precision is to be maintained, electronic filtering must be employed or slit width increased (with an accompanying decrease in resolution). This produces slower pen response and requires slower scanning rates. For example, analyses have been attempted where the cell thickness chosen resulted in background solvent absorption of all but 2% of the total beam energy. Placing 2% of the beam energy full scale results in a substantial increase in measurement sensitivity. It is interesting to note, however, that at a transmittance of 2%, the absorbance is about 1.6 units. If a cell ¼ as thick were used, the absorbance would be 0.4 unit and the transmittance, 40%. The ratio between 40% and 2% is 20x. Thus, a factor of 20 in energy is given up to gain a factor of 4 in sensitivity. It appears that in differential analysis a large price must be paid in the signal-to-noise ratio for measurement sensitivity once the absorbance of sample plus solvent exceeds 0.4 absorbance unit, i.e., the e^{-1} point.

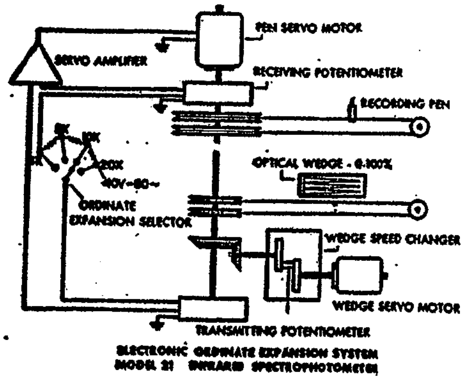


Figure 1

Trace-Gas Analysis

A similar limitation occurs in atmospheric trace-gas analysis where long path lengths are required. As mentioned above, the use of folded-path, multiple-traversal cells of path lengths of up to 500 meters have allowed part per million components to be measured at atmospheric pressure. An examination of the design criteria for these cells which utilize numerous mirror reflections in their operation reveals that maximum efficiency is achieved when,

$$R^n = \frac{1}{e} \quad \text{Where } R = \text{reflectivity}, \; n = \text{number of passes}$$

If gold mirrors are used, reflectivities of the order of 0.975 are obtained in the infrared region. For this value, the optimum number of passes is 40. If the base length of the cell is one meter, then 40 passes will result in a 40-meter sample path length.

In this cell, increasing the sample path length to 160 meters requires 160 passes and results in an efficiency of 2.5%. A factor of 16 in efficiency is given up to gain a factor of 4 in detectability.

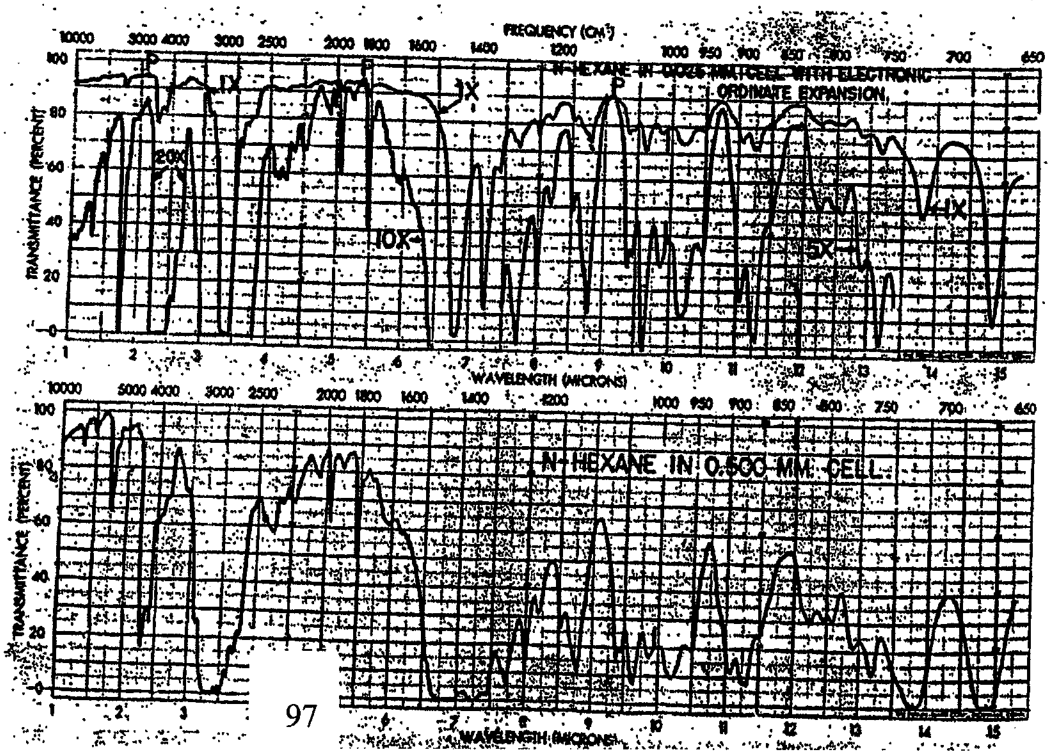
This limiting situation is similar to the example cited above for solvent absorption.

The other area where weak absorption limits measurement precision is in surface film studies. Here, absorbed mono-molecular layers of the order of five to several hundred Ångström units are encountered. Even for bands with large absorptivities, the largest absorbance which will be observed in a single film thickness is of the order of 0.01 absorbance unit. Multiple traversal sampling systems can be constructed, but their efficiencies are limited by factors similar to those encountered in multiple traversal gas cells.

A Novel Approach

This situation has caused us to examine the instrumentation problem to determine if less strain might be placed on the sampling system and more on the measuring instrument itself. In present quantitative work, it is not unusual to set up an analysis to provide detectability of the order of 1 part in 50 or 1 part in 100. To achieve this performance, signal-to-noise ratios of 100 or greater are employed by careful adjustment of basic instrumental para-

Figure 3



Appendix: XVI

Model 21 Scale Expansion System for Trace Analysis

continued from page 11

meters—slit width, amplifier gain, and amplifier band pass.

Under ideal conditions, the best S/N ratios obtainable from single-beam infrared spectrometers are of the order of 400 (the basic noise at this level being caused by source intensity fluctuations due to line voltage changes).

It has not been completely appreciated that double-beam optical-null spectrophotometers which provide automatic cancellation of source fluctuation can be adjusted to achieve S/N ratios of up to 4000. This is achievable by using increased slit widths up to 7-times those used for routine analysis. Since energy is gained as the square of slit width, a 7x slit change results in a 49x increased signal level. (It should be mentioned here that a control for maintaining stable servo mechanism performance under these high signal levels *must* be available.) With a S/N ratio of 1000 to 5000, a problem arises. It is not possible to read transmittance differences of 0.01% or less with a 20 cm. chart plotting 0-100% transmittance. Since it is obviously impractical to increase chart size, ordinate expansion is employed. In other words, for a given transmittance change, a larger change in pen position must occur which will allow the operator to discern these small differences.

Single-Beam Scale Expansion

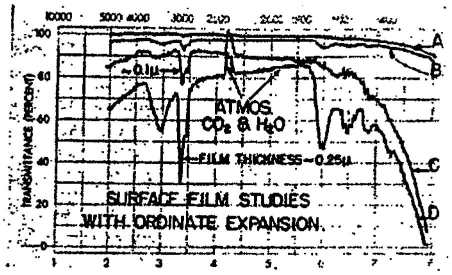
Most single-beam, infrared spectrometers are equipped with "zero suppression" controls which allow the zero transmission level to be depressed, thus expanding the upper transmittance level. Ordinate expansions of 2 or 3 are possible by this means before the basic S/N ratio limitation is observed.

Double-Beam Electronic Scale Expansion

Electronic ordinate scale expansion is a versatile and convenient method to increase sensitivity based on the ultimate signal to noise capability of the double beam spectrophotometer.

In the original Model 21 Infrared Spectrophotometer, the position of the optical wedge is measured through a direct mechanical coupling to the recorder pen.

In Figure 1 (see p. 11), for electronic ordinate expansion, the wedge position is transmitted electrically by means of a precision potentiometer coupled to the wedge-drive shaft. This potentiometer, in turn, is part of a bridge circuit in which a follow-up potentiometer is driven to null balance by a servo motor. The pen is driven at the same time by this motor to its proper ordinate position. An electrical switching circuit allows expansions of 1x, 5x, 10x and 20x. Another control allows any portion of the optical wedge motion to be expanded. Thus, if it is desired to expand the range 30-40% T, the ordinate expansion selector is turned to 10x, and the pen position control is adjusted until the 40% T point is at the top of the recorder paper.



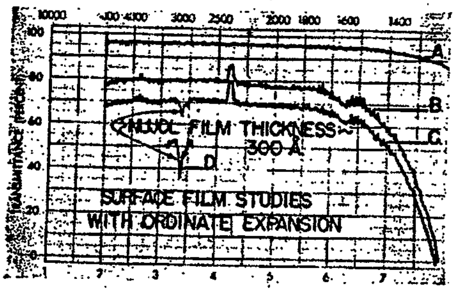
(A) CaF_2 PLATE WASHED WITH CH_3OH.(IX) (SPECTRO GRADE)

(B) CaF_2 PLATE WASHED WITH CH_3OH AND COATED WITH NUJOL.(IX)

(C) SPECTRUM (A) ORDINATE EXPANDED 10X.

(D) SPECTRUM (B) ORDINATE EXPANDED 10X.

Figure 4



(A) CaF_2 PLATE WASHED WITH CCl_4 (SPECTRO GRADE) 1X

(B) SPECTRUM (A) ORDINATE EXPANDED 10X

(C) SAME PLATE COATED WITH THIN FILM OF NUJOL 10X

(D) SPECTRUM (C) 20X

Figure 5

Examination of Iso-Butane

The use of the electronic ordinate expansion is shown in Figure 2 (p. 11). A 10 cm gas cell containing iso-butane is placed in the sample beam of the spectrophotometer and the region 8.0-8.3μ is scanned. Weak absorption structure is absorbed using a 1x ordinate scale with a signal-to-noise ratio of 100. This triplet is difficult to identify or measure with any degree of precision. By decreasing the noise on the recording paper by using increased slit widths and lower amplifier gain, the readability is slightly improved, but not enough to allow a large increase in measurement sensitivity. However, with a signal-to-noise ratio of 400, using a 5x scale expansion, a real gain in sensitivity is observed. Similarly, by increasing the slits to obtain a signal-to-noise ratio of 1000 and using a 10x expansion, a further gain in sensitivity is obtained. When the signal-to-noise ratio is increased to 4000 the triplet is no longer resolved. However, even with low resolution, gain in sensitivity is observed when a 20x scale expansion is used under these conditions. To indicate that a signal-to-noise ratio of 4000 was obtained with stable long-term performance, three separate runs were superimposed, and excellent reproducibility was obtained. The steps observed under the 20x condition indicate that the resolution of the slide wire in the transmitting potentiometer was limiting. By using a potentiometer with a higher resolution degree, these steps are eliminated.

Figure 3 (page 11) shows electronic ordinate expansion for a pure spectra study with a 0.025 mm. cell filled with normal hexane. The various regions of the spectrum were expanded to bring out weak structure. The performance under these conditions can be compared to the spectrum of normal hexane run in an 0.5 mm. cell with a 1x scale.

Mono-Molecular Film Analyses

Figures 4 and 5 show the application of electronic ordinate expansion for surface film studies where extremely thin films which give very weak absorption bands are measured. The application of infrared to mono-molecular film studies now become a distinct possibility.

Conclusion

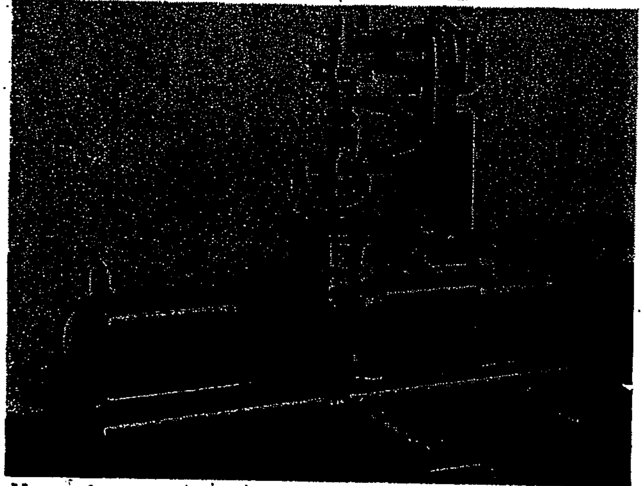
The data indicate that the use of double-beam infrared spectrophotometers equipped with means of ordinate expansion under stable operating conditions lead to a real gain in the ability to perform trace analysis. This gain is obtained basically by using the control of signal-to-noise ratio, servo stability, and resolution available in the double-beam spectrophotometer.

THE PERKIN-ELMER INSTRUMENT NEWS FOR SCIENCE AND INDUSTRY

Published to further research, material analysis and production through electro-optical instrumentation.

Norwalk, Conn. SUMMER 1953 Vol. 4, No. 4

New Infrared Microscope Attachment Gives Routine Spectra of Single Crystals, Fibers or Tissues



New microscope mounts directly on monochromator case of Models 12, 112 or 13 Perkin-Elmer Infrared Spectrometer.

Natural and synthetic fibers, single crystals, tissue sections, bacterial cultures, all have been examined and have yielded excellent, reproducible infrared spectra by means of a new infrared microscope attachment for Perkin-Elmer Model 12, 112 and 13 Spectrometers. Useful spectra have been obtained with as little as 0.1 microgram of sample. The new microscope should find wide applications in crystal and fiber studies as well as in medical infrared studies.

• **Requirements** – The design requirements of infrared microscopes, or more accurately, infrared micro-sampling attachments, have been rather thoroughly explored in recent years, and a number of systems have been constructed. All of these instruments have the common property of measuring, as a function of wavelength, the infrared absorption of minute samples. The resulting absorption spectra are similar to the infrared spectra of macroscopic samples so that the well-known applications of infrared—qualitative analysis, quantitative analysis and molecular-structure investigations—are possible.

Infrared microscopes have been shown to be useful where:

(a) amount of sample is small,
(b) dimensions of sample are small,
(c) sample is non-homogeneous.

• **Design** – The arrangement of optical parts is indicated on page 7. Energy from the exit slit of the monochromator is incident on a field mirror which directs it upward and forms a reduced image of the pupil of the monochromator (the Littrow mirror) near the convex mirror of the condenser. By this means, light from the entire useful slit length is concentrated at the sample. The condenser forms a reduced image of the exit slit at the sample space, and at the same time reimages the Littrow mirror near the center of curvature of the objective mirrors. The objective collects the energy which has passed through the sample, and then magnifies

Continued on page 7

Perkin-Elmer TV Color Projector Big Success at AMA Meetings

Live color telecasts of medical procedures were shown on a four by six-foot screen for the first time to thousands of doctors during the recent American Medical Association meetings in New York. A special projector, equipped with Perkin-Elmer Schmidt-type optics and Columbia Broadcasting System Laboratories electronics, made the large-scale projections possible. Inspecting the unit are Joseph E. Bambera, left, CBS project engineer, and Dr. Rod S. Scott, of Perkin-Elmer.

Reactions to Proposals for a Pure Spectra Program

Early in 1952, a suggested basis for a pure spectra program was mailed to over three hundred infrared users. Various aspects of this proposal were discussed in INSTRUMENT NEWS (*2*, 3, *2*, and *2*, 4, 2). More than one hundred and fifty replies were received, of which about eighty were sufficiently detailed to permit a statistical study. We wish to take this opportunity to express our appreciation to those who answered, and particularly to those who gave thoughtful, detailed responses.

The aim of the survey was two-fold: first, to obtain information on field preference to guide instrument design; and

Continued on page 6

PURE SPECTRA PROGRAM

Continued from page 1

second, to evaluate the possibility of such a centralized pure spectra program. The results of the five major aspects of the questionnaire were as follows:

I. Spectral Presentation and Sample State Specifications:

70 percent felt the spectral specifications were good, 25 percent felt they were too tight, and 5 percent wanted a tighter specification in one respect or another.

35 percent normally worked to the accuracy specified.

75 percent would accept cm^{-1} as the abscissa scale (this does not imply that only 25 percent would accept wavelength, if such had been proposed).

A small (but vehement) percentage wanted absorptance rather than transmission plotted upward.

With respect to sample state specifications, there was some desire for solids in compensated solution rather than as films or mulls. This is a difficult question, and the KBr pressing technique may prove to be an acceptable solution.

- **Transmittance vs. absorbances** – The most thoughtful and concentrated objection to overall presentation was the choice of transmittance rather than absorbance. This group felt that absorbance or an absorptivity, as common in the ultraviolet and visible, is a more fundamentally useful scale and that it is not prevalent in infrared because of instrument and sampling difficulties which will eventually be overcome, that any thorough spectral collection should presuppose the future in this regard.

II. With regard to the bugaboo of ensuring sample purity, 80 percent felt that the proposed method was as good as could be devised without excessive expense.

III. In general, spectra would be supplied from outside sites only when spectra to the same specifications were requisite for internal programs at that site. The number that could be obtained from outside sites was appreciable, but not sufficient to carry the program.

IV. The eight spectral sample classifications in order of preference were: (1) Basic Organic Chemicals; (2) Solvents and Common Gases; (3) Biochemicals (Food and Drugs); (4) Polymers, Monomers and selected Compounds; (5) Inorganic Materials; (6) Miscellaneous End Products; (7) Petroleum Products; (8) Miscellaneous Raw and Intermediate Materials. The order is fairly predictable with Petroleum Products occupying its position because of intent not to overlap A.P.I. spectra in the early phases.

- **Sales Potential** – A point of major consideration was the question: Would such spectra sell at $3-$5 apiece in sufficient quantity to be economically feasible? A clear-cut answer was not possible from the returned questionnaires because: 1) many "yesses" were accompanied by some qualification; 2) no data could be obtained from people who would become infrared users in the future—and they would be more likely purchasers; 3) allowance for "snowballing" after initial acceptance is difficult to judge. The impression obtained from overall study was that such a proposal could be economically feasible, that a significant number of people would pay $5.00 for an accurate infrared spectrum of a pure material.

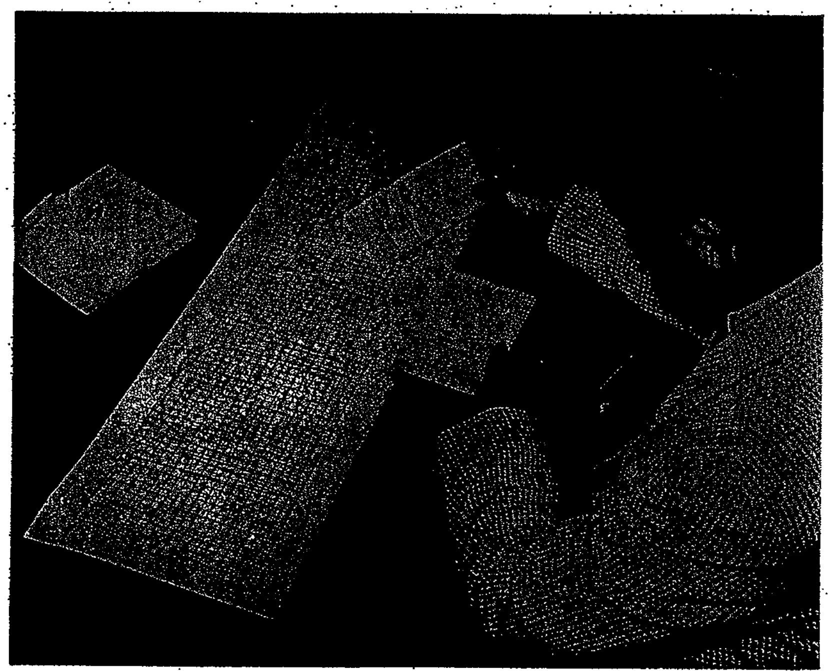
The point that gave pause, however, was as to how the spectrum should be provided. There was complete agreement that the $5.00 should include the original spectrum since this is always required for ultimate measurement, check or comparison. However, the original spectrum is a bulky beast and does not permit casual handling or frequent reference.

A relatively large percentage realized and stated that, in addition to the original spectrum, a McBee or I.B.M. card is required and most felt that this should be provided too. In fact, 75 percent stated that they would not buy the originals if the reduced copies of the spectra were available through the N. R. C.-N. B. S. punched card index. The summary of all the comments gave the impression that what the field would purchase for $5.00 was a punched I.B.M. or McBee card, in which there was mounted a microfilm of the original spectrum with provision for projecting the microfilm spectrum on a laboratory spectrum for comparison.

In summary, it was concluded that the proposal for a Pure Spectra Program should wait for: 1) instrumentation that will provide the spectral range, accuracy, and logarithmic ordinate without the checking and care necessary today; 2) better control of sample thickness and solid state handling; and 3) a good combination of providing both the original spectrum and spectral index card. Of these three, the first is the major effort.

V. Z. W.

What size spectra do you want to record?



Want to record spectra on IBM cards, on McBee cards, or on some other special size forms? You can do it quite easily on any Perkin-Elmer Model 21 Infrared Spectrophotometer simply by choosing the proper abscissa scale (by means of the change gears provided) and by reducing the ordinate with the balance control. Energy is lost directly as the ordinate is reduced but this is usually not serious. An attachment for reducing the ordinate without energy loss is under consideration, as is the use of smaller chart paper for survey spectra. The McBee card pictured was supplied by the National Bureau of Standards.

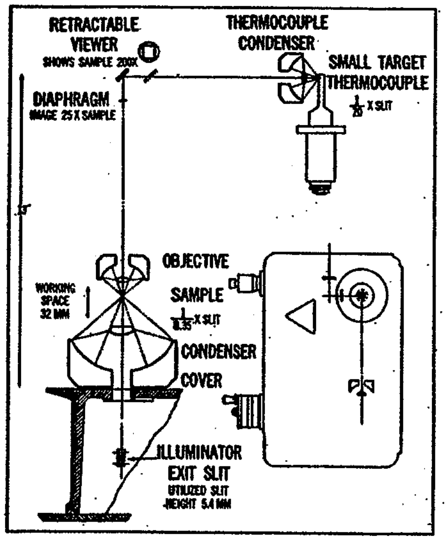
NEW MICROSCOPE

Continued from page 1

the image of the sample at an adjustable diaphragm. The energy then passes to a second field mirror which forms an image of the Littrow mirror near the center of curvature of the thermocouple condenser. The thermocouple condenser forms a reduced sample image on the thermocouple.

The condenser reduction is about 8.5X, the minimum required to fill the aperture of the objective with radiation from the spectrometer. The maximum sample area is 650 x 220 microns which corresponds to a maximum slit height of 5.4 mm and the maximum slit width of 2.0 mm. The sample image at the adjustable diaphragm is magnified 25X. A viewing system consisting of a transfer lens and a 7X eyepiece can be used to observe the image of the sample at the adjustable diaphragm by moving the viewing mirror into the light path. The diaphragm is adjusted to frame the sample while the sample is being observed at 175X magnification. An image of the exit slit is provided in the sample space by an auxiliary diffuse light source so that the sample may be centered.

The system is composed entirely of reflecting elements, so that it is free of chromatic aberration. In addition, the useful wavelength range is not limited by absorption in refracting elements. The Perkin-Elmer Infrared Microscope has been placed in the dispersed beam between the exit slit and the detector, thus avoiding the disadvantages of heat or photochemical effects on samples.



Optical schematic diagram of Infrared Microscope attachment.

The focal length of the objective is 8 mm, and the working distance is 32 mm.

The design allows 5.4 mm of the slit height to be utilized with the microscope. Since this is about half the macroscopic slit height, the height of the thermocouple target was reduced by a factor of two with a resultant gain in the thermocouple detectivity.

• **Optical Elements** – The optical elements are mounted on a convenient and stable mechanical system. Focussing and locking adjustments are provided for the objective. Positioning of the sample is accomplished by coarse and fine focussing adjustments, rotation of the stage and x and y translation using a mechanical stage. The illuminator and viewer allow accurate and reproducible positioning of the sample. The adjustable diaphragm, used to restrict the field to the area covered by the sample, is rectangular in shape, and its length and width are independently and continuously variable. There is also a slide which allows insertion of special diaphragms cut as masks for irregularly shaped samples.

Continued on next page

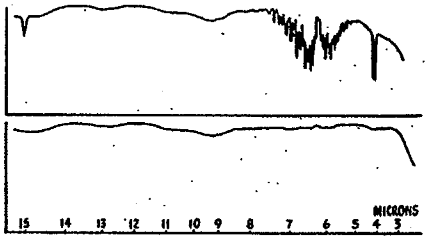
Eliminating H_2O and CO_2 Bands from Infrared Spectra

by E. V. Rouir and H. G. Dietz

Numerous samples of plastics, plasticizers, rubbers (natural and synthetic) and lubricants are examined daily in our laboratory. The rapid analysis of these materials, often available in limited quantities, is only possible by means of infrared spectrometry. In order to reduce the time required for obtaining the absorption curve of the product to be studied, our single beam spectrometer (Perkin-Elmer Model 12C) has been modified so as to obtain a direct recording of transmission percentage.

Before air is passed into the spectrometer enclosure, it is freed of H_2O and CO_2 by being streamed over a column of activated alumina. This product is prepared by the *Société d'Electrochimie d'Ugine* under the registered trademark "Activalum." The S1 grade adsorbs with an efficiency of 100 percent 12 to 14 g of water, using 100 g of dry product, from air at 30° C having a relative humidity of 90 percent. The quantity of CO_2 adsorbed is approximately 3-5 g. The regeneration of the alumina is effected by heating for several hours at 200° C in an air stream, followed by cooling in a closed circuit.

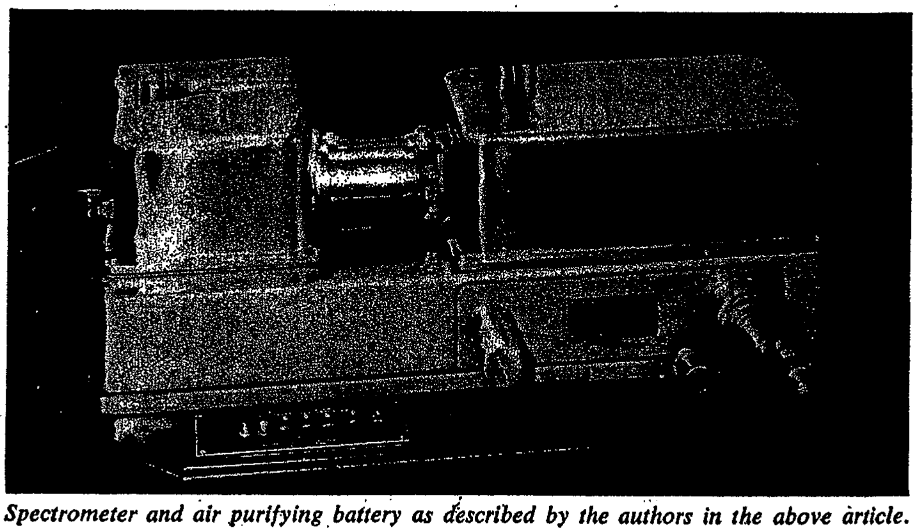
A group of 2 tubes has been connected to a compressed air intake enabling one tube to be used during the regeneration of the second. Each tube contains 3 kg of alumina and permits a three-day working period. In order to obtain a satisfactory purification of the atmosphere, the two housings of the spectrometer have been connected by a bellows.



Globar spectrum before and after elimination of H_2O and CO_2 from the air in the spectrometer enclosure.

The first housing is extended by means of a protruding tube. The cell is fixed at the end of this tube, and it is brought into direct contact with the entrance slit of the monochromator. In this way the optical path remains entirely in an atmosphere free of H_2O and CO_2.

Submitted by E. V. Rouir and H. G. Dietz, Physics Research Laboratory, A.C.E.C., Charleroi, Belgium.



Spectrometer and air purifying battery as described by the authors in the above article.

NEW MICROSCOPE

Continued from preceding page

The microscope attachment is mounted on the monochromator cover. It is so designed that all the standard macroscopic functions of the spectrometer are preserved and the change from macrooperation to micro-operation may be made quickly. Access is provided for easy prism interchange. In addition, provision is made for flushing the optical path with dry nitrogen to reduce CO_2 and water vapor absorption.

- **Operation**—There are two fundamental points which should be mentioned in discussing the operation of a microscope. The first involves the size of sample necessary for obtaining useful spectral data, while the second concerns the purity of the radiation striking the microspectrometer detector.

In practice, sample size is restricted by limitations on both thickness and cross-sectional area. The thickness of a particular sample is restricted to a certain range in order to obtain the proper strength of infrared absorption. The required thickness is almost the same as for macroscopic work, very frequently being about 25 microns, a convenient value for infrared microspectrophotometry. The minimum sample area is that required to provide sufficient energy for satisfactory detection. A significant decrease in minimum sample area can be made by using a high intensity infrared source (carbon arc, zirconium arc or tungsten glower) although this has not been incorporated in this instrument as yet. Additional reduction in sample size may be realized by employing photoconductive detectors in their limited spectral ranges.

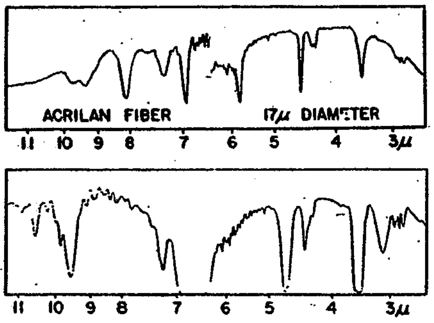


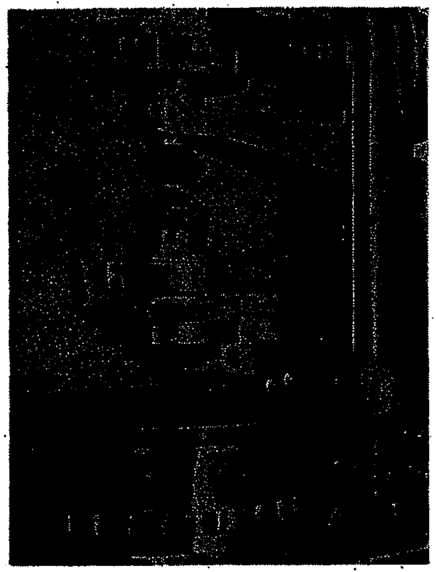
Fig. 1. Infrared spectrum of single fiber of Acrilan, above, and cholesterol in CS_2 (AgCl capillary cell), below.

- **Sample Area**—Because of the variety of factors affecting energy and their variation with the wavelength, no single minimum sample area can be stated as the limit even for a single instrument.

It is possible to assume a particular set of operating conditions for the present instrument and to state the area limit for these conditions. This has been done to provide a *guide* to minimum sample area. The data, which neglect diffraction effects, are given in Table I, below.

The reduction of impurity radiation *i.e.*, radiation passing around the sample, is especially important for quantitative work involving long, narrow samples such as fibers. The Perkin-Elmer Infrared Microscope provides for such reduction in several ways. The viewer system allows precise alignment of the slit image, sample and diaphragm opening. The diaphragm is adjustable so that it can be made slightly smaller than the sample image. The large numerical aperture of the objective reduces the size of the diffraction pattern. The objective is free from coma, astigmatism and chromatic aberration. The spherical aberration is ¼ wave, at the short wavelength end of the infrared spectrum and becomes negligible at long wavelengths.

- **Performance** — The spectra shown here were recorded with the infrared microscope mounted on a Perkin-Elmer Model 112, Single-Beam, Double-Pass Spectrometer. The standard globar source, a 60° NaCl prism, and the usual amplifying and recording system were used. The detector was a Perkin-Elmer small-target (1 x 0.2 mm) thermocouple, designed for use with the microscope. Using the full field of the microscope and identical slit widths, the energy transmitted by the microscope is about 35 percent of that transmitted by the spectrometer.



Close-up detail of microscope. Note diaphragm knobs at top. Sample stage area can be flushed with dry nitrogen to reduce H_2O and CO_2 absorption.

- **Fiber Spectra**—Figure 1 shows a spectrum obtained with a fiber of Acrilan 17μ in diameter. The size of the diaphragm opening when imaged at the sample was 10 x 650μ. The optical path was flushed with dry nitrogen to reduce water vapor and CO_2 absorption. The time of running the spectrum was 15 minutes.
- **Liquid Spectrum**—A short length of silver chloride capillary tubing was filled with a 20 percent solution of cholesterol in carbon disulfide, and used to obtain the spectrum shown in Figure 1. The diameter of the hole in the tubing was 102μ. The diaphragm opening at the sample was 50 x 650μ. Nitrogen flushing was used in taking this spectrum and the recording time was again 15 minutes. It is shown by the complete CS_2 absorption at 6.5μ that no dilution occurs with a sample of this size.

Table I. **GUIDE TO MINIMUM SAMPLE AREA**

Wave-length (in μ)	Signal Peak-to-Peak Noise	Time Constant (in sec)	Sample Height (in μ)	Sample Width (in μ)	Area (in μ²)	Spectral Slit Width (in cm⁻¹)
2	48/2.5	2.1	100	6	600	34
4	40/2.5	2.1	100	8	800	20
6	47/2.5	2.1	100	17	1700	12
8	48/2.5	2.1	100	25	2500	6
10	44/2.5	2.1	100	35	3500	3.2
12	41/2.5	2.1	100	50	5000	3
14	33/2.5	2.1	100	100	10000	3

Abstracted from a paper to appear in J. Opt. Soc. Am.

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Interferometric Method of Measuring the Spectral Slit Width of Spectrometers*

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(Received December 20, 1954)

The spectral slit width of a monochromator is defined as the half-intensity band width of frequencies passed by the exit slit. This paper discusses its measurement using methods similar to those used to measure the frequency transmission of electrical networks. A Fabry-Pérot interferometer, used as a sinusoidal signal generator, produces interference fringes constantly spaced in wave numbers at a known amplitude input to the monochromator. It can be shown that the attenuation of the fringe pattern as the spacing is changed is a function of the exit-slit output-intensity distribution function. From this information the spectral slit width in any spectral region can be measured and the exit-slit function indicated.

The sensitivity of measurement is limited by the fringe amplitude obtainable from the interferometer, which is a function of the reflectivity of the interferometer plates and their adjustment. Rays passing through the interferometer at other than normal incidence tend also to damp the fringe pattern.

INTRODUCTION

SPECTRAL slit width, defined as the frequency interval, $\Delta\nu$, between the two points at which the energy passing through the exit slit of a monochromator is one-half its maximum value, is a concept used to describe the output intensity distribution of a monochromator.[1] When applied as a measure of the performance of the dispersive system of a monochromator, it has validity.[2] It is also one of the factors influencing the shape and intensity of spectral bands passed by the monochromator. In the ideal case where the entrance and exit mechanical slit widths are equal, a triangular intensity distribution is obtained neglecting diffraction. Spectral slit width, as defined above, adequately describes this function. However, when narrow mechanical slit widths are used, imperfections in the slit jaws, inequality in the widths of the entrance and exit slits, optical aberrations, diffraction effects, image curvature, and misalignment of the optical system can greatly affect the shape of the output intensity distribution from the exit slit.

It may be desirable to transfer the Beer's law extinction coefficients determined with one spectrometer to another. The accuracy with which this transfer can be accomplished depends to a great extent on how well the output intensity functions of the two instruments are matched.

This paper proposes a method of obtaining an indication of the output intensity distribution function and a precise measurement of spectral slit width. Using this method, the mechanical slit widths with which it is possible to transfer spectral absorption data between two or more instruments with a given accuracy might be determined for any frequency or wavelength.

THEORETICAL BASIS

In determining the band-pass characteristics of an electrical network, it is general practice to apply a sinusoidal signal generator to the input of that network and observe the transmission of the signal as the frequency of the sine wave is varied. The plot of transmission *versus* frequency is the Fourier transform of the response of the network to a unit impulse. A monochromator can be considered analogous in many respects to an electrical network which has ideally a triangular band-pass characteristic. The Fourier transform of a triangular function is expressed by

$$V_0=\frac{\sin^2 x}{x^2}, \tag{1}$$

where x is related to the band width of the system.

Interferometers of the Fabry-Pérot type placed in the sample space of a spectrometer, produce interference patterns which for low reflectivities vary sinusoidally with frequency. The interference fringes produced in this manner are equally spaced in frequency for a given separation of the interferometer, d, and can be applied to the calibration of spectrometer wavelength scales.[3]

The frequency interval, ν', between succeeding interference fringes is given by the formula

$$\nu'=\frac{1}{2\mu d}, \tag{2}$$

where μ = index of refraction of the medium between the surfaces. By varying the separation, d, any fringe spacing, ν', can be achieved.

If the interferometer is applied to a monochromator, much as a signal generator is applied to an electrical network, then an expression can be written similar to (1) for the amplitude of the interference pattern, A_0,

$$A_0=\frac{\sin^2\alpha}{\alpha^2}, \tag{3}$$

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* Presented in part at the Pittsburgh Conference of Analytical Chemistry and Applied Spectroscopy, March, 1952.

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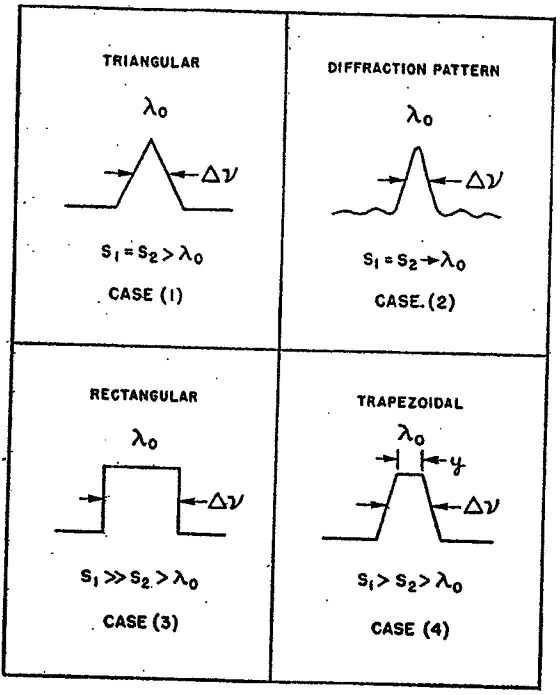

FIG. 1. The four cases shown represent in idealized form the general exit-slit intensity distribution functions which may exist. S_1 and S_2 are the mechanical widths of the entrance and exit slits. λ_0 is the center wavelength and $\Delta\nu$ is the spectral slit width

where α is a function of the fringe spacing, ν', and the spectral slit width, $\Delta\nu$, of the monochromator, and may be expressed as follows:

$$\alpha = \frac{\pi\Delta\nu}{\nu'}. \tag{4}$$

Equation (3) is written to express the Fourier transform of the triangular slit intensity distribution, which occurs at wide slits when the entrance and exit slits are equal, and the optical system is in good adjustment (Case 1, Fig. 1). When the slits are made narrow and equal such that their width approaches the wavelength of the radiation passing through, then a diffraction pattern is obtained (Case 2). The intensity of a diffraction pattern can be expressed

$$I_0 = \frac{\sin^2 y'}{y'^2} \tag{5}$$

which is similar to Eq. (3). Furthermore it can be shown that the Fourier transform of Eq. (5) is a triangular function. It is interesting to note that the distributions for the two cases are Fourier transforms of each other.

When one slit is very wide with respect to the other, and both slit widths are large with respect to the wavelength passing through, a rectangular distribution similar to that shown in Fig. 1, Case (3) is obtained.

The Fourier transform of the rectangular function can be expressed

$$A_0 = \frac{\sin\alpha}{\alpha}. \tag{6}$$

Case (3) is almost never observed in practice in infrared spectroscopy. However, the condition indicated in Fig. 1, Case (4) can often exist. Here one slit is slightly larger than the other, and a trapezoidal distribution is obtained. The Fourier transform of this function is expressed

$$A_0 = \frac{\sin\alpha}{\alpha}\left[\frac{\sin(\alpha - y)}{\alpha - y}\right], \tag{7}$$

where y is a function of the constant difference between the slits. When $y = \alpha$, a rectangular function obtains. When $y = 0$, a triangular function obtains.

The four cases are idealized. In practice diffraction always exists at the edges of the slits and imperfections in the optical system introduce other effects which change the shape of the slit intensity function.

Figure 2 shows a plot of the transforms expressed in Eqs. (3), (5), and (6).

THE METHOD

According to formula (2), by continuously varying the distance, d, between the windows of an interferometer placed in a spectrometer adjusted to a given wavelength and mechanical slit widths, the fringe spacing, ν', can be varied until an attenuation in fringe amplitude which approximates the shape of one of the functions plotted in Fig. 2 is observed. At some point, the fringe amplitude should equal zero. The fringe spacing computed at this point ($\alpha = \pi$) would theoretically equal the spectral slit width of the spectrometer, regardless of the intensity distribution function. A plot of fringe amplitude *vs* α should reveal the intensity-distribution function which exists.

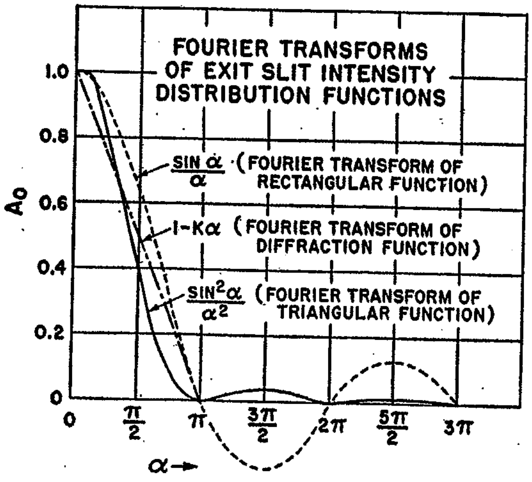

FIG. 2. The three curves plotted are the Fourier transforms of the exit-slit intensity distribution functions shown in Fig. 1, Cases (1), (2), and (3). Note that when $A_0 = 0$, $\alpha = \pi$ for the three cases. α is a function of spectral slit width.

This method of measurement is limited by the ability to change accurately and continuously the interferometer spacing over the required interval.

An alternate method offers more promise for the direct measurement of spectral slit width. An interferometer is constructed with a separation, d, calculated to provide a given fringe spacing, ν'. The mechanical slit width is set to a given value and the interference pattern recorded over the frequency range of interest. As the spectral slit width varies in accordance with the dispersive properties of the prism,[2] the interference fringe amplitude changes. At the frequency where the spectral slit width is equal to the fringe spacing ($\alpha=\pi$), the amplitude is observed to equal zero.

The data shown in Fig. 3 was obtained with a silver chloride plate 0.25 mm thick. The fringe spacing, approximately 9 cm^{-1}, varied slightly as the refractive index of silver chloride changed, but was assumed constant over the frequency interval used. (More constant spacing is obtained using two crystalline windows with an air space between.)

Curve A indicates the interference pattern obtained over the range 1500 to 700 cm^{-1} on a Perkin-Elmer Model 21 infrared spectrometer, recording linearly in frequency. The slits were adjusted to give approximately maximum resolution. It was observed that the fringe amplitude near 800 cm^{-1} approached approximately 27 percent. This is a region where the spectral slit width of the spectrometer is of the order of 2 cm^{-1}. This spectral slit width is considerably smaller than the fringe spacing of 9 cm^{-1}.

Curve B indicates the interference pattern obtained using a constant slit width of 0.400 mm. Note that the amplitude of the fringe pattern approaches zero at approximately 1110 cm^{-1} ($\alpha=\pi$). Note that a secondary fringe pattern also appears which reaches a maximum at 1220 cm^{-1} ($\alpha=3\pi/2$). This is in accordance with the $\sin^2\alpha/\alpha^2$ curve in Fig. 2. It can be deduced from these data and formula (4) that spectral slit widths of 9 cm^{-1} at 1110 cm^{-1} and 13.5 at 1220 cm^{-1} are obtained when the mechanical slit width is set to 0.400 mm, and that a triangular distribution function exists.

The pattern in Fig. 3 was obtained using rather large mechanical slit widths. It would be expected with very narrow slits that the exit-slit function would approach a diffraction pattern and the amplitude of the fringe pattern would tend to follow a linear function as shown in Fig. 2. The spectral slit width could be calculated by observing the frequency at which the fringe amplitude approached zero for a given mechanical slit width. For any exit-slit intensity function, it can be shown that the fringe amplitude will reach zero at the $\alpha=\pi$ point. This fortunate situation relieves this method of ambiguity in the measurement of spectral slit width.

FRINGE AMPLITUDE

The accuracy of this method of measuring spectral slit width depends to some extent on the fundamental amplitude of the interference pattern. As the amplitude of the interference pattern is increased, the accuracy with which spectral slit widths may be measured is improved. The reflectivity of the interferometer windows determines the theoretical interference fringe amplitude which can be obtained. The reflectivity, R, near normal incidence for transparent crystalline materials can be determined from the following expression, which neglects absorption:

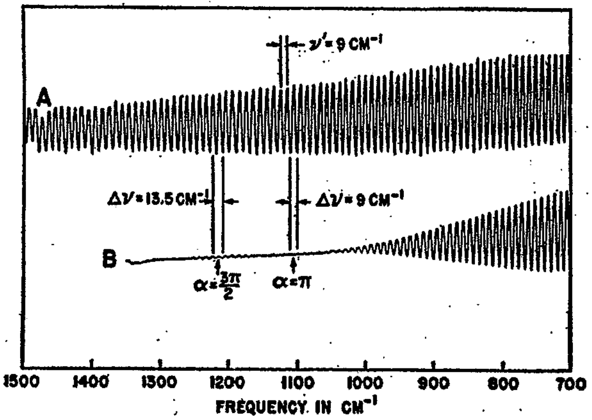

FIG. 3. *Curve A* shows the interference pattern obtained on a double beam infrared spectrometer recording linearly in frequency and transmittance under maximum resolution conditions (slit width 0.090 mm). *Curve B* was obtained on the same instrument with the same interferometer with a wide mechanical slit width of 0.400 mm. Note the secondary fringe pattern which appears near 1200 cm^{-1}. The fringe spacing, ν', was about 9 cm^{-1}.

$$R=\left(\frac{\mu-1}{\mu+1}\right)^2. \tag{8}$$

The reflectivity, R, can be applied to the following expression to determine the theoretical peak-to-peak amplitude, A_T, of the interference fringes in terms of percent transmittance where collimated radiation passes through the interferometer near normal incidence:

$$A_T=1-\left(\frac{1-R}{1+R}\right)^2\times 100. \tag{9}$$

It is assumed in the above discussion that the interference pattern is a true sine wave. For values of A_T below 30 percent this assumption is essentially correct. However, as the amplitude increases the maxima sharpen and the minima broaden. Interference patterns involving multiple reflections arise and these are superimposed on the fundamental fringe pattern.

Each of these can be treated as a separate set of fringes whose path lengths are multiples of the path length, d, in which the fundamental interference takes place. They are sinusoidal and can be treated as harmonics of the fundamental pattern. The fringe separation, ν_n', for any harmonic, n, can be computed by modifying expression (2) as follows:

$$\nu_n'=\frac{1}{2\mu n d} \tag{10}$$

or

$$\nu_n'=\frac{\nu'}{n}, \tag{11}$$

where n is an integer greater than 1. Thus, the fringe spacing of the harmonics, ν_n', must be less than ν'. Their contribution near the point $\alpha=\pi$ (Fig. 2) is small and can be considered negligible. However, they can represent a major portion of the amplitude, A_T, observed at $\alpha=0$. When determining spectral slit width at the minimum, $\alpha=\pi$, only the fundamental fringe pattern, A_F, is present. The higher harmonics have been, in effect, "filtered."

Equation (9) can be written more generally in the form[4]

$$T=\frac{(1-R)^2}{1+R^2}\times\frac{1}{1-\dfrac{2R}{1+R^2}\cos\beta}, \tag{12}$$

where T is the transmittance, and β is the phase difference between successive rays. When $\beta=0$, T is maximum, and when $\beta=\pi$, T is minimum.

For convenience, let

$$B=\frac{(1-R)^2}{1+R^2}, \tag{13}$$

and

$$C=\frac{2R}{1+R^2}. \tag{14}$$

Equation (12) may now be written

$$T=B(1-C\cos\beta)^{-1}. \tag{15}$$

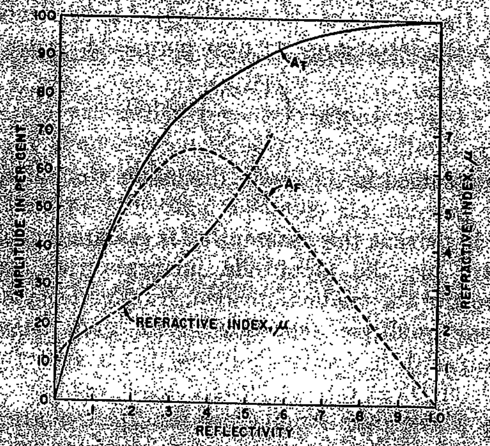

FIG. 4. A_T, the total fringe amplitude, and A_F, the fundamental fringe amplitude resulting from interference occurring after a single reflection, are functions of the reflectivity of the windows of the interferometer. The reflectivity in turn is a function of refractive index for transparent crystalline materials.

A binomial expansion of (15) provides an infinite series of the form

$$T=B(1+C\cos\beta+C^2\cos^2\beta+C^3\cos^3\beta+\cdots C^n\cos^n\beta) \tag{16}$$

or,

$$T=B\left[1+C\cos\beta+\frac{C^2}{2}(\cos2\beta+1)+\frac{C^3}{4}(\cos^3\beta+3\cos\beta)+\frac{C^4}{8}(\cos4\beta+4\cos2\beta+3)+\frac{C^5}{16}(\cos5\beta+5\cos3\beta+10\cos\beta)+\frac{C^6}{32}(\cos6\beta+6\cos4\beta+15\cos2\beta+10)+\cdots\right]. \tag{17}$$

The coefficients associated with the fundamental can be extracted from (17), and an expression written which allows the calculation of the peak-to-peak amplitude of the fundamental fringe pattern, A_F, for any reflectivity,

$$A_F=2B\left[C+\frac{3C^3}{4}+\frac{5C^5}{8}+\frac{7C^7}{16}+\cdots\frac{nC^n}{\dfrac{n+1}{2}}\right]. \tag{18}$$

B and C are both functions of the reflectivity, R, and can be determined using Eqs. (13) and (15).

Figure 4 shows plots of index refraction, total amplitude, A_T, and fundamental amplitude, A_F, *versus* R. Table I provides a comparison of some suitable crystalline materials available for the construction of interferometers.

Figures 5 and 6 compare the suggested method of measuring spectral slit width with that of observing the rotational structure of gases. Figure 7 is a plot of spectral slit width *vs* frequency and compares the calculated resolution data based on the prism material, slit width, and the optical arrangement of the Model 21 monochromator with the spectral slit widths measured in Figs. 5 and 6.

Interferometers constructed using windows coated with thin films of highly reflecting materials do not provide the constant reflectivity and fringe spacing

TABLE I.

		Refractive index (av)	Reflectivity	Total amplitude	Amplitude of fundamental
Crystal		μ	R	A_T	A_F
CaF$_2$		1.40	0.028	11%	11.0
NaCl		1.50	0.04	15%	15.0
AgCl		2.0	0.112	36%	35.5
KRS-5		2.35	0.164	48.5	46.5
Germanium		4	0.36	77.8	66.0
Tellurium	Parallel	5	0.44	84.8	63.0
	Perpendicular	7	0.56	91.1	52.0

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[4] F. A. Jenkins and H. E. White, *Fundamentals of Physical Optics* (McGraw-Hill Book Company, Inc., New York, 1937), Chap. 4, p. 83.

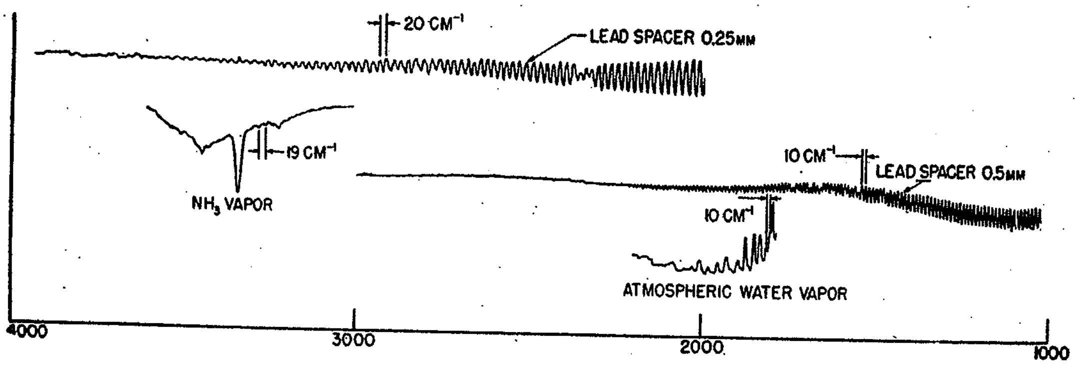

FIG. 5. The spectra compare the suggested method of measuring spectral slit width to the normal one of observing the separation of the rotational bands of gases in the region above 2000 cm^{-1}.

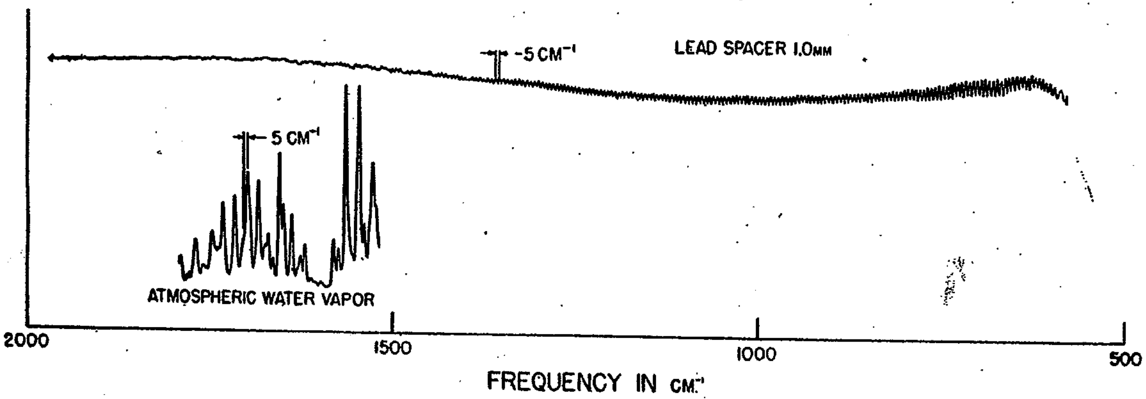

FIG. 6. Fringes produced by interferometer with rock salt windows coated with germanium. It is similar to Fig. 5 for the region near 1700 cm^{-1}.

obtainable from thick crystalline windows. Also, phase shifts occurring on reflection from the film will vary with frequency and alter the effective spacing of the interferometer, introducing considerable complication in the measurement. (Figure 8 compares the specular reflectance of the germanium-coated rock salt windows with a KRS-5 crystal window. Note that the KRS-5 provides nearly constant reflectivity over the spectral range of interest. However, its reflectivity at high frequencies is considerably less than that of the germanium-coated windows.)

The values of A_T and A_F given in Table I and Fig. 3 assume an interferometer with perfect windows, parallel and flat. Interferometers can be constructed

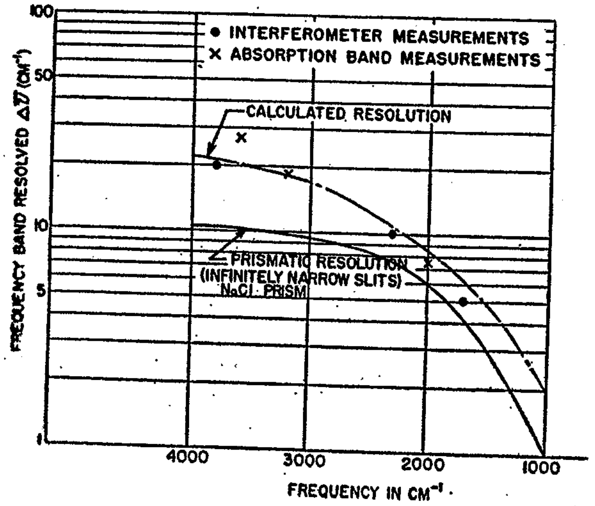

FIG. 7. Fringes produced by interferometer with rock salt windows coated with germanium. The figure is a plot of calculated spectral slit widths for the Perkin-Elmer Model 21 using a rock-salt prism. The spectral slit widths measured in Figs. 5 and 6 using the closely spaced absorption bands method and the interferometric method are plotted as crosses and dots.

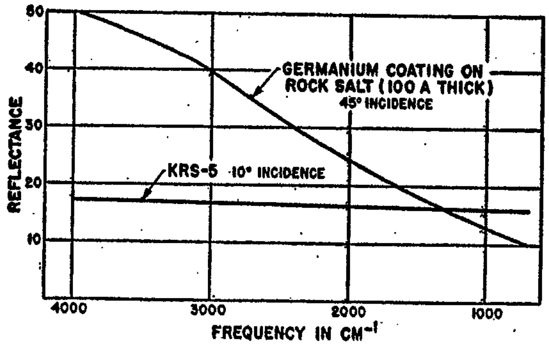

FIG. 8. The spectra shown above were obtained experimentally and represent reflectance from a single surface. Note that the KRS-5 crystal provided nearly constant reflectance over the region measured. This agrees with reflectivity data calculated using published refractive indices for this material.

using CaF_2 or NaCl windows but they do not provide large enough fringe amplitudes to allow a critical measurement of spectral slit width. AgCl and KRS-5 windows properly adjusted are satisfactory, but are so ductile that they are difficult to maintain constant. Pure germanium, if available, offers the possibility of providing large fringe amplitudes. It is opaque in the visible and must be adjusted by trial and error.

There is a further problem associated with placing the interferometer in a converging beam of radiation.[5,6] The path, d', for various rays will differ according to the formula

$$d'=\left(1+\frac{\theta^2}{4}\right)d,$$

where θ=the angle of convergence of the ray. It is important therefore to measure the fringe spacing, ν', near the frequency where the spectral slit width is being measured. At high frequencies this effect, of itself, will damp the interference pattern irrespective of the spectral slit width, and must be taken into consideration. The angle of convergence, θ, can be reduced by masking the collimator of the spectrometer. However this affects the diffraction pattern and the signal-to-noise ratio of the spectrometer.

ACKNOWLEDGMENT

We would like to acknowledge the assistance and encouragement of Mr. Larkin Scott, Dr. Harry Polster, and Dr. Van Zandt Williams of The Perkin-Elmer Corporation.

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[5] J. H. Jaffe, J. Opt. Soc. Am. 43, 1170 (1953).
[6] H. D. Polster, J. Opt. Soc. Am. 44, 503 (1954).

JOURNAL OF THE OPTICAL SOCIETY OF AMERICA VOLUME 45, NUMBER 6 JUNE, 1955

Ratio Measuring Twin-Beam Photometer*

MARCEL J. E. GOLAY
Signal Corps Engineering Laboratory, Fort Monmouth, New Jersey

(Received July 19, 1955)

A ratio measuring spectrophotometric system is described in which the percentage transmission or reflection of a sample is obtained as a directly recordable dc output.

INTRODUCTION

SINCE the measurement of the optical transmission of a substance implies the experimental procedure of measuring the two electrical signals obtained from two beams of radiation and forming their ratios, the reduction of this procedure to that of measuring the electrical phase angle between a detector signal and a reference signal offers the promise of doing electronically certain tasks otherwise performed by optical and electromechanical links such as beam weakening combs and servos.

A phase discrimination method of radiation ratio measurement has been developed and described by Halford and Savitzky,[1] in which the radiation beams through a standard cell and a sample cell are interrupted at a 90° phase interval before being recombined on the entrance slit of a monochromator. The resultant detected signal is decoded by means of break-make contacts into two dc outputs which represent the standard and the sample beam. These are applied, respectively, to the potentiometer and the nulling servo (regular input) of a recording instrument which registers their ratios.

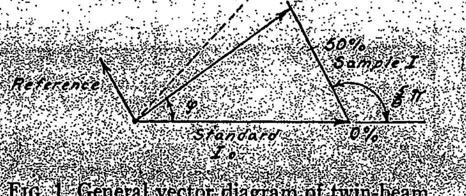

FIG. 1. General vector diagram of twin-beam phase recording system.

The phase shifting method described here consists in interrupting the beams of radiation through the standard and sample cell at a $112\frac{1}{2}°$ ($\frac{5}{8}\pi$) phase interval, recombining them in the proper proportion on the entrance slit of the monochromator, and obtaining a dc output which is proportional to the phase angle between the actual signal and the zero transmission signal, as sketched in Fig. 1. This signal is taken as the measure of the sample to standard radiation ratio and is recorded directly. It is nearly proportional to this ratio, the departure from strict proportionality having the extreme values -1.0 percent and $+1.0$ percent at the 22 percent and 78 percent transmission values, respectively, when the 0 percent, 50 percent, and 100 percent values are adjusted correctly. The system based on this method is believed to offer the advantages of simplicity, directness, and freedom from moving parts others than the radiation chopper and, of course, the

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* The experimental work discussed in this article was carried out at the Eppley Laboratory, Inc., Newport, Rhode Island.
[1] A. Savitzky and R. S. Halford, Rev. Sci. Instr. 21, 203 (1950).

Appendix: XIX

Society for Applied SPECTROSCOPY

PROGRAM

NINTH ANNUAL MEETING

May 27-28, 1954

HOTEL NEW YORKER
NEW YORK, N. Y.

Society For Applied Spectroscopy

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Utica Drop Forge & Tool Corp.
Westinghouse Electric Corp.

Appendix: XIX

ABSTRACTS

tion ago. However, the instrument has been commercially available for only a few years.

Research and commercial control have found the method useful and its acceptance has been rapid. Several hundred units are in use at the present time and there is a rapidly-growing wealth of information available touching on such factors as fields of applicability, techniques, analyses of data, etc. These points are summarized.

The future progress of the method can be predicted in the light of the past progress, the inherent features, and the known lines of instrumentation changes in design and contemplation. There are certain limitations imposed by the basic physics involved, and the practical limits that these impose are becoming evident.

(9) ANALYSIS OF NON-FERROUS ALLOYS BY THE X-RAY SPECTROGRAPH

Garton L. Crumrine, North American Smelting Co.

A brief review of the analysis of aluminum base, copper base, and lead-tin base alloys. Presenting the methods now in use in production control. Also presenting comparative results with wet chemical methods.

(10) DETERMINATION OF METALLIC ELEMENTS IN NONMETALLIC MATRICES

by: A. A. Shurkus, Applied Research Laboratories

The determination of metallic elements in nonmetallic matrices has been studied, using both emission spectroscopy and x-ray fluorescence. This paper will compare the results obtained using these two supplementary methods.

(11) A NEW SPECTROPHOTOMETER FOR USE WITH VOLUMES OF 0.01-5 MILLILITERS

Frederick Brech and David Glick, Jarrell-Ash Co., Newtonville, Mass. and Department of Physiological Chemistry, University of Minnesota, Minneapolis, Minnesota

A double-beam instrument will be described for use in the ultra-violet and visible regions between approximately 200 and 700 mu. A grating monochromator with a reciprocal dispersion of 4.4 mu/mm provides two beams, each focused on a separate exit aperture. One beam passes through a cuvette containing the sample and the other passes through a reference cell. A rotating alternator permits each beam in turn to fall on the same area of a single photomultiplier, the output of which is coupled to a logarithmic amplifier. This provides a signal of the form $\log\,\frac{A}{(B)}$ which is displayed on a meter graduated linearly with optical density, and also carrying transmission scales. A feature of the instrument is that it employs special cuvettes with a 1 cm. light path having volumes of 0.010 ml. Micro cuvettes of this type with light paths up to 5 cm. can be accommodated. Macro cuvettes with light paths from 1 to 5 cm. having volumes of several ml. can also be used. Examples of the use of the instrument in biochemical and histochemical research will be detailed.

(12) SOME APPLICATIONS OF THE MICRO-VOLUME TECHNIQUE TO EMISSION SPECTROCHEMISTRY AND METALLURGY

by J. K. Hurwitz

Mines Branch, Ottawa, Ontario, Canada

There has been a growing metallurgical interest in segregation of alloying constituents and trace elements in casting and in the study of solid state diffusion phenomena. To obtain the necessary analytical data, a rapid quantitative spectrographic technique has been developed. In principle, the specimen is moved over a spark discharge while the photographic plate is lowered so that each point on the spectrum corresponds to the anlysis of a point on the specimen. A theory has been developed which takes into consideration the effects of the measurable instrumental variables and predicts quantitatively the observed intensity changes in the spectrum when a region of rapidly changing composition is traversed. The practical significance of this theory is indicated. Illustrative examples of the analysis of segregates are given, and an outline of the method to be applied to specimens in which diffusion has occurred is described. The results of a prespark-effect investigation in the spectrochemistry of zinc base alloys indicate a procedure by which this effect may be eliminated. Future developments in the micro-volume technique are discussed.

(13) SOME NOTES ON THE ILLUMINATION OF THE SPECTROGRAPH

R. H. Bell, Lucius Pitkin, Inc.

The selection of a suitable lens system for the illumination of the spectrograph will be discussed, particularly in relation to the proper "sampling" of the light source and the ways in which the practical resolving power of the spectrograph is affected. Results of tests on the use of ground diffusion screens, and gratings, both transmission and reflection, will be presented. It is believed that the proper illumination of the spectrograph should be given much more consideration than it has been given previously.

(14) WEIGHING PIPET METHOD FOR PREPARING INFRARED GAS STANDARDS

By: Frank Pristera, Chief, Spectrographic Laboratory, Picatinny Arsenal

(no abstract)

(15) AN EVACUABLE DIE FOR THE PRESSED POTASSIUM BROMIDE TECHNIQUE

Harry Hausdorf, The Perkin-Elmer Corporation, Norwalk, Connecticut

In February, 1953 a general paper on this invaluable technique was presented at the Society for Applied Spectroscopy. Since, the author had the opportunity to compare various types of dies for pressing potassium bromide and sample into thin transparent disks. The experience gained during this evaluation period led to the choice of a design of a special evacuable die which provides optimum results.

This die will be shown and described.

(16). THE PERFORMANCE OF THE PERKIN-ELMER MODEL 21 IN THE SPECTRAL REGION 210 mu to 2300 mu

V. J. Coates, T. Miller, A. Savitzky

Perkin-Elmer Corporation

The Perkin-Elmer Model 21 Double Beam Recording Infrared Spectrophotometer employs a completely achromatic optical system. The controls provided on this instrument allow the operator to vary its operating conditions over a wide range, optimizing its performance for a given problem. It might be expected that by the addition of the proper sources, prism and detectors satisfactory performance can be obtained in the ultraviolet, visible and near infrared portions of the spectrum, without affecting its infrared performance which presently extends to 37.5 microns.

On an experimental basis, a standard instrument has been converted for operation in this region while maintaining its function as an infrared spectrophotometer. The design of the various attachments and the problems involved in providing simple interchange are discussed. The performance obtained with this system is shown.

THE PERFORMANCE OF THE PERKIN-ELMER MODEL 21 IN THE REGION 210mu TO 2000mu

VINCENT COATES, THOMAS MILLER, ABRAHAM SAVITZKY

The Perkin-Elmer Corporation, Norwalk, Connecticut

Introduction

The Perkin-Elmer Model 21 Infrared Recording Spectrophotometer presently has the ability to provide accurate, high resolution absorption spectra in the infrared portion of the spectrum from 2 to 37μ. (1-3) This paper describes the conversion of this instrument for operation in the ultraviolet, visible and near infrared from 210 to 2000mμ. This is a logical extension of the performance of this instrument. Its original design principles make it well suited in many respects for operation as a universal transmittance recording absorption spectrophotometer. It employs an all-reflecting f4.5 optical system and its null balance principle is not limited to any one region of the spectrum. The mechanical and electronic systems are stable and capable of handling the signals produced by photocell as well as thermal detectors.

The prism interchange feature of the Model 21 allows quick and simple interchange of prisms for any region and their specific linear wavelength cams and slit programming potentiometers without tedious recalibration of the system.

Design Principles

In order to obtain optimum performance in the ultraviolet, visible and near infrared regions of the spectrum, it is necessary to separate these portions of the spectrum and to utilize the source and detector combinations which will provide the best performance in each region. It would be possible to scan this region with a single source and a single detector. However, such a procedure would yield relatively poor performance over most of the range.

The sources, prisms and detectors required to provide optimum performance in various parts of the spectrum are shown in Figure 1. The sources and detectors are placed to provide dispersed radiation in the sample space. This is important if one wishes to avoid photochemical effects in the sample which might occur if the full undispersed beam were to fall upon it. The monochromator is operated in a reversed direction from that used in the infrared region.

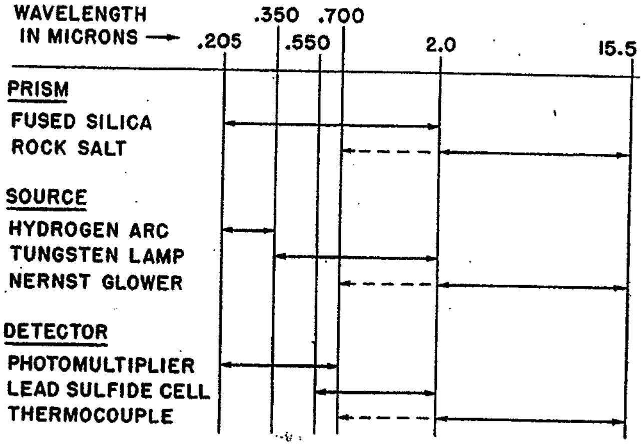

FIGURE 1—Prisms, sources, and detectors used to cover the region 0.205μ to 15.5μ with optimum performance.

Description

The optical system of the Universal Model 21 Spectrophotometer can be seen in Figure 2. The radiation from the hydrogen lamp H_2 falls on the off-axis ellipsoid M17. This mirror, in conjunction with lens L3, forms a five times magnified image of the source on the slit S2. Mirrors M16 and M14 are flat mirrors. The window, W, in the monochromator housing is made of calcium fluoride which is transparent to ultraviolet and visible radiation.

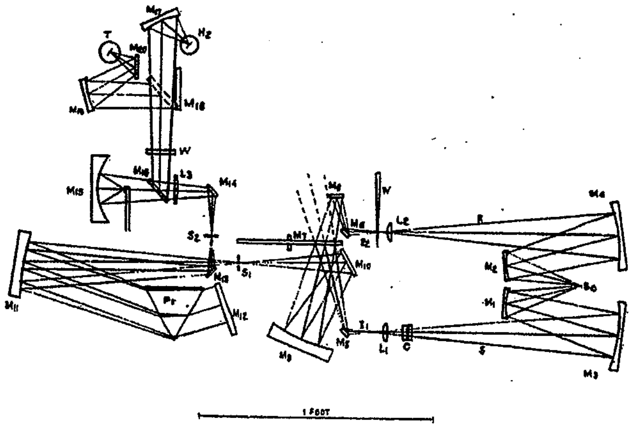

FIGURE 2—Optical system of the Universal Spectrophotometer.

The radiation from the Tungsten lamp falls on mirror M20 which directs the radiation to the spherical mirror M19 which, in conjunction with lens L3, forms a two times magnified image of the Tungsten filament on the slit S2. The mirror M18 is rotated into the beam and chooses either the Tungsten ribbon filament optical path or the hydrogen lamp optical path. Its position is fixed by a detent arrangement and is reproducible.

The mirror M16 and the lens L3 serve to take the energy from the Hydrogen or Tungsten lamps and direct it into the monochromator. These two elements are mounted as a unit and can be removed or readily assembled on the instrument. They utilize a kinematic mounting arrangement and are held in place by spring clips. When they are removed, the radiation from mirror M14 is directed to the thermocouple ellipsoid M15 which focuses the radiation from the exit slit S2 onto the thermocouple detector. The lens L3 is used to form an image of the pupil of the monochromator (the Littrow mirror) on the surface of mirrors M17 or M19.

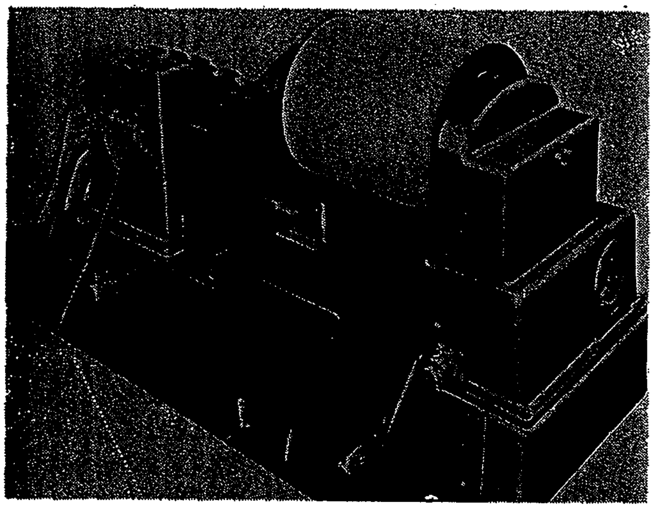

FIGURE 3—Rear view of instrument showing source assembly and sample space cover in place.

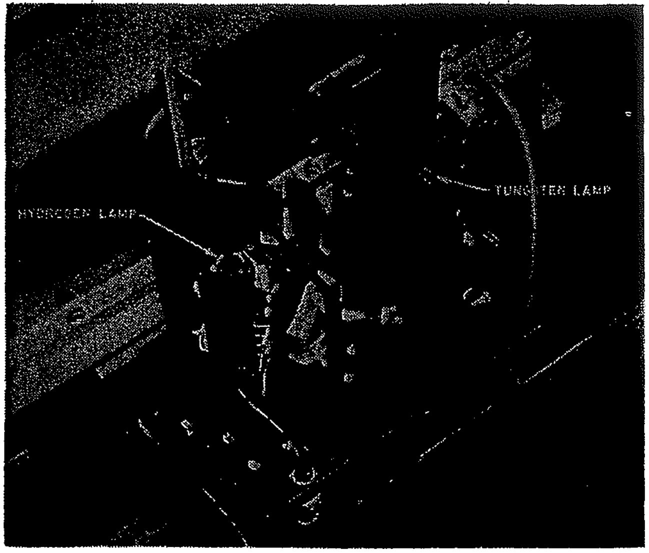

FIGURE 4—Hydrogen and Tungsten source optical assembly. Cover removed.

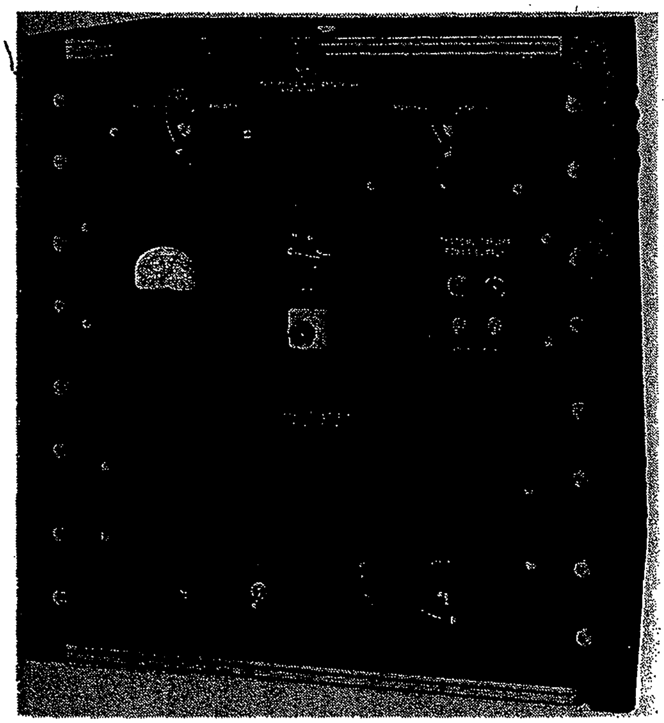

FIGURE 5—Cabinet containing source and detector power supplies.

Figure 3 shows the source system which is contained in a rigidly mounted box fastened on the rear of the instrument base. Figure 4 shows a view of this box with the cover removed. The mirror mounts are designed for rigidity and simple adjustment of their optical elements. Suitable baffles are provided to eliminate stray radiation. Vent holes are provided in the base and in the cover to allow dissipation of heat from the sources. Cables bring the power from the source power supplies which are cabinet mounted as shown in Figure 5.

The Hydrogen lamp is a standard Beckman 2230 and the power supply is the standard Beckman 2965. It has been found that this high intensity lamp has long life and stable operating characteristics. The Tungsten lamp is a standard General Electric type 9AT - 8½ with vertical ribbon filament. Its supply consists of a step-down transformer (115 VAC to 6.3 VAC) and a voltage dropping resistor of 25 ohms in series with the transformer primary. This resistor reduces the voltage applied to the lamp to 6.0 VAC.

The prism interchange assembly utilizes a 50° Corning fused silica prism. This material has been developed by the Corning Glass Works of Corning, New York and its properties are particularly good in the ultraviolet and visible regions of the spectrum. (See Figure 6.) It does not exhibit the strong absorption band at 240mμ which is observed in standard commercial fused quartz and it has good transmission to 2000mμ. However, it exhibits some strong absorption bands in the near infrared region of the spectrum. It has a moderate band at 1385mμ and a second strong band in the vicinity of 2200mμ. The double beam symmetry of the Model 21 was adequate to cancel these two bands.

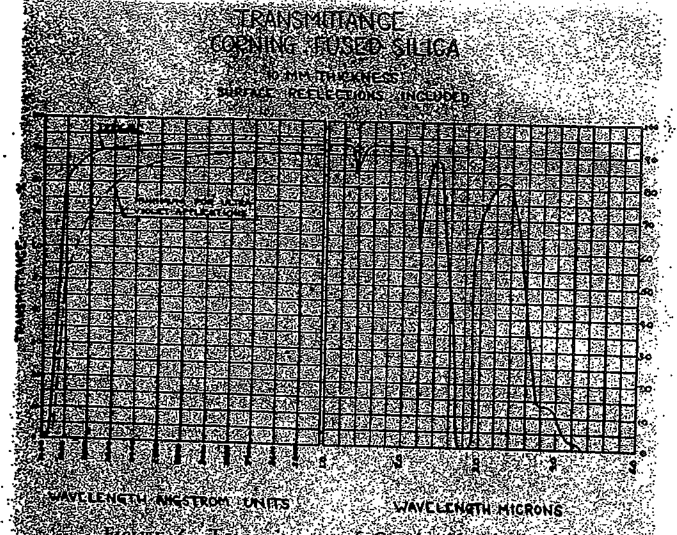

FIGURE 6—Transmittance of Corning fused silica.

In Figure 7 the linear wavelength cam is shown which is used with the fused silica prism. The first region of the cam extends from 205.0mμ to 800.0mμ. The second portion of the cam extends from 550mμ to 2300mμ at a 10 times faster rise than the first section. The gearing to the wavelength counters, shown in Figure 8, is such that the two counters read directly in millimicrons. The left hand counter, reading to four decimal places, is used for the first portion (UV-visible) of the cam and the right hand counter, reading to three decimal places, is used for the second portion (near infrared). A counter mask indicates the proper counter to be used in each region. There is an overlap between the two sections of the cam between 550 and 800mμ. This allows the operator to choose the best point in the spectrum at which he wants to change from photomultiplier to lead sulfide detector operation. The non-linear slit potentiometer used in this instrument is also divided into two sections, one for operation from 210.0 to 800mμ and one for use from 550 to 2300mμ. The slits are automatically adjusted by this potentiometer in a balance servo to provide a constant energy background.

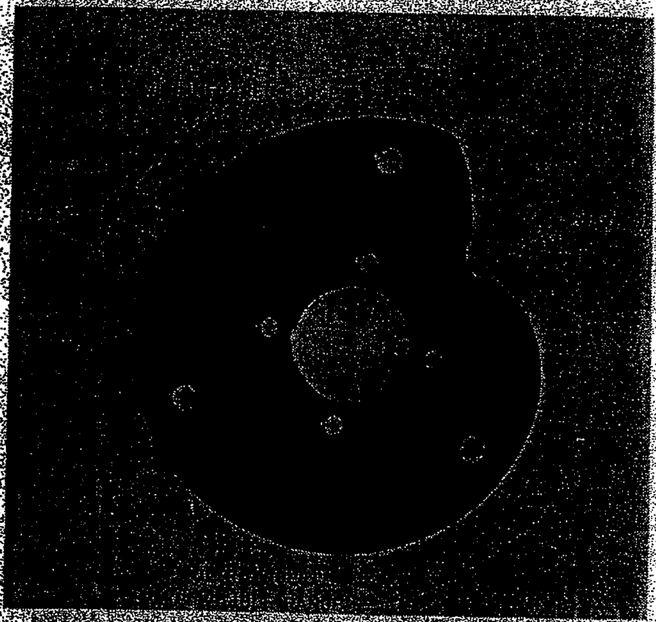

FIGURE 7—Littrow mirror cam designed to provide linear wavelength recording using Corning fused silica prism.

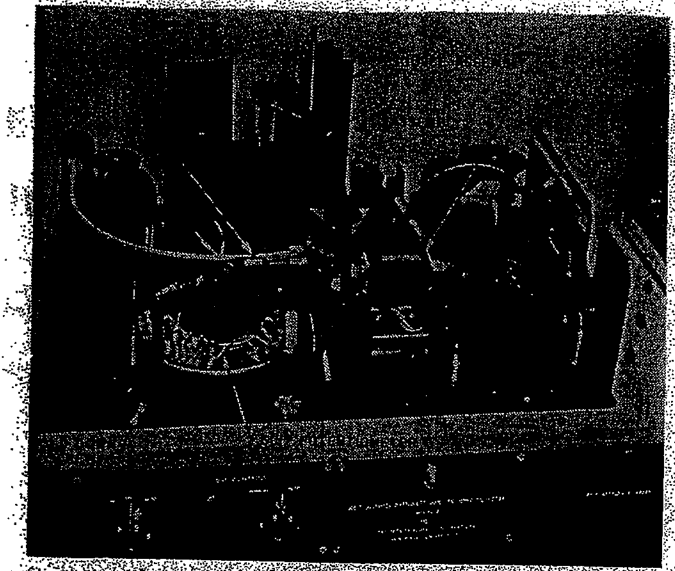

FIGURE 8—Monochromator with recorder cover removed showing fused silica prism and interchange assembly in place.

The dispersed radiation from the monochromator appears in the sample space of the instrument when operating in the ultraviolet and visible regions. Provided with the instrument is an enclosure for the sample space which excludes room light from the system. It is important to insure that the detector is suitably shielded from room light. A small amount of room light can effect the photometric accuracy of the instrument by adding light to one beam or the other, or by saturating the detector, causing it to operate in a noisy and non-linear manner. This precaution is, of course, unnecessary when the instrument is operated in the infrared region of the spectrum, and the sample space enclosure may be removed from the instrument. Suitable cell mounts have been provided so that normal sampling procedures can be carried out.

For infrared operation, the Nernst glower is at a focus of the entrance slit of the monochromator, and is focussed by means of mirrors M1, M2, M3, M4 and M9 onto the entrance slit. These mirrors in the opposite sense serve to form a two times reduced image of the entrance slit on the Nernst glower. The photomultiplier tube and the lead sulfide cell are placed at this image position after removal of the Nernst glower mount. In order to accomplish this, an access hole, shown in Figure 9, is machined in the floor of the source optics cover underneath the hinged control panel of the Model 21. In this space there also has been provided two electrical receptacles. The lower one accepts the signal from either the photomultiplier tube or the lead sulfide cell and the upper one provides power to the Nernst glower when it is being used. A kinematic mechanical system is placed on the base of the instrument to accept the Nernst and photo-detector assemblies which are shown in Figure 10, and to allow reproducible interchange.

The lead sulfide and photomultiplier tube detectors

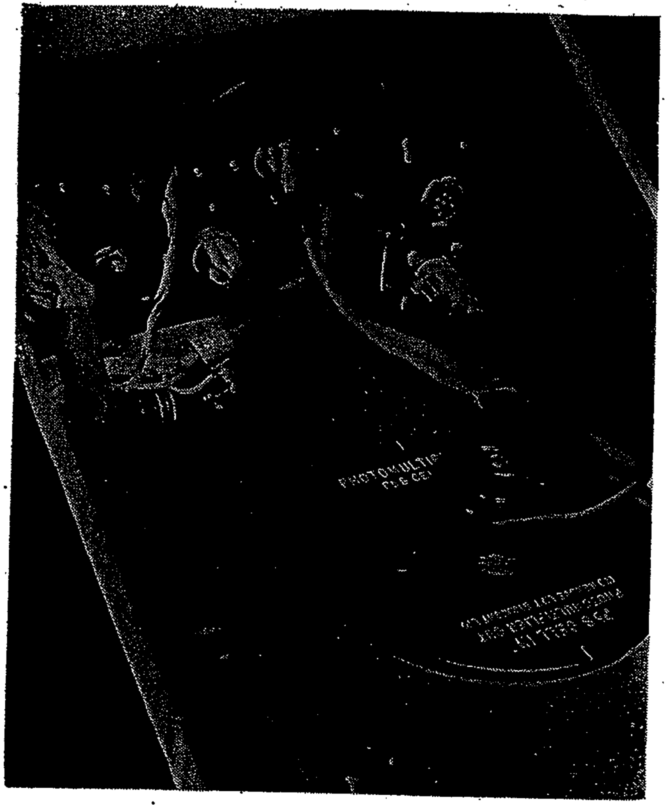

FIGURE 9—View of access hole under instrument control panel. Wing nut holds detector assembly in place. Lower receptacle is used for detector assembly. Upper receptacle for Nernst Glower Assembly.

have been mounted on a single assembly which fits through the access hole of the source optics housing floor. It is guided in place by a guide shaft and then bolted down. In one position, the lead sulfide cell is reproducibly located; 180° of rotation places the potomultiplier tube in the proper position. A cover plate closes off the access hole and prevents room light from entering the detection section.

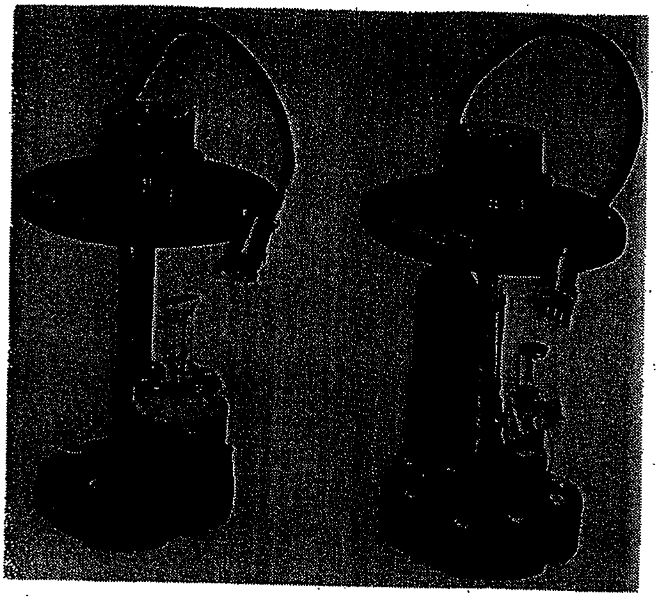

FIGURE 10—The Nernst assembly, left, and the detector assembly, right, are designed to be interchangeable. The detector assembly mounts a photo-multiplier tube and lead sulfide cell.

The 1P28 Photomultiplier Tube is a high sensitivity U.V.-visible detector. Its peak sensitivity falls in the region of 450mμ. Its envelope is transparent to below 205mμ. The lead sulfide cell is a photo-conductive detector which has a useful range of from 500mμ to 2700mμ. It has a target size of 1 x 6mm. The thermocouple, mounted in its normal position after the exit slit, is operated through the range from 1 to 37μ.

Performance

Figure 11 illustrates the survey performance obtained in the ultraviolet region of the spectrum. It was discovered that the noise level near maximum resolution was low enough in this region (0.25% peak-to-peak) to allow optimum performance at very fast scanning speeds. The

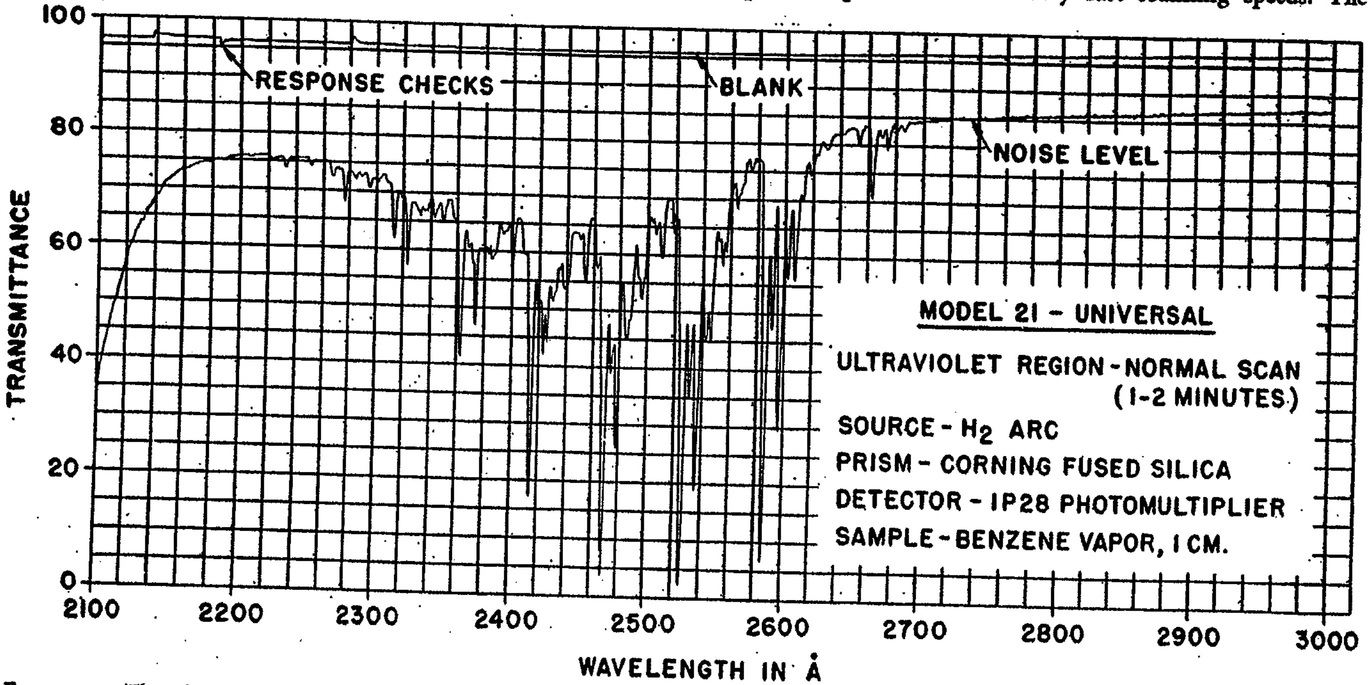

FIGURE 11—The above recording illustrates the survey performance in the ultra-violet region at high scanning speed.

scanning time for a normal survey run was limited only by the slewing speed of the servo motor driving the optical wedge. The fine structure in the benzene vapor spectrum is well resolved when the scanning time for the full region is 1 - 2 minutes. Maximum resolution was obtained, as shown in Figure 12, in a 10 minute scan using narrower slits. The resolution observed at 248mμ is 0.15mμ. For liquid samples, where the bands are considerably broader, an accurate spectrum can be obtained in less than 2 minutes. A change in slit curvature, which is presently optimized for the infrared, would bring a further improvement in resolution.

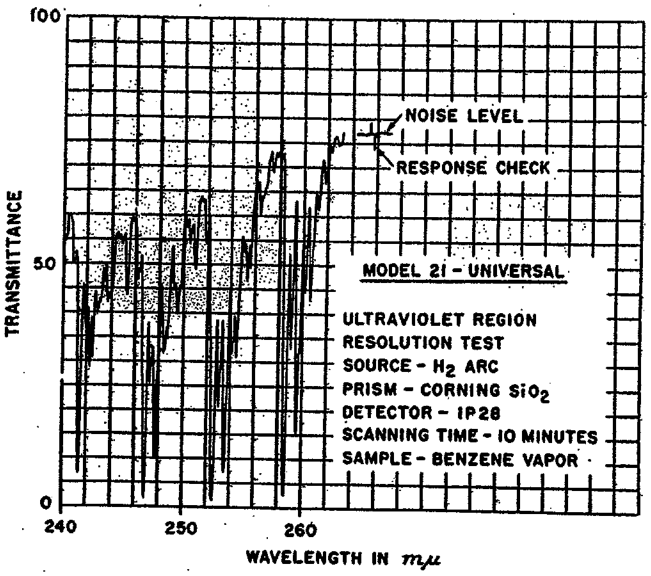

FIGURE 12—The resolution of benzene vapor structure shown above is near the maximum capabilities of the instrument. The band separation at 248.5mμ is about 0.15mμ.

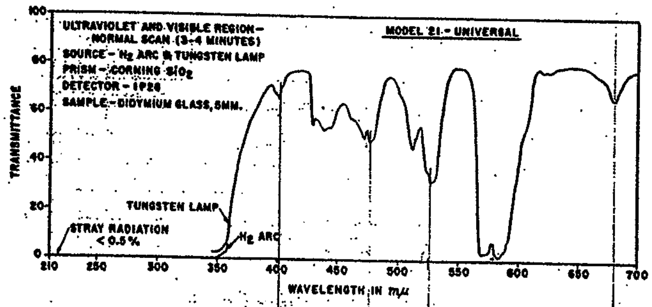

FIGURE 13—The Didymium glass spectrum shown above was recorded in the visible region. The zero line in the ultraviolet region shows the low scattered light.

The well-resolved spectrum of Didymium glass shown in Figure 13 indicates the performance obtained in the visible. The stray light, which was negligible in the ultraviolet, is a maximum of 1% through this region. Optical filters can be utilized to reduce this to a small value.

Figure 14 shows a Didymium glass spectrum recorded from 550 to 1000mμ, using two different detectors, the 1P28 photomultiplier and the PbS cell. The Tungsten lamp was used as the source for each run. It is in this spectral region that the sensitivity of both detectors has fallen to a low value; the 1P28 (peak sensitivity about 450mμ) is decreasing toward long wavelengths and the PbS cell (peak sensitivity about 2000mμ) is falling off toward short wavelengths. Thus it is here that a small amount of stray light of wavelengths near the peak sensitivity region of either detector will produce a relatively large signal, thus causing a zero offset. This zero error can be minimized by changing detectors at the wavelength at which the stray light observed with either detector crosses. The crossover point for this instrument occurs at 700mμ. Fortunately, the spectral sensitivities of the two detectors also cross in this region.

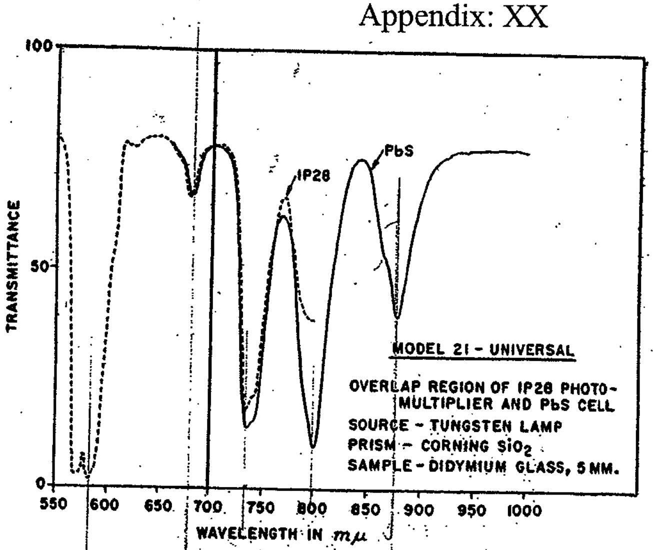

FIGURE 14—The above spectra illustrate the cross-over region of the photo-multiplier and lead sulfide detectors. Note the difference in readings obtained in the range above 700mμ. The lead sulfide cell provides accurate performance in this region.

The performance of the instrument in the near infrared region from 700mμ to 2100mμ is shown in Figure 15. The noise level is low (0.25% peak-to-peak and the resolution is high (approximately 2mμ at 1500mμ) for a 5 minute scan. The cancellation of atmospheric water vapor absorption near 1800mmμ and prism absorption at 1385mμ is complete.

The photometric linearity of the instrument in the ultraviolet region is shown in Figure 16. The errors in linearity observed at absorbances below 1.0 are small. Errors in the optical attenuator are apparent at higher absorbance values. Maximum accuracy is maintained for strongly absorbing materials by placing calibrated absorbers such as screens in the reference beam, thus utilizing the transmittance scale in its most linear region. In the same manner, solvent absorption is compensated by placing a cell filled with solvent in the reference beam. Thus, only the spectrum of the sample is recorded and measured. Such differential recording methods allow the measurement of small traces of sample in heavily absorbing solvents.

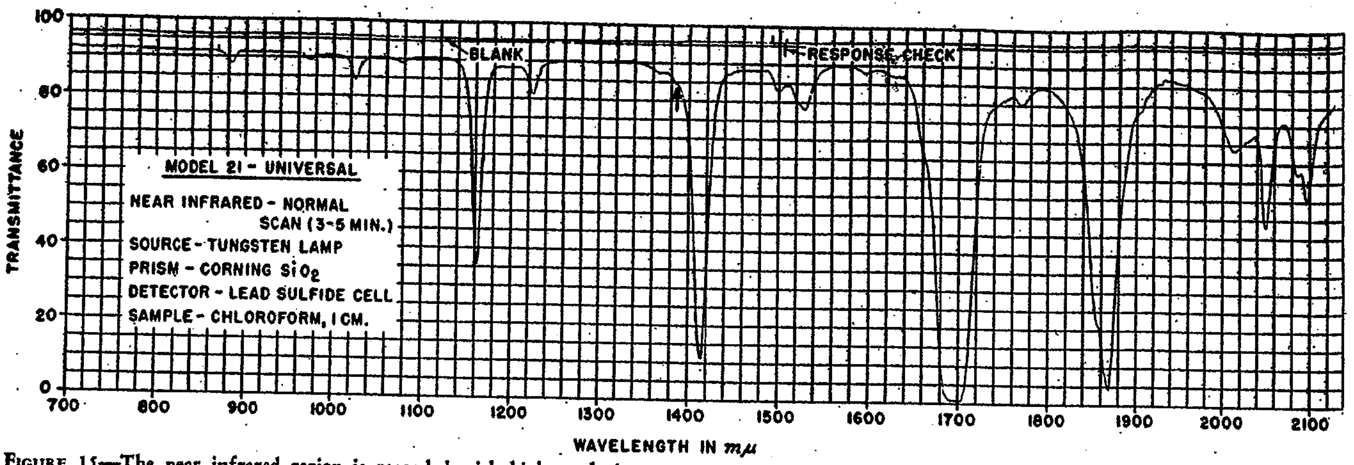

FIGURE 15—The near infrared region is recorded with high resolution and low noise level. The survey spectrum of chloroform shown above illustrates the performance in this region.

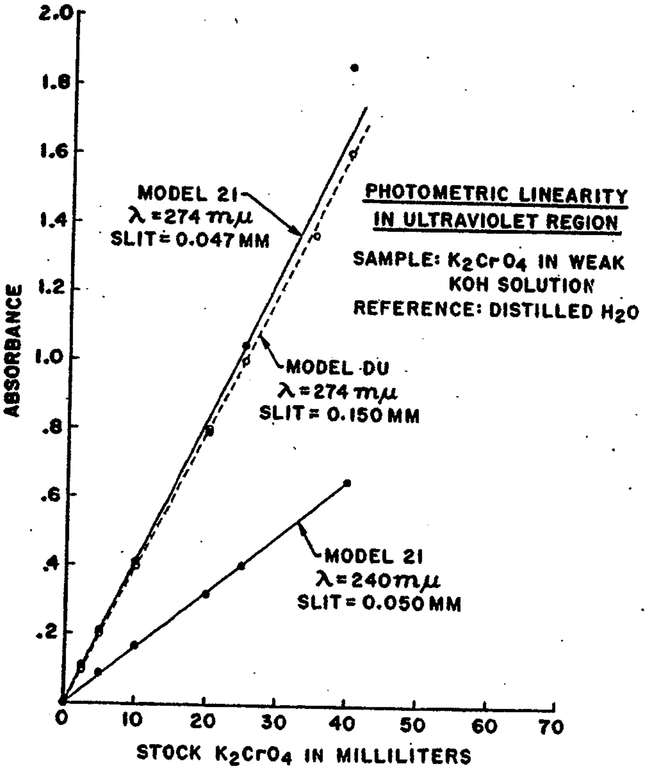

FIGURE 16—The photometric linearity of the instrument indicated above allows its application to accurate quantitative analyses.*

Conclusion

The addition of the proper sources, prism and detectors has extended the range of an infrared spectrophotometer, providing high performance operation in the ultraviolet, visible and near infrared regions. Simple and quick conversion from region to region is provided. The conversion from ultraviolet to visible or near infrared operation requires only a few seconds. The conversion back and forth from infrared operation requires approximately 30 minutes.

This instrument provides absorption data throughout the presently useful spectral range of liquid, solid or gaseous samples.

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(1) J. U. White and M. D. Liston, J. Opt. Soc. Am. *40*, 29 (1950)

(2) M. D. Liston and J. U. White, J. Opt. Soc. Am. *40*, 36 (1950)

(3) J. U. White and M. D. Liston, J. Opt. Soc. Am. *40*, 93 (1950)

*This graph was plotted from data kindly supplied by Dr. William Mason of the University of Rochester, Atomic Energy Project, Rochester, N. Y.

UNIVERSAL SPECTROGRAPHIC METHOD FOR THE ANALYSIS OF IRON AND STEEL

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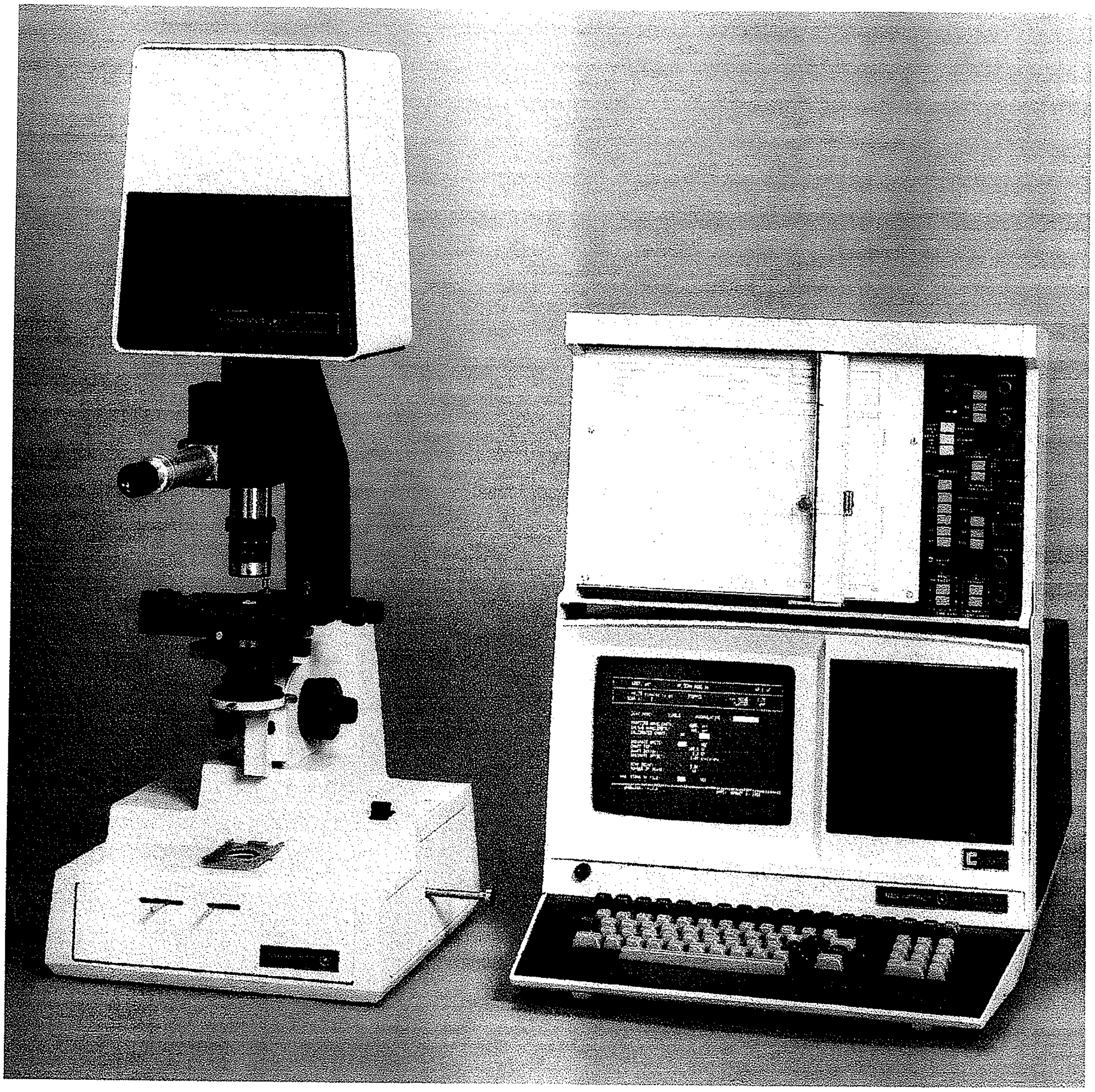
Summary

A universal spectrographic method has been developed for the determination of the following elements in iron and steel: Silicon, Manganese, Nickel, Chromium, Cobalt, Molybdenum, Titanium, Niobium (Columbium), Vanadium, Aluminum, Copper, Silver, Lead and Tin. The method has proven itself highly satisfactory in classifying or characterizing samples of iron or steel. It is universal in that it can be applied to any sample of 0.010 gram or



NANOSPEC 20IR INFRARED MICROSCOPE SPECTROPHOTOMETER
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