

The Birth of Infrared Filtometry

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literally thousands of filter-based infrared (IR) analyzers — IR filtometers — are used today throughout the world. Yet these ubiquitous instruments rarely receive mention in the original literature, which is concerned primarily with applications of their big brothers — Fourier transform-IR (FT-IR) spectrometers. IR filtometers perform countless measurements daily in the real world of quality control and process and environmental monitoring. This is the story of how IR filtometers came into being, how they are being applied today, and where the technology is going in the future.

The West Coast chapter of the Society for Applied Spectroscopy (Frederick, MD) held a meeting and exhibition in spring 1969 in Anaheim, California. Wilks Scientific's chief engineer, Tony Gilby, and I attended to present papers — Tony to talk about his IR goniometer and I to discuss gas chromatography-IR (GC-IR).

While touring the exhibition, we came across a brand-new instrument from Beckman (now Beckman Coulter, Fullerton, CA) designed to be a rapid-scan GC-IR instrument. It had in it a device we had never seen before, called a circular variable filter (CVF). It could be rotated in front of a set of slits to pass a continuously variable wavelength of IR energy. It covered the spectral region from 2.5 to 15 μm in three segments.

While discussing the Beckman instrument at dinner that evening, we came to the conclusion that the CVF was an extremely significant development for IR spectroscopy and that Beckman had completely missed the boat in how to put it to work effectively and efficiently.

Beckman engineers, as was their wont with their ultraviolet (UV) instruments, used long-focal-length, high- f -number (around $f10$) optics. This practice is common with UV equipment where source energy levels are extremely high and low-efficiency optics still produce acceptable signal-to-noise ratios. However, in IR, energy levels are so low that short-focal-length, low- f -number, aspheric optics are generally required to produce high signal-to-noise ratios.

Tony and I thought that with the CVF, we could build a very compact IR spectrometer that would eliminate expensive prisms and gratings, along with their complicated optics.

Furthermore, the CVF could be operated with short-focal-length $f1$ optics with more than 100 times the energy throughput possible with the Beckman instrument. We learned at the Anaheim meeting that the CVF was developed by Optical Coating Laboratory (OCLI, Santa Rosa, CA).

On my return home, I called OCLI to see if I could learn more about the availability of the CVF. The operator said, "Oh, the CVF is Mr. Illsley's special project. I'll see if I can locate him to talk to you. He is

our company president and not easy to locate."

Rolf Illsley came on the phone shortly, and it was immediately obvious that he was very interested in finding applications for the CVF. He indicated that developing the variable filter had turned out to be more expensive than originally thought, and that he was not happy with the Beckman relationship, but he felt that the CVF had lots of promise. He ended the conversation by inviting me to come to Santa Rosa to discuss the project in detail — an invitation I was happy to accept.

On arriving at the San Francisco, California, airport, I was paged with a message that I should proceed to the hanger for private planes. Here I found Rolf waiting for me. He flew from Santa Rosa in his own plane to pick me up.

My visit to Santa Rosa turned out to be very fruitful (in more ways than one). I learned that OCLI had developed an ingenious procedure for making optical elements that passed successively varying wavelengths from one end to the other. Interference filters are made up of many layers of coatings with different indices of refraction — the thickness of the individual layers determines the specific wavelength passed by the filter. The OCLI procedure involved rotating the optical element out from behind a mask as each layer was deposited so that the overall thickness of each layer varied uniformly from one end to the other; hence the variation in transmission along the filter.

Rolf and I worked out an agreement whereby Wilks Scientific would buy, on a time basis, all of the CVFs OCLI had made for Beckman (which had decided to drop their CVF instrument), including the units already in Beckman's hands. The price per CVF at that time was around \$300 — about twice the cost of a rock salt prism. I was also entertained at a delightful dinner at Rolf's home at which a different local wine (from the heart of the Napa Valley) was served with each course.

Wilks Scientific at this time was a manufacturer of IR sample-handling accessories. The CVF gave us the opportunity for us to move up to the manufacture of complete instruments.

We went right to work building the CVF into a versatile IR analyzer that we introduced in 1971 as the MIRAN — for "miniature infrared analyzer" — portable IR spectrometer. In the ensuing 25 years until the MIRAN line was finally discontinued, more than 5000 CVF instruments were produced. Interestingly enough, widespread use of the variable filter instruments brought about the introduction of a variety of single-wavelength, fixed-filter instruments that today are becoming the workhorses of IR analytical devices.

The characteristics of IR filtometers — their ruggedness, ease of operation, and low cost — led to their widespread use for routine quantitative measurements wher-

ever needed. Because most gases and vapors have characteristic IR absorption bands in the mid-IR, a variety of filter-based gas analyzers became available from General Analysis (a division of OI Analytical, College Station, TX), Thermo Environmental (Franklin, MA), and Mine Safety Appliances (Pittsburgh, PA), among others. The instruments monitor toxic vapors in the workspace and anesthetic gases in operating areas, as well as keep surveillance on heavy refrigeration equipment. Another important use of IR filter gas analyzers is emission monitoring of exhaust gases from vehicles at test stations and at factory stacks.

Because IR analysis is a non-destructive technique, it is increasingly used in process stream monitoring — both liquid and gas. For example, most of the large soft drink packaging plants are equipped with on-line IR filter analyzers that continuously monitor Brix (sugar) and CO_2 levels. With production rates as high as 2000 containers per minute, the instant warning they give regarding the occurrence of out-of-specification fluids is essential in the operation of the plants. In-line IR sensors making use of narrow band filters to isolate specific absorption bands are coming into use to follow concentrations of specific components in a variety of processes.

Probably one of the most interesting developments in the application of IR filtometers is their use as a supplement to FT-IR spectrometers, both inside and outside the analytical laboratory. Perhaps 30,000 FT-IR spectrometers are in use in analytical laboratories. Many of them are used to develop analytical procedures for tasks in the plant or in the field. Frequently, these procedures involve measuring IR absorption at one or two wavelengths — measurements that are ideally suited to IR filtometers. FT-IR instruments are necessarily complex, requiring trained operators and not suited for use outside the benign atmosphere of the laboratory. In contrast, IR filtometers can be used anywhere by relatively unskilled operators. Hence, they make ideal partners to their FT-IR brothers, carrying out the FT-IR developed procedures in the real world where analyzers are needed.

Rolf Illsley and OCLI, with their circular variable and narrow band pass filters, made it possible to take IR analytical devices out of the laboratory and into the real world, where they can perform their measurements when and where needed.

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