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The SPEX Speaker

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SPECTROMETRIC BITS AND PIECES

THROUGHPUT AND SCATTERED LIGHT IN SPEX 1400 MONOCHROMATOR

D. Berger of Temple University's Skin and Cancer Hospital cooperated with us in appraising the throughput and scattered light of the 1400 Double Spectrometer in connection with his planned experiments to correlate the spectral sensitivity of human skin with sunburn. Although work along these lines was done in the '30s, results are now subject to question because of limitations of the Hg source then available and spectral purity of then existing monochromators. Spectral purity is, of course, essential in such an experiment to eliminate the possibility that a trace of scattered light rather than the indicated bandpass is responsible partially or wholly for the sunburn.

Scaling up our preliminary measurements, based on a 150-watt xenon lamp, throughput has been calculated for the General Electric XE-5000, 5000-watt lamp. According to the manufacturer's literature, its output is 1.70 watt/ steradian/ 100A in the region 3000-3100A. It has a "hot spot" about 2x2 mm which, because of the old law limiting man's ability to increase the brightness of a source, means that the slit should be kept the same size. Taking into account reflection losses of about 10% at each mirror surface and assuming a peak efficiency of 70% for each grating, the maximum throughput of the 1400 turns out to be about 25%. Further, the acceptance angle of the 1400's f/6.8 system is only about 1/60 steradian. Both factors reduce the overall output at the exit to around 8 mW/100A bandpass, a figure roughly half that of our single 1700-II monochromator.

The stray light characteristics of a monochromator are relatively easy to specify with a source of monochromatic light but they are not so readily pinned down for a continuous source. Throughput of the instrument, spectral output of the source and detector response are all parameters with which one must contend. Obviously, an ordinary incandescent lamp will give rise to no scattered light below the transmission limit of its glass envelope. It is not so obvious that two gratings with different blaze properties will cause differences in the scattered light output. Similarly, photomultipliers with different spectral response curves will indicate the same scattered light differently.

The conditions chosen were as follows:

Gratings	pair of 1200 groove/mm blazed at 3000A
Slits	2 mm wide, bandpass 20A
Filter	Corning 7-54, bandpass 2300-4100A
Detector	EMI 6256S
Measured	Intensity 2100A: Intensity 3100A

The only radiation detectable at indicated 2100A would have to be scattered in the region of the filter bandpass, 2300-4100A. Under the conditions cited, the intensity detected at 2100A as ratio to that at 3100A was found to be less than 0.001%, a value too low to be considered causative in the proposed sunburn experiments.

At short wavelengths there is another source of scattered light, the direct image. Between 0° and about 4.5° rotation of the grating, the direct image travels across the focusing mirror. Beyond this angle, the "white light" reflection falls off the mirror edge. When the direct image does impinge on the mirror, more light may scatter, reducing the spectral purity of the indicated wavelength at the exit slit.

Since 4.5° corresponds to about 3500A with a 600 groove/mm grating, it is a good plan to specify 1200 groove/mm gratings below this wavelength when high spectral purity is desired. With the latter, the problem arises below 1750A—a good reason to advise a 2400 groove/mm grating or the second order of a 1200 groove/mm grating in an evacuable monochromator.

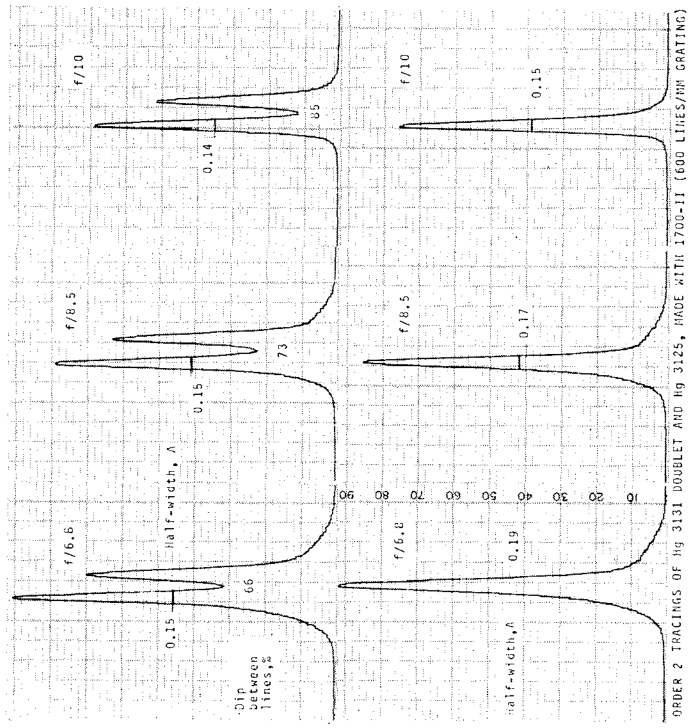
RESOLUTION OF 1700-II

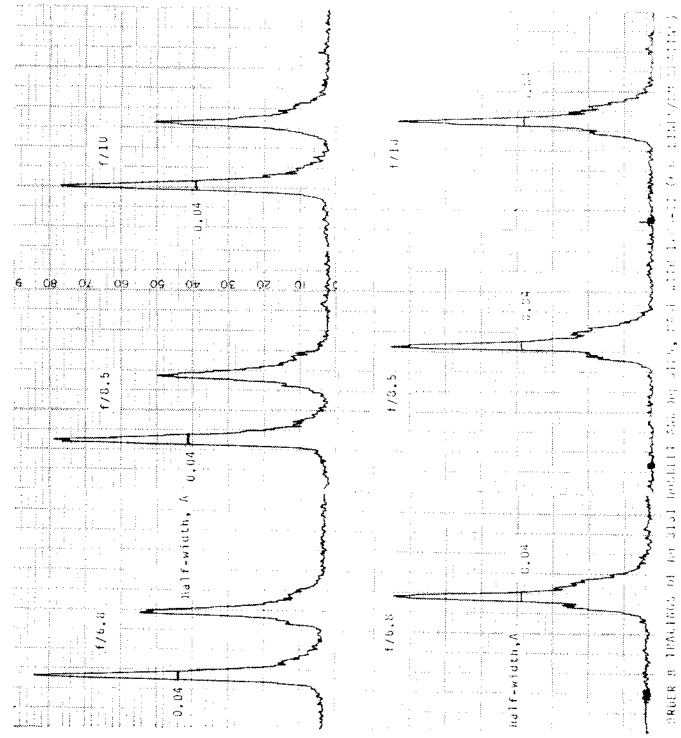
The subject of resolution is besieged with ambiguities, claims and counterclaims. First of all, we come upon a real difference between resolution values of a He-Ne laser source at 6328A and of the 3131A doublet from a penlight source. The former consistently shows appreciably smaller half-width figures. Because precedent seems to have been set with comparison of Hg lines, however, we conducted some tests with the penlight source, obtaining a series of curves with which we hope to disperse some of the fog shrouding the facts of the matter. (Curves on p. 2.)

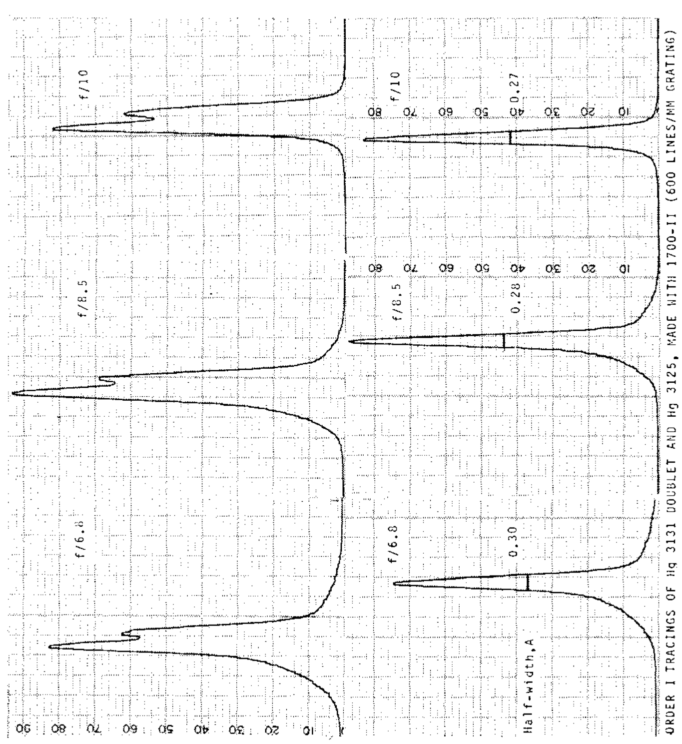
The tracings are of the Hg doublet and its neighboring 3125A line with a 600 groove/mm grating, Spex 1700-II spectrometer in Orders I, II, IV and VIII with f/6.8 aperture followed by f/8.5 and f/10. The progressive improvement in half-width and dip between the peaks is apparent with increasing orders and decreasing optical speed. The dip is an inverse function of both coma and spherical aberration. Note that a line straight down from the peak of 3125A would not divide it in half; the root structure is not exactly symmetrical, indicating some coma.

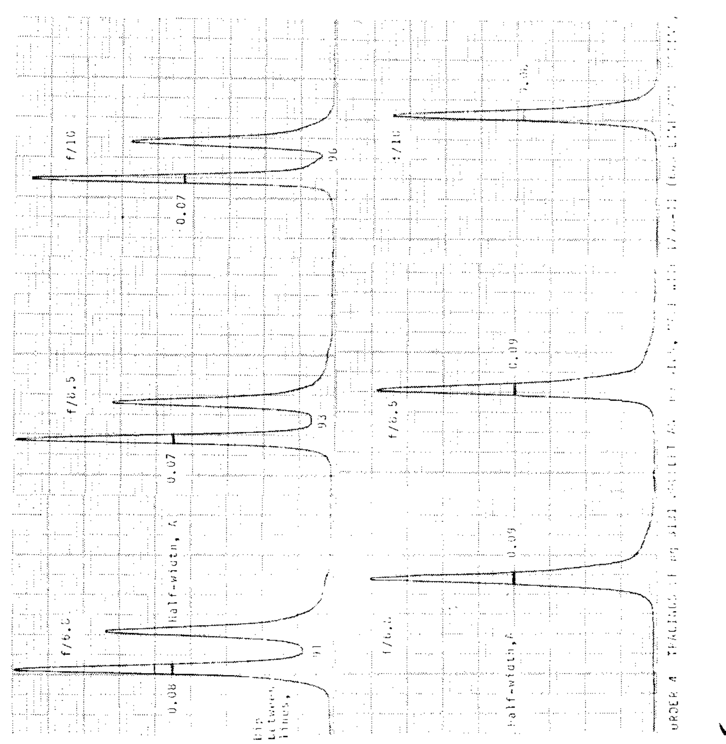
In the eighth order isotope and hyperfine lines, hidden up to now behind the skirts of the strong lines, begin to peek out. The side-band structure in each of the three lines is clearly reproduced and the stronger of the doublet peaks is always freest of parasites. Again we conclude that the most accurate resolution measurements derive from a source devoid of both Doppler broadening and fine structure lines: a laser.

RESOLUTION TRACINGS





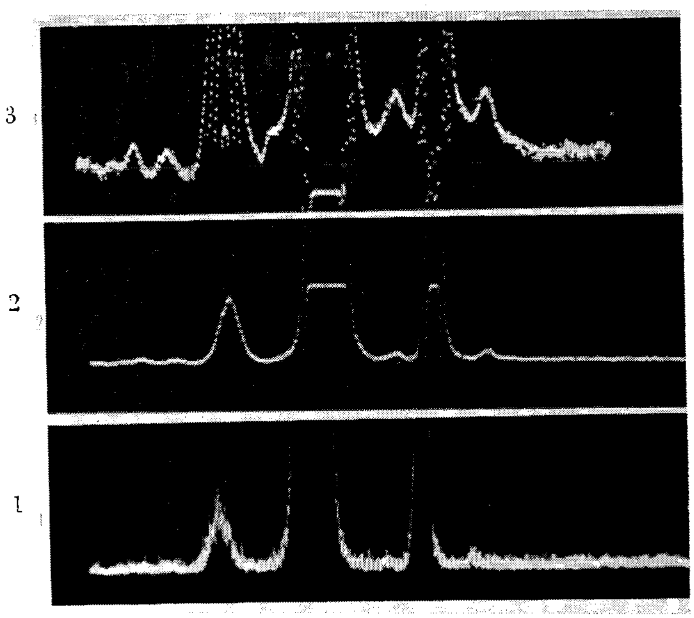




NOISE ABATEMENT

Coping with spectroscopic noise is a matter of concern if you are engaged in ferreting out feeble signals from background. One detection technique gaining foothold for such measurements is that of signal averaging. While the spectrum is repetitively scanned very slowly, a computer divides the total scan interval into a number of narrow bands then processes the digitized signal in each band separately. Operation depends on the principle that if a signal appears in a particular wavelength slot during *every* sweep, the signal is indeed real. Should the slot receive a signal only during some of the sweeps, it is considered spurious and discarded.

One signal averaging digital computer is the Nuclear Data Enhancetron, a portable device about the size of a laboratory oscilloscope. It can have as many as 1024 channels although less expensive models are available with 512 or 256. We connected one to the output of our 1700-II, fitted with a slowly rotating 1626 Refractor Plate, and a 50A "raw" portion of the spectrum of an iron hollow cathode appeared on our oscilloscope screen—the lowest spectrum in the accompanying figure. After about 100 sweeps the Enhancetron processing began to show results, oil was seemingly dumped on the background swells. In 1000 sweeps of the refractor plate (position 3) several lines were seen to emerge. Watching the oscilloscope one finds the emergence almost miraculous; lines grow out of nowhere. Incidentally, the dotted lines between the squared-off overloaded lines represent true segmented extensions of the latter.



Yet if you get a group of physicists together to discuss signal averaging surely one will bring up the photographic plate as a means of accomplishing as much as an $8000. assemblage of electronic gear. Statistically, he will point out, both rely on time—that great healer—to effect a cure. He is right. But over the photographic plate signal averaging boasts at least one big advantage: peak height is proportional to intensity, no H & D excuses.

Can we encourage someone to investigate matters more thoroughly?

NEON LAMP FOR WAVELENGTH CALIBRATION

Spectrometers are subject to two types of wavelength errors. One is run-out, where the real wavelength departs more or less linearly from that indicated over the entire span. The other error is periodic. With a 600 groove/mm grating, one turn of our lead screw results in a sweep of 100A. While a mercury lamp, with lines and various orders of lines, is excellent for checking the run-out, it emits too few lines over a short span for checking the periodic error. For this purpose, a neon lamp has proved satisfactory. A 115-vac General Electric NE-40 pilot lamp has proved exceptionally valuable in that it has a standard lamp thread and a uniform brightness over a circular area about 20 mm in diameter.

Note the indications of periodicity at 5341A, 6143A; also at 5400A and 6402A in the chart of errors over a span of about 1350A in one of our 1500 Evacuable Spectrometers

Line, A		
ACTUAL	READ	ERROR, A
5330.8	5332.4	+1.6
5341.1	5342.5	+1.4
5400.6	5401.6	+1.0
5764.4	5765.6	+1.2
5820.2	5821.3	+1.1
5852.5	5854.0	+1.5
5881.9	5883.2	+1.3
5944.8	5946.2	+1.4
5975.5	5976.4	+0.9
6030.0	6031.2	+1.2
6074.3	6075.7	+1.4
6096.2	6097.8	+1.6
6143.1	6144.6	+1.5
6163.6	6165.2	+1.6
6217.3	6218.5	+1.2
6266.5	6267.8	+1.3
6304.8	6306.1	+1.3
6334.4	6335.7	+1.3
6383.0	6383.9	+0.9
6402.3	6303.4	+1.1
6506.5	6508.1	+1.6
6532.9	6533.9	+1.0
6599.0	6600.5	+1.5
6678.3	6679.8	+1.5

A CONTRIBUTION TO THE J. NEG. RESULTS

When beginning to produce spectrometers some years ago, we considered reversing the exit slit, i.e., mounting it with the bevel facing away from the spectrometer. Preliminary measurements indicated no performance differences between the two mounting methods, however, and we naturally drifted toward the simpler approach. Recently, the subject came up again. This time, with access to a He-Ne laser, we were able to conduct careful line profile and scattered light measurements. Happily, our earlier conclusion was confirmed. Our tracings show virtually no difference when slit bevels are reversed.

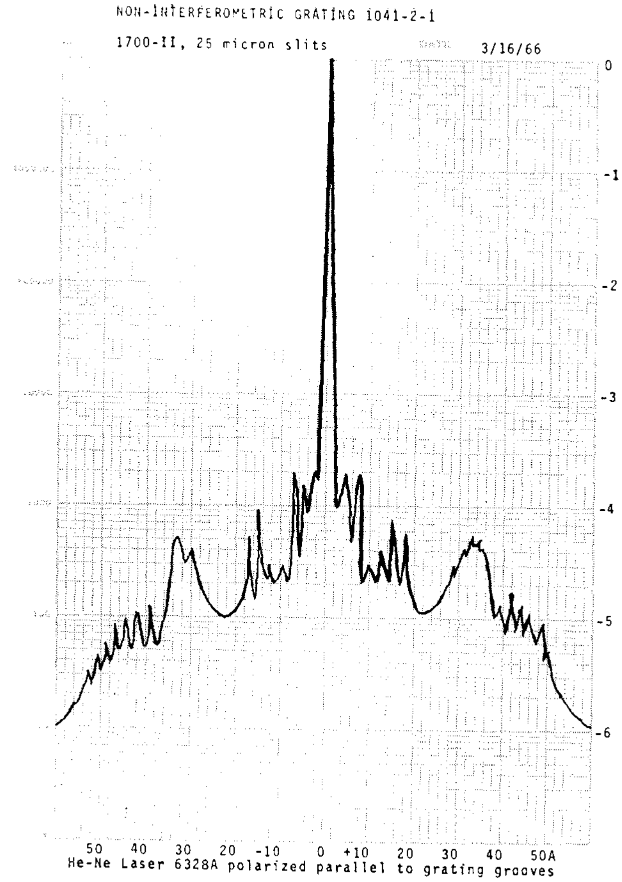
GRATINGS—INTERFEROMETRIC OR NOT

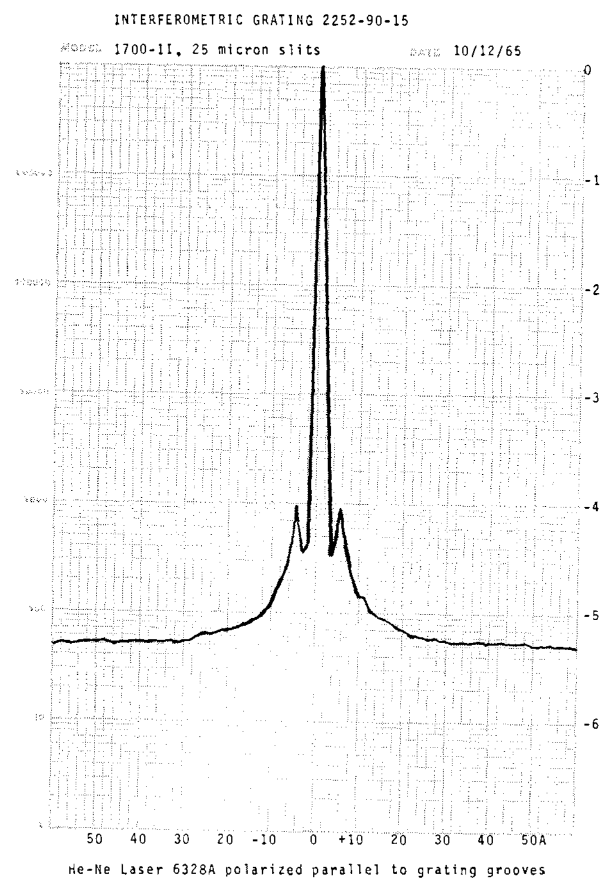
Whether to recommend interferometrically or non-interferometrically ruled gratings for the 1400 Double Spectrometer is a question we tackle anew with each new customer. Our conflict is depicted in the two curves below of a He-Ne 6328A line taken with one grating of each type under otherwise identical conditions. They are from gratings most often specified for Raman spectroscopy with the Ar^{+} laser.

The interferometrically ruled grating (serial 2252) shows a single ghost pair at an intensity of around 10^{-4} of the parent line and the scattered light curve then drifting down to a constant 3×10^{-6}. With a cursory glance at its chart, the non-interferometrically ruled grating appears much worse, its multiple ghost structure extending out to 50A on either side of the parent. Its ultimate scattered light drops to 10^{-6}, however, some three times as good as the other grating. This factor of three is about the lowest we have seen. Some interferometrically ruled gratings exhibit ten times more far scattered light than their counterparts made on the Chicago engine.

At the present state of the art far (beyond 50A) scattered light can be expected to be considerably higher in all interferometrically ruled gratings. As the diamond is drawn across the master blank, its lateral position is continuously controlled. During the excursion, there is a tendency for the servo control to hunt and oscillate, the task of achieving critical damping being characteristic of all servo mechanisms. This results in diamond jitter and, in turn, a microscopic upheaval of the aluminum surface to increase scatter. Incidentally, this jitter may be more pronounced as gratings are ruled finer. The loading on the diamond must be reduced appropriately and so it is more prone to vibration pickup. We have not, however, noticed any systematic increase in scattered light from B&L gratings with increase in the number of grooves/mm.

A conclusion? With the exception of close rotational lines, Raman spectra are best detected with gratings having the least far scattered light. Therefore, a pair of non-interferometric gratings are normally recommended. Since ghost intensity, like scattered light, is reduced to that of the product of both gratings ghosts rarely interfere with real spectra. On the other hand, if the 1400 is ordered as a dual-purpose instrument—the first half as a single monochromator with photographic accessories—it is often better to trade a little total scattered light for an improved photographed spectrum. In such circumstances, we recommend an interferometric grating in the monochromator section.





Send A "Cheer" Card . . .

Bausch & Lomb's David Richardson is waging his biggest fight, this time involuntarily, for his health. Dave's lifelong sparring partner—the diffraction grating ruling engine—is well on its way to being licked. He has had the satisfaction of leading a sucessful campaign to eke out of the machine larger and better gratings and turn a tricky process into one from which scientists could expect a grating tailored to their needs at a reasonable price in a reasonable time.

Formidable as it has been, that contest has proved not nearly as severe as his long bout for his life. We think that many of the friends he has made may want to mail him a few words of cheer to brighten some moments in these difficult days, so here's his home address:

DAVID RICHARDSON
Apartment 4
Colonial Parkway
Pittsford, N. Y.

Won't you join us in a friendship mailathon?

GRATINGS FOR LASER STUDIES

Popular continuous lasers emit lines such as:

Laser	*Principal lines emitted*
Ionized Argon	Many from 4545A upward
He-Ne	6328A
Nd-glass	1 micron

Ordinarily we recommend 1200 groove gratings blazed at 5000A for Ar^{+} lasers, at 7500A for He-Ne and at 1 micron for Nd-glass lasers. Since, however, with lasers there is rarely confusion of overlapping orders (the laser emitting at a single wavelength) a 600 groove/mm grating set for the second order is often equally applicable. Such a grating, blazed at 1 micron, would be suitable for the Nd-glass laser in the first order and for the Ar^{+} and He-Ne lasers in the second order. Typical data on current replicas as compared with those of 1200 grooves follow:

Cat. Description	No. 1501, 1200 g/mm, 5000A	No. 1502, 600 g/mm, 1 μ
Price in Mount	$1235	$755
Efficiency	69% 5790A Order I	84% 5086 Order II
	76% 4800 Order I	90% 4800 Order II
	74% 4047 Order I	92% 4678 Order II
Ghost Int.	0.02%, 5461A, Order I	0.05%, 5461A, Order II
Scattered light	approx. 10^{-6} at 50A from monochromatic line	approx. 10^{-6} at 50A from monochromatic line

PAST ERRATUM

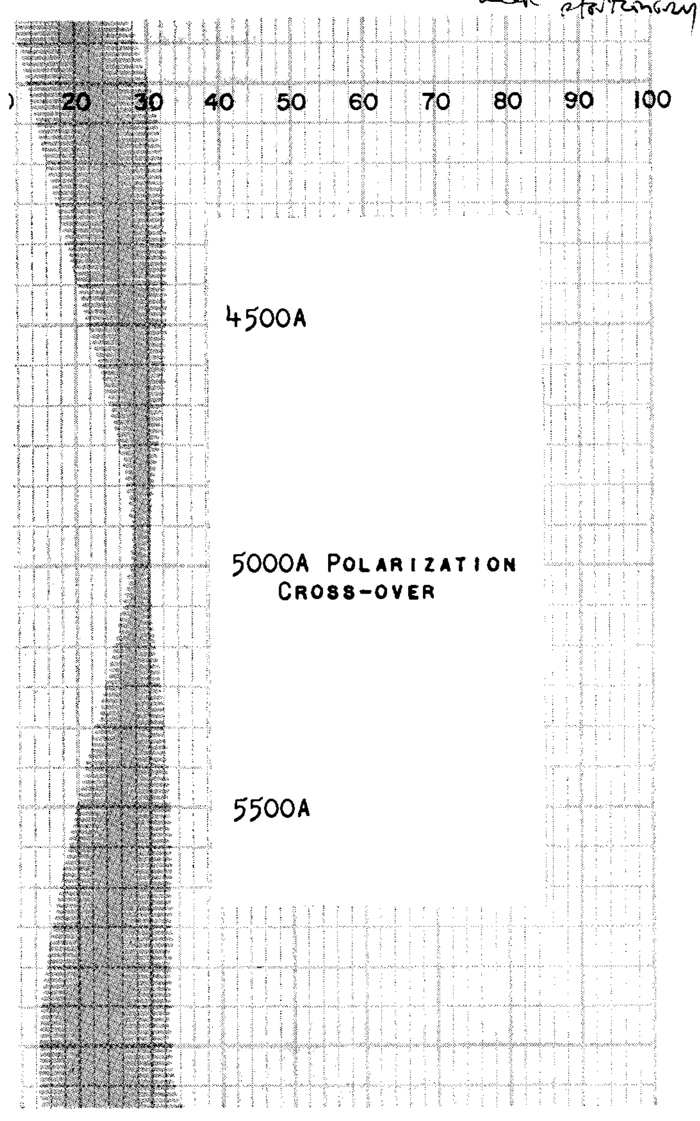
"I wish to congratulate you on the fine article you have written on Laser Raman Spectroscopy in the SPEX SPEAKER Vol. X, No. 1. I found it informative and very appropriate, coming at this time. However, I wish to point out several errors made in the article on page 3 (paragraph below Fig. 2). You point out that the four bands observed in Raman for CCl_4 are stretching vibrations. Actually, two are stretching and two are bending. These, you say, are not observed in the infrared, and thus you conclude that a center of symmetry is present in the molecule. Your final conclusion is that the molecule is of *Td* symmetry. Molecules in *Td* symmetry do not possess a center of symmetry. Further, two bands at 314 and 791 cm^{-1} are also seen in the infrared (See Herzberg, "Vibrational IR and Raman Spectra," p. 311, Table 83.) The final conclusion that the molecule has a *Td* structure is correct, but your reasons for this conclusion are incorrect."

John R. Ferraro
Chemistry Division
Argonne National Laboratory

AUTOMATED POLARIZATION MEASUREMENTS

In the last issue of the SPEX SPEAKER, we called attention to the work of J. G. Skinner and W. G. Nilsen of the Bell Laboratories concerning the variable polarization of gratings. The conclusion reached was that it is a good idea not to tarry below the blaze wavelength if undue polarization is to be avoided.

Not described was the method of making the measurements, a procedure refined by our own Research Director, D. O. Landon, to a point where results are plotted out automatically on a strip chart recorder. The trick is to use a fast cross-chart recorder, one capable of following the fluctuations in output as a polarizer is rotated continuously at a rate of about 1 rps. An incandescent lamp is directed at a stationary Polaroid window in front of the entrance slit of the spectrometer. With a second motor-driven Polaroid window rotating between the exit slit and the photomultiplier the wavelength scanning drive and strip-chart recorder are turned on. The result is a repetitive oscillation of the recorder pen, one end reading maximum transmittance, the other minimum through the rotating window. As the chart paper advances, the envelope of the swinging pen represents polarization *vs.* wavelength. The top part of the envelope follows the parallel polarization, the bottom the perpendicular. Gratings tested thus far indicate a cross-over in polarization at the blaze wavelength.



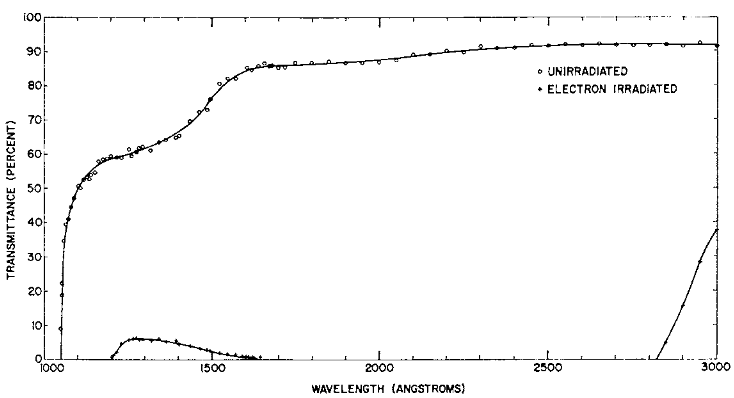
RECOMMENDED WINDOW MATERIALS

In the past we have offered LiF windows and lenses for the region below 1650A, the cut-off wavelength of fused quartz. That of LiF is around 1060A, the lowest wavelength of any practical material. It has been known, however, that under bombardment of x-rays and hydrogen Lyman-alpha at 1216A, coloration develops and the transmittance falls. Although the initial clarity can be restored by heating, this procedure is at best an annoying, time-consuming one.

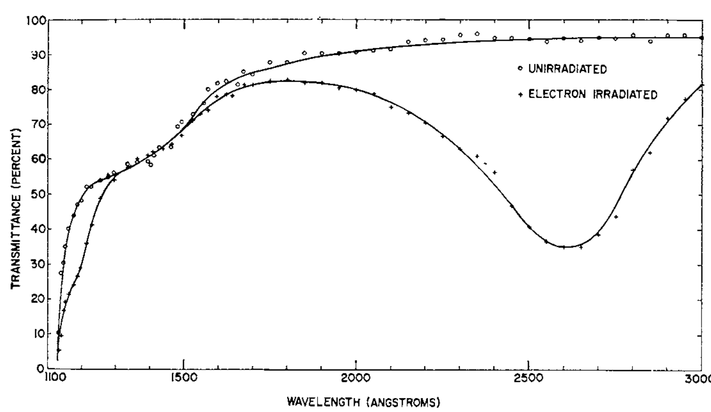
A recent NASA report (*App. Optics*, *5*, 937, 1966) by D. F. Heath and P. A. Sacher shows that MgF_2, while having a somewhat longer terminal wavelength than LiF, is almost unaffected by intense radiation below 1800A. Under the same radiation conditions (10^{14} e/cm_2 at 1.0 Mev for 30 minutes, chosen to simulate passage of a space vehicle through the radiation belt produced by Starfish, the nuclear explosion at 400 km in 1962), the transmittance of LiF below 2800A drops to virtually zero; CaF_2 drops to about one-half of its initial value below 3000A; BaF_2 remains unaffected over its range of transmittance but is extremely costly. The paper also furnishes extensive graphs for sapphire, fused quartz and ADP.

Under many conditions it appears that our 1500 Evacuable Spectrometer can be equipped with permanent entrance and exit windows of MgF_2 to permit changing of sources, samples and detectors without disturbing the vacuum. Sorption pumping can be utilized to maintain a static rather than the normal highly dynamic vacuum.

The 1541 Microwave-Powered Discharge Source can be operated with MgF_2 windowed tubes of hydrogen or any of the rare gases while the spectrometer is kept evacuated with an ion pump. If it is desired to run the lamp dynamically and so vary its pressure with, say, hydrogen or helium, a small mechanical pump will maintain the pressure while flushing.



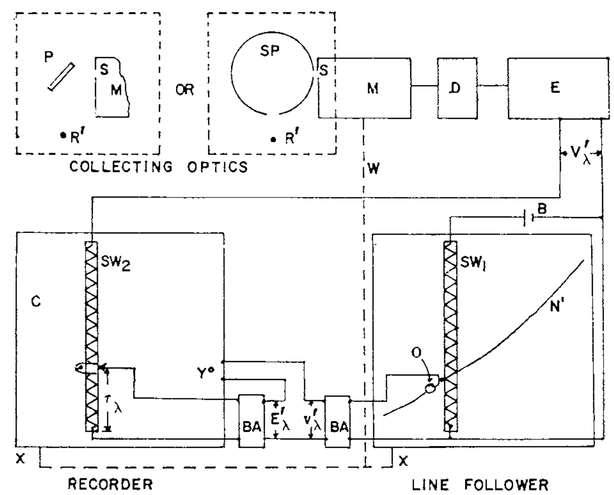
LiF



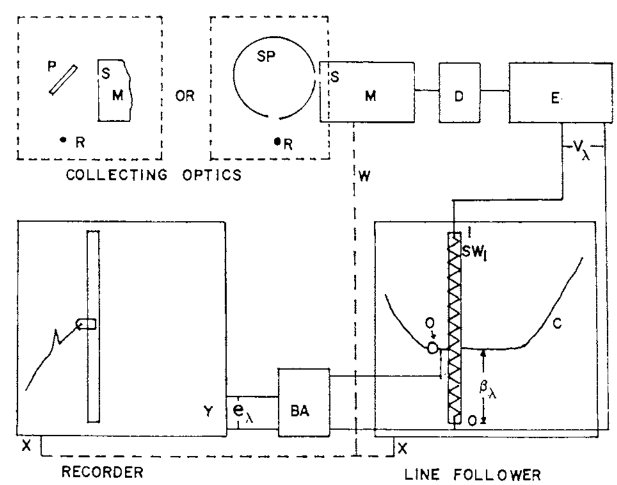
MgF_2

SPECTRORADIOMETRY SYSTEM

(Described on p. 7)



Pictorial diagram of system for automatic preparation of compensator function: P=reflection plaque, SP=sphere, R′=standard source, S=entrance slit, M=monochromator, D=detector, E=electrometer amplifier, W=voltage proportional to wavelength, X=recorder and line follower X-axis input, Y°=recorder Y-axis servo input, BA=buffer amplifier, SW_1=line follower slidewire, SW_2=recorder slidewire, O=optical pickup, B=battery, N′=plot of spectral distribution of standard source, C=compensator function in preparation.



Pictorial diagram of spectroradiometer: P=reflection plaque, SP=sphere, R=test source, S=entrance slit, M=monochromator, D=detector, E=electrometer amplifier, W=voltage proportional to wavelength, X=recorder and line follower X-axis input, Y=recorder Y-axis input, BA=buffer amplifier, SW_1=line follower slidewire, O=optical pickup, C=compensator function.

(Courtesy ILLUMINATING ENGINEERING)

A SAMPLING OF STRANGE DOINGS

1700 In Spectroradiometry

A curve of energy output *vs.* wavelength for a lamp is not easy to come by; throughput of the spectrometer and response of the detector both vary considerably with wavelength. R. L. Brown of General Electric Co., Nela Park, Cleveland, Ohio devised a radiometric scheme (*Illum. Eng., LXI*, 230, 1966) utilizing an NBS standardized lamp as reference to obtain an overall compensator function. The NBS curve is drawn by hand and placed on an X-Y recorder bed fitted with an optical follower in such a way that the compensating curve is automatically plotted on a second X-Y recorder. The Spex 1700 Spectrometer with a single grating records the spectrum from 2500 to 10,000A, the compensating function correcting the final curve of energy *vs.* wavelength.

The block diagrams on p. 6 show the system employed.

The 1400 Feeds a Computer

Computers have wended their way even into research laboratories! C. A. Penchina, at the University of Massachusetts in Amherst is modifying his double spectrometer to feed Raman data to a memory bank, there to be compared with other spectra, retrieved or discarded by the press of the boss's button finger. The three slits on the spectrometer have been ganged to maintain constant output with changing wavelength, which is recorded digitally through an absolute shaft encoder. When completed, his system will rely on the computer to identify the Raman spectrum of an unknown substance or, failing that, at least provide worthy, educated guesses.

The 1500 Accounts for Itself in the Space Program

J. C. Burger of Westinghouse's Tube Division in Elmira, N. Y., is appraising the spectral sensitivity of their Uvicon, an ultra-violet image orthicon to be used in space explorations. A mercury Penlight calibrated by NBS is the starting point from which our evacuable spectrometer takes over. Incidentally, an excellent technique for approximating spectral sensitivity from 1942A to 5790A is to reference the relative intensities of about 40 lines of the mercury penlight measured by Childs of NBS (C. B. Childs, *App. Optics, 1*, 711, 1962).

Reclining 1700s, or Upside-down?

Several experimental souls have reported that the non-evacuable single spectrometer is relatively insensitive to attitude changes (although temperature must be maintained fairly constant). We have always known that for its resolution it is a lightweight, at 80 pounds, and wavelength can be scanned remotely and telemetered. To J. Bohse, University of Maryland, who is planning to send his spectrometer 36 km high in a balloon, such particulars have import. He intends to measure the intensity of sunlight, from a vantage above atmospheric ozone, alternately with that of the absorption at 2800-3000A of the ozone layer below.

The 1800 Photographs Psuedo-Micrometeorite Impact Flashes

The impact of a micrometeorite on a space vehicle could be so violent as to vaporize metals. Concerned with the possibility of dangerous penetration, NASA is sponsoring a research project at Computing Devices, Ottawa, where, in a long evacuated tunnel, B. Jean fires 1/2″ diameter balls at almost escape velocity against a target. To gain insight into the impact reactions, in the hope of developing materials able to withstand such encounters in space, the light flashes emitted are studied with our high-speed spectrograph.

Reading Wavelength with a Digital Voltmeter

With a digital voltmeter and a 15-volt dc supply R. E. Shrader, RCA Laboratories, Princeton, N. J. converted our 1542 Wavelength Analog Takeoff into a remote-reading device. Noting that the wavelength range extends over about 15,700A, he reasoned that a digital voltmeter would, to a first approximation, read angstroms directly in millivolts. The fact that the range is not exactly 15,000A leads to a small error which was "swept out of the way" with trimming resistors. He connected a 200-ohm trimmer to either end of the precision potentiometer and with two adjustments achieved (Case C) what he was interested in: high wavelength accuracy in the region 4000-6500A.

Shrader first set the high and low trimmers so that 1.000 volts was equivalent to 1000A and 14.000 volts to 14000A, thus coming by the values in Case A. Case B represents his first attempt at improving the wavelength accuracy in the visible region.

Of course, once digitized electrically, the signal can be further processed for computer applications.

Setting, A	*Case A Error, A*	*Case B Error, A*	*Case C Error, A*
2000	—16	+32	+6
3000	—25	+22	+1
4000	—31	+11	0
4500			—1
5000	—39	0	—3
5500			—2
6000	—41	—6	+1
6500			+1
7000	—40	—9	+7
8000	—28	—1	+26
9000	—16	+9	+41
10000	—13		+50*
11000	—20	0	+50
12000	—10	+10	+60
13000	—6	+20	+80
14000	0	+10	+100

*Above 10V the least count is 10 millivolts.

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He will look forward to the opportunity of working among people in the finest industrial, government and academic laboratories in the Middle Atlantic States, picking up ideas here and dispensing them there.

If you have the qualifications and ambitions along these lines, sharing the growth potential of our small but solid company may appeal to you.