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Conversion of the Perkin-Elmer Infrared Microscope Attachment for Double-Beam Operation

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The Perkin-Elmer Infrared Microscope Attachment, used together with model 13 spectrometer, has been converted to double-beam operation by removing the beam-chopper and inserting a shutter-disk in the image plane of the microscope. The shutter-disk, which is revolved synchronously with the spectrometer breakers, is equipped with two semi-circular slits so situated that the light is passed alternatively from the sampling and reference area to the detector. The arrangement allows for recording of spectra from microgram quantities with medium resolution.

The reflecting microscopes employed in infrared microspectrometers have been placed in two different positions in the spectrometer: (a) Between the source and the entrance slit of the monochromator. (b) Between the exit slit of the monochromator and the detector. Temperature and photochemical effects in the sample are generally considered to be of importance when the microscope is situated in position (a), where the sample is subjected to a concentrated beam of undispersed radiation. With the microscope in position (a) Ford *et al.*[1], however, found the rise to be only about 1°, when using a Nernst source. They consider also the ultraviolet radiation from this source to be of minor importance. On the other hand, the possible effects of concentrating an undispersed beam of radiation on the sample are certainly dependent on the material under investigation and the radiation source used. Since there is a general need of more intensive radiation sources in infrared microspectrometry than the Nernst and Globar sources, the disadvantage of position (a) will no doubt be more pronounced when, for instance, a carbon arc is employed. Therefore, the construction of microspectrometers which take advantage of both double-beam operation and of position (b) is of importance. As far as the present authors know, the only apparatus hitherto designed according to these specifications is that described by Haggis [2] which employs a rocking mirror to displace the optic axis through the microscope, so that the beam passes alternatively through the sample and an adjacent reference area.

The present paper describes the conversion of the Perkin-Elmer Infrared Microscope Attachment [3] for double-beam operation. The spectrometer used together with the microscope was a Perkin-Elmer model 13, equipped with a Nernst source and a sodium chloride prism.

GENERAL DESCRIPTION

In order to dispose the sample and the reference alternatively into the beam, experiments with a vibrating stage, placed in the sampling plane of the microscope, were performed by the present authors. However, neither a solenoid driver, nor a pure mechanical system synchronized with the beam-chopper and breakers of the filter circuits were found to give a reliable operation. The difficulties involved were no doubt connected with the rather high frequency of the oscillatory movement necessary to fit the 13-cps system of the spectrometer. Ford *et al.*[1], who employed such a technique successfully, used an oscillatory frequency of 4-cps only.

In consequence of these experiments, another method was developed, utilizing a rotating shutter-disk, placed in the image plane of the microscope. The shutter-disk is equipped with two semi-circular slits. Being of different radii, the two shutter-slits, termed I for sample and I_0 for reference, alternatively pass light from the sample and an adjacent clear area or reference. The relation between the rotating shutter-slits and the image of the spectrometer exit-slit is shown schematically in Fig. 1. The slits are situated in such a relation to each other, that the four operations described below take place when the shutter-slits cross the image of the spectrometer slit during one complete revolution of the shutter-disk:

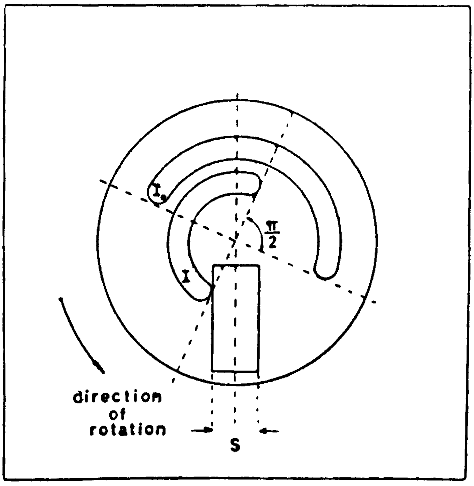

Fig. 1. Schematic diagram of the two semi-circular slits and the image of the spectrometer exit-slit of the width *S*.

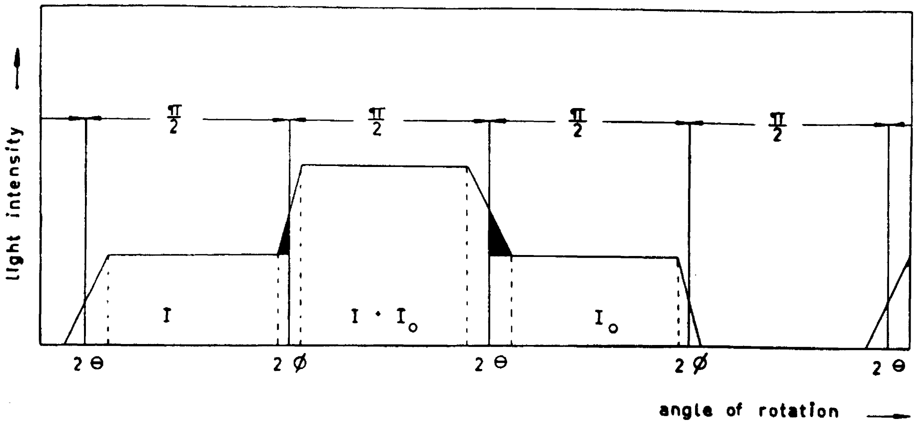

Fig. 2. Schematic diagram of the angular distribution and light transmission of the four spectrometer operations during one revolution of the shutter-disk. The solid vertical lines indicate the position in the cycle where the breakers are actuated. The dashed lines indicate the positions where the leading and trailing edges of the shutter-slits cross the lateral sides of the spectrometer slit image. The shaded areas correspond to the cross-talk. The diagram is constructed for a spectrometer slit-width of 500 μ.

(1) Light from the sample area passes through the I-slit to the detector.
(2) Light from both the sample and the reference areas passes simultaneously through the appropriate shutter-slit to the detector.
(3) Light from the reference area passes through the I_0-slit to the detector.
(4) No light passes through the shutter-disk.

By these means the cycle of operation of the model 13 ratio recording system is reproduced when the shutter-disk is revolved synchronously and in proper phase with the spectrometer breakers. In order to match the spectrometer system, the duration of each of the four operations listed above has to be equal to a quarter of a revolution. Such a condition, however, prevails only when the spectrometer slits are infinitely narrow. Obviously, the individual lengths of the four operations are functions of the actual slit-width, as seen from Fig. 1. These lengths, together with the distribution of light passed through the rotating shutter-disk are shown in Fig. 2. The solid vertical lines drawn in the diagram indicate the position in the cycle, where the spectrometer breakers are actuated. Under the conditions outlined in Fig. 2 it is evident that a certain amount of light from the I_0-channel is introduced into the I-channel and *vice versa* when the spectrometer slit is > 0. The cross-talk, $C(I)$ and $C(I_0)$, introduced into the sampling and reference channel, respectively, may be expressed as the ratio of the shaded area to the total area bordered by the solid vertical lines in Fig. 2 or, provided no sample is inserted into the beam, thus:

$$C(\mathrm{I}) = \frac{b \cdot \varphi}{\frac{1}{2}\pi - a \cdot \vartheta + b \cdot \varphi} \quad (1)$$

$$C(\mathrm{I_o}) = \frac{a \cdot \vartheta}{\frac{1}{2}\pi + a \cdot \vartheta - b \cdot \varphi} \quad (2)$$

where $\varphi = \sin^{-1} \frac{S}{2\,R_{I_o}}$ and $\vartheta = \sin^{-1} \frac{S}{2\,R_I}$

R_I denotes the radius of the sampling shutter-slit, and R_{I_o} that of the reference shutter-slit. S designates the width of the spectrometer slit in the image plane of the microscope objective. When the operating positions of the spectrometer breakers are in exact quadrature, and the centre of rotation of the shutter-disk is aligned symmetrically with the image of the spectrometer slit, the constants a and b are equal, and may be set approximately to 1/4. $C(\mathrm{I})$ and $C(\mathrm{I_o})$ are shown in Table 1 for some various slit-widths. The numerical values of R_I and R_{I_o} are those of the apparatus to be described, and are equal to 5.0 and 8.0 mm, respectively. The evaluation was performed under the assumption of an image reduction by the microscope condenser of 8.4 × and a magnification by the objective of 25 ×.

The percentage cross-talk will increase enormously in a region of low transmission, and the absorption is thus erroneously decreased. In a 100 % transmission run, the presence of cross-talk will cause a deviation of the recorded curve. Being of negligible magnitude in the short-wave region, the error will increase towards that of long waves. The error introduced by the cross-talk can be reduced by the following procedures:

(1) By selecting the channel of less cross-talk, see Table 1, as the sampling channel, *i.e.* the channel corresponding to the shutter-slit of minor radius.

(2) By increasing the energy output from the source in order to obtain a narrower slit, especially in the long wave range.

(3) By inserting a rectangular diaphragm into the image plane of the microscope objective, thus limiting the effective maximum slit-width of the spectrometer.

(4) By moving the trigger position of the spectrometer breakers, so that the shaded areas of Fig. 2 are reduced.

Of the four procedures outlined above, (1) and (4) have been applied in the present work. Since no light source other than the standard Nernst filament

Table 1. Percentage cross-talk introduced into the sampling and reference channels for some different spectrometer slit-widths, evaluated from eqns (1) and (2). No adjustment for reduction of cross-talk.

Slit-width, μ	$C(\mathrm{I})$	$C(\mathrm{I_I})$
0	0	0
500	1.7	2.5
1 000	3.3	5.1
1 500	5.3	7.7
2 000	6.9	10.5

has been employed, the procedures (2) and (3) were not tried. In order to reduce the error by procedure (4), the cross-talk of either channel can be reduced by readjusting the phase between the rotating shutter-disk and the breaker assembly, or by displacing the centre of rotation of the shutter-disk. It is thus possible to displace all the vertical lines, *i.e.* the trigger positions of the breakers in Fig. 2, to the left, resulting in reduced cross-talk in the I-channel. To reduce the cross-talk of the I_0-channel also, the solid vertical line between the $(I + I_0)$ — and the I_0-operations has to be moved to the right by adjusting the individual position of the appropriate spectrometer breaker. It is not possible, however, to displace the trigger position of the breakers over a certain limit from the position of quadrature without seriously disturbing the operation of the model 13 rectifier system.

MECHANICAL DESIGN AND CONSTRUCTION

A photograph of the modified model 13 Spectrometer and Microscope Attachment is given in Fig. 3. Fig. 4 shows the rotating shutter-disk with the semi-circular slits.

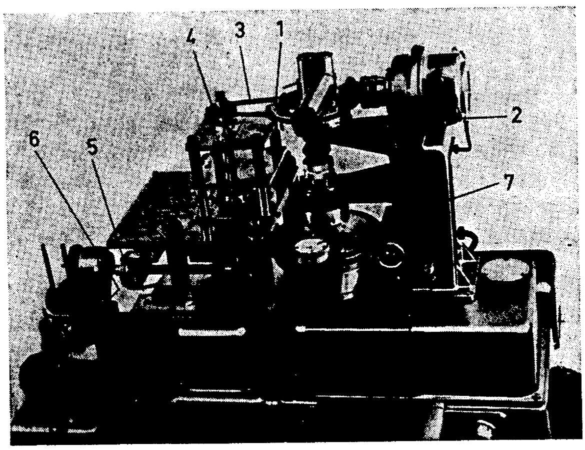

Fig. 3. Complete view of the apparatus. Refer to the text for numerical designations.

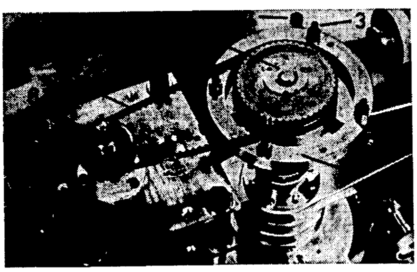

Fig. 4. The rotating shutter-disk with the semi-circular slits. The moulded housing containing the diaphragm field mirror has been removed. Refer to the text for numerical designations.

The diaphragm leaves of the microscope attachment, together with the leaf controls, were removed, and the standard diaphragm holder was replaced by a special holder (Fig. 3:1, 2, Fig. 4:2), intended to support the rotating shutter mechanism. The new holder was equipped with the appropriate spacers (Fig. 4:3) for supporting the moulded housing containing the diaphragm field mirror assembly, for the purpose of maintaining the accurate position of this mirror. The rotating shutter-disk with the two semi-circular slits (Fig. 4:1) was attached to the centre of a socket fitted into the inner ring of a ball bearing (SKF type 16007 X, 62 × 35 × 9 mm). The outer ring of the bearing was supported in a frame machined to the shape of a frustrum of a cone. This frame, together with the ball bearing and the rotating shutter-disk, was placed in a horizontal position on the special holder and was then accurately secured in position by three adjusting screws (Fig. 4:4).

The shutter-disk was caused to revolve by the aid of a Power Grip Timing Belt system. The driven pulley was attached to a flange on the socket supporting the shutter-disk. The timing belt (type 190 XL 025) (Fig. 3:3, Fig. 4:5) was led outside from the microscope assembly through the open space between the special holder and the diaphragm field mirror housing.

The driving pulley (Fig. 3:4, Fig. 4:6) was attached to the end of a vertical shaft supported by ball bearings in a heavy frame-work mounted on the lower of the two spectrometer source covers. After removal of the spectrometer beam-chopper, the pulley shaft was linked to the breaker shaft of the spectrometer by a gear and shaft train and a second timing belt system (Fig. 3:6) to provide direct transmission between the breaker shaft and the shutter-disk. As the standard chopper motor was not powered for driving this assembly, a synchronous motor (1500 rpm, 25 watts) was employed. This motor was placed beside the spectrometer. A pulley (Fig. 3:5) and belt system was used for transmission, so that the frequency of rotation of the breaker shaft was approximately 13-cps. This transmission system has been found to be very reliable and devoid of mechanical vibrations. The rotating shutter and the heavy frame-work were made of brass, the special holder and pulleys of duralu-minium.

The two semi-circular slits are cut at a slightly larger angle than the theoretical value of π radians. After the spectrometer had been properly adjusted for the standard macro-operation, the angular values, to be used when cutting the slits, were obtained by reading the angular distribution of the four spectrometer operations from the position of a fixed index on a dial, engraved on the driven pulley (Fig. 3:5) of the spectrometer breaker assembly. The chopper shaft was rotated manually, and the positions at which the light from the source was obscured or just passed by the chopper-blade, were noted.

With shutter-slits of 2.0 mm width, the width of the sampling area amounts about 0.08 mm. The length of the sampling area depends upon the spectrometer slit-width and consequently varies with the wave-length. When the slit is fully opened, *i.e.* 2.00 mm, the sampling area becomes $0.08 \times 0.240 = 1.92 \times 10^{-4}$ mm^2. In the sampling plane, the free space between the sampling and the reference areas is about 0.12 mm.

When adjusting the double-beam operated microscope to "Zero" and "100 % transmission", edge-formed shutters could be inserted into the appro-

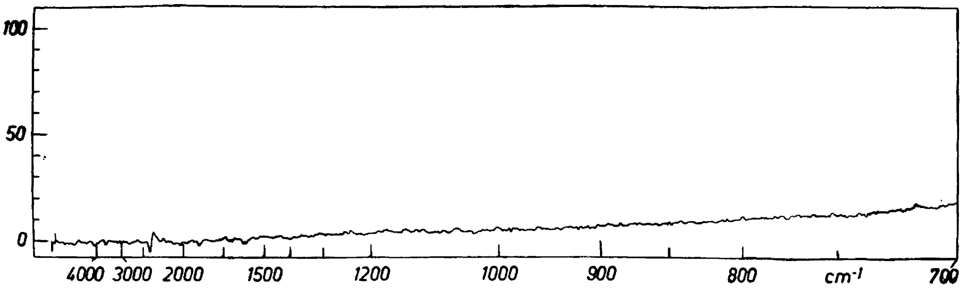

Fig. 5. Recorded curve of a 100 % transmission run. The y-coordinate gives the percentage absorption. Operating positions of the model 13 spectrometer controls: Ratio I/I_0, constant I_0, slit-servo 1.60, gain 18, full scale 6, response 3, speed 2 (1:1).

priate path in the sampling plane of the microscope. One of these shutters was also utilized for supporting the sample. The position of the shutters and the sample was controlled by a vernier screw and a dial displacement gauge (Fig. 3:7), mounted on the microscope stage. Precautions have to be taken to prevent the aperture of the microscope condenser and objective from being impaired when using these shutters.

ADJUSTMENTS

The apparatus was adjusted to minimum cross-talk by observing the position of the shutter-slits through the microscope viewer in visible light when the transmission system was manually revolved. The trigger position of each breaker was indicated by the lighting of a low-voltage lamp, connected in

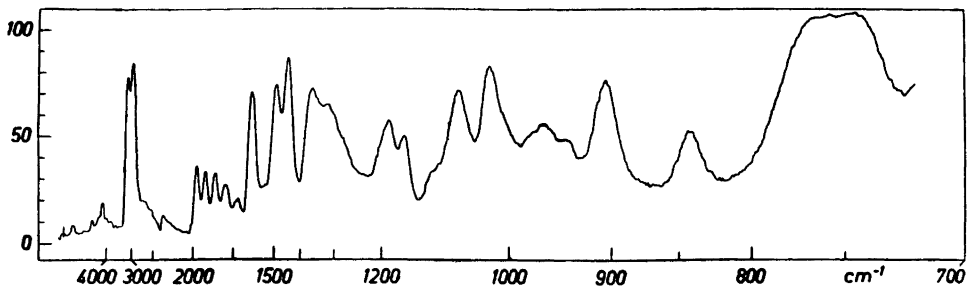

Fig. 6. Recorded spectrum of a polystyrene film. Maximum amount of substance in the sampling beam about 1.3 µg. The y-coordinate gives the percentage absorption. Operating positions of the spectrometer controls are the same as those given for Fig. 5. Reference channel: air.

series with the breaker and a small battery *. By these means it was possible to eliminate the cross-talk of the sampling channel. The cross-talk of the reference channel, however, could not be decreased much below the values given in Table 1 for the I_0-cross-talk without interfering with the spectrometer operation.

In order to make sure that the light, passed through both shutter-slits, falls well inside the sensitive area of the thermocouple, its position was aligned when observing the signals of the sampling and reference channels on a dual trace oscilloscope.

PERFORMANCE

The curve of a 100 % transmission record is shown in Fig. 5. The deviation from a constant level, increasing towards the long wave extreme, is a consequence of the light distribution already discussed above, and shown in Fig. 2. The compensation of atmospheric disturbances is found to be rather effective. The humidity of the air was about 80 % when the record was made.

The recorded spectrum of a polystyrene film of 0.07 mm thickness is shown in Fig. 6. Assuming the density of polystyrene to be equal to 1.0, the effective amount of substance present in the sampling beam is about $0.08 \times 0.240 \times 0.07 \times 1.0 = 1.3\ \mu g$ when the spectrometer slits are opened at their maximum width. Due to lack of sufficient energy, the record had to be interrupted at about 725 cm^{-1}, since the spectrometer slits had opened at maximum width at this wave-number. For the same reason the resolution is slightly lower than that generally obtained in macro-operation. The actual slit-width at 3 000 cm^{-1} was about 70 μ.

DISCUSSION

The problem associated with the presence of cross-talk might be simplified by the use of a breaker and filter system different from that of the model 13 spectrometer. However, the modification described above has the advantage of simplicity as it involves no changes either in the electronic system, or in the optics apart from the insertion of the shutter-disk instead of the beam-chopper. As compared with the system described by Haggis [2], the present arrangement has the advantage of avoiding abberations caused by the off-axis displacement inherent to this method.

The preparation of samples suitable for use with the present method will largely depend on the material to be investigated. Methods for preparation and handling of samples for infrared microspectroscopy have been described elsewhere [4]. With some forms of material, the problem of providing a clear space close to the sample will no doubt constitute a limiting factor of the present method.

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* A similar technique is recommended by the makers when adjusting the breakers in the standard operation of model 13 spectrometer.

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