Experimental Study of Monochromators in UV–Vis and IR Spectrophotometers

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The use of monochromators to select narrow wavelength bands is well-known. Monochromators generally consist of several parts: a component for dispersing the radiation; entrance and exit slits; and collimator and focusing elements. The dispersion or separation of different wavelengths is achieved in one of two ways: refraction using a prism or diffraction using a grating. Today most optical instruments use a reflection grating due to the good dispersion that results.

Teaching chemical instrumentation, we have noticed that most textbooks give only a numerical example of the dispersion of UV–vis radiation, omitting the IR radiation. Thus, the student may never learn the similarities and differences that distinguish the usual mountings of UV–vis spectrophotometers from those of IR spectrophotometers. Thus, they may be unable to answer certain types of questions: What are the actual values of grating angles? What are the dimensions of the gratings? If we simply explain the role of monochromators on the blackboard, students may continue to think of these chemical instruments as a "black box".[1]

In this article, we relate a laboratory excercise that solves the above-mentioned problem: The students strip down a UV–vis spectrophotometer and an IR spectrophotometer. They observe the position of several components of the optical instruments (source, lens, slits, detector), and they study the different mountings of the monochromators. They meausure the grating angles and calculate the corresponding wavelengths from the grating equation, comparing them with those shown on the control panel of the spectrophotometers. This is the first experiment that the students carry out in a course on chemical instrumentation, and they have shown great interest.

Experimental Apparatus

In the UV–vis range we used a Varian 634 spectrophotometer equipped with a deuterium lamp and a tungsten lamp. The monochromator grating is a 32 × 27-mm ruled area with 1276 grooves per mm. In the IR range we used a Beckman Acculab 100 spectrophotometer whose grating is 50 × 40 mm with 100 grooves per mm. However, for this experiment any UV–vis (molecular or atomic) and IR spectrophotometers (even the old models) can be used, as long as stripping them down does not disturb their analytical application.

Results

UV–Vis Monochromator

The UV–vis spectrophotometer has a monochromator with a Czerny–Turner mounting. (See Fig. 1.) Two small concave mirrors are used as collimating and focusing elements. The slit widths are 0.2, 0.5, 1.0, and 2.0 nm. The sum $i + r$ is fixed at 2α, and the experimental measuring result is $2\alpha = 20°$. The grating angle β is variable from 0° to 36°. The known grating equation is

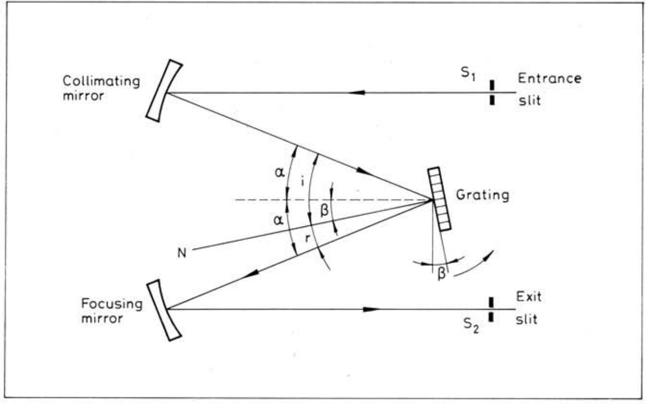

Figure 1. Czerny–Turner mounting in UV–Vis spectrophotometers. *i*, incidence angle. *r*, refraction angle. *N*, grating normal. β, grating angle. α is fixed to 10°.

$$m\lambda = d\,(\sin i - \sin r) \qquad (1)$$

The negative sign shows that angles i and r are on opposite sides of the normal. This equation can be changed in the following way. From Figure 1, we can see that $i = \alpha + \beta$ and $r = \alpha - \beta$. Substituting them into eq 1, we obtain

$$m\lambda = 2d\,(\cos\alpha)\,(\sin\beta) \qquad (2)$$

We use the following values. For the first order, $m = 1$. The distance between grooves is given by

$$d = \frac{1}{1276 \times 10^{-6}}\ \text{nm} = 783.7\ \text{nm}$$

and $\alpha = 10°$. Then we obtain $\lambda\ (\text{nm}) = 1544 \sin\beta \qquad (3)$

Table 1. Wavelengths as a in Function of Grating Angles

β	sin β	λ, nm
5	0.087	134
7.5	0.129	200
10	0.173	268
15	0.258	399
20	0.342	528
25	0.422	652
30	0.500	772
35	0.573	885
35.7	0.583	900

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[1] Laitinen, H. A. *Anal. Chem.* **1979**, *51*, 177.

Table 2. Limits of Wavelengths

slit, nm	deuterium lamp lower λ, nm	tungsten lamp upper λ, nm
0.2	190	785
0.5	185	830
1.0	110	850
2.0	108	875

Table 1 shows the range of β values used to obtain the corresponding wavelengths in the UV–vis region. To avoid wavelength overlapping of several orders, cutoff filters must be used. Although the wavelengths dispersed by the grating can take any value given by eq 3, the radiation actually obtained will depend on whether sufficent energy reaches the detector. Thus, it depends on the radiation source and the slit width. Table 2 shows the lower and higher limits of the wavelengths for the UV–vis spectrophotometer used by us. On the other hand, in the UV region the lower wavelength for absorption of radiation in liquid samples is about 185 nm, which is the transmision limit of a quartz envelope.

IR Monochromator

In the IR region the Littrow mounting (Fig. 2) is the most widely used for IR spectrophotometers. The difference with a UV–vis spectrophotometer is that it requires a single paraboloid mirror for focusing and collimating the beam of radiation. As a result, the incidence and refraction angles are on the same side of the grating normal and are nearly equal. Thus, the grating equation in terms of wavenumber can be written as

$$\bar{\nu} = \frac{1}{\lambda} = \frac{m}{2d \sin i} \tag{4}$$

giving values of distance between grooves

$$d = \frac{1}{10^3} \text{ cm}^3$$

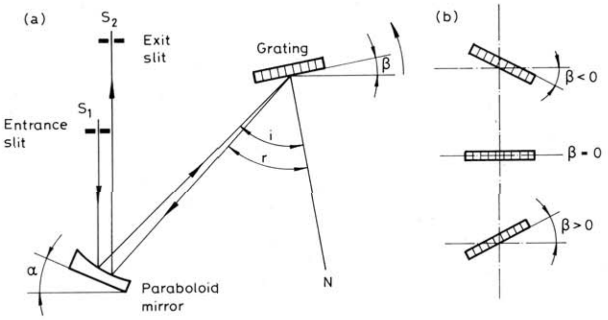

Figure 2. (a) Littrow mounting in IR spectrophotometers. *i*, incidence angle. *r*, refraction angle. *N*, grating normal. β, grating angle. α is fixed at 19°. (b) Position of the grating angle, β.

we obtain:
for second order, $m = 2$

$$\bar{\nu}\ (\text{cm}^{-1}) = \frac{1000}{\sin i} \tag{5}$$

for first order, $m = 1$

$$\bar{\nu}\ (\text{cm}^{-1}) = \frac{500}{\sin i} \tag{6}$$

The incidence angle is $i = \alpha + \beta$, where α is fixed at 19°, and the grating angle β is variable from –4.5° to 37.5°. Table 3 shows the values of grating angle β and the incidence angle i for the suitable wave number in the IR region.

From 4000 to 2000 cm^{-1} the wavenumbers are selected with the second-order difraction. On reaching 2000 cm^{-1}, the grating angle comes back to the initial position. To select the suitable wavenumber, filters are mounted on an eight-position wheel. The first three positions corresponds to transmision filters that allow the second-order difraction radiation to pass, while blocking the first-order radiation. For the remaining five filter positions, pass filters are used to allow the first-order difraction radiation to pass.

Some IR spectrophotometers, for example Perkin–Elmer Model 781, have a second grating with 25 grooves per mm, and $d = 1/250$ cm. This allows us to select wavenumbers in the range 600–200 cm^{-1}. In this case the grating equation for the first order is given by the following equation.

for first order, $m = 1$

$$\bar{\nu}\ (\text{cm}^{-1}) = \frac{125}{\sin i} \tag{7}$$

Table 4 shows the incidence angles to obtain the corresponding wavenumber.

Table 3. Wavenumbers as a Function of Grating Angles

β	$i = \alpha + \beta$	sin *i*	Range 4000–2000 $m = 2$ $\bar{\nu}$, cm^{-1}	Range 2000–600 $m = 1$ $\bar{\nu}$, cm^{-1}
–4.5	14.5	0.250	4000	2000
–4	15	0.259	3860	1930
0.5	*19.5*	*0.333*	*3000*	*1500*
11	30	0.500	2000	1000
21	40	0.643	—	777
31	50	0.766	—	652
37.5	56.5	0.863	—	600

Table 4. Wavenumber as a Function of Incidence Angles

i	sin *i*	$\bar{\nu}$, cm^{-1}
12	0.208	600
15	0.259	482
20	0.342	365
30	0.500	250
38.7	0.625	200