Infrared Spectrophotometry: A Semi-Micro Die for Oblong KBr-pellets

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A macro-die for oblong pellets has been described by Kirkland [1]. His pellet was pressed in a rather complicated tool, which was afterwards inserted in the spectrophotometer.

We needed a similar device, but as we wanted to store the samples afterwards and also wanted a smaller pellet (we had earlier made a beam-condensing microscope for our Perkin-Elmer model 21) [2], Kirkland's die was not suitable for our purpose. We therefore constructed a die to press 2.5 × 0.3 mm^2 pellets in simple plate disks. Semi-micro and micro dies pressing pellets in plate disks have been described [3] and are also commercially available, but these make cylindrical pellets, which obviously means either a waste of substance or a pellet much too small to fill the image of the monochromator's entrance slit. Manufacturing difficulties seem to have prevented the constructions of semi-micro dies for oblong pellets.

Fig. 1 shows our die. The plunger slides in a guide made in two parts forming a cylinder, with the plane of intersection longitudinal but slightly off axis. Into the plane intersection surface of the larger part is milled a space for the plunger and pusher.

Straight through the guide an evacuating hole is bored (across the plunger-space), terminating at each end in a circular groove around the guide. The heavy-duty contact-surface between plunger and pusher is cylindrical. This is to avoid any tilting moment on the plunger.

The power is transmitted to the pusher by a cylindrical piston. Two O-rings ensure tightness, and their compressibility allows a sufficient stroke for the piston.

The disk is punched from an 0.3 mm stainless steel plate. It is kept in position in the die by 3 pins protruding from the guide. One of the pins is slimmer than the

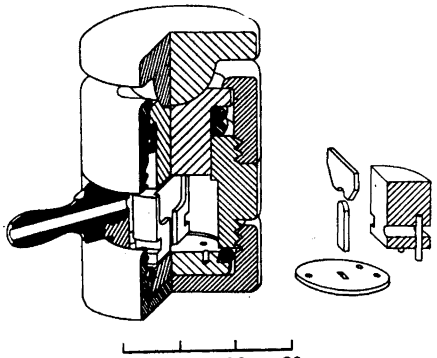

Fig. 1. Die in section. Disk, plunger, pusher and one of the guide halves (in section) lifted out and shown beside the die.

others and does not fit in its hole in the disk, it serves only to prevent the disk being turned wrongly. A jig is used when the pin-holes are bored in the disk.

Beneath the disk is a polished bottom-plate kept firmly in position by a screw cap and sealed by an O-ring.

The guide should be made of stainless steel and the plunger, pusher and bottom-plate of hardened alloyed steel.

It proved difficult to make the oblong pellet-hole in the right place in the disk. We finally solved the problem by using the die as a punching tool. Before the punching, the plunger is exchanged for a punch and the bottom-plate for a bolster made in two halves in the same way as the guide.

To make a pellet one turns the die upside down, removes the cap and bottom-plate and pushes down the plunger with a clean plate-strip. Then a mixture of about 35 μg of substance and 1 mg of KBr is filled into the plunger-space. (The operator should wear a mask while handling the tiny amount of powder, to protect it against his breath). The disk, bottom-plate and cap are placed in position (cap tightened firmly) and the pellet can be pressed in the usual way. The force on the piston should be 820 newtons.

The best results are obtained with 30–40 μg of substance, but even less will do (if some loss of detail may be tolerated), as shown in Fig. 2.

To make the pellet 20 μg of 2DL-methyltetracosanoic acid and 1 mg of KBr were

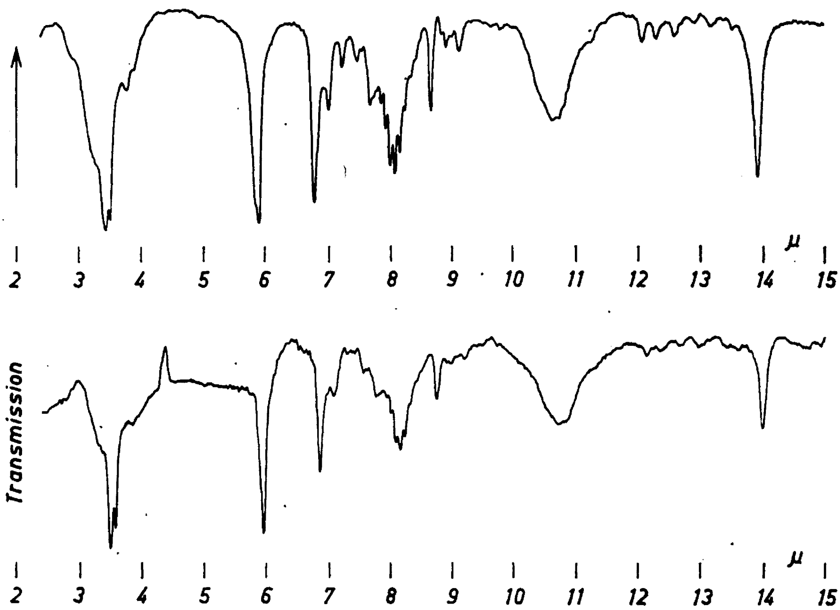

Fig. 2. Spectra of 2 DL-methyltetracosanoic acid. The macro spectrum (top) was made from a circular pellet measuring 13 mm in diameter and containing 3 mg of substance in 300 mg of KBr. The semi-micro spectrum (bottom) was made from a 2.5 × 0.3 mm² pellet containing 20 μg of substance in 1 mg of KBr. Spectra scanned at 4 and 6 min/micron, respectively. Slit program was 927 for both.

weighed out and mixed thoroughly with a small spatula (not ground in a mortar, as this would entail considerable loss of material). The slow scanning (6 min/micron) is due not so much to the sample as to the microscope, which transmits only a fraction of the available energy, most of it being masked off by the Schwarzschild condenser. (The construction with elliptic mirrors developed simultaneously by Perkin-Elmer is much better in this respect. But unfortunately it is not a twin device and cannot be used for the comparison of two samples.)

The oblong pellet transmits as much energy as a circular pellet of 6.5 times its area. This of course applies also to a macro pellet, provided it can be brought sufficiently close to focus. The construction described here seems also to be well suited for macro dies.

In a future version of the die we are planning to make the guide conical instead of cylindrical, so that it can be easily lifted out and taken apart for cleaning.

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