

Figure 2 C.O.P. of albumin solutions obtained with the concentrator (Δ - Δ), Prather *et al.* osmometer (\blacktriangle - \blacktriangle) and by calculation (\bullet - \bullet). For clarity S.D. is not shown ($n = 6$ for each set of measurements).

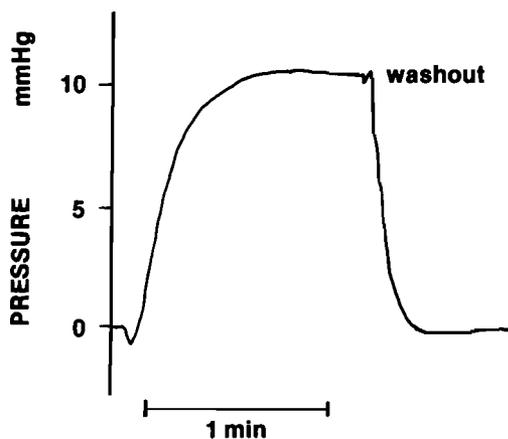


Figure 3 C.O.P. of an albumin solution measured with the concentrator showing response time.

tended to the measurement of C.O.P. of plasma, lymph and tissue fluid samples from small laboratory animals.

The concentrator also manifests potential in being adapted as an implantable osmometer such as described by Reed (1979). The polyethylene core would resist any deformatory forces applied on the membrane, ensuring that an equilibrium is obtained which would accurately reflect tissue C.O.P. The only possible disadvantage at present is that care must be exercised not to allow air into the system (through the membrane) and the concentrator should thus always be stored in saline.

It is therefore apparent that the concentrator provides a means for rapid, accurate estimation of C.O.P. On a comparative basis much remains to be learnt concerning this important variable, not only from plasma but also other body fluids. The methodology reported here, makes this possible.

Acknowledgement

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A pellet mould for small powdery specimens for X-ray microanalysis in the SEM

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This paper describes a simple mould to press small powdered samples into pellets for X-ray microanalysis. The pellet can be pressed on the sample holder (stub), but in some cases it has to be glued to the sample holder or stub. The drawings indicate how the mould is made (Figure 1).

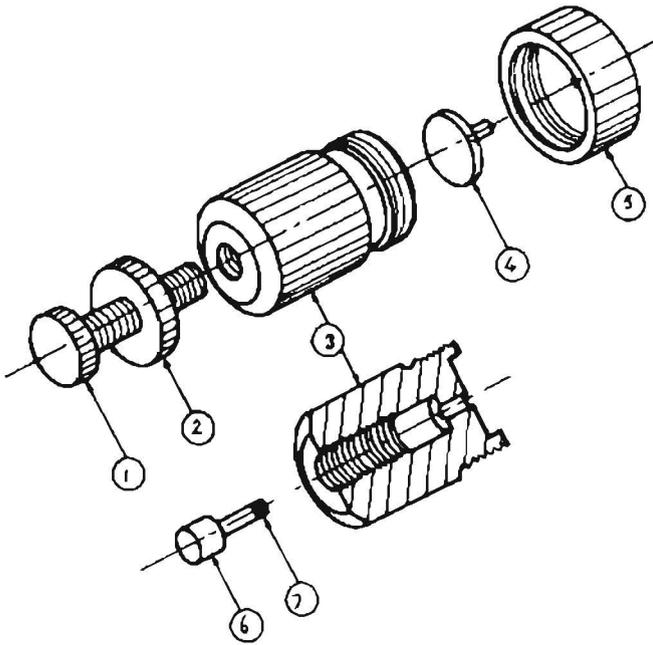


Figure 1 An isometric and exploded view of the mould with plunger (6,7), screw and locknut (1,2), body (3), stub (4) and stub retainer (5). Diameter of body 20 mm, length of body 27 mm.

As we did not have the convenience of a laboratory press and due to the problems we encountered when mounting small samples of various powders which can include incinerated biological material to chemical deposits for semi- or quantitative microanalysis, we devised a small mould-cum-press that would take the place of the laboratory press and which could handle the small powder samples.

This mould can be manufactured from materials available in most machine workshops. To prevent any introduction of foreign material contaminating the sample especially with metals from the mould, we decided to manufacture the mould from Wesconite and Teflon. The plunger (Figure 1.6) is made of stainless steel capped with Wesconite (Figure 1.7). In this way no metals are in contact with the sample. This is an improvement on the mould described by Bistricki (1980).

The diameter of the plunger (Figure 1.6) is 2,5 mm with a contact area of 5 mm² and a close fit in the tube (Figure 1.3). The retainer of the stub (Figure 1.5) is threaded to screw on the body of the mould (Figure 1.3). At the rear end of the body of the mould a threaded bolt (Figure 1.1) screws into the body to exercise a pressure on the plunger and to compress the powder in a pellet onto the specimen stub. The threaded bolt (Figure 1.1) and the holder for the stub (Figure 1.5) can either be machined from stainless steel or from Wesconite and Teflon as is the body of the mould.

The pressure exerted on the powder is of the order of approximately 30N m⁻² and can be maintained for as long as necessary provided that the locknut (Figure 1.2) is tightened to prevent the bolt slipping. Wesconite screws are known to slip as time goes by, and therefore a locknut is essential.

In preparation of a pellet, double-sided sticky tape (Bistricki 1980), can be stuck to the stub, but experience has shown that the pellet is so small and that the adhesion

to the plunger, so large, that the pellet stays stuck to the plunger. The amount of powder depends on the type of powder, but experience has shown that 10 mg is sufficient to fill the plunger tube. The pellet can be removed without any problems by inserting a fine scalpel between the tip of the plunger and the pellet. To prevent any contamination with a foreign material, a very thin Wesconite blade, similar to a scalpel, can be manufactured and used instead of a metal one. This operation has to be conducted underneath a stereo dissecting microscope.

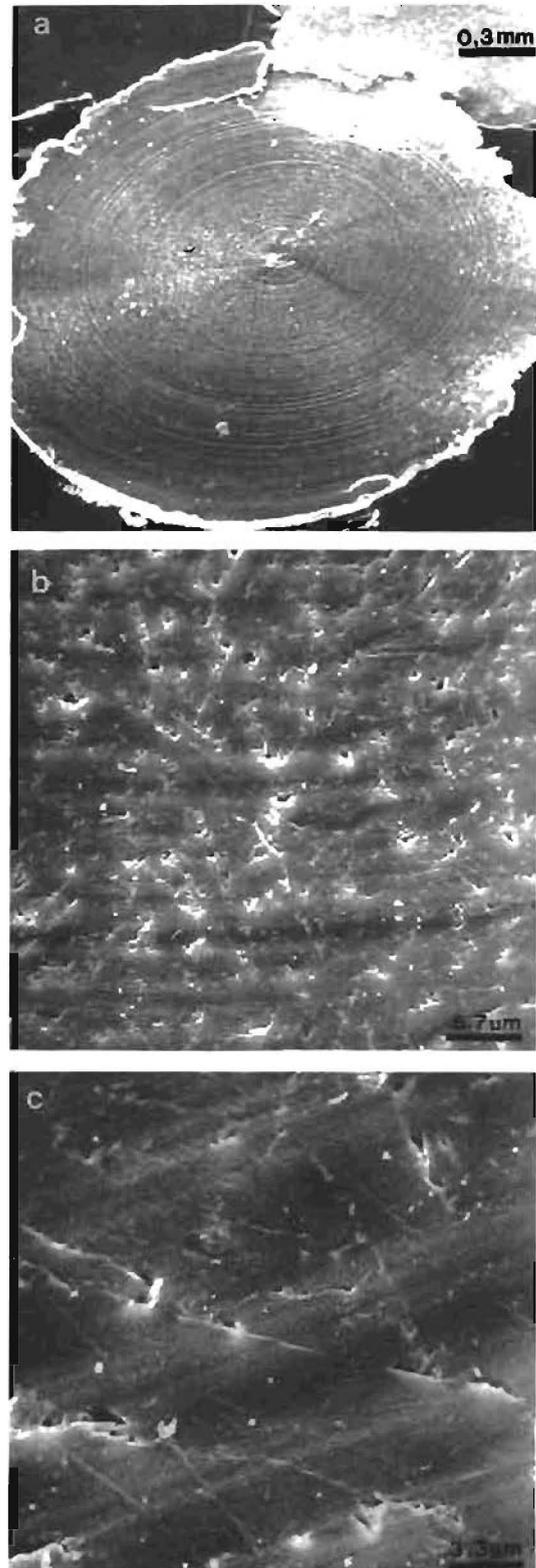


Figure 2 A pellet photographed at different magnifications to illustrate shape and surface features.

The small size (Figure 2a) (area 5 mm²) of the pellet proves to be an advantage in X-ray microanalysis as the whole sample can be covered during analysis thus minimizing the possibility of analysing non-homogeneous areas and allowing very small samples to be handled. Although the morphology of the particles is lost, this method is used to analyse the powder and not to study the morphology of the particles. When used in the correct perspective the advantages and speed of this method are apparent.

As depicted in Figure 2b and c the surface of the pellet is fairly flat, and polished to such an extent that it is possible to do semi-quantitative analysis of a small area or an analysis of the entire pellet, without reorientation of the pellet or specimen stub. The flat polished surface can be attributed to the rubbery properties of the Wesconite. By polishing the tip of the plunger and the stub to a higher sheen, the sample can be polished to such an extent that quantitative analysis is possible. A double-sided polished pellet can be obtained by using a very smooth Wesconite stub in combination with a Wesconite plunger.

When a powder is very coarse and difficult to compound Bistricki (1980) advises impregnating the powder with Spurr embedding medium and then polishing the pellet to remove excess resin. An easier method is to use the cap of a Beem capsule as a mould in which the powder and Spurr embedding medium are poured and polymerized with heat at 100 °C for 24 h. After polymerization the surface can be polished to facilitate quantitative analysis.

This pellet mould is of great help in X-ray microanalysis of large numbers of minute samples of fine powders.

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References

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