NEW APPARATUS

AN APPARATUS FOR SEMI-MICRO CRYSTALLISATION

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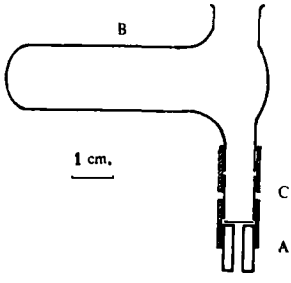
Repeated recrystallisation of quantities of material of the order of 20 to 40 mg. often presents a difficult problem. Application of the usual micro-methods or of the conventional macro-methods, using small apparatus, frequently results in disappointing losses of material owing to premature crystallisation during filtration or to repeated transferences of small quantities of solutions or solids from vessel to vessel. We have found that an apparatus, consisting essentially of a crystallisation vessel attached to a form of the Schwinger filter, is very convenient for repeated and rapid crystallisations. The principle of the apparatus is somewhat similar to that of the Bergkampf[1] filter-beaker.



The apparatus, having the dimensions shown in the figure, is convenient for crystallisations from about 0·4 to 4 ml. of solvent. The surfaces, which meet inside the rubber sleeves, are ground flat. Cell C is tared together with the filtering unit A, and the disc of hardened filter paper secured by the lower rubber sleeve. The crude reaction product is collected, washed and dried in cell C, slight positive pressure being applied to B for rapid filtration. After weighing the product, the cell C and filtering unit are again attached to the crystallising vessel B and the thick-walled capillary tube is replaced by a plug of glass rod of the same dimensions. Most of the solid is tapped down into B and dissolved in hot solvent; by tilting the apparatus, the hot solution is run to and fro into C to dissolve solid adhering to the sides. Cell C is then removed and the filtering unit is attached directly to the crystallising vessel. The solution is reheated for a short time to warm the whole apparatus and then filtered, under slight positive pressure, into a micro-beaker by turning the apparatus through 90°. If crystallisation occurs during filtration, the crystals are easily dissolved in more hot solvent run to and fro from B and then filtered into the bulk of the solution.

The crystals which separate are collected in the same manner as the crude reaction product. Since filtration of the hot solution in further crystallisations is not normally necessary, the crystals are transferred to B as before and dissolved; crystallisation is then allowed to take place in B. The crystals are collected again in cell C. If some crystals adhere to the sides of B, they are redissolved in some of the mother liquor, allowed to crystallise and collected with the main bulk in cell C. The crystals are then washed and dried.

The process can be repeated as often as necessary. After the first hot filtration, there are no losses owing to transference from vessel to vessel, and the material is accessible for weighing and melting-point determination between each recrystallisation.

In the first two trials with this apparatus, hyoscyamine picrate, obtained

from hyoscyamine sulphate solution produced in a partition chromatogram of *Atropa Belladonna*[2] was used. The results of crystallising two samples of the picrate from aqueous alcohol are shown in the Table. We have since obtained equally satisfactory results in many other crystallisations.

Recrystallisations	Weight recovered mg.		Melting-point °C. (uncorrected)	
	(a)	(b)	(a)	(b)
Crude precipitate	21	40	162—3	162—3
First crystallisation with filtration of hot solution ...	14·5	34	164—5	164—5
Second crystallisation without filtration of hot solution	11·5	32	164—5	164—5
Third crystallisation without filtration of hot solution ...	10·5	30	164—5	164—5
Fourth crystallisation without filtration of hot solution	9·5	27	164—5	164—5

REFERENCES.

1. von Bergkampf, *Z. anal. Chem.*, 1926, **69**, 321.
2. Evans and Partridge, *Quart. J. Pharm. Pharmacol.*, 1948, **21**, 126.

NEW REMEDIES (*continued from page 350*)

is less irritating; its low viscosity also makes it easier to handle, and a comparatively small gauge needle may be used. Ethiodan is specifically indicated for use in the radiological diagnosis and localisation of cord tumours, herniated nucleus pulposus, intraspinal protrusion of intervertebral discs, and any other conditions in which obstructions in the cerebrospinal canal or compression of the cord are suspected. Normally, 3 ml. is injected immediately below the level at which the obstruction is suspected. It is issued in boxes of 3 ampoules each containing 3 ml. S. L. W.

Ferosan Tablets* contain exsiccated ferrous sulphate 3 gr., copper sulphate 1/25 gr., and manganese sulphate 1/25 gr. Their use is indicated in all cases of hypochromic microcytic anæmia, including anæmia due to chronic or acute hæmorrhage, idiopathic hypochromic anæmia and anæmia of pregnancy or lactation. The adult dosage is 1 or 2 tablets 3 times daily after meals. Ferosan tablets are supplied in bottles of 100 tablets. S. L. W.

Priscol* is the hydrochloride of 2-benzyl-4:5-imidazoline; in colourless crystals, freely soluble in water; m.pt. 171°C. Its principal action is to dilate the peripheral vessels. This effect is primarily on the arterioles and the smaller arteries, and its use is therefore followed by hyperæmia and acceleration of the blood flow in the capillaries. The improved circulation is usually accompanied by a fall in blood pressure. It is indicated particularly for the treatment of peripheral vascular disorders, by intravenous, intramuscular or intra-arterial injection; arthritic conditions are treated by peri-articular injections or the local use of an ointment. It may also be employed as a local application, combined with parenteral or oral therapy, for the treatment of slow-healing wounds and ulcers. It is claimed to be especially valuable in ophthalmic conditions where active hyperæmia is desired; for this purpose, it is employed either in the form of drops of a 10 per cent. solution or by subconjunctival injection. Priscol is supplied in bottles of 40 or 200 tablets containing 25 mg., in boxes of 10 ampoules containing 1 ml. (10 mg.), in bottles of 10 ml. of 10 per cent. solution, and in tubes containing 20 g. of 10 per cent. ointment. S. L. W.