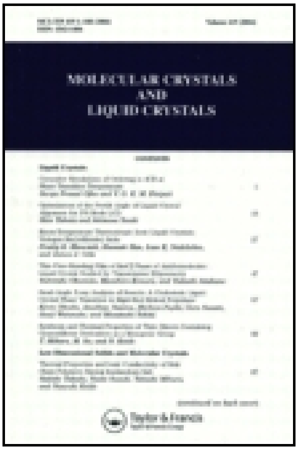

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An All Glass Apparatus for "Continuous" Chromatography and its Application to Chrysene

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A technique for the 'continuous' chromatography of large batches, greater than 10-20 g, of organic scintillators for removal of strongly adsorbed impurities was extensively used in 1956 by Sangster and Irvine[1] and is shown in Fig. 1(a). The solvent percolates through the batch of crude material extracting the most soluble fraction and carrying it onto the adsorption chromatography column. The highly adsorbed impurities are retained on the column, usually highly polar impurities, and the purified solution enters the lower reservoir. A heating mantle is then used to boil up the solvent which recondenses and falls on the crude material again, thus making a continuous solvent cycle. The process is continued until the impurities, observed either by their color or fluorescence, approach the lower reservoir at which point the elution can be temporarily stopped and a fresh charge of adsorbent introduced.

This arrangement suffers from the disadvantage that it is difficult to control the boil up rate over long periods of time. Some workers have circumvented this problem by using a constant leveling device, e.g. a photoelectric cell[2] which is operated by an opaque float in the liquid. This controls the heater on-off switch and maintains the solvent at a predetermined level in the upper reservoir.

One potential danger exists with this mechanism – that of an explosion where a volatile flammable solvent is used in conjunction with poor ventilation. The following simple all glass design obviates this danger and also the need for photoelectric switching.

The apparatus is shown in Fig. 1(b). The only requirement for successful operation is that a larger boil up rate be used than the minimum needed for

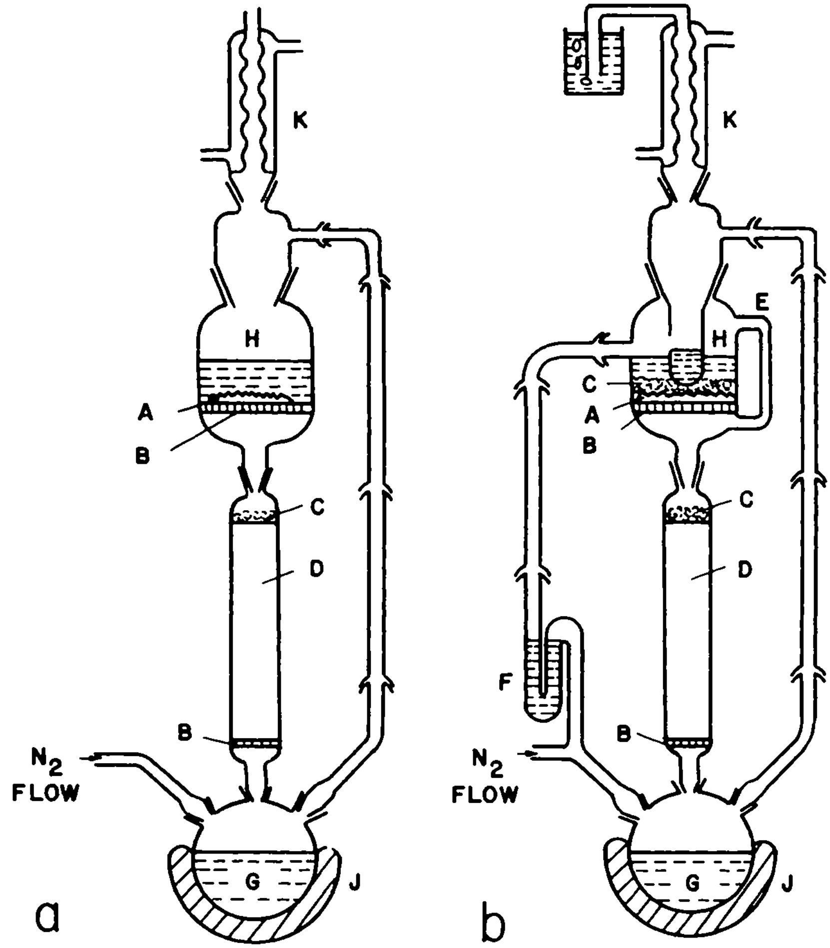

FIGURE 1 (a) Original Sangster and Irvine apparatus. (b) Modified Sangster and Irvine apparatus

A – crude material; B – coarse frit; C – glass wool; D – adsorbent; E – pressure relief line; F – liquid trap; G – lower reservoir; H – upper reservoir; J – heating mantle; K – condenser.

equilibrium under any ambient temperature fluctuations. Excess solvent overflows and returns to the bottom reservoir through a liquid trap which prevents nitrogen flow or boil up through this return line. The crude material is supported on a coarse frit and is covered with a layer of glass wool which prevents mixing of the solute in the upper reservoir with the possibility of overflow down the return line to contaminate the bottom reservoir. The design of the condensate return is such that mixing in the upper reservoir, due to falling droplets, is

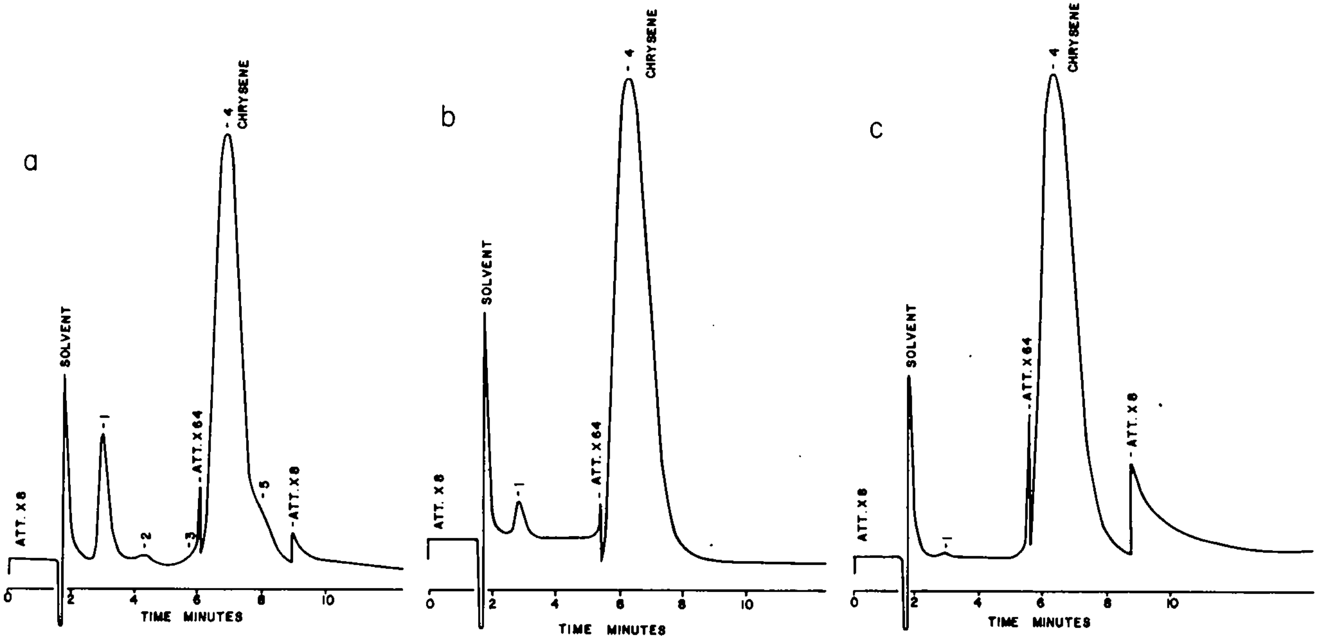

FIGURE 2 Liquid Chromatograms of Chrysene (a) as received, Aldrich Puriss; (b) after sublimation and zone melting; (c) after chromatography.

Chromatographic operating conditions:
Column = "Permaphase" ODS (octadecylsilane), lm x 2. lmm I.D.
Solvent = 1.4 Dioxane
Sample = 5μℓ
Mobile Phase = 50% 1.4 Dioxane/50% Water (V/V)
Column Temperature = 25°C
Flow Rate = 0.8 ml/min
Detector = UV photometer at 254 nm

minimized. A pressure relief line is also included to prevent pressure build-up above the adsorbent with subsequent blockage of the solvent flow through the column.

This apparatus has been succesfully used in the purification of chrysene. A sample of chrysene, 50 g (Aldrich, Puriss.), was initially purified by sublimation and zone melting, 100 zone passes on a Sloan-McGowan zone melter with ℓ:L = 1:20 and zoning speed of 1 mm./h. G.C. analysis showed no detectable impurity, but analytical liquid chromatography showed a persistent polar impurity which was slowly removed. After zone melting, this impurity concentration was reduced by only a factor of 6 in the purest section of the charge. The zone melted material, 16.5 g, was placed in the upper reservoir and chromatographed through a 50 cm × 50 mm O.D. column charged with 750 g of Woelm aluminium oxide neutral, activity grade 1, using 2 liters of benzene (Fisher, A.C.S. certified) as eluting solvent.[3] After 21h, the charge had totally disappeared and a 4 cm band of blue fluorescent impurity was strongly retained at the top of the column with no chrysene fluorescence observed on the remainder of the column. The solution in the lower reservoir was allowed to cool and the first crop of chrysene crystals were filtered. The solvent was then evaporated to half volume and a second crop obtained. The total yield from both crops was 13.2 g. Fig. 2 shows the increase in purity obtained by using both techniques, zone melting and chromatography, as determined by high speed analytical liquid chromatography (DuPont LC model 830). The polar impurity concentration was reduced to an almost undetectable level.

This apparatus is easily constructed, relatively inexpensive, and should be a useful addition to any laboratory where large scale chromatographic purification (greater than 10 g) is a recurring problem.

Acknowledgements

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References

1. Sangster, R. C. and Irvine, J. W., *J. Chem. Phys.* 24, 670 (1956)
2. Sherwood, J.N., in *Purification of Inorganic and Organic Materials*, Ed., Zief, M., Marcel Dekker, New York, 1969 p 158.
3. Peter, L., Engel, W. and Vaubel, G., *Mol. Cryst. and Liq. Cryst.* 19, 207 (1973).