For the best results it is advisable in this case, as also in the separation of aluminium, gallium and indium, to maintain the relative humidity in the chamber at 65 per cent. Nickel, cobalt and copper are identified by means of rubeanic acid; manganese by reduction of ammoniacal silver solution; and iron by potassium ferrocyanide. Nickel and cobalt have been determined quantitatively by polarographic measurements. Some typical results are illustrated in the accompanying reproductions.

Limits of detection and estimation. The procedure provides a delicate means of separating and estimating small quantities of metallic ions. The limits of detection vary with different elements and depend on the sharpness of the bands and the sensitivity of the spot tests. Quantities of the order of 1·0–0·1 microgram have been detected, while the normal range for quantitative work is in the region of 5–200 micrograms.

Mechanism. The processes involved in this type of separation are being investigated; but it is clear that a number of factors must be taken into consideration. These are: (*a*) selective extraction of the salts by the organic solvent, an effect prevailing at the site of the test patch; (*b*) where substantially immiscible solvents are used, partition of the inorganic substances between organic solvent and aqueous layer, since water is present as a normal constituent in the absorbent paper and is usually added to the organic solvent; this partition, therefore, takes place as the liquid mixture moves down the paper, and separations are due to slight changes in conditions at different points on the absorbent strip; (*c*) adsorption of metallic ions by the paper; (*d*) the formation of complexes with high solubility in organic media under specific conditions.

In view of the uncertainty as to mechanism, it seems to us best for the present to refer to this method non-committally as 'extraction analysis'. Further study will, it is hoped, provide a logical theoretical background for the processes taking place.

The investigations so far carried out indicate that the broad application of this and closely related techniques should make possible the separation, detection and estimation of a very wide range of inorganic products. The method is simple and fairly rapid, requires only very small quantities of material, operates with complicated mixtures of ions and does not demand any specialized equipment, at least for qualitative examination. The work is being actively pursued at this Laboratory.

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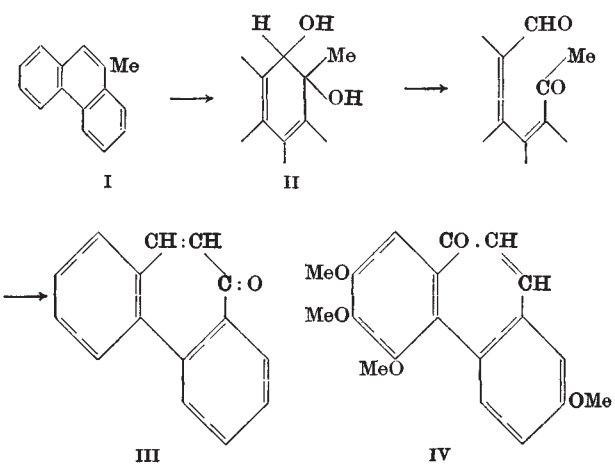
Synthesis of Colchicine Derivatives

While the methoxylated carbon-system of (IV) is conclusively established for certain degradation products of colchicine[1,2], the synthesis of such unsymmetrically substituted dibenz*cyclo*heptatrienes, necessary for further work, has hitherto miscarried, because of difficulties which are partly concealed and partly apparent[2–4]. A synthesis which promises to afford the requisite degree of adaptability has now been found and is based on the oxidation of appropriate 9- or 10-methylphenanthrenes with osmium tetroxide in benzene-pyridine[5,6]. In a model experiment, 9-methylphenanthrene (I) was thereby oxidized to 9 : 10-dihydroxy-9 : 10-dihydro-9-methylphenanthrene (II), m.p. 130–131° (found: C, 79·9; H, 6·1; $C_{15}H_{14}O_2$ requires C, 79·7; H, 6·2 per cent), from which, by cleavage with lead tetra-acetate and renewed cyclization, there was obtained the known 3 : 4 : 5 : 6-dibenz*cyclo*hepta-1 : 3 : 5-trien-7-one (III)[2]. Similarly, 2 : 3 : 4 : 7-tetramethoxy-10-methylphenanthrene, m.p. 134–135° (found: C, 73·2; H, 6·4; $C_{19}H_{20}O_4$ requires C, 73·1; H, 6·4 per cent), was converted *via* the corresponding 9 : 10-dihydro-9 : 10-diol, m.p. 156° (found: C, 66·0; H, 6·4; $C_{19}H_{22}O_6$ requires C, 65·9; H, 6·4 per cent) into the unsaturated ketone (IV), which was identical with the part-product previously obtained[1] by oxidizing deaminocolchinol methyl ether with sodium dichromate in acetic acid.



2 : 3 : 4 : 5- and 2 : 3 : 4 : 7-Tetramethoxy-9-methylphenanthrenes[7] have given apparently analogous results, and full experimental details will be published when the developments now in hand, which include the attempted synthesis of colchinol methyl ether, have been completed. One of us (J. M.) thanks the Department of Scientific and Industrial Research for a maintenance allowance.

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