TABLE I

Complexometric determination of M^{2+}, Ca^{2+}

Sl. No.	M^{2+} (mg)			Calcium (mg)		
	Theo.	Expt.	% Error	Theo.	Expt.	% Error
1	112·40	112·00	0·35	120·24	120·24	0·00
2	61·20	60·90	0·45	60·12	60·00	0·20
3	207·20	207·10	0·04	80·16	80·08	0·10
4	103·60	103·50	0·09	40·08	40·08	0·00
5	65·37	65·37	0·00	60·12	60·12	0·00
6	49·03	49·03	0·00	40·08	40·08	0·00
7	111·70	111·35	0·30	80·16	80·04	0·15
8	55·85	55·60	0·44	60·12	60·12	0·00
9	200·59	199·59	0·50	120·24	120·24	0·00
10	100·03	100·03	0·00	60·12	60·12	0·00

(Where M^{2+} for serial numbers 1 and 2 is Cd, 3 and 4 is Pb, 5 and 6 is Zn, 7 and 8 is Fe, 9 and 10 is Hg).

is changed from wine red to blue. The volume of EDTA solution consumed in the titration corresponds to the total content of Ca^{2+} and Pb^{2+} (or Cd^{2+} or Zn^{2+}) in the filtrate taken. To the same volume of another aliquot of the filter 4 ml of buffer of pH 10 and 1·0 g of solid NH_4F were added and titrated against 0·01 M standard EDTA solution using Eriochrome Black T as indicator till the colour was changed from wine red to blue. The volume of EDTA consumed in this titration corresponds to the content of lead (or Cd^{2+} or Zn^{2+}) alone. The difference between the titre values of the above experiments corresponds to the content of calcium in the filtrate taken. In the determination of a mixture of Calcium and Iron (or Mercury) to a convenient volume of the filtrate a known excess of standard 0·01 M EDTA was added and then back titrated with 0·01 M $MgSO_4$ till the colour changed from blue to wine red. The volume of $MgSO_4$ consumed in the titration corresponds to the total content of Calcium and Iron (or Mercury) in the filtrate taken. Calcium content in the same volume of another aliquot of the filtrate was masked as described earlier, and a known excess of 0·01 M EDTA was added and then back titrated with 0·01 M $MgSO_4$ till wine red colour was obtained. From the volume of $MgSO_4$ consumed in this titration the content of Iron (or Mercury) in the filtrate was calculated. The difference between the titre values of the above experiments

corresponds to the content of calcium in the filtrate. Representative sets of the results are given in Table I. The method suggested was found to be very rapid and accurate.

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SEPARATION OF IRON (III), COBALT (II) AND NICKEL (II) BY PAPER CHROMATOGRAPHY

IRON (III), cobalt (II) and nickel (II) have been separated by paper chromatography using several solvent mixtures by various workers[1-7]. We have studied the separation and determination of Fe (III), Co (II) and Ni (II) by ascending paper chromatography at various conditions using 85·0% acetone, 5·0% con. HCl and 10·0% methyl isobutyl ketone as solvent system. The minimum time required for the separation is 15 minutes, whereas the methods recommended so far require minimum of 90 minutes for the separation[1-3].

All the chemicals used were of A.R. quality. One ml of each of the solution (1·0 M) of Fe (III), Co (II) and Ni (II) as sulphates was mixed together in a beaker and 5 μl of this mixture was applied to the strips of Whatman paper No. 1 (30 × 30 cm) at the base line and the paper strips were allowed to dry. The paper strips were then kept in the developing solvent, taken in a glass jar which was previously saturated with the vapours of the solvent and chromatogram was run for various intervals. The positions of these ions on strips were located with the aid of 0·1% alcoholic solution of rubeanic acid followed by exposing the strips to ammonia vapours. The R_f values of these ions have been calculated under various conditions.

After trying various solvents, it has been observed that reasonably good separation can be achieved within 15–30 minutes with a solvent mixture of 85·0% acetone, 5·0% con. HCl and 10·0% methyl isobutyl ketone although the best separation is possible within 45·90 minutes (Table I).

TABLE I

Effect of time on separation of Fe(III), Co(II) and Ni(II)

Solvent composition : 85 ml acetone + 5 ml HCl (32%) + 10 ml methyl isobutyl ketone

Time min.	R_f Values			Separation
	Fe (III)	Co (II)	Ni (II)	
15	1·0	0·85	0·17	Good
30	1·0	0·81	0·15	Better
45	1·0	0·67	0·13	Best
60 to 90	1·0	0·64	0·08	do.

The presence of impurities like Zn (II) and Al (III) interferes with the separation of Fe (III), Co (II) and Ni (II), since they move with Fe (III). However, the presence of Cu (II) does not interfere. From the foregoing account it can be concluded that this method which involves methyl isobutyl ketone as one of the components of the solvent system renders quick and satisfactory separation of these three ions.

These three ions have been determined in micro quantities after their separation on paper strips using solvent extraction and colorimetric techniques. The experimental values in micro grams for Ni (II), Fe (III) and Co (II) were found to be 45·2, 18·4 and 44·4 against theoretical values, 48·7, 20·7, and 48·1 respectively.

The authors are highly grateful to Prof. R. H. Sahasrabudhey for providing the facilities.

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A SIMPLE POTENTIOMETRIC METHOD FOR THE ESTIMATION OF BHC

GENERALLY, for the estimation of lower concentration of BHC (1, 2, 3, 4, 5, 6-Hexachlorocyclohexane, commonly called benzenehexachloride), a widely used insecticide, gas chromatographic[1] or colorimetric[2] methods are commonly employed. But most of the laboratories do not have the costly gas chromatograph equipped with either an electron capture detector or a microcoulometric detector. The other methods, usually employed for the estimation of this compound in its technical and commercial formulations[2,3,4], are not very sensitive to low concentrations as that found in its aqueous solutions. As such, at present it is difficult to estimate traces of BHC in solutions. The potentiometric method described below is sensitive, simple and can be adopted even for residue analysis. The method is based on the hydrolysis of BHC under alkaline condition, to trichlorobenzene and inorganic chloride. The inorganic chloride thus released is estimated using a chloride ion-specific electrode. The concentration of chloride is taken as an index of the concentration of BHC. The procedure for the new method is given below.

Partition the BHC to hexane phase from a known volume of its aqueous solution, by repeatedly extracting its aqueous solution with 10 ml portions of *n*-hexane. Similarly, the hexane extracts of any other substance containing BHC can be used for analysis. The combined hexane extracts are transferred to a 50 ml test tube and the contents are evaporated to dryness on a water-bath at 60° C. Moisten the residue in the tube with 2 ml of distilled water, treat with 2 ml of 0·5 N ethanolic potash and keep the tube on a water-bath at 55°–60° C, till the contents are almost evaporated. Cool and extract the residue with 2 ml portions of water, finally making the extract to 10 ml. Mix well and determine the concentration of chlorides in the solution using a chloride ion-specific electrode (Beckman Silver-Silver chloride electrode or any other make similar electrode). The electrode potential is measured on a potentiometer (pH meter) having an expanded scale (Corning model No. 12 pH meter or its equivalent). Determine the concentration of BHC in the sample by referring to a calibration curve previously prepared by adopting the same procedure with 1 to 10 mg of BHC.

This method has been found quite satisfactory for solutions containing more than 0·1 ppm of BHC. Although the aqueous solubility of this compound is small, its concentration in water solutions can be estimated even in such small quantities, by partitioning the compound from a large volume of the solution to hexane phase, and then further con-