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Extraction of Scandium(III) with Tri-*n*-butyl Phosphate from Aqueous Chloride Solutions

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The distribution of scandium(III) between tri-*n*-butyl phosphate and aqueous solutions containing chloride has been measured using ^{46}Sc as a tracer. The extraction of anionic scandium chlorocomplexes seems to be unimportant in the systems studied and it appears that different solvates of the trichloride $ScCl_3$ can exist in tributyl phosphate extracts.

The recent paper by Sekine, Hamada and Sakairi¹⁾ has prompted us to report the results of a study of the distribution of scandium(III) between tri-*n*-butyl phosphate (TBP) and aqueous solutions containing chloride.

Experimental

Materials. Scandium-46 (half-life 84 days) of high specific activity was supplied as the chloride in aqueous solution by the Radiochemical Centre, Amersham. TBP was purified by the method of Alcock *et al.*²⁾ and was dried by warming under reduced pressure. Benzene used as a diluent in some experiments was material "for molecular weight determinations" from British Drug Houses Ltd. Hydrochloric acid was of "AnalaR" quality and lithium chloride was purified as described previously.³⁾

General Procedure. Distribution was obtained by stirring together mechanically 5 m*l* of TBP phase and 5 m*l* of aqueous phase containing ^{46}Sc as tracer. Agitation for 30 min always sufficed for equilibration and all exp eriments were performed at 20±1°C. After centrifuging, the phases were separated by means of transfer pipettes for analysis. Aliquot parts were counted in polythene counting cups under identical conditions with NaI(Tl) - scintillation counter. Values of the distribution ratio for scandium(III) were obtained as:

$$D=\frac{\text{Count rate per m}l\text{ of org. phase}}{\text{Count rate per m}l\text{ of aq. phase}},$$

the observed count rates having been corrected for background.

For each distribution experiment the concentration of chloride $[Cl^-]$, M, in the aqueous phase after equilibration was determined by titration.

No adsorption of radioscandium was detected on the walls of vessels used for the equilibrations.

Results and Discussion

Figure 1 shows plots of D against $[Cl^-]$, for (a) distribution between TBP and hydrochloric acid and (b) distribution between TBP and aqueous solutions 0.1 M in HCl containing lithium chloride. Extraction is greater from concentrated lithium chloride than from concentrated hydrochloric acid, and in this respect the behaviour of scandium follows that of other metals.⁵⁾ Values for D were

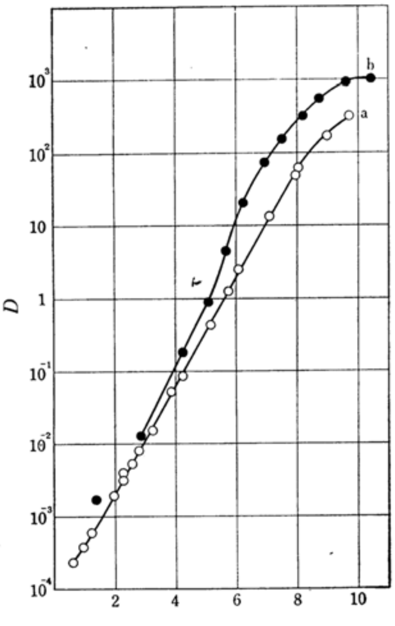

Fig. 1. Relationship between D and the chloride concentration of the equilibrium aqueous phase. Extraction by TBP from (a) HCl (b) from aqueous LiCl 0.1 M in HCl. The curve (a) matches the corresponding plot of results due to Peppard, Mason and Maier.⁴⁾

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1) T. Sekine, T. Hamada and M. Sakairi, This Bulletin, **39**, 244 (1966).

2) K. Alcock, S. S. Grimley, T. V. Healy, J. Kennedy and H. A. C. McKay, *Trans. Faraday Soc.*, **52**, 39 (1956).

3) D. F. C. Morris and E. L. Short, *J. Inorg. Nucl. Chem.*, **25**, 291 (1963).

4) D. F. Peppard, G. W. Mason and J. L. Maier, *ibid.*, **3**, 215 (1956).

5) D. F. C. Morris, E. L. Short and D. N. Slater, *Electrochim. Acta*, **8**, 289 (1963); D. F. C. Morris and M. W. Jones, *J. Inorg. Nucl. Chem.*, **27**, 2454 (1965).

found to be independent of the initial aqueous concentration of scandium in the range 2×10^{-5} to 1×10^{-4} M, suggesting that scandium(III) complexes in both organic and aqueous phases are mononuclear.

Measurements analogous to those effected with TBP were made of the distribution of tracer scandium(III) between 0.1 M tri-*n*-octylamine hydrochloride (TOA) in benzene and (i) hydrochloric acid and (ii) aqueous solutions 0.1 M in HCl containing lithium chloride. In these cases extraction of scandium was very slight (max. D 8×10^{-3} for distribution between 0.1 M TOA and 0.1 M HCl - 12 M LiCl). This suggests that formation of anionic chlorocomplexes of scandium(III) in the systems is quite unimportant.

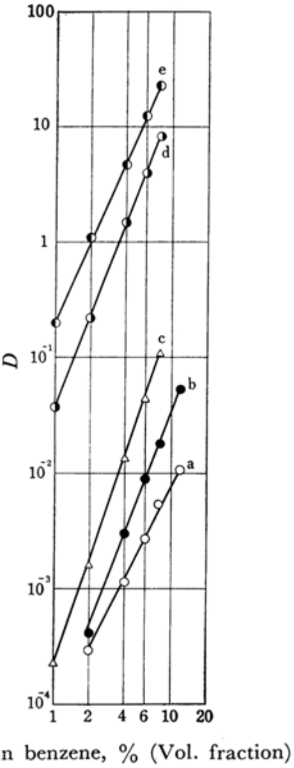

Fig. 2. Distribution of scandium(III) as a function of TBP concentration for solutions of hydrochloric acid of fixed concentration as the aqueous phase. (a) $[Cl^-]=5.77$ M, slope 2.0; (b) $[Cl^-]=6.39$ M, slope 2.6; (c) $[Cl^-]=6.93$ M, slope 3.0; (d) $[Cl^-]=9.10$ M, slope 2.6; (e) $[Cl^-]=10.77$ M, slope 2.3.

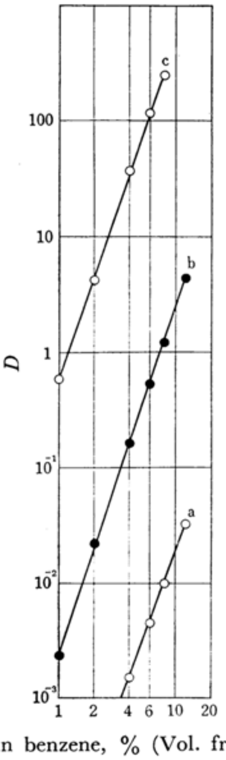

Fig. 3. Distribution of scandium(III) as a function of TBP concentration for solutions of lithium chloride (0.1 M in HCl) of fixed concentration as the aqueous phase. (a) $[Cl^-]=6.66$ M, slope 2.8; (b) $[Cl^-]=8.59$ M, slope 3.0; (c) $[Cl^-]=11.03$ M, slope 3.0.

In addition to experiments with 100% TBP, values of D were measured with aqueous phases of constant composition and dilute solutions of TBP in benzene as the organic phase. Results are plotted in Figs. 2 and 3 and the slopes of the graphs may be taken as approximating to the average number of molecules of TBP directly associated with extracted scandium(III) species.

The interesting possibility arises from the present study that scandium(III) may exist in TBP extracts from aqueous chloride solutions as complexes $ScCl_3\cdot x$TBP which may have different stereochemistry. It is intended to pursue the problem further using spectroscopic methods.