

### A NEW SOLVENT EXTRACTION METHOD FOR THE SEPARATION OF NIOBIUM AND TANTALUM

Sir:

We wish to make a preliminary report of the separation of niobium and tantalum by a new solvent extraction technique. It has been found that niobium may be extracted essentially quantitatively from strong hydrochloric acid with a solution of methyldioctylamine in xylene. Under these conditions the extraction of tantalum appears to be negligible. The niobium may then be "stripped" from the organic phase with nitric acid, sulfuric acid or dilute hydrochloric acid.

Methyldioctylamine, a water-insoluble tertiary amine, is known<sup>1</sup> to form the corresponding amine acid salts which are also water-insoluble, in general, and preferentially extract into organic solvents. The work to date suggests that approximately 8 M hydrochloric acid concentration is satisfactory for the separation.

In a typical experiment an aqueous phase containing Nb<sup>95</sup> tracer or Ta<sup>182</sup> tracer was extracted for five minutes with an equal volume of a 5% solution of methyldioctylamine in xylene. Each phase was checked for Nb<sup>95</sup>  $\gamma$  or Ta<sup>182</sup>  $\gamma$  radioactivity by use of a scintillation counter. The results of the initial investigation of the effect of hydrochloric acid concentration on the extraction of niobium and tantalum are given in Tables I and II, respectively.

TABLE I

THE EFFECT OF HYDROCHLORIC ACID CONCENTRATION ON THE EXTRACTION OF Nb<sup>95</sup> WITH METHYLDIOCTYLAMINE IN XYLENE

HCl, M	Nb <sup>95</sup> Extracted, %
2	4.0
3	2.4
4	2.2
6	21.5
8	99.3
9.6	100.0
(control) 9.6 (no MDOA)	0.04

TABLE II

THE EFFECT OF HCl CONCENTRATION ON THE EXTRACTION OF Ta<sup>182</sup> WITH METHYLDIOCTYLAMINE IN XYLENE

HCl, M	Ta <sup>182</sup> Extracted, %
2.91	0.25
4.85	0.11
7.28	0.55
8.85	1.10
10.10	1.35
11.20	1.40

It has been found that Nb<sup>95</sup> does not extract appreciably from nitric acid concentrations up to 10.6 M and from sulfuric acid concentrations up to 12 M. Ta<sup>182</sup> does not extract from nitric acid concentrations up to 10.6 M nor from higher concentrations of sulfuric acid, but appears to extract appreciably from 2 M H<sub>2</sub>SO<sub>4</sub>. The extraction behavior of these elements in dilute sulfuric acid is being investigated.

(1) R. L. Smith and J. E. Page, *J. Soc. Chem. Ind.*, **67**, 48 (Feb., 1948).

Using the procedure given above, Nb<sup>95</sup>-Ta<sup>182</sup> tracer mixtures have been separated and non-radioactive Nb-Ta separations at one milligram/milliliter concentrations have been effected.

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### THE DIPOLE MOMENT OF DECABORANE

Sir:

In the course of a comprehensive investigation of decaborane,<sup>1</sup> B<sub>10</sub>H<sub>14</sub>, we have measured the dielectric constant of benzene solutions and have obtained a surprising value for its dipole moment which should be of considerable interest to those concerned with the structures and bonding of the boron hydrides.

Preliminary experiments showed that even short exposure to moist air led to variable results. Therefore the decaborane and benzene were purified with great care and stored under vacuum conditions. The solutions were prepared in a dry box and transferred to the dielectric cell without exposure to the air. The dielectric constants of solutions ranging in concentration from 0.0071345 to 0.018691 mole fractions of decaborane were measured at 25°, using a heterodyne-beat oscillator equipped with a precision condenser and operating at a frequency of 1.79 megacycles. The dielectric constant of benzene was taken to be 2.2773 at 25°. Subsequently the densities of the solutions were determined pycnometrically and values were calculated for the polarization of decaborane according to the method described by Smyth.<sup>2</sup> Graphical extrapolation to infinite dilution gives the value 297.0 for the total molar polarization of B<sub>10</sub>H<sub>14</sub> in benzene solution. If the sum of the electronic and atomic polarizations is assumed to be 1.05 times the molar refraction this sum equals 43.9, and the dipole moment of B<sub>10</sub>H<sub>14</sub> is then calculated to be 3.52 ± 0.02 Debye.

Mole fraction B <sub>10</sub> H <sub>14</sub>	Total molar Polarization of B <sub>10</sub> H <sub>14</sub>
0.0071345	295.5
.0073878	295.1
.0078698	294.8
.013943	293.3
.018691	291.2

This is an unexpectedly high value in the light of the Kasper, Lucht and Harker model<sup>3</sup> for the structure of decaborane and present theories of electronegativity and bonding. It appears, however, that high polarity may be characteristic of asymmetric boron hydrides having a number of hydrogen bridge bonds. This is supported by a recent microwave investigation of pentaborane, B<sub>5</sub>H<sub>5</sub>, by Pimentel and associates.<sup>4</sup> They report a dipole

(1) We wish to thank Dr. A. E. Newkirk of the Research Laboratory of the General Electric Company for providing the decaborane and for information concerning the characteristics of this compound.

(2) C. P. Smyth, "Dielectric Constant and Molecular Structure" (The Chemical Catalog Co.), Reinhold Publ. Corp., New York, N. Y., 1931.

(3) J. S. Kasper, C. M. Lucht and D. Harker, *Acta Cryst.*, **3**, 436 (1950).

(4) Professor G. C. Pimentel and associates describe this in a letter recently submitted to the *Journal of Chemical Physics*.