Notes

larger temperature interval (of about 400° K). CAVALIER's data are given in Table 1 and compared with our previous values (see Table 1 of reference 3). The new values are twice as high and therefore our empirical curve requires a revision.

Additional H_{η} -data⁽⁸⁾ used are 5100 for Ca ($T_M = 1123^{\circ}$ K) and 5300 for Ag ($T_M = 1233^{\circ}$ K). For Sb, an average of Cavalier's value⁽⁸⁾ and a value from recalculated Second Liquid Metal Handbook's data was taken: $H_{\eta} - 4600$, $T_M = 903^{\circ}$ K. The rest of the values used are identical with those given in Table 1 of reference 3.

Based on the new data, the empirical relationship is simplified. The log of H_{ij} is a simple straight line function of the log of T_M , as shown in Fig. 1, in contrast to the old curve (see Fig. 2 of ref. 3 or Fig. 11 in ref. 1).

Expressed algebraically, the relationship is:

$$\log_{10} H_{\eta} = 1.348 \log_{10} T_M = 0.366,$$

an equivalent from is:

$$H_{\eta} = 0.431 \cdot T_{M}^{1.348}$$

where T_M is the melting point of the metal in ${}^{\circ}K$.

TABLE 1.—VISCOSITY CONSTANTS, H_{η} and a, of andrade's viscosity equation from Cavalier's data⁽⁸⁾

Metal	<i>Т_м</i> , (°К)	η in cP. $(T_{\mathcal{M}})$	Constant a (P \times 10 ^s)	H_{η} (cal/g atom)	Old H_{η} vaules ^(1,2,3)
Cu	1357	4.09	2.58	7450	3680
Ni	1728	5.55	1.69	12000	
Co	1768	5.25	3.04	10300	_
Fe	1812	5.84	3.49	10200	5070

The viscosity of *any metal at any temperature* can thus be calculated by the use of the above equations and in conjunction with the well-known Andrade equation (see refs. 1, 2, 3) for the viscosity of a metal at its melting point.

It is obvious that additional experimental measurements of the viscosity of metals melting above 1000°K, and particularly those melting above 2000°K, are highly desirable to further strengthen the empirical basis of the relationship.

Our previous estimate of the viscosity of liquid plutonium⁽⁹⁾ requires revision. The new estimated value of H_{η} is 4350, instead of the old value of 3200; the new estimated value at 2000°K, is 1.48 cP, instead of the value⁽⁹⁾ of 1.72 cP (see also Table 2 of ref. 3).

A similar revision has to be made of the H_{η} of the lanthanide metals.⁽⁹⁾

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The preparation of ²¹⁰Bi by a Szilard-Chalmers reaction

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MAURER and RAMM,⁽¹⁾ and MURIN and NEFEDOV⁽²⁾ reported the separation of ²¹⁰Bi from neutron irradiated organo-bismuth compounds by a Szilard–Chalmers process. In both cases carrier bismuth was added to aid the isolation of the ²¹⁰Bi; consequently no enrichment factors were quoted. The enrichment factors obtained in separated "carrier-free" ²¹⁰Bi from irradiated triphenyl bismuth and triphenyl bismuth dichloride have been determined and are reported in this note.

The results show that the highest specific activity ²¹⁰Bi was that separated from very pure triphenyl bismuth. The yields of ²¹⁰Bi were low and somewhat variable.

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Notes

EXPERIMENTAL

Triphenyl bismuth was dissolved in benzene and the solution extracted with water to remove hydrophilic bismuth impurities. The triphenyl bismuth was then twice recrystallized from benzene. Triphenyl bismuth dichloride was prepared by a standard method⁽³⁾ and was recrystallized from 50/50VV chloroform-methanol.

The organo-metallic compounds were sealed in small polythene bags and irradiated in BFPO at Harwell, usually for 2 hr at a flux of 2×10^9 neutrons cm⁻² sec⁻¹. Varying the time of the neutron irradiation between 1 and 24 hr caused no deterioration in the performance of the solvent extraction process described below.

Triphenyl bismuth		Recrystallized Triphenyl bismuth		Recrystallized Triphenyl bismuth dichloride		Aqueous phase
(%) Yield	E.F.	(%) Yield	E.F.	(%) Yield	E.F.	
19	70	13*	200	14	140	- <u> </u>
49	250	26	430	29	very low	b
		35*	840			с
9	190	6	450			с
		7	520			С
		11	500			d
36	80					e
43	270	*				f
33	400					f
34	400					g

 TABLE 1.—YIELDS AND ENRICHMENT FACTORS (E.F.) BY SOLVENT

 EXTRACTION SEPARATIONS

* The experiments were also carried out in presence of 0.5 mg of bismuth carrier added to the aqueous extracting phase. Recoveries of ²¹⁰Bi were approximately 80 per cent.

Triphenyl b	oismuth	Recrystallized triphenyl bismuth		Recrystallized triphenyl bismuth dichloride	
Yield (%)	E.F.	Yield (%)	E.F.	Yield (%)	E.F.
12	150 190	34 25	175 (low)	27	(low)
23	165	20 23	500 465		

 TABLE 2.—YIELDS AND ENRICHMENT FACTORS (E.F.) BY ZINC

 REDUCTION COLUMN METHOD

The two separation methods used in this work were solvent extraction and retention on a zinc reductor column. In the first method a weighed portion of the irradiated compound was dissolved in benzene and the solution extracted by shaking with one of various aqueous phases (see below) in a polythene ampoule. Two successive extractions were carried out. The aqueous phases were combined, backwashed with benzene and evaporated to dryness with a few drops of perchloric acid. Bismuth was isolated from the residue by quantitative extraction into carbon tetrachloride of the diethyl-dithiocarbamate complex⁽⁴⁾. The amount of bismuth extracted was then determined spectrophotometrically. The carbon tetrachloride solution of the complex was evaporated to dryness and decomposed by nitric acid. ²¹⁰Bi was then β -counted through a 7 mg cm⁻² aluminium absorber using a General Electric Company 2B2 Geiger Müller counter. By direct counting a weighed amount of the irradiated starting material, the specific activity of the ²¹⁰Bi produced could be compared with the specific activity of the enriched ²¹⁰Bi (Table 1). The aqueous phases used for the solvent extractions

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(4) K. L. CHENG, R. H. BRAY and S. W. MELSTED, Analyt. Chem. 27, 24 (1955).

were: (a) distilled water; (b) lanthanum nitrate solution, 1 mg/ml, pH \sim 5; (c) 2% ammoniacal disodium ethylendiamine tetra-acetate, pH > 10; (d) 2% potassium iodide, pH \sim 4; (e) 5% antimonyl potassium tartrate, pH \sim 5; (f) 2% ammonium oxalate, pH \sim 7; (g) disodium hydrogen phosphate and citric acid buffer, pH 6.0. These reagents were chosen because it was thought that (b) and (e) might act as non-isotopic carriers, and (c), (d) and (f) might act as complexing agents, while the function of (g) was primarily to act as buffer.

Reduction separations (Table 2) were performed based on the assumption that zinc would electrochemically reduce bismuth species in solution and that the bismuth metal so formed would be deposited on the surface of the zinc metal. A benzene solution of the irradiated bismuth compound was passed through a column of zinc dust and the column then washed with several portions of benzene. The zinc was dissolved in nitric acid and the solution evaporated to dryness. The residue was dissolved in 0.5 M HCl, 1 mg of copper carrier added and then cupric sulphide precipitated. Bismuth was then extracted as the diethyldithiocarbamate, as previously described, from the mixed copper-bismuth solution.

RESULTS

In spite of the variable yields and enrichments obtained, the best method discovered of preparing enriched ²¹⁰Bi was from irradiated pure triphenyl bismuth using as aqueous extractant ammoniacal 2% disodium ethylendiamine tetra-acetate.

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Homogeneous precipitation and X-ray diffraction studies of urea uranate

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GENTILE, TALLEY and COLLOPY⁽¹⁾ first reported that urea uranate $(H_2O \cdot UO_3 2urea)$ may be obtained by simple addition of ammonium hydroxide to the water solution of uranyl nitrate and urea. The authors also reported the X-ray data of powder photographs without, however, attempting to index the reflections. More detailed studies of reactions in the system $H_2O-NH_4OH-UO_2(NO_3)_2 NH_2CONH_2$, were made by some of us⁽²⁾ by means of potentiometric titrations.

We now report that urea uranate may be obtained also by homogeneous precipitation from the system $H_2O-NH_4OH-UO_2(NO_3)_2-NH_2CONH_2$. Such a method of precipitating uranium-bearing compounds has been previously reported by HERMANS⁽³⁾. He did not identify the products as urea uranate, but as a nonstoichiometric compound containing 1% of urea and 9% of NH₄HCO₃. Our applied precipitation conditions were similar to those employed by Hermans except that the uranium concentration was about 50 per cent higher, and no excess of nitrate was added.

The chemical analyses have shown the composition close to that expected for urea uranate H_2UO_4 (NH_2CONH_5)₂. The analytical procedure was the same as already described⁽²¹⁾.

The preliminary X-ray identification was done by means of a 57.3 mm diameter camera and CoK radiation. The data from X-ray powder patterns are shown in Table 1. For comparison, data for the samples precipitated at room temperature are included.

Further powder photographs of the samples obtained by homogeneous precipitation were taken with a camera of 57.3 nm diameter and CuK α radiation. The diffraction patterns checked in the range $\Theta = 0-20^{\circ}$ with the Siemens Interferenz Goniometer. It was possible to index all the reflections on the basis of an orthorhombic cell with the following dimensions: a = 7.33 Å, b = 10.12 Å, c =12.10 Å. The unit cell has a volume of 897.6 Å³ and the theoretical density is 3.14 g/cm³, assuming four formula units per cell. This is in good agreement with the observed pycnometric density -3.10 g/cm³. The visually-estimated intensities of the diffraction lines, and the measured and calculated sin⁴ Θ values are given in Table 2, together with the reflection indices. The conditions limiting all reflections are: (0kl), k = l - 2n; (h0l), l + h - 2n; h = 2n; (h000), h - 2n; (0k0), k - 2n; (00l), l = 2n; the space group is consequently Pnna $-D_{2h}^{e}$. The general position of Pnna $-D_{2h}^{e}$ is eight-fold and there are four sets of four-fold positions. As there are only four U atoms in the cell,

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