516. *Eight Co-ordinate Complexes of Niobium*(IV)

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The reaction of excess of *o*-phenylenebisdimethylarsine (Diarsine) with niobium pentahalide, tetrahalide, or oxytrihalide in sealed tubes leads to the formation of $Nb^{IV}X_4$, 2Diarsine (X = Cl, Br, or I). The chloro- and bromo-complexes are isomorphous with the known eight co-ordinate, dodecahedral compounds MX_4,2Diarsine (X = Cl; M = Ti, Zr, Hf, or V: X = Br; M = Ti, Zr, or Hf).[1] The iodo-complex is not isomorphous with the chloro- or bromo-complexes, but an examination of the visible spectra indicates that the metal has the same stereochemistry. These compounds appear to be the first for which eight co-ordination of niobium has been established, and among the very few paramagnetic niobium compounds which are magnetically dilute. Although the magnetism is complicated by the presence of a ferromagnetic decomposition product, the existence of two paramagnetic species is confirmed by the observation of two *g* values in the electron spin resonance spectrum. Attempts to isolate the corresponding tantalum complexes were unsuccessful.

We have shown elsewhere[1] that the bidentate ligand *o*-phenylenebisdimethylarsine (Diarsine),[2] $o\text{-}C_6H_4(AsMe_2)_2$, is capable of stabilising eight co-ordination of Group IV metals by the formation of compounds of the type MX,2Diarsine (M = Ti, Zr, or Hf; X = Cl or Br). In addition, the paramagnetic complex $V^{IV}Cl_4$,2Diarsine was obtained from vanadium tetrachloride (magnetic moment of 1·74 B.M.). Nearly all the formally d^1 compounds of Nb^{IV} and Ta^{IV} are diamagnetic, and the structures show that the metal atoms occur in pairs linked by a metal–metal bond. For example, the tetrahalides NbX_4 and TaX_4 (X = Cl, Br, or I) are diamagnetic,[3,4] and the structures show the presence of metal–metal bonds.[5] Similarly, the oxyhalides $NbOI_2$ [6] and $TaOCl_2$,[4] the disulphides NbS_2 and TaS_2,[7] and the dioxides NbO_2 and TaO_2 [7,8] are diamagnetic or weakly paramagnetic, and the

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[1] (*a*) R. J. H. Clark, J. Lewis, and R. S. Nyholm, *J.*, 1962, 2460. R. J. H. Clark, W. Errington, and R. S. Nyholm, unpublished results. (*b*) R. J. H. Clark, D. L. Kepert, R. S. Nyholm, and J. Lewis, *Nature*, 1963, **199**, 559.

[2] J. Chatt and F. G. Mann, *J.*, 1939, 610.

[3] R. F. Rolsten, *J. Amer. Chem. Soc.*, 1958, **80**, 2952; R. E. McCarley and B. A. Toup, *Inorg. Chem.*, 1963, **2**, 540.

[4] H. Schafer, E. Sibbing, and R. Gerkin, *Z. anorg. Chem.*, 1961, **307**, 173.

[5] L. F. Dahl and D. L. Wampler, *J. Amer. Chem. Soc.*, 1959, **81**, 3150.

[6] H. Schafer and R. Gerkin, *Z. anorg. Chem.*, 1962, **317**, 105.

[7] E. I. Krylov, *Zhur. neorg. Khim.*, 1958, **3**, 1487.

[8] G. Brauer, *Z. anorg. Chem.*, 1948, **256**, 10.

structure of TaO_2 confirms the presence of metal–metal bonds.[9] The compounds Cs_2NbCl_6 and Cs_2TaCl_6 show complex magnetic behaviour.[10] It was decided to attempt to prepare Diarsine complexes of niobium and tantalum of the same type as those mentioned above, as it was thought that the high co-ordination number may sterically hinder the formation of metal–metal bonds, and the complex would, therefore, remain paramagnetic. Since the preliminary report of this work,[1b] paramagnetic niobium compounds have been reported by others. Thus, the reaction of niobium and tantalum tetrahalides with pyridine forms the adducts $MX_4 \cdot 2py$, which are non-electrolytes in pyridine and have magnetic moments ranging from 0·43 B.M. for $TaBr_4,2py$ to 1·37 B.M. for $NbCl_4,2py$.[11] Electrolytic reduction of alcoholic solutions of $NbCl_5$ forms $[Nb(OR)Cl_5]^{2-}$ (where R = Me, Et, or Pr^i), which has a magnetic moment of 1·72 B.M. for the pyridinium salt;[12] however, it is interesting that reduction in the presence of pyridine forms the diamagnetic dimer $[NbCl(OEt)_3py]_2$, which with sodium ethoxide forms the diamagnetic alkoxide $Nb(OEt)_4$.[13]

We have found that reaction of Diarsine with niobium pentahalide, tetrahalide, or oxytrihalide in sealed tubes leads to the formation of NbX_4,2Diarsine (X = Cl, Br, or I). In the reaction with the pentahalides, three moles of Diarsine were used for each mole of pentahalide; if only two moles were used, a mixture of niobium(IV) and niobium(V), compounds was obtained.[14] The pentaiodide reacts at 70°, the pentabromide at 100° and the pentachloride at 250°, but the pentafluoride did not react below 400°, at which temperature the sealed tubes exploded.

The chloro- and bromo-complexes are isomorphous with the known eight co-ordinate dodecahedral compounds MX_4,2Diarsine mentioned above. The infrared spectra of these compounds are also very similar, and characteristic of these compounds. The iodo-complex is not isomorphous with the chloro- or bromo-complexes.

These compounds are insoluble in all solvents tried. The diffuse reflectance spectra of the three compounds are related, with a general trend to lower wavelengths along the series Cl, Br, and I (Table 1 and Figure 1). The most plausible eight co-ordinate stereochemistries for molecules of this type are the dodecahedron and the square antiprism. The *d* orbitals are split into four energy levels under the influence of a dodecahedral field,[15] and into three energy levels under the influence of a square antiprismatic field,[16] and so the visible absorption spectrum would be expected to change if there was a change in stereochemistry. It therefore appears that NbI_4,2Diarsine has the same stereochemistry as the dodecahedral $NbCl_4$,2Diarsine and $NbBr_4$,2Diarsine. The spectrum of $TiCl_4$,2Diarsine (d^0) shows no absorption below 18,000 cm.$^{-1}$, whereas VCl_4,2Diarsine (d^1) shows a single absorption at 13,250 cm.$^{-1}$.[1a]

TABLE 1

Bands in the diffuse reflectance spectra (cm.$^{-1}$) of the eight co-ordinate Diarsine complexes

$NbCl_4$,2Diarsine	$NbBr_4$,2Diarsine	NbI_4,2Diarsine
10,700	10,600	10,250
13,800	13,800	13,100sh
(15,300w, sh)	—	—
17,300	16,700	15,200
22,700sh	21,200sh	19,700sh

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[9] B.-O. Marinder, *Acta Chem. Scand.*, 1961, **15**, 707.

[10] D. J. W. IJdo, Thesis, University of Leiden, 1960.

[11] R. E. McCarley and B. A. Toup, *Inorg. Chem.*, 1963, **2**, 540; R. E. McCarley and J. C. Boatman, *ibid.*, p. 547.

[12] R. A. D. Wentworth and C. H. Brubaker, *Inorg. Chem.*, 1962, **1**, 971; 1963, **2**, 551.

[13] R. A. D. Wentworth and C. H. Brubaker, *Inorg. Chem.*, 1964, **3**, 47.

[14] R. J. H. Clark, D. L. Kepert, and R. S. Nyholm, following Paper.

[15] M. Randic, *J. Chem. Phys.*, 1962, **36**, 2094.

[16] M. Randic, *Croat. Chem. Acta*, 1960, **32**, 189 (*Chem. Abs.*, 1961, **55**, 23,031); E. Konig, *Theor. Chim. Acta*, 1962, **1**, 14.

The observed magnetic moments of these NbX_4,2Diarsine compounds are higher than expected for d^1 niobium(IV) compounds. The magnetic moment increases with the number of packings (carried out in a dry-box) indicating probable decomposition to a compound of higher magnetic moment. The dependence of magnetic susceptibility on field strength (Figure 2, centre curves) indicates the presence of a ferromagnetic impurity. This field-dependent paramagnetism is characteristic for these compounds, and is not observed in the original niobium pentachloride, which is diamagnetic, or in any other niobium–Diarsine compounds which have been prepared.[14]

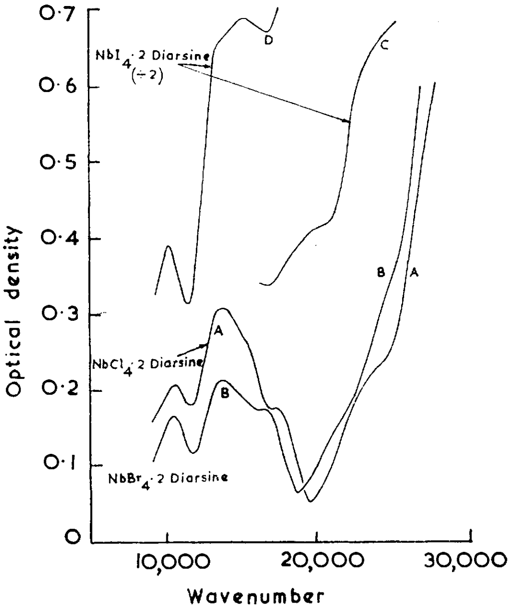

FIGURE 1

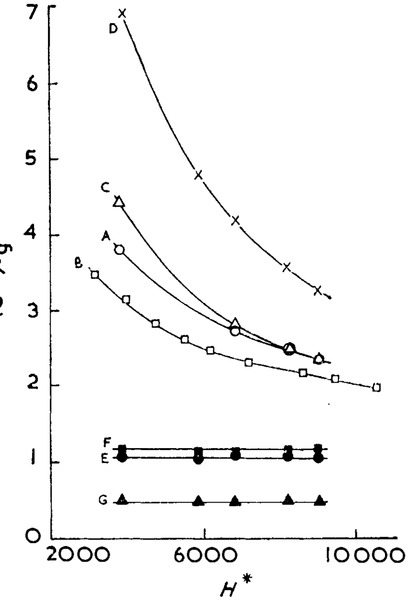

FIGURE 2

FIGURE 1. Diffuse reflectance spectra of NbX_4,2Diarsine compounds
Curve A: $NbCl_4$2Diarsine. Curve B: $NbBr_4$,2Diarsine. Curve C: NbI_4,2Diarsine (÷2). Curve D: NbI_4,2Diarsine

FIGURE 2. Gram-magnetic susceptibilities of NbX_4,2Diarsine compounds as function of field strength (H^*)
Curve A: $NbCl_4$,2Diarsine. Curve B: $NbBr_4$,2Diarsine. Curve C: NbI_4,2Diarsine. Curve D: Nb–O–As (Susceptibility × 0·1). Curve E: $NbCl_4$,2-Diarsine—3·9% Nb–O–As. Curve F: $NbBr_4$,2Diarsine—2·9% Nb–O–As. Curve G: NbI_4,2Diarsine—5·6% Nb–O–As

The magnetic susceptibility is inversely proportional to the effective field strength (H^*) over the range studied, and extrapolation to infinite field strength leads to the magnetic susceptibility of the non-ferromagnetic material.[17] This procedure gives effective magnetic moments of 1·8, 2·0, and 1·8 B.M. for the chloro-, bromo- and iodo-complexes, respectively: these moments are consistent with the spin-only value for one unpaired electron occupying a non-degenerate orbital (cf. the magnetochemistry of second-row transition metals forming octahedral complexes).

Some decomposition to form the ferromagnetic impurity invariably occurred, although elaborate precautions were taken to exclude oxygen and water. Attempts to prepare the complexes in sealed tubes, in the absence of oxygen and water, and to study the magnetic behaviour of the products, still contaminated with the niobium(V) and the oxidised

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[17] H. J. Hrostowski and A. B. Scott, *J. Chem. Phys.*, 1950, **18**, 105.

Diarsine by-products, were unsatisfactory due to the volume change accompanying the reaction. The compounds could not be purified by crystallisation because of their low solubilities. Attempts to isolate and identify the decomposition products were unsuccessful, but it was noted that on exposure to air the compounds developed the strong characteristic odour of phenyldimethylarsine, although the presence of small quantities of other arsines is also possible.

In an attempt to ascertain the nature of the ferromagnetic impurity, the samples were analysed for iron, and the magnetic properties of possible decomposition products were studied.

Analyses showed that small amounts of iron impurities are present in these compounds in approximately the same proportion in the reactants and products. For example (figures are expressed as % of iron in niobium):

$$\underset{0{\cdot}002}{\mathrm{NbCl_5}} \longrightarrow \underset{0{\cdot}003}{\mathrm{NbCl_4,2Diarsine}} \qquad \underset{0{\cdot}09}{\mathrm{Nb}} \longrightarrow \underset{0{\cdot}12}{\mathrm{NbI_4,2Diarsine}}$$

This small amount of iron would be insufficient to cause the observed magnetic behaviour unless it was present largely as metal (but it is difficult to envisage reduction under the conditions of the experiment) or as a ferromagnetic oxide of sufficient particle size to form domains. It is also important to note that similar magnetic behaviour is shown by complexes prepared from different sources of niobium, even although the iron content varied by a factor of 100.

Another course of action appeared to be the preparation of the decomposition product and the measurement of its magnetic properties. It was thought that this may be a niobium arsenide or oxyarsenide since it is known that niobium is capable of abstracting the donor atoms from ligands, for example, oxygen from triphenylphosphine oxide, with the resultant decomposition of the ligand. Although $\mathrm{NbO_{2{\cdot}3-2{\cdot}4}}$ shows a field-dependent susceptibility,[8] it is not of sufficient magnitude to explain our experimental results. No other ferromagnetic niobium compound could be found in the literature. $\mathrm{NbAs_{1{\cdot}8}}$ was prepared,[18] but it showed only normal paramagnetic behaviour. Heating of this compound in air produced a mixture of phases, presumably of arsenides, oxyarsenides, and oxides. This mixture is ferromagnetic, and the dependence of susceptibility on field strength is similar to that of the Diarsine compounds (Figure 2, upper curve; note change of scale). If it is assumed that the observed susceptibility of the Diarsine compounds is the sum of a field-independent susceptibility of the niobium(IV) atom, and of an impurity with the same field strength dependence as the " niobium oxyarsenide," the field-independent term can be estimated, and an approximate magnetic moment found (lower curves, Figure 2). The values obtained are 1·7, 1·9, and 1·6 B.M. for the chloro-, bromo-, and iodo-complex, respectively. Within the uncertainties of this calculation, these moments are again consistent with one unpaired electron.

These magnetic results are substantiated by the electron spin resonance spectra. The chloro-compound $\mathrm{NbCl_4}$,2Diarsine shows two discrete bands having g values of 1·951 and 2·002, respectively, whereas the niobium oxyarsenide shows only the latter band at $g =$ 2·002. The value of 1·951 is therefore due to the Diarsine compound, and leads to a magnetic moment of 1·69 B.M. The spectra of the bromo- and iodo-complexes, which are more sensitive to air than the chloro-complex, show much stronger bands due to the decomposition product, and the spectra due to the Diarsine compounds could not be located.

Hoard[19] has suggested that in the dodecahedral $\mathrm{Mo(CN)_8}^{4-}$, the filling of the d_{xy} orbital results in a repulsion of ligands in these positions. In the isomorphous d^0 and d^1 dodecahedral compounds prepared in this laboratory a significant distortion arising from the

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[18] E. Heinerth and W. Biltz, *Z. anorg. Chem.*, 1931, **198**, 168.

[19] J. L. Hoard and J. V. Silverton, *Inorg. Chem.*, 1963, **2**, 235.

occupation of this orbital should be visible as slight changes in the cell dimensions; all lines of the powder photographs are unchanged within experimental error.

All attempts to prepare the corresponding tantalum complexes failed. The easier reduction of niobium compared with tantalum is an important difference in their chemical behaviour; for example: (*a*) the mixed pentachlorides of niobium and tantalum can be separated by reduction with aluminium or hydrogen, as the nonvolatile $NbCl_{\sim 3}$ is formed, but the volatile $TaCl_5$ remains unchanged;[20] (*b*) reaction of niobium and tantalum pentahalides with the higher lithium dialkyl amides forms the reduced $Nb(NR_2)_4$ and the unreduced $Ta(NR_2)_5$.[21]

Even reaction of tantalum tetrachloride or tantalum tetrabromide with Diarsine did not yield the eight co-ordinate complexes. There is no reaction at low temperatures, and at high temperatures the tetrahalides disproportionated to TaX_3 and TaX_5, the latter forming TaX_5,Diarsine.[14]

It is convenient at this stage to summarise the eight co-ordinate dodecahedral compounds of the type MX_4,2Diarsine now known (Table 2).

TABLE 2

Known eight co-ordinate dodecahedral compounds of the type (MX_4,2 Diarsine)^0. Univalent cations of technetium and rhenium of the type (MX_4,2 Diarsine)$^+$, where X = Cl or Br, have also been reported (J. E. Fergusson and R. S. Nyholm, *Chem. and Ind.*, 1958, 1555. J. E. Fergusson, W. J. Kirkham, and R. S. Nyholm, " Rhenium," Elsevier, 1962)

$TiCl_4$,2Diarsine	$ZrCl_4$,2Diarsine	$HfCl_4$,2Diarsine	VCl_4,2Diarsine	$NbCl_4$,2Diarsine
$TiBr_4$,2Diarsine	$ZrBr_4$,2Diarsine	$HfBr_4$,2Diarsine		$NbBr_4$,2Diarsine
				NbI_4,2Diarsine

EXPERIMENTAL

The starting materials and products were stored in vacuum manifolds, and all manipulations carried out in a conventional nitrogen-filled dry-box. " Oxygen-free " nitrogen was further purified by passing it through a column containing activated copper at 400°, and then thoroughly scrubbing it with chromous solutions (0·4M) in sulphuric acid (0·1M). The gas was then dried with sulphuric acid and magnesium perchlorate. The solvents were dried over calcium hydride for several weeks, deoxygenated with the purified nitrogen, and then fractionally distilled.

Niobium was determined by ignition of the complex to the oxide, and halogen by potentiometric titration with silver nitrate after fusing the sample with potassium hydroxide. Microanalyses for carbon, hydrogen, and arsenic were determined by the Max-Planck Institut für Kohlenforschung, Mülheim, Germany. Trace amounts of iron were determined colorimetrically with *o*-phenanthroline, after the sample had been decomposed by repeated evaporation with hydrofluoric acid and concentrated nitric acid until a clear solution was obtained.[22]

The diffuse reflectance spectra were measured by using the standard attachment to a Unicam S.P. 500 spectrophotometer with magnesium carbonate as the blank reflector. The magnetic susceptibilities were measured by the conventional Gouy method.

Materials.—Niobium pentachloride. Obtained from Johnson, Matthey and Co. (Found: Cl, 66·4. Calc. for $NbCl_5$: Cl, 65·7%). Magnetic susceptibility $\chi_g = 0{\cdot}56 \times 10^{-6}$, therefore $\chi'_{Nb} = -52 \times 10^{-6}$ cgsu.

Tantalum pentachloride. Obtained from Johnson, Matthey and Co. (Found: Cl, 49·3. Calc. for $TaCl_5$: Cl, 49·5%).

Niobium pentabromide. Prepared from cleaned niobium sheet (L. Light and Co.) and excess of AnalaR bromine in a sealed tube at 450°. It was separated from $NbOBr_3$ by fractional sublimation (Found: Br, 81·0. Calc. for $NbBr_5$: Br, 81·2%).

Tantalum pentabromide. Prepared from cleaned tantalum sheet (L. Light and Co.) and excess of AnalaR bromine in a sealed tube at 450° (Found: Br, 69·1. Calc. for $TaBr_5$: Br, 68·8%).

Niobium pentaiodide. The pentaiodide was prepared from cleaned niobium sheet and AnalaR iodine in a sealed tube at 260° (Found: I, 83·0. Calc. for NbI_5: I, 87·2%).

[20] H. Schafer and C. Pietruck, *Z. anorg. Chem.*, 1951, **266**, 151.

[21] D. C. Bradley and I. M. Thomas, *Canad. J. Chem.*, 1962, **40**, 449, 1355.

[22] F. T. Sisco and E. Epremian, " Columbium and Tantalum," John Wiley, New York, 1963.

Tantalum pentaiodide. This pentaiodide was prepared from cleaned tantalum sheet and AnalaR iodine at 375° (Found: I, 77·9. Calc. for TaI_5: I, 77·9%).

Niobium oxytrichloride. The oxytrichloride was sublimed as a less volatile, silky white material from commercial pentachloride. The infrared spectrum shows a strong –Nb–O–Nb– stretching frequency at 765 cm.$^{-1}$.

Niobium tetrachloride. Reduction of the pentachloride with niobium sheet, at 400° under a large partial pressure of pentachloride, and fractional sublimation, also under a large partial pressure of pentachloride (to prevent disproportionation of the $NbCl_4$ to $NbCl_5$ and $NbCl_{\sim 3}$), yielded the tetrachloride and the less volatile $NbCl_{2\cdot 90}$ (Found: Cl, 61·2. Calc. for $NbCl_4$: Cl, 60·5%).

Tantalum tetrachloride. The pentachloride was reduced with aluminium wire, at 400° under a large partial pressure of pentachloride at 200°, and the products were fractionally sublimed, under the same partial pressure of pentachloride, to produce a low yield of crystalline tetrachloride (Found: Cl, 43·9. Calc. for $TaCl_4$: Cl, 44·0%).

Tantalum tetrabromide. A very low yield of $TaBr_4$ (0·01 g.) was obtained from tantalum pentabromide and tantalum in a manner similar to that described for the tetrachlorides.

Tetrachlorodi-(o-*phenylenebisdimethylarsine*)*niobium*(IV).—*Preparation* 1. Niobium tetrachloride (0·56 g.) was heated with Diarsine (1·5 ml.; 3 mol.) in a sealed tube at 150° for several hours. The pale green *product* was washed with acetonitrile (20 ml.) and pumped dry (Found: C, 30·0; H, 4·0; As, 36·9; Cl, 17·5; Nb, 11·9. $C_{20}H_{32}As_4Cl_4Nb$ requires C, 29·8; H, 4·0; As, 37·1; Cl, 17·6; Nb, 11·5%).

Preparation 2. Niobium pentachloride (0·65 g.) was heated with Diarsine (1·2 ml.; 2·5 mol.) in a sealed tube at 200—250° for eight hours. The product was thoroughly washed, with boiling acetonitrile until free from red niobium(V) species,[14] and pumped dry (Found: C, 29·3; H, 4·0; Cl, 17·1%).

Preparation 3. Niobium oxytrichloride (*ca.* 0·05 g.) was heated with excess of Diarsine (*ca.* 0·2 ml.) in a sealed tube at 200° for 8 hrs. The product was washed with hot acetone until free from red niobium(V) compounds.[14] The powder pattern showed the residue to be identical with the above preparations.

The magnetic susceptibility increased for successive packings of the same sample χ_g = 3·89, 5·07, 5·37, and 5·90 × 10^{-6} cgsu. (magnetic moment μ_{eff} = 2·91, 3·27, 3·36, and 3·51 B.M.). The magnetic moment determined as a function of field strength is given below:

H^* (gauss)	3790	6800	8180	8950
$10^6\chi_g$ (cgsu.)	3·81	2·70	2·49	2·32
μ_{eff} B.M.	2·87	2·48	2·39	2·32

A sample which was decomposed by leaving it in air for some hours (with some oxidation to red niobium(V) compounds [14]) had a magnetic susceptibility, χ_g = 14·95 × 10^{-6} cgsu. (μ_{eff} = 5·5 B.M.)

Tetrabromodi-(o-*phenylenebisdimethylarsine*)*niobium*(IV).—Niobium pentabromide (0·32 g.) was heated with Diarsine (0·45 ml.; 3·5 mol.) in a sealed tube at 100° for 1 hr. The pale green *product* was washed with hot acetonitrile and pumped dry (Found: C, 24·2; H, 3·4; As, 30·4; Br, 32·5. $C_{20}H_{32}As_4Br_4Nb$ requires C, 24·6; H, 3·3; As, 30·4; Br, 32·4%).

The magnetic susceptibilities for two successive packings were, χ_g = 2·00 × 10^{-6} and 2·05 × 10^{-6} cgsu. (μ_{eff} = 2·37 and 2·39 B.M.). The magnetic moment determined as a function of field strength is:

H^* (gauss)	3150	3950	4740	5480	6150	7120	8580	9410	10,050
$10^6\chi_g$ (cgsu.)	3·48	3·14	2·83	2·62	2·48	2·31	2·18	2·11	1·98
μ_{eff} B.M.	3·02	2·89	2·76	2·67	2·60	2·53	2·47	2·44	2·38

The susceptibility was also determined as a function of temperature at a field strength of 6200 gauss:

T(°K)	291·6	226·8	185·0	131·8	92·9	81·4
$10^6\chi_M$	2080	2370	2650	3270	4075	4530

Tetraiododi-(o-*phenylenebisdimethylarsine*)*niobium*(IV).—Niobium pentaiodide (0·83 g.) was heated with Diarsine (0·69 ml.; 3 mol.) in a sealed tube at 70° for 1 hr. The tube was opened, the contents were ground, resealed in another tube, and reheated. This procedure was repeated until apparently no black niobium pentaiodide remained in the yellow *complex*. This procedure is necessary to ensure that the Diarsine complex is not contaminated with unchanged niobium iodide which cannot be washed out. The compound was washed with carbon tetrachloride and pumped dry (Found: C, 20·2; H, 3·2; I, 45·0; Nb, 7·4. $C_{20}H_{32}As_4I_4Nb$ requires C, 20·7; H, 2·7; I, 43·3; Nb, 7·9%).

The magnetic susceptibility, $\chi_g = 5{\cdot}14 \times 10^{-6}$ cgsu., indicates a magnetic moment $\mu_{eff} =$ 3·93 B.M. The magnetic moment determined as a function of field strength is given below:

H* (gauss)	3790	6800	8180	8950
$10^6\chi_g$ (cgsu.)	4·48	2·84	2·50	2·32
μ_{eff} B.M.	3·69	3·00	2·83	2·75

Niobium–Oxygen–Arsenic Compound.—Niobium sheet (2·27 g.) was heated with arsenic (1·79 mol.) in a sealed tube at 800° for about 2 months. Unchanged niobium was picked out, and excess of arsenic sublimed off, leaving a small black residue (0·1 g.). This was then heated in air at about 500° for 30 min. producing a mixture of black and white particles. The mixture was finely ground and the magnetic susceptibility determined as a function of field strength:

H* (gauss)	3830	5890	6810	8190	8960
$10^6\chi_g$ (cgsu.)	69·2	48·0	41·9	35·6	32·8

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