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# Some Characteristics of Sodium Titanium Bronze and Related Compounds[1]

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Sodium titanium bronze, $Na_xTiO_2$ ($0.20 < x < 0.25$), has been prepared in powder form by solid-state reaction of $Na_2Ti_3O_7$, $TiO_2$, and Ti. The material shows relatively high conductivity, low, temperature-independent paramagnetic susceptibility, and an electronic spectral absorption consistent with a delocalized-electron model involving band formation through Ti(3d $t_{2g}$)–Ti(3d $t_{2g}$) or Ti(3d $t_{2g}$)–O(p$\pi$)–Ti(3d $t_{2g}$) overlap. Compounds isomorphous with $Na_xTiO_2$, $Na_2Fe_2Ti_6O_{16}$ and $Na_2Sc_2Ti_6O_{16}$, have also been prepared; they are not bronzes and show little sign of free-electron characteristics. The iron isomorph has low conductivity, a normal field-independent paramagnetism, and an electronic absorption characteristic of a spin-free $d^5$ iron in an octahedral oxygen environment. The scandium compound, which is a new one, is nonmagnetic and nonconductive; its unit cell dimensions are $a = 12.442 \pm 0.004$, $b = 3.845 \pm 0.002$, $c = 6.578 \pm 0.003$ A, and $\beta = 107.49 \pm 0.05°$.

## Introduction

The preparation and crystal structure of a sodium titanium bronze, of composition $Na_xTiO_2$, was originally described by Andersson and Wadsley,[3] and a number of compounds isomorphous with it were subsequently prepared by Bayer and Hoffman.[4] Among these are the compositions $Na_2O \cdot Fe_2O_3 \cdot (6 + m)TiO_2$, $m = 0$–4, which correspond to that of the mineral freudenbergite.[5,6] The establishing of $Na_xTiO_2$ as a bronze is of interest in that it extends the range of such structures known[3,7–9] and provides an example for which complete structure determination, including oxygen atom positions, has been made.[3] The structure consists of an extended network of octahedra joined at edges or corners, and no crystallographic distinction is made, or can be made, between $Ti^{3+}$ or $Ti^{4+}$, as the eight Ti atoms per unit cell occupy two sets of four equivalent positions. Special positions at the unit cell corners and *c*-axis faces, comprising the centers of groups of four corner-sharing octahedra, are available for sodium atoms. These are filled to the extent to which the $TiO_2$ host lattice is reduced. The $Na_xTiO_2$ lattice is of a structure type different from anatase, brookite, or rutile and is possibly stabilized by the presence of sodium ions.

Two limiting models for bronze behavior present themselves: in one, typified by the sodium tungsten bronzes, the electron from Na in $Na_xWO_3$ is donated with essentially zero activation energy to the conduction band of the host $WO_3$ structure;[7] in the other, typified by the sodium vanadium bronzes, the electron from Na in $Na_xV_2O_5$ is transferred to a $V^{5+}$ to form a localized $V^{4+}$ center from which electron excitation to the host conduction band can occur.[10] The measurements of this investigation were undertaken to establish whether $Na_xTiO_2$ conformed to a delocalized $Ti^{3+}$ model and to investigate the effect of having $Fe^{3+}$ or $Sc^{3+}$ in place of the hypothetical $Ti^{3+}$.

## Experimental Section

**Starting Materials.**—$TiO_2$ was Fisher Certified reagent. Ti metal was 99.8% Ti and was obtained as 300-mesh powder from Alfa Inorganics. After being dried in air at 100°, the sieved metal powder was analyzed for Ti metal content by its weight gain on heating at 800–1000° in air, a value of 96.0% free metal being found. Sodium carbonate and oxalate were analytical reagent grade. $Fe_2O_3$ was Baker laboratory reagent grade. $Sc_2O_3$, 99.5%, was obtained from the Australian Mineral Development Laboratories, Adelaide. Sodium trititanate, $Na_2Ti_3O_7$, was prepared by heating finely ground 1:3 mole ratio mixtures of sodium carbonate or oxalate with $TiO_2$ at 1000° for 15 hr.[11]

**Preparation of Compounds.**—Appropriate quantities of $Na_2Ti_3O_7$, Ti metal, and dry $TiO_2$ were separately sieved or ground to below 300-mesh particle size and then thoroughly mixed and ground before reaction. Reaction was effected in sealed platinum capsules held in evacuated silica ampoules heated at 800–1000° for periods of 12–40 hr. The X-ray powder patterns for all such preparations were identical. Reaction was also carried out at a continuously pumped low pressure of $10^{-6}$ torr. In this case pressed pellets of the reaction mixture were supported on platinum disks in a covered nickel crucible which was inductively heated at 850° for 4–6 hr. However, even after grinding and reheating for a similar period, a small amount of a phase identifiable as $Na_2Ti_6O_{13}$[12] was formed as a by-product, and some sodium metal, as vapor, was lost from the heated preparation. Reaction in sealed enclosures therefore seems preferable. The stoichiometry of the $Na_xTiO_2$ preparations was determined by the proportions of the reactants used in sealed-capsule preparations, and the $Ti^{3+}$ content of the products was verified by measuring the gain in weight of a sample heated in air at 650°. The magnetic, spin resonance, and optical studies were performed on samples containing no free $TiO_2$ or $Na_2Ti_6O_{13}$.

$Na_2Fe_2Ti_6O_{16}$ was prepared by reaction of appropriate quantities of sodium oxalate, $Fe_2O_3$, and $TiO_2$, thoroughly mixed and ground, then fired first at 900° for 15 hr and, after further grinding, at 1000° for 15 hr. No trace of any other phase could be detected, and the X-ray diffraction intensities and peak positions were closely similar to those for $Na_xTiO_2$. $Na_2Sc_2Ti_6O_{16}$

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[3] S. Andersson and A. D. Wadsley, *Acta Cryst.*, **15**, 201 (1962).

[4] G. Bayer and W. Hoffman, *Z. Krist.*, **121**, 9 (1965).

[5] D. McKie, *ibid.*, **119**, 157 (1963).

[6] A. D. Wadsley, *ibid.*, **120**, 398 (1964).

[7] M. J. Sienko, Advances in Chemistry Series, No. 39, American Chemical Society, Washington, D. C., 1963, p 224.

[8] D. Ridgley and R. Ward, *J. Am. Chem. Soc.*, **77**, 6132 (1955).

[9] F. Galasso, L. Katz, and R. Ward, *ibid.*, **81**, 5898 (1959).

[10] M. J. Sienko and J. B. Sohn, *J. Chem. Phys.*, **44**, 1369 (1966).

[11] S. Andersson and A. D. Wadsley, *Acta Cryst.*, **14**, 1245 (1961).

[12] S. Andersson and A. D. Wadsley, *ibid.*, **15**, 194 (1962).

was prepared similarly but contained a proportion of $Na_2Ti_6O_{13}$ as a separate phase, together with a corresponding amount of free $Sc_2O_3$. Using slow scan rates and high resolution the major phase and the two impurity phases were easily recognized and indexed. The monoclinic unit cell dimensions of $Na_2Sc_2Ti_6O_{16}$ were found to be $a = 12.442 \pm 0.004$, $b = 3.845 \pm 0.002$, $c = 6.578 \pm 0.003$ A, and $\beta = 107.49 \pm 0.05°$, and the diffraction intensities corresponded closely to those for $Na_xTiO_2$ and $Na_2Fe_2Ti_6O_{16}$.

**X-Ray Diffraction Spectra.**—Powder diffraction spectra were obtained on a General Electric XRD-5 diffractometer. Flat samples 2.5 × 2 cm were used, and a beam collimation angle of 1° ensured that the beam did not overlap the sample. Intensities were determined by measuring with a planimeter the areas of peaks obtained at slow scan rates. The $2\theta$ scale on the diffractometer was calibrated with tungsten powder and silicon powder samples, which gave calibrations agreeing within ±0.02° based on the NBS tables.[13]

**Magnetic Susceptibility.**—Susceptibilities were determined from 77 to 360°K using a Gouy balance arrangement sensitive to $2 \times 10^{-9}$ cgs unit. $HgCo(NCS)_4$,[14] sieved to a series of mesh sizes, was employed as the standard, and measurements were taken at fields from 2500 to 10,000 gauss at each temperature point. Pyrex sample tubes with a septum at the pole gap center and an evacuated extension 10–11 cm below the septum were used to obviate tube corrections. No change in weight, within 0.00002 g, could be detected when full field was applied to an empty tube.

**Electron Spin Resonance.**—The esr spectra were examined from room temperature to 77°K on a standard homodyne X-band system with balanced crystal detection and 100-kc/sec field modulation. Through the courtesy of the physics department group of Professor R. Silsbee, additional experiments also at X band were performed at 77, 4.2, and 1.2°K.

**Optical and Infrared Spectra.**—Reflection spectra from 40,000 to 3800 $cm^{-1}$ were obtained by diffuse reflectance using a Beckman DK-2A double-beam recording spectrophotometer fitted with a reflectance sphere and housing. It was found that sodium titanates have a strong ultraviolet absorption falling to an absorption edge near 330 mμ; the position of this edge, presumably a function of the Ti–O bond in octahedrally coordinated titanium, is very constant. While $Na_2Sc_2Ti_6O_{16}$, which we have found to be isomorphous with $Na_xTiO_2$, would appear to be the best choice of reference substance, very similar results were obtained by use of this substance and of a number of other titanates, including $NaScTiO_4$[15] and $Na_2Ti_6O_{13}$. The infrared spectra were obtained from KBr pressings containing 0.5% of $Na_xTiO_2$, using a conventional infrared transmission instrument.

## Results and Discussion

The preparative reaction employed

$$x\mathrm{Na_2Ti_3O_7} + 0.5x\mathrm{Ti} + (2 - 3.5x)\mathrm{TiO_2} \longrightarrow 2\mathrm{Na}_x\mathrm{TiO_2}$$

where $x$ ranges from 0.20 to 0.25, was found readily to give powder samples of the titanium bronze $Na_xTiO_2$. The powder pattern given by Andersson and Wadsley is incomplete, since their sample consisted of relatively few single crystals with consequent preferred orientation and absent reflection effects.[16a] The powder data were accordingly examined in full in the present work.[16b] The observed intensities, after allowance for Lorentz polarization and multiplicity, show good agreement with those observed by Andersson and Wadsley for a single crystal and, for the 16 strongest reflections, give a structure factor discrepancy factor of 16.6% when compared with their single-crystal data for which $R = 16.7\%$. Intensities did not vary greatly with change in sodium content over the range $x = 0.23$ to $x = 0.20$. Lattice constants for the monoclinic cell with $x = 0.23$ were found to be $a = 12.159 \pm 0.006$, $b = 3.844 \pm 0.003$, $c = 6.464 \pm 0.004$ A, and $\beta = 106.99 \pm 0.05°$.

Attempts to produce samples with $x < 0.20$ always resulted in the separation of $TiO_2$ (rutile form) as a distinct phase. Calibration of the areas of the relatively intense $TiO_2$ diffraction peaks by addition of weighed quantities of rutile to $Na_{0.20}TiO_2$ allowed the relatively small (10–20%) amount of *uncombined* $TiO_2$ to be calculated. For compositions nominally containing $x = 0.16$ and $x = 0.18$, it was found, in fact, that the values of $x$ were both $0.200 \pm 0.005$. The composition is thus limited to the range $NaTi_4O_8$ to $NaTi_5O_{10}$. For $Fe^{3+}$ and $Al^{3+}$ isomorphs the composition can vary from $NaFeTi_3O_8$ to $NaFeTi_5O_{12}$ and from $NaAlTi_3O_8$[4,16a] to $NaAlTi_5O_{12}$.

Hagenmüller[17] has shown that the reduction $Ti_2O_3 + Na_2O \rightarrow 2TiO_2 + 2Na$ occurs at temperatures above 800°. This side reaction occurred to a small extent in the present preparations when open capsules were heated in evacuated silica tubes and more particularly when the preparation was pumped continuously under vacuum.

Attempts to form the related bronze $Na_2Ti_6O_{12}$ ($Na_{0.33}TiO_2$) postulated by Andersson and Wadsley[3,12] using similar methods led to heavy loss of Na (as vapor) from the initially sealed reaction mixtures and production of mixed phases. In an attempt to avoid the side reaction of $Ti_2O_3$ with $Na_2O$, synthesis of the "model" compound $Na_2Sc_2Ti_4O_{12}$ was attempted. However, here also the compound $Na_2Sc_2Ti_6O_{16}$—*i.e.*, the $Na_xTiO_2$ isomorph—was formed together with $NaScTiO_4$.[15] The $Na_2Ti_8O_{16}$ structure type thus appears to be particularly stable, as evidenced by $Na_2Sc_2Ti_6O_{16}$ analogy, its slight loss of sodium on heating, and its numerous isomorphs.[4]

The magnetic susceptibility of $Na_{0.215}TiO_2$ was small and practically constant with temperature. Observed values of $\chi_M$, the susceptibility per mole of $Na_xTiO_2$, are shown in the last line of Table I. Because the titanium metal powder was not free of all traces of iron and because there was observed a linear dependence of $\chi$ on reciprocal magnetic field, values shown are those extrapolated to zero reciprocal field, where the susceptibilities were about half those observed at 10,000 gauss. The calculated values of $\chi_M$ are based on two models. In one, labeled "$Ti^{3+}$" in Table I, it is assumed that each Na atom in $Na_xTiO_2$ transfers an electron to $Ti^{4+}$ to form a localized $Ti^{3+}$ state, for which the magnetic susceptibility is calculated on a spin-only basis assuming a simple Curie law dependence. In the other, labeled "$e^-$ gas" in Table I, it is assumed that each Na contributes an electron to a degenerate elec-

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(13) H. E. Swanson and E. Tage, National Bureau of Standards Circular 539, Vol. I, U. S. Government Printing Office, Washington, D. C., 1962.

(14) B. N. Figgis and R. S. Nyholm, *J. Chem. Soc.*, 4190 (1958).

(15) $NaScTiO_4$, $NaFeTiO_4$, and $NaFe_xSc_{1-x}TiO_4$ will be discussed in a separate paper.

(16) (a) A. D. Wadsley, private communication. (b) A Table of these data has been deposited as Document No. 9153 with the ADI Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington 25, D. C. A copy may be secured by citing the document number and by remitting $1.25 for photoprints or $1.25 for 35-mm microfilm. Advance payment is required. Make checks or money orders payable to: Chief, Photoduplication Service, Library of Congress.

(17) P. Hagenmüller, private communication.

tron gas, for which the susceptibility can be calculated by the Pauli–Peierls equation.[7] The latter model is equivalent to a conduction band model in which the $Ti^{3+}$ state is delocalized, possibly by overlap of $t_{2g}$ orbitals of the titanium 3d manifold or, possibly more likely, by indirect overlap involving a $p\pi$ interaction with the oxygen atoms joining titanium atoms. If, as Morin predicts,[18] the band width in $TiO_2$ is quite large, then the effective mass of the conduction electrons, $m^*$, would be close to the rest mass, $m_0$. In Table I, two possibilities have been considered: (1) $m^* = m_0$, corresponding to a free-electron gas; (2) $m^* = 2.5m_0$, corresponding to an almost-ideal electron gas. It is noteworthy that the observed $\chi_M$ of $Na_xTiO_2$ can be most satisfactorily interpreted by an electron gas model with $m^* = 2.5m_0$, approximately the same situation that holds in the alkali metal tungsten bronzes.[7] In calculating the predicted susceptibilities of $Na_xTiO_2$, there is a problem in deciding what to use for the susceptibility of the host lattice. Published values for $TiO_2$ range from $30 \times 10^{-6}$ reported by Meyer[19] to $-24 \times 10^{-6}$ reported by Hill and Selwood.[20] The most likely value is the temperature-independent value $6 \times 10^{-6}$ reported by Ehrlich and others.[21] (The work of Ehrlich is particularly interesting; it covers a range of composition $TiO_x$, $1.90 < x < 2.00$, and shows that when $x$ is less than 2, the magnetic susceptibility becomes fairly large and inversely proportional to temperature. This means that the $TiO_2$ system is unlike the $WO_3$ system, where addition of Na atoms is magnetically equivalent to removal of O atoms.[22])

TABLE I

MAGNETIC SUSCEPTIBILITY OF $Na_{0.215}TiO_2$ (CGS UNITS × $10^6$)

| | Temp, °K | | | |
|---|---|---|---|---|
| | 77 | 128 | 248 | 297 |
| $\chi_M$, calcd ($Ti^{3+}$) | 1070 | 650 | 338 | 283 |
| $\chi_M$, calcd ($e^-$ gas, $m^* = m_0$) | 13 | 13 | 13 | 13 |
| $\chi_M$, calcd ($e^-$ gas, $m^* = 2.5m_0$) | 26 | 26 | 26 | 26 |
| $\chi_M$, obsd | 30.6 | 29.8 | 22.5 | 25.5 |

Electron spin resonance studies at room temperature showed no absorption lines, but behavior on insertion of the sample in the resonant cavity was indicative of a highly conducting material. At 77°K, a line centered at $g = 1.92$ appeared; its intensity followed a $1/T$ dependence down to 1.2°K and corresponded to about $10^{16}$ spins/cc. Initially, the line had been attributed to $Ti^{3+}$ donor centers, but the lack of an exponentially increasing concentration with decreasing temperature suggests it was due to trace impurities.

Attempts to prepare large single crystals of $Na_xTiO_2$ for conductivity studies were unsuccessful. In the absence of single-crystal specimens, electrical conductivity as a function of temperature was not measured. However, a powder compact of area 1 $cm^2$ and length 1 cm prepared under vacuum had a resistance of only 20 ohms, strongly dependent on contact pressure. It is expected that single crystals would show resistivities several orders of magnitude less than this. Using Morin's mobility of 0.1 $cm^2/v$ sec[18] and assuming each Na in $Na_xTiO_2$ contributes one free carrier to the conduction band, giving a carrier density of about $6.5 \times 10^{21}$ electrons/cc, we predict a resistivity for $Na_{0.2}TiO_2$ of 0.01 ohm-cm.

The above conclusion that in $Na_xTiO_2$ delocalization of electrons into a conduction band has occurred is supported further by the electronic absorption spectrum, curve A in Figure 1. The spectrum shows no evidence of a $Ti^{3+}$ "ligand-field band," as found for example in $Ti^{3+}$ aqueous solution and in solid $(C_5H_5)_2TiCl$,[23] but is characteristic of electrons in conduction bands. The discrete absorption bands at 970, 860, and 690 $cm^{-1}$ are probably due to Ti–O stretching vibrations; similar bands were observed for $Na_2Sc_2Ti_6O_{16}$. (M–O bands for other oxide systems have been described by Cotton[24] and others.[25] The Ti–O–Ti stretching vibration in an oxygen-bridged cyclopentadienyl derivative has also been found in this region.[26]) The strong electronic absorption at 29,000 $cm^{-1}$ continues to 3000 $cm^{-1}$, and the tail of this absorption may well extend under the vibrational absorption bands in the 600–1000-$cm^{-1}$ region. The dark blue tint evident in the substantially black $Na_xTiO_2$ must be due to the slight decrease in absorption at the blue end of the visible spectrum.

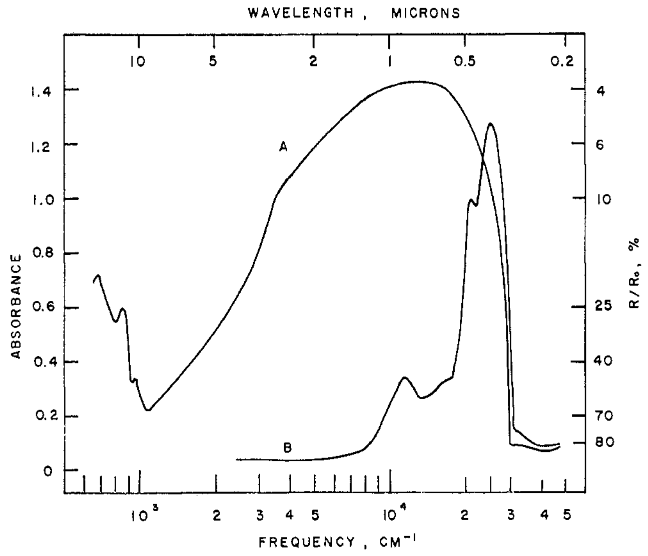

Figure 1.—Electronic absorption spectra: curve A, $Na_xTiO_2$; curve B, $Na_2Fe_2Ti_6O_{16}$.

By contrast with the physical properties of $Na_xTiO_2$, the isomorph $Na_2Fe_2Ti_6O_{16}$ was found to be a much poorer conductor (resistivity of a pressed pellet about

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(22) M. J. Sienko and B. Banerjee, *ibid.*, **83**, 4149 (1961).

(23) A. F. Reid and P. C. Wailes, *Australian J. Chem.*, to be published.

(24) F. A. Cotton, *Inorg. Chem.*, **4**, 1545 (1965).

(25) C. G. Barraclough, J. Lewis, and R. S. Nyholm, *J. Chem. Soc.*, 3552 (1959).

(26) S. A. Giddings, *Inorg. Chem.*, **3**, 684 (1964).

$10^7$ ohm-cm), and the isomorph $Na_2Sc_2Ti_6O_{16}$, a nonconductor at ordinary voltages. The iron compound shows the normal, field-independent paramagnetic susceptibility for $Fe^{3+}$ ions (Figure 2), a $g = 2.00$ esr signal, and an electronic absorption spectrum typical of a spin-free $d^5$ ion with all electrons localized in an octahedral oxygen environment (curve B of Figure 1). The magnetic susceptibility per gram of $Na_2Fe_2Ti_6O_{16}$ is given by

$$\chi_g = \left(\frac{11{,}560}{T + 120.8} \pm 0.04\right) \times 10^{-6} \text{ cgs unit}$$

for the temperature range 200–370°K, with $\mu_{eff} = 5.73$ BM for each $Fe^{3+}$ atom. Below 200°K the plot of $\chi_g^{-1}$ *vs.* $T$ deviates from linearity, as expected from the relatively high Weiss constant. However, the Weiss constant of 120.8° is very close to the value of 125° observed for the (structurally different) compound $NaSc_{0.5}Fe_{0.5}TiO_4$,[15] the unit cell formulas being $Na_2Fe_2Ti_6O_{16}$ and $Na_4Fe_3Sc_2TiO_{16}$, respectively, and is consistent with the bulk concentration of $Fe^{3+}$ ions. It is evident that for $Na_2Fe_2Ti_6O_{16}$ delocalization *via* band formation does not occur to any appreciable extent nor was it to be expected. In $Na_xTiO_2$ it is presumably, therefore, a function both of the presence of like atoms in the two sets of crystallographically equivalent metal atom positions and of the low donor ionization energy of $Ti^{3+}$. Failure to observe an esr signal for $Ti^{3+}$ at 1.2°K suggests that the donor state may actually lie in the conduction band. It is probable that in $Na_xTiO_2$ all of the Ti atoms participate in band formation, but the character of the band, particularly whether d–p$\pi$ overlap between metal and oxygen atoms is involved as in $ReO_3$,[27] remains to be established. To this end, carrier density and carrier mobility measurements on single crystals would be most useful.

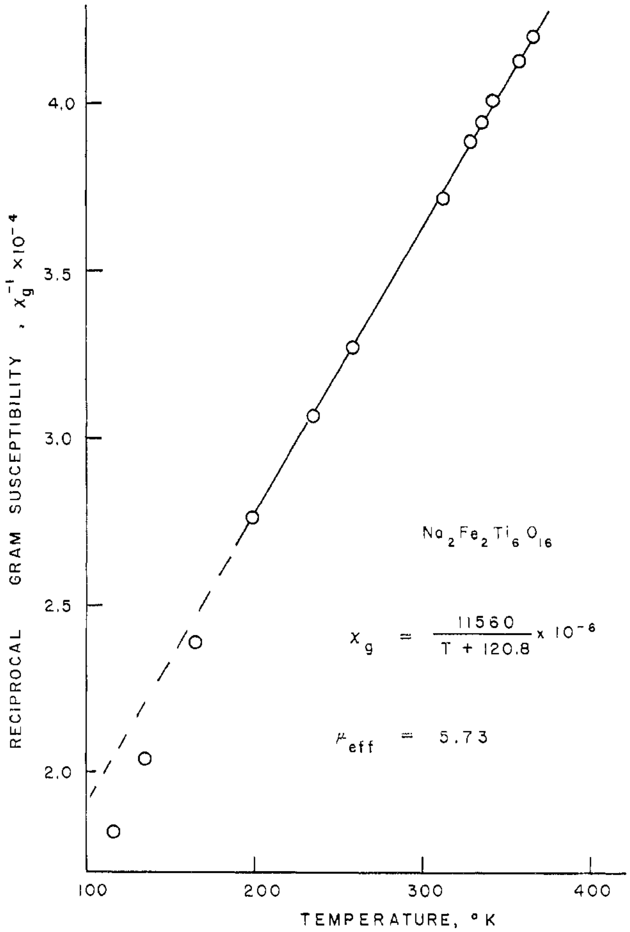

Figure 2.—Reciprocal gram susceptibility of $Na_2Fe_2Ti_6O_{16}$ as a function of temperature.

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# Magnetic Susceptibility of Uranium Trifluoride in the Range 2–300°K[1]

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The magnetic susceptibility of $UF_3$ has been measured by the Gouy method over the temperature range 1.9–300°K. Behavior follows the Curie–Weiss law $\chi = C/(T - \theta)$ with $\theta = -110 \pm 5°$. The effective moment is $3.67 \pm 0.06$ BM, corresponding closely to a $5f^3$ configuration. There is no sign of a Néel inversion even at the lowest temperature reached. Comparison of data for $UF_3$, $UCl_3$, and $UBr_3$ suggests that the ratio of spin–orbit coupling to ligand-field splitting increases in the order $UF_3$, $UCl_3$, $UBr_3$.

## Introduction

The rather great carrier mobility (10 cm$^2$/v sec) observed in single-crystal specimens of $WO_3$ can be explained in terms of electron delocalization from overlap of d orbitals.[2] Occasional findings of lustrous fragments in powder preparations of $UF_3$ suggest that similar delocalization might also occur in $UF_3$. However, preparation of single crystals of $UF_3$ for conductivity

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