

The reaction of the cubic sodium bronzes,  $\text{Na}_x\text{WO}_3$ , with powdered iron metal has been studied by heating samples *in vacuo* and also at high pressure. Evidence for reaction is found at unexpectedly low temperatures. The reaction is an overall reduction which proceeds via an increase in the sodium content of the bronze phase up to some temperature-dependent limiting composition for which  $x < 1$ . The existence of this limit, its temperature dependence, and the identity of the other products of reduction have been explained in terms of the partial oxygen pressure of the system. The course of the reduction has been followed through the evolution of the bronze lattice parameter and a reaction mechanism is postulated. No evidence of significant incorporation of iron into a stable cubic sodium bronze phase has been found.

*NMR Study of Hydrogen Molybdenum Bronzes:  $\text{H}_{1.71}\text{MoO}_3$  and  $\text{H}_{0.36}\text{MoO}_3$ .* R. C. T. SLADE, T. K. HALSTEAD, AND P. G. DICKENS. Department of Chemistry, University of York, Heslington, York YO1 5DD, England.

Proton NMR relaxation times ( $T_2$ ,  $T_1$ , and  $T_{1\rho}$ ) and absorption spectra are reported for the compounds  $\text{H}_{1.71}\text{MoO}_3$  (red monoclinic) and  $\text{H}_{0.36}\text{MoO}_3$  (blue orthorhombic) in the temperature range  $77 \text{ K} < T < 450 \text{ K}$ . Rigid lattice dipolar spectra show that both compounds contain proton pairs, as  $\text{OH}_2$  groups coordinated to Mo atoms in  $\text{H}_{1.71}\text{MoO}_3$  and as pairs of OH groups in  $\text{H}_{0.36}\text{MoO}_3$ . The room temperature lineshape for  $\text{H}_{1.71}\text{MoO}_3$  shows that the average chemical shielding tensor has a total anisotropy of 20.1 ppm. The relaxation measurements confirm that hydrogen diffusion occurs and give  $E_A = 22 \text{ kJ mole}^{-1}$  and  $\tau_c^0 = 10^{-13} \text{ sec}$  for  $\text{H}_{1.71}\text{MoO}_3$  and  $E_A = 11 \text{ kJ mole}^{-1}$  and  $\tau_c^0 = 3 \times 10^{-8} \text{ sec}$  for  $\text{H}_{0.36}\text{MoO}_3$ .

*Crystal Structures of Some Niobium and Tantalum Oxides. VIII. The  $5\text{Rb}_2\text{O} : 14.6\text{Ta}_2\text{O}_5$  Phase—A Tunnel Structure.* G. D. FALLON AND B. M. GATEHOUSE. Department of Chemistry, Monash University, Clayton, Victoria, 3168, Australia.

$\text{Rb}_{10}\text{Ta}_{29.20}\text{O}_{78}$  crystallizes in the hexagonal system with unit-cell dimensions (from single-crystal data)  $a = 7.503(4) \text{ \AA}$ ,  $c = 36.348(4) \text{ \AA}$  and space group  $P6_3/mmc$ ,  $z = 1$ . The structure was solved using three-dimensional Patterson and Fourier techniques. Of the 666 unique reflections measured by counter techniques, 515 with  $I \geq 3 \sigma(I)$  were used in the least-squares refinement of the model to a conventional  $R$  of 0.057 ( $wR = 0.039$ ). The structure of  $\text{Rb}_{10}\text{Ta}_{29.20}\text{O}_{78}$  consists of layers of corner-sharing groups of six edge-shared octahedra separated by layers of single octahedra and double hexagonal tungsten bronze-like layers, these layers being perpendicular to the hexagonal  $c$ -axis. Nine-coordinate tricapped trigonal prismatic sites between the hexagonal tungsten bronze-like layers are partially occupied by Ta(V) ions.

*Ion Exchange Properties of  $\beta$ -Eucryptite ( $\text{LiAlSiO}_4$ ): EPR Investigation on Copper-Doped Single Crystals.* JEAN-LOUIS BERCHOT, DANIEL VIVIEN, DIDIER GOURIER, JEANINE THERY, AND ROBERT COLLONGUES. Laboratoire de Chimie Appliquée de l'état Solide, 11, Rue P. et M. Curie 75231 Paris Cedex 05, France.

Investigation of the ion exchange properties of  $\beta$ -eucryptite ( $\text{LiAlSiO}_4$ ) single crystals indicates that it is impossible to substitute  $\text{Li}^+$  with other bigger univalent cations such as  $\text{Na}^+$ ,  $\text{K}^+$ , or  $\text{Ag}^+$ . On the contrary,  $\text{Li}^+$  exchange with bivalent cations,  $\text{Cu}^{2+}$  or  $\text{Mn}^{2+}$ , is very easy. For a general orientation of the crystal with respect to the magnetic field, the EPR spectrum of  $\text{Cu}^{2+}$  ions in  $\beta$ -eucryptite consists of 12 sharp lines partially superimposed on a broad one. The sharp lines are attributed to isolated copper ions in the conducting channels.  $\text{Cu}^{2+}$  lies in sixfold, coordinated  $\text{Li}''$  sites, but not in the fourfold coordinated  $\text{Li}'$  ones. The corresponding spin-Hamiltonian parameters at  $T = 140 \text{ K}$  are found to be:  $g_x = 2.362$ ,  $g_y = 2.340$ ,  $g_z = 1.990$ ;  $|A_x| = 85 \times 10^{-4} \text{ cm}^{-1}$ ,  $|A_y| = 71 \times 10^{-4} \text{ cm}^{-1}$ ,  $|A_z| = 203 \times 10^{-4} \text{ cm}^{-1}$ . The broad line is attributed to clusters of  $\text{Cu}^{2+}$  located in neighboring  $\text{Li}''$  sites.

*Ferrimagnetism in the Rare Earth Titanium (III) Oxides,  $\text{RTiO}_3$ ;  $R = \text{Gd, Tb, Dy, Ho, Er, Tm}$ .* CARL W. TURNER AND J. E. GREEDAN. Institute for Materials Research and Department of Chemistry, McMaster University, Hamilton, Ontario, L8S 4M1, Canada.