109.3(1)°. The structure was determined using three-dimensional X-ray diffraction data gathered on multiple-film equi-inclination, integrated Weissenberg and precession photographs taken about two crystal axes. The structure, excluding the hydrogen atoms, was solved from Patterson and electron density maps and refined by least-squares methods to a final R of 0.081. The coordination about the yttrium atom is sevenfold, best described by a capped trigonal prism. Each *ortho*-aminobenzoate ligand acts as a bridging bidentate ligand, resulting in six *ortho*-aminobenzoate residues coupled to each yttrium atom. The water molecule occupies the seventh position. This bonding configuration generates a structure in which each yttrium atom in (100) is attached to two other yttrium atoms via carboxylate bridges to give parallel sets of polymeric chains coincident with (100). It is suggested that this polymeric character accounts for the extreme insolubility of $Y(H_2NC_6H_4COO)_3 \cdot H_2O$.

Self-Compensation in Niobium-Doped TiO_2. N. G. Eror, Oregon Graduate Center, Beaverton, Oregon 97005. Gravimetric measurements on pure and niobium-doped TiO_2 have shown that the reversible change of oxygen content, between specified states of oxidation and reduction, is proportional to the dopant concentration. These measurements indicate that the donor–dopants are electronically compensated by additional oxygen uptake in the oxidized state. The range of this reversible change in oxygen stoichiometry is up to more than an order of magnitude larger than the oxygen nonstoichiometry of the undoped oxides. Self-compensation has been measured in TiO_2 with donor–dopant concentrations of up to 8 atom% Nb^{+5} over the oxygen partial pressure range 10^0–10^{-15} atm. For the case of Nb^{+5} doped TiO_2 the monoclinic phase "$TiNb_2O_7$" was found to exsolve under oxidizing conditions for Nb^{+5} concentrations >8 atom%. The exsolved phase transformed into niobium doped rutile under reducing conditions. The gravimetric measurements for Nb^{+5} concentrations <8 atom% can be explained by the creation of metal deficit point defects (self-compensation) or a model involving a shear structure.

Crystal Structure of the Ordered Pyrochlore $NH_4Fe^{II}Fe^{III}F_6$: Structural Correlations with $Fe_2F_5 \cdot 2H_2O$ and Its Dehydration Product $Fe_2F_5 \cdot H_2O$. G. Ferey, M. Leblanc, and R. De Pape, Laboratoire des Fluorures et Oxyfluorures Ioniques, Faculté des Sciences, Route de Laval, 72017 Le Mans Cédex, France. Crystal structure of $NH_4Fe^{II}Fe^{III}F_6$ is studied in order to explain further its peculiar antiferromagnetic behavior compared to the spinglass one of the pyrochlore family. $NH_4Fe^{II}Fe^{III}F_6$ is orthorhombic, space group $Pnma$ with $a = 7.045$ (4) Å, $b = 7.454$ (4) Å, $c = 10.116$ (6) Å, $Z = 4$. Diffraction data on single crystals obtained by hydrothermal synthesis, collected on an automatic four-circle diffractometer, have been refined by full-matrix least-squares calculations to a weighted value of 0.029 (unweighted $R = 0.024$) for 798 observed reflections. This structure is derived from the pyrochlore structure, with a cationic order between Fe^{2+} and Fe^{3+} ions. $(Fe^{II}F_6)^{4-}$ octahedra form infinite *trans* chains along [100] by sharing corners although similar chains of $(Fe^{III}F_6)^{3-}$ octahedra lie along [010]. This type of Fe^{II}–Fe^{III} order is related to a similar one existing in $Fe_2F_5 \cdot 2H_2O$, the dehydration of which leads to the pyrochlore $Fe_2F_5 \cdot H_2O$. A mechanism is proposed to explain the formation of this compound.

Crystal Growth and Electrical Properties of Lithium, Rubidium, and Cesium Molybdenum Oxide Bronzes. Pierre Strobel and Martha Greenblatt, Department of Chemistry, Rutgers University, New Brunswick, New Jersey 08903. Crystals of $Li_{0.33}MoO_3$(blue), $Rb_{0.23}MoO_3$(blue), and $Cs_{0.31}MoO_3$ (red) were grown by electrolysis from MoO_3–M_2MoO_4 melts (M = alkali metal) with composition 70–77 mole% MoO_3. Melts richer in M_2MoO_4 produced MoO_2 only. Correlation is made between bronze formation and the coordination of Mo in the melt and in the equilibrium solid phase $M_2Mo_4O_{13}$. $Li_{0.33}MoO_3$ and $Cs_{0.31}MoO_3$ are semiconductors with high-temperature range activation energies 0.16 and 0.12 eV. $Rb_{0.23}MoO_3$ has an electrical behavior similar to that of blue K_xMoO_3 with a semiconductor–metal transition at (170 ± 5) K. ESR spectra observed in $Li_{0.33}MoO_3$ and $Rb_{0.23}MoO_3$ single crystals at 4.2 K show extensive delocalization of the $4d^1$ electron associated with Mo(V) centers. Attempts to grow molybdenum bronzes containing Ca or Y were unsuccessful.

Synthesis of Rare Earth Monoxides. I. M. Leger, N. Yacoubi, and J. Loriers, E. R. 211, 1 place A. Briand, 92190 Meudon, France. The standard Gibbs energy changes for the formation of an ionic or metallic monoxide from rare earth metal and sesquioxide have been calculated. Under high pressures ionic ytterbium monoxide and lighter rare earth metallic monoxides should be obtained which is confirmed by experiments in a belt-type apparatus in the range 15–80 kbar and 500–1200°C. For *Ln* = La, Ce, Pr, Nd, Sm, a face-centered cubic compound is obtained from each reaction. The cell parameters are respectively 5.144, 5.089, 5.031, 4.994, and 4.943 ± 0.005 Å. The compounds appear golden yellow with a metallic luster. From chemical analyses and cell parameter considerations it is concluded that these compounds are the rare earth monoxides. For *Ln* = Gd, Dy, Tm, no reaction is observed at 50 kbar and 1000°C. The rare earth monoxides show a variety of properties: LaO, CeO, PrO, and NdO are metallic with the rare earth in the trivalent state; EuO and YbO are semiconductors with the rare earth in the divalent state; SmO is metallic with samarium in an intermediate valence state close to 3.

An Electron Microscope Study on Substitutional Order and Disorder in the System MnS-Ln_2S_3 *(Ln = Yb or Y)*. M. Bakker and C. M. Plug, Gorlaeus Laboratories, State University, P.O. Box 9502, 2300 RA Leiden, The Netherlands. The substitution of 2 Ln^{3+} (*Ln* is Yb or Y) for 3 Mn^{2+} at about 1873 K in the α-MnS structure (ccp) was investigated by means of electron diffraction (at room temperature). Diffuse intensity contours suggest that cuboctahedral clusters of Mn^{2+} and Ln^{3+} around a vacancy are formed. In the close-packed plane, the cluster is a hexagon of 4 Mn^{2+} and 2 Ln^{3+} around a vacnacy, a seven-point cluster. Clusters remain independent up to composition 4 MnS · Ln_2S_3. With higher Yb_2S_3 content spinel domains in the S.R.O. matrix are formed. Samples MnS–Y_2S_3 heated at 1873 K showed formation of extended defects (in this case twin planes) when they contained more than 20 mole% Y_2S_3 (14% vacancies).

Synthesis of New Luminescent Materials Activated with Divalent Samarium. A. Gros, F. Gaume, and J. C. Gâcon*, Université Lyon I, 43 Bd du 11 Novembre 1918, 69622 Villeurbanne, France. A simple technique for the preparation of powder compounds doped with divalent samarium is described. The reaction is carried out in nickel containers sealed in an inert atmosphere. The samarium impurity is introduced as samarium trifluoride SmF_3 and metallic samarium powder acts as the reducing agent to change Sm^{3+} into Sm^{2+}. Using this method, samarium has been stabilized in the divalent state in various fluorides: $KMgF_3$, $BaLiF_3$, BaY_2F_8, and KY_3F_{10}. The resulting compounds show under ultraviolet or visible excitation an intense luminescence in the red region characteristic of Sm^{2+}-doped materials. The emission and excitation spectra of these phosphors are presented and briefly discussed.