The data crystal was also examined by the precession method with the cone axis along both $\vec{c}$ and $\vec{a}$, but in neither case was any indication of additional superstructure reflections found. Secondly, a single crystal that had been cooled from 750 to 200 °C over 2 weeks was also checked by long exposures of Weissenberg photographs (Cu Kα) of the $l = 0, 1, 2$ levels as well as precession data (Mo Kα) for the $k = 0, 1$ levels. Both were again negative. Finally, the Guinier pattern of a sample equilibrated for 4.5 months at 550 °C showed nothing new. Evidently an ordering of the 7–8% vacancies in the metal sites on a scale sufficient for coherent X-ray scattering is not present. Smaller domain/twin composites or disorder along $\vec{c}$ could still be involved. This disorder feature (to X-rays) together with the compound's evident fixed composition (above) seems relatively unusual, but the same characteristics have been seen in Lu_3S_4[20] and $Na_{0.6}ScI_3$.[21]

A few other properties of this "diiodide" phase have also been examined. The magnetic susceptibility of a fully reduced sample was examined only briefly. The molar susceptibility corrected only for core diamagnetism is small, 334×10^{-6} emu/mol at room temperature or about 3 times the equivalent value for LaI_2,[10] and this quantity increases only moderately on cooling to 504×10^{-6} emu/mol at 118 K and 465×10^{-6} emu/mol at 77 K, with some possibility of a transition near 103 K. This aspect was not pursued further except to note that the ^{45}Sc NMR spectrum for the compound showed no changes in the 150–77 K range.

A polycrystalline rod of a reduced composition ~0.25 × 3 in. in size was grown in tantalum tubing, and its conductivity was examined by four-probe ac methods. The procedure was only marginally satisfactory for this compound because portions of rods in which crystals of the laminar diiodide grew roughly normal to the rod axis were easily fractured. It was also difficult to achieve complete reduction of such a large sample as it grew away from the metal reservoir. Notwithstanding, a sample with an overall composition corresponding to about 91 mol % $Sc_{0.93}I_2$, the rest ScI_3, was found to exhibit a resistivity of 1.54×10^{-3} Ω cm at 355 K, and this decreased fairly linearly to 0.89×10^{-3} Ω cm at 222 K. This quantity then appeared to increase ~20% on cooling to 160 K, but this may have been spurious as electrical contact was lost at lower temperatures and, shortly thereafter, the sample was lost as well during attempts to adjust the contacts.

The room-temperature resistivity for this sample was about 24 times that for a sample of pure LaI_2 but only about half as large as that obtained for a similarly impure sample containing ~86% of the incongruently melting CeI_2 mixed with $CeI_{2.5}$.[10] Since both the LaI_2 and the "CeI_2" samples showed very similar (positive) temperature coefficients of resistivity, we conclude the impurity level does not seriously hazard the conclusion that $Sc_{0.93}I_2$ is also metallic in character. The compound does not exhibit superconductivity down to 1.5 K. Such a metallic "dihalide" is not found in either the scandium chloride or the scandium bromide systems.[7] We conclude that a π^* description of the conduction band with a significant contribution by the iodide is apt for this phase as well as for LaI_2, CeI_2, etc.[10]

Acknowledgment. We are indebted to Norm Linder for the construction of the thermal analysis apparatus, to R. E. McCarley's group for assistance with the magnetic susceptibility apparatus, and to R. G. Barnes for the NMR examination. The earlier part of this research was supported by Ames Laboratory of the U.S. Atomic Energy Commission, and the later work, by the National Science Foundation, Solid State Chemistry, Grant DMR-8318616.

Supplementary Material Available: A table of information on the single-crystal diffraction study (1 page); a table of observed and calculated structure factors for $Sc_{0.93}I_2$ (1 page). Ordering information is given on any current masthead page.

---

(20) Hariharan, A. V.; Powell, D. R.; Jacobson, R. A.; Franzen, H. F. *J. Solid State Chem.* **1981**, *36*, 148.

(21) Lachgar, A.; Dudis, D. S.; Corbett, J. D. Unpublished research.

Additions and Corrections

1989, Volume 28

Kattesh V. Katti and Ronald G. Cavell*: First Examples of an Isomeric Methylene-Bridged Free Phosphano Phosphoranimine and Metalated Phosphano Phosphoranimine. Synthesis, Characterization, and Isomerization of the Heterodifunctional Ligand $Me_3SiNPPh_2CH_2PPh_2$ and the Transmetalation to the Titanium Derivative $(\eta^5\text{-}C_5H_5)\text{-}TiCl_2NPPh_2CH_2PPh_2$.

Page 416. In the second column, the heading "Thermal Isomerization of **2R**" and the first sentence of text following should read as follows:

Thermal Isomerization of 2. A sample of **2** (1.512 g, 3.2 mmol) (as the waxy solid recovered from the reaction of dppm and Me_3SiN_3 and *without* recrystallization) was placed in a Schlenk tube and connected to a dynamic vacuum line....

The confusing consequence of this error in the experimental description is that the recrystallized form, **2R**, can be thermally isomerized, which is not the case. In fact, only the isomeric distribution of the waxy solid product **2** can be changed by heating **2** to 160 or 240 °C etc. and this gives the form that we have labeled as **2D**. All statements made in the text with regard to **2D** (and **2** without the modifying label) are correct.—Kattesh V. Katti and Ronald G. Cavell