Additions and Corrections

Efficient Photoinduced Charge Separation in Layered Zirconium Viologen Phosphonate Compounds [*J. Am. Chem. Soc.* **1993**, *115*, 11767–11774]. LORI A. VERMEULEN, JONATHAN L. SNOVER, LINDA S. SAPOCHAK, AND MARK E. THOMPSON*

In our recent paper we reported that the powder X-ray diffraction pattern shown in Figure 1 was due to a chloride-containing compound, $Zr(O_3PCH_2CH_2(bipyridinium)CH_2CH_2PO_3)Cl_2$. We have found that this X-ray diffraction pattern is in fact due to a fluoride compound, present in the sample. The structure of this fluoride compound has been determined by Rietveld analysis of the powder data and indicates that the stoichiometry is $(ZrF_3)_2(O_3PCH_2CH_2(bipyridinium)CH_2CH_2PO_3)\cdot 2H_2O$. Ion microprobe analysis of single crystals (1–5 μm) of this material supports this formula (Zr:F:P = 1:3:1). Fluoride ion was only present in the preparation of microcrystalline samples; the thin films discussed in this paper contain no fluoride ion. Microcrystalline samples of the chloride and bromide compounds prepared in the absence of fluoride ion have very poor crystallinity but have the same relative photochemical response as the materials reported in our paper [*i.e.* **ZrPV(Cl)** is photoreduced more readily than **ZrPV(Br)**]. Similar reactivity for the materials without the fluoride impurity suggests that the reported photochemistry is not related to the fluoride compound. The structural study of $(ZrF_3)_2(O_3PCH_2CH_2(bipyridinium)CH_2CH_2PO_3)\cdot 2H_2O$ will be reported in the near future (M. D. Poojary, L. A. Vermeulen, M. E. Thompson, E. Vicenzi, and A. Clearfield, manuscript in preparation).