aluminum into some of the sodalite units. We consider the dehydration of $Ca(OH_2)_x^{2+}$ to $Ca(OH)^+$ and the stabilization of the anionic aluminum species to be important in the dealumination process as observed with divalent ions as opposed to monovalent cation-exchanged zeolite A. The terminal OH^- group is available for nucleophilic attack at the aluminum site to help initiate the dealumination reaction. Infrared and X-ray and neutron diffraction studies of these samples are presently being pursued to determine the role of the divalent species in the dealumination process.

Acknowledgment. Discussions with A. J. Vega, D. B. Chase, and L. Abrams are appreciated. The excellent technical assistance of R. F. Carver is acknowledged.

Registry No. $Al(OH)_4^-$, 14485-39-3; ^{29}Si, 14304-87-1; Al, 7429-90-5.

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An Improved Synthesis of TeF_5OF

Sir:

Recently, we reported[1] the synthesis and characterization of the previously unknown hypofluorite TeF_5OF, according to

$$\mathrm{CsTeF_5O} + \mathrm{FOSO_2F} \xrightarrow{-45\ ^\circ\mathrm{C}} \mathrm{CsSO_3F} + \mathrm{TeF_5OF}$$

This reaction represented a new synthetic route to hypofluorites, but it did entail drawbacks. Among these were the need for careful low-temperature control, long reaction times (9 days), the requirement for excess $CsTeF_5O$, and the less than optimal yields (~70%). Furthermore, the necessity to prepare and handle the treacherously explosive $FOSO_2F$[2,3] was a disadvantage.

We now report a new and improved process for the synthesis of TeF_5OF by means of the reaction

$$\mathrm{B(OTeF_5)_3} + 3\mathrm{F_2} \xrightarrow{\Delta} 3\mathrm{TeF_5OF} + \mathrm{BF_3}$$

This reaction provides TeF_5OF in 80–95% yield at 100–115 °C after 1–3 days. Either stainless steel or Monel reactors may be used with the latter, giving consistently higher yields in shorter reaction times. Separation of byproducts, mainly BF_3 and TeF_5OH, is easily accomplished. Since commercial F_2 is the fluorinating agent, the need for synthesizing the hazardous $FOSO_2F$ is eliminated. To our knowledge this preparation of TeF_5OF is the first example of the formation of a hypofluorite involving the fluorinative scission of a boron–oxygen bond.

Experimental Section. Materials and Apparatus. Volatile materials were manipulated in a stainless steel vacuum line equipped with Teflon-FEP U-traps, 316 stainless steel bellows-seal valves, and a Heise Bourdon tube-type pressure gauge. Pentafluorotelluric acid was prepared from telluric acid, $Te(OH)_6$, and fluorosulfuric acid, HSO_3F.[1] Reaction of BCl_3 with excess TeF_5OH at ambient temperature provided $B(OTeF_5)_3$.[4] Spectroscopic measurements were performed with equipment and techniques previously described.[1]

Synthesis of TeF_5OF. Caution! While we have not encountered any difficulties or incidents in preparing and handling TeF_5OF, its hypofluorite nature demands treatment as a potentially violent oxidizer. Suitable safety precaustions must be followed.

In the dry-nitrogen atmosphere of the glovebox, a sample of $B(OTeF_5)_3$ (0.95 mmol) was weighed and transferred to a prepassivated 16-mL Monel pressure cylinder equipped with a Monel Hoke valve. The cylinder was transferred to the vacuum line, evaluated, and cooled to −196 °C, and F_2 (~7 mmol) was added. The closed cylinder was warmed to ambient temperature before being placed in an oven at 115 °C. After 24 h the cylinder was cooled to ambient temperature before it was cooled further to −196 °C (*hot* Monel should not be cooled directly to −196 °C since it may rupture catastrophically). The excess F_2 was pumped away through a scrubber, and then, while the reactor was allowed to warm from −196 °C to room temperature, the volatile products were separated by fractional condensation through a series of U-traps cooled at −78, −126, and −196 °C. In the lowest temperature trap were BF_3 and a little TeF_6 (1.05 mmol total), while the trap at −78 °C contained TeF_5OH (0.05 mmol). Retained in the trap cooled at −126 °C was pure TeF_5OF (2.68 mmol, 94% yield based on $B(OTeF_5)_3$). In all respects the properties of this material matched those reported.[1]

With use of the Monel vessel and a 100 °C reaction temperature for 22 h, the yield of TeF_5OF was 55%. This yield had increased to 95% when the reaction was examined after a total of 67 h at 100 °C. When a 30-mL stainless steel cylinder was employed as the reactor at 100 °C, the yield of TeF_5OF was 74% after 64 h and 80% after 83 h. At 115 °C in the stainless steel reactor, the hypofluorite was formed in 43% yield after 24 h and 81% after 51 h total. No unreacted $B(OTeF_5)_3$ remained at the conclusion of these reactions.

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