

aluminum into some of the sodalite units. We consider the dehydration of $\text{Ca}(\text{OH}_2)_x^{2+}$ to $\text{Ca}(\text{OH})^+$ 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.

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

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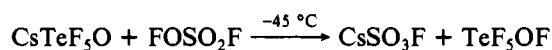
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An Improved Synthesis of TeF_5OF

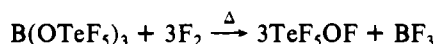
Sir:

Recently, we reported¹ the synthesis and characterization of the previously unknown hypofluorite TeF_5OF , according to



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 ($\sim 70\%$). Furthermore, the necessity to prepare and handle the treacherously explosive $\text{FOSO}_2\text{F}^{2,3}$ was a disadvantage.

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



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, $\text{Te}(\text{OH})_6$, and fluorosulfuric acid, HSO_3F .¹ Reaction of BCl_3 with excess TeF_5OH at ambient temperature provided $\text{B}(\text{OTeF}_5)_3$.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 precautions must be followed.

In the dry-nitrogen atmosphere of the glovebox, a sample of $\text{B}(\text{OTeF}_5)_3$ (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 $\text{B}(\text{OTeF}_5)_3$). 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 $\text{B}(\text{OTeF}_5)_3$ remained at the conclusion of these reactions.

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(4) Sladky, F.; Kropshofer, H.; Leitzke, O. *J. Chem. Soc., Chem. Commun.* 1973, 134.

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(1) Schack, C. J.; Wilson, W. W.; Christe, K. O. *Inorg. Chem.* 1983, 22, 18.

(2) Cady, G. H. *Inorg. Synth.* 1968, 11, 155.

(3) Shreeve, J. M. *Adv. Inorg. Chem. Radiochem.* 1983, 26, 119.

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