863, and 592 cm^{-1} are characteristic of the $-SF_5$ group,[2,10,11] and a band at 702 cm^{-1} is also attributable to $-SF_5$.[2,11] A peak at 795 cm^{-1} probably arises from an S—F stretch in $=SF_2=$. Peaks at 725 and 663 cm^{-1} have not been assigned.

The mass spectrum (Table I) was obtained on a Hitachi Perkin-Elmer RMU-6A spectrometer. The molecule ion peak was present at *m/e* 352 with a peak at *m/e* 354, about 13% as intense as the *m/e* 352 peak. This was consistent with three sulfurs in the molecule. Smaller peaks corresponded to the ions containing ^{33}S and ^{15}N isotopes. The largest peak was that for SF_5^+ with the second largest being the SF_3^+ peak. This differs from SF_5NH_2 and $SF_5N{=}SF_2$ mass spectra in which the SF_3^+ peak was more intense than the SF_5^+ peak.

TABLE I

MASS SPECTRUM OF $(SF_5N{=})_2SF_2$

m/e	Rel intens	Species
46	5.5	NS^+
51	2.3	SF^+
65	2.2	NSF^+
70	8.8	SF_2^+
83	2.1	S_2F^+
84	2.0	NSF_2^+
85	2.1	SiF_3^+
89	27.5	SF_3^+
91	1.6	$S^{34}F_3^+$
102	1.3	$S_2F_2^+$
103	5.5	NSF_3^+
108	1.0	SF_4^+
127	100.0	SF_5^+
128	0.8	$S^{33}F_5^+$
129	5.0	$S^{34}F_5^+$
206	1.1	$S_2F_6N_2^+$
333	8.8	$S_3F_{11}N_2^+$
335	1.1	$S^{34}S_2F_{11}N_2^+$
352	5.5	$S_3F_{12}N_2^+$ (molecule ion)
354	0.74	$S^{34}S_2F_{12}N_2^+$

Acknowledgment.—This work was supported by the National Science Foundation under Grant No. GP-4974.

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A Simple Preparation of Oxygen Difluoride in High Yield

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Received January 13, 1969

Oxygen difluoride is commonly prepared by bubbling fluorine through a 1-cm layer of 0.5 *N* NaOH with yields up to 50%.[2] A modification of this technique has been reported to produce OF_2 in a yield of 86.79%.[3] The compound has also been produced by various electrolytic methods.[4,5]

In view of recent successes in using CsF or KF to catalyze the preparation of hypofluorites,[6,7] it was thought that OF_2 could be prepared by allowing H_2O to react with F_2 in the presence of an alkali fluoride. This was found to be the case. When F_2 was admitted to a glass bulb containing H_2O and excess CsF, KF, or NaF, a reaction occurred producing OF_2 in yields that ranged from 55 to 80%. Yields were usually above 70% when CsF or KF was used but were generally lower (about 60%) when NaF was employed. Similar experiments were performed in which the alkali fluoride was replaced by powdered NaOH or CaF_2, but in neither case was any appreciable amount of OF_2 obtained.

As a laboratory preparation, this method has the advantage of giving a high yield of OF_2 from a simple reaction. Moreover, the yield is not especially sensitive to either reaction time or variations in the molar ratios of the reactants. Reaction times varied from 15 min to 3 hr, fluorine to water molar ratios varied from 2:1 to 4:1, and alkali fluoride to water molar ratios varied from 2:1 to 100:1. The product is obtained in a very pure state, since HF and SiF_4 (formed from attack of HF on glass) are removed by combining with the catalyst. Oxygen, formed as a by-product, and unreacted fluorine are easily removed by pumping at −196°.

Experimental Section

Standard vacuum-line techniques were employed in all preparations.

When KF was used as a catalyst, H_2O was conveniently added in the form of the commercially available dihydrate, $KF \cdot 2H_2O$. In a typical experiment, 0.327 g (3.48 mmol) of $KF \cdot 2H_2O$, 0.807 g (13.9 mmol) of anhydrous KF, and 0.544 g (14.3 mmol) of F_2 were allowed to react for 1.5 hr in a 500-ml glass bulb. A yield of 5.18 mmol (74%) of OF_2 was obtained. The identity and purity of the product were established from the characteristic infrared and ^{19}F nmr spectra and from the molecular weight (calcd, 54.0; found, 53.4). When CsF or NaF was used as a catalyst, water was added by condensing the desired quantity onto the surface of the anhydrous, powdered catalyst. Since the alkali fluoride tended to cake as the reaction progressed, a few stainless steel balls were added to the glass bulb. Occasional agitation of the balls kept the catalyst powdered. While pumping away O_2 and F_2 from OF_2 at −196°, the pumping time was kept as short as possible in order to minimize loss of product, since OF_2 has an appreciable vapor pressure (~1 mm) at −196°.

Acknowledgments.—The authors are grateful to Professor George H. Cady of the University of Washington for helpful suggestions in connection with this work. A. H. B. was a participant in the National Science Foundation Undergraduate Research Program, Grant No. GY4235.

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