The titrations were made at room temperature in a high beaker, provided with a stirrer, in carbon dioxide-free air.

The results were calculated by the Henderson–Hasselbalch formula

$$\log K = \log \frac{x}{A - x} - p\text{H}$$

where A is the initial quantity of hypobromous acid and x is the quantity of added alkali, both in moles.

The results of one experiment are reported in Table I. In this experiment 49.84 cc. of 0.0235 molal hypobromous acid was titrated by 0.0255 molal sodium hydroxide at 20°.

TABLE I

NaOH, cc.	$x/(A-x)$	Mv.	pH	$10^9 K$
00		153.2	4.97	
1	0.0224	270.4	7.02	(2.14)
2	.0458	288.6	7.36	2.00
4	.096	306.9	7.67	2.05
6	.1511	318.4	7.87	2.04
8	.212	325.8	8.01	2.07
12	.3546	338.1	8.22	2.14
16	.5335	348.4	8.40	2.12
20	.771	358.0	8.58	2.03
25	1.193	368.0	8.75	2.13
30	1.890	379.7	8.96	2.07
34	2.866	390.0	9.14	2.11
36	3.645	395.8	9.25	2.05
				2.07

The mean value K_{HOBr} in this experiment is 2.07×10^{-9}. The titration of 0.0437 molal hypobromous acid by 0.046 molal alkali has given mean value $K_{\text{HOBr}} = 2.04 \times 10^{-9}$.

The probable mean value of all (five) experiments is $K_{\text{HOBr}} = 2.06 \times 10^{-9}$ at 20°.

The procedure was controlled by determining the dissociation constant of hypochlorous acid. The value obtained, $K_{\text{HOCl}} = 3.16 \times 10^{-8}$ at 20°, is in agreement with the values of Britton and Dodd[7] and Ingham and Morrison.[7]

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The Preparation of Benzotrifluoride

BY J. H. SIMONS AND C. J. LEWIS

Benzotrifluoride is made in yields approaching the theoretical and without the formation of tars or resins by the action of anhydrous hydrogen fluoride on benzotrichloride. Previous methods[1-3] have used antimony trifluoride, but the authors have found that the reaction in a copper vessel with hydrogen fluoride gives fewer side reactions than when antimony trifluoride is used either alone or with hydrogen fluoride.

The benzotrichloride is placed in a copper flask kept at 0° in an ice-bath. Gaseous hydrogen fluoride is admitted slowly and continuously through a copper tube that extends to the bottom of the flask, and the exit gases escape through another copper tube from the top of the flask. The reaction mixture is stirred or agitated during the course of the reaction.

The exit gases consist chiefly of hydrogen chloride with some hydrogen fluoride and small amounts of benzotrifluoride. As the reaction nears completion, the concentration of hydrogen fluoride and the desired product increase; and they should be trapped in a copper trap kept in a salt-ice bath. The exit gases can be analyzed qualitatively for hydrogen chloride and hydrogen fluoride and relative amounts estimated by the simple procedure of testing with beads of aqueous solutions of silver nitrate and calcium chloride held in nichrome wire loops. Although the reaction can be carried to 95% completion or further, the concentration of hydrogen fluoride in the exit gases rises at about 70% completion, and it is economical to stop the reaction at this point, separate the benzotrifluoride from the mixture by distillation, and return the residue (consisting chiefly of mono- and difluorides) with a new supply of benzotrichloride to the reaction flask for further conversion.

A charge of 500 g. of starting material required about seventy-two hours for the conversion. When the reaction is stopped, the temperature is allowed to rise to room temperature, and a small amount of sodium fluoride is added to remove any hydrogen fluoride present. After standing for a short time with occasional stirring, it is ready for distillation.

Yields of from 75 to 95% were obtained. Other than mechanical losses the chief cause for less than theoretical yield is the amount of the product escaping as vapor with the exit gases.

The benzotrifluoride made by this method was used to make trifluoroacetic acid by the method described by Swartz.[1] With careful technique a yield of about 50% of acid from the starting material, benzotrichloride, can be obtained.

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(1) Swartz, *Bull. acad. roy. sci. belg.*, **35**, 375 (1898).

(2) Aelony, THIS JOURNAL, **56**, 2063 (1934).

(3) Booth, Elsey and Burchfield, *ibid.*, **57**, 2066 (1935).