Pure (99.5%) nitrogen gas was first passed through concentrated sulfuric acid and bubbled through 20 g. of the pure hexane in a distilling flask. The arm of this flask was connected with a bubbler containing water with an excess of bromine. Five hours were required to vaporize the hexane, and examination of the bromine solution revealed no benzene bromides. Repeating with the bromine solution kept below zero and the hexane vaporized in the course of forty hours, no benzene bromides were found. A large amount of the hexane was recovered.

Repeating under the same conditions, but with the bromine solution exposed to a 100-watt Mazda lamp, again no benzene bromides were obtained.

When one gram of benzene was added to the hexane, and the vaporization carried out in the cold and in the dark, some benzene bromides were readily obtained.

The production of the derivatives mentioned in the literature can therefore be traced to hexane slightly contaminated with benzene.

This work was done at the suggestion of Dr. M. S. Kharasch, of the University of Chicago.

CONTRIBUTION FROM THE
DEPARTMENT OF CHEMISTRY
UNIVERSITY OF CHICAGO
CHICAGO, ILLINOIS.

FRANCIS M. PARKER

RECEIVED MAY 23, 1930
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Determination of Halogens in Organic Compounds.—We wish to thank Professor Robertson[1] for calling our attention to his papers, which we overlooked, probably because we were searching for a general method for the determination of chlorine, bromine and iodine and Robertson's method is, as he states, not suitable for iodine compounds.

We have tested his method using samples of about 0.15 g. of bromobenzene and obtained low results, a tendency to which Robertson also calls attention. Duplicates checked less closely than by our method.

The use of sodium peroxide instead of arsenite is obviously optional in either method, but the latter was decided upon after a trial of both reagents.

The time required depends upon whether the gravimetric or volumetric method is used. We needed eighty-five minutes by the gravimetric method with Robertson's apparatus.

The principal advantages of our method[2] are: (1) it is a general method applicable to compounds of chlorine, bromine and iodine.

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[1] Robertson, THIS JOURNAL, **52**, 3023 (1930).

[2] Thompson and Oakdale, *ibid.*, **52**, 1195 (1930).

(2) It can be used with low-boiling compounds such as ethyl bromide. (3) Metals in the residue may be very easily determined. (4) The accuracy is greater than in Robertson's method, as shown by a comparison of analyses. (5) The use of oxygen and platinized asbestos is never necessary. (6) There is no danger of the reaction becoming too violent or of the projection of particles onto the walls of the flask. (7) The time required is less, particularly because it has been found since publication of the last paper by Willard and Thompson,[3] that the apparatus described by them for the micro determination of halogens can also be used for samples as large as 0.1–0.2 g. This results in a saving of time as well as of space required for the apparatus. For samples of 0.01–0.02 g. an apparatus about half the size of this, or only 28 cm. high, works perfectly.

CONTRIBUTION FROM THE
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U. O. OAKDALE

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COMMUNICATIONS TO THE EDITOR

THE HEAT OF FORMATION OF MOLECULAR OXYGEN

Sir:

In a recent communication Copeland[1] has reported a value for the heat of formation of molecular oxygen of 165,000 cal. We have been engaged in a similar determination by an experimental method which differs only in minor details from the method used by Copeland. The results of our first determinations were in substantial agreement with those reported by Copeland, the average of a number of runs being about 160,000 cal. When we checked the method for possible errors we discovered that we were getting spurious heat effects in the calorimeter. When we took precautions to eliminate these heat effects we obtained as the average of a number of fairly consistent runs 131,000 cal. We do not find any evidence that metastable atoms reach the calorimeter.

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[3] Willard and Thompson, THIS JOURNAL, **52,** 1893 (1930).

[1] Copeland, *ibid.*, **52,** 2581 (1930).