NOTES

Polyethylene Container in the Schöniger Oxygen Flask Combustion

A survey of the recent literature shows the increasing popularity of the Schöniger oxygen flask method for the decomposition of materials prior to elemental analysis. Decomposition of 50-mg. samples of fluorine-containing polymers in Pyrex flasks results in severe attack on the containers. Recent work in this laboratory has demonstrated that an ordinary polyethylene bottle may be used in place of the conventional glass flask. Combustion in this manner proceeds smoothly with no attack. Encouraged by the safety, simplicity, and economy of this approach, as well as the inherent inertness of the container, all Schöniger combustions are now conducted in this manner.

Apparatus. The polyethylene bottle and combustion assembly are shown in Figure 1. This bottle is an ordinary 1-qt. reagent container. The combustion assembly consists of a 24/40 ⅋ glass stopper, to the bottom of which is sealed a length of No. 18 platinum wire. Platinum mesh is used to hold the sample. Commercially available filter flags (A. H. Thomas, Cat. No. 6471-F) are used. The safety aspects of the combustion were carefully considered. On several occasions an improperly centered combustion unit caused ignition of the bottle. The procedure finally adopted involves surrounding the bottle with ice in a 3-liter stainless steel beaker and conducting the combustion behind a shield. This minimizes fire hazard and also the chance of a violent ejection of the combustion assembly from the flask. Many combustions have been carried out safely in this manner.

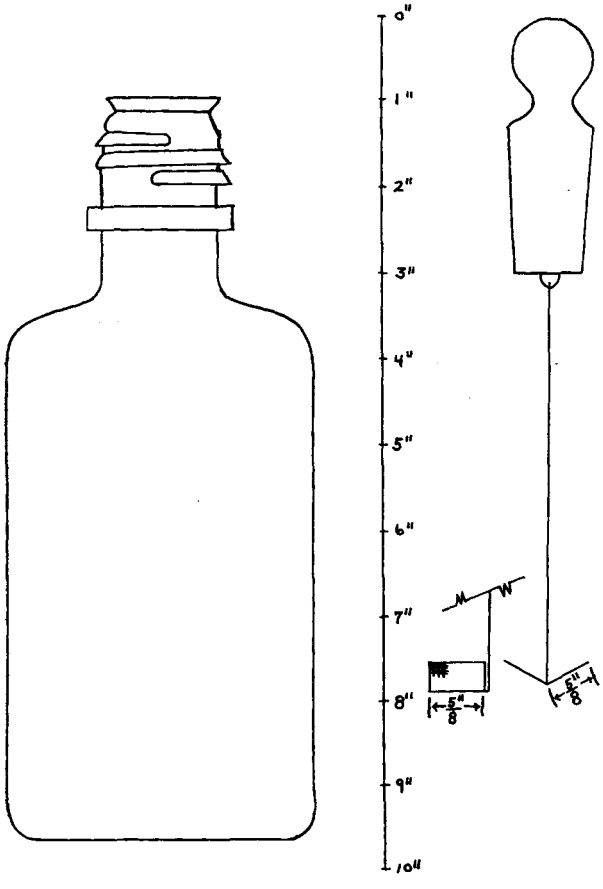

Figure 1.

TABLE I

Polymer or film	Fluorine content, % Theory	Found
Teslar* PVF film	41.26	41.46
Polyvinylidene fluoride	59.34	59.59
Teflon* TFE–fluorocarbon resin	76.0	74.1
Teflon* FEP–fluorocarbon film	76.0	73.9
Viton* A–synthetic rubber	65.99	65.51
Kel-F†–elastomer	48.9	50.9

* Du Pont trademark.

† M. W. Kellogg trademark.

Procedure. The procedure described is for fluorine-containing polymers or films as this was the most frequent application. The absorbing solution (50 ml. of 0.1*N* NaOH) is added to the bottle and oxygen passed in at a moderate rate for 1 to 2 min. After insertion of a 24/40 ⅋ glass stopper, the bottle is surrounded with ice in a stainless steel beaker and placed behind a shield. The weighed sample (5–50 mg.) is transferred to a filter flag which is then folded and positioned in the holder. The fuse is lighted, the stopper removed, and the combustion assembly inserted. After combustion is complete (about 10 sec.) the bottle is shaken at intervals for 30 min. Titration with thorium nitrate is conducted as described by Sweetser.[1] Results are given in Table I.

Discussion. Results for perfluorinated polymers are not entirely satisfactory. Steyermark[2] states that low results are caused by the formation of tetrafluoromethane. Our experience indicates that best results are obtained by burning 5 mg. samples of finely divided material using 50 mg. of powdered sucrose as an accelerator. Double burnings using only one absorbing solution are recommended when larger samples are required.

The glass stopper of the combustion assembly showed no evidence of attack by fluoride; results indicate that error from this source is insignificant.

The polyethylene bottle in no way limits the applicability of the Schöniger combustion. Although the data presented are for solid materials only, liquids have been analyzed by established procedures.

References

1. Sweetser, P. B., *Anal. Chem.*, **28**, 1766 (1956).
2. Steyermark, A., *Microchem. J.*, **3**, 523 (1959).

Howard R. Kanner
Joseph N. Carmody

Film Department
Yerkes Research Laboratory
E. I. du Pont de Nemours & Co., Inc.
Buffalo, New York

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