directed by Feuge et al. and stored in the chamber of a Machlett automatic buret.

HBr reagent was also prepared from HBr generated from a saturated aqueous solution of KBr on the addition of concentrated sulfuric acid. The HBr generated by the reaction was dried by passing it through concentrated sulfuric acid, and the bromine vapors, resulting from the oxidation of HBr by sulfuric acid, were condensed before the gas stream was passed into dry toluene. Qualitative tests for Br_2 in the toluene solution were negative. This solution was also stored in the chamber of a Machlett automatic buret. Continuous loss of HBr was observed with each preparation we studied.

In each case the automatic buret was equipped with a three-way Teflon stopcock, and the delivery tip was modified to receive a no. 18 gauge hypodermic needle.

Titration of HBr

A Teflon-coated magnetic bar and two drops of phenolphthalein indicator were placed in a 250 ml narrow neck bottle, and the bottle was warmed on a water bath at 50-55 C and immediately stoppered with a serum bottle stopper with a retractable sleeve. The bottle was then cooled to room temperature to reduce the pressure inside to facilitate subsequent operations.

The hypodermic needle attached to the automatic buret was plunged through the serum stopper, and a metered quantity of HBr solution was delivered into the bottle and the needle withdrawn. The bottle was then placed above a rotating magnetic bar, and standard KOH (0.1077 N in ethyl alcohol) was delivered into the bottle through a hypodermic needle by means of a mechanically driven and calibrated infusion pump (Aminco No. 4-2340, Patent 2,925,198) until a faint pink color developed and remained permanent. The delivery of the KOH solution (either continuous or intermittent) was positive.

Very careful observations revealed no loss of HBr to the atmosphere under the conditions used. No stopper was reused for a second titration, and in no instance did the hypodermic needle serve as an opening to the atmosphere.

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Preparation of Methyl Esters of Long Chain Fatty Acids

Sir: For some time we have been successful in using $HClO_4$ in place of boron trifluoride for transesterification of triglycerides of fatty acids to form corresponding methyl esters. This has several advantages. Perchloric acid is commercially more available and less expensive than boron trifluoride, and under the following reaction conditions perchloric acid is not hazardous. The transmethylation technique is (AOCS Method Ce 2-66): Introduce 0.5 g oil or fat into 50 ml round bottom reaction flask. Add 0.8 ml 0.5 N methanolic NaOH. Attach condenser and reflux on steam bath until fat globules dissolve (10 min). Add 0.8 ml 60% $HClO_4$ through condenser and continue refluxing for 2-3 min. Add 5 ml heptane and boil 1 min. Remove from head and add enough saturated NaCl solution to float heptane solution of methyl esters into neck of flask. Transfer as much heptane solution as possible into dry test tube. Add small amount of anhydrous Na_2SO_4. Dry heptane solution is ready to be injected directly into gas chromatograph.

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