AOAC Official Method 933.03
Fluorine (Total) in Pesticide Formulations

Distillation Method
First Action 1933
Final Action

(Applicable to H_2O-soluble or H_2O-insoluble insecticides in absence of gelatinous SiO_2, B, and Al.)

A. Reagents

(**a**) *Sodium alizarin sulfonate indicator*.—Dissolve 0.1 g sodium alizarin sulfonate in 200 mL H_2O.

(**b**) *Thorium nitrate solution*.—Approximately 0.0125M. Standardize in terms of g F/mL by titrating F obtained by distillation from standard NaF as in **933.03B**. In standardizing for use with **B**(**b**), add 5 mL saturated $KMnO_4$ solution in addition to other reagents in distillation flask.

B. Determination

(**a**) *In absence of organic matter*.—Weigh test portion containing ca 0.09 g F, and with aid of little H_2O transfer to 250 mL Claisen distillation flask (B) containing 12 glass beads. Adjust to ca 30 mL and close flask with 2-hole rubber stopper, through which pass thermometer (D) and 4 mm glass tube, both of which extend into solution. (The 4 mm glass tube extends ca 5 cm above rubber stopper and by means of rubber tube, E, connects still with 1 L Florence flask [A] containing H_2O for steam generation. Flask is equipped with steam discharge, H, and pressure tube, G. *See* Figure **933.03**.)

Bring H_2O in steam generating flask to boil with pinchcock, F, in release tube open. Connect distilling flask to condenser, C, and add 25 mL H_2SO_4 through top of 4 mm tube, using pipet or special funnel. With pinchcock, F, open, connect rubber tubing to 4 mm tube. Light burner under Claisen flask. Regulate flow of steam by adjusting burner flames and pinchcock, F, so that volume of solution is held constant and temperature in flask, B, is kept at 145–150°C. Continue distillation until 400 mL distillate collects. Dilute to 500 mL in volumetric flask, transfer 50 mL aliquot to tall-form 150 mL beaker, and add 5 drops indicator, **A**(**a**). Adjust acidity with 1% NaOH solution (w/v) and HCl (1 + 249) until pink just disappears. Add 2 mL of the HCl, and titrate with 0.0125M $Th(NO_3)_4$ to permanent pink, using buret graduated in 0.05 mL.

(**b**) *In presence of organic matter*.—In presence of moderate amounts of organic matter, transfer test portion containing ca 0.09 g F and containing ≤0.2 g organic matter, with aid of little H_2O, to 250 mL Claisen distillation flask containing 12 glass beads. Add 5 mL saturated $KMnO_4$ solution, adjust to ca 30 mL, and proceed as in (**a**), beginning "…close flask with 2-hole rubber stopper,…".

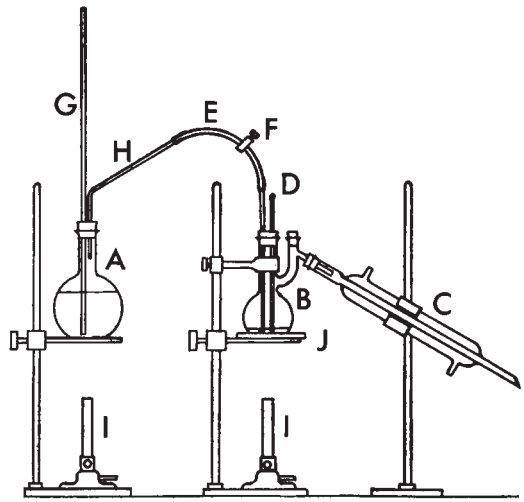

Figure 933.03—Apparatus for determining fluorine.

In presence of large amounts of organic matter, transfer test portion to medium size Pt dish, add 15 mL H_2O and enough F-free CaO to make mixture distinctly alkaline to phenolphthalein, mix with glass rod, and evaporate to dryness on steam bath and in oven at 105°C. Ignite at low heat, preferably in furnace (≤600°C), until organic matter is thoroughly charred. Pulverize any lumps present in charred ash with glass rod, transfer to 250 mL Claisen distillation flask by brushing, and finally wash out dish with 30 mL H_2SO_4 (1 + 9). Except to add 22 mL instead of 25 mL H_2SO_4, proceed as in (**a**), paragraph 2.

(*Note*: If coating of precipitated SiO_2 forms on inside of distillation flask, remove by treatment with hot concentrated alkali solution, as it is capable of retaining F during distillation of some products and giving it up, at least in part, in later distillations.)

References: *J. Am. Chem. Soc*. **55,** 1741(1933).
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JAOAC **21,** 459(1938); **53,** 378(1970).

CAS-7782-41-4 (fluorine)

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