adding SiO_2, in which case formation of SiO_2 skeleton is conclusive evidence of presence of fluosilicate. In ash contg appreciable amt of SiO_2, H_2SO_4 liberates SiF_4 rather than HF. Therefore presence of fluosilicate, not fluoride, is indicated.

Ref.: Mon. Sci. (4th Ser.) **9,** Part 1, 324(1895).

C. Qualitative Test II

Ash filter contg insol. portion from **975.28A** in Pt crucible, mix with little pptd SiO_2, and add 1 mL H_2SO_4. Cover crucible with watch glass from underside of which drop of H_2O is suspended, and heat 1 hr at 70–80°, keeping watch glass well cooled. The H_2O decomposes SiF_4 formed, leaving gelatinous deposit of SiO_2 and etching ring at periphery of drop of H_2O. Test filtrate for H_3BO_3 as in **970.33**.

D. Quantitative Method

See **944.08**.

931.08 Formaldehyde in Food

First Action

(*See also* **964.21**.)

A. Preparation of Sample

If sample is solid or semisolid, macerate 100 g with 100 mL H_2O in mortar. Transfer to 800 mL Kjeldahl flask, acidify with H_3PO_4, add 1 mL excess, connect with condenser thru trap, and slowly distil 50 mL. For milk, dil. 100 mL with 100 mL H_2O, and acidify and distil as for solids. With other liq. foods, acidify 200 mL and distil as for solids.

B. Chromotropic Acid Test

(a) *Reagent.*—Prep. satd soln of 1,8-dihydroxynaphthalene-3,6-disulfonic acid (ca 500 mg/100 mL) in ca 72% H_2SO_4 (pour 150 mL H_2SO_4 into 100 mL H_2O and cool). Soln is light straw-colored.

(b) *Test.*—Place 5 mL reagent in test tube and add, with mixing, 1 mL distillate, **931.08A**. Place in boiling H_2O bath 15 min, and observe during heating period. Presence of HCHO is indicated by appearance of light to deep purple (depth of color depending on amt of HCHO present).

Ref.: Z. Anal. Chem. **110,** 22(1937).

C. Hehner-Fulton Test

(*Caution:* See safety notes on sulfuric acid and bromine.)

(a) *Oxidizing soln.*—To cold H_2SO_4 add, in small portions, equal vol. satd Br-H_2O, cooling thruout operation.

(b) *Test.*—To 6 mL cold H_2SO_4 add 5 mL distillate, **931.08A**, slowly and with cooling. Place 5 mL mixt. in test tube, and add, slowly and with cooling, 1 mL aldehyde-free milk, then 0.5 mL oxidizing soln. Mix. Purplish-pink indicates HCHO.

Ref.: Ind. Eng. Chem. Anal. Ed. **3,** 199(1931).

CAS-50-00-0 (formaldehyde)

970.35 Formic Acid in Food

Final Action

See **938.09**; **971.11**.

957.08 Hydrogen Peroxide in Milk

Qualitative Color Test

Final Action

(a) *Reagent.*—Dissolve 1 g V_2O_5 in 100 mL H_2SO_4 (6 + 94).

(b) *Test.*—Add 10–20 drops reagent to ca 10 mL sample and mix. Pink or red indicates H_2O_2.

Ref.: JAOAC **40,** 789(1957).

CAS-7722-84-1 (hydrogen peroxide)

942.09 Monochloroacetic Acid in Liquids and Preservatives

Qualitative Tests

Final Action

A. Optical-Crystallographic Properties of Barium Salt

(Applicable to com. preservatives)

Dil. 4–5 mL sample to 100 mL, add 6 mL H_2SO_4 (1 + 1), and ext with equal vol. ether in separator. If emulsions form, ext in continuous extractor 1 hr. Transfer ether ext to separator, add few drops phthln and 5 mL 0.1*N* $Ba(OH)_2$, and shake 30 sec. If aq. layer takes on pink typical of phthln, filter thru paper into small beaker. Add ca 0.05*N* HOAc until colorless and evap. to 1–2 mL on steam bath. Let remaining liq. evap. spontaneously in air and finally in desiccator. If 5 mL 0.1*N* $Ba(OH)_2$ does not give pink aq. layer, add 5 mL more before sepg. Repeat extn with $Ba(OH)_2$ soln several times or until pink soln is obtained, evapg each Ba soln in sep. beaker. Examine crystals under polarizing microscope.

Barium monochloroacetate monohydrate crystallizes from H_2O in plates, many of which are hexagonal in habit and frequently form in overlapping layers. Even in material that has been finely powd for microscopic examination, pointed terminations of the plates, often in pairs, can be observed. In parallel polarized light (crossed nicols) extinction is parallel and sign of elongation is neg. on more elongated plates. Plates invariably extinguish sharply with crossed nicols and therefore interference figures are not observed in convergent polarized light (crossed nicols). Since plates persistently lie in one orientation, significant refractive indices are detd by statistical method, lowest and highest indices resp. being measured on plates showing max. double refraction. These 2 indices are therefore arbitrarily designated as $n\alpha$ (min. value) and $n\gamma$ (max. value). Two significant refractive indices are: $n\alpha$ = 1.582 and $n\gamma$ = 1.611, both ±0.002, frequently shown on the platey fragments.

B. Indigo Test

(*Caution:* See safety notes on distillation, flammable solvents, and diethyl ether.)

(a) *Commercial preservatives.*—Dil. 2 mL sample to 100 mL, add 3 mL H_2SO_4, and shake with 100 mL ether. Add 3 mL *anthranilic acid reagent* (1 g + 0.3 g NaOH/50 mL) to ether ext, evap. at low temp., filter off any insol. matter, and proceed as in (c), beginning "Test with litmus paper."

(b) *Carbonated beverages, orange juice, and wine.*—Acidify 100 mL sample with 3 mL H_2SO_4 and ext, using either continuous extractor or separator. Add 3 mL anthranilic acid reagent, (a), to ether ext and evap. at low temp. If any insol. matter seps, filter thru small wet paper. To clear liq. in 50 mL