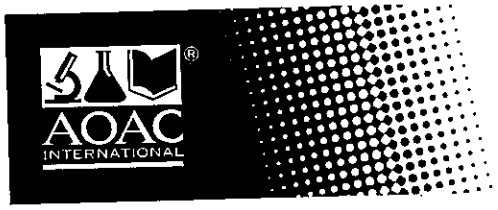

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ninum 8-hydroxyquinolate.—Warm 250 mL aluminum 50°–60°C and add excess of oxine reagent. Slowly add H_4 solution until permanent precipitate forms. Then add more to ensure complete precipitation. Let precipitate ilter through fritted glass crucible. Wash precipitate well st seven or eight 30 mL portions cold water and dry at C. Store in desiccator.

oform solution of aluminum 8-hydroxyquinolate.—Dissolve oxine in $CHCl_3$ to prepare 0.5 mg/mL solution. Prepare

ric acid.—Concentrated. If blank determination reveals f F, purify as follows: Carefully mix equal volumes H_2O, boil down to fumes, dilute carefully, boil down , and dilute to 1 + 1 volume.

as in **905.03(a)** (*see* 47.3.17), adding 3 mL CH_3COOH to addition to K_2SO_4 and $Ba(CH_3COO)_2$ solutions. Transfer due to small porcelain crucible (≤5 mL).

e of filter paper with $CHCl_3$ solution of aluminum oxine ger in diameter than top of crucible and let air dry. Add over ash, crimp paper over crucible edge, and put weight er) on paper. Heat crucible covered with paper 5 min at

Observe paper under UV light. In presence of F, ce of the aluminum oxine is quenched in area of spot over imit of identification is ca 0.05 mg F. Conduct blank ion on H_2SO_4.

JAOAC **37,** 381(1954).

AOAC Official Method 975.28
Fluorides (Insoluble) in Food

First Action 1975
Final Action

ates, fluosilicates, etc.)

ation of Test Solution

200 g test portion alkaline with lime–water, evaporate to id ash. Extract crude ash with H_2O containing enough l to decompose carbonates; filter, ignite insoluble tract with CH_3COOH (1 + 2), and again filter. Insoluble w contains $CaSiO_3$ and CaF_2, while filtrate contains all sent.

itive Test I

C. Qualitative Test II

Ash filter containing insoluble portion from A in Pt crucible, mix with little precipitated 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 h at 70°–80°C, 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** (*see* 47.3.07).

D. Quantitative Method

See **944.08** (*see* 9.2.11).

47.3.20

AOAC Official Method 931.08
Formaldehyde in Food

First Action 1931

(*See also* **964.21** [*see* 44.5.14].)

A. Preparation of Test Solution

If food 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 through trap, and slowly distil 50 mL. For milk, dilute 100 mL with 100 mL H_2O, and acidify and distil as for solids. With other liquid foods, acidify 200 mL and distil as for solids.

B. Chromotropic Acid Test

(a) *Reagent.*—Prepare saturated solution 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). Solution is light straw-colored.

(b) *Test.*—Place 5 mL reagent in test tube and add, with mixing, 1 mL distillate, A. Place in boiling water 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 amount of HCHO present).

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

C. Hehner-Fulton Test

(a) *Oxidizing solution.*—To cold H_2SO_4 add, in small portions, equal volume saturated Br_2–H_2O, cooling throughout operation.

(b) *Test.*—To 6 mL cold H_2SO_4 add 5 mL distillate, A, slowly and

47.3.22

AOAC Official Method 957.08
Hydrogen Peroxide in Milk

Qualitative Color Test
First Action 1957
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 test portion and mix. Pink or red indicates H_2O_2.

Reference: *JAOAC* **40,** 789(1957).
CAS-7722-84-1 (hydrogen peroxide)

47.3.23

AOAC Official Method 942.09
Monochloroacetic Acid in Liquids and Preservatives

Qualitative Tests
First Action 1942
Final Action

A. Optical-Crystallographic Properties of Barium Salt

(Applicable to commercial preservatives.)

Dilute 4–5 mL test portion to 100 mL, add 6 mL H_2SO_4 (1 + 1), and extract with equal volume ether in separator. If emulsions form, extract in continuous extractor 1 h. Transfer ether extract to separator, add few drops phenolphthalein and 5 mL 0.05M $Ba(OH)_2$, and shake 30 s. If aqueous layer takes on pink typical of phenolphthalein, filter through paper into small beaker. Add ca 0.05M CH_3COOH until colorless and evaporate to 1–2 mL on steam bath. Let remaining liquid evaporate spontaneously in air and finally in desiccator. If 5 mL 0.05M $Ba(OH)_2$ does not give pink aqueous layer, add 5 mL more before separating. Repeat extraction with $Ba(OH)_2$ solution several times or until pink solution is obtained, evaporating each barium solution in separate 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 powdered 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 negative 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