

8.1.07

AOAC Official Method 930.11 Phenol in Hazardous Substances

Colorimetric Method

First Action 1930

Final Action

Method I

(Applicable to commercial cresols, saponified cresol solutions, coal tar dips, and disinfectants, and to kerosene solutions of phenols in absence of salicylates or *m*-naphthol.)

A. Reagents

(a) *Dilute nitric acid*.—Aerate HNO₃ until colorless and dilute 1 in 4 (v/v) with H₂O.

(b) *Millon reagent*.—(*Danger*: Millon reagent is extremely poisonous.) (*Caution*: Perform in a fume hood.) To 2 mL Hg in 200 mL Erlenmeyer, slowly add 20 mL HNO₃. After first violent reaction subsides, shake as needed to disperse Hg and maintain reaction. After ca 10 min, when reaction practically ceases (undissolved Hg may be present), add 35 mL H₂O. If basic salt separates, add enough dilute HNO₃ to redissolve it. Add 10% NaOH solution dropwise with thorough mixing until the curdy precipitate that forms after each drop no longer redissolves but disperses as permanent turbidity. Add 5 mL dilute HNO₃ and mix well. Prepare fresh daily.

(c) *Phenol standard solution*.—Dissolve weighed amount pure phenol (congealing point 40°C) in enough H₂O to make 1% solution. Prepare the working standard fresh daily by diluting with H₂O to make 0.025% solution (final standard). (*Caution*: Phenol is extremely toxic and may be fatal if swallowed, inhaled, or absorbed through the skin.)

(d) *Formaldehyde solution*.—Dilute 2 mL 37% HCHO solution to 100 mL with H₂O. (*Caution*: Formaldehyde is a suspected carcinogen. Use caution when handling to avoid exposure.)

(e) *Methyl orange indicator*.—0.5% aqueous solution.

B. Apparatus

(a) *Nessler cylinders*.—50 mL tall-form, matched.

(b) *Test tubes*.—Approximately 180 × 20 mm, with stoppers, marked at 25 mL.

(c) *Boiling water bath*.—Size sufficient to hold four test tubes.

C. Preparation of Test Solution

(a) *Commercial cresol*.—Weigh by difference ca 2.5 g test portion into 250 mL volumetric flask, dissolve in 10 mL 10% NaOH solution (w/v), and dilute to volume with H₂O.

(b) *Saponified cresol solutions, coal tar dips and disinfectants, kerosene solutions of phenols, etc.*—Weigh by difference ca 5 g test portion (or use 5 mL and calculate weight from density) into 250 mL volumetric flask and dilute to volume with H₂O. With products consisting largely of kerosene, bring water level to mark and take aliquots from aqueous phase only.

D. Determination

Transfer 5 mL aliquot prepared test solution to 200 mL volumetric flask and promptly dilute to ca 50 mL with H₂O. Add 1 drop methyl orange indicator, and then dilute HNO₃ until solution is practically neutral (yellow color). Dilute to volume and shake well.

Place 5 mL diluted solution in each of 2 marked test tubes; in each of 2 additional test tubes place 5 mL standard phenol solution. Flow 5 mL Millon reagent down side of each tube, mix, and place tubes in boiling water bath. Continue boiling for exactly 30 min, cool immediately and thoroughly by immersion in bath of cold water

10 min. After cooling, add 5 mL dilute HNO₃ to each tube. Mix well and add 3 mL HCHO solution to one of each pair of tubes. Dilute all tubes to 25 mL mark with H₂O, stopper, shake well, and let stand overnight. (Tubes containing only HCHO fade to yellow; others show orange or red color.)

Pipet 20 mL from each of the 2 standard phenol tubes to 100 mL volumetric flasks; add 5 mL dilute HNO₃, dilute to volume with H₂O, and mix. (Red solution = “phenol standard,” yellow solution = “phenol blank.”) Transfer these solutions to burets. Pipet 10 mL of each test solution into Nessler tubes. The orange/red constitutes the “test solution unknown” and the yellow the “test solution blank.” To “test solution blank” tube add measured amount of “phenol standard” from buret and add the same volume “phenol blank” to “unknown.” Agitate thoroughly (by shaking, if necessary), and compare colors. When solution colors match, each mL phenol standard used = 1% phenol if test portion weighing exactly 5 g was used, or 2% if exactly 2.5 g was used.

(*Note*: Pair of phenol standard tubes provides enough final solution to assay several unknowns. All the unknowns *must* have accompanied phenol standard solutions through entire process with identical reagents and treatment. Delay in matching tubes must be avoided after titration is started; otherwise excess HCHO present in blanks may, after mixing, affect intensity of red color.)

References: *USDA Bull.* **1308**, p. 17.

JAOAC **13**, 160(1930).

Method II

E. Determination

(Applicable to determination of phenol in presence of salicylates.)

Weigh by difference a 10 g test portion into 250 mL separatory funnel. Add 50 mL kerosene and extract with three 100 mL portions H₂O. Filter aqueous extracts through wet filter into 500 mL volumetric flask, dilute to volume with H₂O, and proceed as in **D**.

When tubes are brought to match, each mL phenol standard used = 1% phenol if test sample weighing exactly 10 g was used.

Reference: *Ind. Eng. Chem. Anal. Ed.* **1**, 232(1929).

CAS-108-95-2 (phenol)