39.1.15 - Meat and Meat Products

AOAC Official Method 928.08
Nitrogen in Meat

Kjeldahl Method
First Action 1928
Final Action 1974

A. Reagents

(**a**) *Kjeldahl catalyst*.—15 g K_2SO_4 + 0.7 g HgO (commercially prepared catalysts are available containing pumice, if desired).

(**b**) *Sulfuric acid*.—H_2SO_4, ACS.

(**c**) *NaOH solution*.—Prepare 1200 mL NaOH (1 + 1). Let stand until clear (ca 10 days).

(**d**) *Metallic zinc*.—Powder, ACS, to be used if catalyst does not contain pumice.

(**e**) *Indicator solution*.—Fleisher Methyl Purple, or equivalent.

(**f**) *Acid potassium phthalate*.—NIST Standard.

(**g**) *Standardized NaOH solution*.—0.2000 0.0004M. Add 108 mL NaOH (1 + 1) to CO_2-free H_2O and dilute to 10 L. Standardize against potassium acid phthalate, using phenolphthalein indicator.

(**h**) *Standardized acid solution*.—0.2000 0.0004M. HCL or 0.0002M H_2SO_4

(**i**) *Hydrochloric acid*.—Dilute 178 mL 35-37% HCl to 10 L. Standardize against standard NaOH solution, and adjust strength accordingly.

(**j**) *Sulfuric acid*.—Dilute 55 mL 98% H_2SO_4 to 10 L. Standardize against standard NaOH solution, and adjust strength accordingly.

(**k**) *Sodium hydroxide*.—*Sodium thiosulfate solution*.—Dissolve 460 g $Na_2S_2O_3 \cdot 5H_2O$ in H_2O; dilute to 1 L with H_2O, and add this solution to 15,250 g NaOH dissolved in 14,250 mL H_2O. This will yield 20 L of 50% (w/v) NaOH solution. If other volumes are desired, adjust weights of NaOH and $Na_2S_2O_3 \cdot 5H_2O$ accordingly. Specific gravity of final solution should be 1.45. 40 g K_2S or Na_2S per liter of final solution may be used in lieu of $Na_2S_2O_3 \cdot 5H_2O$. (*Note*: Prepared in excess to precipitate residual Hg.)

Standardize each standard solution with primary standard (*see* Appendix A, Standard Solutions and Certified Reference Materials) and check one against the other. Test all reagents before using by blank determination with 2 g sugar, which ensures partial reduction of any nitrates present and provides organic material to assure acid requirements and digestion characteristics similar to those encountered in meat standards.

B. Determination

Place weighed test portion (2.0-2.2 g) in digestion flask. Add 0.7 g HgO or 0.65 g metallic Hg, 15 g powdered K_2SO_4 or anhydrous Na_2SO_4, and 40 mL H_2SO_4. If test portion >2.2 g is used, increase $H_2SO_4$by 10 mL for each g test portion used. Place flask in inclined position and heat gently until frothing ceases (if necessary, add small amount of paraffin to reduce frothing); boil briskly until solution clears, and then 30 min longer (2 h if organic material is present).

Cool, add ca 200 mL H_2O, cool <25°C, add 25 mL of the sulfide of thiosulfate solution, and mix to precipitate Hg. Add few Zn granules to prevent bumping, tilt flask, and add layer of NaOH without agitation. (For each 10 mL H_2SO_4 used, or its equivalent in diluted H_2SO_4, add 15 g solid NaOH or enough solution to make contents strongly alkaline.) (Thiosulfate or sulfide solution may be mixed with the NaOH solution before addition to flask.) Immediately connect flask to distilling bulb on condenser, and, with tip of condenser immersed in standardized acid and 5-7 drops indicator in receiver, rotate flask to mix contents thoroughly; then heat until all NH_3 has distilled (150 mL distillate). Remove receiver, wash tip of condenser, and titrate excess standardized acid in distillate with standardized NaOH solution. Correct for blank determination on reagents.

$$\text{N, \%} = [(\text{mL standardized acid} \times \text{M acid}) - (\text{mL standardized NaOH} \times \text{M NaOH})] \times 1.4007/\text{g test portion}$$

References:

JAOAC **11**, 408(1928).

Alternative II (Copper-Based Catalyst)
First Action 1993

(Applicable to determination of nitrogen in meat and meat products.)

Results of the interlaboratory study supporting the acceptance of the method:

Ground beef (protein value = 17%)
s_r = 0.22; s_R = 0.26; RSD_r = 1.30%; RSD_R = 1.51%

Canned ham (protein value = 18%)
s_r = 0.23; s_R = 0.27; RSD_r = 1.27%; RSD_R = 1.48%

Smoked ham (protein value = 15%)
s_r = 0.20; s_R = 0.24; RSD_r = 1.36%; RSD_R = 1.58%

Pork sausage (protein value = 10.5%)
s_r = 0.16; s_R = 0.19; RSD_r = 1.54%; RSD_R = 1.80%

Cooked sausage (protein value = 12%)
s_r = 0.18; s_R = 0.21; RSD_r = 1.47%; RSD_R = 1.72%

Dry cured ham (protein value = 23%)
s_r = 0.28; s_R = 0.31; RSD_r = 1.16%; RSD_R = 1.36%

C. Principle

Test sample is digested in H_2SO_4, using $CuSO_4$ as catalyst, converting N to NH_3 which is distilled and titrated.

D. Apparatus and Reagents

See **A**, except a copper catalyst is used in place of mercury catalyst.

(**a**) *Copper catalyst.*—Prepare using 15.0 g K_2SO_4 and 0.45 g $CuSO_4$. As boiling aid, 0.1 g pumice may be added. (Propac 15p, Alfie Packers, Inc., Omaha, NE, USA, or other commercially prepared catalysts are suitable.)

E. Preparation of test samples

See **983.18** (*see* 39.1.01).

F. Determination

See **B**, except in paragraph 1, after solution clears, boil 45 min longer (2 h for test portions containing organic material).

G. Calculations

See **B**. (*Note:* Alternative II yields results that are, on average, 99.0% of results from Alternative I.)

Reference:
J. AOAC Int. **77**, 1542(1994).