9.2.36 - Metals and Other Elements at Trace Levels in Foods / Single Element Methods

AOAC Official Method 973.36
Titanium in Cheese

Spectrophotometric Method
First Action 1973
Final Action 1976

A. Standard Solution

Titanium dioxide standard solution.—0.1 mg/mL. Accurately weigh 50 mg TiO_2 and transfer to 250 mL beaker; add 15 g anhydrous Na_2SO_4 and 50 mL H_2SO_4. Add boiling chips, cover with watch glass, and heat to bp on hot plate to dissolve. Cool, and cautiously add 100 mL H_2O with stirring. (Warm on steam bath if solution becomes cloudy.) Cool, transfer solution to 500 mL volumetric flask containing 200 mL H_2O, and dilute to volume with H_2O.

B. Preparation of Material

Weigh, to nearest 0.1 g, 10 g prepared test sample, **955.30** (*see* 33.7.02), into 100 mL Pt dish and char under IR lamp. Place in cold furnace and ignite at 850°C to white ash.

Cool, add ca 1.5 g anhydrous Na_2SO_4 and 10 mL H_2SO_4, cover with watch glass, and bring to bp on hot plate to dissolve. Turn heat off and let cool on hot plate. Cautiously rinse cover, carefully add ca 30 mL H_2O, and mix with stirring rod to disperse any insoluble salts. Heat on steam bath if insoluble material forms cake on bottom of dish.

Transfer quantitatively to 100 mL volumetric flask with aid of ca 40 mL H_2O. If solution is cloudy, heat on steam bath or in boiling H_2O bath to clarify. Cool, and dilute to volume with H_2O.

C. Preparation of Standard Curve

Transfer 0, 1, 2, 3, 4, and 5 mL TiO_2 standard solution to separate 5 mL glass-stoppered graduates (or volumetric flasks) and dilute to volume with H_2SO_4 (1 + 9).

Add 0.2 mL 30% H_2O_2, mix, and determine *A* on recording spectrophotometer in 1.0 cm cells from 650 to 325 nm against 0.2 mL 30% H_2O_2 in 5.0 mL H_2SO_4 (1 + 9). Determine *A* at maximum, ca 408 nm, and prepare standard curve.

D. Determination

Transfer 3.0 mL test solution to 5 mL glass-stoppered graduate (or volumetric flask), dilute to volume with H_2SO_4 (1 + 9), and continue as in **C**, beginning "Add 0.2 mL 30% H_2O_2, . . .".

Determine mg TiO_2 in test solution from standard curve, and calculate as percent TiO_2.

Reference:
JAOAC **56,** 535(1973).

CAS-7440-32-6 (titanium)