

5.1.19

AOAC Official Method 977.36
Dibutyltin Dilaurate (DBTD) in Feeds
Atomic Absorption Spectrophotometric Method
First Action 1977
Final Action 1979

A. Principle

DBTD is extracted from feed with CHCl_3 , extract is filtered to remove feed particles, and aliquot is concentrated in presence of methanol until CHCl_3 is removed. Methanol solution is diluted and filtered to remove feed interference, and Sn is determined by AA using air- C_2H_2 flame.

B. Apparatus and Reagents

(a) *Atomic absorption spectrophotometer.*—Double beam, operated at 286.3 nm with air- C_2H_2 flame and direct readout using 10 mV recorder. Optimize instrument according to manufacturer's instructions.

(b) *Hot plate.*—Regulated to ± 3 C.

(c) *Mechanical shaker.*—Wrist-action type (Burrell Corp., or equivalent).

(d) *Tin standard solutions.*—(1) *Stock solution.*—500 g/mL. Accurately weigh ca 0.217 g dibutyltin bis(2-ethylhexanoate) (NIST SRM No. 1057 or Eastman Kodak No. 10427, percent Sn certified) into 100 mL volumetric flask, and dissolve and dilute to volume with methanol. (2) *Working solution.*—10 g/mL. Pipet 2 mL stock solution into 100 mL volumetric flask, add 1.0 mL HCl, and dilute to volume with methanol.

C. Preparation of Test Sample and Extraction of Test Portion

Accurately weigh test portion of ground feed containing ca 10 g Sn/mL in final solution (see Table 977.36), and transfer to 125 mL Erlenmeyer. Add 50 mL CHCl_3 , mix, and place flask in 55–60 C water bath. Let test portion reach bath temperature; then stopper tightly. Continue heating additional 30 min, swirling occasionally. Remove from water bath and shake mechanically 20 min. Filter through Whatman No. 4 paper, and collect 30 mL filtrate in 50 mL Erlenmeyer.

Pipet 25 mL filtrate into 100 mL graduated beaker, add 2 boiling chips and 0.25 mL HCl, and concentrate to ca 10 mL at gentle boil on

Table 977.36. Test portions weights for DBTD-containing feeds

DBTD, %	Feed test portions, g
0.020 (Polystat)	13.00
0.0375 (Tinostat)	7.00
0.0700 (Wormal)	3.75

hot plate. Add 20 mL methanol and concentrate test portion to ca 10 mL again; then repeat with additional 20 mL and 30 mL portions methanol. (Raise temperature of hot plate to maintain gentle boiling as ratio of methanol to CHCl_3 increases.) Remove from heat and let cool to room temperature. Transfer methanol solution to 25 mL volumetric flask, washing beaker and funnel with two to three 5 mL portions methanol, dilute to volume with methanol, and mix thoroughly. Filter through Whatman No. 42 paper and collect filtrate in another 25 mL volumetric flask.

Prepare blank by diluting 1 mL HCl to 100 mL with methanol.

D. Determination

Let spectrophotometer warm up thoroughly and equilibrate by aspirating methanol 15 min, using air- C_2H_2 flame and triple slot burner head. Zero spectrophotometer by aspirating blank; then aspirate test portion and standard solutions, using conditions given in B(a). Repeat sequence for each test portion.

$$\text{DBTD, \%} = \frac{A}{C} \frac{50}{5.32} \frac{10^{-6}}{(100/A) W} \\ = (A/A) \frac{0.266}{W}$$

$$\text{DBTD, mg/kg} = \frac{A}{A} \frac{C}{W} \frac{50}{5.32} \\ = (A/A) \frac{2660}{W}$$

where A and A refer to test portion and standard, respectively; C = g standard/mL; and W = g test portion.

Reference: *JAOAC* 60, 1054(1977).

CAS-77-58-7 (dibutyltin dilaurate)