

5.1.41

AOAC Official Method 978.30 Pyrantel Tartrate in Feeds

Spectrophotometric Method
First Action 1978
Final Action 1981

(Applicable to range 106–881 mg/kg. Pyrantel tartrate solutions are light sensitive. Extracts must be protected from direct sunlight or artificial light.)

A. Apparatus

- (a) *Spectrophotometer*.—For use at 311 nm, with 1 cm cells.
- (b) *Centrifuge*.—International Model EXD (International Equipment Co.) or equivalent, equipped to hold 50 mL glass-stoppered centrifuge tubes.
- (c) *Filter aid*.—Celite 545, acid-washed (Celite Corp.), or Millipore prefilter pad (No. AP2507500; Millipore Corp.) or equivalents.
- (d) *Filtrator*.—Fisher No. 9-788, low form (Fisher Scientific Co.), or equivalent.
- (e) *Mixer*.—Vortex Genie mixer (Scientific Products), or equivalent.

B. Reagents

- (a) *Hydrochloric acid*.—0.1M. Dilute 8.33 mL HCl to 1 L with H₂O.
- (b) *Leaching solution*.—Dissolve 100 g NaCl in 1 L H₂O, add 1 L methanol, and mix vigorously. Prepare fresh daily.
- (c) *Perchloric acid*.—0.24M. Dilute 20 mL HClO₄ to 1 L with H₂O.
- (d) *Sodium hydroxide*.—0.1M. Dissolve 4 g NaOH in H₂O and dilute to 1 L with H₂O.
- (e) *Pyrantel tartrate standard solutions*.—(1) *Stock solution*.—0.80 mg/mL. Weigh 80.0 mg Pyrantel Tartrate Reference Standard (available from Phibro Animal Health, 65 Challenger Rd, Third Floor, Ridgefield, NJ 07660, USA, Tel: +1-888-403-0074, Fax: +1-201-329-7070; www.philbroah.com) into 100 mL volumetric flask, dissolve in leaching solution, (b), and dilute to volume with same solvent. Use ultrasonic bath to speed dissolution. If standard does not dissolve readily, remake. Prepare fresh daily. (2) *Working solution*.—0.16 mg/mL. Pipet 20 mL stock solution into 100 mL volumetric flask, dilute to volume with leaching solution, and mix well. Prepare fresh daily.

C. Preparation of Test Samples

Weigh duplicate test portions of ground test sample into 125 mL Erlenmeyers: 15.000 ± 0.010 g for 106 mg/kg pyrantel tartrate; 1.800 ± 0.010 g, 881 mg/kg; and 1.000 ± 0.010 g, 8810 mg/kg. Add 60 mL leaching solution to 1 portion; add 50 mL leaching solution and, by pipet, 10 mL working standard solution, **B(e)(2)**, to other portion of 106 or 881 mg/kg pairs or 10 mL stock standard solution, **B(e)(1)**, to other of 8810 mg/kg pair. Cap with polypropylene stopper and shake mechanically 1 h.

Quantitatively transfer mixture to Büchner pre-coated with Celite or containing prefilter pad, **A(c)**, rinsing flask with small portions leaching solution, **B(b)**, and collecting filtrate under vacuum with Filtrator, **A(d)**, containing 250 mL Erlenmeyer, until volume filtrate is 150 mL. Transfer filtrate to 200 mL volumetric flask, and dilute to volume with leaching solution. For 8810 mg/kg level, pipet 50 mL diluted filtrate into 250 mL volumetric flask, and dilute to volume with leaching solution.

D. Determination

Pipet 25 mL prepared filtrate into 250 mL separator containing 5 g finely ground KI, and shake to dissolve. Pipet in 100 mL CHCl₃ and shake 45 s. Transfer lower CHCl₃ layer to another 250 mL separator containing 25 mL 0.24M HClO₄, and shake 10 s. Transfer ca 35 mL lower CHCl₃ layer to 50 mL glass-stoppered centrifuge tube containing 10.0 mL 0.1M NaOH. Stopper tube, and mix on Vortex mixer, **A(e)**, 15 s while shaking back and forth. Centrifuge 10 min at 990 g (1800 rpm). Pipet 25 mL lower CHCl₃ layer from tube into another 50 mL glass-stoppered centrifuge tube containing 10.0 mL 0.1M HCl. Stopper tube, mix, and centrifuge as before. Rapidly record *A* of upper aqueous layer at 311 nm against CHCl₃-saturated 0.1M HCl as reference solvent.

$$\text{mg/kg Pyrantel tartrate} = (A/W) [W/(A - A)] F 10^6$$

where *A* and *A* refer to absorbance of test portion and spiked test portion, respectively; *W* = g feed, supplement, or concentration; *W* = g pyrantel tartrate reference standard used to prepare standard solution; *F* = [(1/100 mL) (20 mL/100 mL) 10 mL] for 106 and 881 mg/kg levels; and *F* = [(1/100) 10] for 8810 mg/kg level.

Reference: *JAOAC* **61**, 296, 473(1971).

CAS-33401-94-4 (pyrantel tartrate)