

## 7.3.14A

**AOAC Official Method 997.14**  
**Thiodicarb in Technical Products**  
**and Formulations**  
**Liquid Chromatographic Method**  
**First Action 1997**  
**Final Action 2001**

**CIPAC–AOAC Method**

(Applicable to technical, suspension concentrate, wettable powder, and water dispersible granule formulations.)

See Table 997.14 for the results of the interlaboratory study supporting the acceptance of the method.

**A. Principle**

Test portion is dissolved in dichloromethane, internal standard in methanol is added, and analysis is performed on a reversed-phase C<sub>8</sub> column with a water–methanol mobile phase. Thiodicarb is measured by absorbance at 254 nm by peak area, using internal standard.

**B. Apparatus**

(a) *Liquid chromatograph*.—With 10 µL loop injector (or autoinjector). Operating conditions: mobile phase flow rate, 1 mL/min (constant flow pump); temperature, ambient.

(b) *Column*.—250 × 4.6 mm id, stainless steel packed with reversed-phase C<sub>8</sub> on 5 µm silica.

(c) *UV detector*.—Measuring at 254 nm wavelength and set to give 80–90% full scale deflection for the concentration of standard solution used for calibration.

(d) *Electronic integrator*.—Preferred for peak area measurements.

(e) *Filtration device*.—Glass syringe fitted with a membrane filtration unit compatible with organic solvents and filter (0.5 µm pore size).

(f) *Ultrasonic bath or mechanical shaker*.

**C. Reagents**

(a) *Calibration solution*.—Weigh in duplicate (to the nearest 0.1 mg) ca 0.08 g thiodicarb standard of known purity (available from Rhône-Poulenc, Secteur Agro, 14–20 Rue Pierre Baizet, 69009 Lyon France; Chem Service, Inc., PO Box 599, West Chester, PA 19381-0599, USA, www.chemservice.com) into separate 100 mL volumetric flasks. Add to each, 5 mL dichloromethane, 10.0 mL internal standard solution (b, Solution I) from a pipet and

dilute to volume with methanol, after allowing solution to adjust to room temperature. Mix thoroughly (solutions C<sub>1</sub> and C<sub>2</sub>). Filter through a 0.5 µm filter.

(b) *Internal standard solution*.—Dissolve 2 mL dimethyl phthalate in 200 mL methanol (Solution I). Prepare a sufficient quantity of this solution for all test and calibration solutions to be used in analysis. Filter through membrane filter B(e).

(c) *Solvents*.—H<sub>2</sub>O, methanol, and dichloromethane, LC grade.

(d) *LC mobile phase*.—Methanol–water (60 + 40, v/v).

(e) *Column flush*.—Methanol.

**D. Preparation of Test Solutions**

Into a 100 mL volumetric flask weigh (to the nearest 0.1 mg) test portion to contain ca 0.08 g thiodicarb. Add 5 mL dichloromethane, dissolve by shaking thoroughly. For suspension concentrate formulations, shake thoroughly or place flask in an ultrasonic bath for 2 min. Add by pipet 10 mL internal standard solution (b, Solution I) and dilute to volume with methanol after temperature equilibration. Mix thoroughly and filter through a 0.5 µm filter, B(e).

**E. Determination**

Equilibrate column with mobile phase for 10 min. After each analysis sequence, flush column for 10 min with methanol and again equilibrate column with mobile phase before additional analysis.

Inject first calibration solution (C<sub>1</sub>), first test solution twice, and then the second calibration solution, etc. (C<sub>1</sub>, S<sub>1</sub>, S<sub>1</sub>, C<sub>2</sub>, flush, equilibrate, C<sub>1</sub>, S<sub>2</sub>, S<sub>2</sub>, C<sub>2</sub>...). Determine peak areas for test and standard injections.

**F. Calculations**

Calculate response factor, *R*, for each injection as follows:

$$R = \frac{\text{Peak Area}}{\text{Concentration}}$$

Calculate thiodicarb in test sample as follows:

$$\text{Thiodicarb, \%} = (R/R') \times (W'/W) \times P$$

where *R* and *R'* = average response factor for test and standard solution injections, respectively; *W* and *W'* = mg test portion and average weight in mg standard, respectively; and *P* = purity (%) of standard.

Reference: *J. AOAC Int.* **81**, 341(1998).

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**Table 997.14 Interlaboratory study results for determination of thiodicarb in technical products and formulations by liquid chromatography**

Product	No. of labs	Mean, g/kg	s <sub>r</sub>	s <sub>R</sub>	r <sup>a</sup> , g/kg	R <sup>b</sup> , g/kg	RSD <sub>r</sub> , %	RSD <sub>R</sub> , %
Technical	19 (17) <sup>a</sup>	950.0	9.25	9.25	25.90	25.90	0.97	0.97
	19 (18)	949.2	8.95	9.16	25.06	25.64	0.94	0.96
Suspension concentrate	19 (15)	342.3	3.94	6.43	11.04	17.99	1.15	1.88
Wettable powder	19 (17)	760.4	6.70	10.22	18.76	28.63	0.88	1.34
Water dispersible granule	19 (17)	791.8	5.54	7.30	15.22	20.44	0.70	0.92

<sup>a</sup>  $r = 2.8 \times s_r$ .

<sup>b</sup>  $R = 2.8 \times s_R$ .