

Appendix VI Laboratory Details

Gas Chromatographic Conditions for the Determination of Propamocarb\

Instrument:	Perkin Elmer AutoSystem GC with Nitrogen-Phosphorus Detector, operating in the hot, on-column injection mode.	
Column:	J & W DB – 1, 15 m x 0.53 mm, 3.0 µm film thickness	
Gases:	Helium –	5.0 mL/min
	Hydrogen –	2.0 mL/min
	Air –	100 mL/min
Temperatures:	Injection Port -	225 °C
	Detector -	250 °C
Oven Profile:	Temp.1 = 130 °C	Time 1 = 0.5 min
	Temp 2 = 200 °C	Time 2 = 4.0 min
	Rate = 10 C°/min	
Injection Volume:	3 µL	
Approximate Retention Time:	6.5 - 6.7 min.	

The following analytical standard was used in this study:

Propamocarb	AgrEvo analytical standard number RL894-01/95, 96.8%, Dissolved in di-isopropyl ether on 06/09/97. Lot/Code #AK H039744 00 1C97 0001 Certificate of Analysis #AZ 06584 Expiration Date: 08/09/99
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Appendix VII Analytical Method

A. PRINCIPLE

Extractable residues of propamocarb are extracted from tomato matrices by blending with acidified methanol. The extract is evaporated to leave the aqueous phase, which is made basic and diluted with saturated brine. The resulting solution is extracted with dichloromethane (DCM) which is then dried and evaporated to dryness. The residue is taken up in di-isopropyl ether for GC-NPD analysis.

B. PROCEDURE

1. Matrix Extraction

Weigh out 20 g of sample into a one-pint Mason jar. Fortify the recovery sample as appropriate. Add methanol (100 mL) and 1.0 N hydrochloric acid (1 mL) to the samples. Blend 1-2 minutes with an Omni Mixer and filter through a Büchner funnel containing Whatman 934-H glass microfiber filter paper into a 1 L boiling flask. Rinse the Mason jar with additional methanol (50 mL) and filter the rinsate into the boiling flask.

2. Dichloromethane Extraction

1. Evaporate the methanolic extract in a rotary evaporator at 40 °C (\pm 5 °C) until only the aqueous phase remains. Add 5 N NaOH (10 mL) to each sample and verify that the pH > 9 with pH paper. Transfer the extract to a 250-mL separatory funnel.
2. Rinse the boiling flask with saturated sodium chloride solution (50 mL) and add this rinsate to the separatory funnel.
3. Partition with dichloromethane (DCM, 3 x 50 mL). Drain the lower DCM layer through a sodium sulfate pad in a powder funnel into a 250-mL boiling flask. Rinse the sodium sulfate pad with additional DCM (20 mL). Discard the aqueous phase.
4. Evaporate the dried, combined DCM solution in a rotary evaporator to dryness at 40 °C (\pm 5 °C).

5. Dissolve the residue in di-isopropyl ether (2.0 mL). Filter the sample through a Spartan disposable syringe filter using a 5-cc Becton Dickinson disposable Luer-Lok syringe. Dilute as necessary with additional di-isopropyl ether for GC analysis.

3. Preparation of Analytical Standards

Prepare a stock solution of approximately 1000 $\mu\text{g/mL}$ of analytical standard propamocarb in di-isopropylether. Prepare serial dilutions for fortification solutions and for GC injection standards by diluting appropriate volumes of the above solution with additional di-isoproylether.

4. Calculations

The peak height of the analyzed sample is compared with least squares derived standard curve and is determined from the equation for the line:

$$\mu\text{g/mL Found} = (y - b) / m \quad (\text{Eq. 1})$$

where: y = peak height (counts)
 b = y-intercept (counts)
 m = slope of the standard curve line
 (counts mL/ μg)

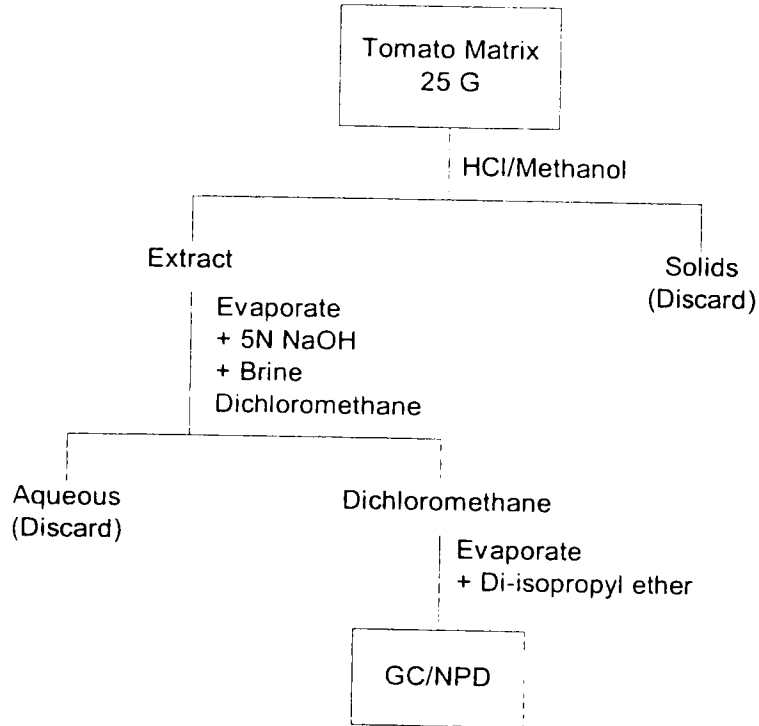
$$\text{ppm Analyte} = ((\mu\text{g/mL Found} \times V_f) / W) / D_f \quad (\text{Eq. 2})$$

where V_f = Final volume of the sample
 W = Sample weight
 D_f = Dilution factor

$$\% \text{ Recovery} = \frac{\text{ppm analyte Found}}{\text{ppm fortification Level}} \times 100 \quad (\text{Eq. 3})$$

Appendix VII (continued)

Figure AVII-1 Flow Diagram of the Analytical Method



Appendix VII (continued)

Figure AVII-2 Residue Decline Curves

