



**Ricerca**

Pacific Ag Research Protocol Amendment to 425USATOM00.0069X  
Ricerca Protocol 012921-0

**Determination of Methyl Iodide (TM-425) in Fruiting Tomatoes by Gas Chromatography Headspace Analysis**

**Introduction**

This method defines the procedure for the analytical determination of methyl iodide in tomatoes in field RAC studies.

In Ricerca Protocol 12157-0, this method was validated under GLP and a Limit of Quantitation (LOQ) was established at 10 ppb methyl iodide residue in tomatoes. Additionally, this same protocol will determine the freezer storage stability of methyl iodide after fortification to homogenized tomatoes.

**Apparatus**

- Gas Chromatograph (Hewlett Packard Model 5890 or equivalent with an Electron Capture Detector) and an automated headspace sampling system (Hewlett Packard Model 7694, or equivalent)
- Chromatography data system (Perkin Elmer Turbochrom or equivalent)
- J&W Gaspro column, 30 Meter x 0.32mm id (Cat Number 113-4332)
- Analytical balance capable of weighing to 0.00001 g
- Volumetric flasks and Miscellaneous glassware (e.g. 100 mL Class A volumetric flasks, 100 ml graduated cylinders, and disposable pipettes)
- 250 mL polyethylene sample bottles (Fisher Cat. 05-562-23)
- Brinkman Polytron Tissue Homogenizer with a 2-cm.-diameter generator (Brinkman Cat. 027-13-066-6)

**Reagents and Solutions**

- Water, HPLC grade
- N,N-Dimethyl Formamide, Anhydrous (Acros Cat Number 610320010, or equivalent)
- Methyl Iodide (Iodomethane) test material/reference standard (as received from sponsor)

**Extraction Procedure**

1. Tomatoes from the field are assumed to be received homogenized. However, if the tomatoes samples have not previously been homogenized then the samples will be processed with dry ice in a hobart chopper according to Ricerca SOP 03-K014-00. The dry ice will be allowed to sublime before subsamples are weighed for analysis.



**Ricerca**

Pacific Ag Research Protocol Amendment to 425USATOM00.0069X  
Ricerca Protocol 012921-0

2. A 20 gram portion of the chopped homogenized sample is weighed into a 250 mL polypropylene bottle with screw cap.
3. Samples to serve as concurrent recoveries are fortified at this stage.
4. A 10 mL aliquot of dimethyl formamide and 40 mL of HPLC grade water are added to each of the samples. The bottles are capped and shaken briefly and placed in an ice water bath.
5. The samples are centrifuged for 20 minutes at 7000 rpm with the centrifuge temperature set to 5 Celsius.
6. The supernatant is carefully poured-off from the pellet and measured in a 100 ml graduated cylinder. The solution is reconstituted to 100 mL with water and stirred briefly to insure uniform distribution.
7. A 2-mL aliquot of the extracts from each sample is transferred to a 20 mL headspace vial and sealed with a crimp-top cap.
8. Likewise, 2 mL aliquots of the calibration solutions are transferred to a headspace vial for calibration and linearity determinations.

#### **Gas Chromatographic System Conditions**

The headspace analyzer (Hewlett Packard Model 7694, or equivalent) will be set with the following operating parameters:

Equilibration of each vial for 15 minutes at 43 °C.

Injection loop of 1 mL.

Loop temperature of 60 °C.

Transfer line temp of 60 °C.

Pressurization time of 0.2 min.

Loop fill time of 0.02 min.

Loop equilibration time of 0.05 min.

Injection time of 1.0 min.

The GC (Hewlett Packard Model 5890 or equivalent with an Electron Capture Detector) will be set with the following operating parameters:

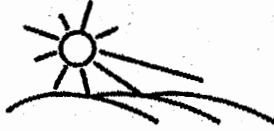
Initial temperature of 180°C for 10 minutes then ramped at 30°C per minute to 260°C and held for 1.0 minute.

Injector temperature of 180°C.

Detector (ECD) set at 300°C.

Column description: J&W Gaspro column, 30 Meter x 0.32mm id.

Column head pressure set at 20 psi.



**Ricerca**

Pacific Ag Research Protocol Amendment to 425USATOM00.0069X  
Ricerca Protocol 012921-0

**Calculations**

Quantitation of methyl iodide residues are made by injecting with the samples a series of calibration standards (0.10, 0.05, 0.025, 0.005 and 0.001  $\mu\text{g}/\text{mL}$ ). The response of the standards will be plotted in area or height versus concentration. The sample concentration in  $\mu\text{g}/\text{mL}$  will be determined from the first order line generated from the calibration standards. The final concentration of the sample in ppb is calculated using the following formula:

$$\frac{\text{sample response from line } (\mu\text{g}/\text{mL}) \times \text{final volume } (100 \text{ mLs}) \times 1000 \text{ ng}/\mu\text{g}}{\text{weight of sample } (20 \text{ g})}$$