Soil Extraction Method – Iodomethane *(see also Reference 3)*

See Figure 2 for a schematic of the extraction method used. For each sample to be analyzed, ten grams of soil (± 0.1 gram) was weighed into a 60 mL amber jar. The sample was fortified with an appropriate concentration of iodomethane, if needed. Twenty mL of chilled ethyl acetate was added, followed by ~ 10 grams of anhydrous sodium sulfate. The jar was closed, and shaken on a wrist action shaker for 30 minutes.

After shaking, the jar was placed on dry ice. The soil was allowed to settle while the jar was chilled. Once chilled, portions of the extract were removed for analysis. If needed, extracts were diluted with ethyl acetate to bring expected iodomethane concentrations into the linearity range of the analytical method.

Soil Extraction Method Validation – Iodomethane

The extraction method for iodomethane was validated at four levels, 0.0025 ppm, 0.005 ppm, 0.050 ppm, and 50.0 ppm. For each level, five 10 g aliquots of soil were weighed and fortified, using the iodomethane fortification solutions described above.

Desired Concentration	Fortification Solution	Volume Added
0.0025 ppm	0.5 µg/mL	50 µL
0.0050 ppm	0.5 µg/mL	100 µL
0.050 ppm	5.0 µg/mL	100 µL
50.0 ppm	5.0 mg/mL	100 µL

In addition, two 10 g control soil samples were extracted, as well as an empty jar (procedural blank). All fortifications were performed using cold solutions onto cold soil (all jars kept on ice during fortification procedures). The samples were extracted as shown in the iodomethane extraction method. Dilutions of extracts were made using cold ethyl acetate as needed to bring the expected iodomethane concentrations within the range of the linearity standard curve.

All extracts were analyzed by GC-ECD using the method discussed in the GC Analytical Method section.

Soil Extract Analysis – Iodomethane

Soil samples were extracted using the validated method described above (see Soil Extraction Method). Two of the control soils weighed out were fortified with a known amount of iodomethane at 0.5 ppm, 0.1 ppm, or 0.01 ppm of iodomethane, using the standard fortification solutions. These fortification samples were used to determine procedural recovery.

Dilutions of extracts were made using cold ethyl acetate as needed to bring the expected iodomethane concentrations within the range of the linearity standard curve. All extracts were analyzed by GC-ECD using the method discussed in the GC Analytical Method section below.

GC Analytical Method

The following method was used to detect iodomethane.

Column:	J&W Scientific GS-GasPro 30m x 0.32mm i.d.
Guard Column:	10m x 0.32mm i.d.
Carrier Gas / Flow:	Helium – 2 mL per minute
Detector Gas / Flow:	Nitrogen or 95% Argon / 5% Methane – 40 mL per minute
Injector Temperature:	200° C
Detector Temperature:	300° C
Injection Volume:	1 µL
Initial Oven Temperature:	80° C
Temperature Program:	5 minute hold at 80° C 30° C per minute to 200° C 1 minute hold at 200° C 50° C per minute to 260° C 8 minute hold at 260° C
Retention Time:	8.4-9.2 minutes

Methods of Calculation

Preparation of Stock Standards

$$\text{Volume of solvent (mL)} = \frac{(W)}{(FC)} \times \frac{P}{100}$$

where W = Micrograms of neat standard
P = Chemical purity of neat standard
FC = Final Concentration (µg/mL)

Recoveries

The recoveries of iodomethane from fortified soil samples were calculated as follows where 1µL samples were injected:

Linear regression formula from calibration curve $y = mx + b$ (generated with Excel® program)

$$x = \text{Nanogram analyte} = \frac{y - b}{m}$$

where y = Sample peak area
b = Calibration intercept
m = Slope

$$\text{ppm analyte} = \frac{\text{ng analyte}}{\mu\text{L inj.}} \times \frac{1\text{ mL}}{\text{Sample Wt (g)}} \times \frac{1\ \mu\text{g}}{1000\text{ ng}} \times \frac{1000\ \mu\text{L}}{1\text{ mL}}$$

Percent Recovery =

$$\frac{\text{Conc. of Fortified Sample } (\mu\text{g/g}) - \text{Conc. of Control Sample } (\mu\text{g/g})}{\text{Fortification Level } (\mu\text{g/g})} \times 100$$

An acceptable percent recovery (70-110%) of each analyte from soil and application targets demonstrated validity of the analytical method and determined the limit of quantitation. The actual analyte ppm in treated soil was corrected for soil moisture content as follows:

$$\text{ppm analyte in dry soil} = \text{ppm analyte in wet soil} \times \frac{\text{wet soil weight (g)}}{\text{dry soil weight (g)}}$$

Half-Life Determination

Average iodomethane ppm in a 0-24" soil core was calculated from ppm values for 0-6", 6-12", 12-18" and 18-24" segments. Data (as natural log) were plotted *vs.* time (days) to produce the best-fit straight line.

Half-life (t½) was calculated: $\frac{\ln 2}{-\text{slope}}$

Statistical Analysis

The residue data included the following statistical calculations: means, averages, standard deviations, and linear regressions.

Limit of Quantification and Limit of Detection of Iodomethane

The limit of quantification in soil was at least 0.0025 ppm, which was the lowest fortification level for which the method was validated.

The limit of detection was at least 0.0001 µg/mL, which was the lowest standard used in generating linearity curves.

Time Required for Analysis

The amount of time required for a typical extraction of a soil set consisting of 15-30 samples was approximately 6-8 hours. A sample extract set with a full linearity curve was then normally run using a GC-ECD, and took about 10 hours. Wet-Dry ratio analysis typically required 3-6 hours. Data analysis, including spreadsheet preparation generally took another 2-4 hours.

Figure 2. Flow Chart Summarizing Soil Extraction Method – Iodomethane.