

**Method 1659**  
***The Determination of Dazomet***  
***in Municipal and Industrial***  
***Wastewater***

# **Method 1659**

## ***The Determination of Dazomet in Municipal and Industrial Wastewater***

### **1. SCOPE AND APPLICATION**

- 1.1 This method covers the determination of dazomet (CAS 533-74-4) by base hydrolysis to methyl isothiocyanate (MITC; CAS 556-61-6) and subsequent determination of MITC by wide bore, fused-silica column gas chromatography (GC) with a nitrogen-phosphorus detector (NPD).
- 1.2 This method is designed to meet the monitoring requirements of the U.S. Environmental Protection Agency under the Clean Water Act at 40 *CFR* Part 455. Any modification of this method beyond those expressly permitted shall be considered a major modification subject to application and approval of alternative test procedures under 40 *CFR* 136.4 and 136.5.
- 1.3 When this method is applied to analysis of unfamiliar samples, compound identity must be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Gas chromatography mass spectrometry (GC/MS) can be used to confirm dazomet in extracts produced by this method when the level is sufficient.
- 1.4 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limit in Table 1 typifies the minimum quantity that can be detected with no interferences present.
- 1.5 This method is for use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatographic data. Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 8.2.

### **2. SUMMARY OF METHOD**

- 2.1 A 50-mL sample is adjusted to pH 10 to 12 and allowed to stand for 3 hours to hydrolyze dazomet to MITC. After hydrolysis, the sample is saturated with salt and extracted with 2.5 mL of ethyl acetate. Gas chromatographic conditions are described that permit the separation and measurement of MITC in the extract by wide-bore, fused-silica capillary column with nitrogen-phosphorus detector (GC/NPD).
- 2.2 Identification of MITC (qualitative analysis) is performed by comparing the GC retention time of the MITC on two dissimilar columns with the respective retention times of an authentic standard. Compound identity is confirmed when the retention times agree within their respective windows.

**2.3** Quantitative analysis is performed using an authentic standard of MITC to produce a calibration factor or calibration curve, and using the calibration data to determine the concentration of MITC in the extract. The concentration in the sample is calculated using the sample volume, the extract volume, and a factor to convert MITC to dazomet.

**2.4** Quality is assured through reproducible calibration and testing of the extraction and GC systems.

### **3. CONTAMINATION AND INTERFERENCES**

**3.1** Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. All materials used in the analysis shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks as described in Section 8.4.

**3.2** Glassware and, where possible, reagents are cleaned by rinsing with and baking at 450°C for a minimum of 1 hour in a muffle furnace or kiln. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment and thorough rinsing with acetone and pesticide-quality hexane may be required.

**3.3** Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

**3.4** Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

### **4. SAFETY**

**4.1** The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 1 through 3.

**4.2** Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves that will prevent exposure.

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## 5. APPARATUS AND MATERIALS

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*NOTE: Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of this method is the responsibility of the laboratory.*

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- 5.1** Sampling equipment for discrete or composite sampling.
- 5.1.1** Sample bottle: Amber glass, 1-L, with screw-cap. If amber bottles are not available, samples shall be protected from light.
- 5.1.2** Bottle caps: Threaded to fit sample bottles. Caps shall be lined with PTFE.
- 5.1.3** Cleaning.
- 5.1.3.1** Bottles are detergent water washed, then rinsed with solvent or baked at 450°C for a minimum of 1 hour.
- 5.1.3.2** Liners are detergent water washed, then rinsed with reagent water and solvent, and baked at approximately 200°C for a minimum of 1 hour prior to use.
- 5.1.4** Compositing equipment: Automatic or manual compositing system incorporating glass containers cleaned per bottle cleaning procedure above. Sample containers are kept at 0 to 4°C during sampling. Glass or PTFE tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing shall be thoroughly rinsed with methanol, followed by repeated rinsings with reagent water to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.
- 5.2** Extraction bottle: 4-oz with PTFE-lined screw-cap, cleaned by solvent rinse or baking at 450°C for a minimum of 1 hour.
- 5.3** pH meter, with combination glass electrode.
- 5.4** Sample vials: Amber glass, 1- to 5-mL with PTFE-lined screw- or crimp-cap, to fit GC autosampler.
- 5.5** Balance: Analytical, capable of weighing 0.1 mg.
- 5.6** Miscellaneous glassware.
- 5.6.1** Pipettes, glass, volumetric, 1.00-, 5.00-, and 10.0-mL.
- 5.6.2** Pipettes, glass, Pasteur.
- 5.6.3** Volumetric flasks, 10.0-, 25.0-, and 50.0-mL.

- 5.7 Gas chromatograph: Shall have splitless or on-column simultaneous automated injection into separate capillary columns with a nitrogen-phosphorus detector at the end of each column, temperature program with isothermal holds, data system capable of recording simultaneous signals from the two detectors, and shall meet all of the performance specifications in Section 12.
- 5.7.1 GC columns: Bonded-phase fused-silica capillary.
- 5.7.1.1 Primary: 30 m long ( $\pm 3$  m) x 0.5 mm ( $\pm 0.05$  mm) ID, DB-608 (or equivalent).
- 5.7.1.2 Confirmatory: DB-1701, or equivalent, with same dimensions as primary column.
- 5.7.2 Data system: Shall collect and record GC data, store GC runs on magnetic disk or tape, process GC data, compute peak areas, store calibration data including retention times and calibration factors, identify GC peaks through retention times, compute concentrations, and generate reports.
- 5.7.2.1 Data acquisition: GC data shall be collected continuously throughout the analysis and stored on a mass storage device.
- 5.7.2.2 Calibration factors and calibration curves: The data system shall be used to record and maintain lists of calibration factors, and multi-point calibration curves (Section 7). Computations of relative standard deviation (coefficient of variation) are used for testing calibration linearity. Statistics on initial (Section 8.2) and ongoing (Section 12.5) performance shall be computed and maintained.
- 5.7.2.3 Data processing: The data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC analysis. Software routines shall be employed to compute and record retention times and peak areas. Displays of chromatograms and library comparisons are required to verify results.
- 5.7.3 Nitrogen phosphorus detector: Thermionic bead or alkali flame detector, capable of detecting 600 pg of MITC under the analysis conditions given in Table 1.

## 6. REAGENTS AND STANDARDS

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- 6.1 Sample preservation: Sodium thiosulfate (ACS), granular.

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- 6.2** pH adjustment.
- 6.2.1** Sodium hydroxide (10N): Dissolve 40 g NaOH in 100 mL reagent water.
- 6.2.2** Sulfuric acid (1+1): Reagent grade, 6N in reagent water. Slowly add 50 mL H<sub>2</sub>SO<sub>4</sub> (specific gravity 1.84) to 50 mL reagent water.
- 6.2.3** Potassium hydroxide: 37% (w/v). Dissolve 37 g KOH in 100 mL reagent water.
- 6.3** Solvents: Methylene chloride, ethyl acetate, and acetone; pesticide-quality; lot-certified to be free of interferences.
- 6.4** Reagent water: Water in which the compounds of interest and interfering compounds are not detected by this method.
- 6.5** Salt: Sodium chloride, spread approximately 1 cm deep in a baking dish and baked at 450°C for a minimum of 1 hour.
- 6.6** Standard solutions: Purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96% or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at -20 to -10°C in screw-capped vials with PTFE-lined lids. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use.
- 6.7** Preparation of stock solutions: Prepare in ethyl acetate per the steps below. Observe the safety precautions in Section 4.
- 6.7.1** Dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 10 mg MITC in a 10-mL ground-glass stoppered volumetric flask and fill to the mark with ethyl acetate. After the MITC is completely dissolved, transfer the solution to a 15-mL vial with PTFE-lined cap.
- 6.7.2** Stock solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards.
- 6.7.3** Stock solutions shall be replaced after 6 months, or sooner if comparison with quality control check standards indicates a change in concentration.
- 6.8** Secondary mixtures: Using stock solutions (Section 6.7), prepare mixtures for calibration and calibration verification (Sections 7.3 and 12.4), for initial and ongoing precision and recovery (Sections 8.2 and 12.5), and for spiking into the sample matrix (Section 8.3).
- 6.8.1** Calibration solutions: Prepare MITC in ethyl acetate at concentrations of 0.2 µg/mL, 1.0 µg/mL, and 5.0 µg/mL. The midpoint solution (1.0 µg/mL) is used for calibration verification (Section 12.4).
- 6.8.2** Precision and recovery standard: Prepare MITC in acetone at a concentration of 25 µg/mL.

**6.8.3** Matrix spike solution: Prepare dazomet in acetone at a concentration of 25 µg/mL.

**6.9** Stability of solutions: All standard solutions (Sections 6.7 and 6.8) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area remains within ±15% of the area obtained in the initial analysis of the standard.

## **7. SETUP AND CALIBRATION**

**7.1** Configure the GC system as given in Section 5.7 and establish the operating conditions in Table 1.

**7.2** Attainment of minimum level: Determine that each column/detector system meets the minimum level for MITC (Table 1).

**7.3** Calibration.

**7.3.1** Inject 3 µL of each calibration solution (Section 6.8.1) into each GC column/detector pair, beginning with the lowest level mixture and proceeding to the highest. For each compound, compute and store, as a function of the concentration injected, the retention time and peak area on each column/detector system (primary and confirmatory).

**7.3.2** Calibration factor (ratio of area to amount injected).

**7.3.2.1** Compute the coefficient of variation (relative standard deviation) of the calibration factor over the calibration range for MITC on each column/detector system.

**7.3.2.2** Linearity: If the calibration factor is constant ( $C_v < 20\%$ ) over the calibration range, an average calibration factor may be used; otherwise, the complete calibration curve (area vs. amount) shall be used.

## **8. QUALITY CONTROL**

**8.1** Each laboratory that uses this method is required to operate a formal quality control program.<sup>4</sup> The minimum requirements of this program consist of an initial demonstration of laboratory capability, an ongoing analysis of standards and blanks as tests of continued performance, and analysis of spiked samples to assess accuracy. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

**8.1.1** The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

**8.1.2** The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance requirements are met. Each

time a modification is made to the method or a cleanup procedure is added, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance. If detection the detection limit for dazomet will be affected by the modification, the analyst is required to repeat demonstration of the detection limit (Section 7.2).

- 8.1.3** The laboratory shall, on an ongoing basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (Section 6.8.2) that the analysis system is in control. These procedures are described in Sections 12.1, 12.4, and 12.5.
  - 8.1.4** The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Section 8.3.
  - 8.1.5** Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 8.4.
- 8.2** Initial precision and recovery: To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:
- 8.2.1** Extract, concentrate, and analyze one set of four 50-mL aliquots of reagent water spiked with 0.1 mL of the precision and recovery standard (Section 6.8.2) according to the procedure in Section 10.
  - 8.2.2** Using results of the set of four analyses, compute the average percent recovery (X) and the coefficient of variation (C<sub>v</sub>) of percent recovery(s) for MITC.
  - 8.2.3** Compare s and X with the corresponding limit for initial precision and recovery in Table 1. If s and X meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, s exceeds the precision limit or X falls outside the range for accuracy, system performance is unacceptable. In this case, correct the problem and repeat the test.
- 8.3** Method accuracy: The laboratory shall spike (matrix spike) at least 10% of the samples from a given site type (e.g., influent to treatment, treated effluent, produced water). If only one sample from a given site type is analyzed, a separate aliquot of that sample shall be spiked.
- 8.3.1** The concentration of the matrix spike shall be determined as follows:
    - 8.3.1.1** If, as in compliance monitoring, the concentration of dazomet in the sample is being checked against a regulatory concentration limit, the matrix spike shall be at that limit or at 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration is larger.
    - 8.3.1.2** If the concentration is not being checked against a regulatory limit, the matrix spike shall be at 50 µg/L or at 1 to 5 times higher than the background concentration, whichever concentration is larger.

- 8.3.1.3** If it is impractical to determine the background concentration before spiking (e.g., maximum holding times will be exceeded), the matrix spike concentration shall be the regulatory concentration limit, if any; otherwise, the larger of either 5 times the expected background concentration, or 50 µg/L (the concentration produced by 0.1 mL of the matrix spike solution spiked into a 50-mL sample).
- 8.3.2** Analyze one sample aliquot to determine the background concentration (B) of dazomet. If necessary, prepare a standard solution appropriate to produce a level in the sample 1 to 5 times the background concentration. Spike a second sample aliquot with the standard solution and analyze it to determine the concentration after spiking (A) with dazomet. Calculate the percent recovery (P):

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**Equation 1**

$$P = \frac{100 (A-B)}{T}$$

where

$T$  = True value of the spike

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- 8.3.3** Compare the percent recovery for dazomet with the corresponding QC acceptance criteria in Table 1. If dazomet fails the acceptance criteria for recovery, the sample is complex and must be diluted and reanalyzed per Section 15.
- 8.3.4** As part of the QC program for the laboratory, method accuracy for samples shall be assessed and records shall be maintained. After the analysis of five spiked samples of a given matrix type (treated effluent, influent to treatment, produced water) in which the recovery test (Section 8.3.3) is passed, compute the average percent recovery (P) and the standard deviation of the percent recovery ( $s_p$ ). Express the accuracy assessment as a percent recovery interval from  $P - 2s_p$  to  $P + 2s_p$  for each matrix. For example, if  $P = 90\%$  and  $s_p = 10\%$  for five analyses of wastewater, the accuracy interval is expressed as 70 to 110%. Update the accuracy assessment in each matrix on a regular basis (e.g., after each five to ten new accuracy measurements).
- 8.4** Blanks: Reagent water blanks are analyzed to demonstrate freedom from contamination.
- 8.4.1** Extract and concentrate a 50-mL reagent water blank with each sample batch (samples started through the extraction process on the same 8-hour shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (Section 12.5) to demonstrate freedom from contamination.
- 8.4.2** If MITC or any potentially interfering compound is found in an aqueous blank at greater than 2 µg/L (assuming the same calibration factor as MITC for

interfering compounds), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

- 8.5** The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (Section 7), calibration verification (Section 12.4), and for initial (Section 8.2) and ongoing (Section 12.5) precision and recovery should be identical, so that the most precise results will be obtained. The GC instrument will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of the analytes given in this method.
- 8.6** Depending on specific program requirements, field replicates and field spikes may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

## **9. SAMPLE COLLECTION, PRESERVATION, AND HANDLING**

- 9.1** Collect samples in glass containers following conventional sampling practices,<sup>5</sup> except that the bottle shall not be prerinsed with sample before collection. Aqueous samples which flow freely are collected in refrigerated bottles using automatic sampling equipment.
- 9.2** Maintain samples at 0 to 4°C from the time of collection until extraction. If the samples will not be extracted within 72 hours of collection, adjust the sample to a pH greater than 9.0 using sodium hydroxide solution. Record the volume used. If residual chlorine is present in aqueous samples, add 80 mg sodium thiosulfate per liter of water. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine.<sup>6</sup>
- 9.3** Begin sample extraction within 7 days of collection, and analyze all extracts within 40 days of extraction.

## **10. SAMPLE HYDROLYSIS AND EXTRACTION**

- 10.1** Hydrolysis and preparation of QC aliquots.
- 10.1.1** Pour 50 mL of sample into a clean 4-oz bottle. If a matrix spike is to be prepared, pour 50 mL into a second clean bottle.
- 10.1.2** For each sample or sample batch (to a maximum of 20) to be extracted at the same time, place two 50-mL aliquots of reagent water in clean 4-oz bottles. One reagent water aliquot serves as the blank.
- 10.1.3** Spike 0.1 mL of the precision and recovery standard (Section 6.8.2) into the remaining reagent water aliquot.
- 10.1.4** Spike 0.1 mL of the matrix spike solution (Section 6.8.3) into the sample aliquot used for the matrix spike.

**10.1.5** Test the pH of the sample and QC aliquots with a pH meter and adjust to 10 to 12 with potassium hydroxide solution. Cap and shake the bottles vigorously to mix. Allow to stand.

**10.1.6** Test and adjust the pH after 0.5 to 1 hour. Allow to stand for an additional 2 to 3 hours.

**10.1.7** Extract the sample and QC aliquots per Section 10.2.

## **10.2** Extraction.

**10.2.1** Add 20 g of clean NaCl (Section 6.5) and 2.5 mL of ethyl acetate to each sample and QC aliquot and cap tightly.

**10.2.2** Shake vigorously for 2 to 5 minutes. Allow the bottle to stand for 10 minutes for the phases to separate.

**10.2.3** Using a Pasteur pipette, transfer the organic phase to a GC autosampler vial. Measure its volume.

## **11. GAS CHROMATOGRAPHY**

Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table is the retention time for MITC achieved under these conditions. An example of the separation achieved by the primary column is shown in Figure 1.

**11.1** Calibrate the system as described in Section 7.

**11.2** Set the injection volume on the autosampler to inject 3.0  $\mu$ L of all standards and extracts of blanks and samples.

**11.3** Set the data system or GC control to start the temperature program upon sample injection, and begin data collection after the solvent peak elutes. Set the data system to stop data collection after the last analyte is expected to elute and to return the column to the initial temperature.

## **12. SYSTEM AND LABORATORY PERFORMANCE**

**12.1** At the beginning of each 8-hour shift during which analyses are performed, GC system performance and calibration are verified on both column/detector systems. For these tests, analysis of the calibration verification standard (Section 6.8.1) shall be used to verify all performance criteria. Adjustment and/or recalibration (per Section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

**12.2** Retention times: The absolute retention time of the peak maxima shall be within  $\pm 10$  seconds of the retention times in the initial calibration (Section 7.3.1).

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- 12.3** GC resolution: Resolution is acceptable if the peak width at half-height is less than 10 seconds.
- 12.4** Calibration verification.
- 12.4.1** Inject the calibration verification standard (Section 6.8.1).
- 12.4.2** Compute the concentration of MITC based on the calibration factor or calibration curve (Section 7.3).
- 12.4.3** Compare this concentration with the limits for calibration verification in Table 1. If the recovery meets the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, the recovery falls outside the calibration verification range, system performance is unacceptable. In this case, correct the problem and repeat the test, or recalibrate (Section 7).
- 12.5** Ongoing precision and recovery.
- 12.5.1** Analyze the extract of the precision and recovery standard extracted with each sample batch (Section 10.1.3).
- 12.5.2** Compute the percent recovery of MITC.
- 12.5.3** Compare the percent recovery with the limits for ongoing recovery in Table 1. If the recovery meets the acceptance criteria, the extraction and concentration processes are in control and analysis of blanks and samples may proceed. If, however, the recovery falls outside the acceptable range, these processes are not in control. In this event, correct the problem, re-extract the sample batch, and repeat the ongoing precision and recovery test.
- 12.5.4** Add results which pass the specifications in Section 12.5.3 to initial and previous ongoing data. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory data quality for each analyte by calculating the average percent recovery ( $R$ ) and the standard deviation of percent recovery  $s_r$ . Express the accuracy as a recovery interval from  $R-2s_r$  to  $R+2s_r$ . For example, if  $R=95\%$  and  $s_r=5\%$ , the accuracy is 85 to 105%.

### **13. QUALITATIVE DETERMINATION**

- 13.1** Qualitative determination is accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (Section 12.1), and with data stored in the retention time and calibration libraries (Section 7.3.1). Identification is confirmed when retention-time and amounts agree per the criteria below.
- 13.2** On each column/detector system, establish a retention-time window  $\pm 20$  seconds on either side of the retention-time in the calibration data (Section 7.3.1).
- 13.3** If the MITC peak from the analysis of a sample or blank is within a window (as defined in Section 13.2) on the primary column/detector system, it is considered tentatively

identified. A tentatively identified compound is confirmed when (1) the retention time for the compound on the confirmatory column/detector system is within the retention-time window on that system, and (2) the computed amounts (Section 14) on each system (primary and confirmatory) agree within a factor of 3.

#### 14. QUANTITATIVE DETERMINATION

- 14.1 Using the GC data system, compute the concentration of the analyte detected in the extract (in milligrams per milliliter) using the calibration factor or calibration curve (Section 7.3.2).
- 14.2 Compute the concentration in the sample using the following equation:

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#### Equation 2

$$C_s = \frac{(2.22) (V_e) (C_{ex})}{V_s}$$

where

$C_s$  = Concentration in the sample, in  $\mu\text{g/L}$

2.22 = Converts MITC (MW 73.12) to dazomet (MW 162.27)

$V_e$  = Extract total volume, in mL

$C_{ex}$  = Concentration in the extract, in  $\mu\text{g/mL}$

$V_s$  = Volume of sample extracted, in L

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- 14.3 If the concentration of MITC exceeds the calibration range of the system, the extract is diluted by a factor of 10, and a 3- $\mu\text{L}$  aliquot of the diluted extract is analyzed.
- 14.4 Report results for dazomet found in all standards, blanks, and samples to three significant figures. Results for samples that have been diluted are reported at the least dilute level at which the concentration is in the calibration range.

#### 15. ANALYSIS OF COMPLEX SAMPLES

- 15.1 Some samples may contain high levels (>1000 ng/L) of dazomet or of interfering compounds, and/or polymeric materials. Some samples may form emulsions when extracted (Section 10.2); others may overload the GC column and/or detector. In these instances, the extract is diluted by a factor of 10 and reanalyzed (Section 1 4.3).
- 15.2 Recovery of matrix spikes: In most samples, matrix spike recoveries will be similar to those from reagent water. If the matrix spike recovery is outside the range specified in Table 1, the sample is diluted by a factor of 10, respiked, and reanalyzed. If the matrix spike recovery is still outside the range, the method may not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

**16. METHOD PERFORMANCE**

**16.1** This method is based on industry Method 131.<sup>7</sup>

**16.2** Development of this method is detailed in Reference 8.

## References

1. "Carcinogens—Working with Carcinogens." Department of Health, Education, and Welfare; Public Health Service; Center for Disease Control; National Institute for Occupational Health and Safety: Publication 77-206, August 1977.
2. "OSHA Safety and Health Standards, General Industry" (29 *CFR* 1910). Occupational Safety and Health Administration: January 1976.
3. "Safety in Academic Chemistry Laboratories," American Chemical Society Committee on Chemical Safety: 1979.
4. "Handbook of Quality Control in Wastewater Laboratories," U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH: EPA-600/4-79-019, March 1979.
5. "Standard Practice for Sampling Water" (ASTM Annual Book of Standards), American Society for Testing and Materials, Philadelphia, Pennsylvania: 76, 1980.
6. "Methods 330.4 and 330.5 for Total Residual Chlorine," U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH: EPA-600/4-70-020, March 1979.
7. "Determination of Metham (Vapam) in Wastewater" (Method 131), *Methods for Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater*. U.S. Environmental Protection Agency Effluent Guidelines Division (WH-552), Washington, DC: EPA 440/1-83/079-C, January 31, 1983.
8. "Narrative for SAS 1019," Pacific Analytical, Inc.: September 1989. Available from the U.S. Environmental Protection Agency Sample Control Center, 300 N. Lee St., Alexandria, VA 22314 (703-557-5040).

Table 1. GC Data and Method Acceptance Criteria for Dazomet\*

<b>Acceptance Criterion</b>	<b>Specification</b>	<b>Note</b>
Minimum Level	10 µg/L	1
Method Detection Limit	3 µg/L	2
Calibration Verification (Section 12.4)	0.8–1.3 µg/mL	3
Initial Precision and Recovery (Section 8.2)		
Precision [standard deviation (s)]	23 µg/L	
Recovery [mean (X)]	18–75 µg/L	
Ongoing Precision and Recovery (Section 12.5)	15–78 µg/L	
Matrix Spike Recovery (Section 8.3.3)	16–123%	
MITC Retention-time		5
DB-608	2.17 minutes	
DB-1701	3.80 minutes	

\*(3,5-dimethyl-2H-tetrahydro-1,3,5-thiadiazine-2-thione) detected as methyl isothiocyanate (MITC).

1. This is a minimum level at which the analytical system shall give recognizable signals and acceptable calibration points.
2. Estimated; 40 *CFR* Part 136, Appendix B.
3. Test concentration 1.0 µg/mL.
4. Test concentration 50 µg/L.
5. Columns: 30 mm long x 0.53 mm ID. DB-608: 0.83 µ. DB-1701: 1.0 µ. Conditions suggested to meet retention times shown: 50°C for 1 minute, 50 to 200° at 10°C/min. Carrier gas flow rates approximately 7 mL/min.

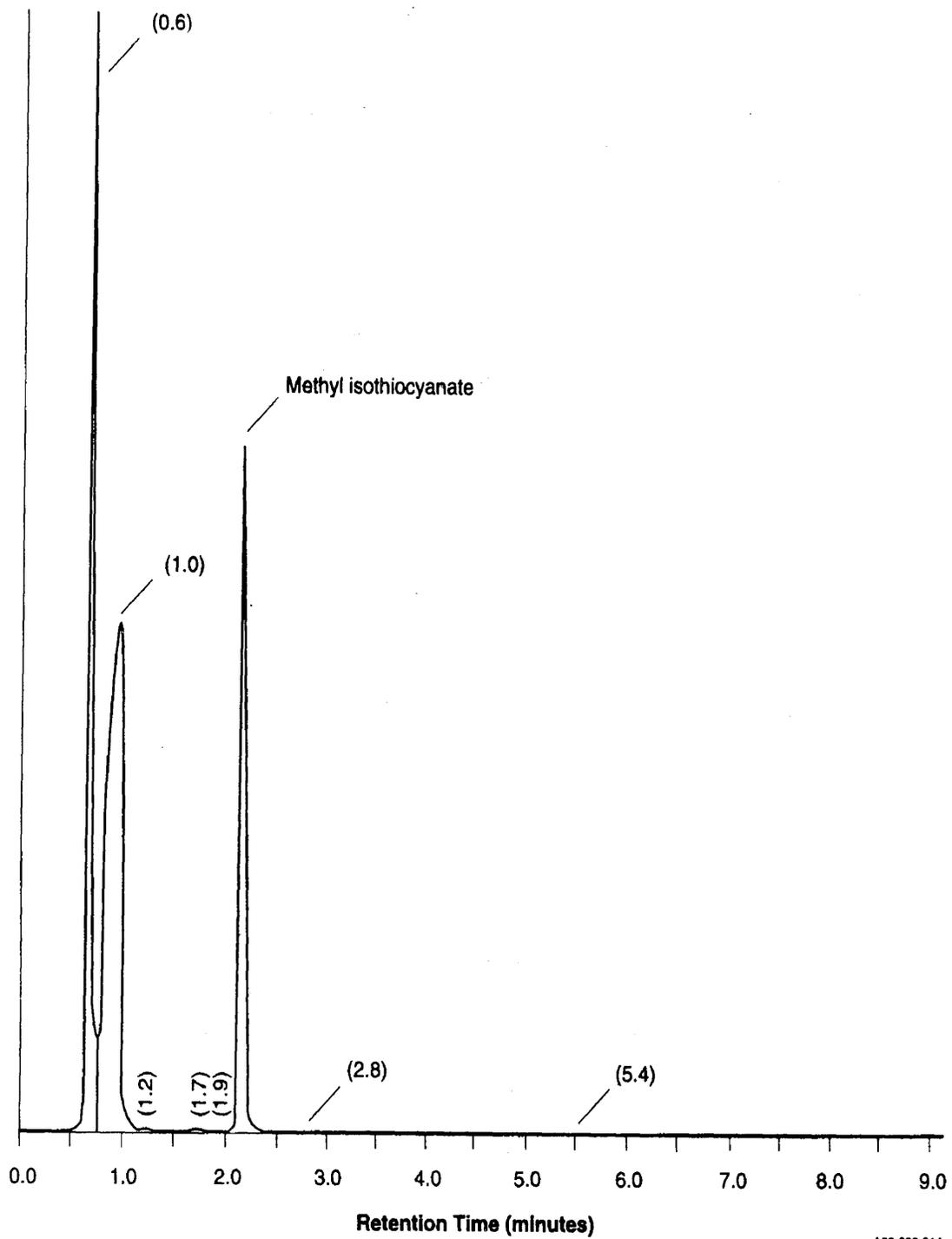


Figure 1. Chromatogram of Methyl Isothiocyanate