

Fast Method for Determination of Nitrobenzene in Water using Agilent 5975T LTM GC/MSD

Application Note

Environmental

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Abstract

Recently, urgent water pollution cases caused by petrochemical plant explosion accidents have increased. Therefore, the requirements of emergency monitoring with transportable instruments continue to grow. This application work developed a simple and fast method for monitoring nitrobenzene in water with the transportable Agilent 5975T LTM GC/MSD. With this method, ppb (ng/mL) levels of nitrobenzene in water can be detected in less than 5 minutes. The calculated MDL (method detection limit) of nitrobenzene here is 0.017 $\mu\text{g}/\text{mL}$ for standard solution. Liquid solvent extraction was used to extract nitrobenzene from water. Sample preparation is easily performed in a mobile lab.



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Introduction

Nitrobenzene is an important raw material widely used in industrial production. It also is an extremely toxic chemical that is harmful to human health. It is often discharged into the water by industrial production processes, resulting in the pollution of surface water and groundwater. With industrial development and increasing discharge of industrial wastewater, determination of nitrobenzene has become one of the most important monitoring targets for major rivers in the world. Nitrobenzene is also one of the target compounds in EPA Method 625.

A fast method for determining nitrobenzene in water is developed following a simple sample extraction procedure. The method is for the Agilent 5975T LTM GC/MSD, which is an integrated and transportable instrument with rapid separation

capability because of its use of low thermal mass (LTM) technology. The experiment proved that this method is accurate, rapid and sensitive enough for determining nitrobenzene in water at low ppm levels, and can be applied in emergency monitoring.

Experimental

This method uses a narrow bore (0.18 mm id) column. LTM column technology provides fast heating and cooling capability, reducing the run time to 4 min. In this method, the cooldown time (from 250 °C to 60 °C) is no more than 1 min. Short cycle times (around 5 min) quickly provides the analyst with quantitative reports for emergency monitoring tasks. A significantly simple and fast sample preparation is adopted in this solution. The instrumental parameters are listed in Table 1.

Table 1. GC/MS Instrument Conditions

GC		MS	
Injector temperature	200 °C	Data acquire mode	Scan and SIM
Injection volume	1 µL	Scan mass range	50–300 amu
Column flow	1 mL/min (He)	Scan speed	Faster
Oven temperature	250 °C	SIM ions	123,77,93
Split ratio	30:1	Transfer line	250 °C
LTM program	60 °C (0.25 min) >150 °C (0 min) at 50 °C/min >250 °C (1.8 min) at 100 °C/min	Ion source	230 °C
LTM column	HP-5ms 8.5 m × 0.18 mm, 0.18 µm	Quad.	150 °C
		Solvent delay	1 min

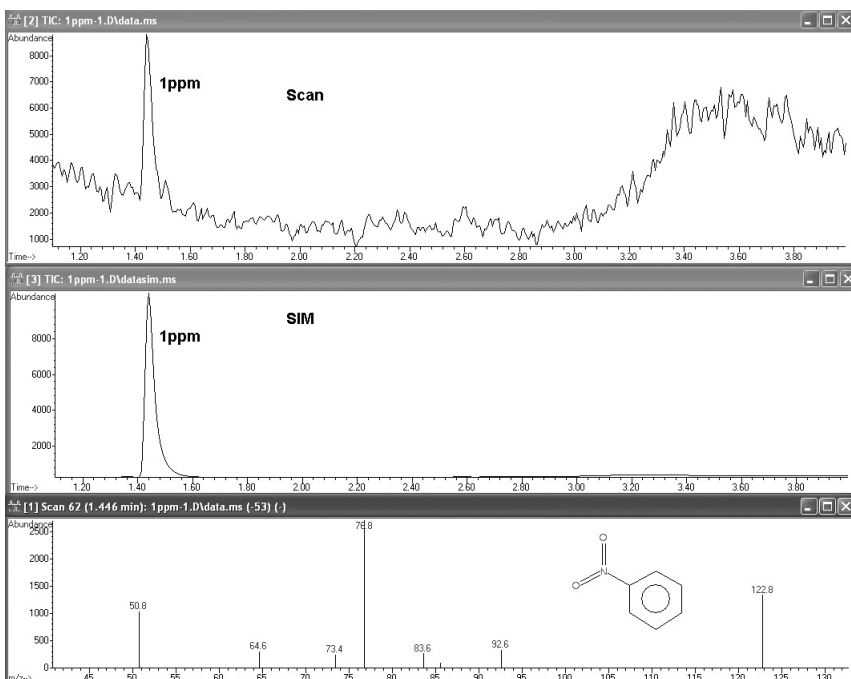


Figure 1. Chromatogram of nitrobenzene standard solution (1 ppm).

Method Performance

Calibration

The calibration curve was measured at a concentration range of 0.1 to 10 µg/mL. Five different concentration levels (0.1, 0.5, 1, 5 and 10 µg/mL) of the standard solution were prepared by diluting the liquid standard using methylene chloride. The calibration curve of nitrobenzene in Figure 2 shows the wide range of linearity.

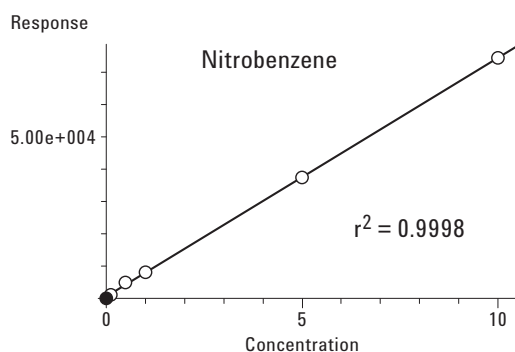


Figure 2. Calibration curve of nitrobenzene.

Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero

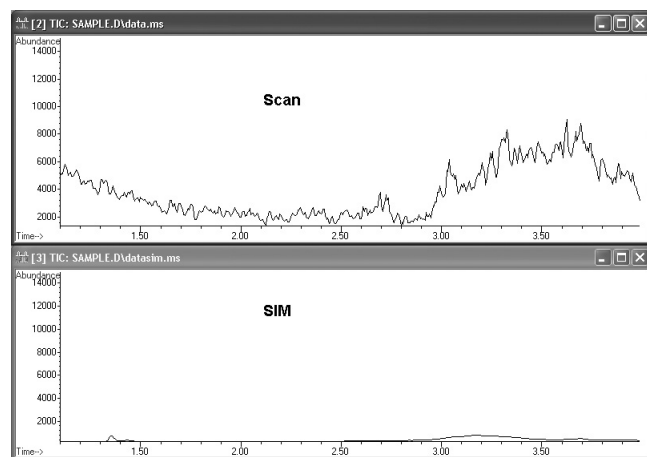


Figure 3. Chromatogram of running water sample (Calculated conc: N.D.).

(EPA 625). To establish the MDL, make seven repeated measurements of the target compound at a concentration near the expected detection limit, calculate the standard deviation for the seven concentrations, and multiply this value by the confidence coefficient 3.14. The method detection limit of nitrobenzene is 0.017 µg/mL using this approach. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

Repeatability

Repeatability is tested by measuring two different concentration levels of nitrobenzene standard solutions, each with ten runs. The relative standard deviations (RSD) of MSD responses were calculated and used in Table 2.

Table 2. Relative Standard Deviations of MSD Responses

ppm	1	2	3	4	5	6	7	8	9	10	% RSD
0.1	1218	1203	1226	1228	1156	1208	1205	1211	1206	1169	1.93
0.5	5259	5118	5313	5315	5175	5328	5298	5344	5100	5077	1.99

Sample Preparation and Recovery

Fifty milliliters of running water sample were placed in the separating funnel and 2 mL of methylene chloride were added. The mixture was manually agitated for one minute to extract nitrobenzene into the methylene chloride layer and the methylene chloride layer was collected. A 1-µL volume of collected sample solution was injected into the Agilent 5975T LTM GC/MSD. The resulting chromatogram is shown in Figure 3. A 2 mL of 2.5-µg/mL amount of standard solution was spiked into 50 mL water to test the recovery. The calculated recovery was 108%.

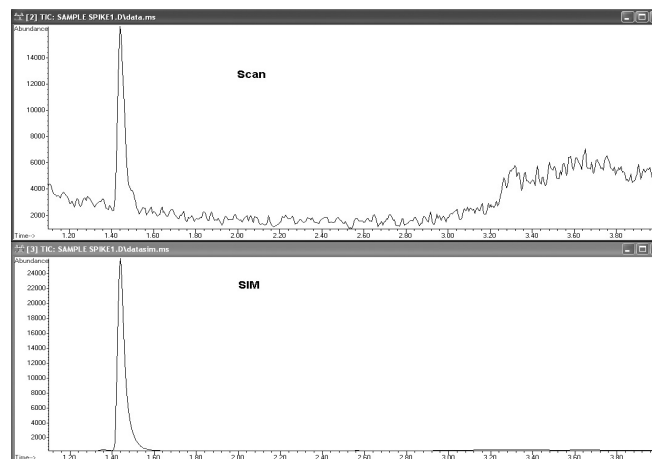


Figure 4. Chromatogram of sample spike solution (2.5 µg/mL).

Recovery was calculated by this formula:

$$\text{Recovery (\%)} = \frac{(\text{conc of spiked sample} - \text{conc of unspiked sample})}{\text{conc added (2.5 } \mu\text{g/mL)}} \times 100$$

The concentration of nitrobenzene in water was calculated by the following formula:

$$\text{Conc of nitrobenzene in water (} \mu\text{g/mL)} = \frac{\text{calculated conc by calibration curve } \mu\text{g/mL}}{\text{volume of water (50 mL)}} \times \text{volume of solvent (2mL)}$$

Conclusions

A simple and fast method for the determination of nitrobenzene in water is described for the Agilent 5975T LTM GC/MSD using the narrow bore LTM column and easy-to-operate sample preparation. This solution was developed for field emergency use with 5 min of injection-to-injection cycle time. It should be highlighted that the method performance shows sensitivity and accuracy comparable to that obtained from stationary lab instruments. This has been the biggest challenge for field-transportable GC/MSDs in the past.

References

U.S. EPA Method 625.

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