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Determination of Aqueous Nitrobenzene in Environmental Samples

Nitrobenzene is a toxic environmental pollutant that is suspected to have effects on the reproductive and nervous systems of those exposed.[1] As such, nitrobenzene is a target analyte for several United States Environmental Protection Agency (EPA) Methods, e.g. 625.2 The instrumental method outlined in this application note is aimed at the rapid, field-based determination of aqueous-phase nitrobenzene following a simplified sample extraction procedure.

Sensitive and selective detection and identification of a wide variety of chemical species, including environmental pollutants, is a necessity in many applications. Field-deployable sensors are preferred in many instances, in that valuable time, resources, and chemical information are conserved by performing analyses directly on-site, rather than retrieving samples to be studied at a later time in the laboratory. The Griffin family of mass spectrometers has been developed to fulfill this need (see Figure 1.).

The Griffin 400™ used in this work was equipped with a low thermal mass gas chromatograph (LTM-GC) for rapid separation prior to mass spectral analysis. Three separations were performed on an Rtx-TNT (5 m, 0.25 mm i.d. and 0.25 um df) with helium carrier gas flowing at 1 mL/min. All injections were performed split (20:1) with 1 µL injection volumes. The GC temperature program was as follows:

40° C - 150° C at 40° C/min

150° C - 250° C at 90° C/min

The mass spectrometer was operated in full MS mode scanning over the mass range m/z 40 -250. A typical nitrobenzene chromatogram is shown in Figure 2. A representative nitrobenzene mass spectrum is shown in Figure 3.

Figure 1. The Griffin 400 - Mobile GC/MS/MS

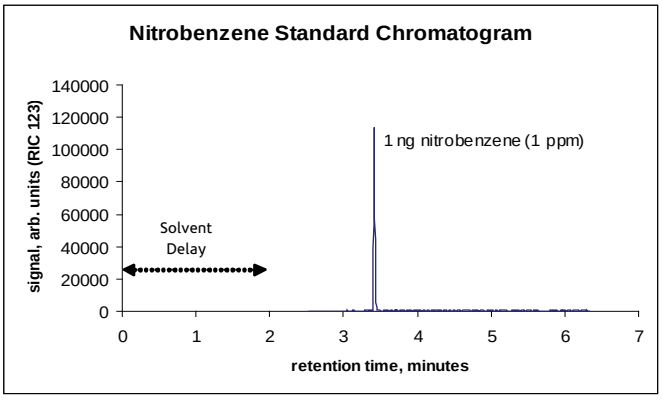

Figure 2. Nitrobenzene standard chromatogram.

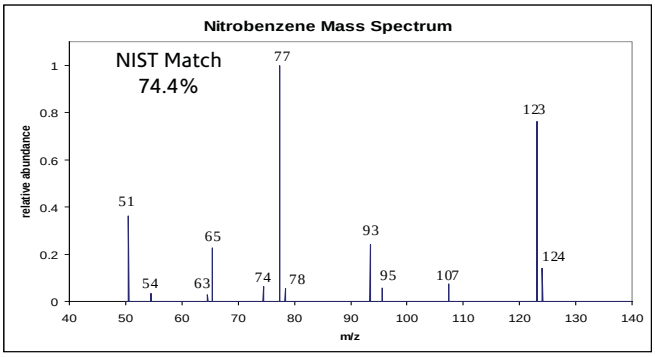

Figure 3. Nitrobenzene mass spectrum.

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Quantitative calibration was performed with liquid standards of nitrobenzene prepared in methylene chloride. Standards were made across the range 50 pg/µL (50 ppb)- 10 ng/µL (10 ppm). It should be noted that the regression is virtually unchanged when the highest point is omitted. This point is included in the graph to confirm the linear dynamic range of the instrument

Figure 4 shows the calibration curve generated prior to the sample extractions. The range of linearity spanned over the 4 orders of magnitude concentration range of the standards. Aqueous mixtures of nitrobenzene were prepared to simulate contaminated environmental samples. A solution of 500 ppt nitrobenzene in water was used to test a rapid sample extraction method. A 50 mL aliquot of the 500 ppt nitrobenzene solution was mixed with 1 mL methylene chloride and the mixture was manually agitated for 1 minute to extract nitrobenzene into the methylene chloride layer. The methylene chloride layer was poured off and a 1 µL volume was injected into the Griffin 400 instrument. The resulting chromatogram is shown in Figure 5.

Subsequent analyses were performed with rapid separations afforded by the LTM-GC. The rapid separation used a temperature ramp of 40° C - 250° C at 90° C/min. The resulting chromatogram is shown in figure 6. The combined rapid extraction (1 min) and fast GC separation (43 s retention time) allows for aqueous nitrobenzene determinations in under 2 minutes. This rapid, simplified analysis is ideal for environmental monitoring at the point of possible contamination in the field without the need for complex sample handling/extraction procedures. The ease of sample preparation in conjunction with rapid separation and sensitive, information-rich mass spectrometric data allows for a complete analysis solution.

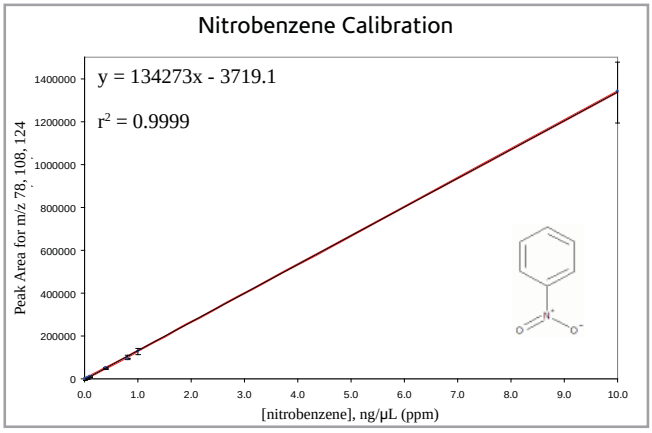

Figure 4. Nitrobenzene calibration curve.

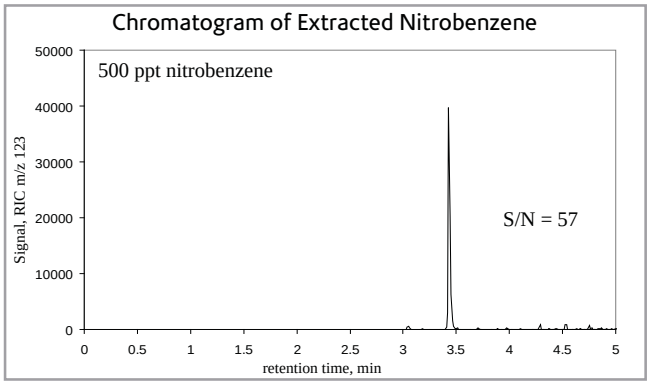

Figure 5. Chromatogram for 500 ppt extracted nitrobenzene sample.

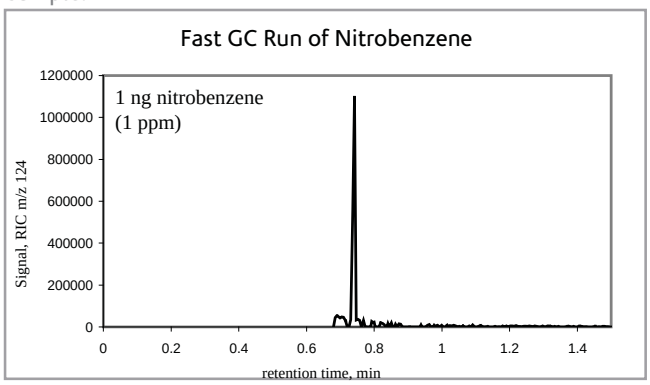

Figure 6. Rapid nitrobenzene separation performed on LTM-GC.

REFERENCES

(1) U.S. EPA, U.S. Environmental Protection Agency Health and Environmental Effects Profile for Nitrobenzene, Office of Solid Waste and Emergency Response, Washington, D.C. ECAO-CIN-P145, 1985.
(2) U.S. EPA Method 625.
(3) Griffin Analytical Technologies, LLC, Application Note 6.

These data represent typical results.