A Sensitive and Selective Method for the Analysis of Hydrazines in Water and Soil

Jia Wang and Chuck Neslund
Lancaster Laboratories
2425 New Holland Pike
Lancaster PA 17601

Introduction

- Hydrazines (hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine) are most commonly known for their use as rocket propellants
- The use of LC/MS/MS for hydrazines analysis is not reported
- Recovery of hydrazines in soil samples is challenging , especially from some site specific matrices

Objective

- Develop LC/MS/MS method for hydrazines analysis
- Develop extraction procedure to recover hydrazines from soil samples

Method

- Hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine in aqueous samples or soil extracts are derivatized with carbonyl compound to form the corresponding hydrazones.
- The resulting solution is analyzed by LC/MS/MS operated in positive atmospheric pressure chemical ionization (APCI) mode.
- Separation and detection is performed using a Thermo Scientific Accela Autosampler and quaternary U-HPLC pump equipped with TSQ Quantum Access Triple Quadrupole mass spectrometer
- Small quantity of sample (1 mL aqueous sample or 1 gram soil sample) is required

Results

Figure 1. Example Chromatogram (TIC and SRM) of Calibration Standard (Level 5)

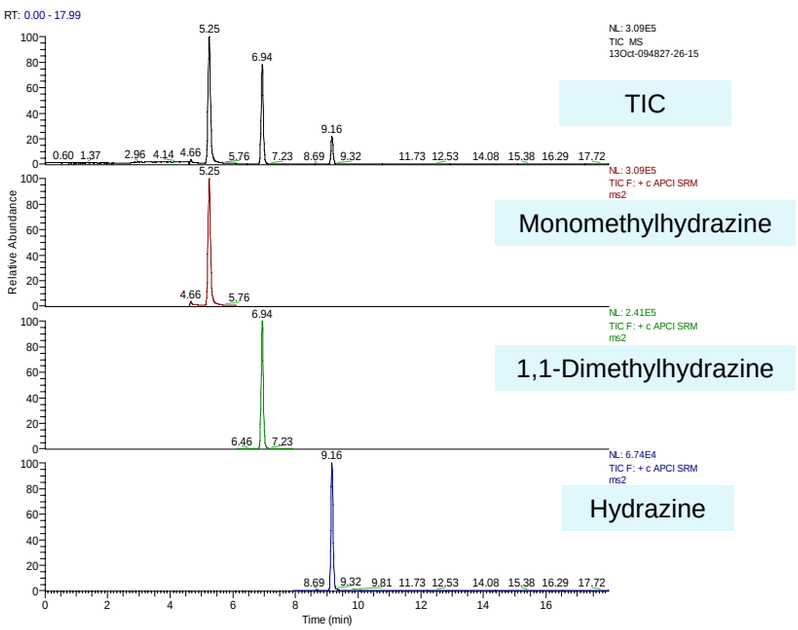
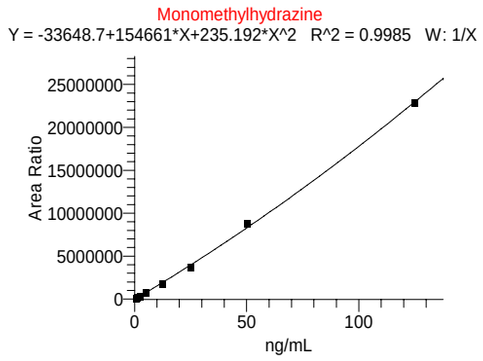
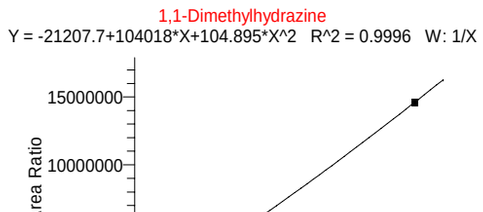


Table 1. Concentration Range of Calibration Curve (ng/mL)

	Hydrazine	Monomethylhydrazine & 1,1-Dimethylhydrazine
Cal. 1	0.1	0.5
Cal. 2	0.25	1.25
Cal. 3	0.5	2.5
Cal. 4	1	5
Cal. 5	2.5	12.5
Cal. 6	5	25
Cal. 7	10	50
Cal. 8	25	125

Figure 2. Calibration Curve of Hydrazine, Monomethylhydrazine and 1,1-Dimethylhydrazine





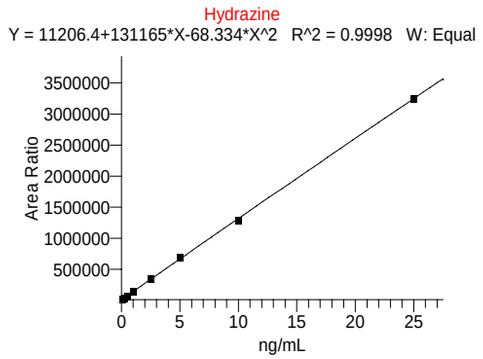


Table 2. Recovery of Hydrazines from Spiked Tap Water[1,2]

	Monomethyl Hydrazine	1.1-Dimethyl Hydrazine	Hydrazine
Milli Q water + spike	59	80	94
Tap water + spike-1	61	80	80
Tap water + spike-2	57	73	79
Tap water + spike-3	59	76	80

[1] Spiking experiment was conducted in tap water containing no measurable analytes

[2] Hydrazine was spiked at 12.5 ng/mL. Monomethylhydrazine and 1,1-dimethylhydrazine was spiked at 62.5 ng/mL.

Table 3. Recovery of Hydrazines from Spiked Soil Sample[3,4]

	Monomethyl Hydrazine	1.1-Dimethyl Hydrazine	Hydrazine
Ottawa Sand + Spike	116	116	104
Soil A + Spike-1	21	55	35
Soil A + Spike-2	19	52	35
Soil B + Spike-1	24	51	48
Soil B + Spike-2	25	54	52
Soil C + Spike-1	23	57	44
Soil C + Spike-2	21	43	44
Soil D + Spike-1	66	88	70
Soil D + Spike-2	87	104	90
Soil E + Spike-1	26	51	57
Soil E + Spike-2	29	63	58
Soil F+ Spike-1	40	77	58
Soil F+ Spike-2	40	75	57
Soil G+ Spike-1	30	46	71
Soil G+ Spike-2	29	43	71

[3] Spiking experiment was conducted in soil samples containing no measurable analytes

[4] Hydrazine was spiked as 125 ng/g. Monomethylhydrazine and 1,1-dimethylhydrazine was spiked as 625 ng/g.

Conclusions

- The use of tandem mass spectrometer provides greater confidence for quantification of hydrazines
- The sensitivity of the tandem mass spectrometer allows for reductions in sample preparation volumes – a “green” analytical technique
- Soil extraction procedure overcomes previous recovery difficulties on site specific matrices