ACROLEIN, ACRYLONITRILE, AND ACETONITRILE BY HS-GC

Demonstration of an HJ Method with an Agilent 7890 GC

Introduction

HJ Method 679-2013 describes the determination of acrolein, acrylonitrile, and acetonitrile from soil and sediments by headspace-gas chromatography. It has similarities to EPA methods 8015C (nonhalongenated organics), 8030A (acrolein and acrylonitrile by gas chromatography), and 8031 (acrylonitrile by gas chromatography). Found in the soil as a byproduct of synthetic processes or from previous pesticide use, these three analytes can quickly degrade and evaporate into the air.

Experimental

The HJ-679 method specifies a column with an internal diameter of 530 µm, which makes it well suited for an Agilent 7890 GC equipped with an FID and Agilent 7697 headspace sample. The HJ method was followed as closely as possible. Five headspace vials were made at each calibration level by filling each vial with ~2 g of quartz sand, adding 10 mL of a matrix modifier (180 g of NaCl in 500 mL of H_2O), and spiking varying amounts of a 2,000 ppm standard to achieve the required levels. Acrolein, acrylonitrile, and acetonitrile were spiked with 2 µg, 5 µg, 20 µg, 40 µg, and 80 µg.

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Instrument conditions

Parameter	Value
Agilent 7890 GC	
Inlet	150 °C, Split 5:1
Column	Agilent J&W DB-WAX UI, 30 m × 0.53 mm, 1.00 µm, 125-7032UI
Column flow	4.5 mL/min
Oven	40 °C (5 minutes) then 5 °C/min to 60 °C then 30 °C/min to 150 °C (5 minutes)
FID	250 °C
Agilent 7697 Headspace Sampler	
Oven	75 °C
Loop	105 °C
Transfer line	150 °C
Vial equilibration	30 minutes
Injection duration	0.1 minutes
Vial	20 mL
Shaking	On, level 1
Vial fill flow	50 mL/min
Vial fill pressure	8 psi
Vial pressure equilibration time	2 minutes
Loop fill ramp rate	20 psi/min
Final loop pressure	1.2 psi
Loop equilibration	0.2 minutes

Results and Discussion

The calibration curves determined for acrolein, acrylonitrile, and acetonitrile were found to be within method requirements (correlation coefficient of ≥0.995). The response factor was calculated over the calibration curve range, and an RSD was determined for each analyte. Acrolein was found to have the highest response factor RSD, at 10 %, likely due to its high volatility. Both acrylonitrile and acetonitrile yielded response factor RSDs of 5.3 %.

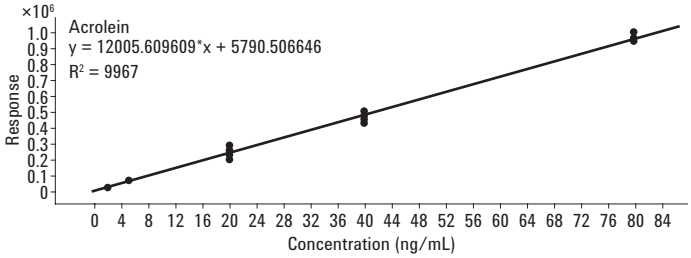

Figure 1. The calibration curve for acrolein from 2 µg to 80 µg gave a correlation coefficient of 0.997.

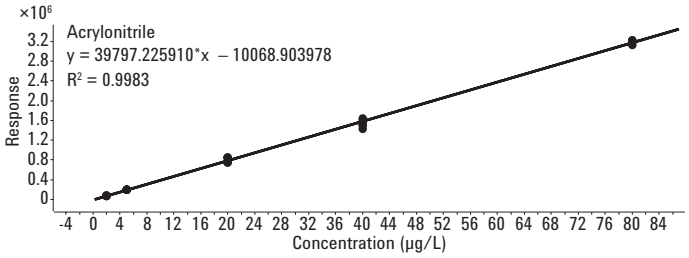

Figure 2. Calibration curve for acrylonitrile gave a correlation coefficient of 0.998 for five levels, ranging from 2 µg to 80 µg.

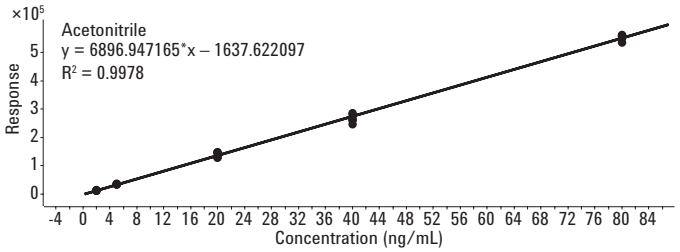

Figure 3. Acetonitrile gave a correlation coefficient of 0.998 for five calibration levels, ranging from 2 µg to 80 µg.

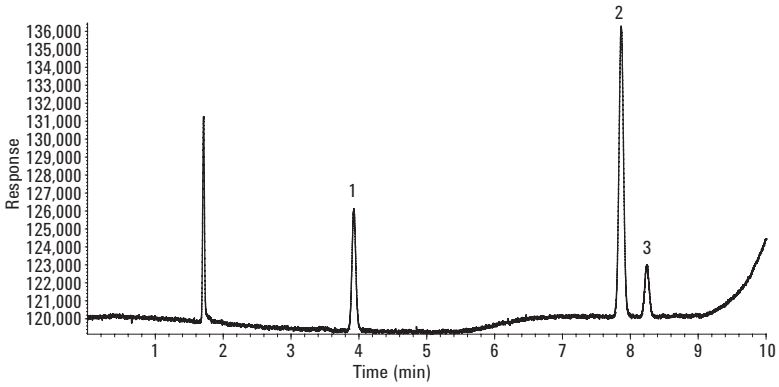

Figure 4. Acrolein (1), acrylonitrile (2), and acetonitrile (3) at 1 mg/kg eluted in under 10 minutes.

Conclusion

The Agilent 7890 GC configured with an Agilent 7697 headspace sampler achieves the performance specifications of HJ679-2013. Correlation coefficients were found to be 0.995 or better, while response factor RSDs are not specified in the method. Acrolein, acrylonitrile, and acetonitrile yielded RSDs of 10 % or better for five replicate vials. The 10-minute run time is typical of HJ679-2013, but could be shortened easily by implementing a smaller id column.

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Information, descriptions and specifications in this publication are subject to change without notice.

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