

Application Note

Analysis of 2-(Bromomethyl)Naphthalene by Atmospheric Pressure Photo Ionization

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

Over the past ten years electrospray (ESI) and atmospheric pressure chemical ionization (APCI) have become the standard ionization techniques for LC-MS(/MS) systems used for qualitative and quantitative analysis of small molecules. A new technique, atmospheric pressure photo ionization (APPI) is now available, which extends the range of compounds that can be analyzed by atmospheric pressure ionization.

The new APPI source is available on the Waters Micromass ZQ, Quattro micro API, and Quattro Premier benchtop quadrupole instruments as well as the LCT Premier and Q-Tof Premier instruments.

Introduction

Figure 1 below shows a schematic of the new APPI source developed in collaboration with Syagen Technology. The sample is introduced into the source by the IonSABRE probe in order to volatilize the sample and solvent. A

10.6 eV krypton lamp ionizes the sample and the ions that are subsequently formed are deflected towards the sample cone by a repeller electrode.

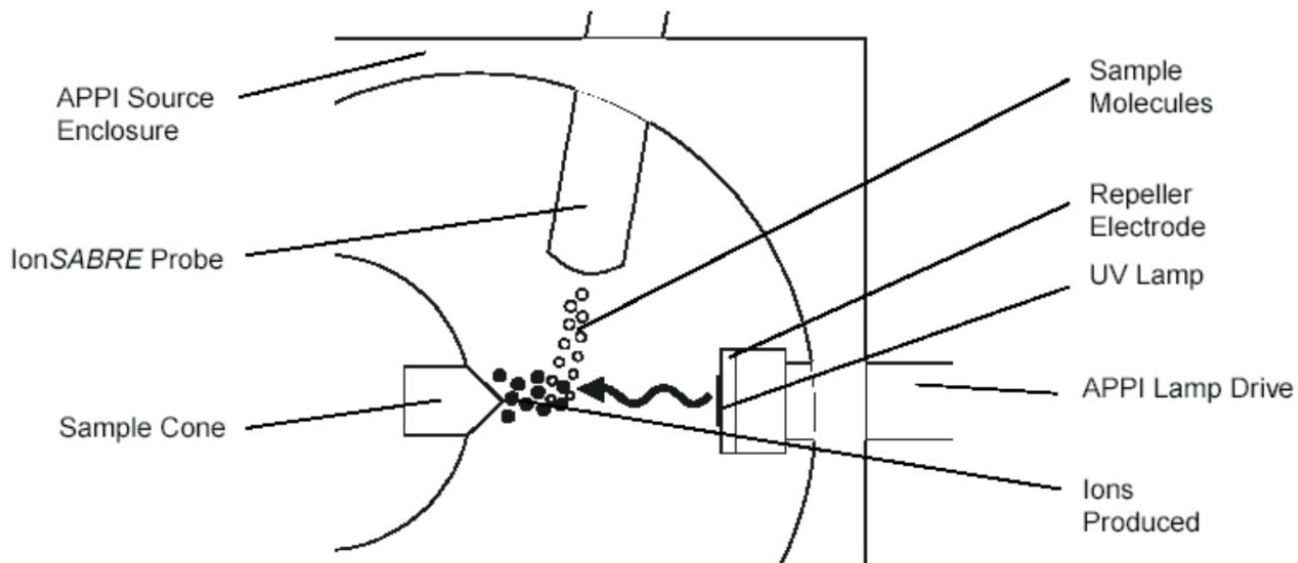


Figure 1. A schematic diagram of the new APPI source.

2-(Bromomethyl)naphthalene is an alkylating agent used in compound synthesis that is usually analyzed by GC-MS, as it does not ionize readily by ESI or APCI to give molecular ions. The new APPI source was used to analyze this compound by LC-MS/MS using the Quattro micro API.

Experimental

A standard solution of 2-(Bromomethyl)naphthalene (1 mg/mL) was prepared in methanol. A calibration series of 2-(Bromomethyl)naphthalene was made over the concentration range of 100–100000 pg/ μ L and analyzed by LC-MS/MS.

LC Conditions

LC system:	Waters Alliance HT 2795
Column:	Waters XTerra MS, C ₁₈ 3.5 µm, 2.1 x 50 mm
Flow rate:	0.3 mL/min
Injection volume:	10 µL
Gradient:	A: Water B:Acetonitrile

Gradient

Time (min)	%A	%B
0	95	5
1	5	95
4	5	95
4.1	95	5

MS Conditions

MS system:	Waters Micromass Quattro micro API
Ion mode:	APPI +ve
Cone voltage:	10 V
Repeller voltage:	0.2 V

Source temp.:	120 °C
APCI heater temp.:	500 °C
Collision energy:	13 eV
Detection mode:	MRM (220.1 > 141.0)
Dwell time:	0.5 seconds
Collision gas:	Argon (5×10^{-3} mbar)

Results and Discussion

A 1 ng/ μ L solution of 2-(Bromomethyl)naphthalene was used to tune the Quattro micro API. Positive ion analysis of 2-(Bromomethyl)naphthalene produced a molecular ion (M^{+}) at m/z 220, MRM analysis of the loss of bromine from this ion to m/z 141 was used for quantification (see Figure 2 for the product ion scan).

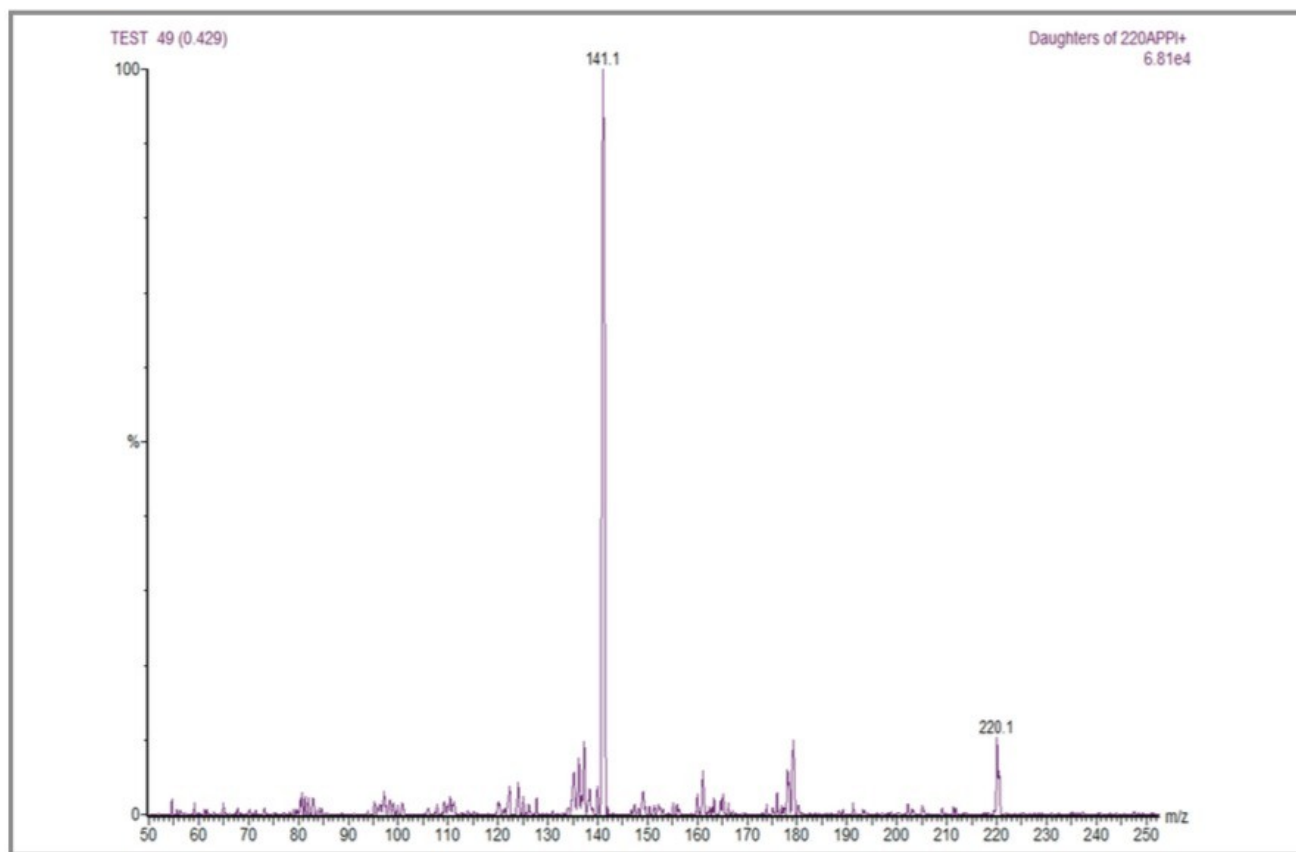


Figure 2. Product ion scan of the ion at m/z 220.

The plot of peak area against concentration showed good linearity over the range 1–1000 ng on column. The calibration line was plotted using a linear fit with $1/x$ weighting and gave a coefficient of determination of 0.9998, with all calibration points resulting in <8% deviation (see Figure 3).

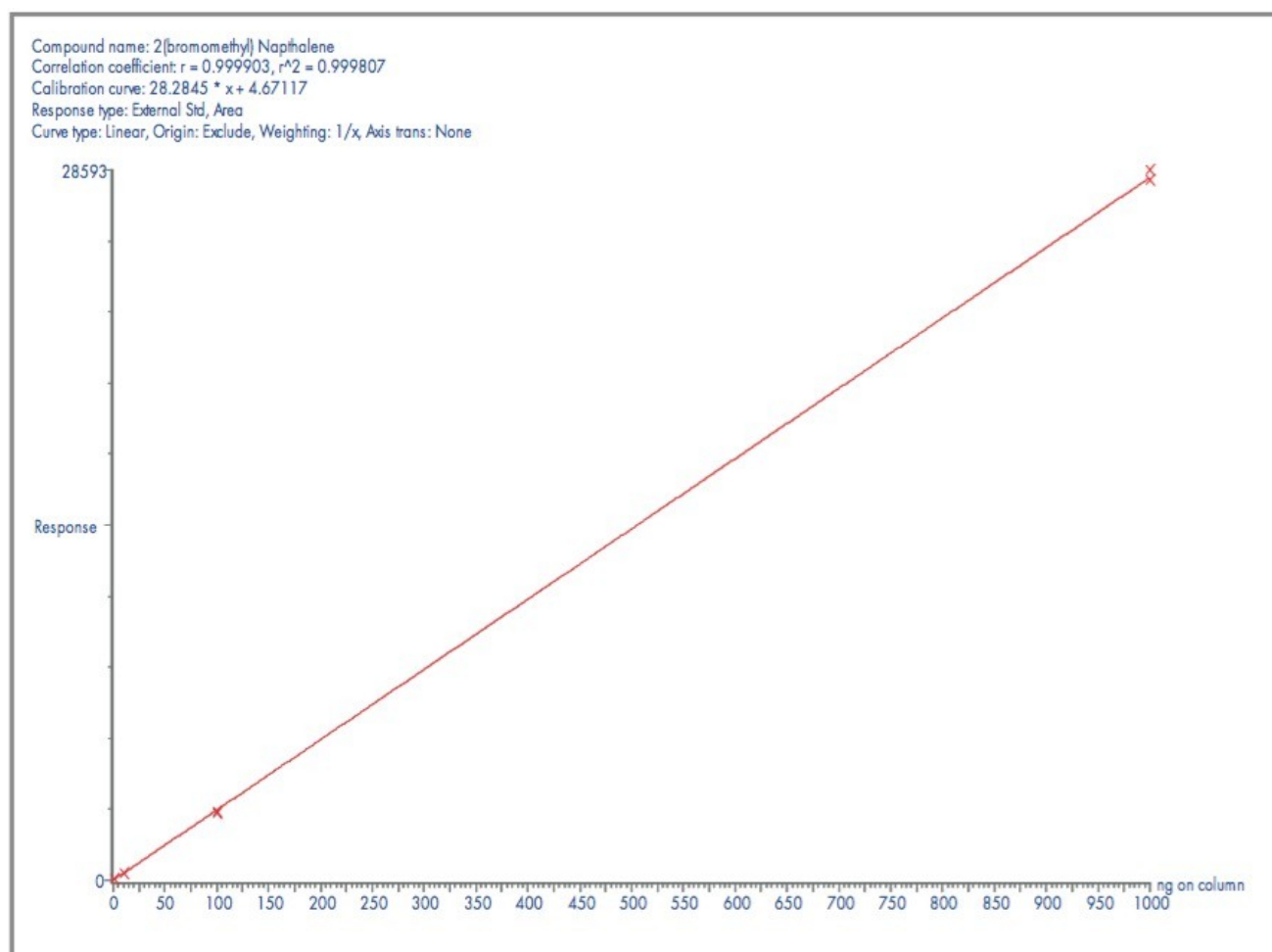


Figure 3. A calibration line for 2-(Bromomethyl)naphthalene over the range 1 to 1000 ng on column.

Conclusion

The Atmospheric Pressure Photo Ionization source provides an alternative ionization technique for LC-MS/MS analysis enabling a wider range of compound classes to be analyzed that were not previously amenable to LC-MS. Analysis of 2-(Bromomethyl)naphthalene demonstrated that photo ionization yielded intact molecular ions that can be quantified over three orders of linear dynamic range, *i.e.* 1–1000 ng on column.

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720000976, November 2004

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