Analysis of Residual Solvents in Hemp-Derived Products using Headspace Combined with Atmospheric Pressure GC/MS

Douglas Stevens, Lindsay Hatch, Otis Beverly, Waters Corporation, Milford MA USA

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Introduction

The active ingredients in hemp, cannabinoids and terpenes, are commonly extracted using solvents. Because many effective extraction solvents are known or suspected to have negative human health effects if consumed, it is necessary to monitor for residual extraction solvents in final hemp-derived products to comply with regulatory requirements, protect consumers and ensure product quality. In this work, the use of GC-APCI MS/MS for the analysis of residual solvents in hempseed oil was investigated.

Experimental

Xevo™ TQ-S micro
Transfer line: 260°C
Cone gas: 45 L/hr
Makeup gas: 350 mL/min
Source: APGC™ @ 120°C
Corona pin: 2.0 µA
Auxillary gas: 100 L/hr

CTC PAL3 RSI
Incubation: 15min @ 80°C
2.5mL HS syringe @ 105°C
Inj vol: 500µL

7890B GC SSL @ 225 °C, Split 10:1, 0.75 mm liner
Rxi-624Sil MS 30m x 0.25mm x 1.4µm, He 1.5mL/min
30°C/6min, to 85°C @ 15°C/min, 260°C @ 35°C/min, hold 1.5min

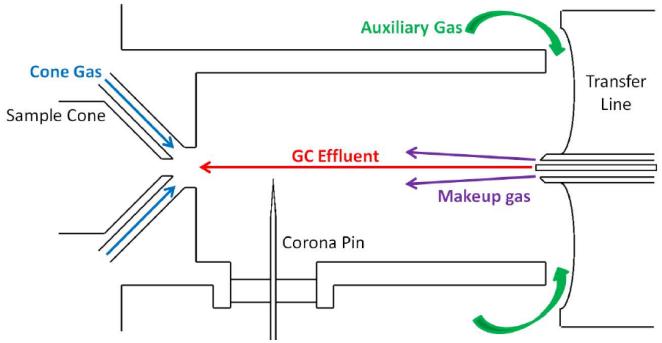

Figure 1. Schematic of the APGC Source

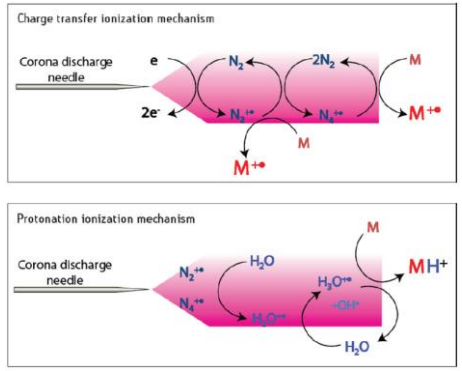

Figure 2. Ionization Mechanisms

Charge transfer, protonation, and mixed ionization modes were investigated. The main parameters that determine the ionization mechanism are the Cone Gas (CG) setting (Figure 1) and primary reagent in the source (N_2 or H_2O; Figure 2).

Results

Table 1 shows the optimized MRM transitions of 20 analytes including alkanes, aromatic hydrocarbons, alcohols, ketones, ethers, and chlorinated species. The analyte list is aligned with typical regulatory guidelines. Calibration standards were obtained from CPI International (p/n Z-G34-115300-03-5PAK & Z-G34-115301-03-5PAK).

The small molecular size of many analytes precluded traditional precursor > product MRM transitions, thus precursor > precursor MRM transitions were monitored.

	Ion. Mech.	Transitions (Collision Energy)
Propane	CT	39 > 39 (3), 53 > 53 (3)
Butane	CT	39 > 39 (3), 53 > 53 (3)
Methanol	PR	33 > 33 (3)
Ethylene Oxide	PR	45 > 45 (3)
Pentane	CT	53 > 53 (3), 71 > 71 (3)
Diethyl ether	PR	75 > 29 (8), 75> 47 (5)
Ethanol	PR	45 > 45 (3), 53 > 53 (3)
Acetone	PR	59 > 31 (6), 59 > 43 (10)
Isopropanol	PR	53 > 53 (3), 59 > 59 (3)
Acetonitrile	PR	42 > 42 (3)
Methylene Chloride	CT	83 > 83 (3), 85 > 85 (3)
Hexane	CT	53 > 53 (3), 57 > 57 (3)
Ethyl Acetate	PR	61 > 43 (7), 61 > 61 (3)
Chloroform	CT	83 > 83 (3), 85 > 85 (3)
Benzene	CT	78 > 52 (12), 78 > 63 (15)
Dichloroethane	CT	62 > 62 (3), 64 > 64 (3)
Heptane	CT	39 > 39 (3), 57 > 57 (3)
Trichloroethene	CT	130 > 95 (16), 132 > 97 (16)
Toluene	PR	93 > 51 (22), 93 > 77 (12)
Xylenes	PR	107 > 91 (12), 107 > 105 (10)

Table 1. MRM Transitions (PR = protonation; CT = charge transfer)

Excellent calibration linearity was observed (Figure 3). Analysis of a hempseed oil proficiency test sample demonstrated quantitative recovery within the acceptance limits of the test (Table 2).

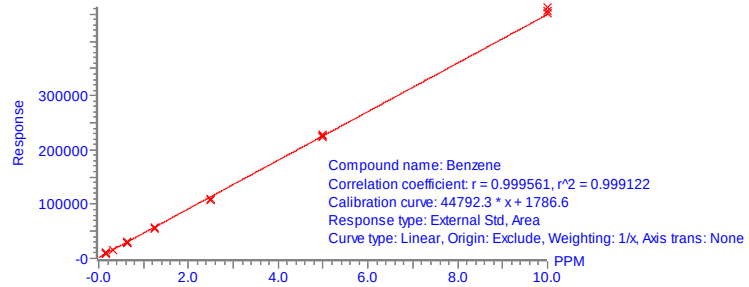

Figure 3. Quantitation example for benzene

	Measured Conc.	%RSD	Lower Acceptance Limit	Upper Acceptance Limit
Chloroform	15.2	1.1%	10.3	24.1
Dichloroethane	25.0	1.9%	16.9	39.5
Ethylene oxide	20.0	6.7%	13.3	31.1
Methylene chloride	12.2	4.8%	7.2	16.8
Trichloroethene	35.0	2.1%	22.9	53.5

Table 2. Quantitation results for hempseed oil PT sample (Absolute Standards p/n 38554); %RSD based on triplicate analyses

Conclusions

- Ionization of residual solvents from multiple compound classes was achieved in the APGC source using mixed-mode ionization combined with headspace sample introduction
- Analysis of a hempseed oil PT sample demonstrates fit-for-purpose quantitative performance for regulatory compliance monitoring

Application Note: Douglas Stevens, Analysis of Residual Solvent in Hemp Oil Using Headspace Sampling and Atmospheric Pressure GC-MS/MS, 2021, Doc. ID 720007150