SHIMADZU
Excellence in Science

Application News

GC-MS GCMS-QP™2020 NX

Semi-Quantitative Analysis of Anions in Water by HS-GC/MS

Y. Nakagawa

User Benefits

- Ions and contaminants other than targets are also identifiable with EI mass spectrum.
- Derivatization performed automatically by HS-20.

■ Introduction

Anions in water are quantitated for various reasons. It can be to check fluoride contamination in drinking water, to monitor an ion permeability or to confirm the effectiveness of reverse osmosis. An ion chromatograph is commonly employed to conduct such analyses.

In this experiment, however, GC-MS was used to monitor anion levels in water. Electron Ionization (EI) yields a mass spectrum to confirm the identity of a peak that would otherwise only be based on the retention time. Ions and contaminants other than the targets were also identified by NIST 20.

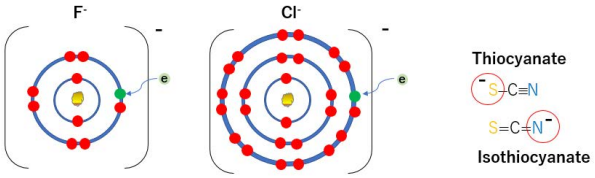

Fig. 1 Targets (i.e., Fluoride, Chloride and Thiocyanate) and an Isomer Isothiocyanate

■ Materials and Methods

To prepare a 10 mg/mL derivatization solution, 100 mg of pentafluorobenzyl p-toluenesulfonate, or PFB-Tosylate for short, was diluted to 10 mL with acetone. 4 mL of water and 1 mL of the derivatization solution were placed in a 20 mL HS vial and analyzed.

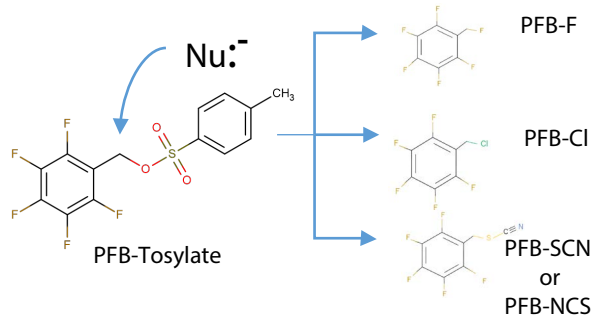

Fig. 2 Reactants and Products in the Derivatization Reaction

The derivatization reaction occurred in the headspace vial when heated in the oven for 5 mins. (Fig. 2) A longer heating time was avoided to ensure the recoveries of the target analytes.

Table 1 Instrument Configurations

GC-MS	: GCMS-QP2020 NX
Headspace	: HS-20
Analytical Column	: SH-I-5MS (30 m × 0.25 mm I.D., df=0.25 µm) *1

*1 P/N: 221-75940-30

Table 2 Analytical Conditions

HS	
Oven Temp.	: 80 °C
Sample Line Temp.	: 150 °C
Transfer Line Temp.	: 150 °C
Pressurizing Gas Pressure	: 100 kPa (Helium)
Equilibrating Time	: 5 mins
Multi Injection Count	: 1
GC	
Injection Mode	: Split
Split Ratio	: 5
Carrier Gas	: Helium
Control Mode	: Constant linear velocity (51.0 cm/s)
Column Oven Temp.	: 40 °C (2 min) → (20 °C /min) → 240 °C (2 mins) Total 14.00 mins
MS	
Ion Source Temp.	: 200 °C
Interface Temp.	: 240 °C
Measurement Mode	: Simultaneous Scan/SIM (FAAST)
Scan Mass Range (*m/z*)	: 35 - 400
SIM ions (*m/z*)	: Refer to Fig. 5
Event Time	: 0.30 second

Fig. 3 GCMS-QP™2020 NX Connected to HS-20 (Left) and HS Vials (Right)

■ Results

Identification was made by a retention time and a mass spectrum (Fig. 4). PFB has a *m/z* of 181 and each derivative has a mass of reactant anion added to 181.

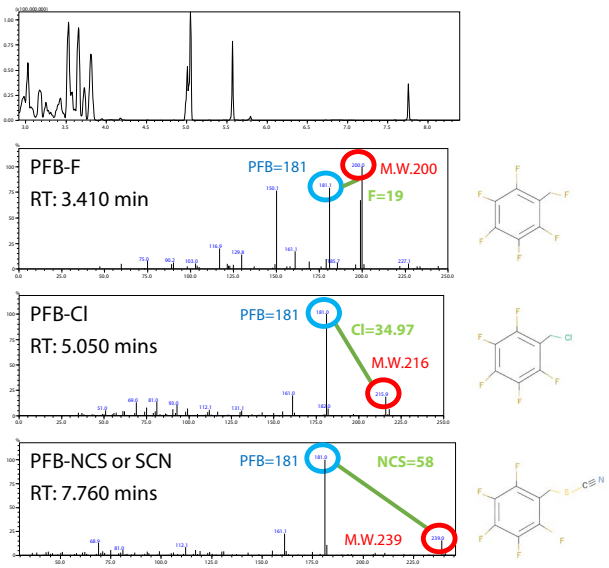

Fig. 4 Mass Spectra Obtained from a 50 µg/mL Mixed Solution

To check sensitivity of the system, 5 µg/mL solution was injected and obtained the chromatograms below.

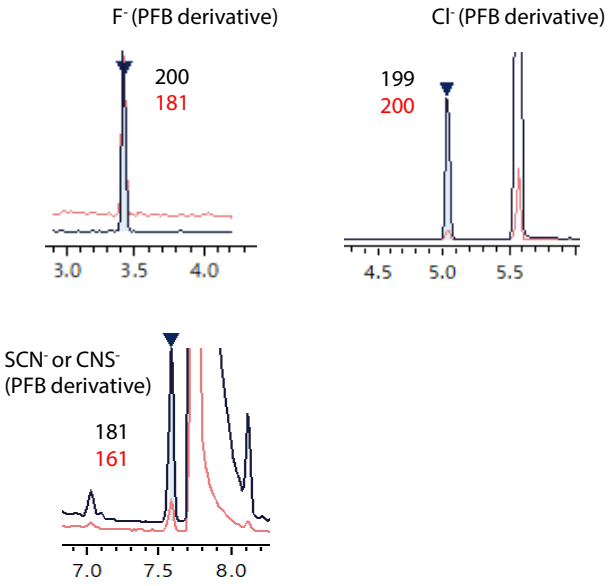

Fig. 5 Mass Chromatograms Obtained from a 5 µg/mL Solution

Since SIM and Scan data were acquired at the same time, compounds other than the targets were also identified.

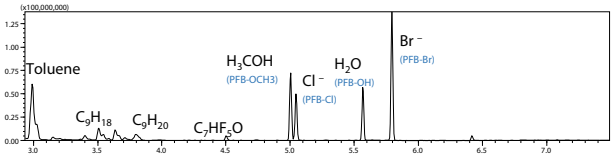

Fig. 6 TIC Chromatogram of a Water Sample

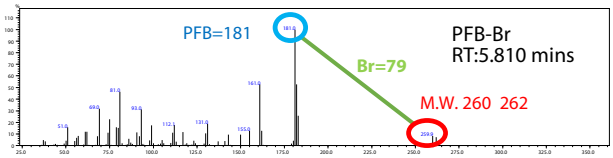

Fig. 7 PFB-Br Mass Spectrum

■ Conclusion

Anions in water were analyzed by HS-GC/MS. The derivatization was performed automatically by a Shimadzu HS-20.

GC/MS not only increased the confidence in peak identification of the targets, but allowed the identification of compounds other than the targets.

Thus, a Shimadzu HS-20 and GCMS-QP2020 NX are excellent tools in monitoring water contents including anions.

SHIMADZU

Shimadzu Corporation
www.shimadzu.com/an/

01-00038A-EN
First Edition: Apr. 2021
Revision A: Mar. 2023