SHIMADZU APPLICATION NEWS

LA146-E022

● GASCHROMATOGRAPHY MASS SPECTROMETRY

No.M207

Highly Sensitive GC/MS Analysis of EDTA (Ethylenediamine Tetraacetic Acid) in Water

The Drinking Water Test Methods in Japan were revised in July 2001 and the method for analyzing ethylenediamine tetraacetic acid (EDTA) was added. EDTA is widely used as an additive for agricultural and food products. It is also used in various industrial fields, including manufacturing various industrial products, plating, drug preparation and (water) softening processes. EDTA waste discharged into the environment does not easily decomposed and exists in the form of a metal chelate. Therefore, trace amounts of EDTA can be contained in environmental water.

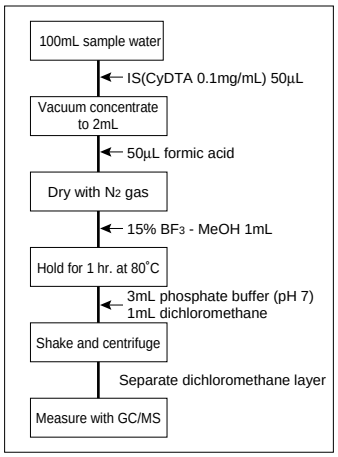


Fig.1 Flow chart of Analysis

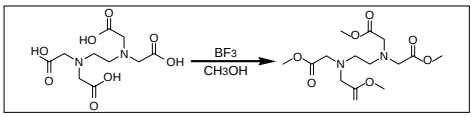


Fig.2 Methyl Ester Derivatization

Table 1 Analytical conditions

Model	: GCMS-QP2010
-GC-	
Column	: Solgel1 30m×0.25mmI.D. df=0.25µm
Col. Temp.	: 60°C (2 min)-15°C / min-270°C (4 min)
Col. Pressure	: 100kPa
Inj. Temp.	: 250°C
Injection	: Splitless
Sampling time	: 1.0min (250kPa)
-MS-	
IF Temp.	: 250°C
Ion Souce Temp.	: 170°C
Ionization	: EI
-Scan Mode-	
Scan Range	: m/z 35-450
Scan Interval	: 0.5sec
-SIM Mode-	
Monitor Ions	: Group1 m/z 174.10, 289.10, 348.10 Group2 m/z 343.15, 402.15
Scan Interval	: 0.2sec

Since animal testing indicated EDTA's slight toxicity to humans, U.N. Food and Agriculture Organization (FAO) and World Health Organization (WHO) have set a guideline value of 0.6mg/L, which is one percent of the acceptable daily intake (ADI) of EDTA at 1.9mg/kg bw per day. Determination limit for EDTA in water stipulated by the Japanese Drinking Water Test Method is 0.5µg/L. In actuality, the sample is concentrated by 100 times before being analyzed. Therefore, the limt of (quantitation) determination required for actual analyses is 0.05mg/L. GCMS-QP2010 has sufficient sensitivity for this requirement. Fig. 1 shows the flowchart of the analytical process.

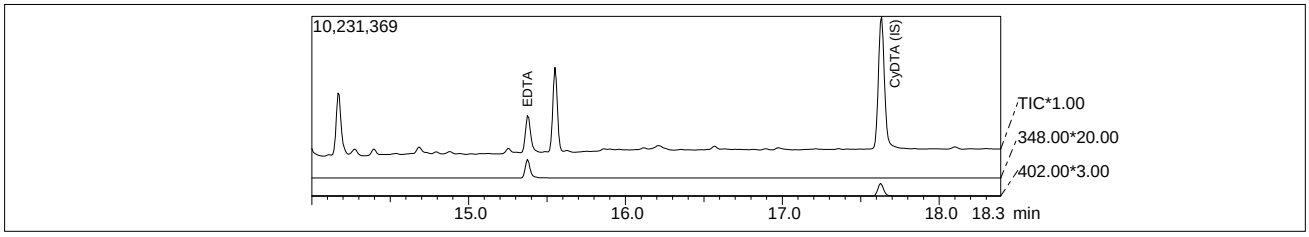


Fig.3 EDTA 0.5 mg/L CyDTA(I.S.) 5.0 mg/L with Scan mode TIC

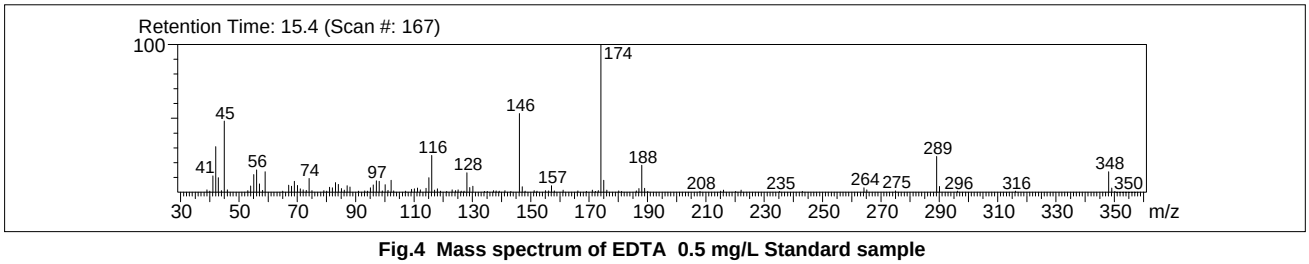


Fig.4 Mass spectrum of EDTA 0.5 mg/L Standard sample

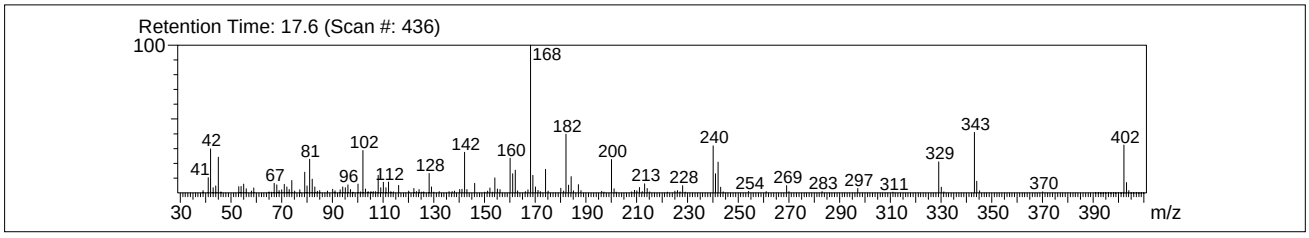
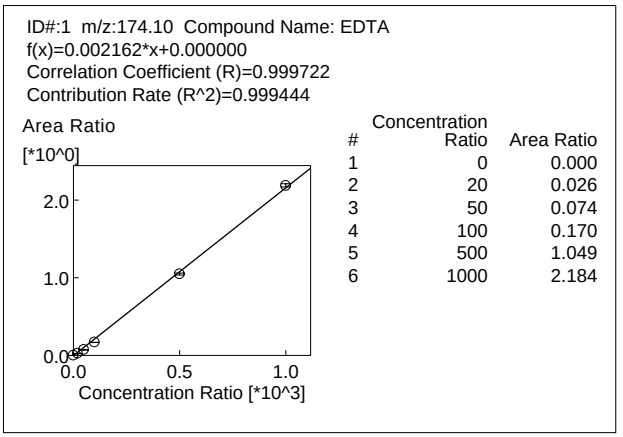


Fig.5 Mass spectrum of CyDTA 5mg/L Internal standard sample

50μL of 0.1mg/mL cyclohexanediamine tetraacetic acid (CyDTA) was added to the 100mL sample water as an internal standard, and concentrated by rotating evaporator to 2mL. The concentrated sample was transferred to a 10mL test tube with ground stopper, spiked with 50μL formic acid, and blown with nitrogen gas while being heated until evaporated to dryness. Then 1mL of boron trifluoride methanol solution (14 - 15% solution is commercially available) was added, the test tube was tightly stoppered and the sample was derivatized for one hour over a 80˚C bath (see Fig. 2 for derivatization reaction). The derivatized sample was left cool. 3mL buffer solution (KH_2PO_4/NaOH at pH 7) and 1mL dichloromethane were added, mixed thoroughly, and separated by centrifuge, to obtain the dichloromethane layer for analysis.
SIM measurement with EI was employed for GC/MS analysis. Table 1 shows the analytical conditions.
Fig. 3 shows the chromatogram for the 0.5mg/L standard sample (5mg/L internal standard sample), measured using the scan mode. Fig. 4 and 5 show the mass spectra. EDTA monitoring ions at 174.1, 289.1 and 348.1m/z, and internal standard at 343.1 and 402.15m/z were used.

The calibration curve was created by measuring



#	Concentration Ratio	Area Ratio
1	0	0.000
2	20	0.026
3	50	0.074
4	100	0.170
5	500	1.049
6	1000	2.184

Fig.6 Calibration Curve

standard samples from 0.02mg/L to 1mg/L (see Fig. 6). A good linearity was obtained. Fig. 7 shows the SIM chromatogram for a standard sample of the lowest concentration at 0.02mg/L. Fig. 8 shows the chromatograms where the standard sample was added to an actual sample, extracted and then analyzed. Fig. 9 shows the quantitative calculation results.

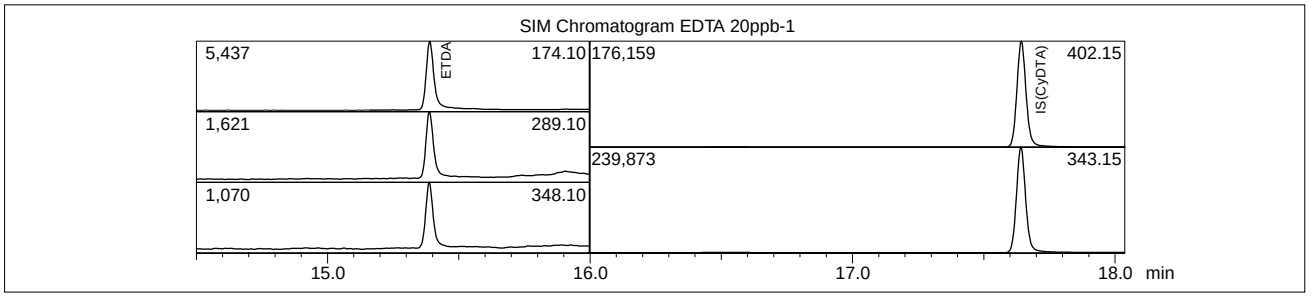


Fig.7 SIM Chromatogram of Standard Sample

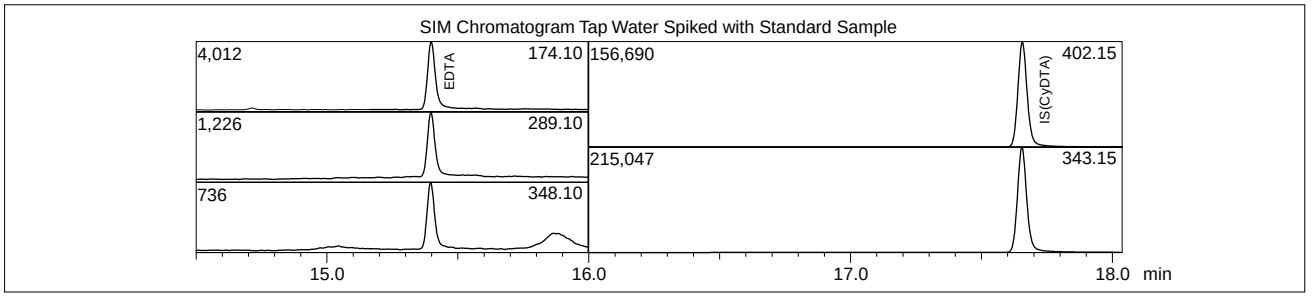


Fig.8 SIM Chromatogram of Sample spiked with 0.02 mg/L Standard

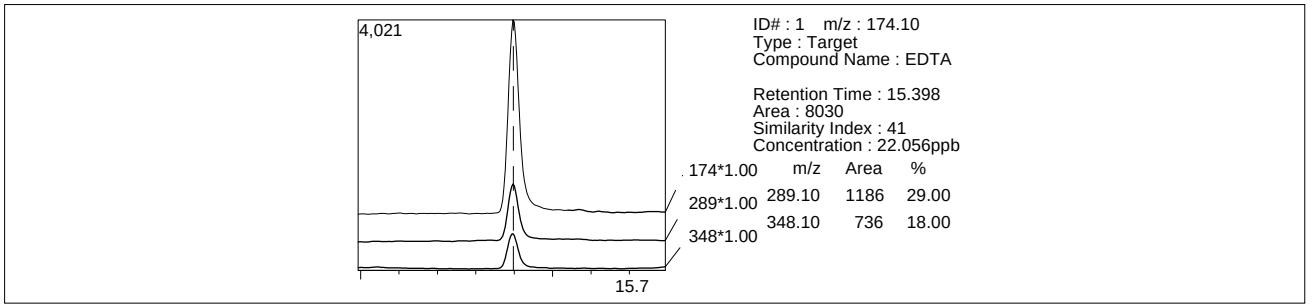


Fig.9 Quantitation of Sample spiked with Standard

SHIMADZU

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