

Qualitative analysis by comprehensive 2D GC / TOFMS [3] - Analysis of sulfur-contained substances in diesel oil -

Comprehensive two-dimensional gas chromatography (GC x GC) is a kind of a continuous hard-cut GC system. Two different types of columns are connected via a modulator in the same GC oven. The GC x GC technique has a very high separating power compared to single GC. GC x GC systems requires a fast acquiring detection system, because the peak width in the GC chromatogram is very narrow. This requirement of very fast data acquisition is fully met in the AccuTOF-GC. Since the maximum spectrum recording interval on JEOL AccuTOF-GC is 25Hz (0.04sec), the system can successfully be used as detection system in combination with a GC x GC system.

This application note shows the results of the qualitative analysis for sulfur-containing substances in diesel oil by GC x GC-TOFMS.

<Sample and measurement conditions>

Sample diesel oil

Measurement conditions

For GC×GC

System:	Agilent 6890GC ZOEX KT2004
Column:	1 st : HP-1ms (30m × 0.25mm I.D., 0.25μm) 2 nd : DB-17 (2m × 0.1mm I.D., 0.1μm)
Oven temp.:	50C(1min) → 5C/min → 280C(6min)
Injection temp.:	280C
Injection volume:	0.5μl [Split mode (1:200)]
Carrier gas:	He (Const. pressure: 680kPa)
Trapping interval:	6 sec

For MS

MS:	JMS-T100GC "AccuTOF GC"
Ionization method:	EI+ (70eV, 300μA)
Acquired m/z range:	m/z 35—500
Spectrum recording interval:	0.04 sec (25Hz)

<Results and discussion>

GC x GC mass chromatogram of m/z 184, 198 and 212 shown in Fig 1, which are the molecular ions of dibenzothiophenes are created by ZOEX GC Image software. In addition, mass spectra for compound A, B and C are shown in Fig.2.

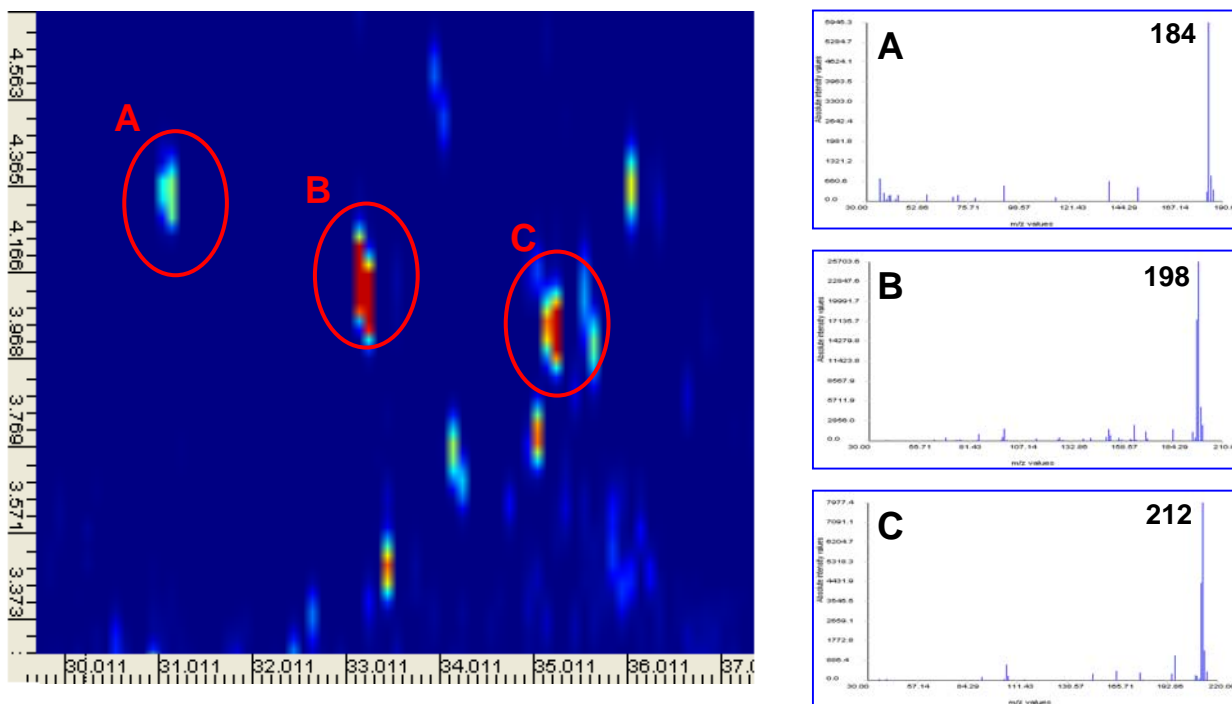


Fig.1 GC x GC mass chromatogram of m/z 184, 198 and 212 (Left) and mass spectra for compound A, B and C (Right)

Compound A, B and C are confirmed as sulfur-containing substances due to the isotopic pattern. The elemental composition of those ions is determined by accurate mass by using 1 point internal calibration using anthracene (M^+ : m/z178.0783) present in the sample. Each estimated elemental composition is shown in Table.1.

Table 1 The result of elemental composition determination for M^+ and ion of m/z152

Substance	Cal. mass	Obs. mass	Diff. (mDa)	Formula
Dibenzothiophene	184.0347	184.0343	-0.4	$C_{12}H_8S$
	152.0626	152.0619	-0.7	$C_{12}H_8$
3-Methyldibenzothiophene	198.0503	198.0495	-0.8	$C_{13}H_{10}S$
	152.0626	152.0620	-0.6	$C_{12}H_8$
2,8-Dimethyldibenzothiophene	212.0660	212.0654	-0.6	$C_{14}H_{12}S$
	152.0626	152.0622	-0.4	$C_{12}H_8$

The proposed formula is the same as the formula of dibenzothiophens and the mass difference of the molecular ion and the fragment ions from the exact mass, is within 1 mDa.

The AccuTOF GC has the capability of high speed spectrum recording to combine with a GC x GC system. In this way, GC x GC-AccuTOF GC can perform very reliable qualitative analysis.

<Acknowledge>

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Zoex's GC x GC system is provided and supported through Zoex's sales and support network and may not be available in your territory. Contact your local JEOL representative for detail.