SHIMADZU

LC-MS Application Data Sheet No. 046

Analysis of Bisphenol A Diglycidyl Ether Using LC-MS

Bisphenol A diglycidyl ether (BADGE) (Fig.1), formed by a condensation reaction (glycidyl reaction) between bisphenol A and epichlorohydrin, is widely used as a raw material of epoxy resin and stabilizer for vinyl chloride. Bisphenol A is a suspected endocrine disrupter. In Europe and the US, elution of bisphenol A from coating material made of epoxy resin is causing greater concern than its elution from polycarbonate material. This data sheet introduces an example of BADGE analysis using LC-MS. Injection of a large amount of sample (500 µL) allows the detection of trace BADGE in the sample.

Fig.2 shows a mass spectrum of standard BADGE. In the atmospheric pressure chemical ionization (APCI) method in the positive mode, BADGE tends strongly to form ammonium adduct ions $[M+NH_4]^+$ at m/z 358. Their fragment ions are also observed at m/z 191. Fig.3 shows SIM chromatograms for 2ppb BADGE. Reliable identification can be conducted by analyzing the ions at m/z 358 and 191. Fig.4 shows a SIM chromatogram of 20ppt BADGE and the calibration curve (n=5) for 20ppt to 100ppb. Good results were obtained with a coefficient of correlation at 0.999 or higher and repeatability at various concentrations at 1.0 to 6.1% RSD (Table 1).

Fig.5 shows the results of an LC-MS analysis where BADGE was added to a leaching solution of a coating resin to adjust the concentration to 10 ppb, and the solution was analyzed after filtration. BADGE was detected without the affection of interfering substances, thereby enabling accurate quantitation.

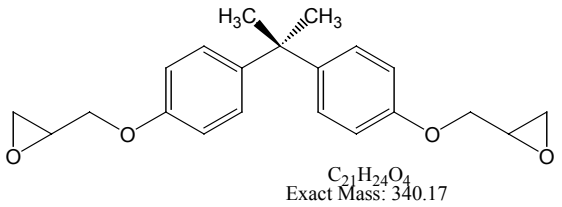

Fig. 1 Chemical structure of bisphenol A diglycidyl ether (BADGE)

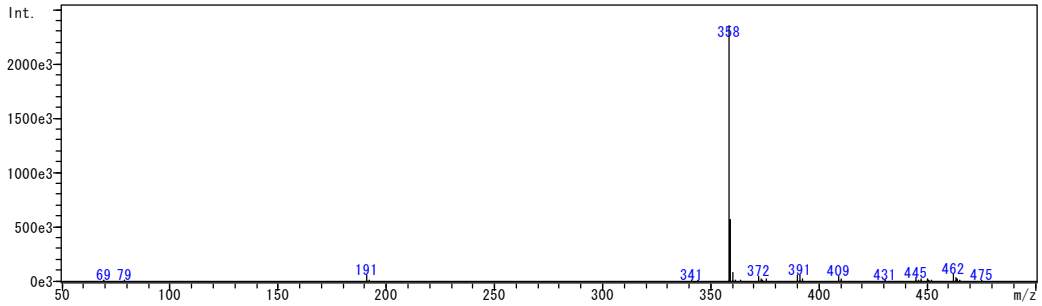

Fig. 2 APCI MS spectrum of BADGE

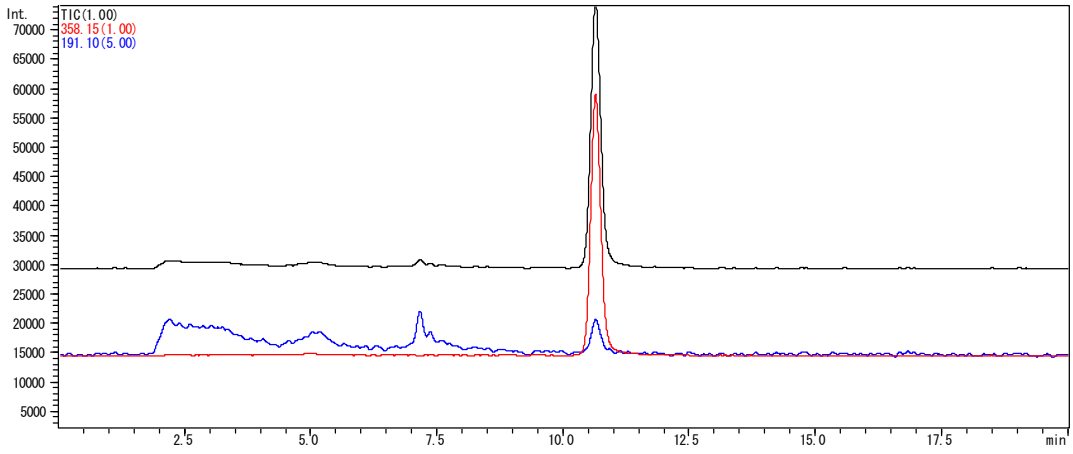

Fig. 3 SIM chromatograms of BADGE (2ppb)

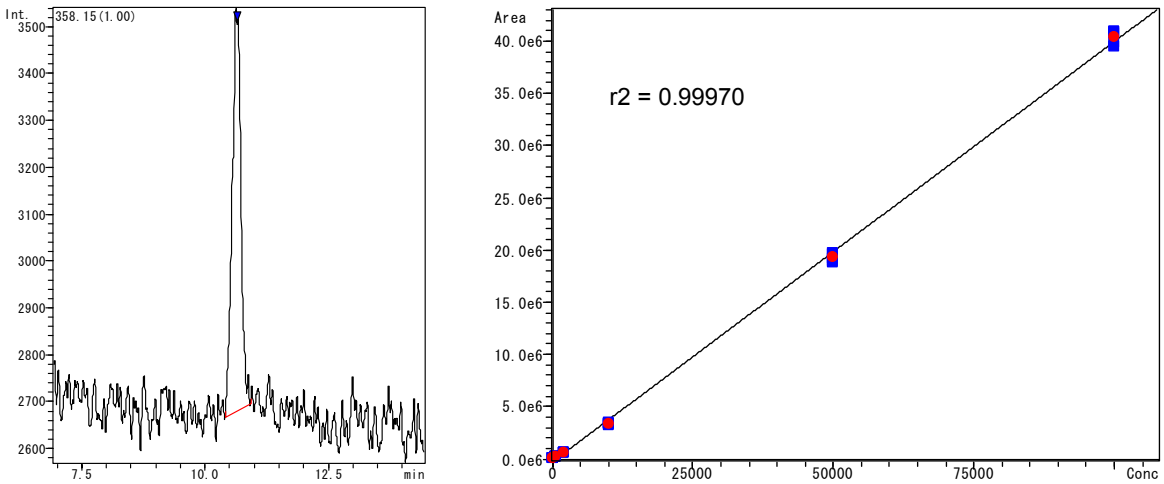

Fig. 4 SIM chromatogram (20ppt) and calibration curve of BADGE (20ppt - 100ppb)

Table 1 Reproducibility of peak area on BADGE

	1	2	3	4	5	average	%RSD
20ppt	9954	11055	10086	9758	9410	10052.6	6.12
100ppt	36331	33754	32960	34134	32271	33890.0	4.55
500ppt	144706	151700	149755	151132	146310	148720.6	2.06
2ppb	604082	592527	597561	600347	607951	600503.0	0.98
10ppb	3262431	3353442	3410712	3382410	3347494	3351298.0	1.66
50ppb	18772383	19598026	19218580	18952694	19741950	19256726.0	2.14
100ppb	39422534	39880234	40755750	40912124	40914658	40377060.0	1.70

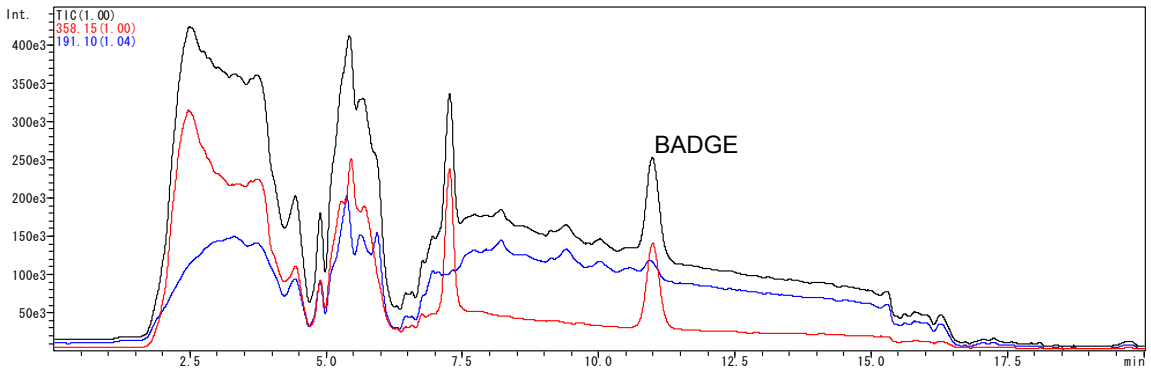

Fig. 5 SIM chromatograms of BADGE (10ppb) in matrix sample

Table 2 Analytical conditions for LC-MS

Column	: Shimadzu Shim-pack VP-ODS (2.0mm I.D.x 150mmL)		
Mobile phase	: 70% methanol - 10mM ammonium acetate buffer (pH 4.0)		
Flow rate	: 0.2 mL/min		
Injection volume	: 500µL	**Column temperature**	: 40°C
Probe voltage	: +2.5 kV (APCI-Positive mode)		
CDL temperature	: 200°C	**Block heater temperature**	: 200°C
Nebulizing gas flow	: 2.5 L/min		
CDL voltage	: -25V		
Q-array DC voltage	: +30V	**Q-array RF voltage**	: 150V
Scan range	: m/z 50-500 (1.0sec/scan)		
SIM	: m/z 358.15, 191.10 (0.2sec/ch)		

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