

Analysis of DNPH-aldehydes using LC-MS

Aldehydes are toxic chemical compounds in the environment, resulting from emission of gases and ozone treatment of water. Long-term exposure to aldehydes can cause a number of health problems. Some of these compounds are carcinogenic. The capture of highly volatile aldehydes from the atmosphere for analysis is extremely difficult. Generally, aldehydes are derivatized using 2,4-dinitrophenyl-hydrazine (DNPH) for analysis. However, as DNPH also reacts with other carbonyl compounds, numerous compounds are detected in samples, which

makes the identification and quantitation of components difficult. This data sheet introduces the batch analysis of DNPH-aldehydes using LC analysis with highly selective mass spectrometric detection.

Fig.1 shows the mass chromatogram of the aldehyde mixture obtained in the scan mode. Using negative atmospheric pressure chemical ionization (APCI), each DNPH-aldehyde yields deprotonated molecules, allowing for excellent detectability and reliable identification of components.

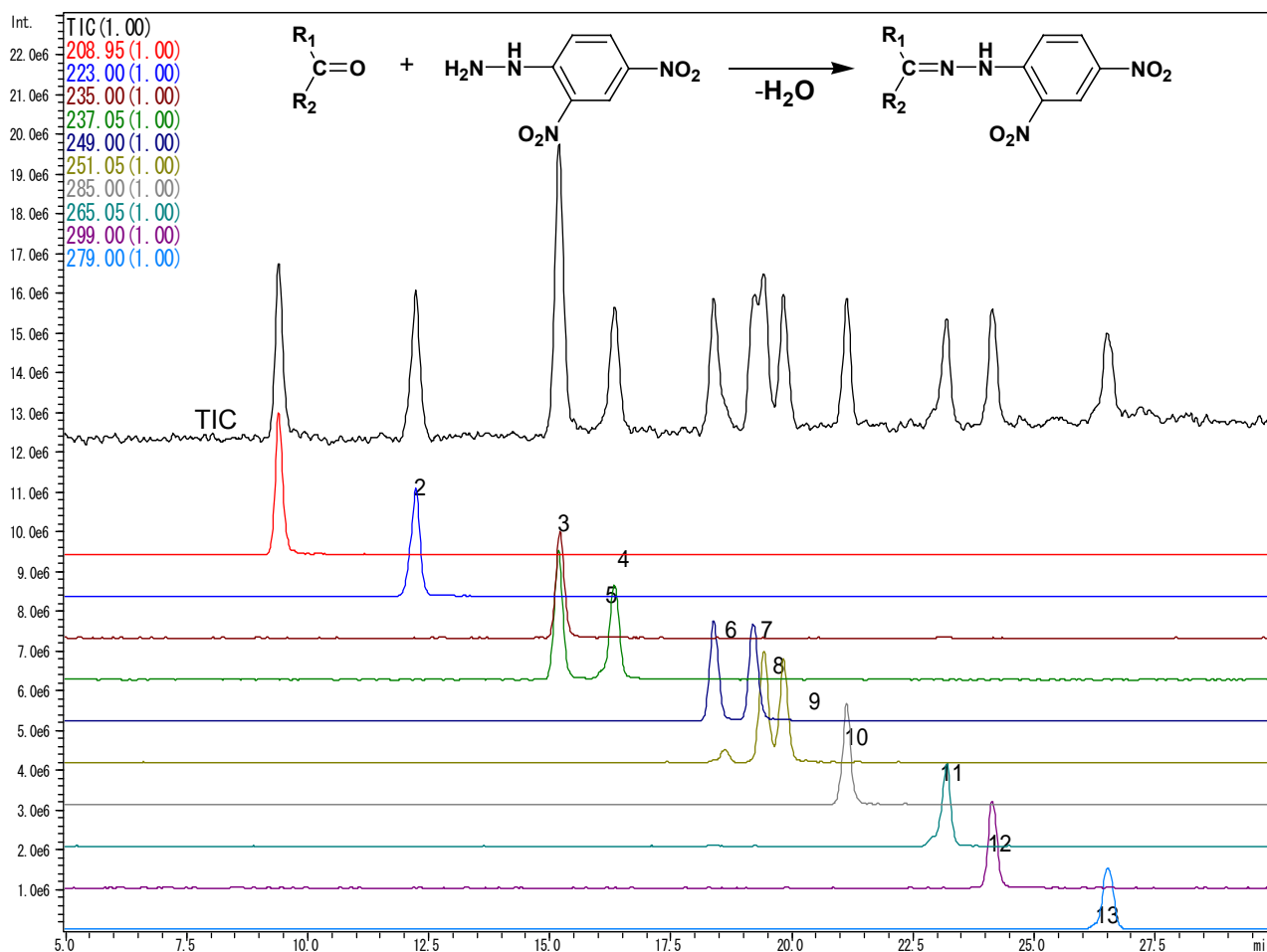


Fig. 1 Mass chromatograms of DNPH-aldehydes

1. DNPH-Formaldehyde	MW 210 ($R_1=R_2= \text{H}$)	8. DNPH-2-Butanone	MW 252 ($R_1= \text{C}_2\text{H}_5, R_2= \text{CH}_3$)
2. DNPH-Acetaldehyde	MW 224 ($R_1= \text{CH}_3, R_2= \text{H}$)	9. DNPH-n-Butyraldehyde	MW 252 ($R_1= \text{C}_3\text{H}_7, R_2= \text{H}$)
3. DNPH-Acrolein	MW 236 ($R_1= \text{CH}_2=\text{CH}, R_2= \text{H}$)	10. DNPH-Benzaldehyde	MW 286 ($R_1= \text{C}_6\text{H}_5, R_2= \text{H}$)
4. DNPH-Acetone	MW 238 ($R_1=R_2= \text{CH}_3$)	11. DNPH-n-Valeraldehyde	MW 266 ($R_1= \text{C}_4\text{H}_9, R_2= \text{H}$)
5. DNPH-Propionaldehyde	MW 238 ($R_1= \text{C}_2\text{H}_5, R_2= \text{H}$)	12. DNPH-m-Tolualdehyde	MW 300 ($R_1= \text{C}_6\text{H}_4\text{CH}_3, R_2= \text{H}$)
6. DNPH-Crotonaldehyde	MW 250 ($R_1= \text{CH}_3\text{CH}=\text{CH}, R_2= \text{H}$)	13. DNPH-Hexanaldehyde	MW 280 ($R_1= \text{C}_5\text{H}_{11}, R_2= \text{H}$)
7. DNPH-Methacrolein	MW 250 ($R_1= \text{CH}_2=\text{CCH}_3, R_2= \text{H}$)		

Figures 2 and 3 show SIM chromatograms for 1 ppb DNPH-formaldehyde and DNPH-acetaldehyde and calibration curves of DNPH-formaldehyde and

acetaldehyde of 1 to 500 ppb. Good area count repeatabilities were obtained at concentrations over 5ppb, with CV values from 1 to 6%.

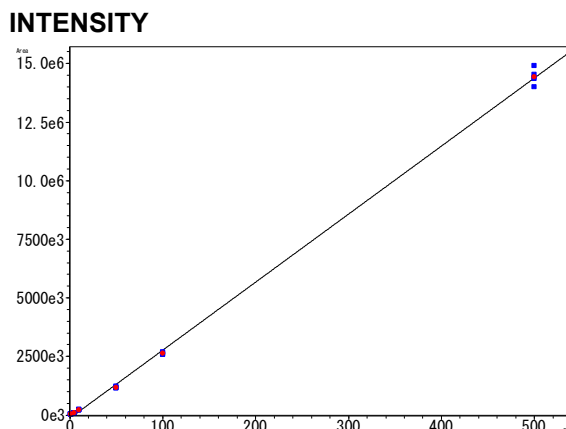
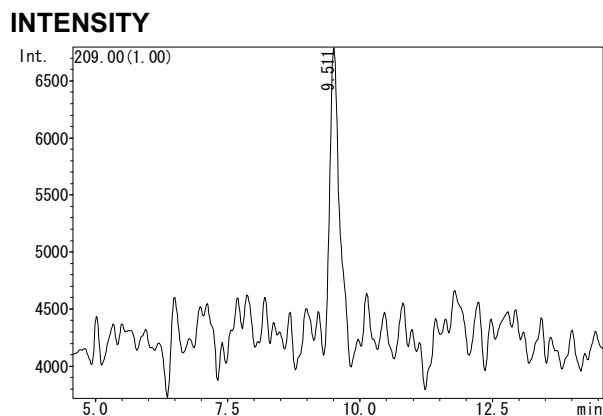


Fig. 2 SIM chromatogram(5pg) and calibration curve of DNPH-Formaldehyde
($Y = 29041X - 128064$, $r^2 = 0.99978$)

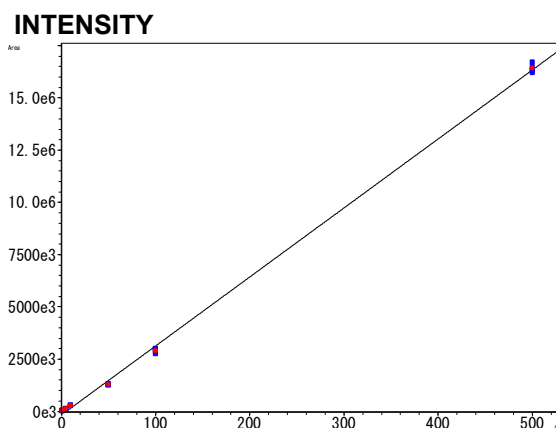
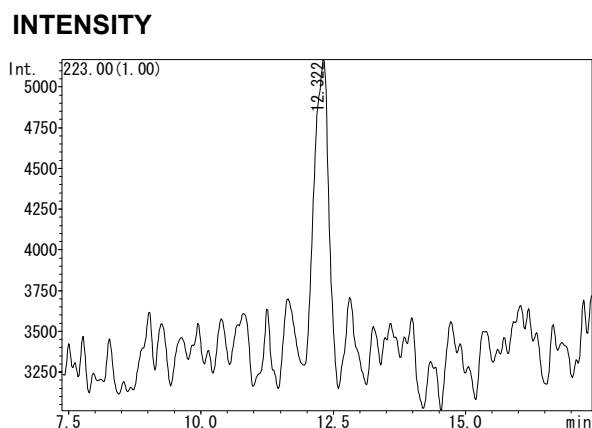


Fig. 3 SIM chromatogram(5pg) and calibration curve of DNPH-Acetaldehyde
($Y = 33007X - 156663$, $r^2 = 0.99961$)

Table 1 Reproducibility of DNPH-Formaldehyde

	1	2	3	4	5	Mean	SD	CV(%)
1ppb	26747	35061	34631	30182	27237	30771.60	3947.542	12.83
5ppb	83517	85552	78105	74654	77693	79904.20	4490.0193	5.62
10ppb	215606	231974	220330	216490	225262	221932.40	6784.7385	3.06
50ppb	1181219	1158296	1230232	1170406	1158225	1179675.60	29839.038	2.53
100ppb	2635500	2572613	2594822	2654021	2672834	2625958.00	41506.266	1.58
500ppb	13988871	14343665	14526266	14401878	14915646	14435265.20	334596.75	2.32

Table 2 Analytical conditions for LC-MS

Column	: Shimadzu VP-ODS (2.0 mmI.D. x 150 mm)	Column temperature	: 40 °C
Mobile phase A	: water	Block Heater temperature	: 200 °C
Mobile phase B	: acetonitrile	Q-array RF	: scan mode
Gradient program	: 40% B—70%B (20-30min)		
Flow rate	: 0.2 mL/min		
Injection volume	: 5 uL		
Probe voltage	: -3.0 kV (APCI-Negative mode)		
Probe temperature	: 400 °C		
CDL temperature	: 230 °C		
Nebulizing gas flow	: 2.5 L/min		
CDL voltage	: +20 V		
Q-array DC voltage	: scan mode		
Scan range	: m/z 50 – 500 (1.0 sec/scan)		

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