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GC-MS Application Data Sheet No. 3 (Environment)

Sensitive Analysis using High-pressure Injection Mode

The split/splitless sample injection method is generally used for GC analysis. As the injection volume increases with these injection methods, the vaporized sample volume exceeds the glass insert volume, such that the signal intensity becomes unrelated to the injection volume (Fig. 1). Consequently, the injection volume is restricted to 1 to 2 μL for splitless injection.

The Programmed Temperature Vaporizer (PTV) method is generally used for injection of large sample volumes. As the sample is injected while the sample injection chamber temperature is maintained below the solvent boiling point, the injection volume can be increased because the vaporized sample volume is small. This method permits injection of sample volumes up to 8 μL. However, the analysis system must be modified because a special PTV sample injection chamber is required.

The high-pressure injection method involves increasing the carrier-gas pressure setting when the sample is injected. This compresses the vaporized sample and permits larger injection volumes. The normal split/splitless sample injection chamber is used, such that the injection volume can be increased simply by changing the analysis condition settings.

Fig. 2 shows the relationship between injection volume and signal intensity. The high-pressure injection mode permits injection of up to 4 μL sample. Injecting 4 μL sample achieves four times the sensitivity achieved with 1 μL. This allows samples conventionally measured in the SIM mode to be measured in SCAN mode. Additionally, SIM mode analysis achieves higher sensitivity than previously possible. The high-pressure injection mode can be used by simply entering the pressure and hold times in GCMSSolution. It permits easy, high-sensitivity measurements.

This Application Data Sheet introduces high-sensitivity measurements on agricultural chemicals obtained using the high-sensitivity injection mode. Fig. 3 indicates that high-pressure injection allows measurement of low-concentration samples in SCAN mode. Additionally, good analysis repeatability (CV) values were achieved below 10% (Table 2). These results indicate that the method is applicable to quantitative analysis.

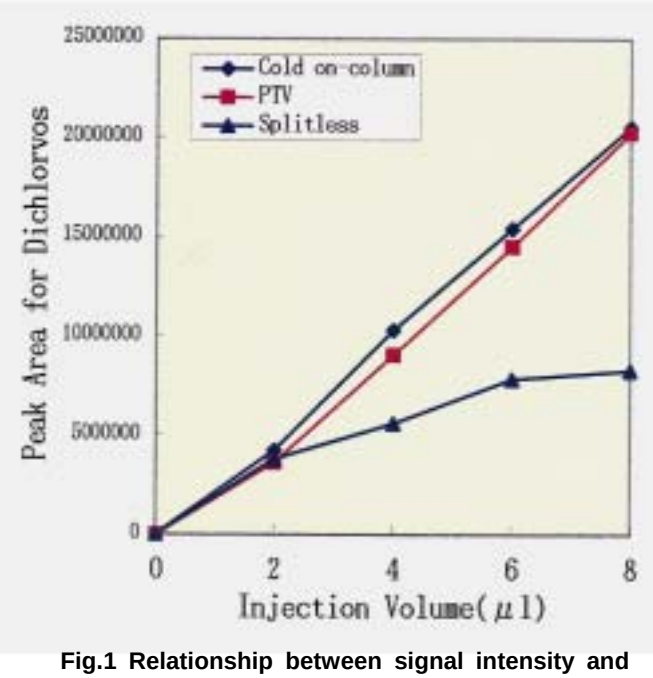

Fig.1 Relationship between signal intensity and injection volume for various injection methods

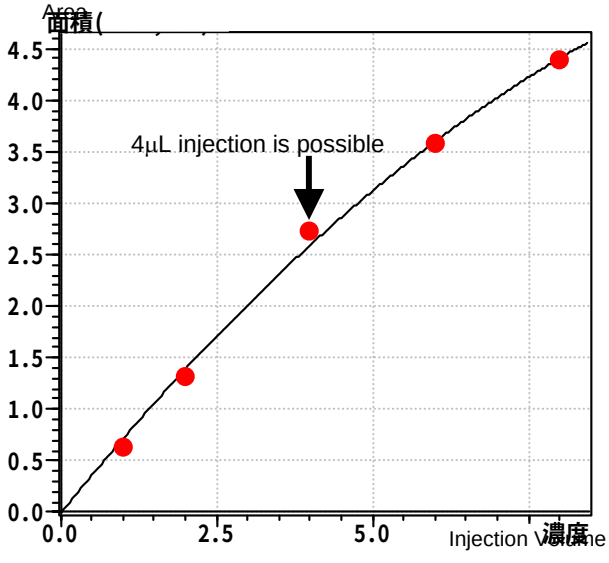

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Fig. 2 Relationship between signal intensity and injection volume in high-pressure injection mode

Table 1 Analytical conditions for GC-MS

Equipment	:**GCMS-QP2010**
Column	: Rtx-5MS (30mx0.25mm i.d. df=0.25μm)
Column Time program	:80°C (1min) -20**℃**/min-140**℃**-10**℃**/min -280**℃**(3min)
Carrier Gas	: Constant Linear Velocity 80.2cm/sec (He 47.7kPa)
Injection Temp.	: 250**℃**
Injection Method	:Splitless (Sampling Time: 1min)
Injection Volume	:4μL
High Press. INJ.	:250kPa (1.5min)
Interface Temp.	: 250**℃**
Ionsource Temp.	: 200**℃**

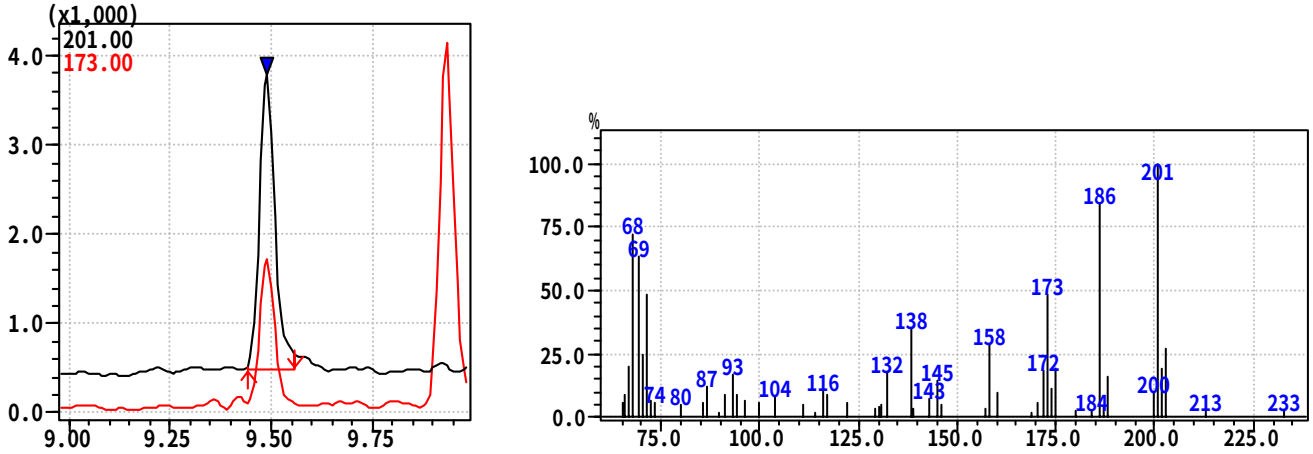

Fig. 3 Mass chromatogram and mass spectrum of simazine (20 μg/L)

Table 2 Reproducibility of High Press. Injection (50μg/L n=5 SCAN)

No	Compound	CV(%)	No	Compound	CV(%)
1	Methamidophos	**6.98**	16	Fenitrothion	**6.03**
2	Acephate	**4.52**	17	Chlorpyrifos	**3.25**
3	Etridiazole	**9.04**	18	Pendimethalin	**6.71**
4	Trichlorfon	**9.37**	19	Methyldymron	**3.90**
5	Chloroneb	**2.32**	20	Isophenphos	**3.67**
6	Benfluralin	**5.25**	21	Captan	**3.97**
7	Pencycuron	**2.60**	22	Butamifos	**8.55**
8	Simazine	**4.25**	23	Napropamide	**4.70**
9	Propyzamide	**5.53**	24	Flutolanil	**8.35**
10	Dimpylate	**6.00**	25	Isoprothiolane	**4.53**
11	Chlorothalonil	**2.19**	26	Isoxathion	**6.17**
12	Terbucarb	**2.37**	27	Mepronil	**3.20**
13	Tolclofos-methyl	**1.84**	28	Pyributicarb	**6.42**
14	Metalaxyl	**4.73**	29	Iprodione	**7.99**
15	Dithiopyr	**4.21**	30	Pyridaphenthion	**3.47**

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