

Application Data Sheet

No.83

GC-MS

Gas Chromatograph Mass Spectrometer

Simultaneous Analysis of 66 Pesticides by GC-MS Using Hydrogen Carrier Gas

Helium gas is typically used as a carrier gas for GC-MS. However, in recent years, the insufficient supply of helium gas and its increase in price have caused problems, prompting global efforts to address these problems. The use of hydrogen gas as an alternative carrier gas holds promise for addressing the issue.

This data sheet presents the application of hydrogen carrier gas to the simultaneous analysis of a standard sample of 66 pesticides using the GCMS-QP2010 Ultra, which features a Dual Inlet Turbo Molecular Pump.

Experimental

Standard samples of pesticides (from Wako Pure Chemical Industries, Ltd.) were diluted with methylene chloride to prepare 0.005 and 0.5 mg/L standard solution mixtures. Table 1 shows the analytical conditions.

Table 1 Analytical Conditions

GC-MS: GCMS-QP2010 Ultra

Column: Rtx $^{\otimes}$ -5MS (Length 30 m, 0.25 mm I.D., df = 0.25 μ m) (Restek Corporation, P/N: 12623)

Glass Liner: Custom SkyLiner, Splitless (Restek Corporation, P/N: 567366)

[GC]

Injection Temp.: 250 °C

Column Oven Temp.: 80 °C (2 min) \rightarrow (20 °C /min) \rightarrow 180 °C \rightarrow (5 °C /min) \rightarrow 280 °C (3 min)

Injection Mode: Splitless (High Pressure Injection 250 kPa, 2.3 min)

Flow Control Mode: Linear Velocity (65 cm/sec)

Injection Volume: 2 μL Sampling Time: 2 min [MS]

Interface Temp.: 250 °C Ion Source Temp.: 230 °C

[Scan Conditions]

Mass Range: m/z 70 - 360 Scan Event Time: 0.5 sec

[SIM Conditions]

Monitoring *m/z*: See below. SIM Event Time: 0.3 sec

SIM Monitoring m/z

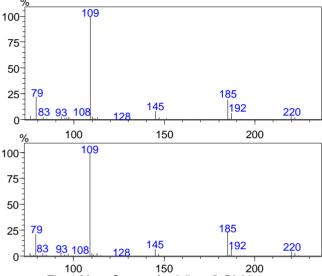
Compound Name	m/z		Compound Name	m/z		Compound Name	m/z		Compound Name	m/z	
Dichlorvos	185.0	109.0	Chlorothalonil	266.0	264.0	Pendimethalin	252.0	281.0	Chlornitrofen	319.0	317.0
Dichlobenil	171.0	173.0	Iprobenfos	204.0	91.0	Methyl dymron	107.0	119.0	Edifenphos	310.0	109.0
Etridiazole	213.0	211.0	Bromobutide	120.0	119.0	Isofenphos	213.0	185.0	Propiconazole-1	259.0	261.0
Chloroneb	193.0	191.0	Terbucarb	220.0	205.0	Captan	79.0	117.0	Endosulfan	272.0	274.0
Isoprocarb	136.0	121.0	Simetryn	213.0	170.0	Dimepiperate	145.0	119.0	Propiconazole-2	259.0	261.0
Molinate	126.0	98.0	Tolclofos-methyl	265.0	125.0	Procymidone	96.0	283.0	Thenylchlor	127.0	288.0
Fenobucarb	150.0	121.0	Alachlor	188.0	160.0	Methidathion	145.0	85.0	Pyributicarb	165.0	108.0
Trifluralin	306.0	290.0	Metalaxyl	206.0	160.0	alpha-Endosulfan	241.0	195.0	Iprodione	314.0	316.0
Benfluralin	292.0	276.0	Dithiopyr	354.0	306.0	Butamifos	286.0	200.0	Pyridaphenthion	340.0	199.0
Pencycuron	125.0	180.0	Fenitrothion	277.0	260.0	Naproramide	128.0	72.0	EPN	157.0	169.0
Dimethoate	125.0	87.0	Esprocarb	91.0	222.0	Flutolanil	173.0	281.0	Piperophos	122.0	140.0
Simazine	201.0	186.0	Malathion	173.0	93.0	Isoprothiolane	189.0	118.0	Bifenox	310.0	343.0
Atrazine	215.0	200.0	Thiobencarb	100.0	72.0	Pretilachlor	238.0	262.0	Pyriproxyfen	136.0	226.0
Propyzamide	175.0	173.0	Fenthion	278.0	125.0	Buprofezin	105.0	175.0	Mefenacet	192.0	120.0
Pyroquilon	130.0	173.0	Chlorpyrifos	314.0	197.0	Isoxathion	177.0	105.0	Cafenstrole	100.0	188.0
Diazinon	304.0	179.0	Phthalide	243.0	241.0	beta-Endosulfan	195.0	241.0			
Disulfoton	89.0	97.0	Dimethametryn	212.0	255.0	Mepronil	119.0	269.0			

Analytical Results

The mass spectral patterns of some compounds are known to change when hydrogen carrier gas is used. To evaluate this, the 0.5 mg/L standard solution was measured in scan mode and checked the resulting mass spectra. Fig. 1 shows the mass spectra for dichlorvos. No change was evident in the mass spectral pattern for the target components.

In addition, it has been reported that using hydrogen carrier gas results in lower sensitivity than when helium is used. To test this, the 0.005 mg/L standard solution was measured in SIM mode using both gases and then compared the S/N ratios. For all the components, the S/N ratio for hydrogen carrier gas was lower, primarily due to the baseline of the chromatogram being higher. Fig. 2 shows the SIM mass chromatograms for dichlorvos, for which the S/N ratio was particularly reduced.

Table 2 shows the repeatability results (n = 5) at a concentration of 0.005 mg/L. For all components, %RSD was less than 10 %.



 $(\times 10,000)$ 185.0 S/N = 661.5 1.0 0.5 4.0 4.5 $(\times 10,000)$ 185 0 S/N = 4904.0 3.0 2.0 1.0 6.0

Fig. 1 Mass Spectra for 0.5 mg/L Dichlorvos (Top: hydrogen carrier gas, Bottom: helium carrier gas)

Fig. 2 SIM Mass Chromatograms for 0.005 mg/L Dichlorvos (Top: hydrogen carrier gas, Bottom: helium carrier gas)

Table 2 Repeatability (n = 5, 0.005 mg/L)

Compound Name	%RSD	Compound Name	%RSD	Compound Name	%RSD	Compound Name	%RSD
Dichlorvos	3.96	Chlorothalonil	1.67	Pendimethalin	4.70	Chlornitrofen	4.18
Dichlobenil	2.19	Iprobenfos	7.26	Methyl dymron	4.93	Edifenphos	7.76
Etridiazole	2.47	Bromobutide	3.79	Isofenphos	8.79	Propiconazole-1	7.72
Chloroneb	2.61	Terbucarb	5.08	Captan	6.86	Endosulfan	3.28
Isoprocarb	4.12	Simetryn	5.60	Dimepiperate	7.67	Propiconazole-2	7.08
Molinate	1.04	Tolclofos-methyl	2.59	Procymidone	6.65	Thenylchlor	3.85
Fenobucarb	3.34	Alachlor	5.80	Methidathion	5.84	Pyributicarb	5.60
Trifluralin	1.44	Metalaxyl	8.22	alpha-Endosulfan	9.82	Iprodione	4.20
Benfluralin	2.04	Dithiopyr	1.67	Butamifos	8.81	Pyridaphenthion	6.36
Pencycuron	3.25	Fenitrothion	5.68	Naproramide	9.34	EPN	7.40
Dimethoate	3.68	Esprocarb	7.13	Flutolanil	3.51	Piperophos	8.33
Simazine	6.79	Malathion	7.01	Isoprothiolane	9.47	Bifenox	9.05
Atrazine	5.35	Thiobencarb	2.40	Pretilachlor	7.38	Pyriproxyfen	1.71
Propyzamide	4.82	Fenthion	4.63	Buprofezin	9.01	Mefenacet	6.06
Pyroquilon	3.94	Chlorpyrifos	6.57	Isoxathion	3.54	Cafenstrole	7.55
Diazinon	7.06	Phthalide	2.41	beta-Endosulfan	2.97		
Disulfoton	7.54	Dimethametryn	2.80	Mepronil	5.13		

Summary

Using hydrogen carrier gas for the simultaneous analysis of 66 pesticides showed no change in the mass spectral pattern. Since the flow rate is high compared to when helium carrier gas is used, however, the baseline was raised, resulting in lower sensitivity. Nevertheless, the results demonstrated that the GCMS-QP2010 Ultra, featuring a Dual Inlet Turbo Molecular Pump, provides satisfactory sensitivity that allows application of hydrogen gas. However, be sure to confirm the required sensitivity and quantitative performance before replacing helium with hydrogen carrier gas.

Hydrogen gas is combustible, so it is necessary to handle it carefully. For safety measures, please refer to our web site: http://www.shimadzu.com/an/gc/support/fag/bombe/bombe1.html

First Edition: July, 2013

