Application Note 10446

Determination of Organochlorine Pesticides Using GC-MS with a Helium-conserving Injector

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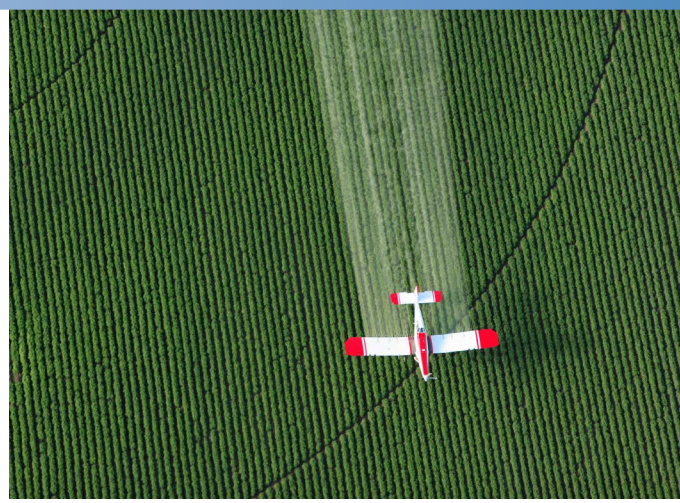
Keywords
Chromeleon CDS software, Environmental, Gas chromatography, GC-MS, Instant Connect Helium Saver injector, ISQ

Goal
To describe the performance of the Thermo Scientific™ ISQ™ LT GC-MS system equipped with an Instant Connect Helium Saver injector and Thermo Scientific Dionex™ Chromeleon™ 7.2 Chromatography Data System (CDS) software in identifying and quantifying organochlorine pesticides.



Introduction
Organochlorine pesticides are among the most toxic synthetic pesticides in the world. For this reason, their presence in the environment must be constantly and carefully monitored. Gas chromatography coupled with mass spectrometry for detection is one of the techniques of choice used to analyze these compounds.

To guarantee the accuracy and reproducibility of the analysis, the sample introduction phase is very important, the injector must provide optimal inertness to prevent compound breakdown and degradation and ensure a constant supply of carrier gas. Modern split/splitless injectors can ably handle this analysis. However, they require a large amount of helium for their operations.

To overcome this problem, Thermo Fisher Scientific introduced the innovative Instant Connect Helium Saver injector module. This injector employs a double-gas system that allows it to minimize helium consumption. Helium is used only as a carrier gas inside the column. All other operations, such as the split and purge flow feeding, use nitrogen. Here we demonstrate the performance of a GC-MS system equipped with the Instant Connect Helium Saver injector for the analysis of organochlorine pesticides.

Method Setup

GC Conditions	
TRACE 1310 GC	
Injection volume:	1 µL
Liner:	Splitless w/glass wool
Carrier gas:	Helium
Column type:	Thermo Scientific™ TraceGOLD™ TG-5 30 m, 0.25 mm, 0.25 µm
Column oven:	100 °C, hold 2 min. Ramp 15 °C/min to 160 °C, hold 5 min. Ramp 5 °C/min to 270 °C hold 2 min
SSL Injector:	225 °C; splitless mode for 2 min with a split ratio of 50:1. Helium delay 0.1 min
Column flow:	Constant flow at 1 mL/min
ISQ LT Mass Spectrometer	
Source temperature:	270 °C
Transfer line temperature:	270 °C

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Table 1. Recommended instrument parameters.

FS Conditions				
Acquisition start	**Mass range**	**Dwell Time**		
4.00	50-350	0.2		
SIM settings				
Compound	**Retention time**	**Polarity**	**Window**	**Ion**
a-Lindane	11.58	Positive	0.3	181, 219
g-Lindane	12.38	Positive	0.3	183, 219
b-Lindane	12.60	Positive	0.3	181, 219
d-Lindane	13.34	Positive	0.3	183, 219
Heptachlor	14.96	Positive	0.3	100, 272
Aldrin	16.14	Positive	0.3	66, 263
Heptachlor epoxyde	17.52	Positive	0.3	81, 263
g-Chlordane	18.35	Positive	0.3	237, 272
Endosulfan I	18.78	Positive	0.3	195, 241
a-Chlordane	18.89	Positive	0.3	237, 272
DDE	19.66	Positive	0.3	246, 318
Dieldrin	19.70	Positive	0.3	79, 263
Endrin	20.46	Positive	0.3	263, 281
Endosulfan II	20.78	Positive	0.3	159, 195
DDD	21.14	Positive	0.3	165, 235
Endrin aldehyde	21.47	Positive	0.3	67, 250
Endosulfan sulfate	22.30	Positive	0.3	229, 272
DDT	22.45	Positive	0.3	165, 235
Endrin ketone	23.93	Positive	0.3	67, 317
Methoxychlor	24.52	Positive	0.3	152, 227

Methods

The TRACE 1310 GC portion of the ISQ LT GC-MS system was equipped with one Instant Connect Helium Saver injector with a splitless w/glass wool liner (P/N 453A1925). A Thermo Scientific™ TraceGOLD™ TG-5MS 30 m, 0.25 mm 0.25 µm (P/N 26098-1420) column was used. The standards used for calibration were ordered from Restek: Organochlorine Pesticide Mix AB # 3 (P/N 32415).

The analysis was first run as a full scan to set up the single ion monitoring (SIM) conditions and then in SIM mode. The data were collected and processed using the Chromeleon 7.2 CDS software.

Results and Discussion

Figure 1 shows a chromatogram of the full scan analysis at a concentration of 1 ppm. The full scan is used to set up the time windows and target ions for the SIM for each compound. The SIM is used for quantitation and identification.

The calibration curve built comprises six points at concentrations of 1, 5, 10, 20, 50, and 100 ppb. Calibration results are reported in Table 2.

The area and retention time repeatability of the system has been assessed performing 10 consecutive runs of a sample at a concentration of 20 ppb.

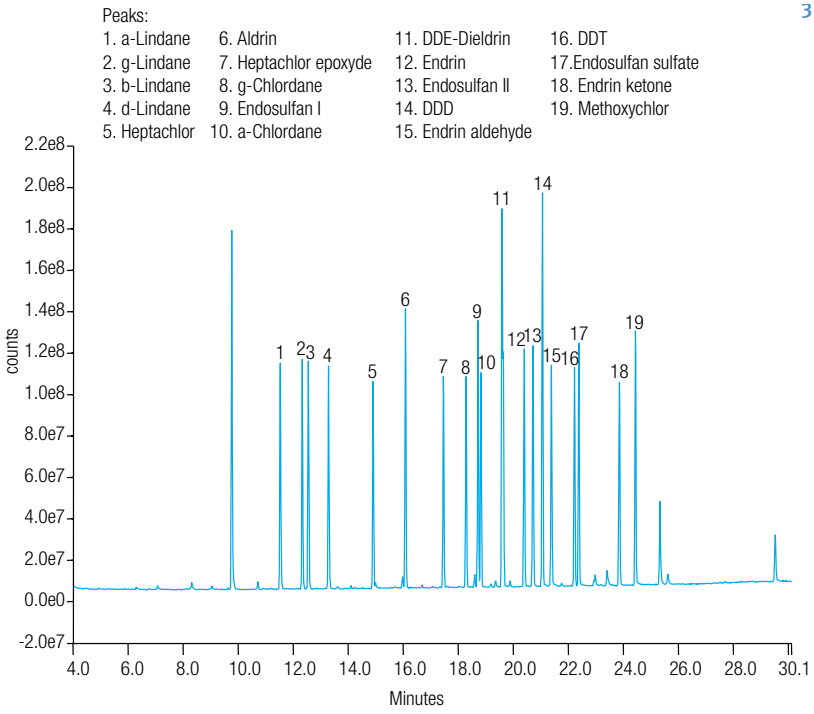

Figure 1. Analysis of organochlorine pesticides at 1 ppm in Full Scan mode.

Table 2. Calibration results.

Peak Name	Ret.Time	Cal.Type	Number of Points	Coeff. of Determination
a-BHC	11.565	Lin, WithOffset	6	0.999
g-BHC	12.362	Lin, WithOffset	6	0.998
b-BHC	12.585	Lin, WithOffset	6	0.998
d-BHC	13.322	Lin, WithOffset	6	0.997
Heptachlor	14.943	Lin, WithOffset	6	0.997
Aldrin	16.125	Lin, WithOffset	6	0.999
Heptachlor epoxyde	17.507	Lin, WithOffset	6	0.999
g-Chlordane	18.334	Lin, WithOffset	6	0.998
Endosulfan I	18.767	Lin, WithOffset	6	0.998
a-Chlordane	18.875	Lin, WithOffset	6	0.998
DDE	19.637	Lin, WithOffset	6	0.997
Dieldrin	19.684	Lin, WithOffset	6	0.997
Endrin	20.445	Lin, WithOffset	6	0.998
Endosulfan II	20.760	Lin, WithOffset	6	0.997
DDD	21.102	Lin, WithOffset	6	0.996
Endrin aldehyde	21.435	Lin, WithOffset	6	0.998
Endosulfan sulfate	22.272	Lin, WithOffset	6	0.996
DDT	22.431	Lin, WithOffset	6	0.996
Endrin ketone	23.907	Lin, WithOffset	6	0.997
Methosychlor	24.488	Lin, WithOffset	6	0.995

Table 3. Area repeatability.

Compound	RSD %
a-BHC	2.62
d-BHC	3.40
b-BHC	2.35
g-BHC	2.42
Heptachlor	2.42
Aldrin	2.56
Heptachlor epoxide	2.57
g-Chlordane	2.53
Endosulfan	2.50
a-Chlordane	2.67
DDE	2.71
Dieldrin	2.32
Endrin	2.54
Endosulfan II	2.83
DDD	2.61
Endrin aldehyde	2.24
DDT	8.32
Endosulfan sulfate	2.64
Endrin ketone	2.17
Metoxychlor	2.89

Table 4 and 5. Retention time repeatability.

	a-BHC	d-BHC	b-BHC	g-BHC	Heptachlor	Aldrin	Heptachlor epoxide	g-Chlordane	Endosulfan	a-Chlordane
	11.6007	12.4201	12.6255	13.3573	14.9839	16.1607	17.5427	18.3694	18.8037	18.9112
	11.5955	12.3966	12.62	13.3519	14.9786	16.1553	17.5373	18.3642	18.7984	18.9059
	11.6007	12.4021	12.6255	13.3573	14.9839	16.1607	17.5427	18.3694	18.8037	18.9112
	11.5906	12.39767	12.6201	13.352	14.7986	16.1556	17.35374	18.3642	18.7986	18.9061
	11.6008	12.4019	12.6252	13.357	14.9839	16.1606	17.5425	18.3693	18.8036	18.911
	11.6007	12.4043	12.6251	13.3569	14.9836	16.1655	17.5472	18.3689	18.8084	18.9108
	11.5957	12.3993	12.6252	13.3569	14.9837	16.1605	17.5423	18.3961	18.8035	18.9059
	11.5957	12.3994	12.6252	13.3572	14.984	16.1608	17.5425	18.3694	18.8037	18.9112
	11.5909	12.3946	12.6154	13.3523	14.974	16.1557	17.5377	18.3644	18.7987	18.9062
	11.591	12.3922	12.6156	13.3473	14.974	16.1508	17.5327	18.3594	18.7937	18.9012
Avg	11.60	12.40	12.62	13.35	14.96	16.16	17.52	18.37	18.80	18.91
Std.Dev	0.0043	0.0077	0.0042	0.0035	0.0578	0.0042	0.0593	0.0100	0.0042	0.0035
RSD%	0.04	0.06	0.03	0.03	0.39	0.03	0.34	0.05	0.02	0.02

	DDE	Dieldrin	Endrin	Endosulfan II	DDD	Endrin aldehyde	DDT	Endosulfan sulfate	Endrin ketone	Metoxychlor
	19.6733	19.7207	20.481	20.7985	21.1411	21.4737	22.3123	22.4647	23.9399	24.5217
	19.6679	19.7203	20.4756	20.7983	21.1358	21.4684	22.3069	22.4643	23.9394	24.5162
	19.6733	19.7207	20.481	20.7985	21.1411	21.4737	22.3123	22.4647	23.9399	24.5217
	19.668	19.72053	20.4756	20.7982	21.1409	21.4685	22.307	22.4645	23.9396	24.5164
	19.6779	19.7254	20.4805	20.8031	21.1407	21.4732	22.3167	22.4692	23.9444	24.5262
	19.6778	19.7253	20.4855	20.803	21.1456	21.4734	22.312	22.4695	23.9446	24.5214
	19.6729	19.7204	20.4806	20.8034	21.1409	21.4735	22.312	22.4695	23.9396	24.5214
	19.673	19.7205	20.4807	20.8032	21.1408	21.4735	22.3119	22.4694	23.9395	24.5214
	19.6682	19.7206	20.4759	20.7986	21.1412	21.4688	22.3073	22.4647	23.9348	24.5166
	19.6631	19.7106	20.4707	20.7884	21.131	21.4586	22.3021	22.4546	23.9298	24.5116
Avg	19.67	19.72	20.48	20.80	21.14	21.47	22.31	22.47	23.94	24.52
Std.Dev	0.0047	0.0040	0.0042	0.0045	0.0039	0.0048	0.0042	0.0045	0.0043	0.0042
RSD%	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02

Conclusions

This work highlights the excellent results that can be achieved using a GC/MS system, such as the ISQ LT GC-MS system. The analytical performance of the Instant Connect Helium Saver injector is remarkable in terms of both area and retention time reproducibility. As with other TRACE 1300 Series injectors and detectors, the Helium Saver benefits from its modularity, providing freedom from helium supply shortages, while maintaining performance identical to that of traditional SSL injectors. These unique features of the Helium Saver reduce cost without the need to change analytical methods or routine. Using a data system, such as the Chromeleon CDS software, provides a powerful tool for data acquisition and reprocessing along with unsurpassed ease of use.

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