Saika Technological Institute Foundation

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No. 001

LVI-S200 Application Data

New PTV Injector for GC, GC/MS
"Unique Stomach Shaped Insert"

Various sort of injectors are used in GC. And also, various type of inserts are used in their injector. Most popular type of conventional injectors has a straight shape. Some of them has glass or quartz wool packed inside. Also PTV injector is used for analyzing thermally labile components or injecting large volume of sample. In PTV injector, the temperature of the injector can be raised on temperature programming.

Recently, we have developed new PTV injector named "LVI-S200" which has a unique "stomach" shaped insert inside as shown in Fig. 1. In this injector, sample introduced can stay as liquid in the insert. This unique shape of the insert makes it possible to carry out Large Volume Injection, Derivatization Injection or Cold Splitless Injection of sample.

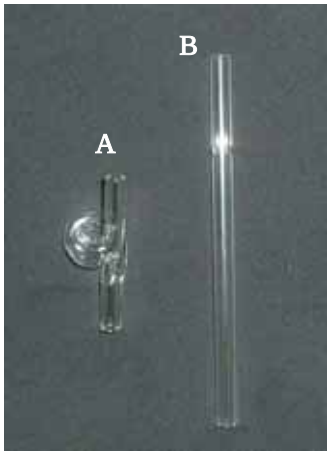


Fig. 1 A. Spiral insert
B. Conventional insert

In this data sheet, we will show you some of these application data obtained using this LVI-S200 injector.

LVI-S200 injector for GC

- ○ Large Volume Injection
- ○ Derivatization Injection
- ○ Cold Splitless Injection

Content

Large Volume Injection

At first, the injector is kept in a low temperature in split mode, then large volume of sample is injected into the insert, and this sample stays as liquid in the stomach shaped insert. In this state, solvent is evaporated and sample is concentrated under carrier gas flow like nitrogen purge concentration process usually done before sample injection. Next, sample is introduced into column by raising the temperature of the insert in splitless mode.

Standard sample analysis: n-C10～C40

Large sample volume of 50 μL is injected

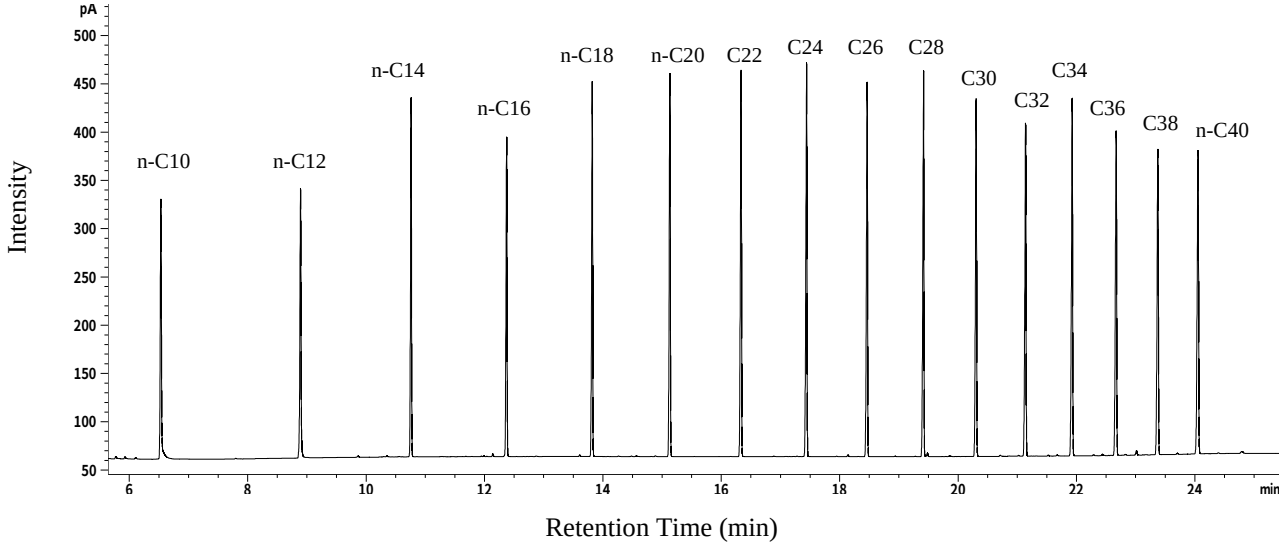


Fig. 2 Chromatogram obtained by injecting 50 μL volume of n-alkenes in hexane (0.5 ng/μL).

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	70℃-120℃/min-290℃(22min)
Solvent Purge Time	18 sec
Auto-Sampler	AT 7683
GC/MS	AT 6890N
Column	DB-5MS 0.25mm×20m, 0.25μm
Column Oven Temp.	50℃(5min)-15℃/min-350℃(2min)
Detector Temp	320℃
Detector	FID
Splitpurge Flow	150 ml/min
Splitless Time	4 min

Large Volume Injection
Peak Area Repeatability

Table 1 Peak areas and the relative standard deviations (R.S.D., n=8)

No.	1	2	3	4	5	6	7	8	RSD(%)
n-C10	185.9	186.2	183.2	187.3	187.8	184.4	186.5	185.5	0.81
n-C12	191.0	192.1	189.8	193.3	193.4	189.7	190.6	191.3	0.75
n-C14	216.3	217.4	215.0	218.6	219.1	214.8	215.2	216.1	0.76
n-C16	203.8	204.6	203.6	206.4	207.4	202.6	203.0	203.7	0.82
n-C18	228.6	229.6	228.0	230.5	231.7	226.9	227.1	229.0	0.72
n-C20	242.5	243.7	242.2	244.3	245.6	240.0	240.7	242.4	0.76
n-C22	249.6	251.0	249.3	251.1	253.0	247.1	247.5	249.2	0.78
n-C24	241.3	242.7	240.9	243.0	244.8	238.9	240.8	241.0	0.74
n-C26	231.7	233.1	231.1	233.1	234.9	229.1	229.7	231.3	0.82
n-C28	235.6	236.9	235.1	237.2	239.1	234.2	233.7	235.5	0.74
n-C30	222.4	223.6	222.0	223.9	225.7	221.4	220.6	222.5	0.72
n-C32	222.2	223.2	221.6	223.4	225.4	220.1	220.6	222.4	0.76
n-C34	233.4	234.2	232.5	234.5	236.6	231.0	231.8	233.8	0.75
n-C36	226.3	227.3	225.1	227.3	230.8	224.0	224.8	227.3	0.94
n-C38	214.6	215.0	213.3	215.1	218.7	212.4	213.3	215.9	0.91
n-C40	227.7	227.9	226.4	227.9	231.8	226.2	227.0	230.7	0.88

Injection volume; 25 μL. Sample; Conc. 0.5 ng/μL in hexane

Linearity between Peak Area and Injection Volume

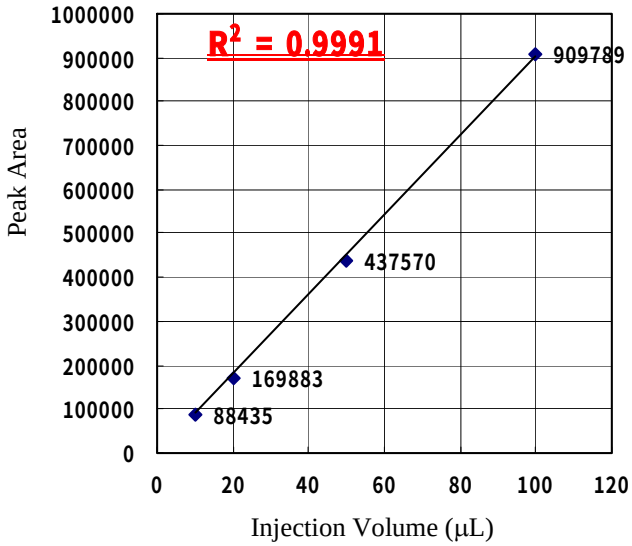
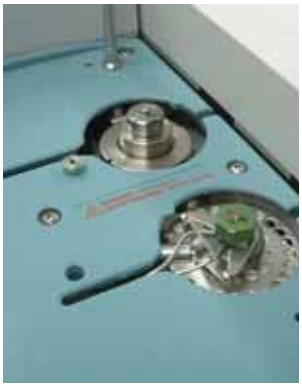


Fig. 3 Relation of injection volume and peak area.
Sample; n-C16 0.02 ng/μL in hexane
Injection volume; 10, 20, 50, 100 μL
※GC/MS (SIM)

Good linearity is obtained between injection volume (10,20,50,100 μL) and its corresponding peak area. Used sample is n-C16.

Comparison between Large Volume Injection and regular volume injection



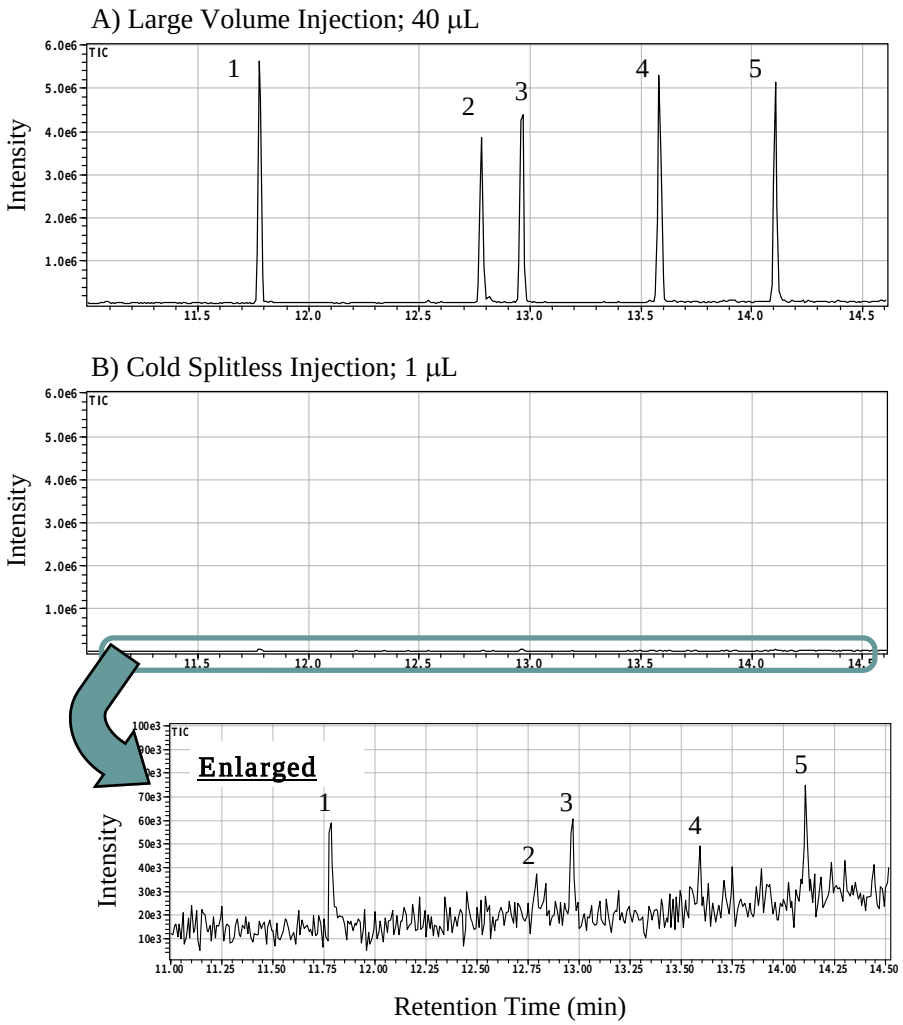
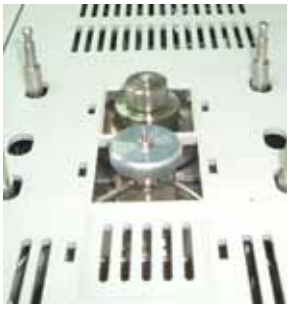


Fig. 4 Comparison of chromatogram between Large Volume Injection and regular volume injection
(Pesticides, Conc. 0.1 ng/mL in hexane)

Chromatogram obtained by injecting the same sample of Large Volume (A: 40µL) and regular volume (B: 1µL) are compared. Chromatogram A shows peaks having sufficient peak area and small noise for obtaining good results of quantification and qualification. While chromatogram B shows poor S/N, and this may be hard for that of quantification and qualification.

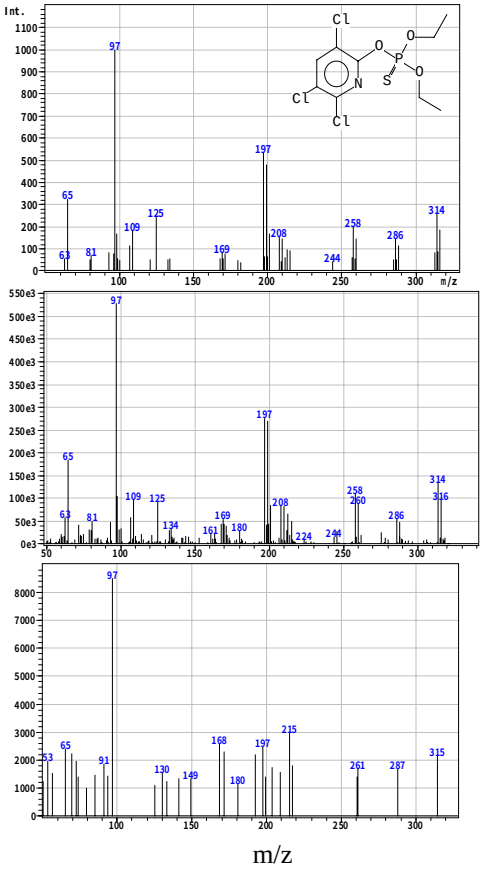
When you cannot obtain enough sensitivity in trace analysis, Large Volume Injection with LVI-S200 will solve your problem. By using this, you can get 10-100 times higher sensitivity, and so can get more reliable results.



MS spectrum registered in the library

Spectrum of No. 3 peak in Fig. 4 A)

Spectrum of No. 3 peak in Fig. 4 B)



m/z

Fig. 5 Comparison of MS spectra when varying injection volume

GC Conditions (LVI method)

Injector	LVI-S200
Injector Oven Temp.	69℃-100℃/min-240℃(20min)
Solvent Purge Time	24 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	Inert Cap 5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	70℃(4min)-20℃/min-190℃-15℃/min-260℃(3min)
Detector Temp	260℃
MS Method	SCAN
Splitpurge Flow	150 ml/min
Splitless Time	4 min

Multi-Scan Analysis of residual pesticides

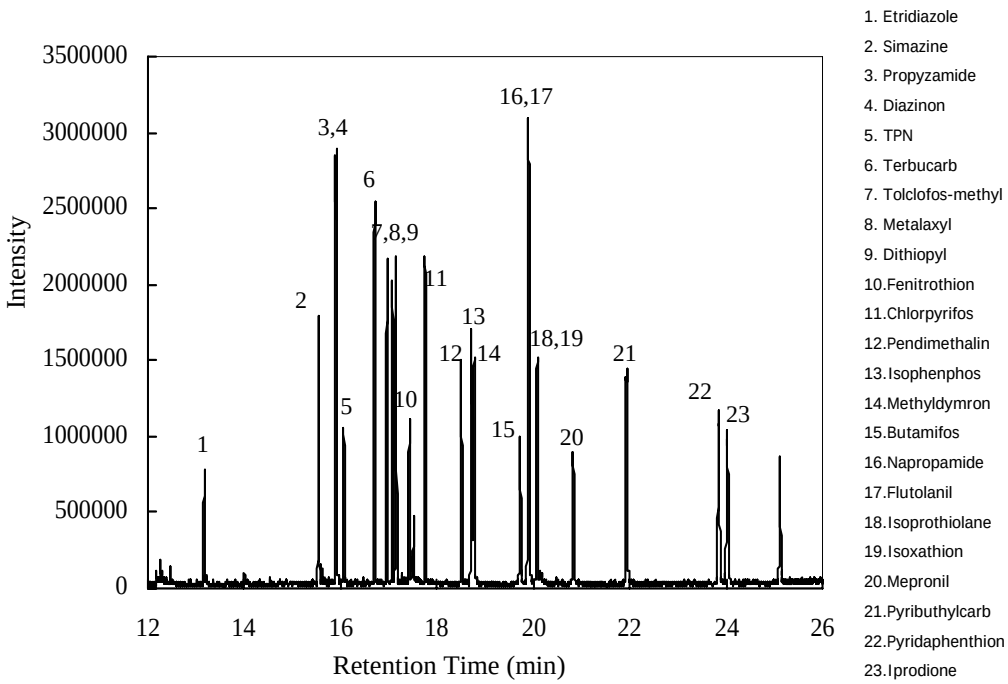


Fig.6 SCAN Chromatogram obtained by injecting 40 μL of pesticides in acetone (0.05 ng/μL).

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	56℃-100℃/min-260℃(20min)
Solvent Purge Time	50 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	50℃(5min)-15℃/min-210℃-4℃/min-245℃-15℃/min-290℃(3min)
Detector Temp	280℃
MS Method	SCAN
Splitpurge Flow	150 ml/min
Splitless Time	4 min

Large Volume Injection Analysis of PCBs(in Toluene)

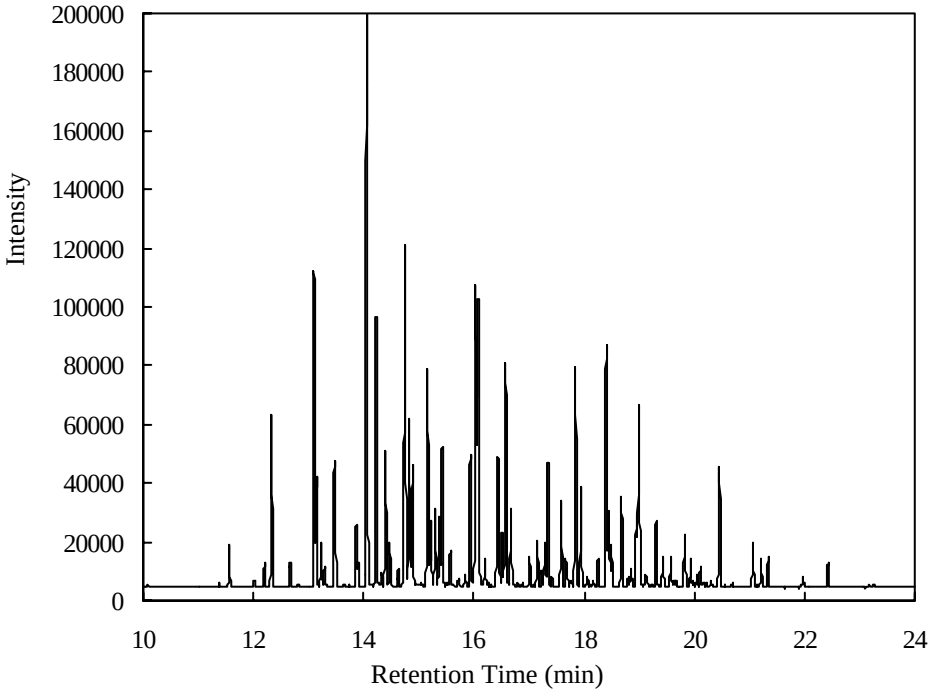


Fig. 7 Total ion chromatogram obtained by injecting 20 μL of PCBs in toluene.
PCBs; KC-300:KC-400:KC-500:KC-600=1:1:1:1

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	90℃(5min)-80℃/min-260℃(21min)
Solvent Purge Time	20 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	70℃(5min)-30℃/min-170℃-8℃/min-300℃(2min)
Detector Temp	300℃
MS Method	SIM
Splitpurge Flow	150 ml/min
Splitless Time	4 min

High Sensitivity Analysis of PAHs using LaviStoma

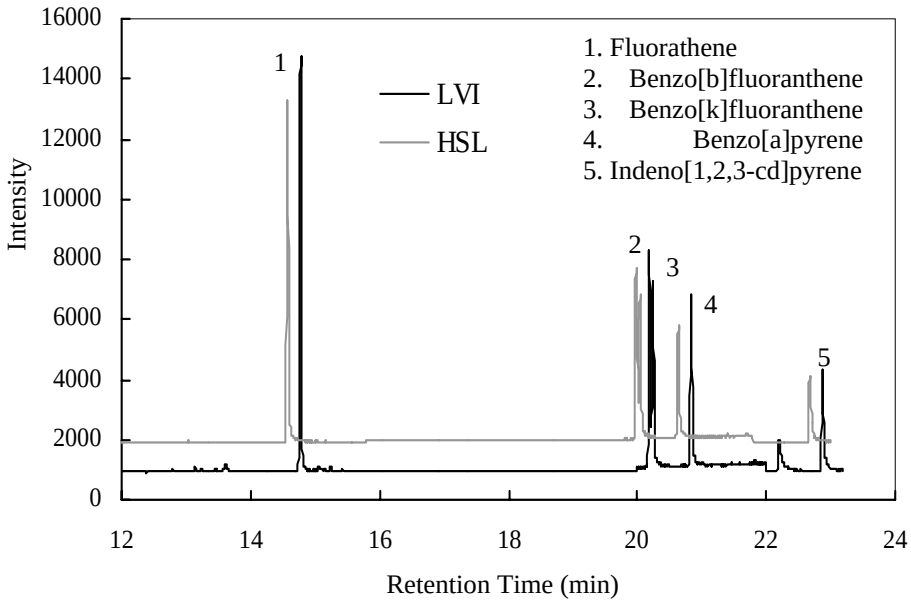


Fig. 8 Total ion chromatogram obtained by injecting 200 μL (LVI) of PAHs in hexane (0.1 pg/μL) and 2 μL (HSL) of PAHs in hexane (10 pg/μL).

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	69℃-100℃/min-280℃(20min)
Solvent Purge Time	100 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	60℃(4min)-15℃/min-300℃(3min)
Detector Temp	300℃
MS Method	SIM
Splitpurge Flow	150 ml/min
Splitless Time	4 min

Analysis of thermally labile pesticides

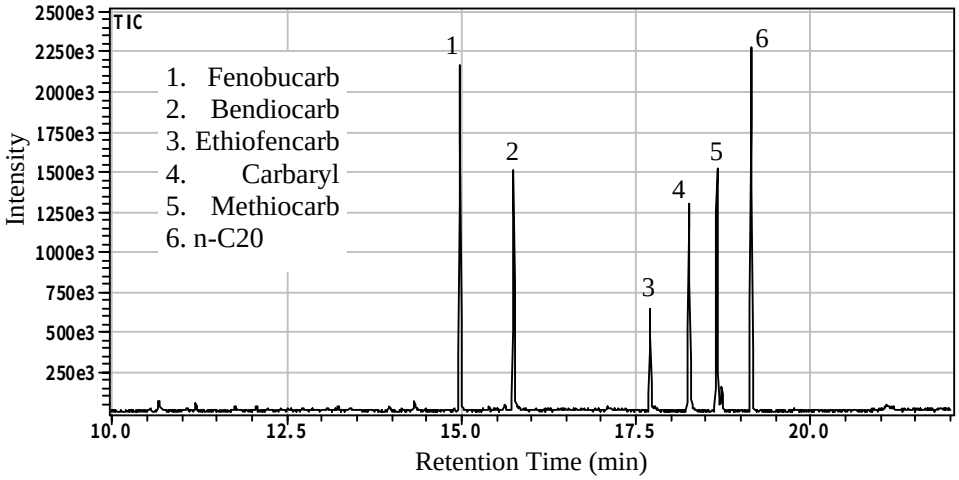


Fig. 9 SCAN chromatogram obtained by injecting 40 μL of N-methyl carbamate in hexane (0.1 ng/μL).

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	70℃-100℃/min-150℃(3min)-50℃/min-240℃(17min)
Solvent Purge Time	24 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	Inert Cap 5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	60℃(3min)-10℃/min-260℃(3min)
Detector Temp	240℃
MS Method	SCAN
Splitpurge Flow	50 ml/min
Splitless Time	3 min

Derivatization Injection

Sample and derivatization reagent are mixed in the stomach shaped insert of LVI-S200 injector. Here, sample is derivatized and concentrated in a short time.

TMS Derivatization of PCP (Pentachlorophenol)

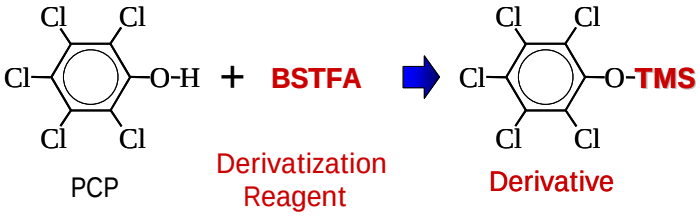


Fig. 11 TMS derivatization of PCP: BSTFA(2,2,2-Trifluoro-N,O-bis (trimethylsilyl)acetamide) is used as TMS derivatization reagent.

SCAN Chromatogram

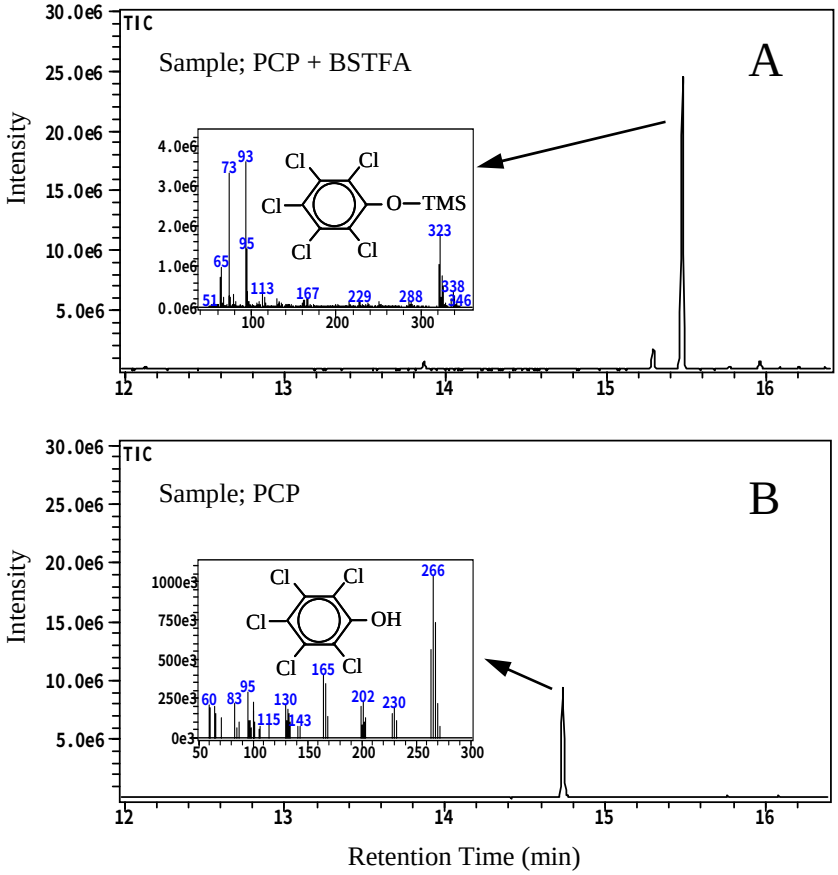


Fig. 12 SCAN chromatogram of PCP-TMS (A) and PCP (B). PCP- TMS (A) is obtained as a result of derivatization in the stomach shaped insert of LVI-S200 injector.

Accuracy of Derivatization Analysis Calibration

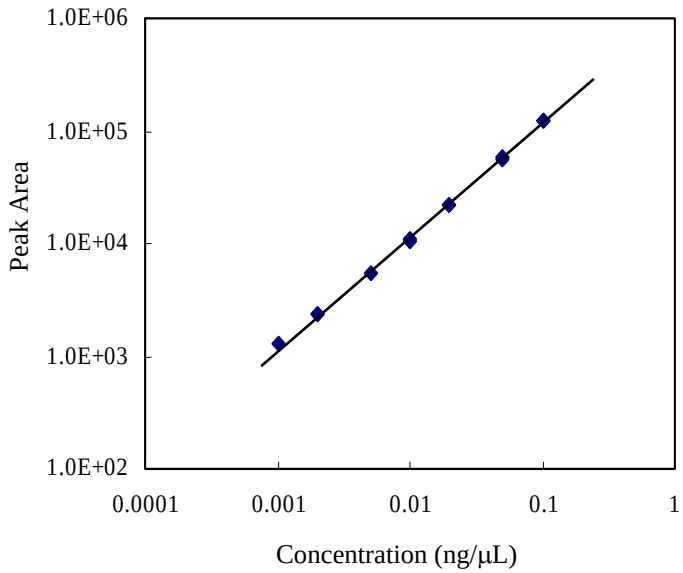


Fig. 13 Calibration curve of PCP-TMS with derivatization injection.

Repeatability of Peak Area

Table 2 Peak areas of PCP-TMS of 7 consecutive analysis, their average area and relative standard deviations (R.S.D.)

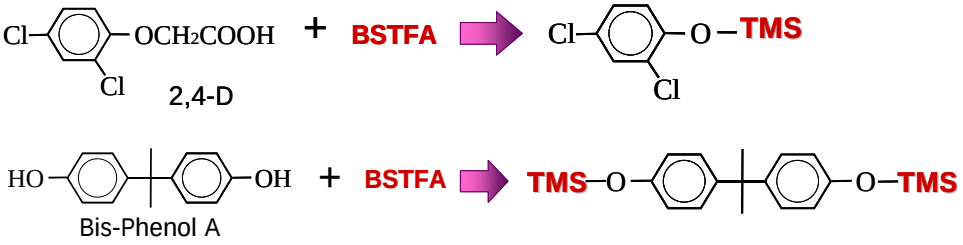
Compound	1	2	3	4	5	6	7	*Ave.*	**RSD**
PCP-TMS	11,206	10,993	11,146	11,285	11,393	11,038	10,910	*11,139*	**1.53**

Sample; 0.01 ng/μL PCP 20μL Derivatization reagent; 1% BSTFA 5μL

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	60℃-80℃/min-240℃(4min)-80℃/min-260℃(2min)
Solvent Purge Time	12 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	60℃(5min)-15℃/min-280℃(2min)
Detector Temp	280℃
MS Method	SIM
Splitpurge Flow	150 ml/min
Splitless Time	5 min

TMS Derivatization of 2,4-D & Bis-Phenol A



SCAN Chromatogram obtained by Derivatization Injection

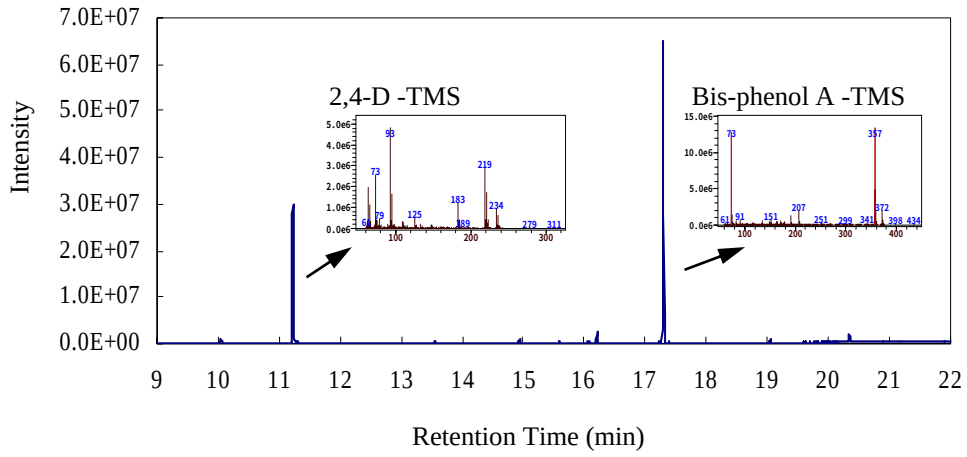


Fig. 15 SCAN chromatogram of 2,4-D -TMS and Bis-phenol A – TMS. They are obtained as a result of derivatization in the stomach shaped insert of LaviStoma injector.

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	60°C-80°C/min-240°C(4min)-80°C/min-260°C(2min)
Solvent Purge Time	12 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	60°C(5min)-15°C/min-280°C(2min)
Detector Temp	280°C
MS Method	SIM
Splitpurge Flow	150 ml/min
Splitless Time	5 min

TMS Derivatization of Fatty Acid

Fig. 16 TMS Derivatization of Fatty Acid

SCAN Chromatogram obtained by Derivatization Injection

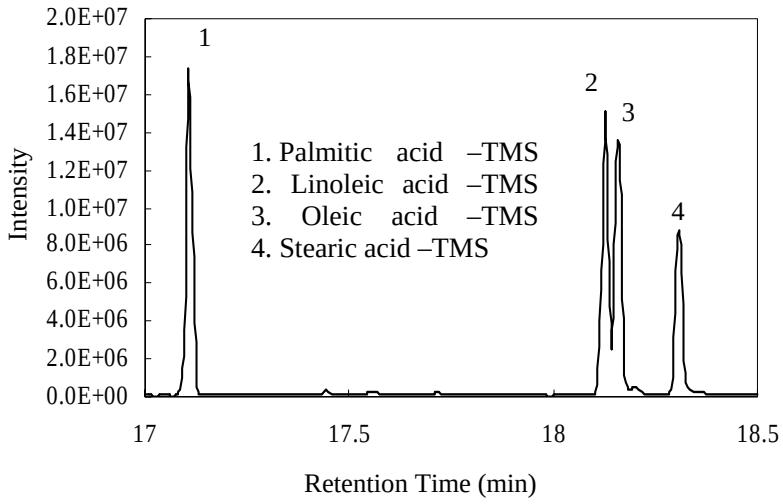


Fig. 17 SCAN chromatogram of fatty acid –TMS. They are obtained as a result of derivatization in the stomach shaped insert of LVI-S200 injector.

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	60℃-80℃/min-240℃(4min)-80℃/min-260℃(2min)
Solvent Purge Time	12 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	60℃(5min)-15℃/min-280℃(2min)
Detector Temp	280℃
MS Method	SIM
Splitpurge Flow	150 ml/min
Splitless Time	5 min

Analysis of Thermally Labile Compounds by Cold Splitless Injection

Analysis of Trichlorfone (DEP) using Various Type of Injections

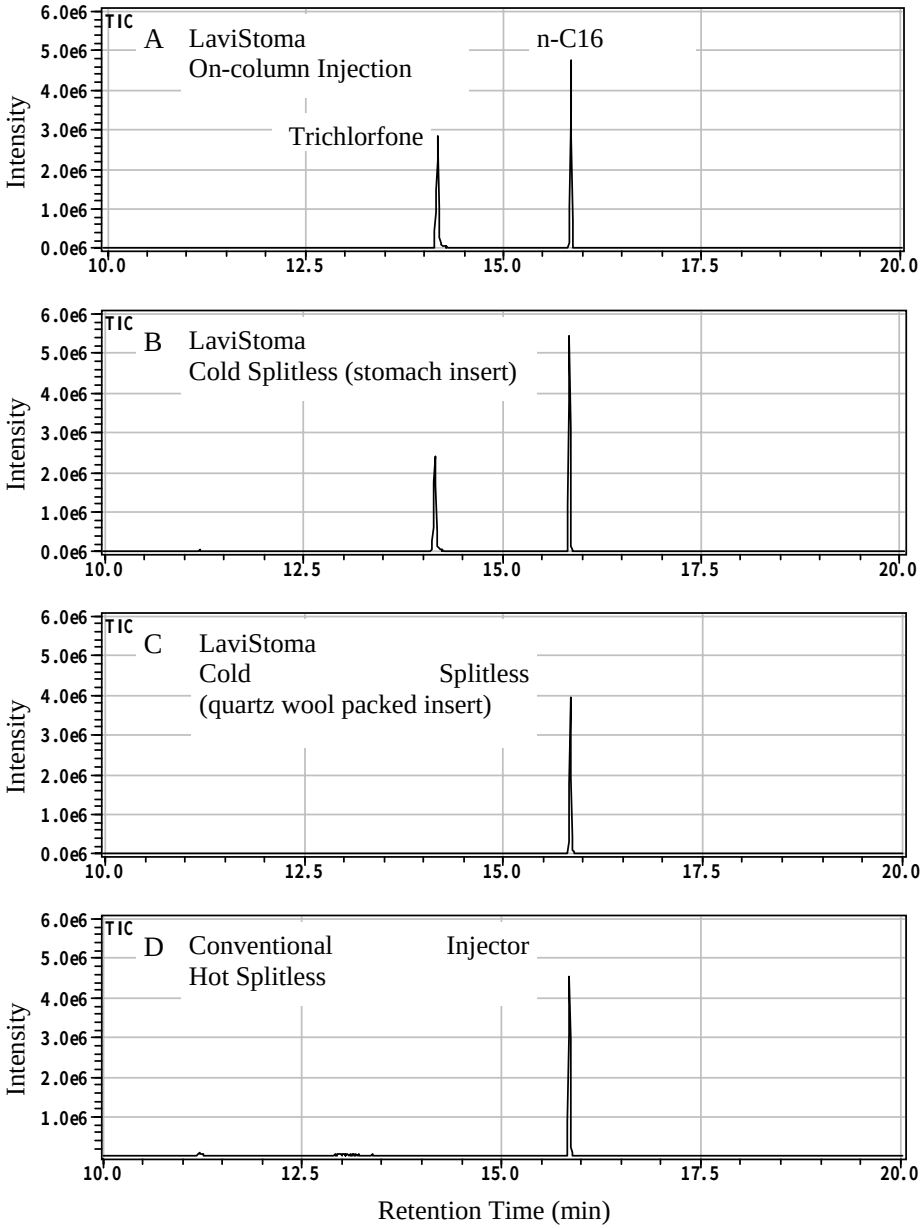


Fig. 18 Comparison of Chromatograms obtained using various type of injections
(DEP 10 ng/µL and n-C16 2 ng/µL in 20% acetone/hexane. Injection volume; 1 µL)

Cold Splitless Injection using LVI-S200 with Stomach Shaped Insert inside

The stomach shaped insert can hold sample as liquid within it. Therefore no quartz wool packing is necessary. At first, the injector is kept in a low temperature in split mode, then large volume of sample is injected into the insert, In this state, solvent is evaporated and sample is concentrated. Next, sample is introduced into column by raising the temperature of the insert in splitless mode. Finally, residual solvent or high boiling point contaminants are purged out in split mode.

GC Conditions

Cold Splitless (stomach insert or quartz wool packed insert)

Injector	LVI-S200
Injector Oven Temp.	60℃-100℃/min-120℃(3min)-50℃/min-240℃
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	Inert Cap 5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	50℃(3min)-10℃/min-240℃(3min)
Detector Temp	240℃
MS Method	SCAN
Pulsed Splitless	150 kPa (3min)
Splitpurge Flow	50 ml/min
Splitless Time	3 min

Hot Splitless

Injector	Conventional
Injector Oven Temp.	240℃

On-Column Injection

Injector	LVI-S200
Injector Oven Temp.	60℃-10℃/min-240℃(2min)

Analysis of Thermally Labile Compounds by Cold Splitless Injection

Analysis of N-methyl carbamate using Various Type of Injection

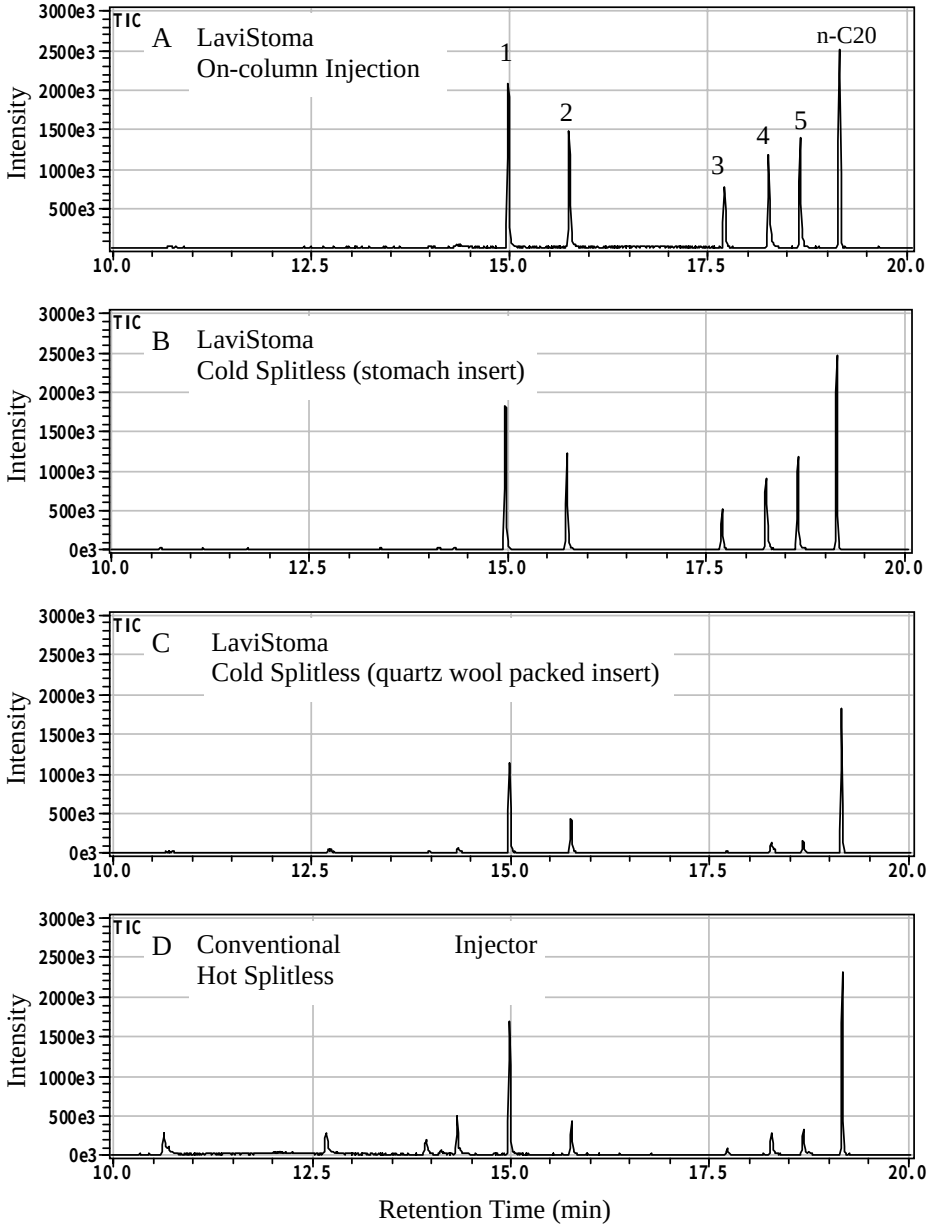


Fig. 19 Comparison of Chromatograms obtained using various type of injections (N-methyl carbamate 2 ng/μL and n-C20 0.5 ng/μL in 20% acetone/hexane. Injection volume; 1 μL)

1. Fenobucarb 2. Bendiocarb 3. Ethiofencarb 4. Carbaryl 5. Methiocarb

Cold Splitless Injection using LVI-S200 with Stomach Shaped Insert inside

In the conventional splitless injection with PTV, quartz wool is usually packed in its straight shaped insert. However, some of sample components might often be adsorbed on the active point of the quartz wool, and we could not obtain good results.

On the other hand, our stomach shaped insert of LVI-S200 does not need quartz wool packing. And we can get good results even for analyzing thermally labile compounds.

We believe that our LVI-S200 is one of the best PTV injection system we have ever obtained.

GC Conditions

Cold Splitless (stomach insert or quartz wool packed insert)

Injector	LVI-S200
Injector Oven Temp.	70℃-100℃/min-150℃(3min)-50℃/min-240℃
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	Inert Cap 5MS 0.25mm×30m, 0.25μm
Column Oven Temp.	60℃(3min)-10℃/min-260℃(3min)
Detector Temp	240℃
MS Method	SCAN
Pulsed Splitless	150 kPa (3min)
Splitpurge Flow	50 ml/min
Splitless Time	3 min

Hot Splitless

Injector	Conventional
Injector Oven Temp.	240℃

On-Column Injection

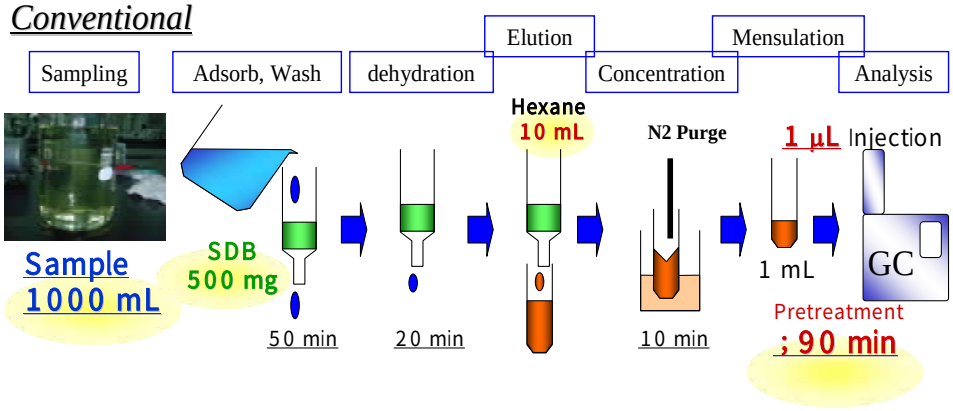
Injector	LVI-S200
Injector Oven Temp.	70℃-10℃/min-240℃(2min)

Analysis of PCBs in Water

When analyzing trace amount of PCBs, large volume of solvent is used for extracting trace amount of them. Then, large volume of solvent has to be evaporated out. We should avoid this for protecting environment. Here we have studied the way to make sample treatment as using less volume of solvent and sample using LVI-S200 injection system.

Make pretreatment process rapid and easy

- *Conventional*



- ***Using LVI-S200 Injector***

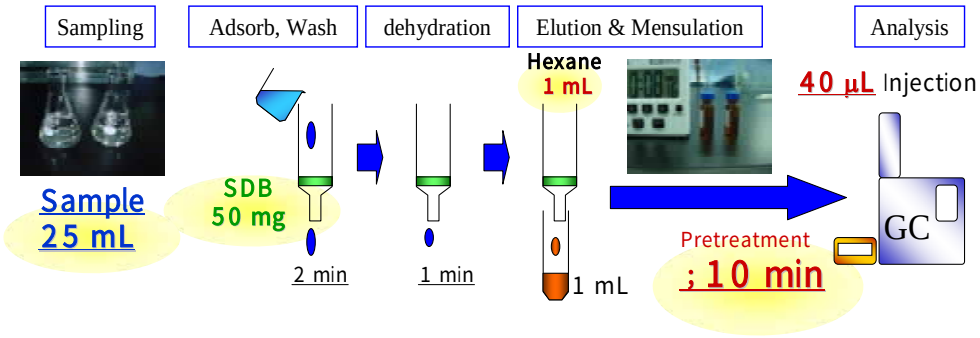


Fig. 20 Comparison of pretreatment process

GC Conditions

Injector	LVI-S200
Injector Oven Temp.	69℃-100℃/min-270℃(20min)
Solvent Purge Time	24 sec
Auto-Sampler	AOC-20i (Shimadzu)
GC/MS	QP5050A (Shimadzu)
Pre-column	Deactivated silica capillary tube 0.53mm×0.5m
Column	DB-5MS 0.25mm×30m, 0.25µm
Column Oven Temp.	70℃(5min)-25℃/min-170℃-7℃/min-250℃-15℃/min-300℃(3min)
Detector Temp	300℃
MS Method	SIM
Splitpurge Flow	150 ml/min
Splitless Time	4 min

Chromatogram

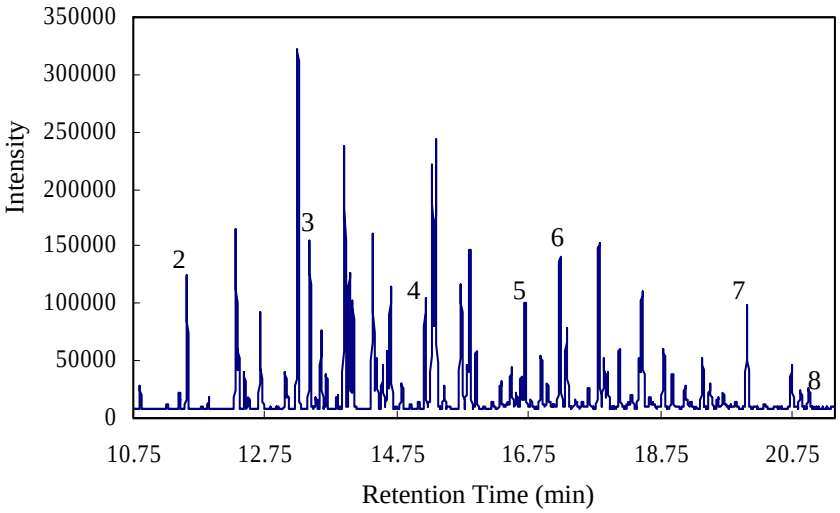


Fig. 21 Total ion chromatogram of river water spiked by 16 μg/L of PCBs.

Spike and Collect experiment

Table 3 Recovery and Reproducibility of PCBs spiked in purified water and river water

Compound	Purified water		River water	
	Rec.(%)	RSD(%, n=5)	Rec.(%)	RSD(%, n=5)
PCB-2	90.2	1.8	96.4	4.6
PCB-3	93.8	2.6	96.1	6.1
PCB-4	91.6	3.7	93.5	4.4
PCB-5	96.0	4.0	95.4	5.7
PCB-6	95.2	2.6	95.3	4.2
PCB-7	103.8	2.5	94.2	5.3
PCB-8	146.4	2.6	108.9	8.5

The sample was 16 mg/L PCB (KC-300, 400, 500, 600) spiked to the purified water and river water.

Life Test

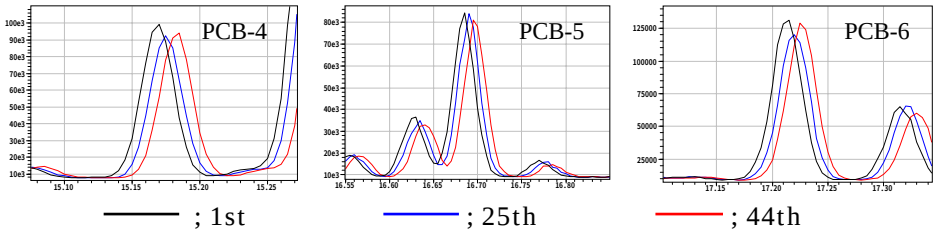


Fig. 22 Life test of PCBs measurement with repeated sample injection using LVI-S200 injection system

Analysis of Residual Pesticides in Golf Course

Here we have studied the way to make sample treatment on the analysis of residual pesticides in a golf course as using less volume of solvent and sample using LaviStoma injection system.

Make pretreatment process rapid and easy

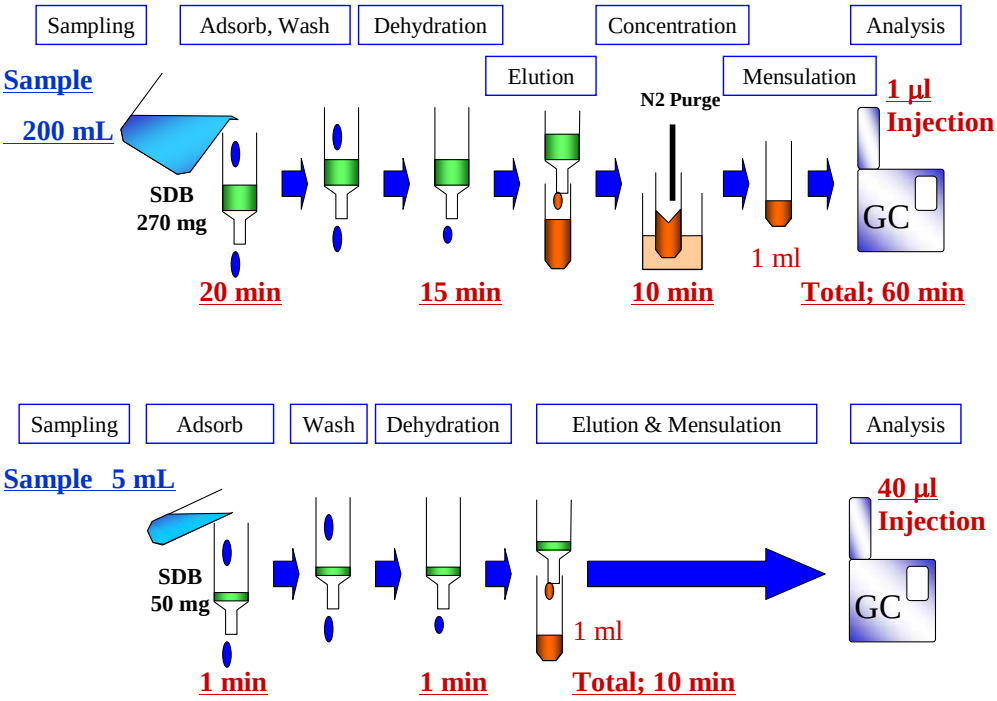


Fig. 23 Comparison of pretreatment process

GC Conditions

Injector	LVI-S200
Injector Oven Temp. Solvent Purge Time	69℃-100℃/min-260℃(20min) 24 sec
Auto-Sampler GC/MS	AOC-20i (Shimadzu) QP5050A (Shimadzu)
Pre-column Column	Deactivated silica capillary tube 0.53mm×0.5m DB-5MS 0.25mm×30m, 0.25µm
Column Oven Temp. Detector Temp MS Method	60℃(5min)-15℃/min-210℃-4℃/min-245℃-15℃/min-290℃(3min) 280℃ SIM
Splitpurge Flow Splitless Time	150 ml/min 4 min

Chromatogram

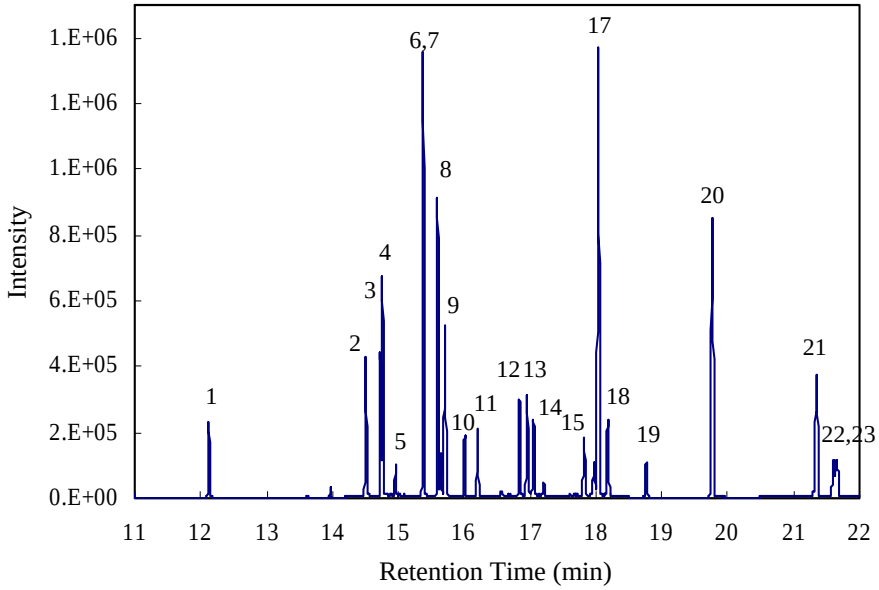


Fig. 24 Total ion chromatogram of waste water in a golf course spiked by 0.01 mg/L of pesticides

Spike and Collect experiment

Table 4 Recovery and Reproducibility of pesticides spiked in purified water and waste water in a golf course

No.	Compound	Purified water		Waste water
		Rec.	RSD (n=4)	Rec.
1	Etridiazole	98.1	1.4	80.8
2	Simazine	99.4	1.1	94.9
3	Diazinon	102.8	2.5	110.4
4	Propyzamide	103.0	1.7	96.5
5	TPN	92.2	3.2	31.6
6	Terbucarb	104.8	1.7	111.9
7	Tolclofos-methyl	100.5	1.4	94.1
8	Metalaxyl	96.3	1.1	86.9
9	Dithiopyl	101.2	1.8	103.2
10	Fenitrothion	109.9	3.5	96.9
11	Chlorpyrifos	98.3	2.4	88.5
12	Pendimethalin	101.2	2.4	103.7
13	Isophenphos	101.6	2.0	101.4
14	Methyldymron	80.2	13.1	36.5
15	Butamifos	105.0	2.8	115.8
16	Napropamide	104.2	1.8	107.1
17	Flutolanil	103.7	2.4	103.6
18	Isoprothiolane	105.1	2.3	107.2
19	Isoxathion	109.5	4.4	82.0
20	Mepronil	105.4	3.0	108.5
21	Pyributhylcarb	100.4	2.8	103.5
22	Pyridaphenthion	102.3	0.8	101.8
23	Iprodione	103.1	1.8	94.7

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