



# Application of derivatization in stomach shaped liner

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[Introduction] Derivatization analysis is usually performed in gas chromatography. In this case, derivatization proceeds in sample preparation step. But many of derivatization reagent are harmful for human body, and also, derivatization process contains laborious steps. In this research, we have studied the possibility of completing derivatization reaction in the stomach shaped liner of LaviStoma injection system, where injected sample is kept as liquid.

[Method] We have studied these samples of 1ppm Pentachlorophenol(PCP), Bisphenol A, 2,4-Dichlorophenol(2,4-D) and 10ppm fatty acids in 20% Acetone/Hexane. Derivatization reagent used here is 1% BSTFA(N,O-bis(trimethylsilyl)trifluoroacetamide) in 20% Acetone/Hexane. Operating conditions of GC/MS and this injection system are shown in Table 1. Using sandwich mode of Auto-injector, 20ul sample and 5ul derivatization reagent are sucked in from each vial consequently, and then injected into the liner. The injector temperature is set just below the boiling point of the solvent to avoid its bumping. Sample is derivatized and concentrated while solvent is vaporized in split mode. Next, injector temperature is raised, and derivatized sample is introduced into column in splitless mode. In this way, derivatization analysis is performed in GC/MS.

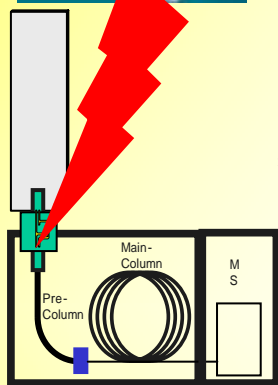


Fig. 1. Installation of large volume injection system

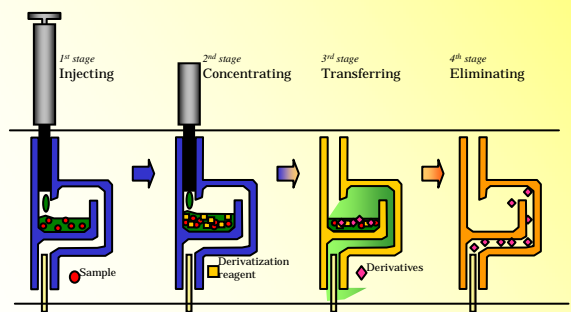
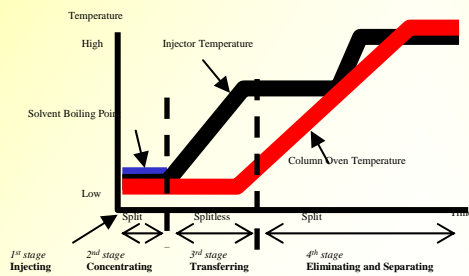


Table 1. Operating conditions of GC/MS and injector

Instrument	: GCMS-QP5050A(SHIMADZU)
	: AOC-201(SHIMADZU) with Sandwich method
Injector	: LaviStoma(EMINET) with middle size liner(0.5ml)
Column	: Pre-column : Deactivated silica capillary tube 0.53mm×0.3m
	: Main-column : DB-5MS 0.25mm×30m, 0.25μm
GC method	: Injection Temp. : 69°C(0.5min)-100°C/min-260°C(18min)
	: Solvent Purge Time : 5sec
GC Temp.	: 60°C(5min)-15°C/-300°C(2min)
MS method	: SCAN Mode, 9-22min, 60-500m/z

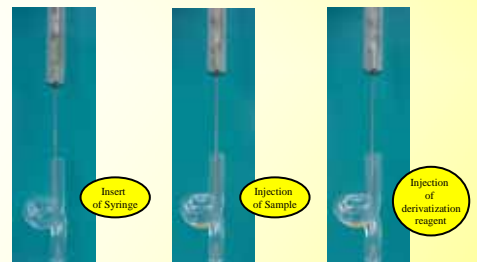


Fig. 2. Scheme of injection method using LaviStoma injection system

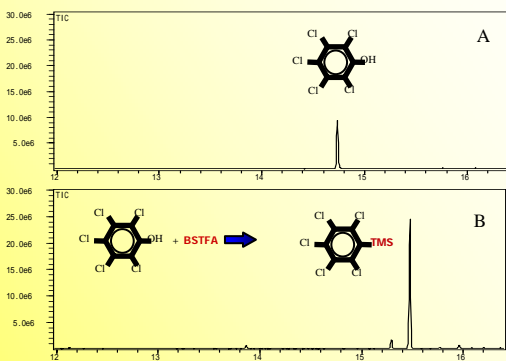


Fig. 3. Chromatogram of PCP and PCP-TMS. PCP-TMS is obtained as a result of derivatization in the stomach shaped liner of LaviStoma injector. BSTFA is used as derivatization reagent.

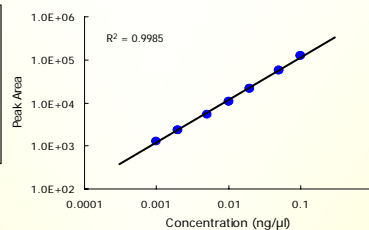


Fig. 4. Calibration curve of PCP-TMS

Table 2. Peak area 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

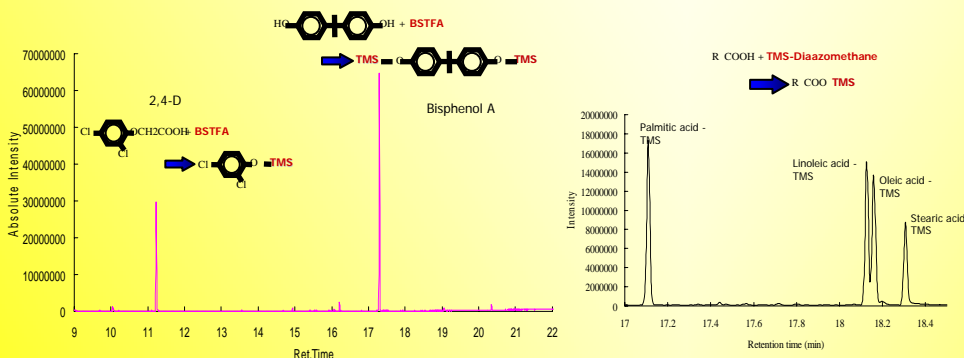


Fig. 5. Chromatogram of TMS-derivatized Bisphenol A and 2,4-Dichlorophenol (left) and fatty acids (right). BSTFA is used as their derivatization reagent.

[Results] Figure 3 is a chromatogram of PCP (A) and PCP-TMS (B). In chromatogram B, BSTFA is used as derivatization reagent and derivatization proceeds in the liner of LaviStoma injection system. Only PCP-TMS peak, and no PCP peak, is found in chromatogram B. This shows that PCP is fully derivatized into PCP-TMS. Good linearity of PCP-TMS between its concentration and peak area is obtained as shown in figure 4. Also good repeatability of the peak area of PCP-TMS is obtained as shown in Table 2. Moreover, Bisphenol A, 2,4-D and fatty acids are completely derivatized as shown in figure 5. In this case, BSTFA is used as derivatization reagent.

[Conclusion] As mentioned above, we can obtain results of derivatization analysis quite easily and we need no laborious preparation process of derivatization before sample injection. In conclusion, derivatization analysis can be performed quite easily, simply and quickly by using LaviStoma injection system.