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Study number	85538

TEST REPORT

Estimation of the adsorption coefficient on soil and on sewage sludge for 6:2 FTOH

This is a correct copy of the original.
Chemicals Evaluation and Research Institute,
Japan, Kurume (CERI Kurume)

Date *March 1, 2018*

Study Director

March, 2018

Chemicals Evaluation and Research Institute, Japan, Kurume

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1. Title

Estimation of the adsorption coefficient on soil and on sewage sludge for 6:2 FTOH

2. Sponsor

Name DAIKIN INDUSTRIES, LTD.

Address 1-1 Nishi Hitotsuya, Settsu-shi, Osaka 566-8585, Japan

3. Test facility

Name Chemicals Evaluation and Research Institute, Japan, Kurume (CERI Kurume)

Address 3-2-7 Miyanojin, Kurume-shi, Fukuoka 839-0801, Japan

4. Objective

This study was performed to obtain the adsorption coefficient on soil and on sewage sludge of 6:2 FTOH.

5. Test method

OECD Guidelines for the Testing of Chemicals, No. 121, January 22, 2001, "Estimation of the Adsorption Coefficient (K_{oc}) on Soil and on Sewage Sludge using High Performance Liquid Chromatography (HPLC)"

6. Dates

Study initiation date January 29, 2018

Study completion date March 1, 2018

7. Personnel

Study Director Hiroko Kawashima (Section 5)

Study personnel Hiroko Kawashima

8. Approval of test report

Date

March 1, 2018

Study Director

9. Summary

Test item

6:2 FTOH

Objective

This study was performed to obtain the adsorption coefficient on soil and on sewage sludge of 6:2 FTOH.

Test method

OECD Guidelines for the Testing of Chemicals, No. 121, January 22, 2001, "Estimation of the Adsorption Coefficient (K_{oc}) on Soil and on Sewage Sludge using High Performance Liquid Chromatography (HPLC)"

Test conditions

Test equipment	Liquid chromatograph-mass spectrometer (LC-MS)
Eluent	Methanol/ultrapure water (50/50 v/v)
Test temperature	25±1°C

Results

Adsorption coefficient of test item

$$\log K_{oc} = 2.39 (25^{\circ}\text{C})$$

10. Test materials

10.1 Test item

a) Chemical name etc.

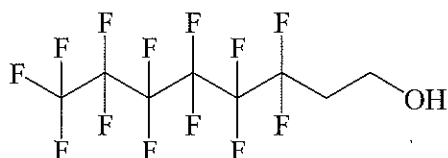
Chemical name 3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluoro-1-octanol

Another name 6:2 FTOH

CAS number 647-42-7

b) Chemical structure etc.

Structural formula

Molecular formula $C_8H_5F_{13}O$

Molecular weight 364.1

c) Test sample

Purity of test item 99.8%

Impurity Unknown components 0.2%

Supplier DAIKIN INDUSTRIES, LTD.

Lot number ST2AV75001

d) Physicochemical property

Appearance Colorless and clear liquid

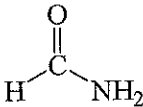
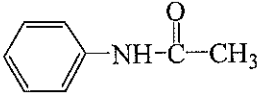
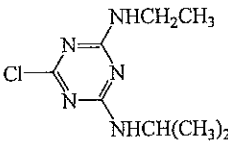
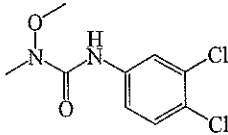
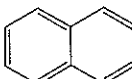
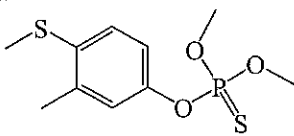
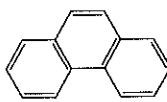
e) Storage conditions

The test sample was stored in a dark storage place at room temperature.

f) Safety and handling

In order to avoid inhalation and contact with the skin and eyes, chemically resistant gloves, a mask, safety glasses, and a white coat were worn when handling the test item.

10.2 Reference item

Name and CAS number	Structural formula	Supplier and grade	Purity (%)	log K _{oc}
Formamide (for dead time) 75-12-7		Wako Pure Chemical Industries JIS special grade	≥98.5	-
Acetanilide 103-84-4		Wako Pure Chemical Industries Wako special grade	≥99.0	1.25
Atrazine 1912-24-9		Tokyo Chemical Industry TCI-EP	>97.0	1.81
Linuron 330-55-2		Wako Pure Chemical Industries Trace Sure	99.8	2.59
Naphthalene 91-20-3		Kanto Chemical Extra pure	≥99	2.75
Fenthion 55-38-9		Sigma-Aldrich No grade	97.9	3.31
Phenanthrene 85-01-8		NACALAI TESQUE GUARANTEED REAGENT	98	4.09

11. Performance of test

11.1 Measurement conditions

a) Test equipment

Instrument	Liquid chromatograph-mass spectrometer (No. LCMS-012)	
LC system	Nexera X2	(Shimadzu)
Mass spectrometer	LCMS-8050	(Shimadzu)

Condition of liquid chromatograph

Column	Inertsil CN-3 (150 mm × 2.1 mm I.D., particle size 5 µm, GL Sciences)
Column temperature	25°C
Eluent	A (50%): Ultrapure water B (50%): Methanol
Flow rate	0.2 mL/min
Measurement wavelength	210 nm (measurement of reference item)
Injection volume	2 µL

Condition of mass spectrometer

Ionization mode	Electrospray ionization (ESI)
Detection ion	Negative
Detection mode	Selected ion monitoring (SIM)
Measurement ion (<i>m/z</i>)	362.9 ([M-H] ⁻)
Interface temperature	120°C
DL temperature	250°C
Nebulizer gas flow	1.5 L/min
Heating gas flow	10 L/min
Heat block temperature	400°C
Drying gas flow	10 L/min

b) Test temperature

25±1°C

11.2 Test procedures

a) Preparation of reference item solution

The reference item solution was prepared as follows.

Reference item	Weight (mg)	Volume (mL)	Solvent	Volume added (mL)	Final volume (mL)	Solvent
Formamide (for dead time: t_0)	500	20	Methanol	1	10	Purified water
Acetanilide	10	10		1		
Atrazine	10	10		0.5		
Linuron	2	2		0.5		
Naphthalene	10	20		0.5		
Fenthion	2	2		1		
Phenanthrene	10	10		0.5		

b) Preparation of test item solution

The test sample (24.20 mg) was put into a 20-mL volumetric flask, and then dissolved in methanol to prepare the stock solution of test item. The stock solution (0.5 mL) was pipetted into a 10-mL volumetric flask and filled up with the eluent of LC-MS analysis to prepare a test item solution. The eluent was used as a solvent blank.

c) Measurement of retention times for reference items, and making of regression line

The reference item solution was injected to the test equipment and the retention times of reference items were measured twice. The capacity factors (k) of reference items were calculated by the following equation.

A regression line was made by the method of least squares using the adsorption coefficient ($\log K_{oc}$) and the logarithmic values of the capacity factors for reference items. The capacity factor (k), the slope of regression equation (a) and the intercept of regression equation (b) were rounded off to three decimal places.

$$k = \frac{t_R - t_0}{t_0}$$

t_R : Retention time of reference item (min)

t_0 : Dead time (min) (average of two measured values)

$$\log K_{oc} = a \times \log k + b$$

a : Slope of regression equation

b : Intercept of regression equation

d) Measurement of retention time for test item

The test item solution was injected to the test equipment and the retention time of the test item was measured twice. The solvent blank was injected once to the test equipment. It was confirmed that no peak existed at the peak position of test item by the analysis of solvent blank.

e) Injection order

The reference item solution was injected before and after the injection of the test item solution to confirm that the retention times of reference item did not change.

Injection order:

Reference item solution (first time) → Test item solution (first time) →

Test item solution (second time) → Solvent blank → Reference item solution (second time)

11.3 Calculation of adsorption coefficient

The capacity factor was calculated from the retention time of test item. The adsorption coefficient of test item was then calculated using the regression equation of regression line and was given as the average value of two calculated values.

11.4 Treatment of numerical values

The adsorption coefficient was represented as logarithm and rounded off to two decimal places.

Values were treated in accordance with Japanese Industrial Standards (JIS) Z 8401:1999 rule B.

12. Results and discussion

12.1 Adsorption coefficient of test item

The test results are shown as follows (see Table 1).

log K _{oc}		
Measured value		Average
2.39	2.39	2.39

12.2 Discussion

The average of measured log K_{oc} values was 2.39 and the difference between two measured values was 0.00. It is judged that the test results are valid because the difference between two measured values was less than 0.25.

Table 1 Calculation table for adsorption coefficient by HPLC method

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		t_R	k	$\log k$	$\log K_{oc}$
Reference item	1-a	2.09	$t_0 =$	2.09	
	1-b	2.09			
	2-a	3.28	0.569	-0.245	1.25
	2-b	3.28	0.569	-0.245	1.25
	3-a	4.26	1.038	0.016	1.81
	3-b	4.26	1.038	0.016	1.81
	4-a	8.50	3.067	0.487	2.59
	4-b	8.47	3.053	0.485	2.59
	5-a	11.92	4.703	0.672	2.75
	5-b	11.89	4.689	0.671	2.75
	6-a	16.44	6.866	0.837	3.31
	6-b	16.43	6.861	0.836	3.31
	7-a	30.87	13.770	1.139	4.09
	7-b	30.96	13.813	1.140	4.09
Test item	a	6.85	2.278	0.357	2.39
	b	6.85	2.278	0.357	2.39
(a,b : individual sample)				Average	2.39

$$k = (t_R - t_0) / t_0$$

$$t_0 : \text{Dead time (average of two measured values) (min)}$$

$$t_R : \text{Retention time (min)}$$

$$\log K_{oc} = 1.948 \times \log k + 1.690$$

$$r = 0.990$$

Reference item

1	Formamide
2	Acetanilide
3	Atrazine
4	Linuron
5	Naphthalene
6	Fenthion
7	Phenanthrene

See Figs. 1,2

January 31, 2018

Name _____

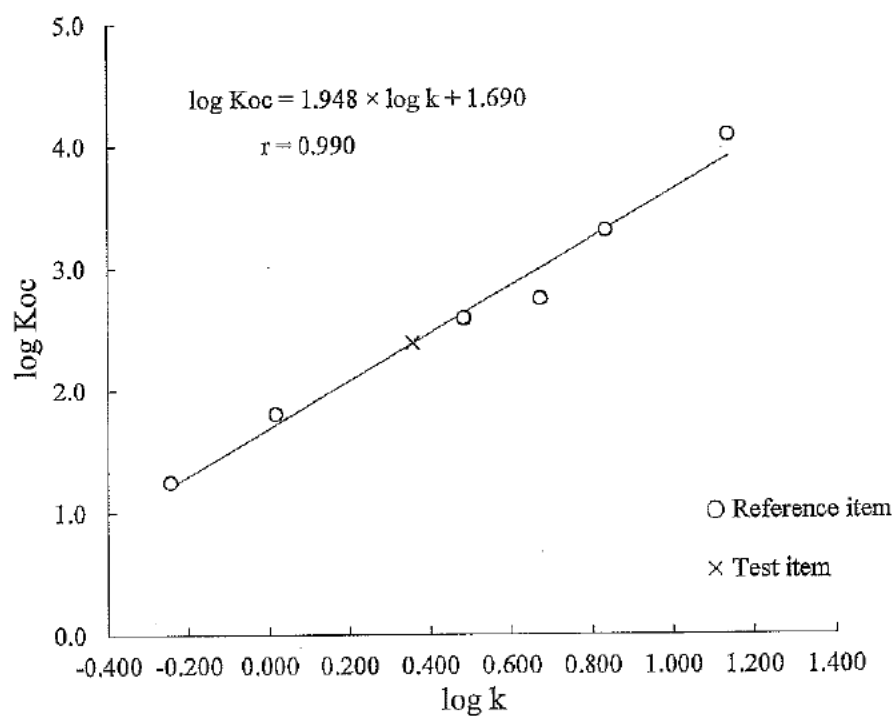


Fig. 1 Calibration curve for adsorption coefficient by HPLC method.

January 31, 2018

Name

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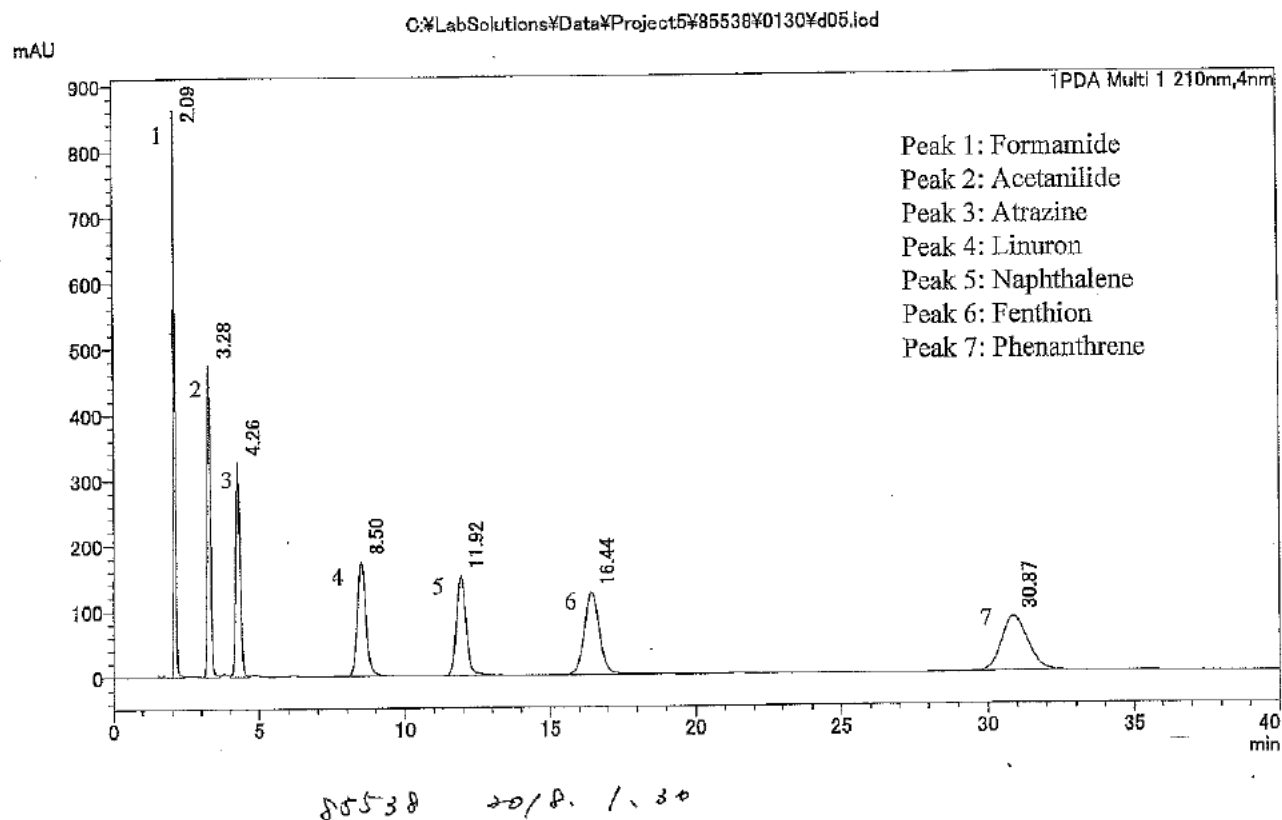


Fig. 2 - 1 Chromatogram of LC-MS analysis for partition coefficient by HPLC method.

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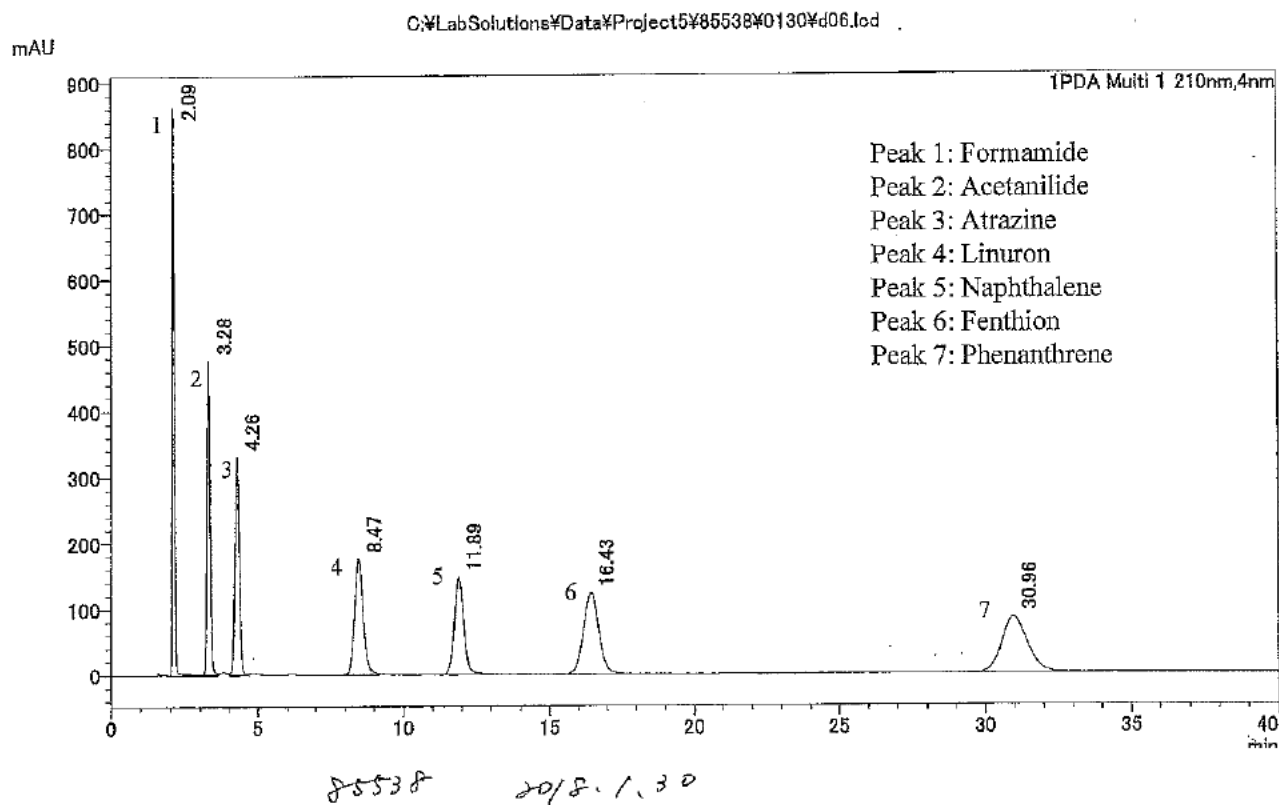
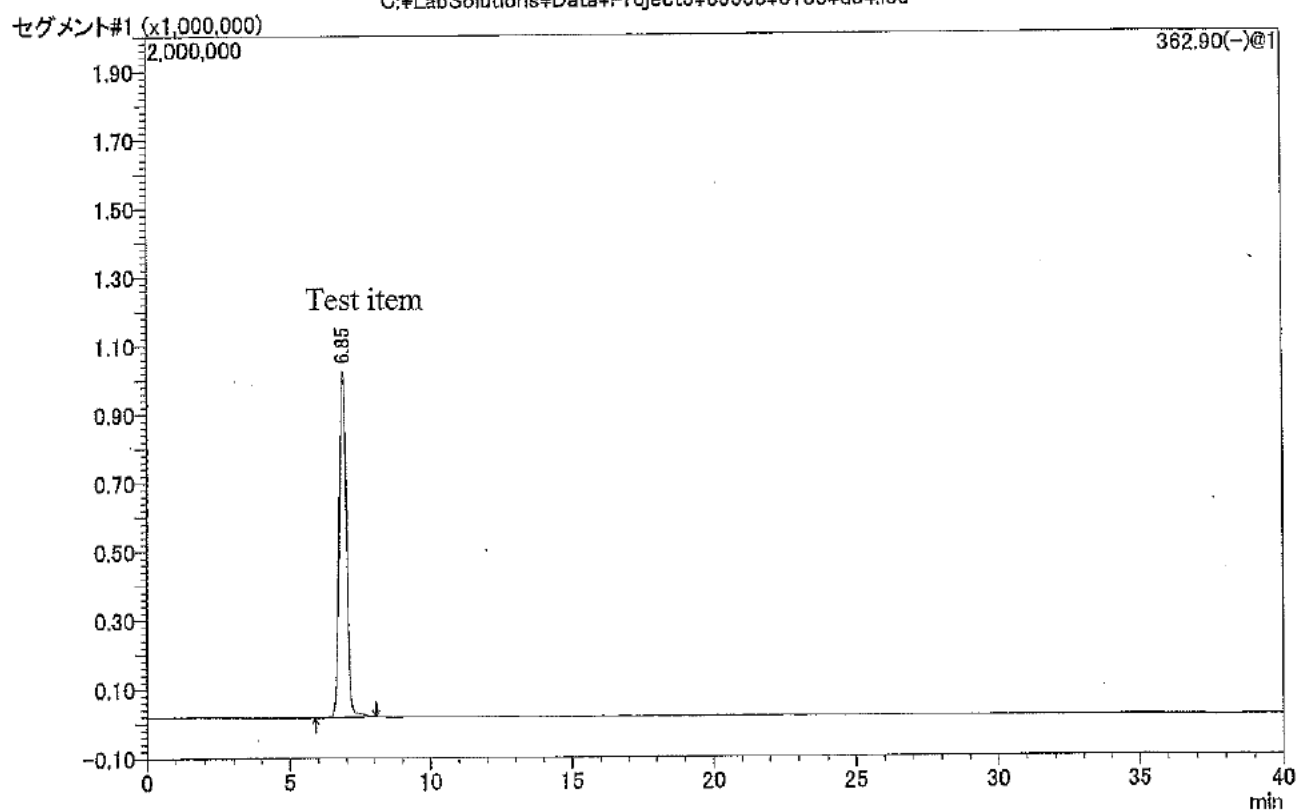


Fig. 2 - 2 Chromatogram of LC-MS analysis for partition coefficient by HPLC method.

分析日 : 2018/01/30
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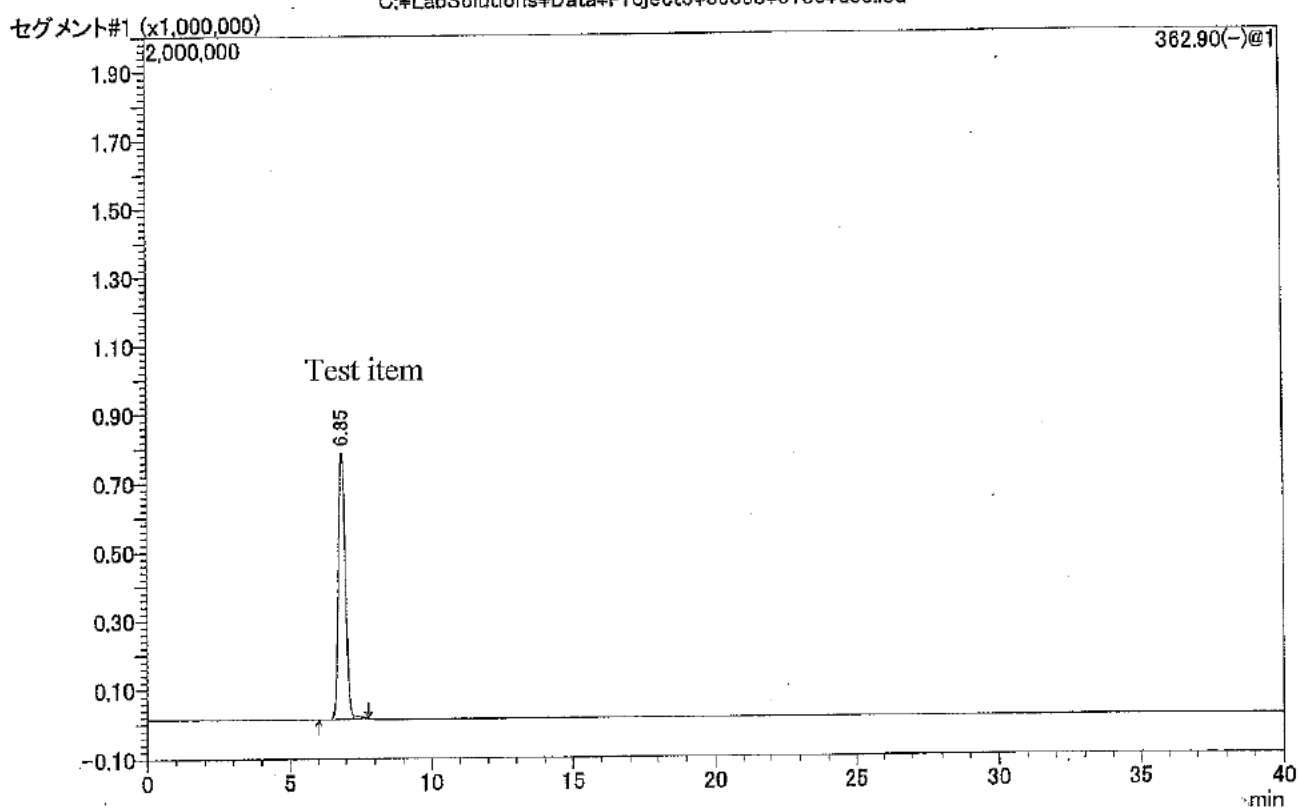


85538 2018.1.30

Fig. 2 - 3 Chromatogram of LC-MS analysis for partition coefficient by HPLC method.

分析日 : 2018/01/30
サンプル名 : Test item - b
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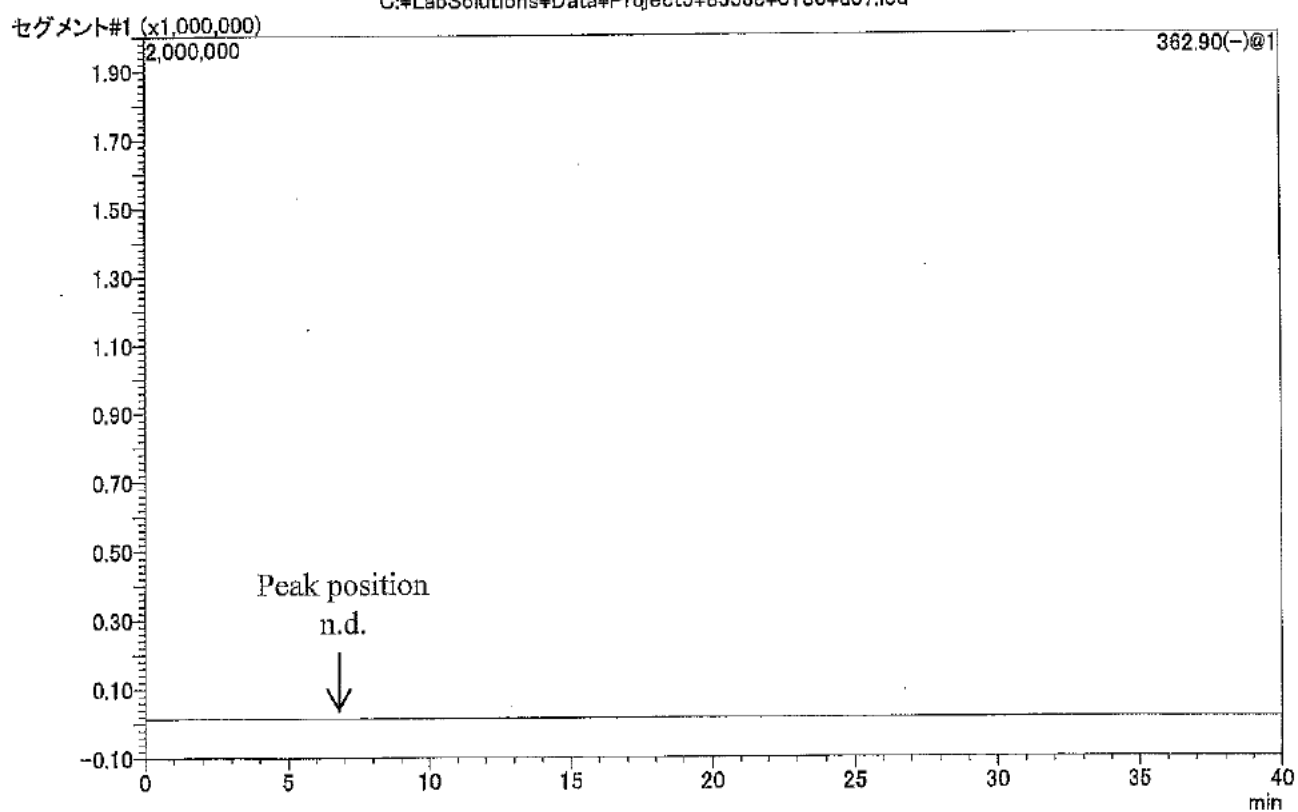


85538 2018.1.30

Fig. 2 - 4 Chromatogram of LC-MS analysis for partition coefficient by HPLC method.

分析日 : 2018/01/30
サンプル名 : Solvent
データファイル : d07.lcd

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Fig. 2 - 5 Chromatogram of LC-MS analysis for partition coefficient by HPLC method.