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Study number	85532

TEST REPORT

Estimation of the adsorption coefficient on soil and on sewage sludge for PFHxA

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

Date *March 15, 2018*

Study Director / *N. Kawashima*

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 PFHxA

2. Sponsor

Name DAIKIN INDUSTRIES, LTD.

Address 1-1, Nishihitotsuya, 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 PFHxA.

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 15, 2018

7. Personnel

Study Director Hiroko Kawashima (Section 5)

Study personnel Tomoko Nakayoshi

8. Approval of test report

Date

March 15, 2018

Study Director

H. Kawashima

Hiroko Kawashima

9. Summary

Test item

PFHxA

Objective

This study was performed to obtain the adsorption coefficient on soil and on sewage sludge of PFHxA.

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	High-performance liquid chromatograph (HPLC)
Eluent	Analysis for undissociated form: Methanol/buffer solution (pH2.0)*1 (6/4 v/v) Analysis for dissociated form : Methanol/buffer solution (pH7.0)*1 (6/4 v/v)
Test temperature	25±1°C

*1 The test item is a dissociative substance ($pK_a = 3.29$). Therefore, this test was performed at pH2.0 and pH7.0. The test item has an undissociated form in pH2.0 and an dissociated form in pH7.0.

Results

Adsorption coefficient of test item

$\log K_{oc} = 4.27$	(25°C, undissociated form at pH2.0)
$\log K_{oc} < 1.25$	(25°C, dissociated form at pH7.0)

10. Test materials

10.1 Test item

a) Chemical name etc.

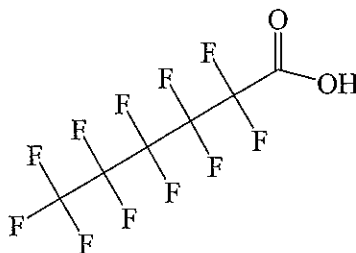
Chemical name (2,2,3,3,4,4,5,5,6,6,6-undecafluorohexanoic acid)

Another name PFHxA

CAS number 307-24-4

b) Chemical structure etc.

Structural formula



Molecular formula $C_6HF_{11}O_2$

Molecular weight 314.05

c) Test sample

Purity of test item 99.8%

Impurity Water 0.2%

Supplier DAIKIN INDUSTRIES, LTD.

Lot number T1221

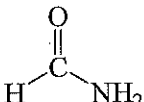
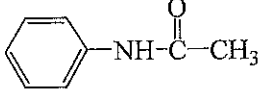
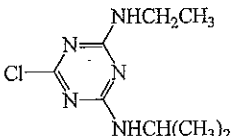
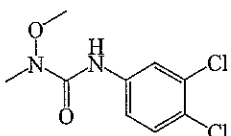
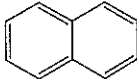
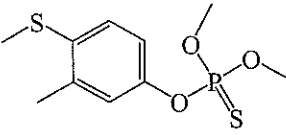
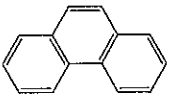
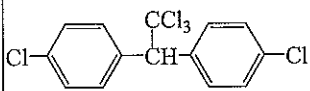
d) Physicochemical property

Appearance White powder

e) Storage conditions

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

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
DDT 50-29-3		Tokyo Chemical Industry TCI-EP	≥98	5.63

11. Performance of test

The test item is a dissociative substance ($pK_a = 3.29$) [refer to the test report "Measurement of dissociation constants in water for PFHxA" (Study No. 85531)]. Therefore, this test was performed at pH2.0 and pH7.0. The test item has an undissociated form in pH2.0 and an dissociated form in pH7.0.

11.1 Test conditions

a) Test equipment

Instrument	High-performance liquid chromatograph (No. LC-147)
Pump	LC-20AD (Shimadzu)
UV-VIS detector	SPD-20A (Shimadzu)
Column oven	CTO-20AC (Shimadzu)
Auto injector	SIL-20A _{HT} (Shimadzu)
Degasser	DGU-20A ₃ (Shimadzu)
Column	Inertsil CN-3 (150 mm × 2.1 mm I.D., particle size 5 µm, GL Sciences)
Column temperature	25°C
Eluent	Analysis for undissociated form Methanol/buffer solution (pH2.0) * ² (6/4 v/v) Analysis for dissociated form Methanol/buffer solution (pH7.0) * ³ (6/4 v/v)
Flow rate	0.2 mL/min
Measurement wavelength	210 nm
Injection volume	2 µL

*2 10 mmol/L potassium dihydrogen phosphate solution was adjusted to pH 2.0 by phosphoric acid.

*3 10 mmol/L potassium dihydrogen phosphate solution was adjusted to pH 7.0 by 1 mol/L sodium hydroxide solution.

b) Test temperature

25±1°C

11.2 Test procedures

a) Preparation of reference item solution

The reference item solutions for analysis of undissociated form and dissociated form were prepared as follows, respectively.

Reference item	Weight (mg)	Volume (mL)	Solvent	Volume added (mL)	Final volume (mL)	Solvent
Formamide (for dead time: t_0)	500	20	Methanol	1	20	Eluent (pH2.0) or Eluent (pH7.0) (see Section 11.1)
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		
DDT	10	10	Acetonitrile	2		

b) Preparation of test item solution

1) Analysis for undissociated form

The test sample (20.4 mg) was weighed with an electronic analytical balance and dissolved in the eluent (pH2.0) for HPLC analysis to prepare the test item solution. The eluent (pH2.0) for HPLC analysis was used as a solvent blank.

2) Analysis for dissociated form

The test sample (20.1 mg) was weighed with an electronic analytical balance and dissolved in the eluent (pH7.0) for HPLC analysis to prepare the test item solution. The eluent (pH7.0) for HPLC analysis 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.

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 place. 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.

	log K_{oc}			Table
	Measured value		Average	
Undissociated form (pH2.0)	4.27	4.27	4.27	1
Dissociated form (pH7.0)	<1.25	<1.25	<1.25	2

12.2 Discussion

The average of measured log K_{oc} values was 4.27 (pH 2.0) and <1.25 (pH7.0). 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 (pH2.0)

Study No. 85532																					
		t_R	k	log k	log Koc																
Reference item	1-a	2.46	$t_0 =$	2.47																	
	1-b	2.47																			
	2-a	3.12	0.266	-0.576		1.25															
	2-b	3.13	0.270	-0.569		1.25															
	3-a	3.49	0.416	-0.381		1.81															
	3-b	3.51	0.424	-0.373		1.81															
	4-a	4.97	1.016	0.007		2.59															
	4-b	4.99	1.024	0.010		2.59															
	5-a	6.28	1.548	0.190		2.75															
	5-b	6.30	1.556	0.192		2.75															
	6-a	6.84	1.775	0.249		3.31															
	6-b	6.86	1.783	0.251		3.31															
	7-a	11.06	3.487	0.542		4.09															
	7-b	11.06	3.487	0.542		4.09															
	8-a	20.45	7.296	0.863		5.63															
	8-b	20.54	7.333	0.865		5.63															
Test item	a	11.24	3.560	0.551	4.27																
	b	11.24	3.560	0.551	4.27																
(a,b : individual sample)				Average	4.27																
<p> $k = (t_R - t_0) / t_0$ t_0 : Dead time (average of two measured values) (min) t_R : Retention time (min) </p> <p> $\log Koc = 2.871 \times \log k + 2.689$ $r = 0.977$ </p> <p> Reference item </p> <table style="margin-left: 20px;"> <tr><td>1</td><td>Formamide</td></tr> <tr><td>2</td><td>Acetanilide</td></tr> <tr><td>3</td><td>Atrazine</td></tr> <tr><td>4</td><td>Linuron</td></tr> <tr><td>5</td><td>Naphthalene</td></tr> <tr><td>6</td><td>Fenthion</td></tr> <tr><td>7</td><td>Phenanthrene</td></tr> <tr><td>8</td><td>DDT</td></tr> </table> <p>See Figs. 1,2</p>						1	Formamide	2	Acetanilide	3	Atrazine	4	Linuron	5	Naphthalene	6	Fenthion	7	Phenanthrene	8	DDT
1	Formamide																				
2	Acetanilide																				
3	Atrazine																				
4	Linuron																				
5	Naphthalene																				
6	Fenthion																				
7	Phenanthrene																				
8	DDT																				

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Table 2 Calculation table for adsorption coefficient by HPLC method (pH7.0)

Study No. 85532

		t_R	k	log k	log Koc																
Reference item	1-a	2.53	$t_0 =$	2.53																	
	1-b	2.53																			
	2-a	3.11	0.229	-0.640	1.25																
	2-b	3.11	0.229	-0.640	1.25																
	3-a	3.33	0.316	-0.500	1.81																
	3-b	3.34	0.320	-0.495	1.81																
	4-a	4.56	0.802	-0.096	2.59																
	4-b	4.58	0.810	-0.091	2.59																
	5-a	5.77	1.281	0.107	2.75																
	5-b	5.78	1.285	0.109	2.75																
	6-a	5.85	1.312	0.118	3.31																
	6-b	5.89	1.328	0.123	3.31																
	7-a	9.40	2.715	0.434	4.09																
	7-b	9.46	2.739	0.438	4.09																
	8-a	15.90	5.285	0.723	5.63																
	8-b	16.02	5.332	0.727	5.63																
Test item	a	1.86	-	-	<1.25																
	b	1.86	-	-	<1.25																
	(a,b : individual sample)				Average	<1.25															
<p> $k = (t_R - t_0) / t_0$ t_0 : Dead time (average of two measured values) (min) t_R : Retention time (min) </p> <p> $\log Koc = 2.944 \times \log k + 2.995$ $r = 0.972$ </p> <p> Reference item </p> <table> <tr><td>1</td><td>Formamide</td></tr> <tr><td>2</td><td>Acetanilide</td></tr> <tr><td>3</td><td>Atrazine</td></tr> <tr><td>4</td><td>Linuron</td></tr> <tr><td>5</td><td>Naphthalene</td></tr> <tr><td>6</td><td>Fenthion</td></tr> <tr><td>7</td><td>Phenanthrene</td></tr> <tr><td>8</td><td>DDT</td></tr> </table> <p>See Figs. 3,4</p>						1	Formamide	2	Acetanilide	3	Atrazine	4	Linuron	5	Naphthalene	6	Fenthion	7	Phenanthrene	8	DDT
1	Formamide																				
2	Acetanilide																				
3	Atrazine																				
4	Linuron																				
5	Naphthalene																				
6	Fenthion																				
7	Phenanthrene																				
8	DDT																				

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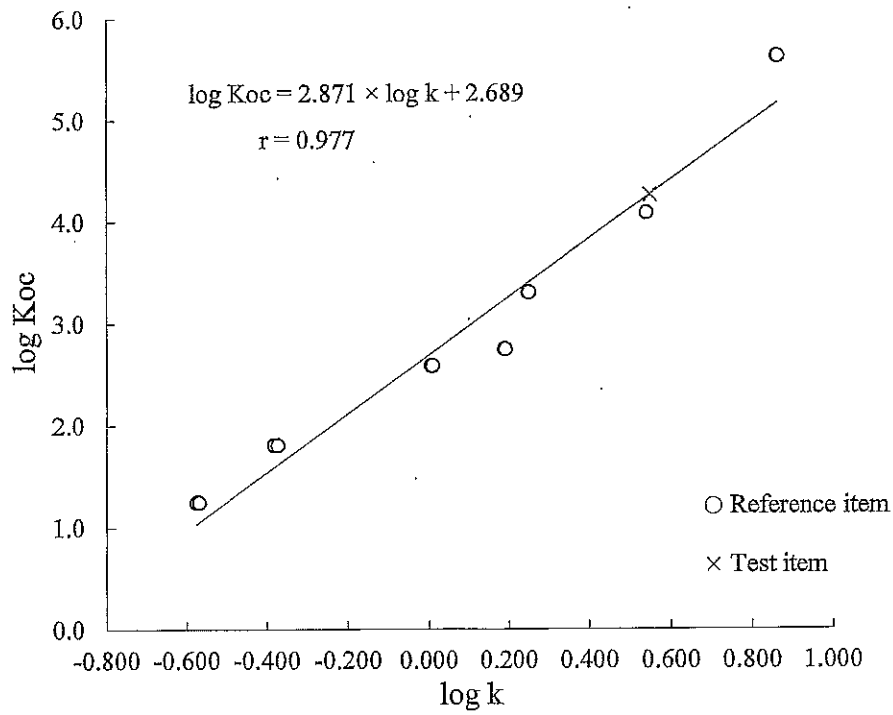


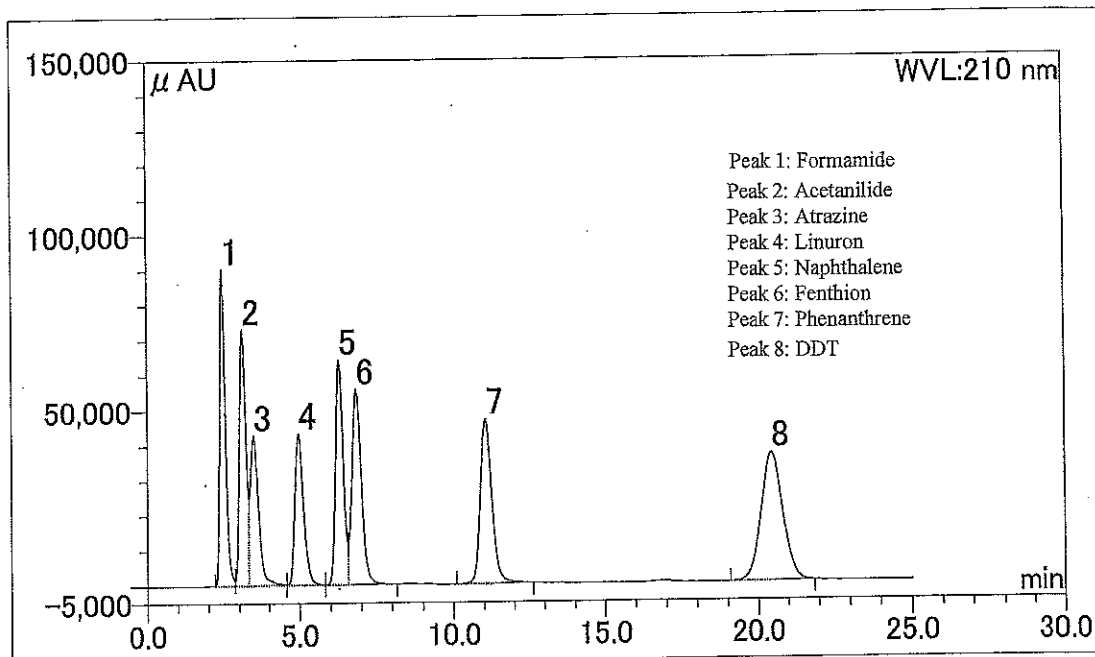
Fig. 1 Calibration curve for adsorption coefficient by HPLC method (pH2.0).

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Name T. Nakayoshi

Reference item - a (pH2.0)

Operator:	Tomoko Nakayoshi
Operating date:	27/Feb/2018
Sample ID:	85532_85545_180227_pH2_12
Program:	85532,85545_pg25_6;4
Vial No.:	1_9
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	2.46	90312	1137321	12.24
2	3.12	73089	1106251	11.90
3	3.49	42778	820154	8.83
4	4.97	43183	844703	9.09
5	6.28	64045	1100019	11.84
6	6.84	55683	1214363	13.07
7	11.06	46939	1239332	13.34
8	20.45	36521	1830706	19.70
Total	-	-	9292848	100.00

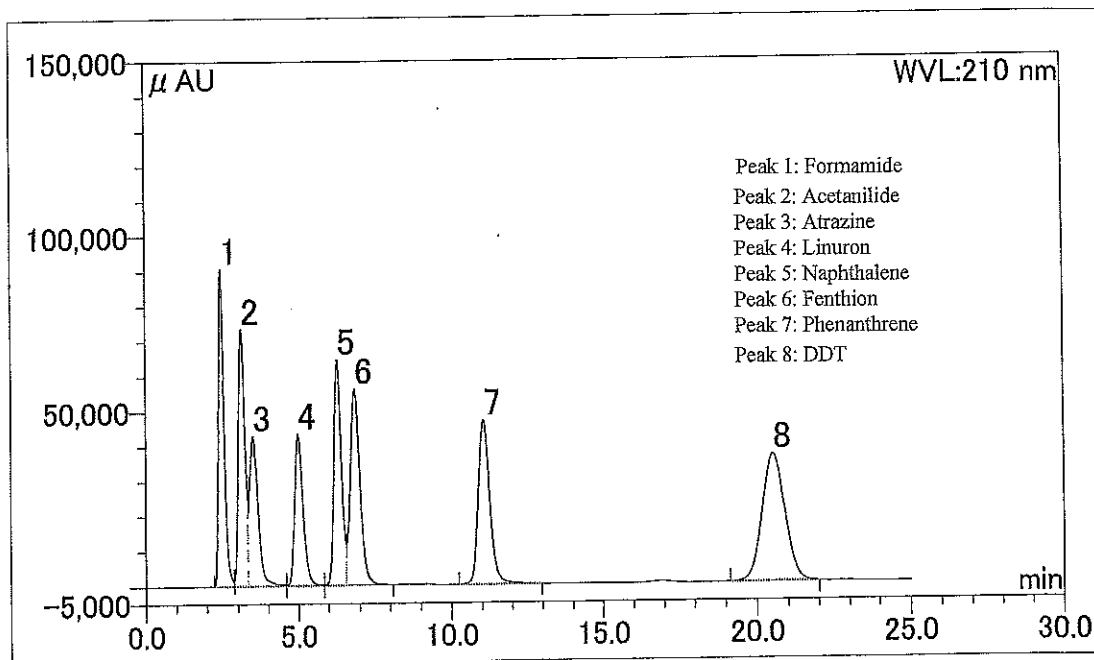


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Fig. 2 - 1 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH2.0).

Reference item - b (pH2.0)

Operator:	Tomoko Nakayoshi
Operating date:	27/Feb/2018
Sample ID:	85532_85545_180227_pH2_13
Program:	85532,85545_pg25_6;4
Vial No.:	1_9
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
1	2.47	90616	1139153	12.22
2	3.13	73370	1109358	11.90
3	3.51	42869	820424	8.80
4	4.99	43385	847604	9.09
5	6.30	64377	1102359	11.83
6	6.86	56041	1215363	13.04
7	11.06	46941	1243448	13.34
8	20.54	36417	1844308	19.78
Total	-	-	9322018	100.00

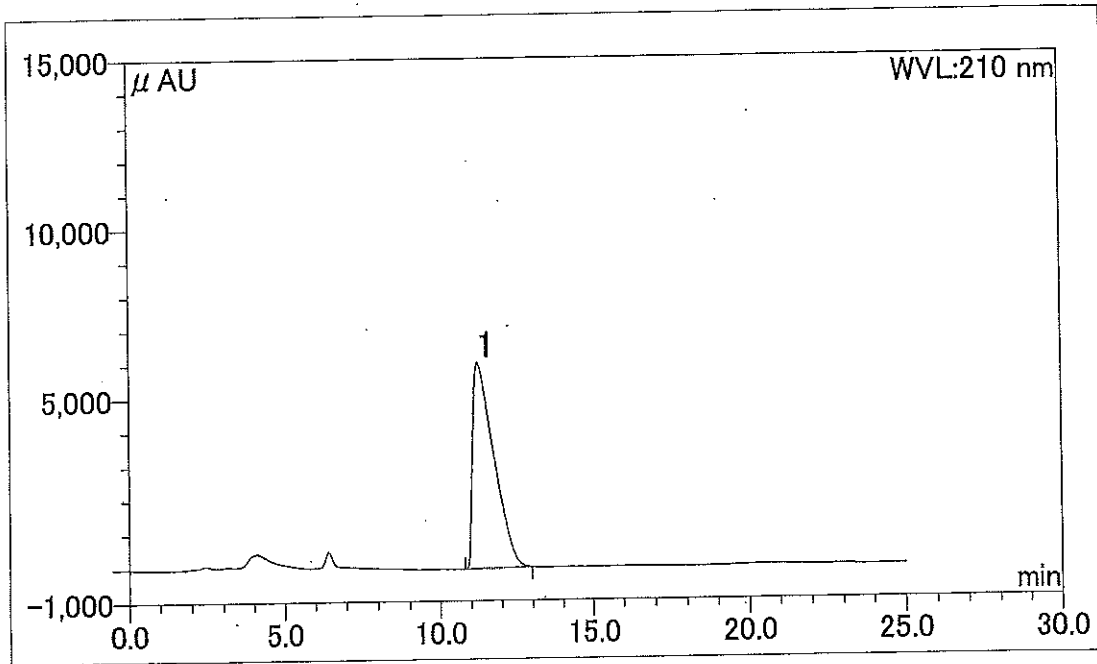


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Fig. 2 - 2 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH2.0).

Test item - a (pH2.0)

Operator:	Tomoko Nakayoshi
Operating date:	27/Feb/2018
Sample ID:	85532_85545_180227_pH2_9
Program:	85532,85545_pg25_6;4
Vial No.:	1_14
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	11.24	6074	288807	100.00
Total	-	-	288807	100.00

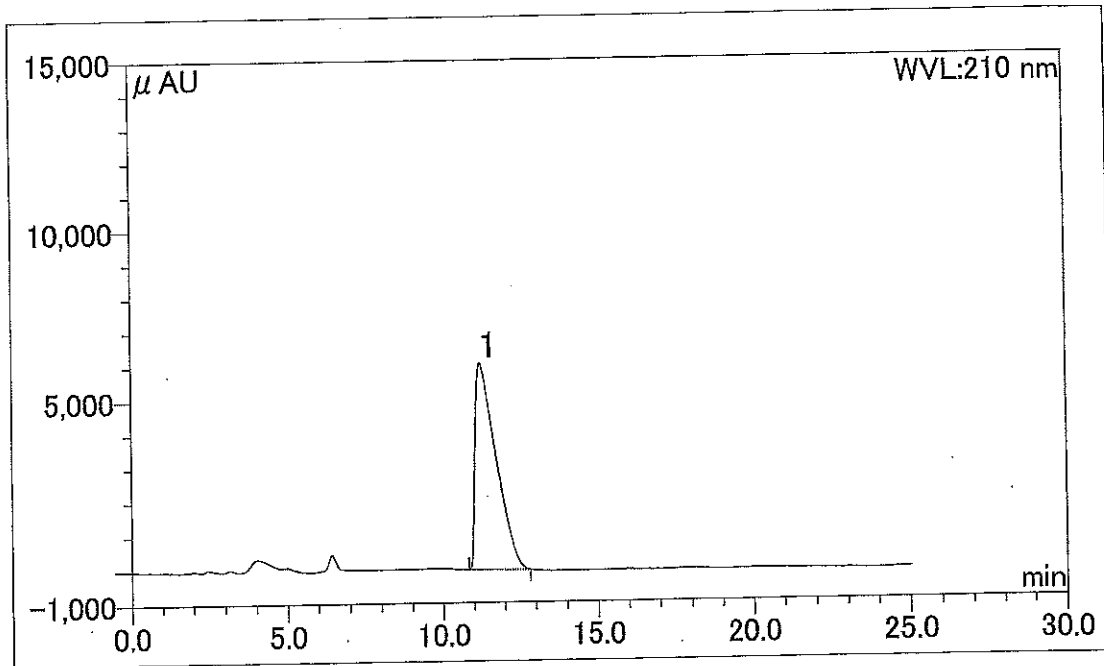


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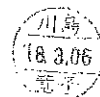
Fig. 2 - 3 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH2.0).

Test item - b (pH2.0)

Operator:	Tomoko Nakayoshi
Operating date:	27/Feb/2018
Sample ID:	85532_85545_180227_pH2_10
Program:	85532,85545_pg25_6;4
Vial No.:	1_14
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	11.24	6091	286939	100.00
Total	-	-	286939	100.00

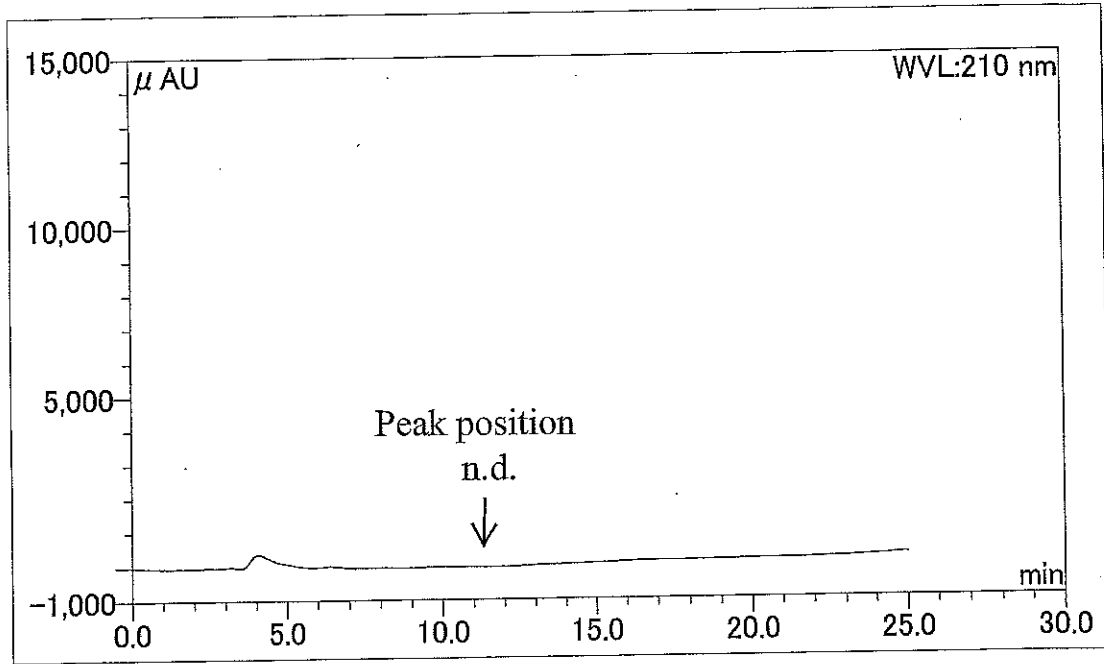


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Fig. 2 - 4 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH2.0).

Solvent blank(pH2.0)

Operator:	Tomoko Nakayoshi
Operating date:	27/Feb/2018
Sample ID:	85532_85545_180227_pH2_11
Program:	85532,85545_pg25_6;4
Vial No.:	1_13
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
Total	-	-	0	0.00



2018.2.27 T. Nakayoshi

Fig. 2 - 5 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH2.0).

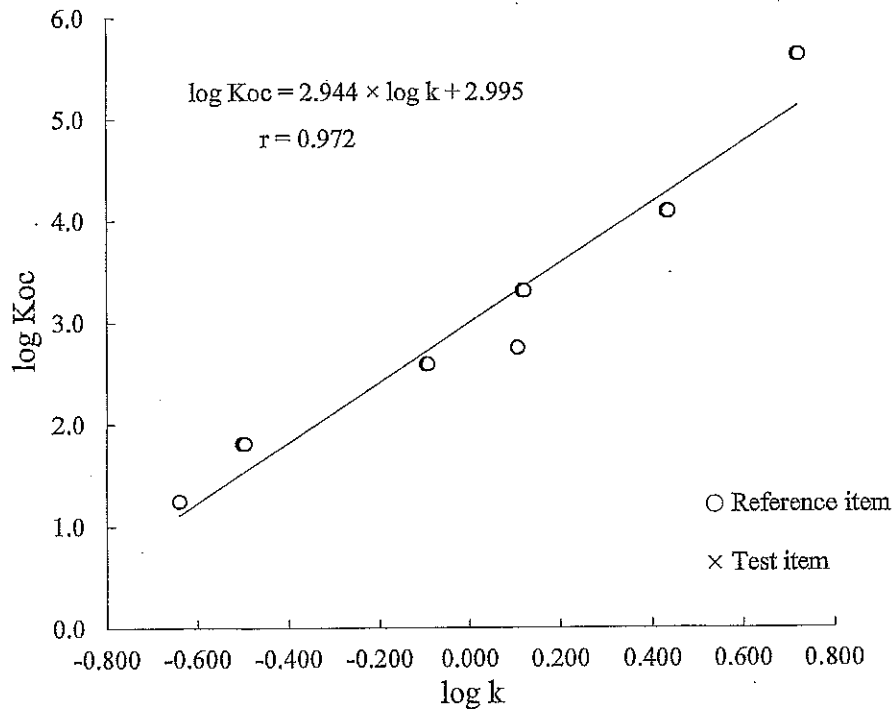


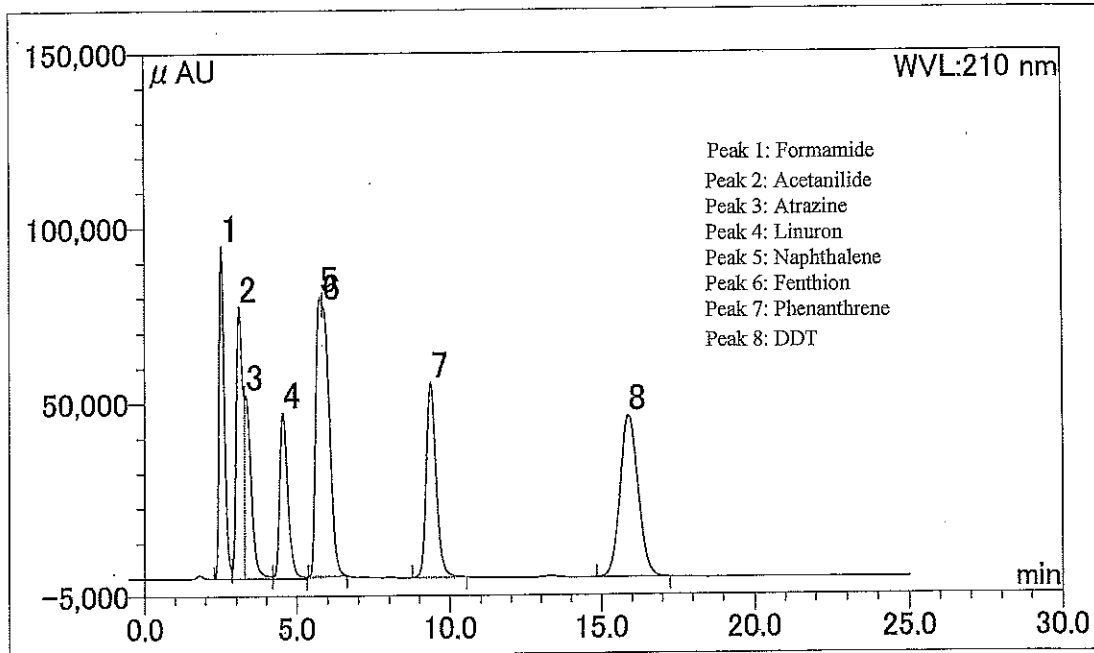
Fig. 3 Calibration curve for adsorption coefficient by HPLC method (pH7.0).

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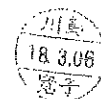
Name 7. Nakayoshi

Reference item - a (pH7.0)

Operator:	Tomoko Nakayoshi
Operating date:	28/Feb/2018
Sample ID:	85532_85545_180228_pH7_3
Program:	85532,85545_pg25_6;4
Vial No.:	1_15
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	2.53	94779	1156906	12.11
2	3.11	77498	1206444	12.63
3	3.33	52296	777320	8.14
4	4.56	47198	885080	9.26
5	5.77	79608	1106707	11.58
6	5.85	77430	1233347	12.91
7	9.40	55571	1287296	13.47
8	15.90	46118	1901324	19.90
Total	-	-	9554425	100.00

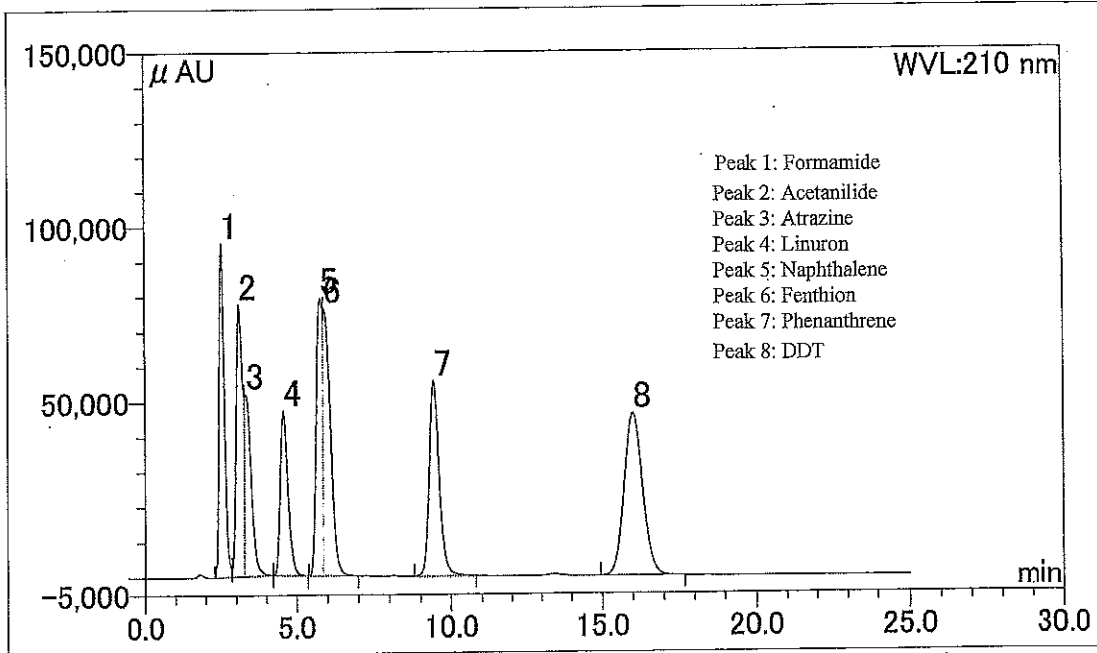


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Fig. 4 - 1 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH7.0).

Reference item - b (pH7.0)

Operator: Tomoko Nakayoshi
 Operating date: 28/Feb/2018
 Sample ID: 85532_85545_180228_pH7_4
 Program: 85532,85545_pg25_6;4
 Vial No.: 1_15
 Channel: UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	2.53	95139	1157125	12.12
2	3.11	77578	1157572	12.12
3	3.34	51895	802713	8.40
4	4.58	46800	855104	8.95
5	5.78	78989	1178442	12.34
6	5.89	76278	1190190	12.46
7	9.46	55589	1300852	13.62
8	16.02	46206	1908954	19.99
Total	-	-	9550952	100.00

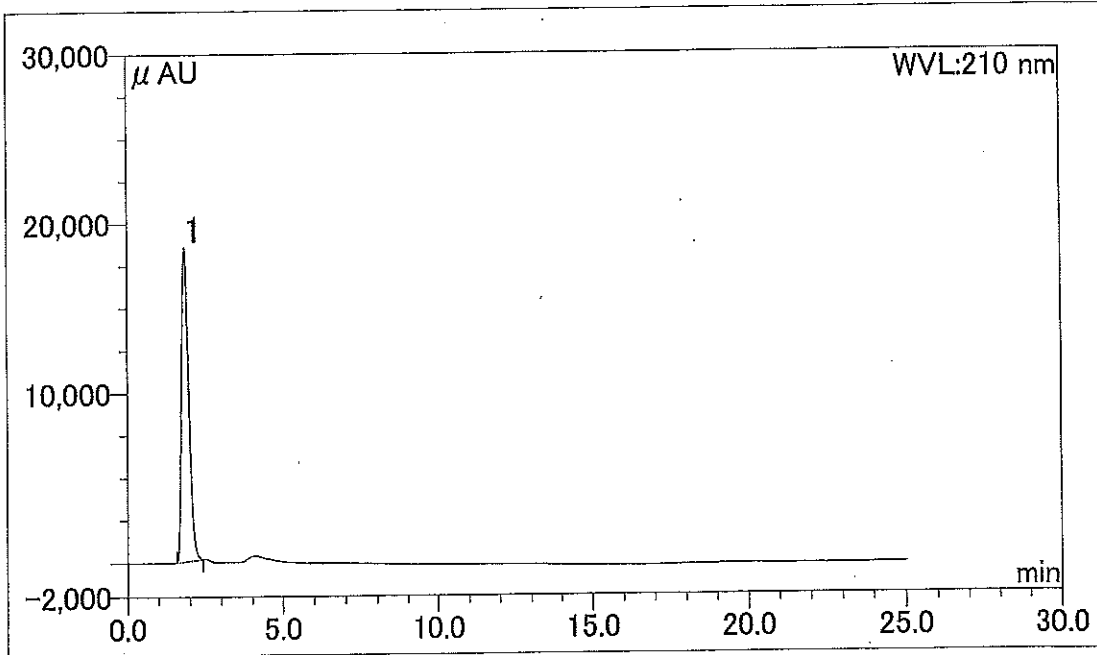


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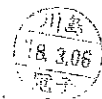
Fig. 4 - 2 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH7.0).

Test item - a (pH7.0)

Operator:	Tomoko Nakayoshi
Operating date:	28/Feb/2018
Sample ID:	85532_85545_180228_pH7_5
Program:	85532,85545_pg25_6;4
Vial No.:	1_16
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	1.86	18523	296609	100.00
Total	-	-	296609	100.00

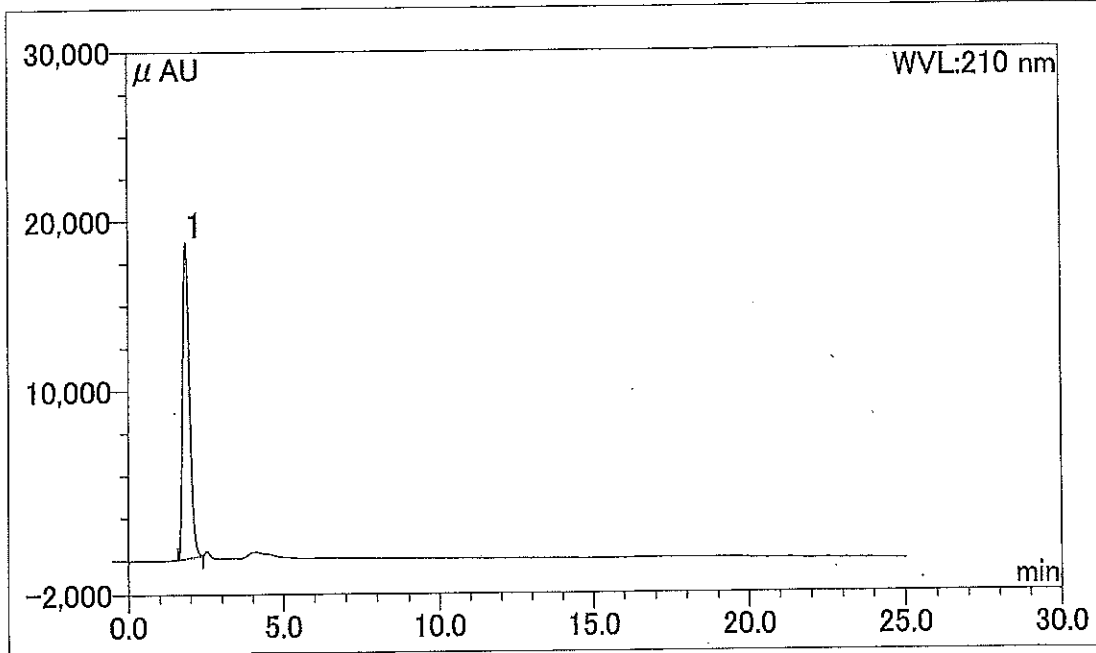


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Fig. 4 - 3 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH7.0).

Test item - b (pH7.0)

Operator:	Tomoko Nakayoshi
Operating date:	28/Feb/2018
Sample ID:	85532_85545_180228_pH7_6
Program:	85532,85545_pg25_6;4
Vial No.:	1_16
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	1.86	18574	295583	100.00
Total	-	-	295583	100.00

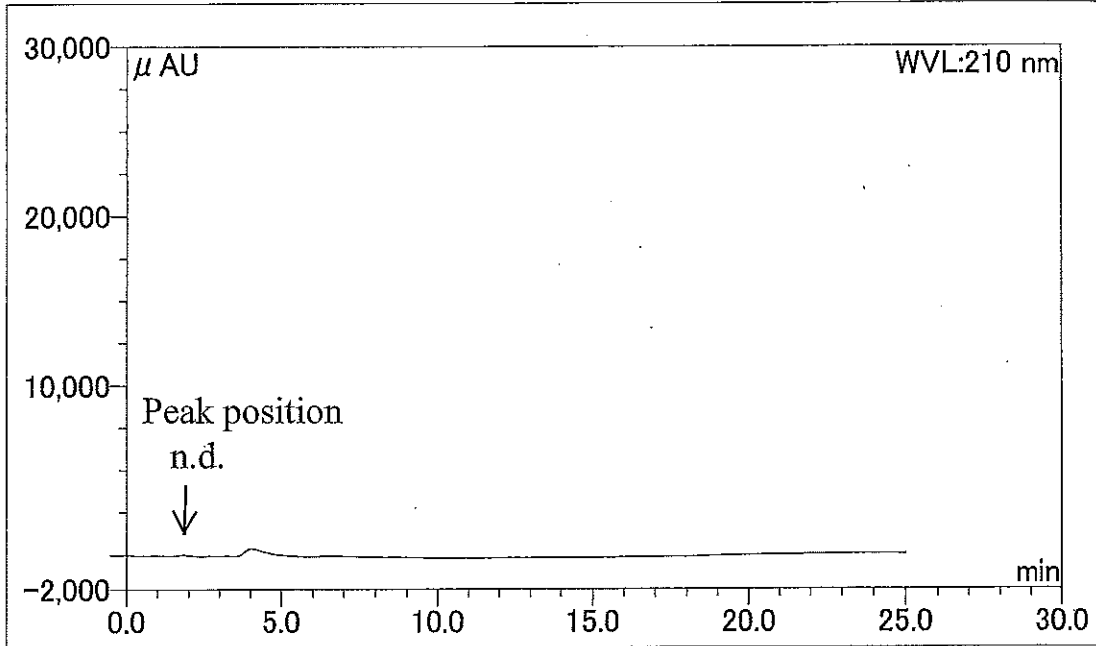


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Fig. 4 - 4 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH7.0).

Solvent blank(pH7.0)

Operator:	Tomoko Nakayoshi
Operating date:	28/Feb/2018
Sample ID:	85532_85545_180228_pH7_7
Program:	85532,85545_pg25_6;4
Vial No.:	1_17
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μAU)	Area (μAU·sec)	Area (%)
Total	-	-	0	0.00



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Fig. 4 - 5 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method (pH7.0).