Receipt number	652-16-P-5000
Study number	85000

FINAL REPORT

Measurement of adsorption coefficient on soil for C6OLF

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

Date March 21, 2017

Study Director

March, 2017

Chemicals Evaluation and Research Institute, Japan, Kurume

GLP STATEMENT

Chemicals Evaluation and
Research Institute, Japan, Kurume

Sponsor	DAIKIN INDUSTRIES, LTD.
Title	Measurement of adsorption coefficient on soil for C6OLF
Study number	85000

The study described in this report was conducted in compliance with the following GLP principles:
OECD Principles of Good Laboratory Practice, November 26, 1997, ENV/MC/CHEM (98)17

This final report reflects the raw data accurately and it has been confirmed that the test data are valid.

Date March 21, 2017

Study Director

QUALITY ASSURANCE STATEMENT

Chemicals Evaluation and Research Institute, Japan, Kurume

Sponsor: DAIKIN INDUSTRIES, LTD.

Title: Measurement of adsorption coefficient on soil for C6OLF

Study number: 85000

I assure that the final report accurately describes the test methods and procedures, and that the reported results accurately reflect the raw data of the study.

The inspections of this study were carried out and the results were reported to the Study Director and the Test Facility Management by Quality Assurance Unit as follows.

Item of inspection	Date of inspection	Date of report
Study plan	March 3, 2017	March 3, 2017
Test conduct	March 6, 2017	March 6, 2017
Study plan amendment No.1	March 13, 2017	March 13, 2017
Raw data and draft final report	March 16, 2017	March 16, 2017
Final report	March 21, 2017	March 21, 2017

Date March 21, 2017

Personnel of Quality Assurance Unit:

CONTENTS

1. Title

Measurement of adsorption coefficient on soil for C6OLF

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 test is performed to obtain the adsorption coefficient on soil of C6OLF.

5. Test method

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

6. GLP principle

OECD Principles of Good Laboratory Practice, November 26, 1997, ENV/MC/CHEM (98)17

7. Dates

Study initiation date	March 1, 2017
Experimental starting date	March 6, 2017
Experimental completion date	March 6, 2017
Study completion date	March 21, 2017

8. Storage of test item, raw data, etc.

The study plan (original), the final report (original), raw data, documents about the study presented by the sponsor and necessary materials will be stored in archives at this laboratory for 10 years from study completion date. Treatment of raw data, etc. after the storage period will be discussed with the sponsor. The test item will be returned to the sponsor.

9. Personnel

Study Director
Study personnel

10. Approval of final report

Date March 21, 2017

Study Director

11. Summary

Test item

C6OLF

Objective

This test is performed to obtain the adsorption coefficient on soil of C6OLF.

Test methods

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

Test conditions

Test equipment	High-performance liquid chromatograph (HPLC) Eluent: Methanol/purified water (60/40 v/v)
Test temperature	25±1°C

Results

The adsorption coefficient (log Koc) of C6OLF was 2.65 at 25°C.

12. Test materials

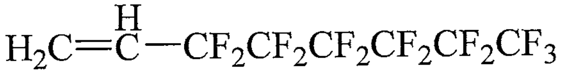
12.1 Test item

a) Chemical name etc.

Chemical name	3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluorooct-1-ene
Another name	C6OLF
CAS number	25291-17-2

b) Chemical structure etc.

Structural formula



$$H_2C{=}\overset{H}{C}{-}CF_2CF_2CF_2CF_2CF_2CF_3$$

Molecular formula	$C_8H_3F_{13}$
Molecular weight	346.09

c) Test sample

Purity of test item	99.95%
Impurity	Unknown 0.05%
Supplier	DAIKIN INDUSTRIES, LTD.
Lot number	C2160215

d) Physicochemical property

Boiling point	106°C (760 mmHg)
Appearance	Colorless transparent liquid
Density	1.560 g/cm^3

e) Storage conditions

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

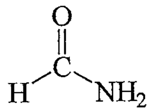
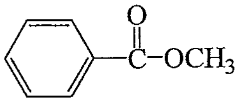
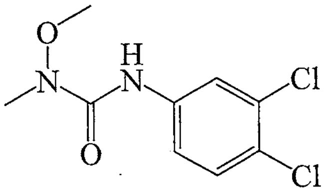
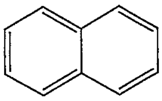
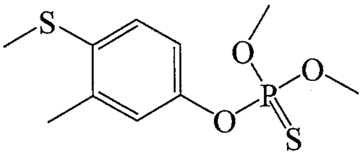
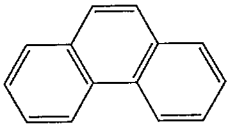
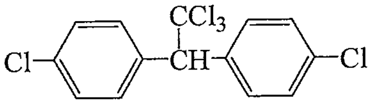
f) Identification and stability of test item

The infrared (IR) spectrum of the test item measured at this laboratory was confirmed to be identical to that provided by the sponsor (see Fig. 4-1 and Reference). The stability of the test item was confirmed by comparing the IR spectrum of the test item after the completion of the experiment with that before the start of the experiment (see Figs. 4-1, 4-2).

g) Safety and handling

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

12.2 Reference item

Name and CAS	Structural formula	Supplier and grade	Purity (%)
Formamide (for dead time) 75-12-7		Wako Pure Chemical Industries, Ltd. JIS special grade	≥98.5
Methyl benzoate 93-58-3		Wako Pure Chemical Industries, Ltd. Wako special grade	≥98.0
Linuron 330-55-2		Wako Pure Chemical Industries, Ltd. Trace Sure	99.8
Naphthalene 91-20-3		Kanto Chemical Co., Inc. Cica special grade	≥99
Fenthion 55-38-9		Sigma-Aldrich Corporation -	97.9
Phenanthrene 85-01-8		NACALAI TESQUE, INC. Guaranteed reagent	98
DDT 50-29-3		Tokyo Chemical Industry Co., Ltd. TCI-EP	≥98

13. Performance of test

13.1 Test conditions

a) Test equipment

Instrument	High-performance liquid chromatograph LC-2010CHT (built-in UV-VIS detector) (Shimadzu Corporation)
Column	Inertsil CN-3 (150 mm × 2.1 mm I.D., particle size 5 µm, GL Sciences Inc.)
Column temperature	25°C
Eluent	Methanol/purified water (60/40 v/v)
Flow rate	0.2 mL/min
Measurement wavelength	210 nm (reference item) 200 nm (test item) (see Fig. 3)
Sample size	2 µL

b) Test temperature

25±1°C

13.2 Test procedures

a) Preparation of reference item solution

Each about 10 mg of methyl benzoate, naphthalene, phenanthrene and DDT was weighed with an electronic analytical balance and dissolved in methanol to prepare each of about 1000 mg/L solution. Each about 2 mg of linurone and fenthion was weighed with the electronic analytical balance and dissolved in methanol to prepare each of about 1000 mg/L solution. About 500 mg of formamide (for dead time) was weighed with the electronic analytical balance and dissolved in methanol to prepare about 50000 mg/L solution. These solutions were mixed and then diluted with methanol to prepare the reference item solution for the measurement of adsorption coefficient.

The concentration of each reference item is shown as follows.

Reference items	log Koc	Concentration (mg/L)
Formamide (for dead time)	-	About 2500
Methyl benzoate	1.80	About 150
Linuron	2.59	About 50
Naphthalene	2.75	About 25
Fenthion	3.31	About 50
Phenanthrene	4.09	About 50
DDT	5.63	About 100

b) Preparation of test item solution

About 100 mg of the test sample was weighed with an electric analytical balance and dissolved in methanol to prepare about 10000 mg/L solution of test item. The test item solution was prepared by use of methanol, because the test sample was not dissolved up to 10000 mg/L in the eluent of HPLC analysis. Methanol 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 twice to the test equipment in Section 13.1 a) and the retention times of the reference items were measured, respectively. The capacity factors (k) of the reference items were calculated by the following equation.

A regression line was made by the method of least squares using the adsorption coefficient and the logarithmic values of the capacity factors for reference items (see Table 1 and Figs. 1, 2). 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)

$$\log Koc = a \times \log k + b$$

a : Slope of regression equation
b : Intercept of regression equation

d) Measurement of retention time for test item

The test item solution was injected twice to the test equipment in Section 13.1 a) and the retention time of the test item was measured. The solvent blank was injected to the test equipment once. It was confirmed that no peak existed at the peak position of the test item by the analysis of the solvent blank.

13.3 Calculation of adsorption coefficient

The capacity factor of the test item was calculated from the retention time. The adsorption coefficient was calculated using the regression equation of the regression line. The average of calculated values was regarded as the adsorption coefficient of the test item.

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

13.4 Treatment of numerical values

These values were treated in accordance with JIS Z 8401:1999 rule B.

14. Factors that affected reliability of test

No adverse effects on the reliability of this test were noted.

15. Results and discussion

15.1 Measurement results

	Name	t_R	k	log k	log Koc
Reference items	Formamide (for dead time: t_0)	2.13	Average $t_0 = 2.13$		
		2.13			
	Methyl benzoate	3.06	0.437	-0.360	1.80
		3.06	0.437	-0.360	1.80
	Linuron	3.98	0.869	-0.061	2.59
		3.98	0.869	-0.061	2.59
	Naphthalene	4.82	1.263	0.101	2.75
		4.82	1.263	0.101	2.75
	Fenthion	5.38	1.526	0.184	3.31
		5.38	1.526	0.184	3.31
	Phenanthrene	8.06	2.784	0.445	4.09
		8.07	2.789	0.445	4.09
	DDT	14.03	5.587	0.747	5.63
		14.05	5.596	0.748	5.63
Test item	C6OLF	4.12	0.934	-0.030	2.66
		4.10	0.925	-0.034	2.64

t_0 : Dead time (min)

t_R : Retention time (min)

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

15.2 Regression equation of regression line in measurement conditions

$$\log Koc = 3.419 \times \log k + 2.760$$

15.3 Adsorption coefficient of test item

log Koc		
Measured value		Average
2.66	2.64	2.65

15.4 Discussion

The average of the measured log Koc values was 2.65 and the difference between two measured values was 0.02. It is judged that the test results are valid because the difference between two measured values is within ±0.25.

Table 1　Calculation table for absorption coefficient by HPLC method

Study No. 85000

		t_R	k	log k	log Koc
Reference item	1-a	2.13	t_0 =	2.13	
	1-b	2.13			
	2-a	3.06	0.437	-0.360	1.80
	2-b	3.06	0.437	-0.360	1.80
	3-a	3.98	0.869	-0.061	2.59
	3-b	3.98	0.869	-0.061	2.59
	4-a	4.82	1.263	0.101	2.75
	4-b	4.82	1.263	0.101	2.75
	5-a	5.38	1.526	0.184	3.31
	5-b	5.38	1.526	0.184	3.31
	6-a	8.06	2.784	0.445	4.09
	6-b	8.07	2.789	0.445	4.09
	7-a	14.03	5.587	0.747	5.63
	7-b	14.05	5.596	0.748	5.63
Test item	a	4.12	0.934	-0.030	2.66
	b	4.10	0.925	-0.034	2.64
	(a,b : individual sample)			Average	2.65

$k = (t_R - t_0) / t_0$
t_0 : Dead time (average of two measured values) (min)
t_R : Retention time (min)

$\log Koc = 3.419 \times \log k + 2.760$
$r = 0.981$

Reference item

1	Formamide
2	Methyl benzoate
3	Linuron
4	Naphthalene
5	Fenthion
6	Phenanthrene
7	DDT

See Figs. 1,2

March 9, 2017　　Name

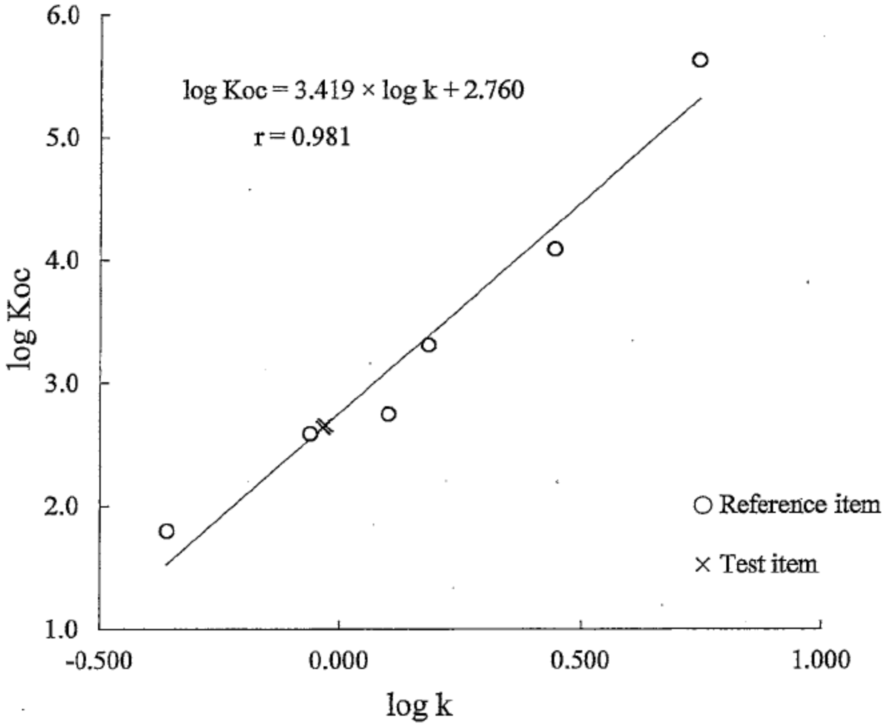
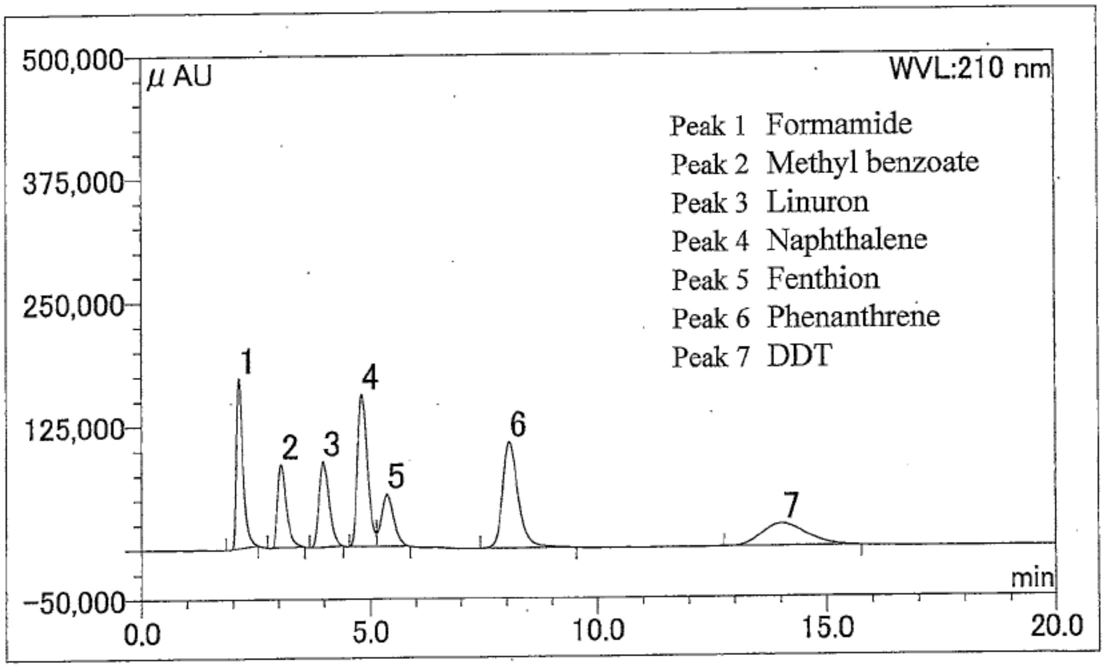


Fig. 1 Calibration curve for absorption coefficient by HPLC method.

March 9, 2017 Name

Reference item − 1

Operator:	
Operating date:	06/Mar/2017
Sample ID:	85000_170306_d1
Program:	85000_Ref_170306_LC138
Vial No.:	1_1
Channel:	UV_VIS_1



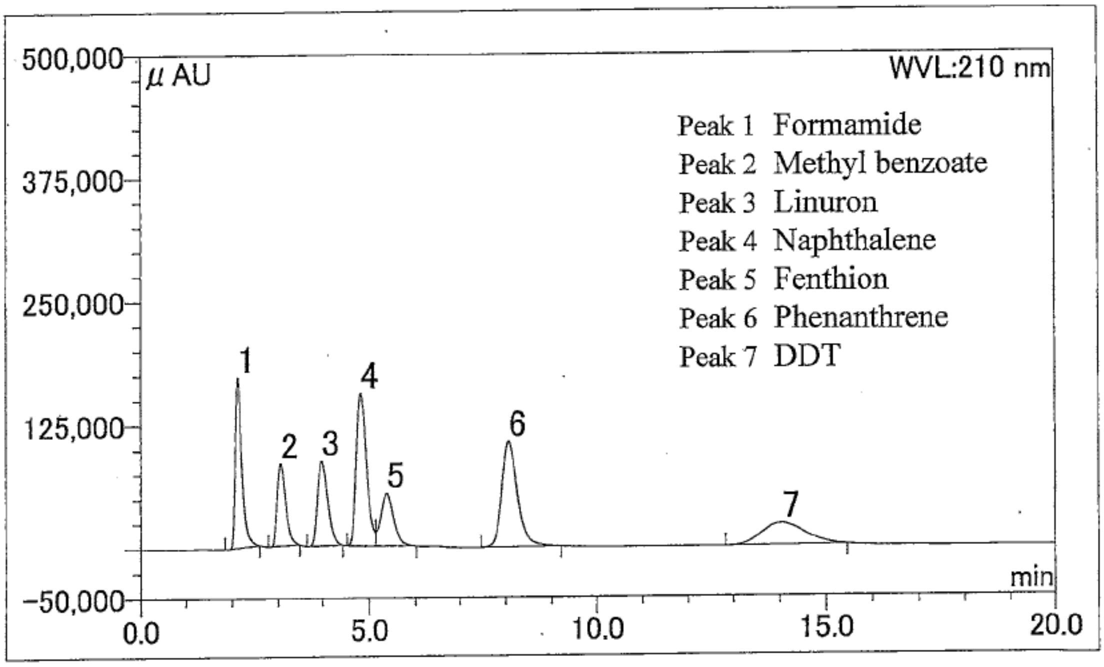
Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	2.13	171639	1712608	14.87
2	3.06	83482	1092496	9.48
3	3.98	86070	1346238	11.69
4	4.82	153481	2323381	20.17
5	5.38	52633	992416	8.62
6	8.06	106803	2538895	22.04
7	14.03	22185	1512555	13.13
Total	−	−	11518589	100.00

2017. 3. 6

Fig. 2- 1 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method.

Reference item – 2

Operator:	
Operating date:	06/Mar/2017
Sample ID:	85000_170306_d5
Program:	85000_Ref_170306_LC138
Vial No.:	1_1
Channel:	UV_VIS_1



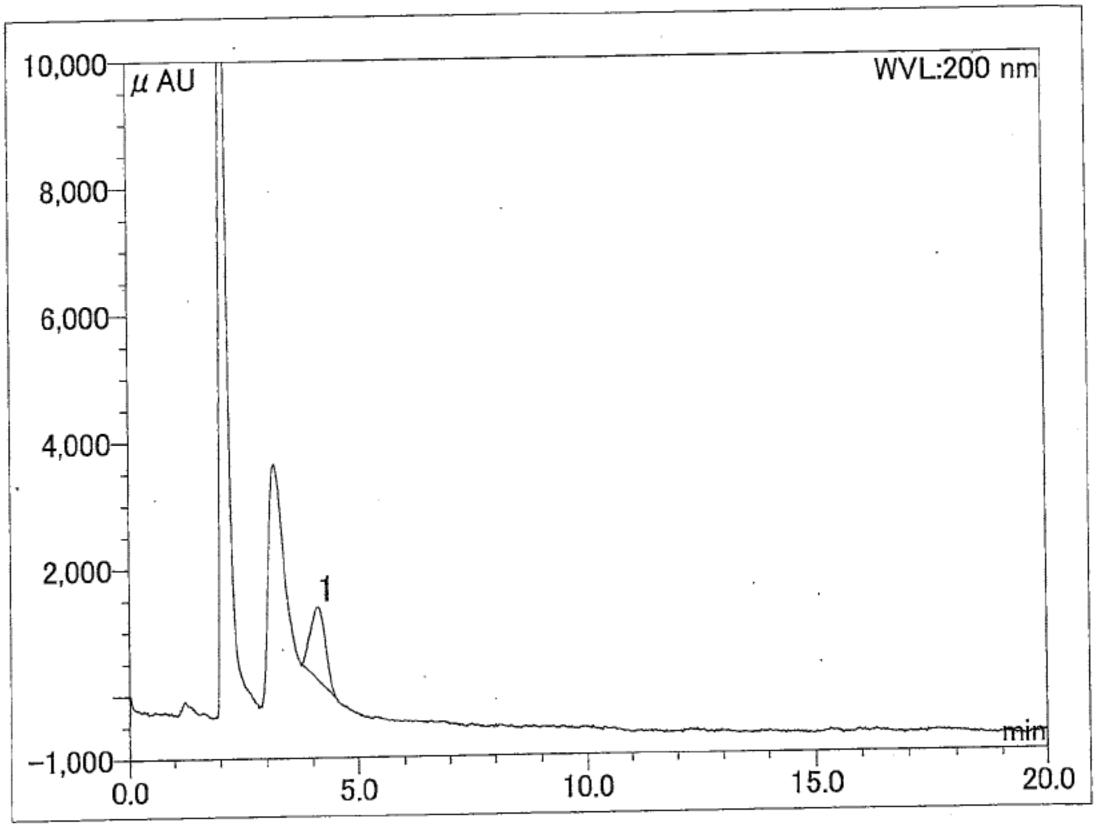
Peak No.	Time (min)	Height (µAU)	Area (µAU·sec)	Area (%)
1	2.13	171843	1735241	15.17
2	3.06	83045	1068615	9.34
3	3.98	85764	1344132	11.75
4	4.82	153709	2340413	20.46
5	5.38	53219	1027529	8.98
6	8.07	106222	2494272	21.80
7	14.05	21673	1431038	12.51
Total	–	–	11441240	100.00

2017. 3. 6

Fig. 2- 2 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method.

Test item − 1

Operator:	
Operating date:	06/Mar/2017
Sample ID:	85000_170306_d2
Program:	85000_Test_170306_LC138
Vial No.:	1_2
Channel:	UV_VIS_1



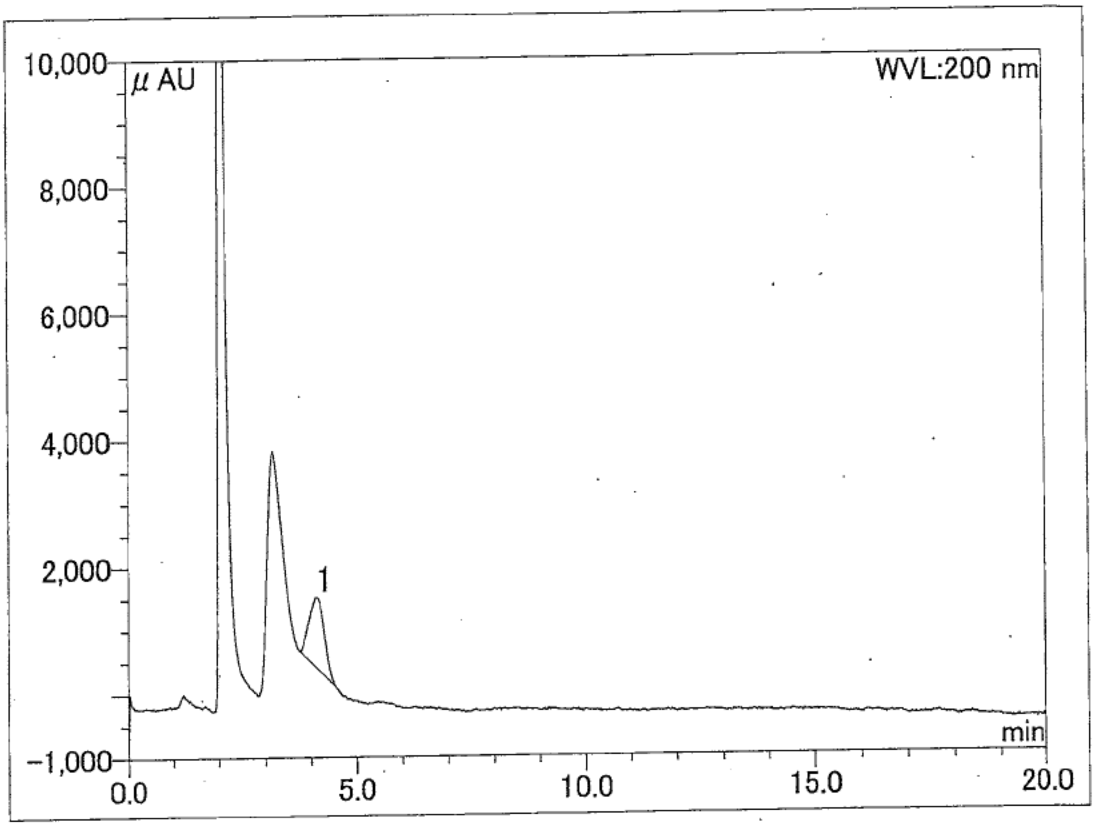
Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	4.12	1153	27041	100.00
Total	−	−	27041	100.00

2017. 3. 6

Fig. 2- 3 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method.

Test item − 2

Operator:	
Operating date:	06/Mar/2017
Sample ID:	85000_170306_d3
Program:	85000_Test_170306_LC138
Vial No.:	1_2
Channel:	UV_VIS_1



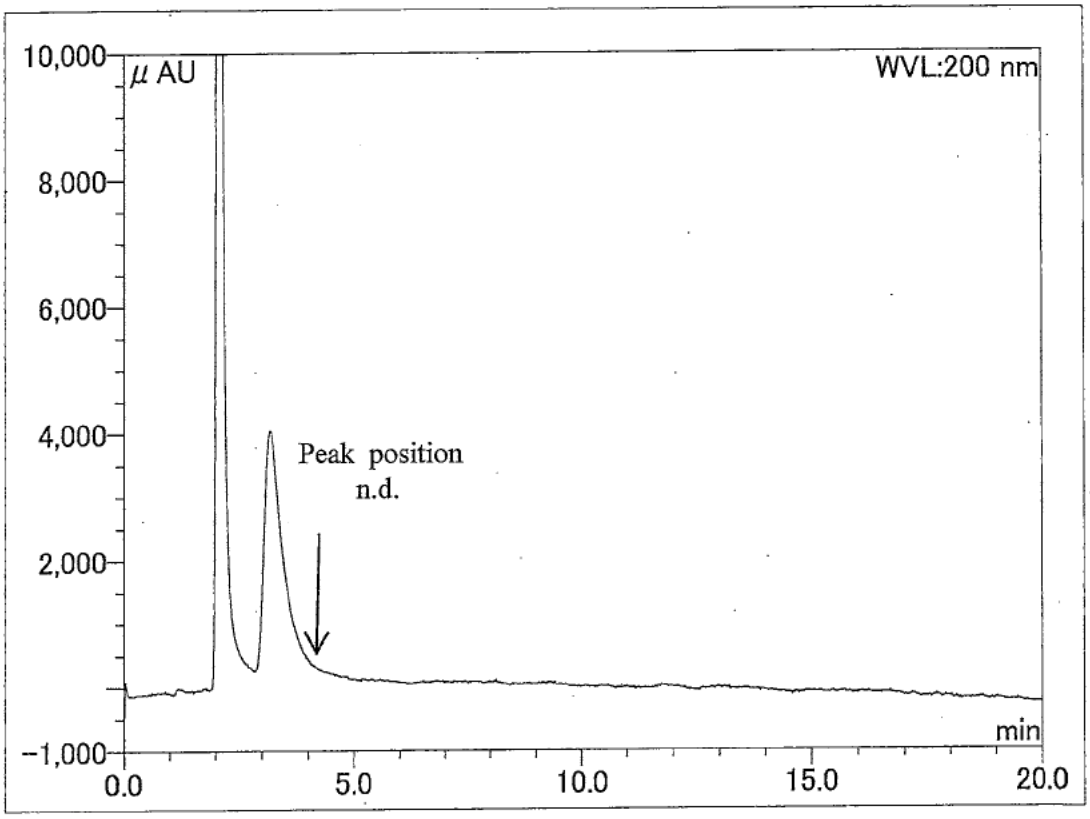
Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	4.10	1102	26173	100.00
Total	−	−	26173	100.00

2017. 3. 6

Fig. 2- 4 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method.

Solvent blank

Operator:	
Operating date:	06/Mar/2017
Sample ID:	85000_170306_d4
Program:	85000_Test_170306_LC138
Vial No.:	1_3
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
Total	–	–	0	0.00

2017. 3. 6

Fig. 2- 5 Chromatogram of HPLC analysis for adsorption coefficient by HPLC method.

2017.2.8 85000
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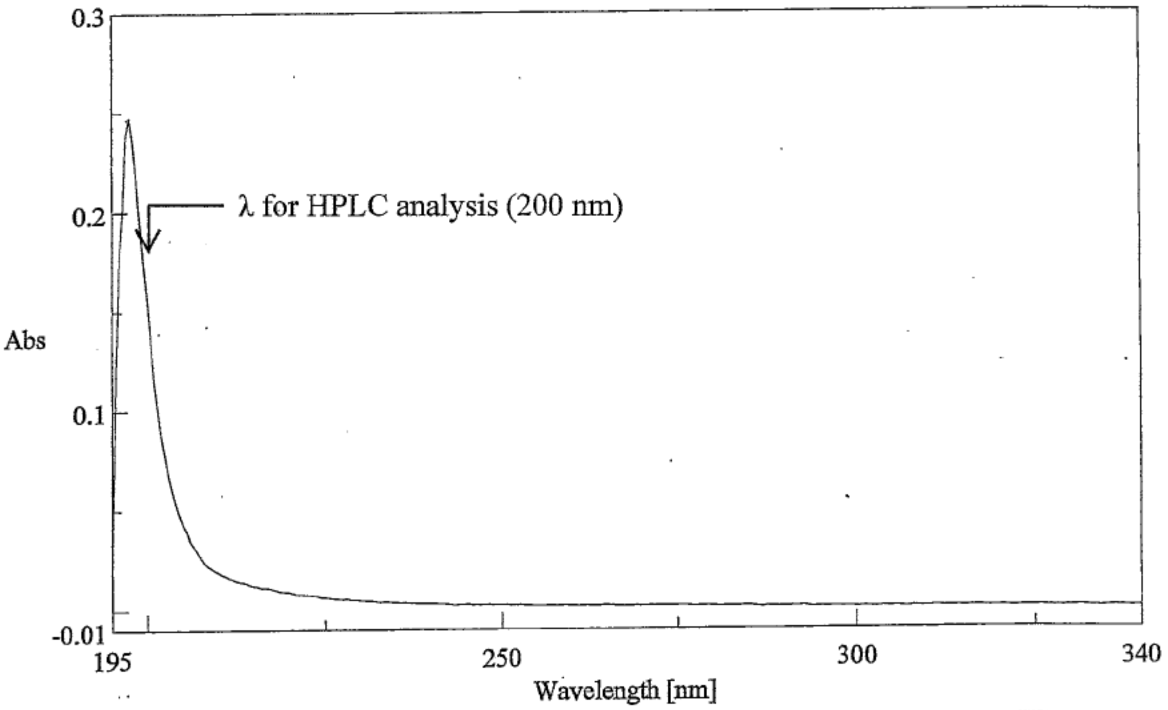
Study No.	84999	Wavelength	195.00-340.00
Date	February. 08, 2017	Scale Limit	-0.01-0.30
Sample	Test item	Slit Width	(UV/VIS)2.0 nm
Solvent	Methanol	Scan Speed	200nm/min
Reference	-	Sampling Pitch	0.500nm
Cell	10mm×10 mm, quartz	Analyst	
Instrument	V-660	Note	about 10000 mg/L
Photometric Mode	Abs		

Chemicals Evaluation and Research Institute, Japan, Kurume



Peak List

Fig. 3 UV spectrum of test item.

85000
2017. 2. 7
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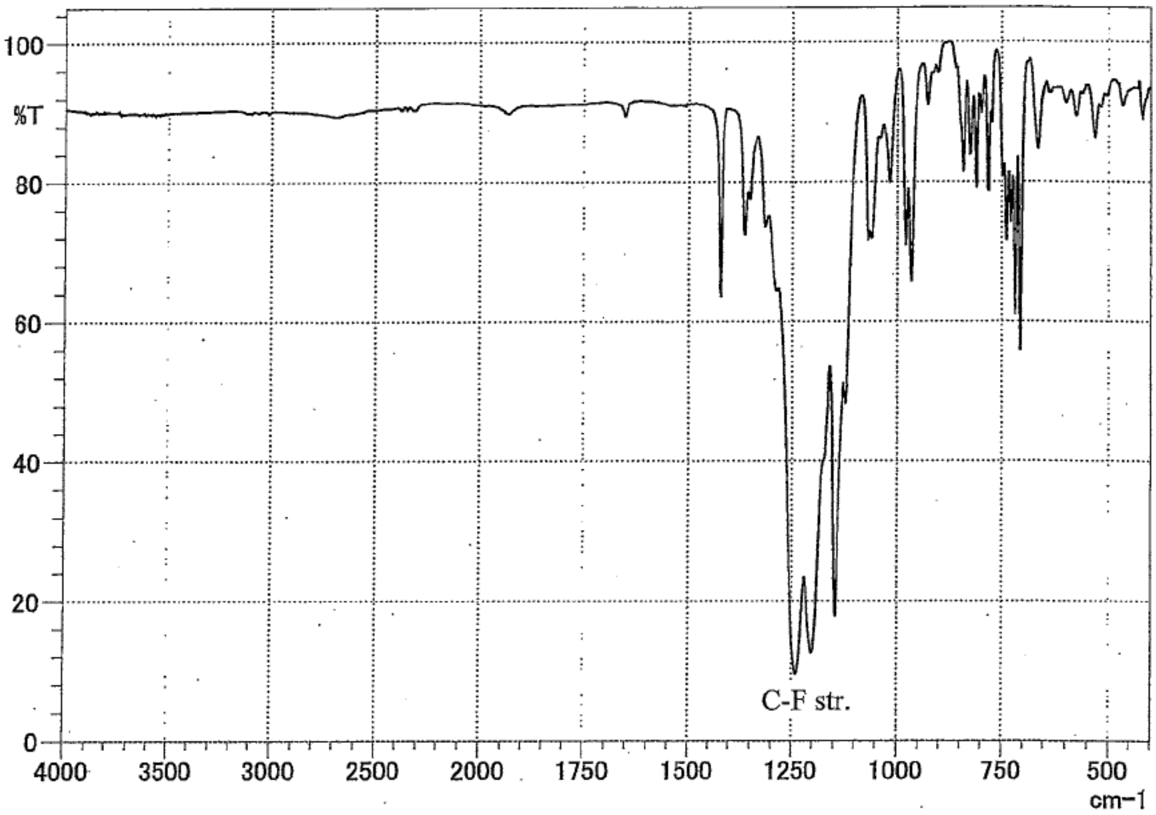
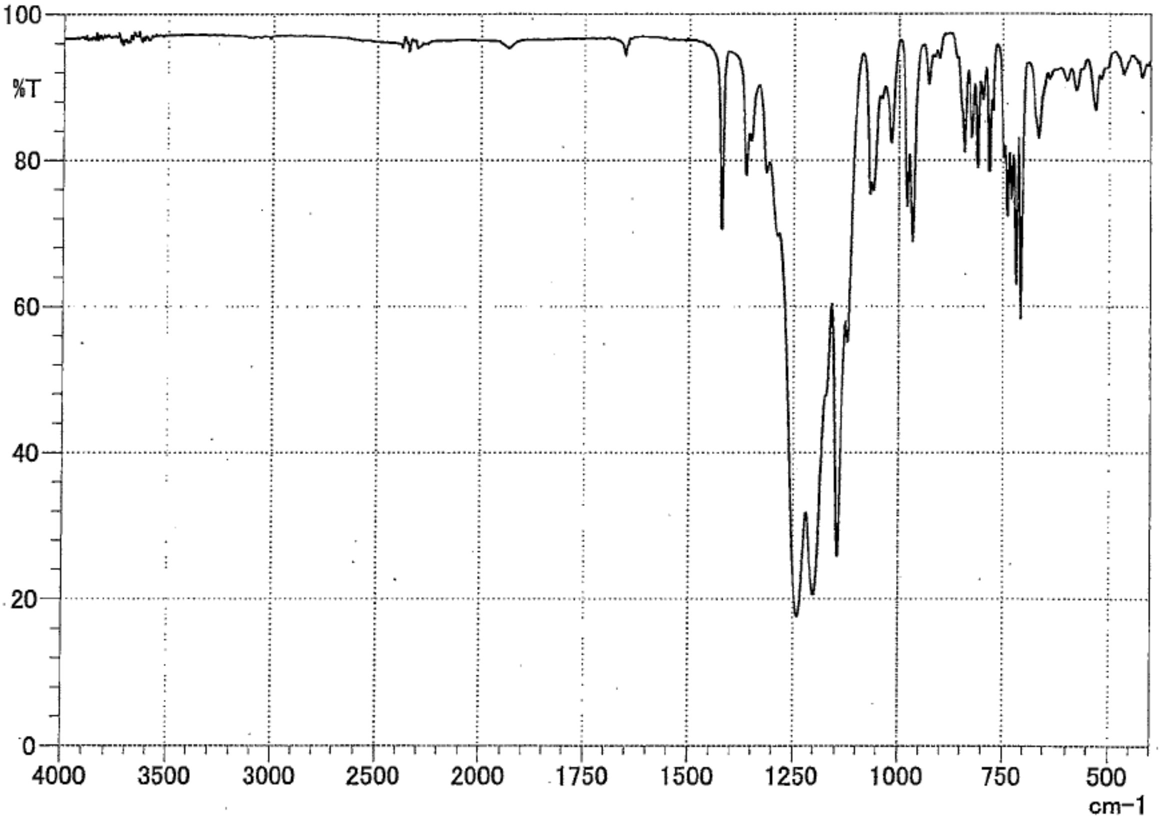


Instrument	: Shimadzu IRAffinity-1S
Study No.	: 84999
Sample	: Test item
Method	: Neat
Date	: February 7, 2017
Name	:

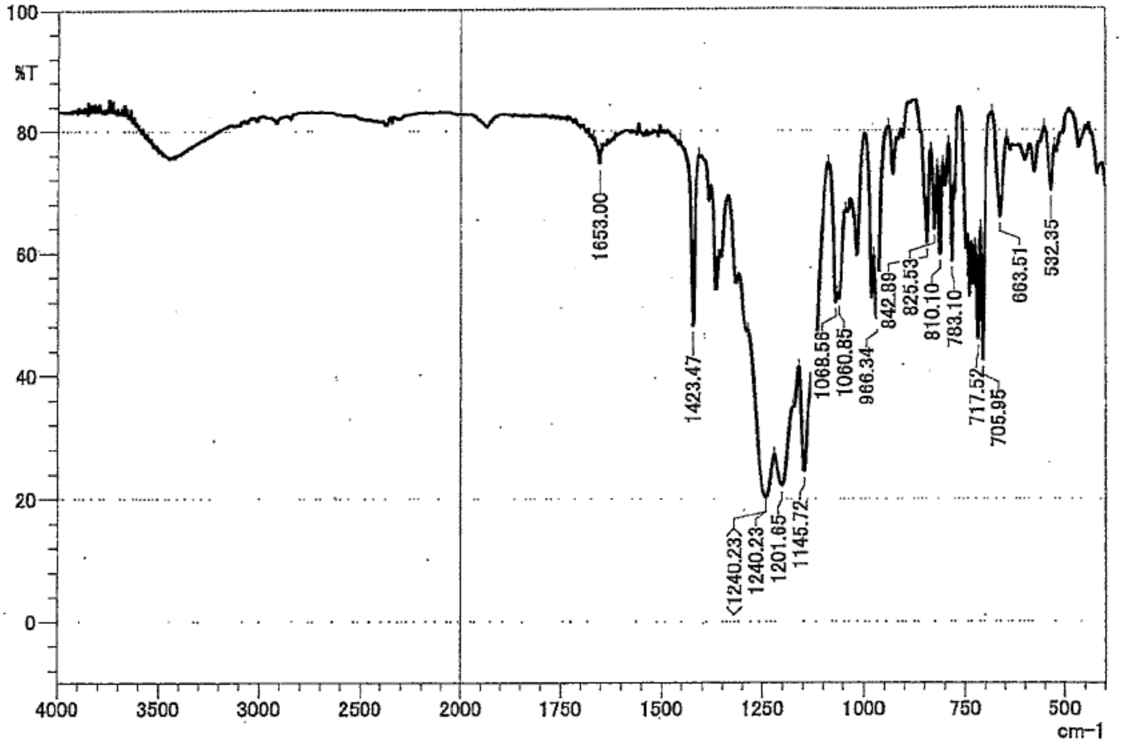
Fig. 4 - 1 IR spectrum of test item measured before experimental start.



Instrument : Shimadzu IRAffinity-1S
Study No. : 85000
Sample : Test item
Method : Neat
Date : March 7, 2017
Name :

Fig. 4 - 2 IR spectrum of test item measured after experimental completion.

SHIMADZU



	パラメータ	値
2	サンプル名	c6オレフィン
3	サンプルID	Lot,o2160215
4	オプション	
5	測定モード	透過率
6	アポダイズ関数	Happ-Genzel
9	積算	20
10	分解	4 cm-1

	ピーク	高さ	補正高さ	ベース (H)	ベース (L)	面積	補正面積	コメント
1	532.35	70.23	8.13	551.64	522.71	711.92	100.74	
2	663.51	65.92	14.72	682.80	646.15	948.91	243.79	
3	705.95	42.56	25.22	711.73	682.80	898.76	130.28	
4	717.52	46.03	16.72	725.23	711.73	606.57	100.94	
5	783.10	58.74	15.42	790.81	777.31	430.21	87.37	
6	810.10	59.88	14.47	817.82	804.32	433.45	86.65	
7	825.53	63.93	11.51	833.25	817.82	465.62	86.60	
8	842.89	61.76	16.20	858.32	833.25	718.20	174.42	
9	966.34	49.43	14.89	974.05	939.33	1120.55	85.27	
10	1060.85	52.51	3.40	1064.71	1043.49	872.64	28.42	
11	1068.56	52.05	5.26	1085.92	1064.71	753.69	-9.11	
12	1145.72	24.57	16.42	1159.22	1126.43	2188.89	251.14	
13	1201.65	22.23	8.17	1220.94	1174.65	3391.08	198.75	
14	1240.23	20.33	12.95	1284.59	1220.94	4501.77	509.03	
15	1423.47	48.20	28.57	1454.33	1408.04	1372.53	315.75	
16	1653.00	74.73	2.36	1668.43	1651.07	400.51	16.32	

11/15/'16

Reference IR spectrum supplied by sponsor.