CERI

FINAL REPORT

Measurement of 1-octanol/water partition coefficient for C6OLF (HPLC method)

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 1-octanol/water partition coefficient for C6OLF (HPLC method)

Study number   84999

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.

March 17, 2017

Date

Study Director

QUALITY ASSURANCE STATEMENT

Chemicals Evaluation and Research Institute, Japan, Kurume

Sponsor: DAIKIN INDUSTRIES, LTD.

Title: Measurement of 1-octanol/water partition coefficient for C6OLF (HPLC method)

Study number: 84999

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	9,	2017	March	9,	2017
Test conduct	March	13,	2017	March	13,	2017
Raw data and draft final report	March	16,	2017	March	16,	2017
Final report	March	17,	2017	March	17,	2017

Date   *March 17, 2017*

Personnel of Quality Assurance Unit:

CONTENTS

1. Title

   Measurement of 1-octanol/water partition coefficient for C6OLF (HPLC method)

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 is performed to obtain the 1-octanol/water partition coefficient of C6OLF.

5. Test methods

   OECD Guidelines for Testing of Chemicals, No. 117, April 13, 2004, "Partition Coefficient (n-octanol/water), High Performance Liquid Chromatography (HPLC) Method"

6. GLP principles

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

7. Dates

Study initiation date	March 8, 2017
Experimental starting date	March 13, 2017
Experimental completion date	March 14, 2017
Study completion date	March 17, 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	(Section 5)
Study personnel	

10. Approval of final report

    Date   March 17, 2017

    Study Director

11. Summary

    <u>Test item</u>

      C6OLF

    <u>Objective</u>

      This study is performed to obtain the 1-octanol/water partition coefficient of C6OLF.

    <u>Test method</u>

      OECD Guidelines for Testing of Chemicals, No. 117, April 13, 2004, "Partition Coefficient (n-octanol/water), High Performance Liquid Chromatography (HPLC) Method"

    <u>Test conditions</u>

Test equipment	High-performance liquid chromatograph (HPLC)
	Eluent: Methanol/purified water (75/25 v/v)
Test temperature	$25\pm1°C$

    <u>Results</u>

      The partition coefficient (log Pow) of C6OLF was 4.9 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

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

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.
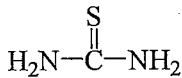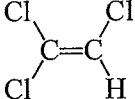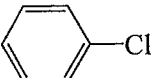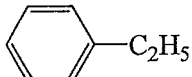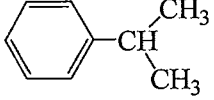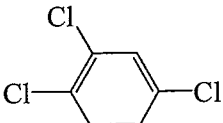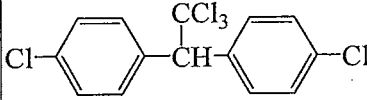
    f) Identification and stability of test item

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. 3-1 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. 3-1, 3-2).

    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 number	Structural formula	Supplier and grade	Purity (%)
Thiourea (for dead time: t0) 62-56-6	$H_2N-\overset{\overset{S}{\|\|}}{C}-NH_2$	Wako Pure Chemical Industries, Ltd. JIS special grade	≥98.0
Trichloroethylene 79-01-6	$\underset{Cl}{\overset{Cl}{>}}C=C\underset{H}{\overset{Cl}{<}}$	Kanto Chemical Co., Inc. JIS special grade	≥99.5
Chlorobenzene 108-90-7	⬡—Cl	Wako Pure Chemical Industries, Ltd. Wako special grade	≥99.0
Ethylbenzene 100-41-4	⬡—C_2H_5	Tokyo Chemical Industry Co., Ltd. TCI-GR	≥98.0
Isopropylbenzene 98-82-8	⬡—CH$\underset{CH_3}{\overset{CH_3}{<}}$	Wako Pure Chemical Industries, Ltd. Wako special grade	≥98.0
1,2,4-Trichlorobenzene 120-82-1	Cl, Cl—⬡—Cl	Tokyo Chemical Industry Co., Ltd. TCI-EP	99
DDT 50-29-3	Cl—⬡—CH$\overset{CCl_3}{|}$—⬡—Cl	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-2010C$_{HT}$ (Shimadzu Corporation)
Differential refractive index detector	
	RI-104 (SHOWA DENKO K.K.)
Column	L-column ODS (150 mm × 2.1 mm I.D., particle size 5 μm,
	Chemicals Evaluation and Research Institute, Japan)
Column temperature	25°C
Eluent	Methanol/purified water (75/25 v/v)
Flow rate	0.2 mL/min
Sample size	2 μL

    b) Test temperature

       25±1°C

13.2 Test procedures

    a) Preparation for reference item solution

About 20 mg of thiourea (for dead time) was weighed with an electronic analytical balance and dissolved in the eluent of HPLC analysis to prepare about 1000 mg/L solution of thiourea. About 100 mg/L solution of thiourea was then prepared from this solution by dilution with the eluent.

Each about 20 mg of trichloroethylene, chlorobenzene, ethylbenzene and isopropylbenzene was weighed with the electronic analytical balance and dissolved in the eluent to prepare each of about 1000 mg/L solution. About 20 mg of 1,2,4-trichlorobenzene was weighed with the electronic analytical balance and dissolved in methanol to prepare about 1000 mg/L solution. About 10 mg of DDT was weighed with the electronic analytical balance and dissolved in methanol to prepare about 1000 mg/L solution. These solutions were mixed and then diluted with the eluent to prepare the reference item solution for the measurement of partition coefficient. The concentration of each reference item is shown as follows.

Reference items	log Pow	Concentration (mg/L)
Trichloroethylene	2.4	About 100
Chlorobenzene	2.8	About 100
Ethylbenzene	3.2	About 100
Isopropylbenzene	3.7	About 100
1,2,4-Trichlorobenzene	4.2	About 100
DDT	6.5	About 100

    b) Preparation for solution of test item

About 100 mg of the test sample was weighed with an electric analytical balance and dissolved in methanol to prepare about 10000 mg/L stock solution of the test item. About 1000 mg/L solution of the test item was then prepared from this solution by dilution with the eluent. The methanol was diluted tenfold with the eluent to use as a solvent blank.

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

The reference item solution and about 100 mg/L solution of thiourea were injected twice to the test equipment in Section 13.1 a) and the retention times of reference items were measured, respectively. 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 partition coefficient 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)

$\log P_{ow} = a \times \log k + b$

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 once to the test equipment. 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 partition coefficient

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

13.4　Treatment of numerical values

Values were rounded off in accordance with JIS Z 8401:1999 rule B.

The partition coefficient value was represented as logarithm and rounded off to one decimal place.

14. Factors that affected reliability of test

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

15. Results and discussion

Measurement results

	Name	t_R	k	log k	log Pow
Reference items	Thiourea (for dead time: t_0)	2.01	Average t_0 = 2.01		
		2.01			
	Trichloroethylene	5.29	1.632	0.213	2.4
		5.28	1.627	0.211	2.4
	Chlorobenzene	5.70	1.836	0.264	2.8
		5.70	1.836	0.264	2.8
	Ethylbenzene	7.83	2.896	0.462	3.2
		7.83	2.896	0.462	3.2
	Isopropylbenzene	10.13	4.040	0.606	3.7
		10.13	4.040	0.606	3.7
	1,2,4-Trichlorobenzene	12.91	5.423	0.734	4.2
		12.91	5.423	0.734	4.2
	DDT	43.47	20.627	1.314	6.5
		43.37	20.577	1.313	6.5
Test item	C6OLF	18.14	8.025	0.904	4.9
		18.14	8.025	0.904	4.9

t_0 : Dead time (min)

t_R : Retention time (min)

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

15.1 Regression equation of regression line in measurement conditions

log Pow = 3.632 × log k + 1.626

15.2 Partition coefficient of test item

log Pow		
Measured value		Average
4.9	4.9	4.9

15.3 Discussion

The average of the measured log Pow values of test item was 4.9 and the difference between two measured values was 0.0. It is judged that the test results are valid because the difference between two measured values is less than ±0.1.

Table 1　Calculation table for partition coefficient by HPLC method

		t_R	k	log k	log Pow
Reference item	1-a	2.01		$t_0 =$ 2.01	
	1-b	2.01			
	2-a	5.29	1.632	0.213	2.4
	2-b	5.28	1.627	0.211	2.4
	3-a	5.70	1.836	0.264	2.8
	3-b	5.70	1.836	0.264	2.8
	4-a	7.83	2.896	0.462	3.2
	4-b	7.83	2.896	0.462	3.2
	5-a	10.13	4.040	0.606	3.7
	5-b	10.13	4.040	0.606	3.7
	6-a	12.91	5.423	0.734	4.2
	6-b	12.91	5.423	0.734	4.2
	7-a	43.47	20.627	1.314	6.5
	7-b	43.37	20.577	1.313	6.5
Test item	a	18.14	8.025	0.904	4.9
	b	18.14	8.025	0.904	4.9
	(a,b : individual sample)			Average	4.9

$k = (t_R - t_0) / t_0$

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

t_R : Retention time (min)

log Pow = 3.632 × log k + 1.626

　　 r = 0.996

Reference item

1	Thiourea
2	Trichloroethylene
3	Chlorobenzene
4	Ethylbenzene
5	Isopropylbenzene
6	1,2,4-Trichlorobenzene
7	DDT

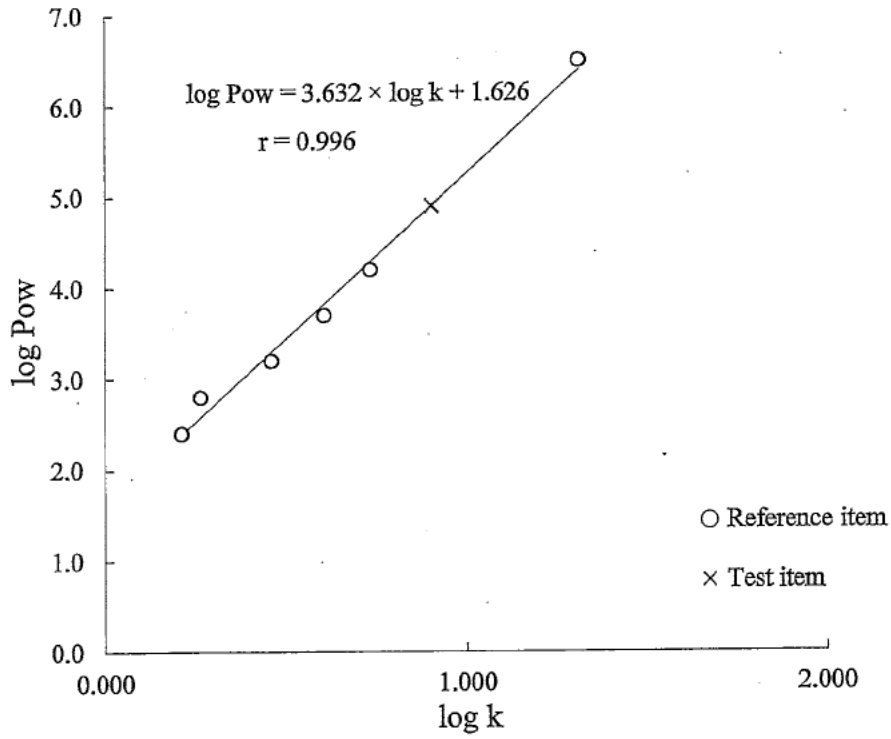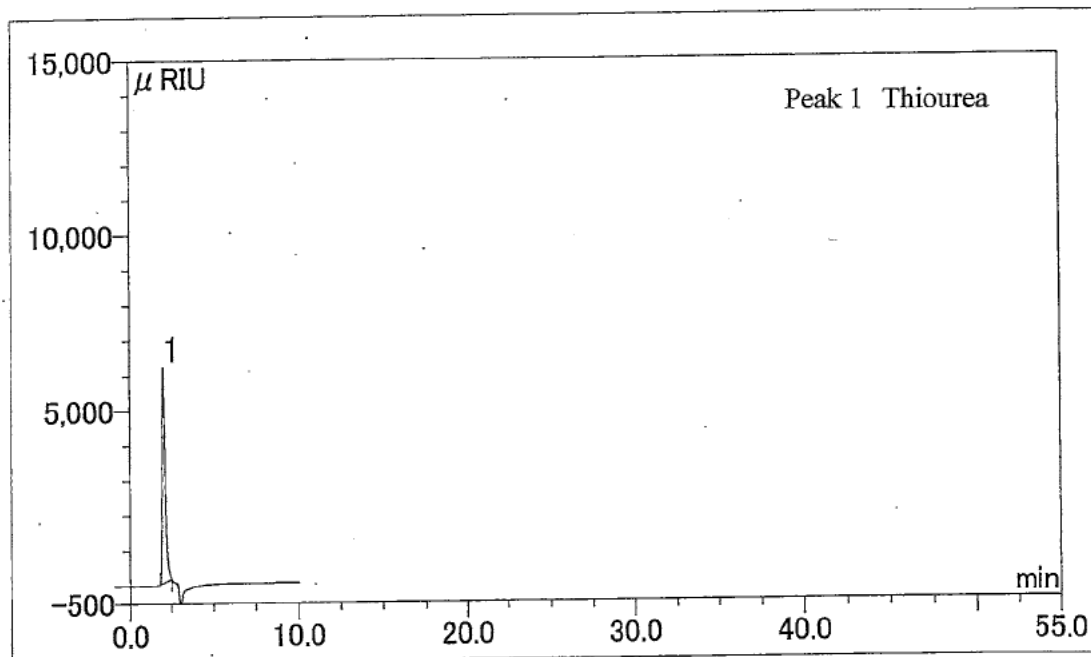See Figs. 1,2

March 14, 2017　　　　Name

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

March 14, 2017　　　Name _____

Thiourea – a

Operator:	
Operating date:	13/Mar/2017
Sample ID:	84999_170313_1
Program:	84999pro_Thio_0313
Vial No.:	1_1
Channel:	RI_1

Peak 1 Thiourea

Peak No.	Time (min)	Height (μ RIU)	Area (μ RIU·sec)	Area (%)
1	2.01	6144	75715	100.00
Total	–	–	75715	100.00

2017. 3. 14

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

Thiourea - b

Operator:	
Operating date:	13/Mar/2017
Sample ID:	84999_170313_2
Program:	84999pro_Thio_0313
Vial No.:	1_1
Channel:	RI_1
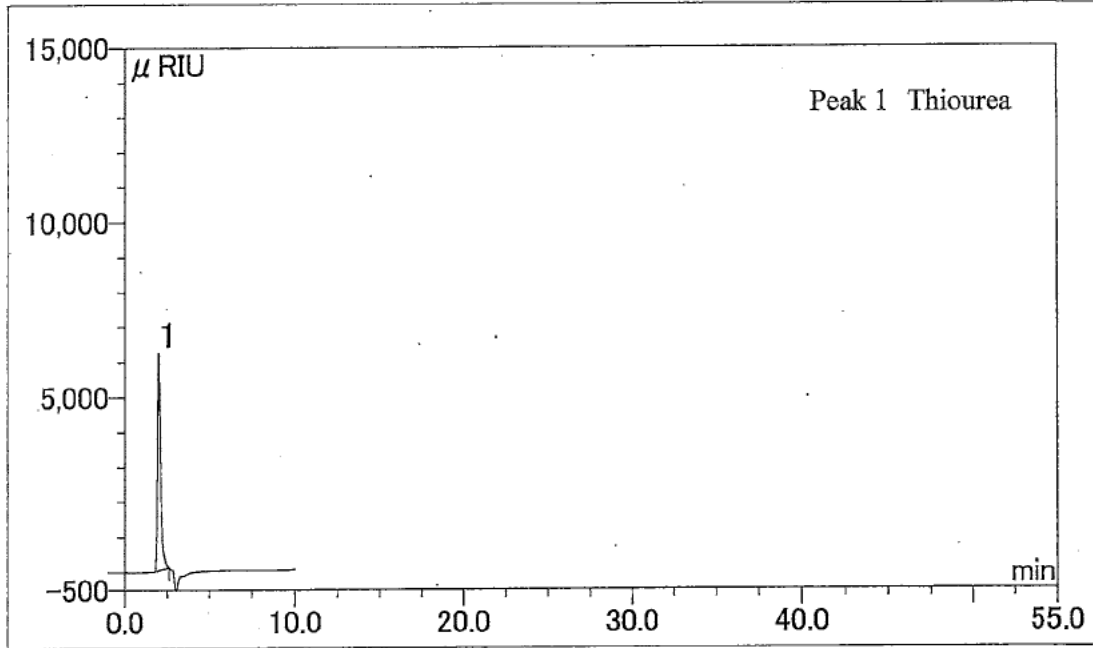
Peak No.	Time (min)	Height (μRIU)	Area (μRIU·sec)	Area (%)
1	2.01	6206	77784	100.00
Total	--	--	77784	100.00

2017, 3. 14

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

Reference item - a

Operator:	
Operating date:	13/Mar/2017
Sample ID:	84999_170313_3
Program:	84999pro_ref_0313
Vial No.:	1_2
Channel:	RI_1

Peak 2 Trichloroethylene
Peak 3 Chlorobenzene
Peak 4 Ethylbenzene
Peak 5 Isopropylbenzene
Peak 6 1,2,4-Trichlorobenzene
Peak 7 DDT
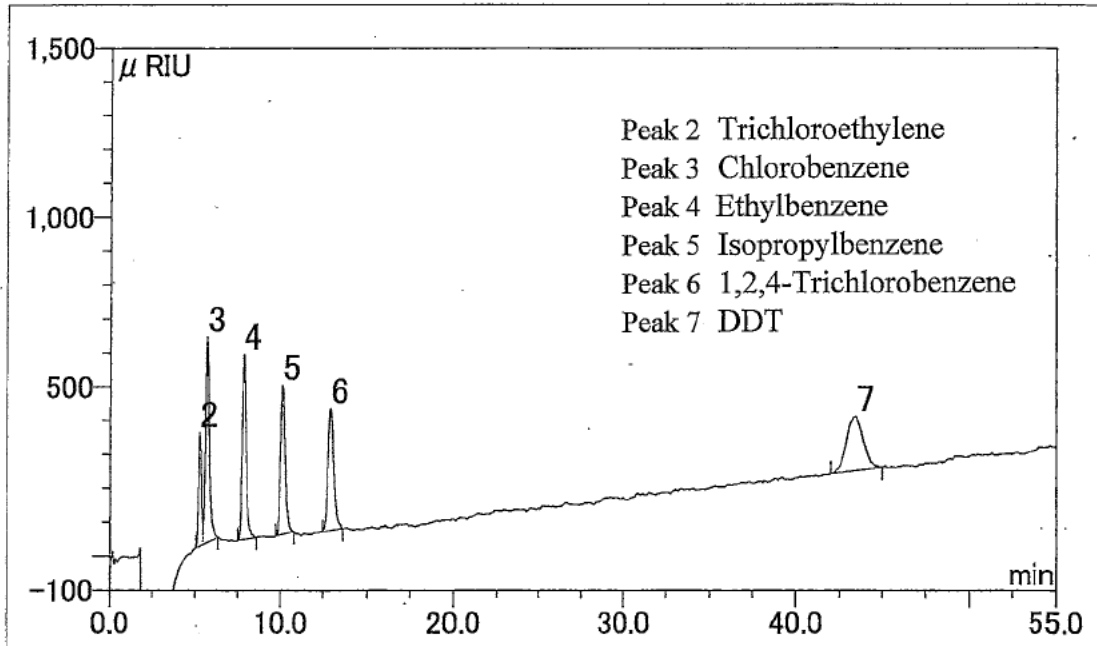
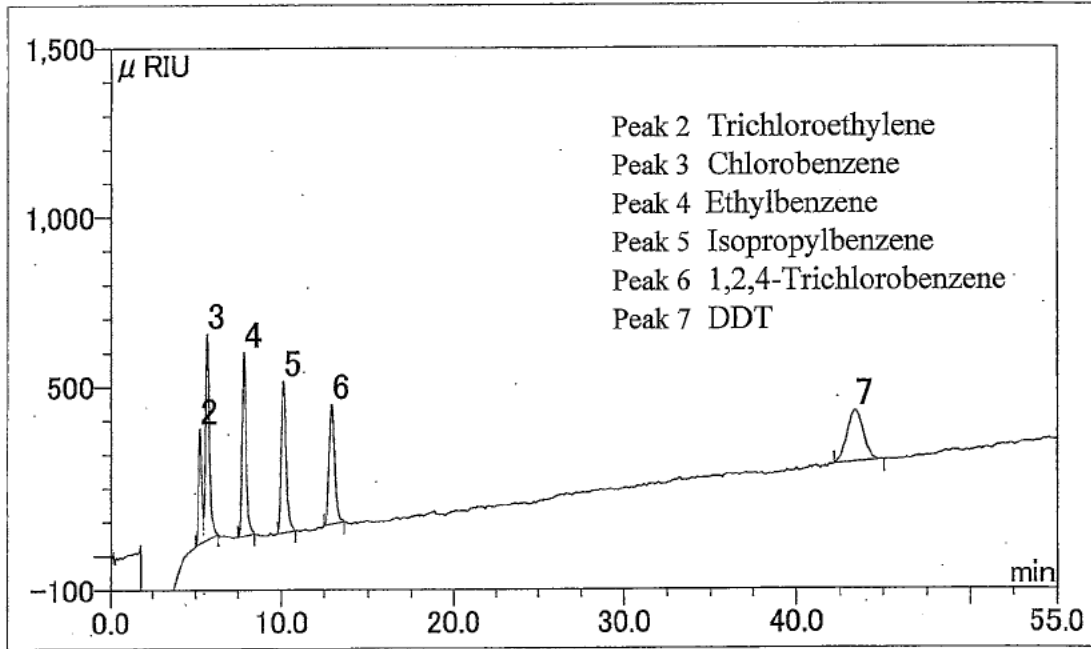| Peak No.	Time (min)	Height (μRIU)	Area (μRIU·sec)	Area (%)
2	5.29	335	4299	8.66
3	5.70	606	9107	18.34
4	7.83	545	9087	18.30
5	10.13	440	8303	16.72
6	12.91	360	8164	16.44
7	43.47	159	10705	21.55
Total	–	–	49664	100.00

2017. 3. 14

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

Reference item - b

Operator:	
Operating date:	14/Mar/2017
Sample ID:	84999_170313_4
Program:	84999pro_ref_0313
Vial No.:	1_2
Channel:	RI_1

Peak 2 Trichloroethylene
Peak 3 Chlorobenzene
Peak 4 Ethylbenzene
Peak 5 Isopropylbenzene
Peak 6 1,2,4-Trichlorobenzene
Peak 7 DDT

Peak No.	Time (min)	Height (μ RIU)	Area (μ RIU·sec)	Area (%)
2	5.28	339	4449	9.14
3	5.70	608	9155	18.81
4	7.83	541	8861	18.21
5	10.13	446	8544	17.56
6	12.91	353	7576	15.57
7	43.37	152	10079	20.71
Total	−	−	48663	100.00

2017. 3. 14

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

Test item – a

Operator:	
Operating date:	14/Mar/2017
Sample ID:	84999_170313_5
Program:	84999pro_testitem_0313
Vial No.:	1_3
Channel:	RI_1
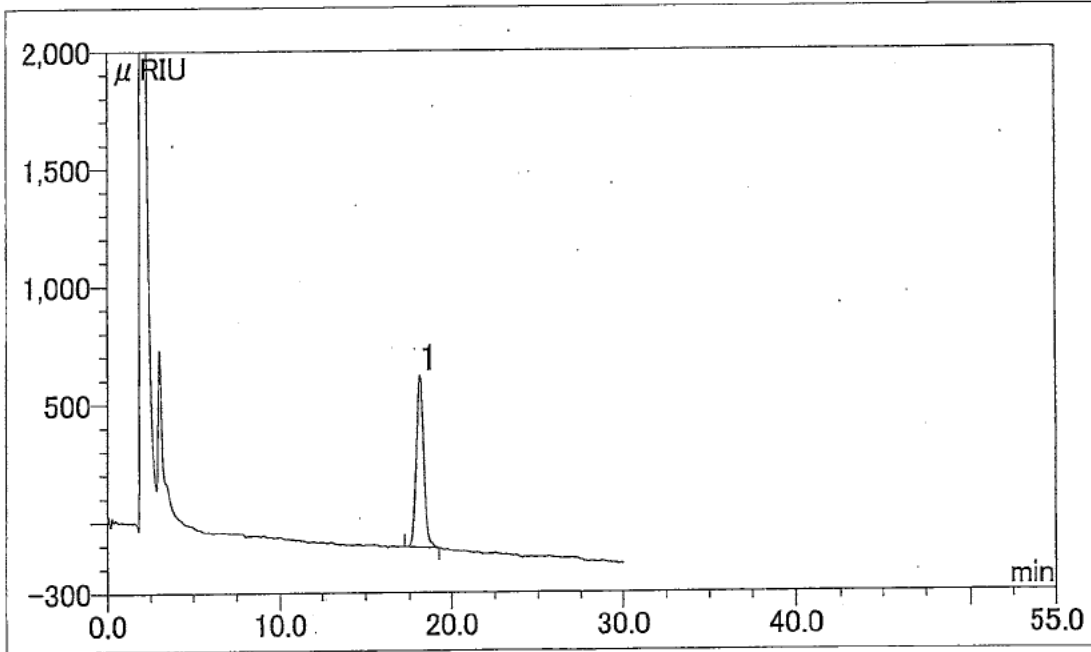
Peak No.	Time (min)	Height (μRIU)	Area (μRIU·sec)	Area (%)
1	18.14	726	21876	100.00
Total	–	–	21876	100.00

2017. 3. 14

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

Test item − b

Operator:	
Operating date:	14/Mar/2017
Sample ID:	84999_170313_6
Program:	84999pro_testitem_0313
Vial No.:	1_3
Channel:	RI_1
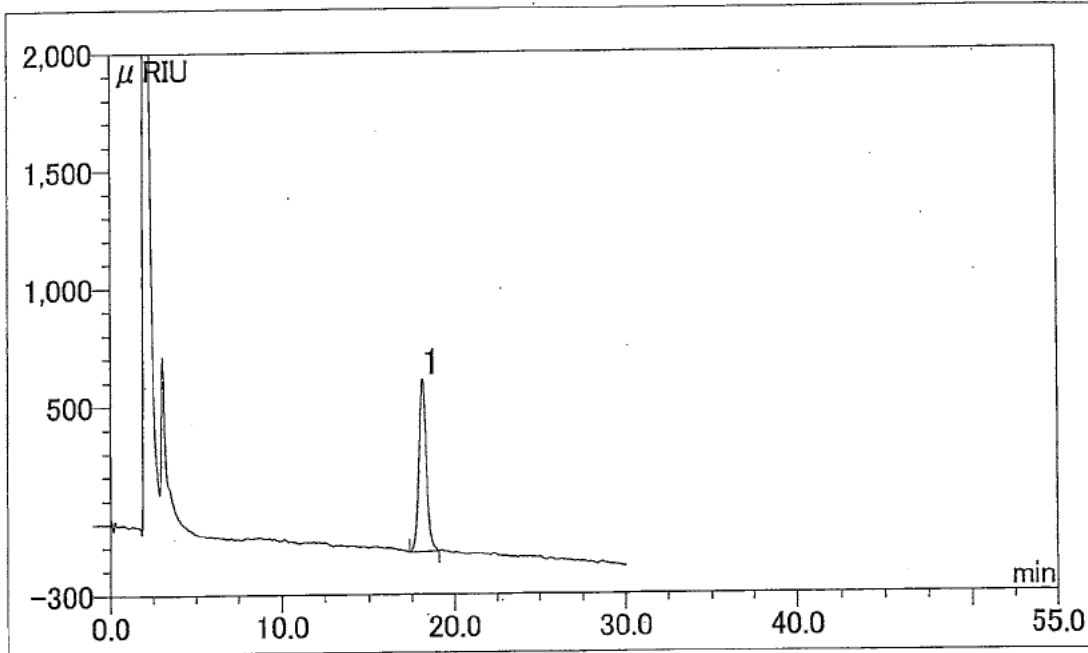
Peak No.	Time (min)	Height (μ RIU)	Area (μ RIU·sec)	Area (%)
1	18.14	728	22154	100.00
Total	−	−	22154	100.00

2017. 3. 14

Fig. 2-6   Chromatogram of HPLC analysis for partition coefficient by HPLC method.
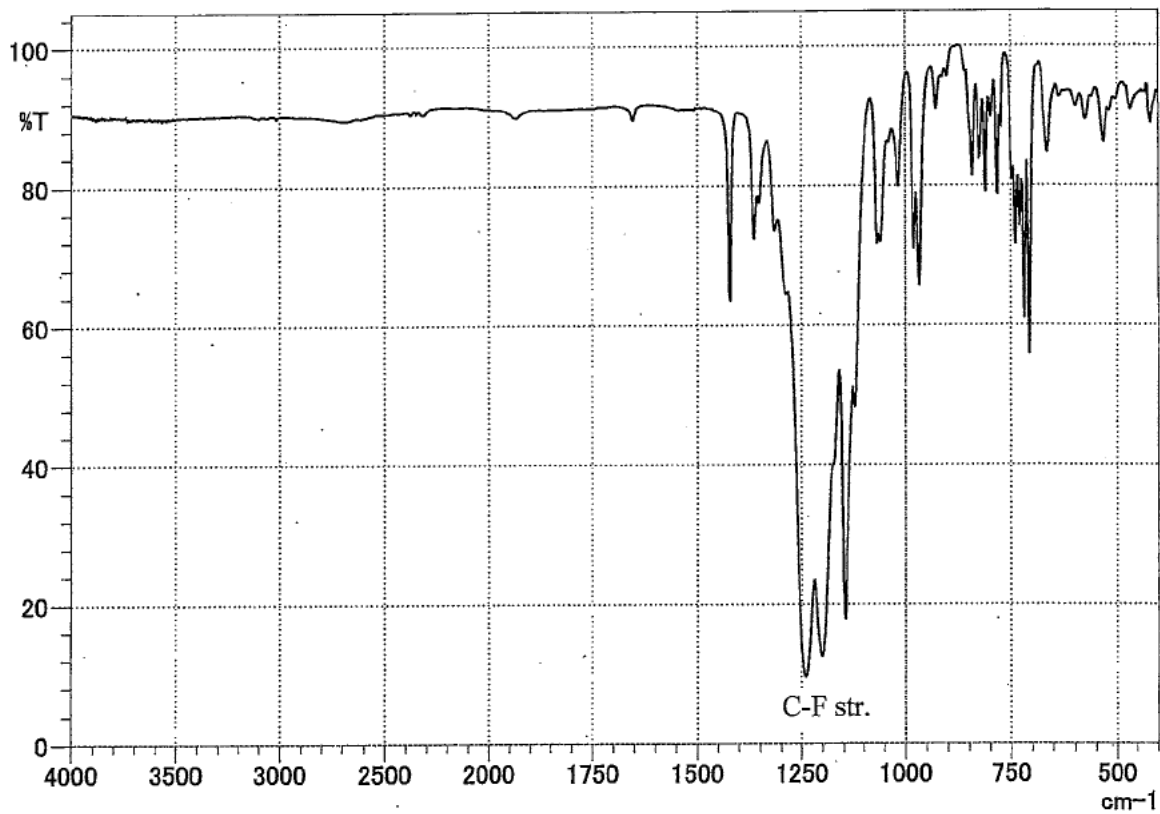
Solvent blank

Operator:	
Operating date:	14/Mar/2017
Sample ID:	84999_170313_7
Program:	84999pro_testitem_0313
Vial No.:	1_4
Channel:	RI_1
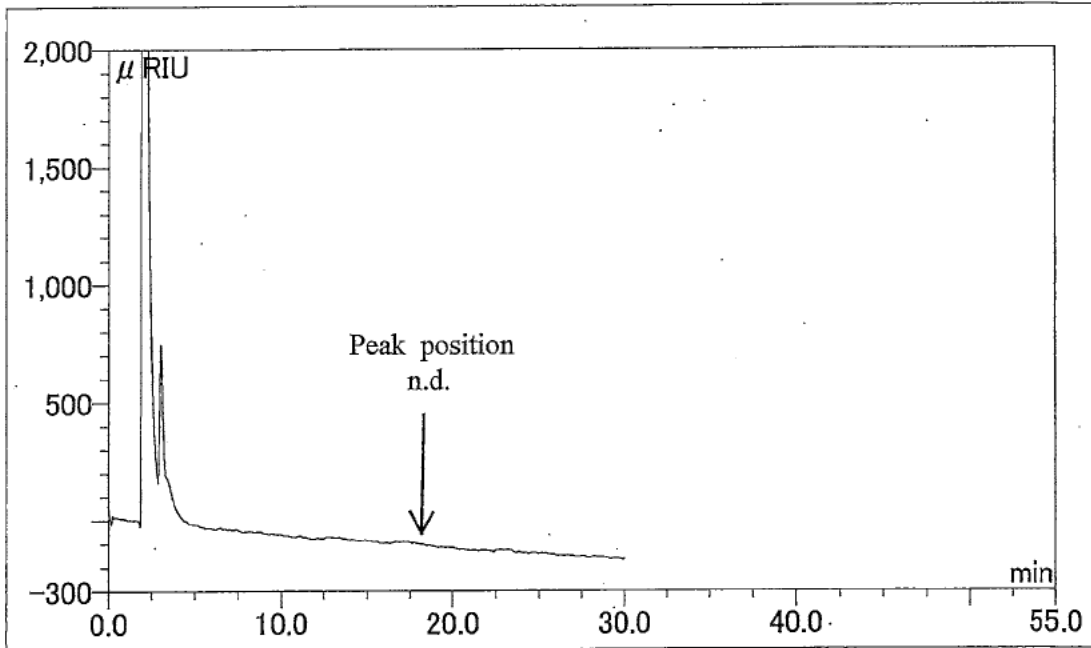
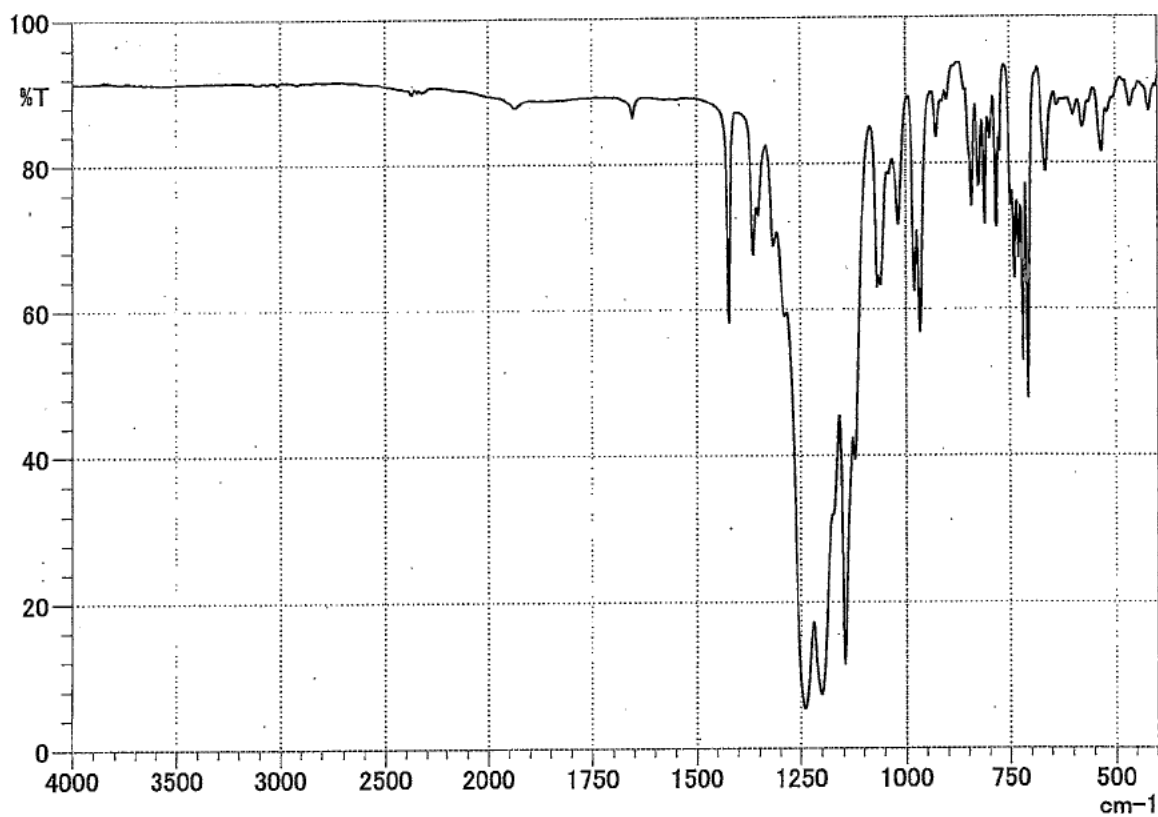| Peak No.	Time (min)	Height (μRIU)	Area (μRIU·sec)	Area (%)
Total	–	–	0	0.00

2017. 3. 14

Fig. 2-7   Chromatogram of HPLC analysis for partition coefficient by HPLC method.

C-F str.

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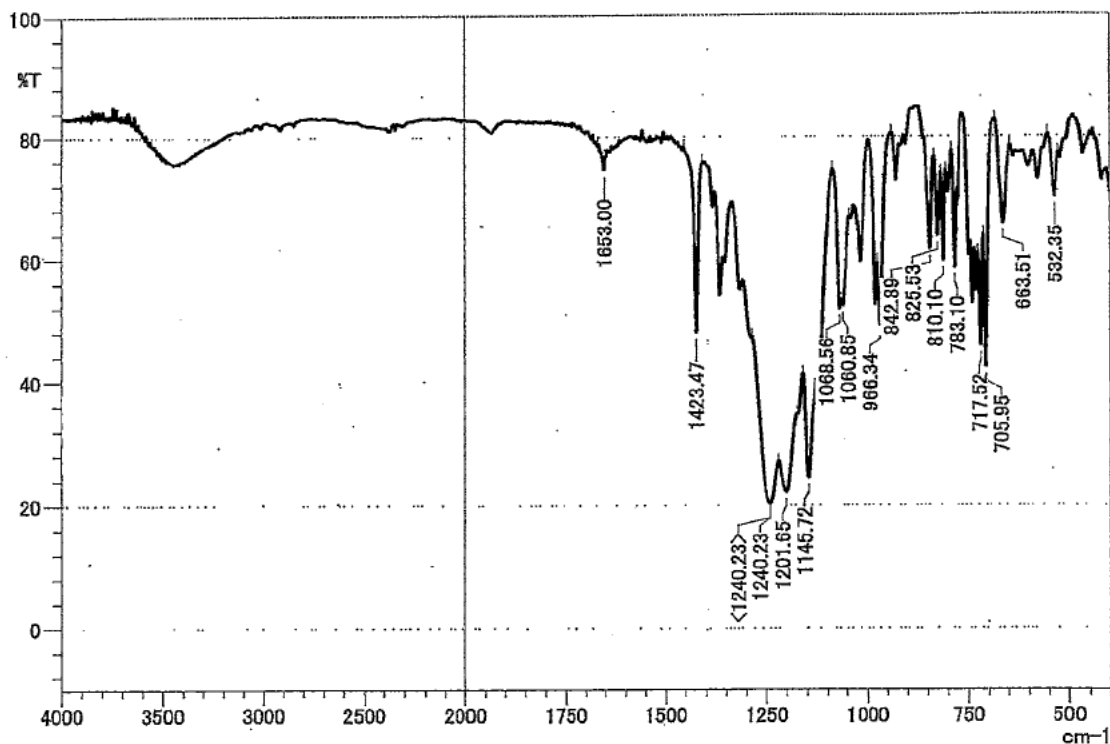Date : February 7, 2017
Name :

Fig. 3 - 1   IR spectrum of test item measured before experimental start.

Instrument : Shimadzu IRAffinity-1S
Study No. : 84999
Sample : Test item
Method : Neat
Date : March 14, 2017
Name :

Fig. 3 - 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	1128.43	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.