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Study number	85530

TEST REPORT

Measurement of water solubility for PFHxA

This is a correct copy of the original. Chemicals Evaluation and Research Institute, Japan, Kurume (CERI Kurume)
Date. <i>March 2, 2018</i>
Study Director / <i>Dr. Kawashima</i>

March, 2018

Chemicals Evaluation and Research Institute, Japan, Kurume

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1. Title

Measurement of water solubility 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 water solubility of PFHxA.

5. Test method

OECD Guidelines for Testing of Chemicals, No.105, July 27, 1995, "Water Solubility: Flask method"

6. Dates

Study initiation date January 29, 2018

Study completion date March 2, 2018

7. Personnel

Study Director Hiroko Kawashima (Section 5)

Study personnel Akemi Inoue

8. Approval of test report

Date

March 2, 2018

Study Director

H. Kawashima

Hiroko Kawashima

9. Summary

Test item

PFHxA

Objective

This study was performed to obtain the water solubility of PFHxA.

Test method

OECD Guidelines for Testing of Chemicals, No.105, July 27, 1995, "Water Solubility: Flask method"

Test conditions

Test water	Purified water
Agitation temperature	30°C
Agitation period	1 day
Test temperature	20±0.5°C
Equilibrium time	24 hours
Number of repetition	3
Analytical method	High-performance liquid chromatography (HPLC)

Result

Water solubility of test item > 250 g/L (20°C)

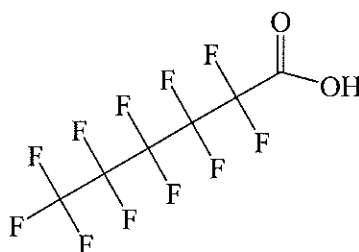
10. Test item

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

The purity of the test item was treated as 100%.

d) Physicochemical property

Appearance	White powder
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e) Storage condition

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

11. Performance of test

11.1 Summary of test method

The water solubility of test item was measured by the flask method.

In this study, it was confirmed that the water solubility of test item was more than the upper limit (250 g/L), because the test item dissolved extremely in water. Therefore, the agitation period of test solution was nominated at only 1 day.

11.2 Test instruments and apparatuses

Water bath incubator	WS-240 (SHIBATA SCIENTIFIC TECHNOLOGY LTD.)
Low constant temperature water bath	TBL320AA (Advantec Toyo Kaisha, Ltd.)
Refrigerated centrifuge	RSL-IV (SAKUMA)
Test vessel	Erlenmeyer flask with glass stopper
Multi function water quality meter	MM-60R(DKK-TOA)

11.3 Test conditions

Test water	Purified water (Takasugi Pharmaceutical, The Japanese Pharmacopoeia)
Agitation temperature	30°C
Agitation period	1 day
Test temperature	20±0.5°C
Equilibration time	24 hours
Number of repetition	3

11.4 Test procedures

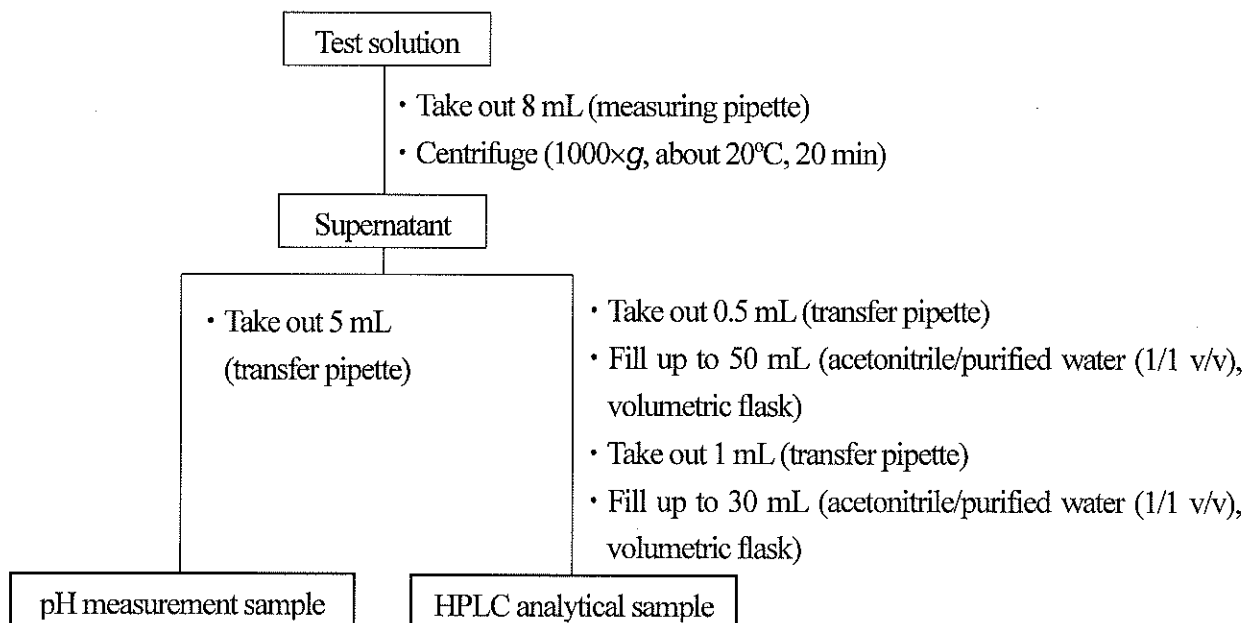
- The test sample (3.00 g) was weighed into each test vessel. Purified water (10 mL) was added into each test vessel to prepare a test solution. Three test solutions were prepared.
- These test solutions were sealed and agitated at 30°C with the shaking bath.
- After 1 day, three test solutions were taken out and equilibrated for 24 hours at the test temperature using the low constant temperature water bath.
- These test solutions were pretreated according to the flow scheme in Section 11.5.1 and analyzed according to the analytical conditions in Section 11.5.2.

11.5 Analysis of test solution

11.5.1 Pretreatment of test solution

The test solutions were pretreated to prepare the high-performance liquid chromatography (HPLC) analytical samples as follows. The pH of each test solution was measured.

Flow scheme



11.5.2 Determination of test item

The test item was analyzed by HPLC.

a) Method of determination

The test item was determined by the absolute calibration curve method using one concentration of the standard solution.

In order to confirm the validity of this determination method, a calibration curve was made using four concentrations of the standard solution, 20.1, 101, 201 and 403 mg/L (see Fig. 1). It was confirmed that the regression line of the calibration curve was a straight line from the origin.

b) Analytical conditions

Instrument	High-performance liquid chromatograph (No. LC-137) LC-2010AHT (Built-in ultraviolet and visible spectrophotometer) (Shimadzu)
Column	L-column ODS (150 mm × 2.1 mm I.D., particle size 5 μm, Chemicals Evaluation and Research Institute, Japan)
Column temperature	40°C
Eluent	A (45%): Purified water/0.5 mol/L tetra- <i>n</i> -butylammonium phosphate (100/2 v/v) B (55%): Acetonitrile
Flow rate	0.2 mL/min
Measurement wavelength	200 nm
Sample size	2 μL

c) Preparation of standard solution and calculation of concentration

The test sample (20.25 mg) was weighed with an electronic analytical balance and dissolved in acetonitrile to obtain 1010 mg/L solution of the test item (20 mL). The standard solution (203 mg/L) was prepared from this solution by dilution with acetonitrile/purified water (1/1 v/v).

The concentration of test item in the HPLC analytical sample was calculated proportionally by comparing the peak area of the HPLC analytical sample with that of 203 mg/L standard solution (see Table 1).

The limit of quantification for test item concentration in the HPLC analytical sample was regarded as 20.1 mg/L, corresponding to the minimum concentration of standard solution used for the calibration curve.

11.6 Calculation of water solubility

The water solubility of test item was determined as >250 g/L, because the measured concentration of test item was more than 250 g/L.

11.7 Treatment of numerical values

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

12. Results

12.1 Test results

Test results are shown as follows.

Sample	pH	Concentration of test item in test solution (g/L)			Table
		Measured value	Average	Standard deviation	
Sample 1	0.6	257	254	3.47	1
Sample 2	0.6	255			
Sample 3	0.6	251			

12.2 Water solubility of test item

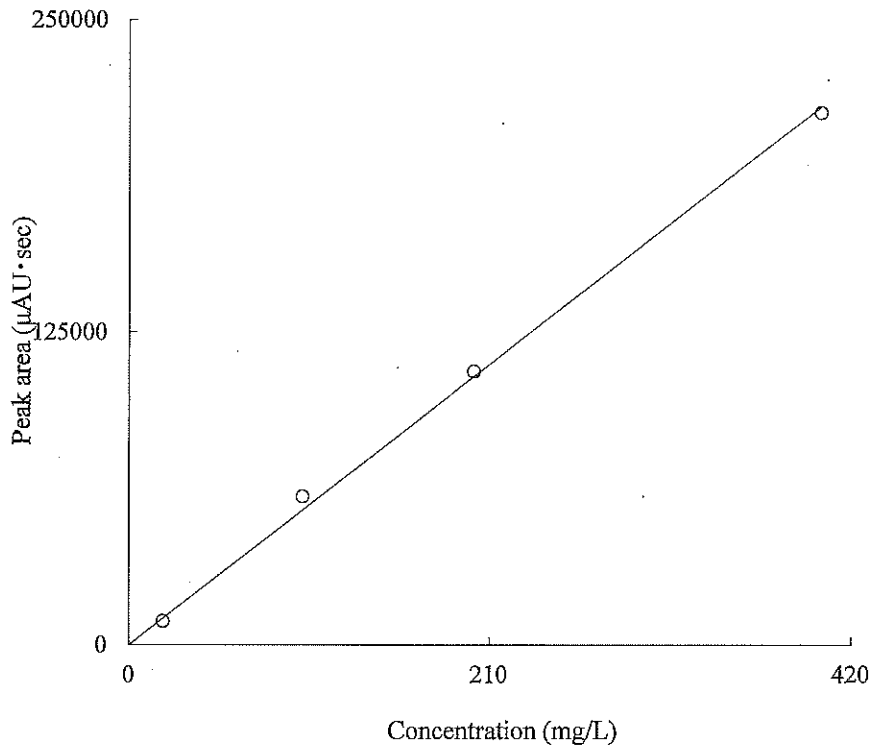
Water solubility >250 g/L (20°C)

Table 1 Calculation table for water solubility

Study No. 85530

Sample description	A	B
Standard solution 203 mg/L	109235	
Sample 1	46167	257
Sample 2	45744	255
Sample 3	44942	251
	Average =	254
	Standard deviation =	3.47
(a, b : individual sample)		
<p>A : Peak area ($\mu\text{AU}\cdot\text{sec}$) A(standard) : Standard solution A(sample) : Sample</p> <p>B : Concentration of test item (g/L) $B = C \times A(\text{sample}) / A(\text{standard}) \times D / 1000$</p> <p>C : Concentration of test item in standard solution 203 (mg/L) D : Dilution factor 3000</p> <p>See Fig.2</p>		

February 28, 2018 Name 1st Kawashima



$y = 534x$
 $r = 0.999$

Concentration (mg/L)	Peak area (μAU·sec)
20.1	9614
101	59449
201	109419
403	212935

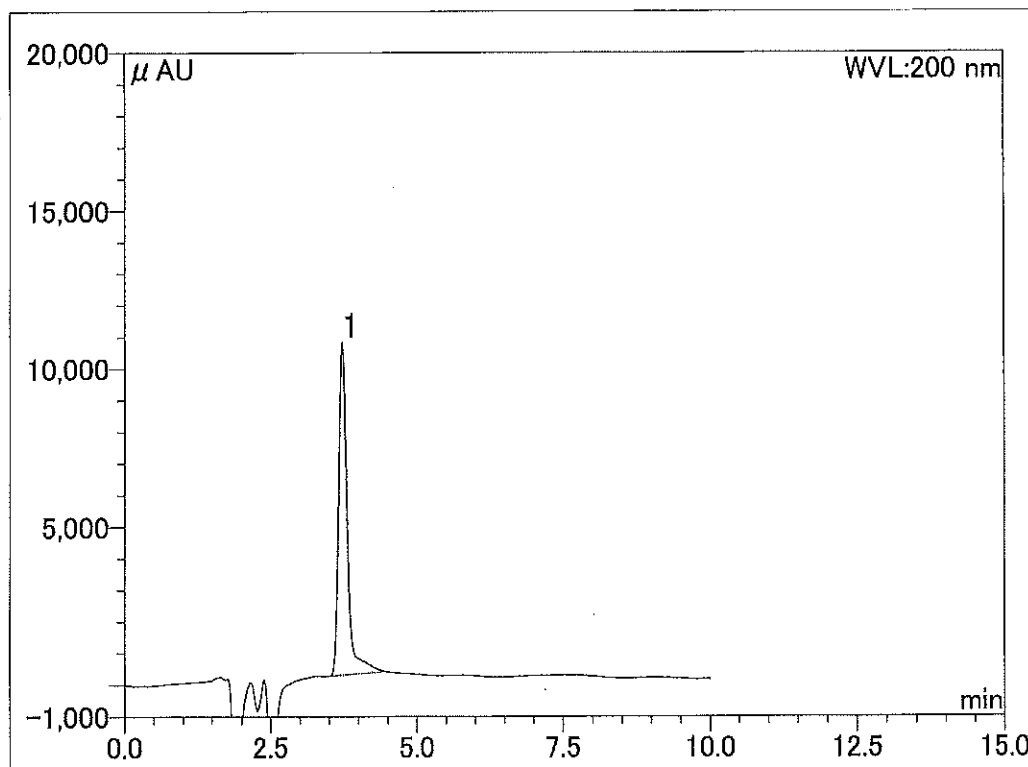
Fig. 1 Calibration curve of test item.

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Name a. inoue

Standard solution 203 mg/L

Operator:	Akemi Inoue
Operating date:	21/Feb/2018
Sample ID:	85530_180221_1
Program:	85530pro0221
Vial No.:	1_1
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	3.72	10513	109235	100.00
Total	-	-	109235	100.00

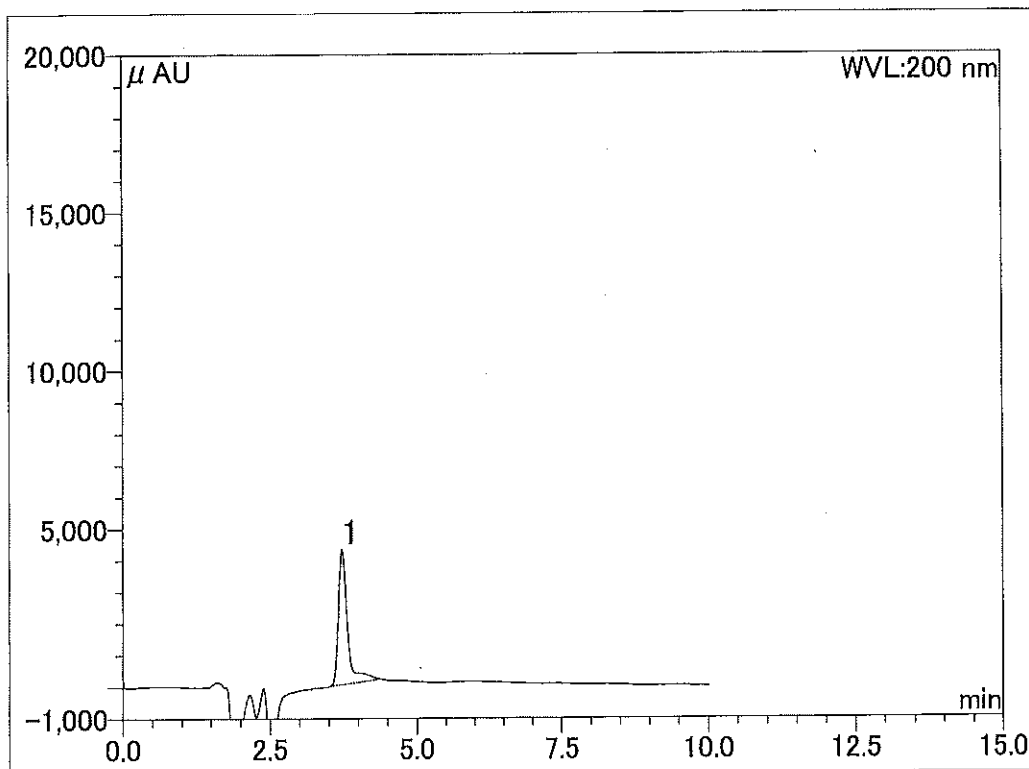


2018. 2. 21 a. inoue

Fig. 2 - 1 Chromatogram of HPLC analysis for water solubility.

Sample 1

Operator:	Akemi Inoue
Operating date:	21/Feb/2018
Sample ID:	85530_180221_2
Program:	85530pro0221
Vial No.:	1_2
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	3.72	4265	46167	100.00
Total	-	-	46167	100.00

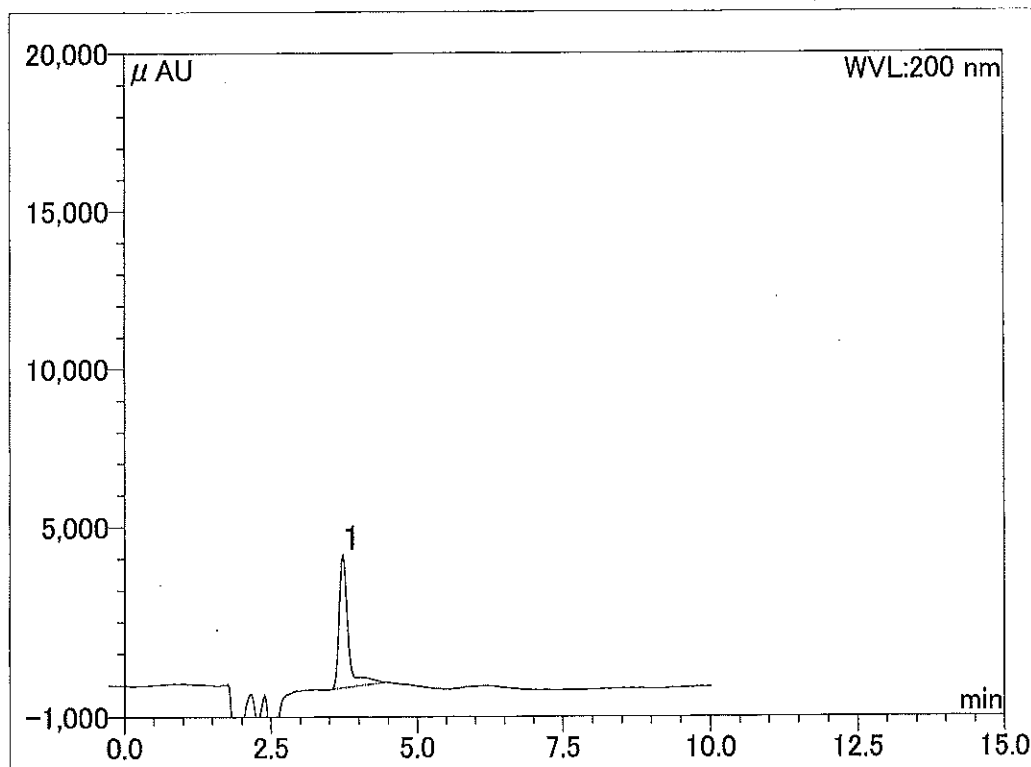


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Fig. 2 - 2 Chromatogram of HPLC analysis for water solubility.

Sample 2

Operator:	Akemi Inoue
Operating date:	21/Feb/2018
Sample ID:	85530_180221_3
Program:	85530pro0221
Vial No.:	1_3
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	3.73	4198	45744	100.00
Total	-	-	45744	100.00

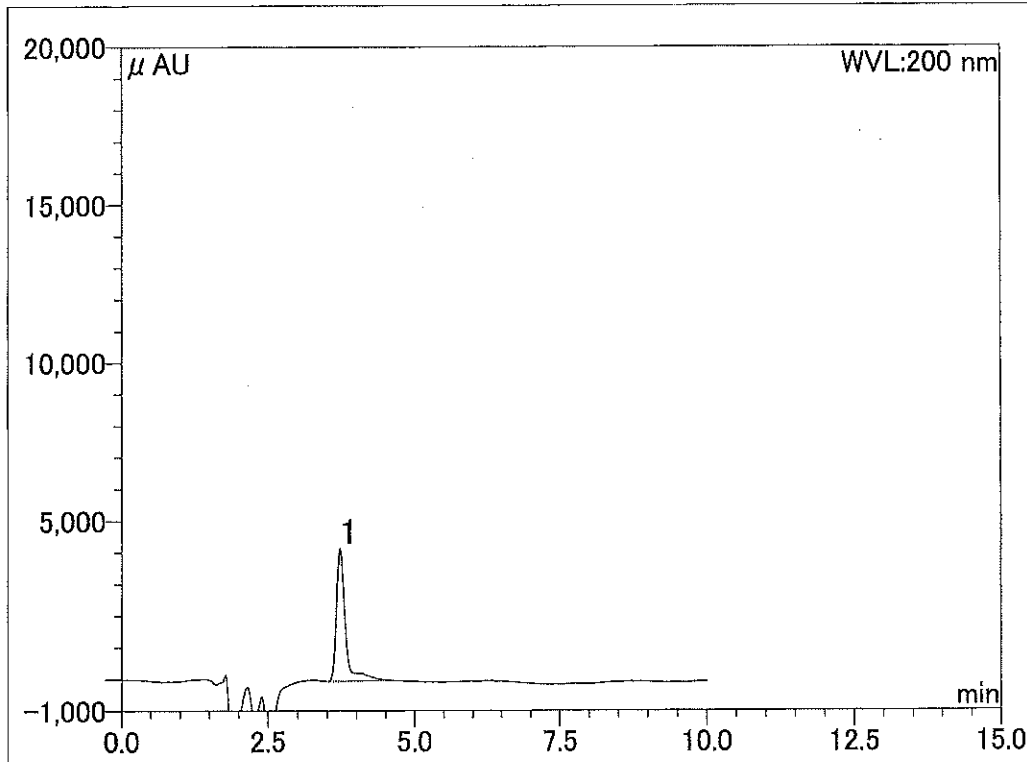


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Fig. 2 - 3 Chromatogram of HPLC analysis for water solubility.

Sample 3

Operator:	Akemi Inoue
Operating date:	21/Feb/2018
Sample ID:	85530_180221_4
Program:	85530pro0221
Vial No.:	1_4
Channel:	UV_VIS_1



Peak No.	Time (min)	Height (μ AU)	Area (μ AU·sec)	Area (%)
1	3.72	4177	44942	100.00
Total	-	-	44942	100.00

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Fig. 2 - 4 Chromatogram of HPLC analysis for water solubility.