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TEST REPORT

Measurement of dissociation constants in water for PFHxA

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

Date *March 1, 2018*

Study Director *I. Kawashima*

March, 2018

Chemicals Evaluation and Research Institute, Japan, Kurume

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1. Title

Measurement of dissociation constants in water 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 dissociation constants in water of PFHxA.

5. Test method

OECD Guidelines for Testing of Chemicals, No.112, May 12, 1981, "Dissociation constants in water: Titration Method"

6. Dates

Study initiation date February 16, 2018

Study completion date March 1, 2018

7. Personnel

Study Director Hiroko Kawashima (Section 5)

Study personnel Tomoko Nakayoshi

8. Approval of test report

Date

march 1, 2018

Study Director

H. Kawashima

Hiroko Kawashima

9. Summary

Test item

PFHxA

Objective

This study was performed to obtain the dissociation constants in water of PFHxA.

Test method

OECD Guidelines for Testing of Chemicals, No.112, May 12, 1981, "Dissociation constants in water:

Titration Method"

Test conditions

Test equipment	Auto titrator
Test concentration	400 mg/L (1.27×10^{-3} mol/L)
Volume of test solution	30 mL
Titrant	0.01 mol/L sodium hydroxide solution (f=1.0, 20°C)
Test temperature	20±1°C
Number of repetition	3

Results

Dissociation constant in water of test item

pKa = 3.29 (20°C)

10. Test item

a) Chemical name etc.

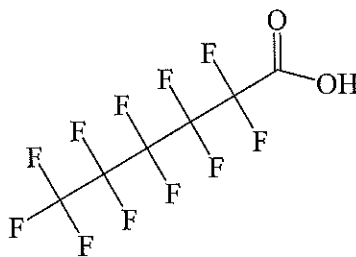
Chemical name 2,2,3,3,4,4,5,5,6,6,6-undecafluorohexanoic acid

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

d) Physicochemical property

Melting point $15^{\circ}C$

Appearance White powder

e) Storage conditions

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

11. Performance of test

11.1 Test equipment

Auto titrator AUT-701 (DKK-TOA)

11.2 Test conditions

Test concentration 400 mg/L (1.27×10^{-3} mol/L)
 Volume of test solution 30 mL
 Titrant 0.01 mol/L sodium hydroxide solution ($f=1.0$, 20°C)
 Test temperature $20 \pm 1^\circ\text{C}$
 Number of repetition 3

11.3 Test procedures

- The test sample (100 mg) was precisely weighed with an electronic analytical balance and dissolved in purified water by means of agitation, and then filled up to 250 mL to obtain 400 mg/L solution of the test item.
- The test item solution (30 mL) was taken into a beaker with a transfer pipette and used as a test solution.
- Three test solutions were titrated with 0.01 mol/L sodium hydroxide solution. The addition volume of titrant and the pH of test solution were measured.

11.4 Calculation of dissociation constant

The dissociation constant (pK_a) of the test item was calculated from the addition volume of the titrant and the pH of the test solution at ten measurement points which were centered on the half volume of the equivalence point, using the following equation (see Table 1). The average of three measured values was regarded as the pK_a value of the test item.

$$\text{pK}_a = \text{pH} - \log \left(\frac{[\text{A}^-]}{[\text{HA}]}\right)$$

$[\text{A}^-]$: Concentration of test item as dissociated form (mmol/L)

$$[\text{A}^-] = \frac{C_t \times F \times V_t}{V + V_t}$$

$[\text{HA}]$: Concentration of the test item as undissociated form (mmol/L)

$$[\text{HA}] = \frac{C \times V}{V + V_t} - [\text{A}^-]$$

Where,

C_t : Concentration of titrant (mmol/L)

F : Factor of titrant

V_t : Addition volume of titrant (mL)

V : Initial volume of test solution (mL)

C : Initial concentration of test item in test solution (mmol/L)

11.5 Treatment of numerical values

Values were rounded off to two decimal places in accordance with Japanese Industrial Standards (JIS) Z 8401-1999 rule B.

12. Results

The test results are shown as follows.

Number of measurement	Dissociation constant		Table
	Measured value	Average	
1	3.29	3.29	1-1
2	3.30		1-2
3	3.29		1-3

Table 1-1 Calculation table of dissociation constant (titrant : 0.01 mol/L NaOH, at first time)

Study No. 85531

Sample	A	B	H	I	J	
1	0.80	3.04	0.26	0.98	3.62	
2	1.05	3.08	0.34	0.89	3.50	
3	1.30	3.12	0.42	0.80	3.41	
4	1.55	3.17	0.49	0.72	3.33	
5	1.80	3.22	0.57	0.63	3.27	
6	2.00	3.26	0.63	0.57	3.22	
7	2.25	3.33	0.70	0.48	3.17	
8	2.45	3.39	0.76	0.42	3.13	
9	2.60	3.45	0.80	0.37	3.12	
10	2.70	3.48	0.83	0.34	3.09	
					Average =	3.29
					Standard deviation =	0.18

A : Added volume of titrant (mL)
 B : pH
 C : Volume of test item solution 30 mL
 D : Concentration of test item in test solution 1.27 mmol/L
 E : Concentration of test item after titrant added (mmol/L)

$$E = C \times D / (A + C)$$

 F : Concentration of titrant 0.01 mol/L
 G : Factor of titrant (NaOH) 1.0
 H : Concentration of dissociated form (mmol/L)

$$H = F \times G \times A / (A + C) \times 1000$$

 I : Concentration of undissociated form (mmol/L)

$$I = E - H$$

 J : Dissociation constant in water (pKa)

$$J = \text{pH} - \log (H / I)$$

 See Fig. 1

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Table 1-2 Calculation table of dissociation constant (titrant : 0.01 mol/L NaOH, at second time)

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Sample	A	B	H	I	J
1	0.80	3.05	0.26	0.98	3.63
2	1.05	3.09	0.34	0.89	3.51
3	1.30	3.12	0.42	0.80	3.41
4	1.55	3.18	0.49	0.72	3.34
5	1.75	3.21	0.55	0.65	3.28
6	2.00	3.28	0.63	0.57	3.24
7	2.20	3.33	0.68	0.50	3.19
8	2.45	3.41	0.76	0.42	3.15
9	2.65	3.48	0.81	0.36	3.12
10	2.80	3.55	0.85	0.31	3.11
Average =					3.30
Standard deviation =					0.17

A : Added volume of titrant (mL)
 B : pH
 C : Volume of test item solution 30 mL
 D : Concentration of test item in test solution 1.27 mmol/L
 E : Concentration of test item after titrant added (mmol/L)

$$E = C \times D / (A + C)$$
 F : Concentration of titrant 0.01 mol/L
 G : Factor of titrant (NaOH) 1.0
 H : Concentration of dissociated form (mmol/L)

$$H = F \times G \times A / (A + C) \times 1000$$
 I : Concentration of undissociated form (mmol/L)

$$I = E - H$$
 J : Dissociation constant in water (pKa)

$$J = \text{pH} - \log (H / I)$$
 See Fig. 2

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Table 1-3 Calculation table of dissociation constant (titrant : 0.01 mol/L NaOH, at third time)

Study No. 85531

Sample	A	B	H	I	J
1	0.80	3.06	0.26	0.98	3.64
2	1.05	3.09	0.34	0.89	3.51
3	1.30	3.13	0.42	0.80	3.42
4	1.55	3.18	0.49	0.72	3.34
5	1.80	3.23	0.57	0.63	3.28
6	2.05	3.29	0.64	0.55	3.22
7	2.25	3.34	0.70	0.48	3.18
8	2.50	3.42	0.77	0.40	3.14
9	2.70	3.50	0.83	0.34	3.11
10	2.85	3.57	0.87	0.29	3.10
Average =					3.29
Standard deviation =					0.18
Total average =					3.29
<p>A : Added volume of titrant (mL)</p> <p>B : pH</p> <p>C : Volume of test item solution 30 mL</p> <p>D : Concentration of test item in test solution 1.27 mmol/L</p> <p>E : Concentration of test item after titrant added (mmol/L)</p> <p style="padding-left: 20px;">$E = C \times D / (A + C)$</p> <p>F : Concentration of titrant 0.01 mol/L</p> <p>G : Factor of titrant (NaOH) 1.0</p> <p>H : Concentration of dissociated form (mmol/L)</p> <p style="padding-left: 20px;">$H = F \times G \times A / (A + C) \times 1000$</p> <p>I : Concentration of undissociated form (mmol/L)</p> <p style="padding-left: 20px;">$I = E - H$</p> <p>J : Dissociation constant in water (pKa)</p> <p style="padding-left: 20px;">$J = \text{pH} - \log (H / I)$</p> <p>See Fig. 3</p>					

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Table 2 Titration data (titrant : 0.01 mol/L NaOH, at first time)

Study No. 85531

Sample No.1					Operating date: February 16, 2018 Equivalence point 3.757 mL				
Intermediate data	Added volume of titrant (mL)	pH	Mode	Temp. (°C)	Intermediate data	Added volume of titrant (mL)	pH	Mode	Temp. (°C)
1	0.00	2.94	pH(MTC)	20.0	46	4.55	10.50	pH(MTC)	20.0
2	0.05	2.95	pH(MTC)	20.0	47	4.70	10.60	pH(MTC)	20.0
3	0.10	2.95	pH(MTC)	20.0	48	4.80	10.63	pH(MTC)	20.0
4	0.20	2.96	pH(MTC)	20.0	49	4.95	10.69	pH(MTC)	20.0
5	0.35	2.98	pH(MTC)	20.0	50	5.05	10.73	pH(MTC)	20.0
6	0.55	3.01	pH(MTC)	20.0	51	5.20	10.80	pH(MTC)	20.0
7	0.80	3.04	pH(MTC)	20.0	52	5.30	10.82	pH(MTC)	20.0
8	1.05	3.08	pH(MTC)	20.0	53	5.45	10.83	pH(MTC)	20.0
9	1.30	3.12	pH(MTC)	20.0	54	5.65	10.89	pH(MTC)	20.0
10	1.55	3.17	pH(MTC)	20.0	55	5.80	10.94	pH(MTC)	20.0
11	1.80	3.22	pH(MTC)	20.0	56	6.00	10.96	pH(MTC)	20.0
12	2.00	3.26	pH(MTC)	20.0	57	6.25	11.00	pH(MTC)	20.0
13	2.25	3.33	pH(MTC)	20.0	58	6.50	11.03	pH(MTC)	20.0
14	2.45	3.39	pH(MTC)	20.0	59	6.75	11.07	pH(MTC)	20.0
15	2.60	3.45	pH(MTC)	20.0	60	7.00	11.11	pH(MTC)	20.0
16	2.70	3.48	pH(MTC)	20.0	61	7.25	11.14	pH(MTC)	20.0
17	2.85	3.56	pH(MTC)	20.0	62	7.50	11.15	pH(MTC)	20.0
18	2.95	3.61	pH(MTC)	20.0	63	7.75	11.18	pH(MTC)	20.0
19	3.00	3.64	pH(MTC)	20.0	64	8.00	11.23	pH(MTC)	20.0
20	3.10	3.71	pH(MTC)	20.0					
21	3.15	3.74	pH(MTC)	20.0					
22	3.25	3.82	pH(MTC)	20.0					
23	3.30	3.88	pH(MTC)	20.0					
24	3.35	3.93	pH(MTC)	20.0					
25	3.40	4.00	pH(MTC)	20.0					
26	3.45	4.08	pH(MTC)	20.0					
27	3.50	4.18	pH(MTC)	20.0					
28	3.55	4.31	pH(MTC)	20.0					
29	3.60	4.50	pH(MTC)	20.0					
30	3.65	4.82	pH(MTC)	20.0					
31	3.70	5.62	pH(MTC)	20.0					
32	3.75	6.86	pH(MTC)	20.0					
33	3.80	8.30	pH(MTC)	20.0					
34	3.85	9.15	pH(MTC)	20.0					
35	3.90	9.52	pH(MTC)	20.0					
36	3.95	9.75	pH(MTC)	20.0					
37	4.00	9.89	pH(MTC)	20.0					
38	4.05	10.01	pH(MTC)	20.0					
39	4.10	10.09	pH(MTC)	20.0					
40	4.15	10.16	pH(MTC)	20.0					
41	4.20	10.22	pH(MTC)	20.0					
42	4.25	10.28	pH(MTC)	20.0					
43	4.30	10.32	pH(MTC)	20.0					
44	4.40	10.41	pH(MTC)	20.0					
45	4.45	10.45	pH(MTC)	20.0					

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Table 3 Titration data (titrant : 0.01 mol/L NaOH, at second time)

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Sample No.2					Operating date: February 16, 2018 Equivalence point 3.743 mL				
Intermediate data	Added volume of titrant (mL)	pH	Mode	Temp. (°C)	Intermediate data	Added volume of titrant (mL)	pH	Mode	Temp. (°C)
1	0.00	2.98	pH(MTC)	20.0	46	4.55	10.56	pH(MTC)	20.0
2	0.05	2.97	pH(MTC)	20.0	47	4.60	10.59	pH(MTC)	20.0
3	0.10	2.97	pH(MTC)	20.0	48	4.70	10.65	pH(MTC)	20.0
4	0.20	2.98	pH(MTC)	20.0	49	4.75	10.67	pH(MTC)	20.0
5	0.35	2.99	pH(MTC)	20.0	50	4.85	10.70	pH(MTC)	20.0
6	0.55	3.02	pH(MTC)	20.0	51	5.00	10.77	pH(MTC)	20.0
7	0.80	3.05	pH(MTC)	20.0	52	5.05	10.76	pH(MTC)	20.0
8	1.05	3.09	pH(MTC)	20.0	53	5.15	10.80	pH(MTC)	20.0
9	1.30	3.12	pH(MTC)	20.0	54	5.30	10.84	pH(MTC)	20.0
10	1.55	3.18	pH(MTC)	20.0	55	5.50	10.88	pH(MTC)	20.0
11	1.75	3.21	pH(MTC)	20.0	56	5.75	10.95	pH(MTC)	20.0
12	2.00	3.28	pH(MTC)	20.0	57	5.95	10.98	pH(MTC)	20.0
13	2.20	3.33	pH(MTC)	20.0	58	6.00	11.01	pH(MTC)	20.0
14	2.45	3.41	pH(MTC)	20.0	59	6.10	11.01	pH(MTC)	20.0
15	2.65	3.48	pH(MTC)	20.0	60	6.25	11.03	pH(MTC)	20.0
16	2.80	3.55	pH(MTC)	20.0	61	6.45	11.06	pH(MTC)	20.0
17	2.90	3.60	pH(MTC)	20.0	62	6.70	11.08	pH(MTC)	20.0
18	2.95	3.63	pH(MTC)	20.0	63	6.95	11.13	pH(MTC)	20.0
19	3.05	3.69	pH(MTC)	20.0	64	7.20	11.15	pH(MTC)	20.0
20	3.10	3.72	pH(MTC)	20.0	65	7.45	11.17	pH(MTC)	20.0
21	3.20	3.80	pH(MTC)	20.0	66	7.70	11.19	pH(MTC)	20.0
22	3.25	3.85	pH(MTC)	20.0	67	7.95	11.21	pH(MTC)	20.0
23	3.30	3.91	pH(MTC)	20.0	68	8.00	11.23	pH(MTC)	20.0
24	3.35	3.97	pH(MTC)	20.0					
25	3.40	4.04	pH(MTC)	20.0					
26	3.45	4.13	pH(MTC)	20.0					
27	3.50	4.23	pH(MTC)	20.0					
28	3.55	4.38	pH(MTC)	20.0					
29	3.60	4.59	pH(MTC)	20.0					
30	3.65	4.96	pH(MTC)	20.0					
31	3.70	5.85	pH(MTC)	20.0					
32	3.75	7.18	pH(MTC)	20.0					
33	3.80	8.49	pH(MTC)	20.0					
34	3.85	9.23	pH(MTC)	20.0					
35	3.90	9.63	pH(MTC)	20.0					
36	3.95	9.82	pH(MTC)	20.0					
37	4.00	9.97	pH(MTC)	20.0					
38	4.05	10.07	pH(MTC)	20.0					
39	4.10	10.17	pH(MTC)	20.0					
40	4.15	10.23	pH(MTC)	20.0					
41	4.20	10.29	pH(MTC)	20.0					
42	4.25	10.35	pH(MTC)	20.0					
43	4.30	10.38	pH(MTC)	20.0					
44	4.40	10.48	pH(MTC)	20.0					
45	4.45	10.50	pH(MTC)	20.0					

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Table 4 Titration data (titrant : 0.01 mol/L NaOH, at third time)

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Sample No.3					Operating date: February 16, 2018				
					Equivalence point 3.728 mL				
Intermediate data	Added volume of titrant (mL)	pH	Mode	Temp. (°C)	Intermediate data	Added volume of titrant (mL)	pH	Mode	Temp. (°C)
1	0.00	3.01	pH(MTC)	20.0	46	4.70	10.56	pH(MTC)	20.0
2	0.05	2.99	pH(MTC)	20.0	47	4.85	10.62	pH(MTC)	20.0
3	0.10	2.99	pH(MTC)	20.0	48	4.95	10.66	pH(MTC)	20.0
4	0.20	2.99	pH(MTC)	20.0	49	5.10	10.71	pH(MTC)	20.0
5	0.35	3.01	pH(MTC)	20.0	50	5.15	10.72	pH(MTC)	20.0
6	0.55	3.03	pH(MTC)	20.0	51	5.25	10.75	pH(MTC)	20.0
7	0.80	3.06	pH(MTC)	20.0	52	5.40	10.78	pH(MTC)	20.0
8	1.05	3.09	pH(MTC)	20.0	53	5.60	10.84	pH(MTC)	20.0
9	1.30	3.13	pH(MTC)	20.0	54	5.85	10.88	pH(MTC)	20.0
10	1.55	3.18	pH(MTC)	20.0	55	6.10	10.93	pH(MTC)	20.0
11	1.80	3.23	pH(MTC)	20.0	56	6.15	10.94	pH(MTC)	20.0
12	2.05	3.29	pH(MTC)	20.0	57	6.25	10.95	pH(MTC)	20.0
13	2.25	3.34	pH(MTC)	20.0	58	6.40	10.96	pH(MTC)	20.0
14	2.50	3.42	pH(MTC)	20.0	59	6.60	10.99	pH(MTC)	20.0
15	2.70	3.50	pH(MTC)	20.0	60	6.85	11.02	pH(MTC)	20.0
16	2.85	3.57	pH(MTC)	20.0	61	7.10	11.06	pH(MTC)	20.0
17	2.95	3.62	pH(MTC)	20.0	62	7.35	11.08	pH(MTC)	20.0
18	3.00	3.64	pH(MTC)	20.0	63	7.60	11.11	pH(MTC)	20.0
19	3.10	3.71	pH(MTC)	20.0	64	7.85	11.13	pH(MTC)	20.0
20	3.15	3.75	pH(MTC)	20.0	65	8.00	11.14	pH(MTC)	20.0
21	3.25	3.84	pH(MTC)	20.0					
22	3.30	3.89	pH(MTC)	20.0					
23	3.40	4.02	pH(MTC)	20.0					
24	3.45	4.11	pH(MTC)	20.0					
25	3.50	4.21	pH(MTC)	20.0					
26	3.55	4.36	pH(MTC)	20.0					
27	3.60	4.57	pH(MTC)	20.0					
28	3.65	4.96	pH(MTC)	20.0					
29	3.70	5.99	pH(MTC)	20.0					
30	3.75	7.58	pH(MTC)	20.0					
31	3.80	8.75	pH(MTC)	20.0					
32	3.85	9.31	pH(MTC)	20.0					
33	3.90	9.61	pH(MTC)	20.0					
34	3.95	9.79	pH(MTC)	20.0					
35	4.00	9.92	pH(MTC)	20.0					
36	4.05	10.02	pH(MTC)	20.0					
37	4.10	10.10	pH(MTC)	20.0					
38	4.15	10.17	pH(MTC)	20.0					
39	4.20	10.21	pH(MTC)	20.0					
40	4.25	10.27	pH(MTC)	20.0					
41	4.30	10.30	pH(MTC)	20.0					
42	4.40	10.41	pH(MTC)	20.0					
43	4.45	10.42	pH(MTC)	20.0					
44	4.55	10.48	pH(MTC)	20.0					
45	4.60	10.51	pH(MTC)	20.0					

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Name 7. Nakayoshi

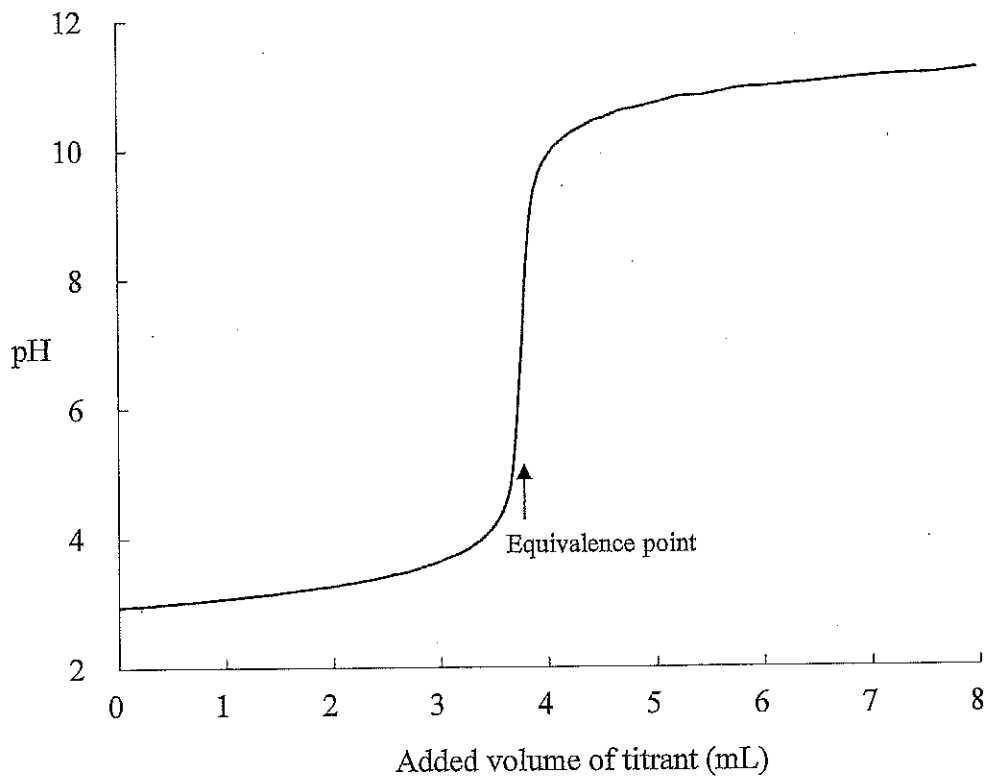


Fig. 1 Titration curve (titrant : 0.01mol/L NaOH, at first time).

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Name T. Nakayoshi

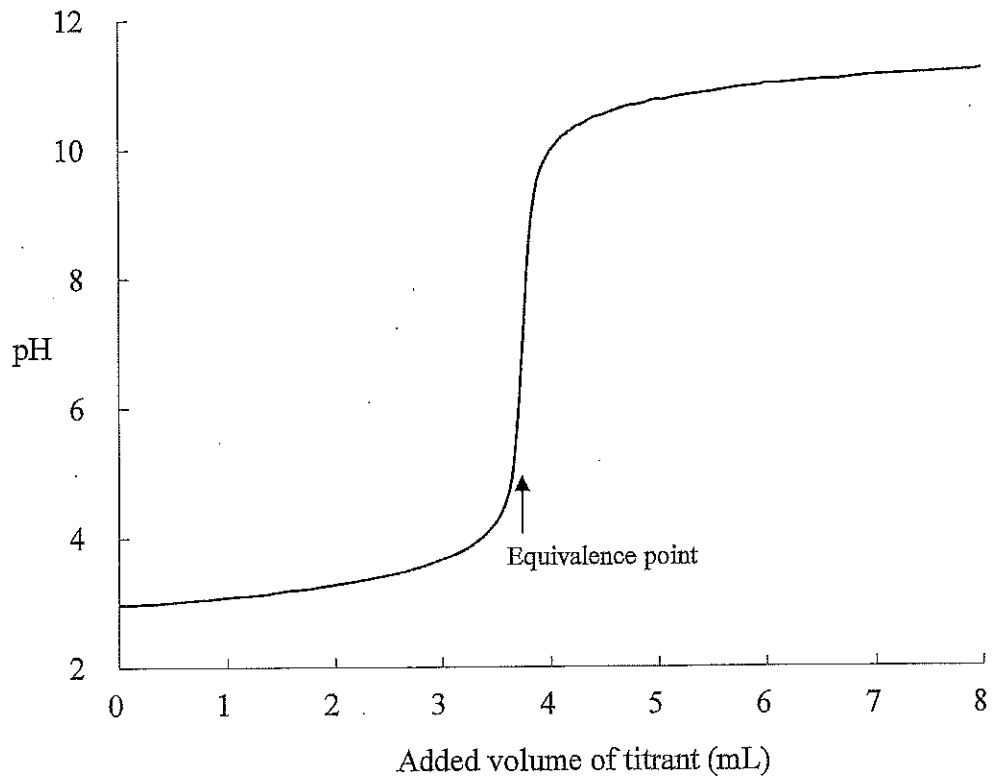


Fig. 2 Titration curve (titrant : 0.01mol/L NaOH, at second time).

February 20, 2018

Name Z. Nakayoshi

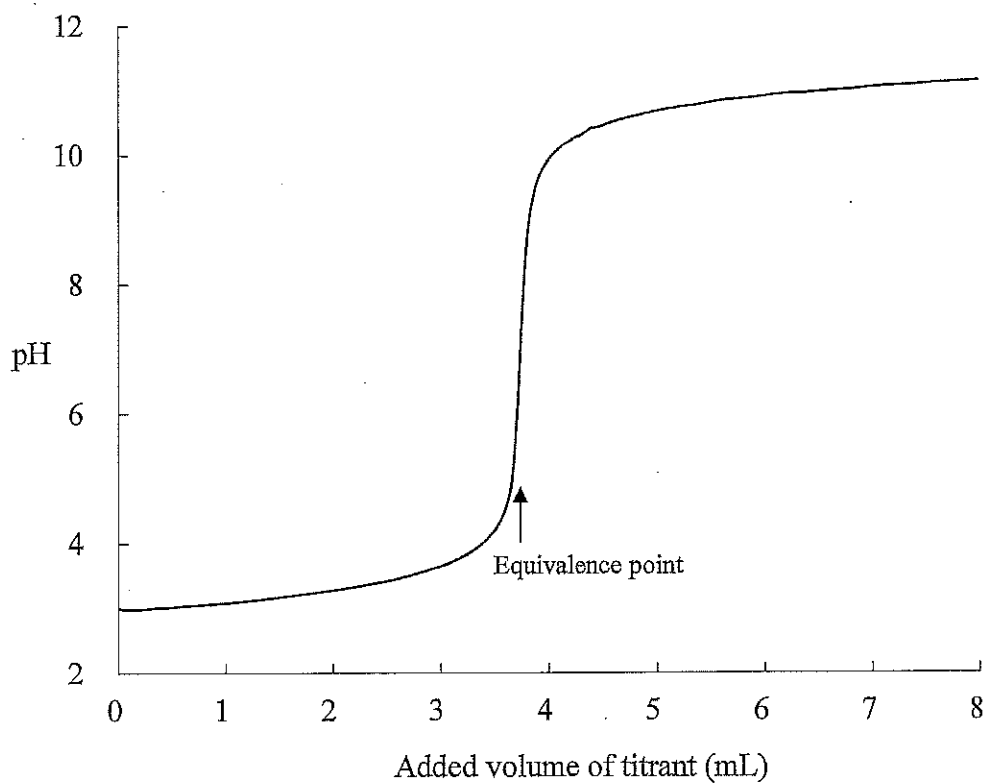


Fig. 3 Titration curve (titrant : 0.01mol/L NaOH, at third time).

February 20, 2018

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