GLP Statement

1. Name of Study: Ready Biodegradability Test of ANO

2. Study No.: B12011

I, the undersigned, hereby certify that this study was conducted in compliance with "Standard for testing facility to conduct studies on new chemical substances" (Yakushokuhatsu 0331 No. 8[1], Heisei 23, 03.29 Seikyoku No. 6[2], Kanhokihatus No. 110331010[3]).

Koei Techno Co., Ltd.

Study Director Seal

Year/Month/Day

The notification was issued by 3 chiefs of the bureaus described below.
1. Ministry of Health, Labor and Welfare, Medicine food bureau
2. Ministry of Economy, Trade and Industry, Production industry of the bureau
3. Ministry of Environment, Synthesis environmental policy of the bureau

FINAL REPORT

(Draft Version)

Ready Biodegradability Test of ANO

(Study No. B12011)

Koei Techno Co., Ltd.

Title: Ready Biodegradability Test of ANO

Study period
From:

To:

Sponsor, name and address:
Repre Co., Ltd.
5-3, Nihonbashi-kayabacho 3 chome, Chuo-ku, Tokyo

Testing facility, name and address
Koei Techno Co., Ltd.
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Study director Seal Year/Month/Day
(Test team)

Testing operator: Yoshikazu Asahi (Ready Biodegradability test)
Mimako Tobimatu (Ready Biodegradability test)

Chiba Testing Facility:
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Chief operator: Minako Yamamoto
Koei Techno Co., Ltd.
Cover Area: Analysis of Inductivity Coupled Plasma – Atomic Emission Spectrometry (ICP – AES)

Author of final report Seal

Date of issue Year/Month/Day

Contents

Purpose of study and summary of the result

As a part of safety assessment of a new substance ANO, a ready biodegradability test was conducted.

The result showed, that the degradability by oxygen demand after 28 days exposure in test substance vessels is -11%[*1]、0%[*1] and 11%[*1] respectively, mean 0%.

The direct quantification was conducted by means of a Liquid Chromatography (LC) for oxalate ion, an Ion Chromatography (IC) for ammonium ion and an Inductive Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)[*2] for niobium ion, since the test item is dissociated to above ions in water. And, based on the result of a preliminary test[*3], the niobium ion is predicted to form a niobium hydroxide (V) which is insoluble degradation product in the test liquid, and hence a retentate of filtered test liquid after the exposure was qualitatively analysed by an Infrared Spectrophotometer (IR). While, the quantification of niobium ion was conducted by ICP-AES after a pretreatment of the test liquid by conc. sulfuric acid.

The analysis showed the following results. The residual rate of oxalate in the test item degradation vessel was mean 0%, in the test item-water was 98% and thus the degradation was mean 100%, of ammonium ion in the test item degradation vessel mean 96%, in the test item-water 98% and thus the degradation was mean 2%, and of niobium ion in the test item degradation vessel mean 0%, in the test item-water 101% and thus the degradation was mean 100%, respectively. Just to be sure, the LC analysis showed no degradation products of more than 1% was found.

An IR analysis was conducted after the exposure of the test item degradation vessel to compare the spectrum with that of standard substance of niobium hydroxide (V), and the identification of both items was confirmed. The generation rate of niobium hydroxide (V) was mean 103% in the test item degradation vessel, and the mass balance of niobium ion and niobium hydroxide in total was 103% in the test item degradation vessel.

The residual rate of DOC was mean 0%[*4] in test item degradation vessels, while 95% in test item-water, and degradation was obtained as mean 104%. And, residual amount of inorganic carbon (IC) was mean 0.8 mg in test item degradation vessels, and the shift of pH towards alkaline was observed, as mean pH8.83.

From the results described above, it is considered that oxalate ion derived from the test item can be degradable by microbes and carbon dioxides generated by the degradation were to retain as IC in the test liquid since the test liquid was alkalinity and hence the IC residual amount wasn't reflected to the oxygen demand. A substantial mineralization rate of the test item by microbes was calculated as mean 18% by using an equation of "Mineralization rate = Degradation (%) by oxygen demand + IC residual rate (residual IC percentage against theoretical DOC) (%)". Further, supplementarily explained, the mineralization rate of mean 18% could not reach to 60% as a criterion of ready biodegradability, and the reason of this lower rate than the expected was considered due to a wide variation of the measurement error attributable to very small values of ANO's theoretical oxygen demand 2.8 mg and the theoretical DOC 4.3 mg.

From analysis results of LC and DOC, it is considered that oxalate as an organic component of ANO is biodegradable and completely mineralized under the present test condition. However, it is also considered that ammonium ion remains as unchanged form, and niobium ion remains as niobium hydroxide (V).

The difference of degradation rate from the maximum to the minimum of replicates was less than 20%, other than calculated by the oxygen demand, and as well, the degradation of aniline

was higher than 40% on 7 days after the exposure and higher than 65% on 14 days after, it was regarded the test was valid.

It is concluded that ANO is not ready biodegradable under the test condition.

*1 The difference of % degradation from the maximum to the minimum of replicates calculated by the oxygen demand was 22%, and this value deviates from the allowance specified as less than 20% in SOP. The reason of this deviation is speculated by a wide variation for the measurement of oxygen demand attributed to the very small theoretical oxygen demand of ANO as 2.8 mg. Since the difference of oxygen demand from maximum to minimum of replicates was 0.6 mg and within the range of coefficient of variation (sd = 1.9 mg in basic respiration vessels obtained by studies in 2012), in this test it seems difficult to judge a validity of the test by using the degradation rate by oxygen demand. Hence, the validity of the test was assessed by LC analysis, IC analysis, ICP-AES analysis and DOC analysis by using their difference from maximum to minimum values of replicates.

*2 ICP-AES analysis was conducted at Chiba Testing Facility by the chef operator.

*3 In a preliminary test, niobium ion in test item degradation vessels formed (an) insoluble product(s). Under the testing condition, it is conjectured to form niobium oxide (V) and niobium hydroxide (V), and its dissolution tests were carried out by using acids and alkalis (conc, sulfuric acid, conc. nitric acid, conc. hydrochloric acid, aqua regia, inverse aqua regia and aqueous ammonia). According to the result that the dissolution was obtained by conc. sulfuric acid, the degradation product was assumed to be niobium hydroxide (V) (Chemical Unabridged Dictionary published by Tokyo Kagaku Dojin). In this study IR analysis was carried out to compare the spectra of the degradation product with that of standard sample of niobium hydroxide (V), resulting the confirmation of identity.

*4 Recorded as 0, because of minus value.

Introduction

As a part of safety assessment of a new substance ANO, a ready biodegradability test was conducted.

Materials and test method

1. Test method

A ready biodegradation test was conducted in accordance with " Degradation Test of Chemical Substance Using Microorganisms"

(Yakushokuhatsu 0331 No. 7[2], Heisei 23, 03.29 Seikyoku No. 5[2], Kanhokihatus No. 110331009[3] , partly revised on 2 April 2012).

2. Test item, components[*5] after dissociation and standard substances

The notification was issued by 3 chiefs of the bureaus described below.
1. Ministry of Health, Labor and Welfare, Medicine food bureau
2. Ministry of Economy, Trade and Industry, Production industry of the bureau
3. Ministry of Environment, Synthesis environmental policy of the bureau

*5 The test item dissociates to oxalate, ammonium and niobium ions in water. Each ion was quantified.

2.1 Test item

Name:	Reaction mass of ammonium oxobis (ethanedioato) bisaquo niobate (V) hydrates and ammonium hydrogen ethanedioate ethanedioic acid dihydrate
Synonym:	Ammonium Niobium Oxalate (abbreviation; ANO)
CAS Number:	-
Chemical formula:	Component 1; $NH_4[NbO(C_2O_4)_2 \cdot 2H_2O] \cdot 3H_2O$ Component 2; $(NH_4C_2HO_4)_2 \cdot (C_2O_4H_2 \cdot 2H_2O)_2$
Molecular formula:	Component 1; $C_4H_{14}NNbO_{14}$ Component 2; $C_8H_{22}N_2O_{20}$
Molecular weight:	368.25 (Component 1; 393.06, Component 2; 466.26)
Lot No.:	AD/4638
Purity:	90% (Component 1; 70.2%, Component 2; 19.8%)
Impurity, Name and Content:	H_2O; 10%
Vapour pressure:	-
Water solubility:	> 5 g/L
1-Octanol/water partition coefficient:	-
Melting point:	-
Boiling point:	-
State at ambient temperature:	ANO is white, water soluble crystalline
Stability:	Stable at ambient temperature
Storage condition:	Ambient temperature
Solubility to solvents:	-
Identification:	Identified by comparison of ANO IR spectrum (Fig.4) with one measured by our lab. (Fig.5)
Stability under storage condition:	Confirmed by IR spectra at the dates of start (Fig.6) and terminate (Fig.7) of exposure
Stability under testing condition:	Not required for degradation test

2.2 Components after dissociation

Name:	Oxalate ion
Synonym:	$C_2O_4^{2-}$
CAS No.:	-
Chemical formula:	$C_2O_4^{2-}$
Molecular formula:	$C_2O_4^{2-}$
Molecular weight:	88.02
Name:	Ammonium ion
Synonym:	NH_4^+
CAS No.:	-
Chemical formula:	NH_4^+
Molecular formula:	NH_4^+
Molecular weight:	18.04
Name:	Niobium ion

Synonym: Nb^{5+}
CAS No.: -
Chemical formula: Nb^{5+}
Molecular formula: Nb^{5+}
Molecular weight: 92.91

The test item was employed as a standard substance for oxalate ion and standard substances described in 2.3 Standard Substance were employed for ammonium ion and niobium ion.

2.3 Standard substances

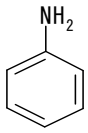
Name: Ammonium Chloride
Lot No.: B07753C (Special Grade, Kishida Chem. Japan)
Purity: 99.7%
Quality, Identification, Stability: Specified as reagent chem.

Name: Niobium ICP Standard solution（NH_4NbF_6 in H2O、1000 mg/L Nb）
Lot No.: HC242206 ICP Standard solution, Merck
Purity: 1001 mg/L
Quality, Identification, Stability: Specified as reagent chem.

Name: Niobium hydroxide (V)
Lot No.: 63515 ICP standard solution, Mitsuwa Chemicals
Purity: 69.2 %
Quality, Identification, Stability: Specified as reagent chem.

3. Control substance

Name: Aniline
Chemical formula



Molecular formula: C_6H_7N
Molecular weight: 93.13
Lot No.: B81714C
Supplier: for Aniline Point, Kishida Chem., Japan
Purity: 99.9%

4. Reagents

Dipotassium hydrogenphospha, Special grade chemical, Kishida Chem. Japan
Potassium dihydrogen-phosphate Special grade chemical, Kishida Chem. Japan
Disodium hydrogen-phosphate 12 H_2O Special grade chemical, Kishida Chem. Japan
Ammonium chloride Special grade chemical, Kishida Chem. Japan
Magnesium sulfite 7 H_2O Special grade chemical, Kishida Chem. Japan
Calcium chloride Special grade chemical, Kishida Chem. Japan
Ferric chloride 6H_2O Special grade chemical, Kishida Chem. Japan

Soda lime	Carbon dioxide absorbent, Kishida Chem. Japan
Potassium hydrogen phthalate	Special grade chemical, Kishida Chem. Japan
Sodium carbonate	Special grade chemical, Kishida Chem. Japan
Sodium bicarbonate	Special grade chemical, Kishida Chem. Japan
Phosphoric acid	Special grade chemical, Wako Pure Chemical Industries Ltd.
Sulfuric acid	Special grade chemical, Wako Pure Chemical Industries Ltd.
Acetonitrile	LC grade, Nacalai Tesque, Inc.
2 mmol/L Methanesulfonate soln,	for Ion chromatography, Wako Pure Chemical Ind.
1 mol/L NaOH soln.	for Volumetric titration , Wako Pure Chemical Ind.
Water	Ultra pure water by own equipment

5. Test organisms

Activated sludge: Standard activated sludge obtained on 11th Jan. 2013 from Chemicals Evaluation and Research Institute, Japan, has been cultured at Koei Techno's Laboratory at condition of temperature 25±2℃, dissolved oxygen >5 mg/L, pH 7.0±1.0 (Sludge No. 201211R).

Suspended solid of the activated sludge (sample taken on 27th Feb. 2013): 3763 mg/L

6. Test equipment

Coulometer: Ohkura Electric Co., Ltd. Model OM-2001A

Data sampler: Asahi Techneion Co., Ltd. DS-3

7. Test condition

Test temperature: 25.0℃

Amount of test liquid: 300 mL

Water of test: Ultra pure water by own equipment

8. Test concentration

Concentration of suspended solid of activated sludge: 30 mg/L

Concentration of test item: 100 mg/L

The following numbers were assigned for detector of coulometer;

1: Aniline, 2: Basic respiration, 3-5: Test item degradation, 6: Test item - water

9. Exposure period

28 days (from 27th Feb. 2013 to 27th March 2013

10. Check items of observation, measurement, inspection and analysis, and their frequency in this test

Observation, once a day, except holidays (9 days in total during the period): temperature, dissolving condition of test item, colour of test liquid, growth of activated sludge

Measurement of oxygen demand: automated continuous measurement for 28 days by coulometer.

Measurement of pH after exposure test: immediately after taking from coulometer after exposure test.

Measurement of residual amount of test item and qualitative analysis of degradation product and its amount:

Measurement of residual amount of test item and qualitative analysis of degradation product and its amount: Test liquids after exposure were pre-treated and analyzed oxalate ion by Liquid Chromatography (LC), ammonium ion by Ion Chromatography (IC) and niobium ion*7 by Inductivity Coupled Plasma – Atomic Emission Spectrometry (ICP – AES) *6.

*6: Analysis of ICP-AES was conducted at Chiba Testing Facility as one of multiple GLP facilities.

*7: Niobium ion in test item vessels formed degradation product (niobium compound). An aliquot of sample was taken for qualitative analysis by IR. The rest was pre-treated for analysis of niobium ion and the degradation amount was determined.

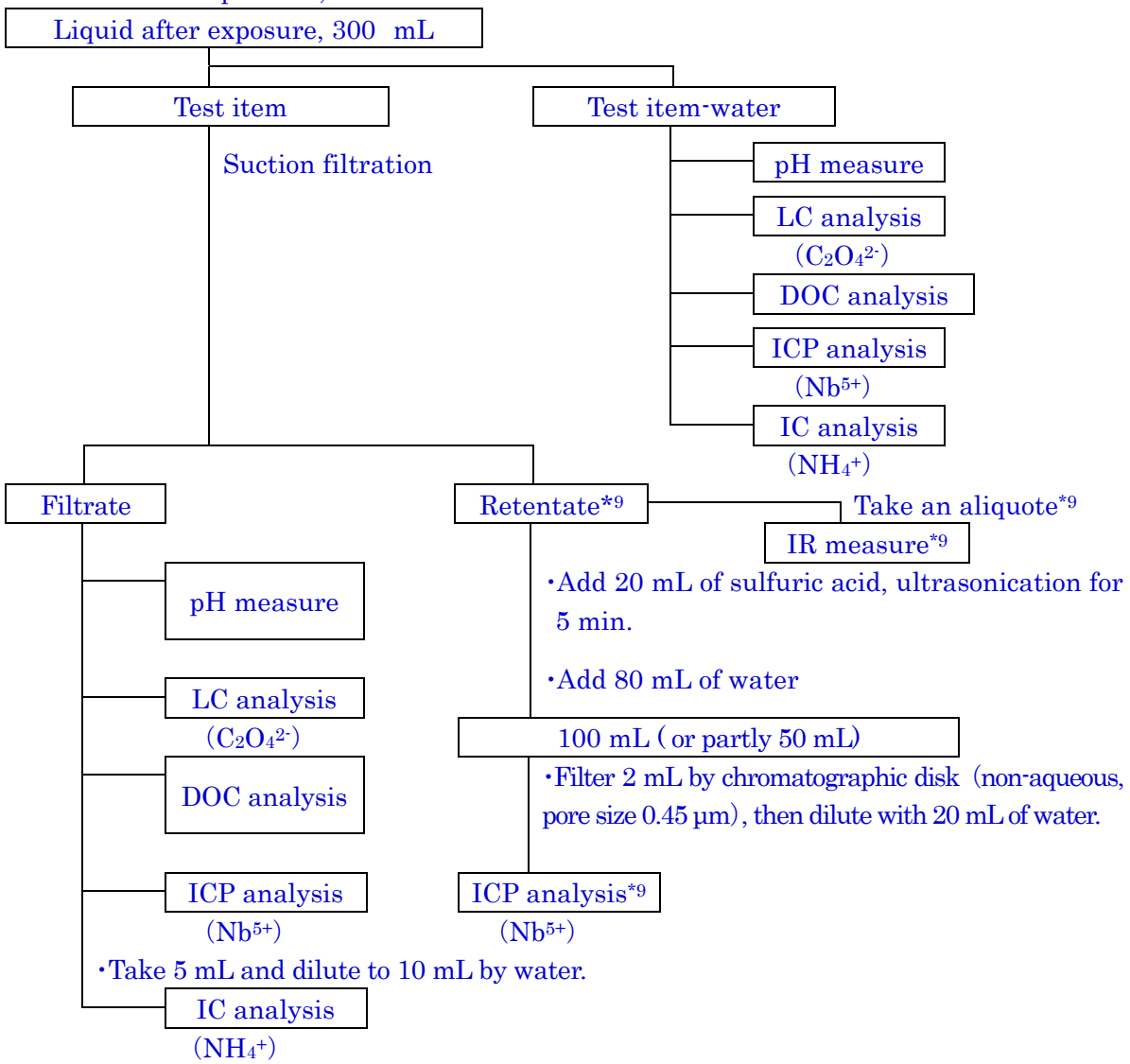
Measurement of dissolved organic carbon (DOC) content in test liquids: analyzed test liquids by TOC analyzer after exposure, since the water solubility of the substance is higher than 100 mg/L*8.

*8: Visual check at the laboratory

11. Measurement method of residual amount of test item and

11.1 Pretreatment of analysis sample

Vessels for basic respiration, test item and test item-water



*9 Since niobium ion was found to be an insoluble substance (niobium compound) in test item vessel, a part was taken for qualitative analysis by IR

spectrophotometry. The rest was pre-treated for niobium ion measurement and the formation of decomposed substance (niobium ion) was measured.

11.2 LC analysis condition（$C_2O_4^{2-}$）

Equipment:	Hitachi L-7000 System
Data processor:	Hitachi D-7000
Column:	COSMOSIL HILIC (5 μm、4.6 mmI.D.×250 mm)
Column Temp:	30℃
Mobile phase:	0.1% phosphate (pH adjusted to 7.0 by 1 mol/L-NaOH)：acetonitrile = 40：60(v：v)
Flow rate:	1.0 mL/min
Detector:	UV210 nm
Detection sensitivity:	1.0 AU/V
Injection volume:	30.0 μL
Analysis time:	20 min

<Preparation of standard solutions>

Prepared standard solutions of 4 concentrations between 7.4 – 73.9 mg/L as $C_2O_4^{2}$.

11.3 IC analysis condition（NH_4^+）

Equipment:	Dionex Japan DX-120
Column:	IonPac CS12A (4 mmI.D.×250 mm)
Mobile phase:	30 mmol/L- methane sulfonic acid aq. solution
Flow rate:	1.0 mL/min
Suppressor:	CSRS (Recycling mode/current value 100 mA)
Injection volume	25 μL
Detector:	Conductance（range 100 μS)
Analysis time:	6 min.

< Preparation of standard solutions >

Prepared standard solutions of 4 concentrations between 1.03 – 10.30 mg/L as NH_4^+.

11.4 ICP analysis condition（Nb^{5+}）

Equipment:	Shimadzu ICPS-7510
Wave length:	309.418 nm
High frequency output	1.2 kw
Amount of blowtorch:	Low
Coolant gas	14.0 L/min
Plasma gas:	1.20 L/min
Carrier gas:	0.70 L/min
Purge gas:	ON

<Preparation of standard solution>

Prepare standard solution of 5.0 and 25.0 mg/L as Nb^{5+} concentration diluted with water.

11.5 DOC analysis condition

Equipment:	Shimadzu TOC-5000A
Range:	AUTO
Injection volume:	AUTO
Catalyst:	TC catalyst
Carrier gas	Air
Flow rate:	150 mL/min

＜Preparation of standard solution＞

(1) Standard solution for TC: Accurately weigh 0.21254 g of KH- phthalate, measure up to 100 mL with water. Further, dilute it 50 times with water and use it as standard solution. (corresponding to 20.0 mg/L TC)

(2) Standard solution for IC: Accurately weigh 0.35005 g of NaH carbonate and ca. 0.44250 g of Na2 carbonate, measure up to 100 mL with water. Further, dilute it 50 times with water and use it as standard solution (corresponding to 20.0 mg/L IC)

11.6 Quantification limit

$C_2O_4^{2-}$; 0.5 mg/L

NH_4^+; 1.0 mg/L

11.7 Recovery test

	$C_2O_4^{2-}$(%)	NH_4^+(%)	Nb^{5+}(%)
Test item	100	97	102
Test item-water	101	97	104

Based on the above result by direct quatification of liquids after exposure, the following were calculated as 100%, i.e., residual amount, residual rate, formation of decomposed substance and its rate, degradation rate and mass balance.

12. Statistical Method for Data Analysis

* Regression equation and regression coefficient of LC and IC analysis calibration curves were calculated with least-squares method.
* Data processing of ICP analysis was carried out by attached data processor with the equipment.
* Data processing of DOC was carried out by attached data processor with the TOC analysis equipment.
* Residue amount of oxalate ion, residue amount of DOC (mass = mg, conc. = mg/L) and oxygen demand (mass = mg) wer expressed by rounding off to the first decimal place, residue amount of ammonium ion, niobium ion and degradation product (niobium compound) to the second decimal place, and residual rate, degradation (%) and compound formation (%) to whole number.
* Mean values are arithmetical mean.
* Rules for rounding of numerical values is round off.

13. GLP compliance

This study was conducted to comply with:
"Standard on Testing Facility for Conducting Studies of New Chemical Substances, etc. (Yakushokuhatsu 0331 No. 8, Seikyoku No. 6 dated 29th March 2011, Kanpoki No. 110331010) Standard Operation Procedure (SOP) on Chemical Substance GLP.

14. Archiving
Any record, sample and data concerning this study are to be kept at storage facility of Koei Techno Co., Ltd. in accordance with GLP.

15. Environmental factor may have affected the quality assurance of the study.
None.

Test Result

1. Validity of test *10
Differences of direct quantifications in test item degradation vessels were found as 0% by LC, 3% by IC and 0% by ICP-AES, respectively from Table 1, and 3% by DOC from Table 3. These differences by direct quantifications are all less than 20% and the degradation of aniline calculated from oxygen demand value were more than 40% on Day 7 and more than 65% on Day 14 of the exposure, therefore, this study is considered to be valid.

*10 Differences from the maximum to the minimum value of the degradation by LC, IC, ICP-AES and DOC analysis were employed for the judgment of validity on test (as shown *1 page 5)

2. Degradation
Mean value of degradation of the test item on Day 28 measured by oxygen demand was 0% as shown in Table 1. And, the direct quantification was conducted by means of a Liquid Chromatography (LC) for oxalate ion, an Ion Chromatography (IC) for ammonium ion and an Inductive Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for niobium ion, since the test item is dissociated to above ions in water.
According to the direct quantification, the % degradation of oxalate was mean 100% of the replicates, mean 2% for ammonium ion and mean 100% for niobium ion, respectively.

Table 1

Degradation by oxygen demand	No.	Test vessel	% Degradation	mean	Attached Table	Attached Figure
	3	Test item	-11	0	AT 1	AF 1
	4	Test item	0			
	5	Test item	11			
	6	Inoculum blank	-			
Degradation of oxalate by direct quantification (LC)	No.	Test vessel	% Degradation	mean	Attached Table	Attached Figure
	3	Test item	100	100	AT 2-1	AF 2-1～2-7
	4	Test item	100			
	5	Test item	100			
Degradation of ammonium ion by	No.	Test vessel	% Degradation	mean	Attached Table	Attached Figure
	3	Test item	0	2	AT 2-2	AF

direct quantification (IC)	4	Test item	3			2-8～2-14
	5	Test item	2			
Degradation of niobium ion by direct quantification (ICP-AES)	No.	Test vessel	% Degradation	mean	Attached Table	Attached Figure
	3	Test item	100	100	AT 2-3	AF 2-15～2-19
	4	Test item	100			
	5	Test item	100			

Remarks: No. indicates a detector of coulometer.

3. Residual rate, degradation rate, formation rate and mass balance (Table 2)

The residual rate was found, mean 0% at test item vessels and 98% at inoculum blank for oxalate ion, mean 96% at test item vessels and 98% at test item-water for ammonium ion and mean 0% at test item vessels and 101% at inoculum blank for niobium ion. According to LC analysis no degradation products of more than 1% was detected.

Further, the formation rate of niobium hydroxide was mean 103% at test item vessels, and combined mass balance of niobium ion and niobium hydroxide was mean 103% at test item vessels.

Table 2

Residual rate of oxalate	No.	Test vessel	% Residue	mean	Attached Table	Attached Figure
	3	Test item	0	0	AT 2-1	付図 2-1～2-7
	4	Test item	0			
	5	Test item	0			
	6	Inoculum blank	98			
Residual rate of ammonium	No.	Test vessel	残存率(%)	mean	Attached Table	Attached Figure
	3	Test item	98	96	AT 2-2	付図 2-8～2-14
	4	Test item	95			
	5	Test item	96			
	6	Inoculum blank	98			
Residual rate of niobium ion	No.	Test vessel	%Residue	mean	Attached Table	Attached Figure
	3	Test item	0	0	AT 2-3	付図 2-15～2-19
	4	Test item	0			
	5	Test item	0			
	6	Inoculum blank	101			
Formation rate of niobium hydroxide (V)	No.	Test vessel	%Formation	mean	Attached Table	Attached Figure
	3	Test item	104	103	AT 2-3	付図 2-15～2-19
	4	Test item	103			
	5	Test item	103			
Mass balance of niobium ion and niobium hydroxide	No.	Test vessel	%Mass balance	mean	Attached Table	Attached Figure
	3	Test item	104	103	AT 2-3	Attached Figure 2-15～
	4	Test item	103			
	5	Test item	103			

4. Qualitative analysis of niobium ion degradation product in test item vessels

An IR analysis was conducted after the exposure of the test item degradation vessels to compare the spectrum with that of standard substance of niobium hydroxide (V), and the identification of both items was confirmed (Attached Figure 9 and 10).

5. Residual amount of DOC

From Table 3 below, it is found that the residual DOC amount was mean 0.0 mg[*12] at test item vessels and 4.1 mg at test item-water vessel against theoretical one, and hence the residual rate was mean 0%[*13] and 95 %, respectively. The degradation rate derived from DOC was mean 104%.

Table 3

No.	Test vessel	Residual DOC (mg)	mean	Residual (%)	mean	%Degradation	mean	Table	Figure
3	Test item	-0.1	0.0[*13]	-2	0[*13]	102	104	AT 3	AF 3
4	Test item	-0.2		-5		105			
5	Test item	-0.2		-5		105			
6	Inoculum blank	4.1		95		-			

*12: The value of test item was calculated by deducting value of the basic respiration vessel and test item-water by deducting that of ultra pure water blank.

*13 The value obtained were minus figures and hence counted as 0.

6. IC residual rate and calculation of mineralization rate

Inorganic carbon (IC) residual rate at test item degradation vessels of DOC analysis were measured as mean 0.8 mg (seen in Table 4) and pH was declined towards alkaline side as 8.8 (seen in Table 6).

These results suggested that oxalate ion derived from the test item can be degradable by microbes and carbon dioxides generated by the degradation were to retain as IC in the test liquid since the test liquid was alkalinity and hence the IC residual amount wasn't reflected to the oxygen demand. A substantial mineralization rate of the test item by microbes was calculated as mean 18% by using an equation of "Mineralization rate = Degradation (%) by oxygen demand + IC residual rate (residual IC percentage against theoretical DOC) (%)". Further, if supplementarily explained, the mineralization rate of mean 18% could not reach to 60% as a criterion of ready biodegradability, and the reason of this lower rate than the expected was considered due to a wide variation of the measurement error attributable to very small values of ANO's theoretical oxygen demand 2.8 mg and the theoretical DOC 4.3 mg.

Table 4

No.	Test vessel	%BOD Degradation	mean	%IC residual rate	mean	% Mineralization	mean	Attached Table
3	Test item	-11	0	14	18	3	18	1 & 5
4	Test item	0		21		21		
5	Test item	11		19		30		

7. Observation and measurement during and after the exposure

Observation carried out once a day during the exposure period was shown in Table 5.

Table 5

Temperature of coulometer thermostatic bath	25.0℃ through the period
Dissolving condition of test item	Test item degradation vessel: insoluble on Day 0 - 28 Inoculum blanc vessel: soluble on Day 0 - 28
Colour of test liquid	Aniline degradation vessel: colourless on Day 0 - 28 Test item degradation vessel: colourless on Day 0 – 28 Inoculum blanc vessel: colourless on Day 0 - 28
Proliferation of activated sludge	Aniline degradation vessel: no proliferation on Day 0 - 7 : observed proliferation on day 8 – 28 Test item degradation vessel: no proliferation on Day 0 - 28

Visual observation and result of pH measurement at the end of exposure were shown in Table 6.

Table 6

End of exposure	Test item vessel			Inoculum blank
Coulometer detector No.	3	4	5	6
Dissolving condition of test item	insoluble	insoluble	insoluble	soluble
Colour of test liquid	colourless	colourless	colourless	colourless
Proliferation of activated sludge	No	No	No	-
pH at the end of exposure	8.55	8.97	8.97	3.65

8. Identification of test item and stability under the storage condition
Identification: IR spectra by sponsor and by Koei Techno were compared to find both are identical. (Attached Figure4 and 5)
Stability: IR measurement on the day of start and end of exposure were carried out and it is confirmed the test item was stable during the test period. (Attached Figure 6 and 7)

Discussion

The difference of both extremes in % degradation of replicates calculated by the oxygen demand was 22% as seen in Table 1, and this value deviates from the allowance of less than 20% as specified in SOP. The reason of this deviation is speculated by a wide variation for the measurement of oxygen demand attributed to the very small theoretical oxygen demand of ANO as 2.8 mg. Since the difference of both extremes in oxygen demand of replicates was 0.6 mg and within the range of coefficient of variation (sd = 1.9 mg in basic respiration vessels obtained by studies in 2012), in this test it seems difficult to judge a validity of the test by using the degradation rate by oxygen demand. Therefore, the validity of the test was assessed by LC analysis, IC analysis, ICP-AES analysis and DOC analysis by using their difference of both extreme values of replicates.

In a preliminary test, niobium ion in test item degradation vessels formed (an) insoluble product(s). Under the testing condition, it is conjectured to form niobium oxide (V) and niobium hydroxide (V), and its dissolution tests were carried out by using acids and alkalis (conc, sulfuric acid, conc. nitric acid, conc. hydrochloric acid, aqua

regia, inverse aqua regia and aqueous ammonia). According to the result that the dissolution was obtained by conc. sulfuric acid, the insoluble substance was assumed to be niobium hydroxide (V) (Chemical Unabridged Dictionary published by Tokyo Kagaku Dojin). In this study IR analysis was carried out to compare the spectra of the insoluble substance with that of standard sample of niobium hydroxide (V), resulting the confirmation of identity.

While, the quantification of niobium ion was conducted by ICP-AES after a pretreatment of the test liquid by conc. sulfuric acid.

The generation rate of niobium hydroxide (V) was mean 103% in the test item degradation vessel, and the mass balance of niobium ion and niobium hydroxide in total was 103% in the test item degradation vessel.

The residual rate of DOC was mean 0%[*4] in test item degradation vessels, while 95% in inoculum blank, and degradation was obtained as mean 104%. And, residual amount of inorganic carbon (IC) was mean 0.8 mg in test item degradation vessels, and the shift of pH towards alkaline was observed, as mean pH8.83.

From the results described above, it is considered that oxalate ion derived from the test item can be degradable by microbes and carbon dioxides generated by the degradation were to retain as IC in the test liquid since the test liquid was alkalinity and hence the IC residual amount wasn't reflected to the oxygen demand.

A substantial mineralization rate of the test item by microbes was calculated as mean 18% by using an equation of "Mineralization rate = Degradation (%) by oxygen demand + IC residual rate (residual IC percentage against theoretical DOC) (%)". Further, supplementarily explained, the mineralization rate of mean 18% could not reach to 60% as a criterion of ready biodegradability, and the reason of this lower rate than the expected was considered due to a wide variation of the measurement error attributable to very small values of ANO's theoretical oxygen demand 2.8 mg and the theoretical DOC 4.3 mg.

From analysis results of LC and DOC, it is considered that oxalate as an organic component of ANO is biodegradable and completely mineralized under the present test condition. However, it is also considered that ammonium ion remains as unchanged form, and niobium ion remains as niobium hydroxide (V).

It is concluded that ANO is not ready biodegradable under the test condition.

Conclusion

It is concluded that ANO is not ready biodegradable under the test condition.

Appendices

Attached Table 1	Analysis data of oxygen demand
Attached Table 2-1	Residual amount of $C_2O_4^{2-}$ by LC analysis
Attached Table 2-2	Residual amount of NH_4^+ by IC analysis
Attached Table 2-3	Residual amount of by ICP analysis and formation of niobium hydroxide (V)
Attached Table 3	Analysis data of DOC
Attached Table 4	Residual rate of IC

Attached Table 5 Mineralization rate

(Data obtained on the final day of exposure, 27th March 2013)

付表 1　酸素消費量分析測定表

No.	試験区	添加量 A(mg)*1	理論的酸素消費量 B(mg)	7日			14日			21日			28日		
				酸素消費量(mg)	分解度 C(%)		酸素消費量(mg)	分解度 C(%)		酸素消費量(mg)	分解度 C(%)		酸素消費量(mg)	分解度 C(%)	
1	アニリン分解区	30.4	91.4	38.5	40		68.2	69		72.5	72		73.3	72	
2	基礎呼吸区	－	－	2.0	－		4.8	－		6.9	－		7.1	－	
3	被験物質分解区	30.4	2.8	3.3	46		5.3	18		6.6	-11		6.8	-11	
4	被験物質分解区	30.4	2.8	3.3	46	50	5.3	18	22	6.9	0	0*2	7.1	0	0
5	被験物質分解区	30.4	2.8	3.6	57		5.6	29		7.1	7		7.4	11	
6	被験物質-水区	30.4	－	0.0	－		0.0	－		0.0	－		0.0	－	

No.: クーロメータの検出部No.

*1 被験物質は純度90%にて換算した。

*2 マイナス値のため、0とした。

B: 理論的酸素消費量[TOD](mg)

(ANO)以下に示す成分1と成分2のTODの合計

(成分1) $C_4H_{14}NNbO_{14} \rightarrow NH_4^+ + Nb(OH)_5 + 2C_2H_2O_4 + 5H_2O$

上記の通り、被験物質分解区において、成分1は、NH^+(アンモニウムイオン)、$Nb(OH)_5$(水酸化ニオブ(V))、$C_2H_2O_4$(シュウ酸)及びH_2O(水)となり、その中で、酸素消費に関与するのは$C_2H_2O_4$(シュウ酸)のみとなる。

よって、TODの算出は以下となる。

$2C_2H_2O_4 + O_2 \rightarrow 4CO_2 + 2H_2O$

被験物質添加量(mg)×(成分1の含量(%))/90×O_2 / $C_4H_{14}NNbO_{14}$

＝A×70.2 / 90×32.00 / 393.06

(成分2) $C_8H_{22}N_2O_{20} \rightarrow 2NH_4^+ + 4C_2H_2O_4 + 2H_2O$

上記の通り、被験物質分解区において、成分2は、NH_4^+(アンモニウムイオン)、$C_2H_2O_4$(シュウ酸)及びH_2O(水)となり、その中で、酸素消費に関与するのは$C_2H_2O_4$(シュウ酸)のみとなる。

よって、TODの算出は以下となる。

$4C_2H_2O_4 + 2O_2 \rightarrow 8CO_2 + 4H_2O$

被験物質添加量(mg)×(成分2の含量(%))/90×$2O_2$ / $C_8H_{22}N_2O_{20}$

＝A×19.8 / 90×64.00 / 466.26

(ANO)被験物質添加量(mg)×(成分1の含量(%))/90×O_2 / $C_4H_{14}NNbO_{14}$ +

被験物質添加量(mg)×(成分2の含量(%))/90×$2O_2$ / $C_8H_{22}N_2O_{20}$ =

＝A×70.2 / 90×32.00 / 393.06 + A×19.8 / 90×64.00 / 466.26

(アニリン) $C_6H_7N + 8.75O_2 \rightarrow 6CO_2 + 3.5H_2O + NO_2$

添加量(mg)×$8.75O_2$ /C_6H_7N = A×280.00 / 93.13

C: 酸素消費量からの分解度(%)

(BOD－b)×100 / TOD

ここに、

BOD: アニリン及び被験物質分解区の生物化学的酸素消費量[測定値](mg)

b: 基礎呼吸区の酸素消費量[測定値](mg)

TOD: アニリン及び被験物質が完全に酸化された場合に必要とされる理論的酸素消費量[計算値](mg)

付表 2-1　LCによる$C_2O_4^{2-}$残留量分析測定表

No.	試験区	被験物質添加量 (mg)*1	理論$C_2O_4^{2-}$添加量 A(mg)	AREA	被験物質濃度 B(mg/L)*2	液量 C(mL)	残留量 D(mg)		残存率 E(%)		分解度 F(%)	
	標準液	−	−	120533	7.4	−	−		−		−	
		−	−	303106	18.5	−	−		−		−	
		−	−	598150	37.0	−	−		−		−	
		−	−	1212758	73.9	−	−		−		−	
3	被験物質分解区	30.4	15.7	不検出	0.0	300	0.0		0		100	
4	被験物質分解区	30.4	15.7	不検出	0.0	300	0.0	0.0	0	0	100	100
5	被験物質分解区	30.4	15.7	不検出	0.0	300	0.0		0		100	
6	被験物質−水区	30.4	15.7	837632	51.2	300	15.4		98		−	

注）No.: ｸｰﾛﾒｰﾀ検出部のNo.

*1 被験物質は純度90%にて換算した。

*2 定量限界未満は0.0とした。

A: 理論$C_2O_4^{2-}$添加量

（成分1）$C_4H_{14}NNbO_{14}$

（成分2）$C_8H_{22}N_2O_{20}$

被験物質添加量(mg)×(成分1の含量(%))/90×2$C_2O_4^{2-}$/$C_4H_{14}NNbO_{14}$

+ 被験物質添加量(mg)×(成分2の含量(%))/90×4$C_2O_4^{2-}$/$C_8H_{22}N_2O_{20}$

= 被験物質添加量(mg)×70.2/90×176.04/393.06

+ 被験物質添加量(mg)×19.8/90×352.08/466.26

D: 残留量(mg) = B×C / 1000

E: 残存率(%) = D×100/A

F: 分解度(%)＝(dh－dg)×100 / dh

dg: 被験物質分解区の残留量(mg)

dh: 被験物質－水区の残留量(mg)

付表 2-2　ICによるNH_4^+残留量分析測定表

No.	試験区	被験物質添加量 (mg)*1	理論NH_4^+添加量 A(mg)	AREA	NH_4^+濃度 B(mg/L)	液量 C(mL)	残留量 D(mg)		残存率 E(%)		分解度 F(%)	
	標準液	－	－	181859	1.03	－	－		－		－	
		－	－	300150	2.06	－	－		－		－	
		－	－	650003	5.15	－	－		－		－	
		－	－	1181925	10.30	－	－		－		－	
3	被験物質分解区	30.4	1.61	567218	2.61	10	1.57		98		0	
4	被験物質分解区	30.4	1.61	561293	2.55	10	1.53	1.55	95	96	3	2
5	被験物質分解区	30.4	1.61	563021	2.57	10	1.54		96		2	
6	被験物質−水区	30.4	1.61	361822	2.62	10	1.57		98		−	

注）No.: クーロメータ検出部のNo.
*1 被験物質は純度90%にて換算した。

A: 理論NH_4^+添加量
（成分1）$C_4H_{14}NNbO_{14}$
（成分2）$C_8H_{22}N_2O_{20}$
被験物質添加量(mg)×(成分1の含量(%))/90×NH_4^+/$C_4H_{14}NNbO_{14}$
+ 被験物質添加量(mg)×(成分2の含量(%))/90×2NH_4^+/$C_8H_{22}N_2O_{20}$
= 被験物質添加量(mg)×70.2/90×18.04/393.06
+ 被験物質添加量(mg)×19.8/90×36.08/466.26

D: 残留量(mg) = B×C/1000×(300/5)*2
*2 暴露試験終了液300 mLから5 mL採取し、水で10 mLに希釈して測定した。

E: 残存率(%) = D×100/A

F: 分解度(%) = (dh－dg)×100/dh
dg: 被験物質分解区の残留量(mg)
dh: 被験物質－水区の残留量(mg)

付表 2-3　ICPによるNb^{5+}残留量及び水酸化ニオブ(V)の生成量分析測定表

＜Nb^{5+}残留量＞

No.	試験区	被験物質添加量 (mg)*1	理論Nb^{5+}添加量 A(mg)	Nb^{5+}濃度 B(mg/L)	液量 C(mL)	残留量 D(mg)		残存率 E(%)		分解度 F(%)	
	標準液	—	—	5.00	—	-		—	—	—	—
		—	—	25.00	—	-		—	—	—	—
3	被験物質分解区(ろ液)	30.4	5.60	0.03	300	0.01	0.01	0	0	100	100
4	被験物質分解区(ろ液)	30.4	5.60	0.03	300	0.01		0		100	
5	被験物質分解区(ろ液)	30.4	5.60	0.03	300	0.01		0		100	
6	被験物質-水区	30.4	5.60	18.84	300	5.65		101		-	

＜水酸化ニオブ(V)の生成量＞

No.	試験区	被験物質添加量 (mg)*1	理論Nb^{5+}生成量A' (mg)	Nb^{5+}濃度 B'(mg/L)	液量 C' (mL)	生成量D' (mg)		生成率 E' (%)	
	標準液	—	—	5.00	—	-		—	—
		—	—	25.00	—	-		—	—
3	被験物質分解区(ろ残)	30.4	5.60	5.81	20	5.81	5.78	104	103
4	被験物質分解区(ろ残)	30.4	5.60	5.79	20	5.79		103	
5	被験物質分解区(ろ残)	30.4	5.60	5.74	20	5.74		103	

＜被験物質分解区におけるNb^{5+}と水酸化ニオブ(V)の物質収支＞

No.	試験区	Nb^{5+}残存率 (%)	水酸化ニオブ生成率(%)	物質収支(%)	
3	被験物質分解区	0	104	104	103
4	被験物質分解区	0	103	103	
5	被験物質分解区	0	103	103	

注)No.: クーロメータ検出部のNo.

*1 被験物質は純度90%にて換算した。

A: 理論Nb^{5+}添加量: (成分1)$C_4H_{14}NNbO_{14}$
被験物質添加量(mg)×(成分1)含量(%)/90×Nb^{5+}/$C_4H_{14}NNbO_{14}$
= 被験物質添加量×70.2/90×92.91/393.06

D: 残留量(mg) = B×C/1000

E: 残存率(%) = D×100/A

F: 分解度(%) = (dh－dg)×100/dh
dg: 被験物質分解区の残留量(mg)d、h: 被験物質－水区の残留量(mg)

A': 理論Nb^{5+}生成量(水酸化ニオブ(V)を前処理し、Nb^{5+}として測定する。)
(成分1)$C_4H_{14}NNbO_{14}$
被験物質添加量(mg)×(成分1)含量(%)/90×Nb^{5+}/$C_4H_{14}NNbO_{14}$
= 被験物質添加量×70.2/90×92.91/393.06

D': 被験物質分解区No. 3;生成量(mg) = B'×C'/1000×(50/2)×2*2
被験物質分解区No. 4及びNo. 5;生成量(mg) = B'×C'/1000×(100/2)*3
*2 被験物質分解区No. 3は、暴露試験終了液全量をろ過したろ残の半分を硫酸10 mLに溶解後、水40 mLを加えて50 mLとし、その溶液から2 mL採取し、水で20 mLに希釈して測定した。
*3 被験物質分解区は、暴露試験終了液全量をろ過したろ残を硫酸20 mLに溶解後、水80 mLを加えて100 mLとし、その溶液から2 mL採取し、水で20 mLに希釈して測定した。

E': 生成率(%) = D'×100/A'

被験物質分解区における物質収支: Nb^{5+}との残存率(%)+水酸化ニオブ(V)の生成率(%)

付表 3　　DOC分析測定表

No.	試験区	被験物質添加量 A(mg)	理論DOC量 B(mg)	TC濃度[*1] (mg/L)	IC濃度[*1] (mg/L)	DOC濃度 C(mg/L)	DOC量 D(mg)	ブランクとの差 E(mg)[*2]	DOC残存率 F(%)		DOC分解度 G(%)	
	標準液	―	―	20.0	20.0	―	―	―	―		―	
	試験使用超純水	―	―	0.2	0.0	0.2	0.1	―	―		―	
2	基礎呼吸区	―	―	1.5	0.3	1.2	0.4	―	―		―	
3	被験物質分解区	30.4	4.3	3.3	2.4	0.9	0.3	−0.1	−2		102	
4	被験物質分解区	30.4	4.3	4.0	3.3	0.7	0.2	−0.2	−5	0[*3]	105	104
5	被験物質分解区	30.4	4.3	3.9	3.1	0.8	0.2	−0.2	−5		105	
6	被験物質−水区	30.4	4.3	14.1	0.0	14.1	4.2	4.1	95		―	

注）No.:ｸｰﾛﾒｰﾀ検出部のNo.

*1 TC濃度及びIC濃度は、DOC測定ﾁｬｰﾄのMN(平均)値を四捨五入し、小数第一位に丸めた値を採用した。

*2 被験物質分解区は基礎呼吸区を、被験物質−水区は試験使用超純水ﾌﾞﾗﾝｸを差し引いた値。

*3 ﾏｲﾅｽ値のため0とした。

B：理論DOC量（mg）

（ANO）

（成分1）添加量×（成分1の含量(%)）/90×C_4/$C_4H_{14}NNbO_{14}$

+（成分2）添加量×（成分2の含量(%)）/90×C_8/$C_8H_{22}N_2O_{20}$

= A×70.2/90×48.04/393.06

+ A×19.8/90×96.08/466.26

C：DOC濃度（mg/L） = TC濃度 − IC濃度

D：DOC量（mg） = C×300 / 1000

F：DOC残存率（%） = E×100 / B

G：DOCによる分解度（%） = （Eb − Es）×100 / Eb

ここに、

Es：E（被験物質分解区） = D（被験物質分解区） − D（基礎呼吸区）

Eb：E（被験物質−水区） = D（被験物質−水区） − D（試験使用超純水）

付表 4　　IC残存率

No.	試験区	被験物質添加量 A(mg)	理論 DOC 量B(mg)	IC濃度*1 C(mg/L)	IC 残留量 D(mg)	ブランクとの差 E(mg)*2		IC 残存率 F(%)	
	標準液	―	―	20.0	―	―		―	
	試験使用超純水	―	―	0.0	0.0	―		―	
2	基礎呼吸区	―	―	0.3	0.1	―		―	
3	被験物質分解区	30.4	4.3	2.4	0.7	0.6	0.8	14	18
4	被験物質分解区	30.4	4.3	3.3	1.0	0.9		21	
5	被験物質分解区	30.4	4.3	3.1	0.9	0.8		19	
6	被験物質-水区	30.4	4.3	0.0	0.0	0.0		0	

注)No.:ｸｰﾛﾒｰﾀ検出部のNo.
*1 IC濃度は、DOC測定ﾁｬｰﾄのMN(平均)値を四捨五入し、小数第一位に丸めた値を採用した。
*2 被験物質分解区は基礎呼吸区を、被験物質-水区は試験使用超純水ﾌﾞﾗﾝｸを差し引いた値。

B:理論DOC量(mg)
(ANO)
(成分1)添加量×(成分1の含量(%))/90×C_4/$C_4H_{14}NNbO_{14}$
+(成分2)添加量×(成分2の含量(%))/90×C_8/$C_8H_{22}N_2O_{20}$
= A×70.2/90×48.04/393.06
+ A×19.8/90×96.08/466.26

D:IC残留量(mg) = C×300 / 1000

F:IC残存率(%) = E/B×100

付表 5　　無機化率

No.	試験区	BOD分解度 A(%)		IC残存率 B(%)		無機化率 C(%)	
3	被験物質分解区	-11	0	14	18	3	18
4	被験物質分解区	0		21		21	
5	被験物質分解区	11		19		30	

注)No.: クーロメータ検出部のNo.

A: 付表 1のﾃﾞｰﾀを採用

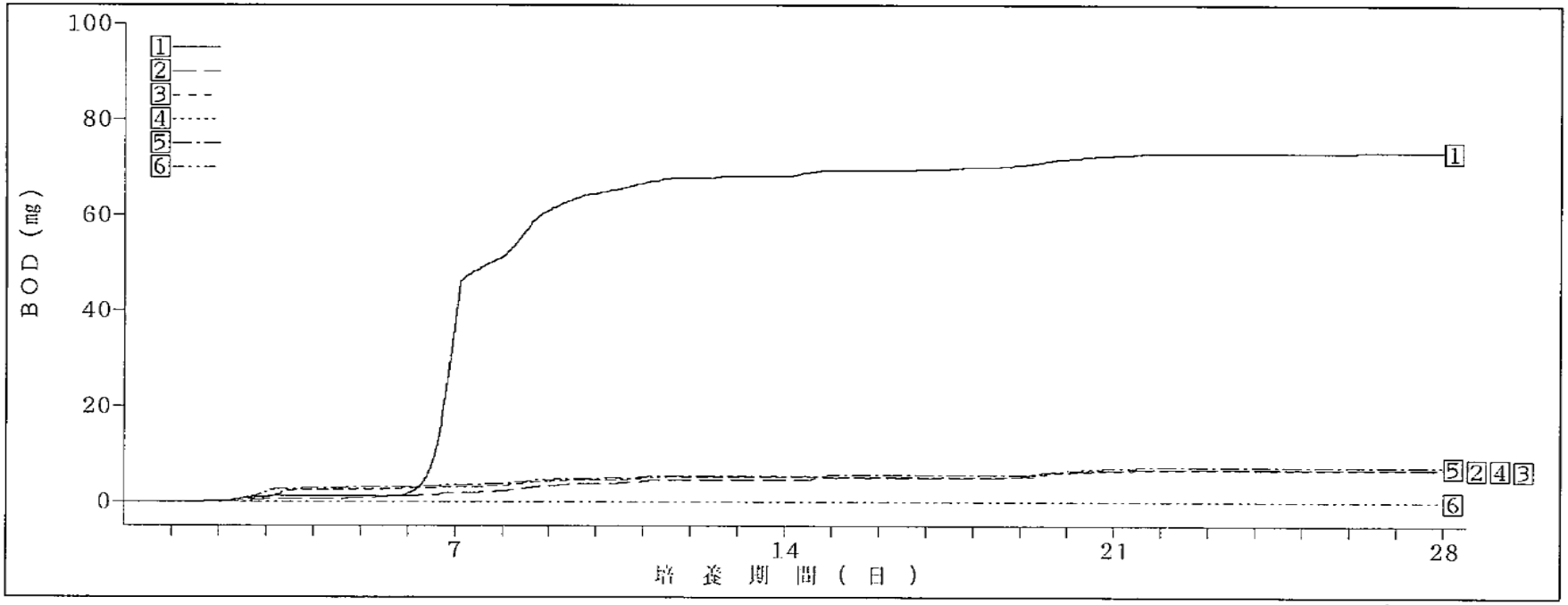
B: 付表 4のﾃﾞｰﾀを採用

C: A+B

図－1　　BODチャート

試験番号：　B12011（被験物質：ANO　）
装置番号：　A-4
試験条件：
　被験物質濃度：　100（mg/ℓ）
　活性汚泥濃度：　30（mg/ℓ）
　培養温度：　25 ± 1°C
　培養期間：　28日(2/27～3/27,2013)
備考：

試料名	BOD（mg）			
	7日	14日	21日	28日
1 汚泥＋アニリン	38.5	68.2	72.5	73.3
2 基礎呼吸	2.0	4.8	6.9	7.1
3 汚泥＋被験物質	3.3	5.3	6.6	6.8
4 汚泥＋被験物質	3.3	5.3	6.9	7.1
5 汚泥＋被験物質	3.6	5.6	7.1	7.4
6 水 ＋ 被験物質	0.0	0.0	0.0	0.0



平成25年3月27日　　氏名　飛松

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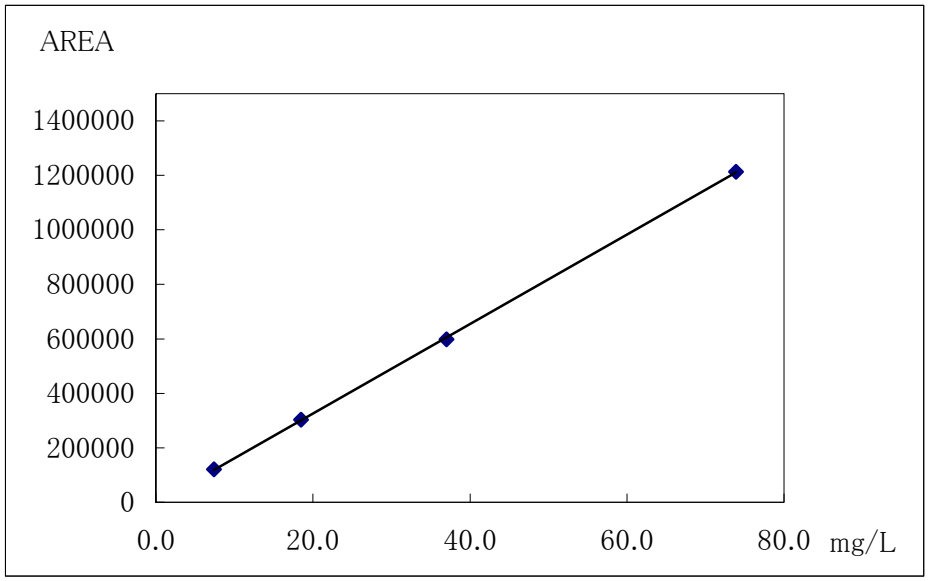
大塚

付図 2-1　$C_2O_4^{2-}$直接定量　LC分析検量線及び分析測定結果



(検量線)

	$C_2O_4^{2-}$ 濃度(mg/L)	AREA
標準液-2	7.4	120533
標準液-3	18.5	303106
標準液-4	37.0	598150
標準液-5	73.9	1212758

a= 16410
b= -2703
R^2= 0.9999

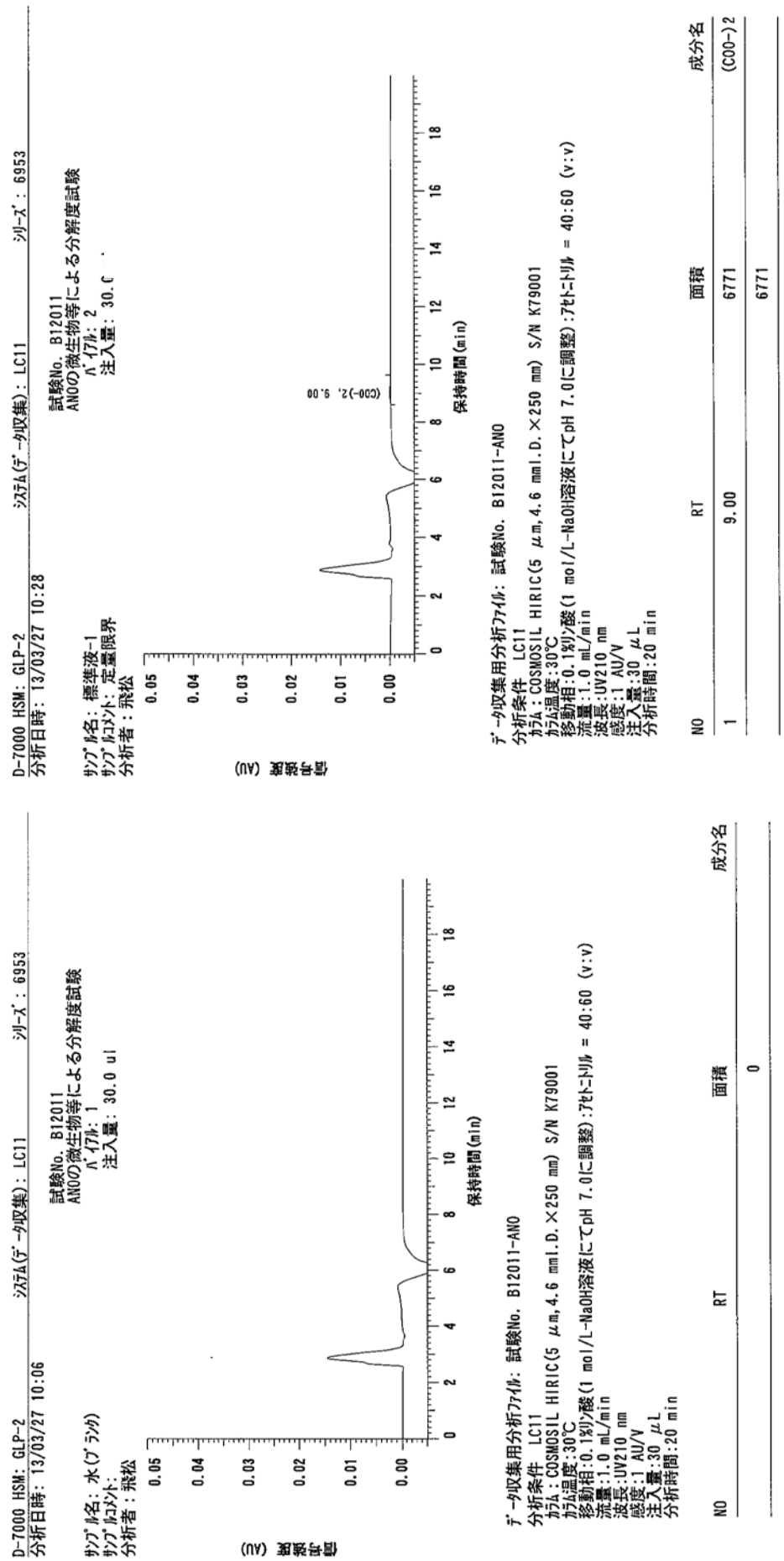
(定量限界)	$C_2O_4^{2-}$ 濃度(mg/L)	AREA
標準液-1	0.5	6771

(分析測定結果)

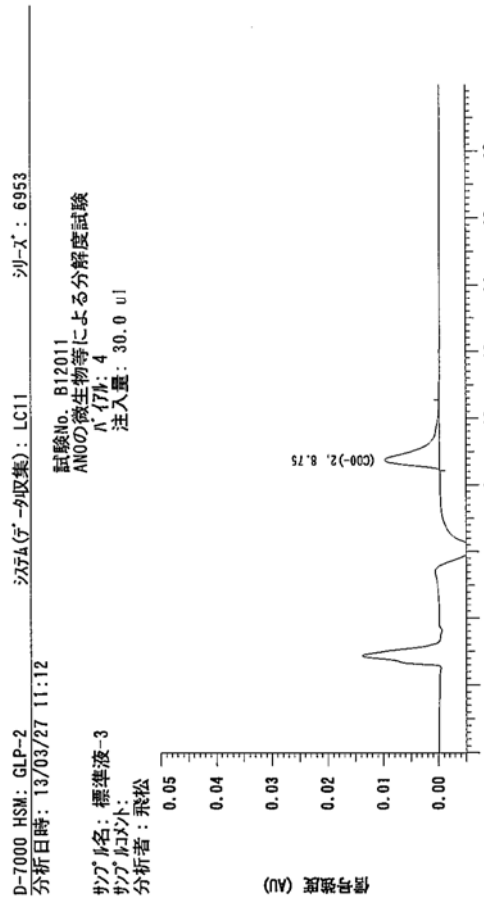
	AREA	$C_2O_4^{2-}$ 濃度(mg/L)
被験物質分解区No.3	不検出	<0.5*1
被験物質分解区No.4	不検出	<0.5*1
被験物質分解区No.5	不検出	<0.5*1
被験物質-水区No.6	837632	51.2

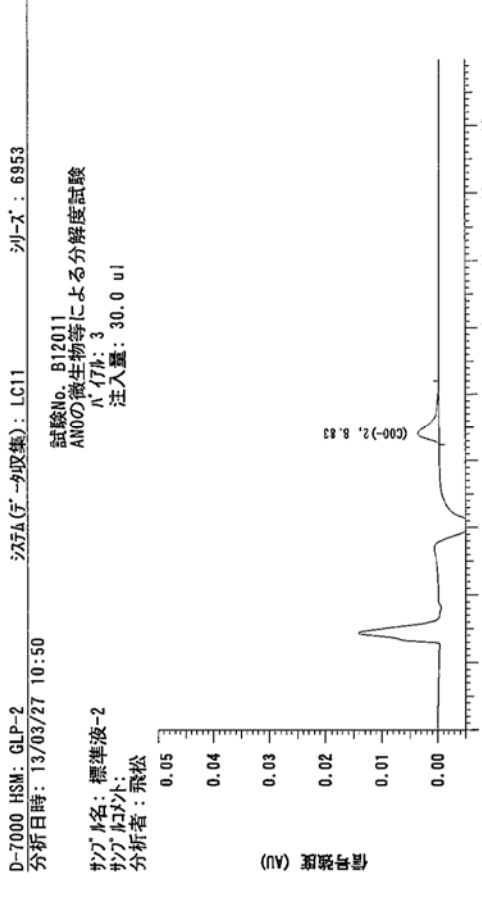
*1 不検出のため定量限界未満とした。

付図 2-2　LC分析 $C_2O_4^{2-}$ 標準液クロマトグラム



付図 2-3　LC分析 $C_2O_4^{2-}$標準液ｸﾛﾏﾄｸﾞﾗﾑ





ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件 LC11
ｶﾗﾑ: COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ｼｭｳ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
1	8.83	120533	(COO-)2
		120533	

ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件 LC11
ｶﾗﾑ: COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ｼｭｳ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
1	8.75	303106	(COO-)2
		303106	

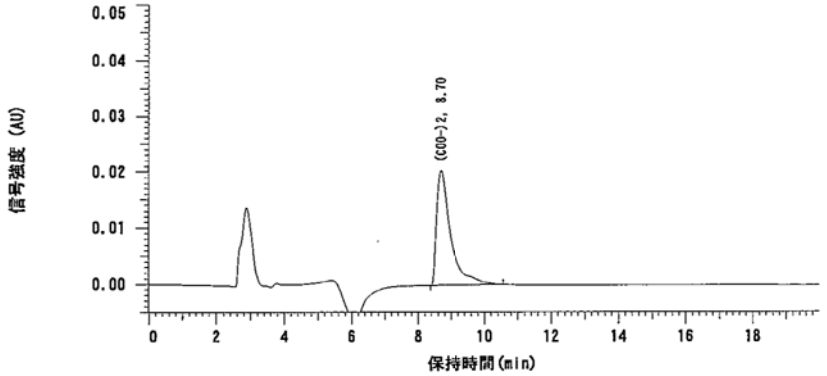
ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

付図 2-4　LC分析 $C_2O_4^{2-}$標準液クロマトグラム

D-7000 HSM: GLP-2　　ｼｽﾃﾑ(ﾃﾞｰﾀ収集): LC11　　ｼﾘｰｽﾞ: 6953
分析日時: 13/03/27 11:34

試験No. B12011
ANOの微生物等による分解度試験
ﾊﾞｲｱﾙ: 5
注入量: 30.0 ul

ｻﾝﾌﾟﾙ名: 標準液-4
ｻﾝﾌﾟﾙｺﾒﾝﾄ:
分析者：飛松



ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件　LC11
ｶﾗﾑ：COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ﾘﾝ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

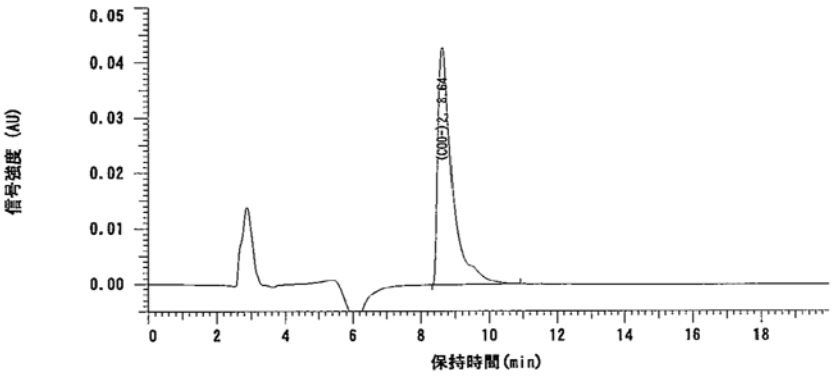
NO	RT	面積	成分名
1	8.70	598150	(COO-)2
		598150	

ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

D-7000 HSM: GLP-2　　ｼｽﾃﾑ(ﾃﾞｰﾀ収集): LC11　　ｼﾘｰｽﾞ: 6953
分析日時: 13/03/27 11:56

試験No. B12011
ANOの微生物等による分解度試験
ﾊﾞｲｱﾙ: 6
注入量: 30.0 ul

ｻﾝﾌﾟﾙ名: 標準液-5
ｻﾝﾌﾟﾙｺﾒﾝﾄ:
分析者：飛松



ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件　LC11
ｶﾗﾑ：COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ﾘﾝ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
1	8.64	1212758	(COO-)2
		1212758	

ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

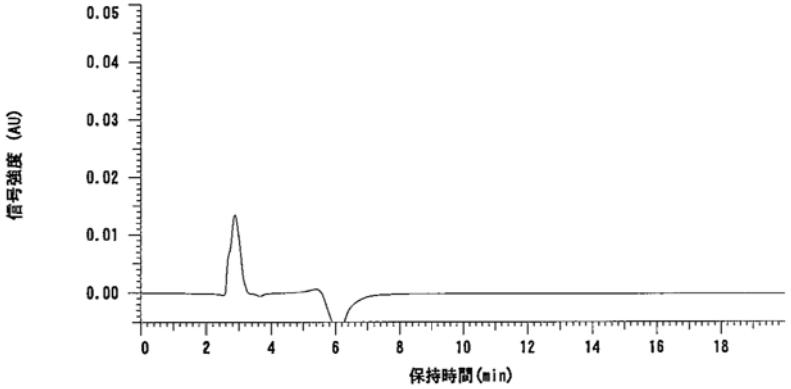
付図 2-5　LC分析 $C_2O_4^{2-}$残留量測定クロマトグラム

D-7000 HSM: GLP-2　　ｼｽﾃﾑ(ﾃﾞｰﾀ収集): LC11　　ｼﾘｰｽﾞ: 6953

分析日時: 13/03/27 12:19

試験No. B12011
ANOの微生物等による分解度試験

ｻﾝﾌﾟﾙ名: 試験開始日使用水
ｻﾝﾌﾟﾙｺﾒﾝﾄ: 被験物質-水区ﾌﾞﾗﾝｸ
ﾊﾞｲｱﾙ: 7
注入量: 30.0 ul
分析者：飛松



ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件　LC11
ｶﾗﾑ：COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ﾘﾝ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
		0	

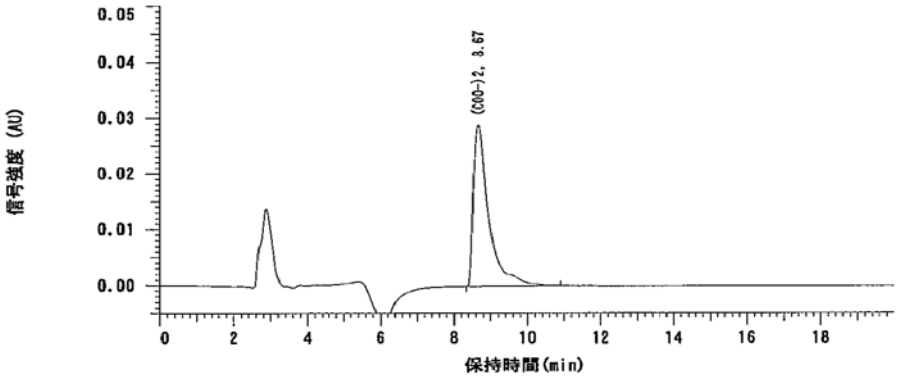
ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

D-7000 HSM: GLP-2　　ｼｽﾃﾑ(ﾃﾞｰﾀ収集): LC11　　ｼﾘｰｽﾞ: 6953

分析日時: 13/03/27 12:41

試験No. B12011
ANOの微生物等による分解度試験

ｻﾝﾌﾟﾙ名: 被験物質-水区(No. 6)
ｻﾝﾌﾟﾙｺﾒﾝﾄ:
ﾊﾞｲｱﾙ: 8
注入量: 30.0 ul
分析者：飛松



ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件　LC11
ｶﾗﾑ：COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ﾘﾝ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
1	8.67	837632	(COO-)2
		837632	

ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

付図 2-6　LC分析 $C_2O_4^{2-}$残留量測定ｸﾛﾏﾄｸﾞﾗﾑ

付図 2-7　LC分析 $C_2O_4^{2-}$残留量測定クロマトグラム

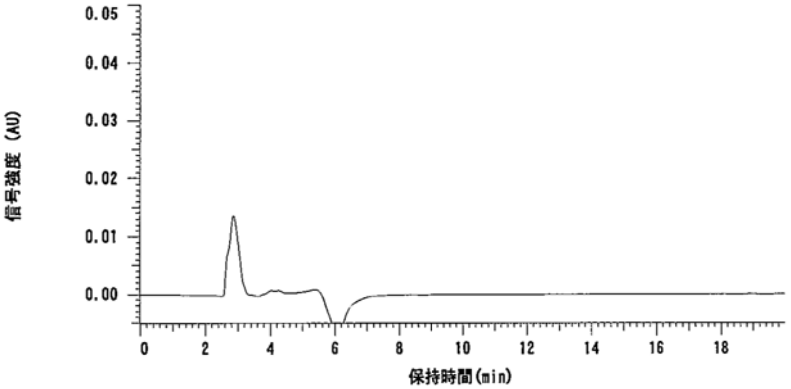
D-7000 HSM: GLP-2　　ｼｽﾃﾑ(ﾃﾞｰﾀ収集): LC11　　ｼﾘｰｽﾞ: 6953

分析日時: 13/03/27 13:47

試験No. B12011
ANOの微生物等による分解度試験

ｻﾝﾌﾟﾙ名: 被験物質分解区(No. 4)
ｻﾝﾌﾟﾙｺﾒﾝﾄ:
分析者 : 飛松

ﾊﾞｲｱﾙ: 11
注入量: 30.0 ul



ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件　LC11
ｶﾗﾑ : COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ﾘﾝ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
		0	

ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

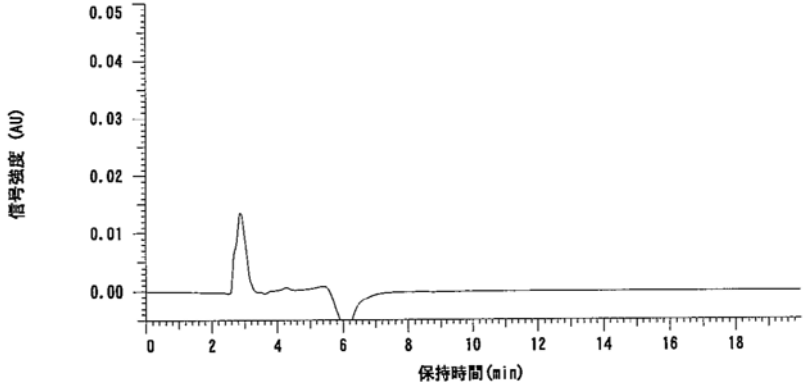
D-7000 HSM: GLP-2　　ｼｽﾃﾑ(ﾃﾞｰﾀ収集): LC11　　ｼﾘｰｽﾞ: 6953

分析日時: 13/03/27 14:09

試験No. B12011
ANOの微生物等による分解度試験

ｻﾝﾌﾟﾙ名: 被験物質分解区(No. 5)
ｻﾝﾌﾟﾙｺﾒﾝﾄ:
分析者 : 飛松

ﾊﾞｲｱﾙ: 12
注入量: 30.0 ul

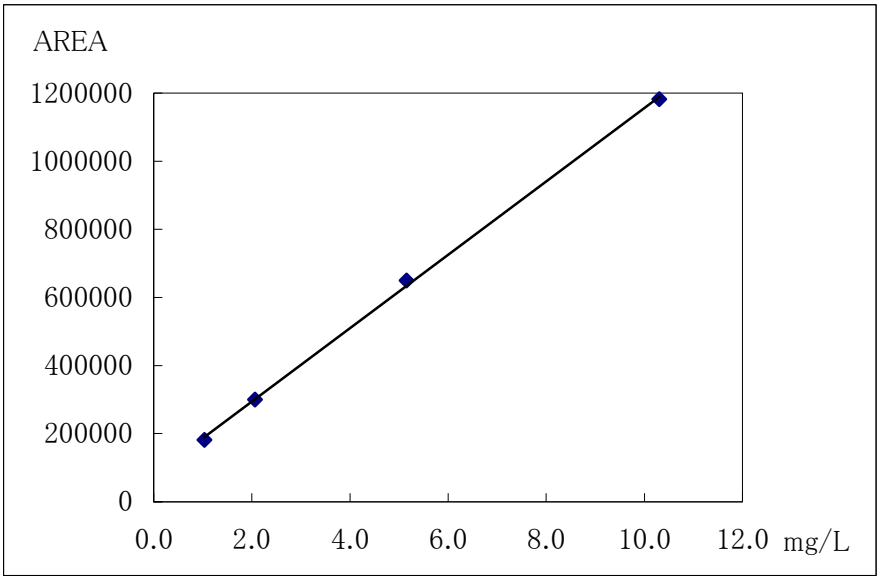


ﾃﾞｰﾀ収集用分析ﾌｧｲﾙ: 試験No. B12011-ANO
分析条件　LC11
ｶﾗﾑ : COSMOSIL HIRIC(5 μm,4.6 mmI.D.×250 mm) S/N K79001
ｶﾗﾑ温度:30℃
移動相:0.1%ﾘﾝ酸(1 mol/L-NaOH溶液にてpH 7.0に調整):ｱｾﾄﾆﾄﾘﾙ = 40:60 (v:v)
流量:1.0 mL/min
波長:UV210 nm
感度:1 AU/V
注入量:30 μL
分析時間:20 min

NO	RT	面積	成分名
		0	

ﾚﾎﾟｰﾄﾋﾟｰｸの判定ﾚﾍﾞﾙ: 10

付図 2-8　NH_4^+直接定量　IC分析検量線及び分析測定結果



(検量線)

	NH_4^+濃度 (mg/L)	AREA
標準液-1*1	1.03	181859
標準液-2	2.06	300150
標準液-3	5.15	650003
標準液-4	10.30	1181925

a= 107800
b= 79010
R^2= 0.9994

*1 定量限界を兼ねる。

(分析測定結果)

	AREA	NH_4^+濃度 (mg/L)*2
基礎呼吸区No.2	285496	1.92
被験物質分解区No.3	567218	4.53
被験物質分解区No.3－基礎呼吸区No.2	－	2.61
被験物質分解区No.4	561293	4.47
被験物質分解区No.4－基礎呼吸区No.2	－	2.55
被験物質分解区No.5	563021	4.49
被験物質分解区No.5－基礎呼吸区No.2	－	2.57
被験物質-水区No.6	361822	2.62

*2 被験物質分解区は、基礎培養基由来のNH_4^+が含まれるため、
ブランクとして基礎呼吸区を差し引いた。

付図 2-9　IC分析 NH_4^+ 標準液クロマトグラム

サンプル分析レポート

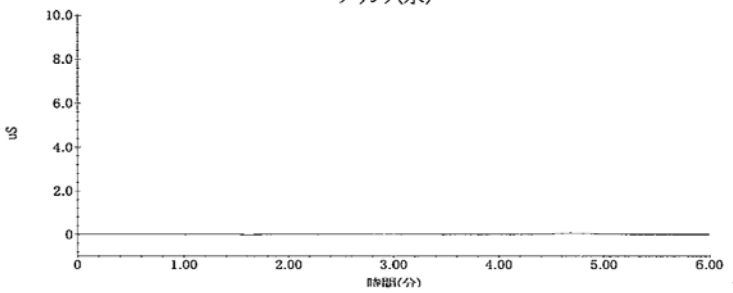
サンプル名：ﾌﾞﾗﾝｸ(水)
データファイル名：...¥B12011-ﾌﾞﾗﾝｸ(水)_001.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 9:35:05
分析者：朝日

ﾋﾟｰｸ情報：全ﾋﾟｰｸ

ﾋﾟｰｸ番号	保持時間	ピーク面積	成分名

ﾌﾞﾗﾝｸ(水)



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
ｻﾌﾟﾚｯｻｰ：CSRS(リサイクルモード/電流値100mA)
ｻﾝﾌﾟﾙ注入量：25μL
検出器：電気伝導度(ﾚﾝｼﾞ100μS)

サンプル分析レポート

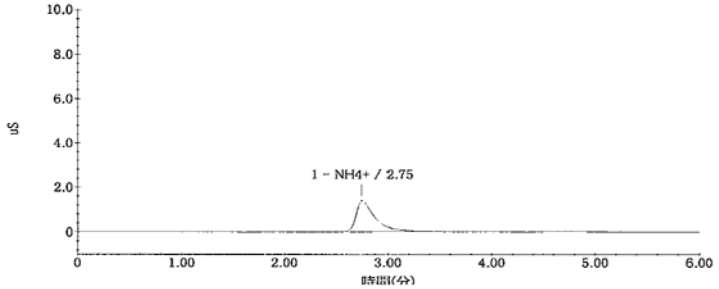
サンプル名：標準液-1
データファイル名：...¥B12011-標準液-1_002.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 9:41:13
分析者：朝日

ﾋﾟｰｸ情報：全ﾋﾟｰｸ

ﾋﾟｰｸ番号	保持時間	ピーク面積	成分名
1	2.75	181859	NH4+

標準液-1



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
ｻﾌﾟﾚｯｻｰ：CSRS(リサイクルモード/電流値100mA)
ｻﾝﾌﾟﾙ注入量：25μL
検出器：電気伝導度(ﾚﾝｼﾞ100μS)

付図 2-10 IC分析 NH_4^+ 標準液クロマトグラム

サンプル分析レポート

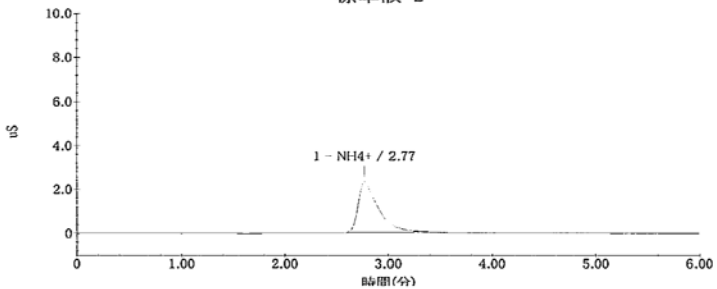
サンプル名 ：標準液-2
データファイル名 ：...¥B12011-標準液-2_003.DXD

メソッドファイル名 ：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時 ：2013/03/27 9:47:22
分析者 ：朝日

ピーク情報 ：全ピーク

ピーク番号	保持時間	ピーク面積	成分名
1	2.77	300150	NH4+

標準液-2



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
サプレッサー：CSRS(リサイクルモード/電流値100mA)
サンプル注入量：25μL
検出器：電気伝導度(レンジ100μS)

サンプル分析レポート

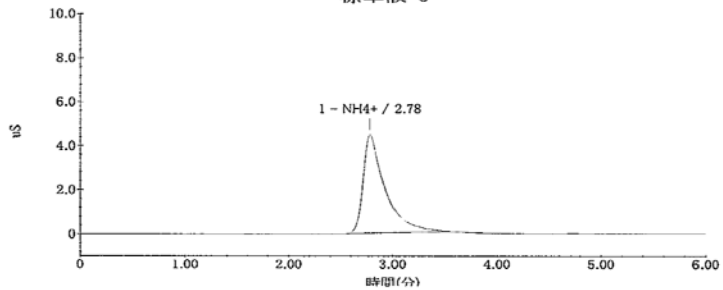
サンプル名 ：標準液-3
データファイル名 ：...¥B12011-標準液-3_004.DXD

メソッドファイル名 ：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時 ：2013/03/27 9:53:30
分析者 ：朝日

ピーク情報 ：全ピーク

ピーク番号	保持時間	ピーク面積	成分名
1	2.78	650003	NH4+

標準液-3



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
サプレッサー：CSRS(リサイクルモード/電流値100mA)
サンプル注入量：25μL
検出器：電気伝導度(レンジ100μS)

付図 2-11　IC分析 NH_4^+ 標準液クロマトグラム

サンプル分析レポート

サンプル名：標準液-4
データファイル名：...¥B12011-標準液-4_005.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 9:59:39
分析者：：朝日

ピーク情報：全ピーク			
ピーク番号	保持時間	ピーク面積	成分名
1	2.80	1181925	NH4+

分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/Lメタンスルホン酸水溶液
流量：1.0mL/min
サプレッサー：CSRS(リサイクルモード/電流値100mA)
サンプル注入量：25μL
検出器：電気伝導度(レンジ100μS)

サンプル分析レポート

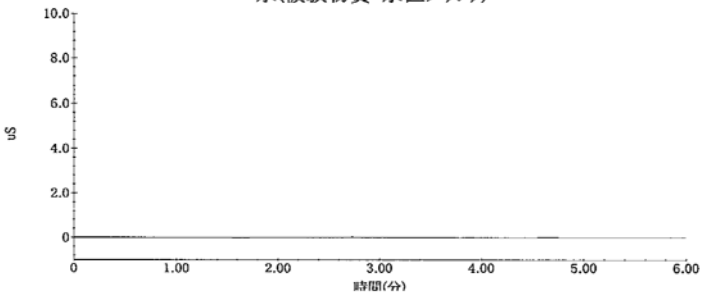
サンプル名：水(被験物質-水区ﾌﾞﾗﾝｸ)
データファイル名：...¥B12011-被験物質-水区ﾌﾞﾗﾝｸ_001.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 10:34:48
分析者：朝日

ﾋﾟｰｸ情報：全ﾋﾟｰｸ

ﾋﾟｰｸ番号	保持時間	ピーク面積	成分名

水(被験物質-水区ﾌﾞﾗﾝｸ)



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
ｻﾌﾟﾚｯｻｰ：CSRS(ﾘｻｲｸﾙﾓｰﾄﾞ/電流値100mA)
ｻﾝﾌﾟﾙ注入量：25μL
検出器：電気伝導度(ﾚﾝｼﾞ100μS)

サンプル分析レポート

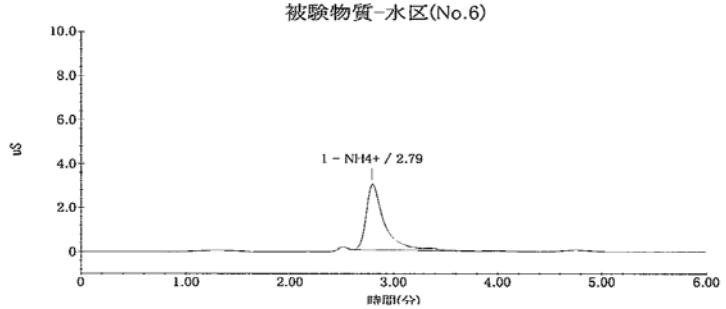
サンプル名：被験物質-水区(No.6)
データファイル名：...¥B12011-被験物質-水区(NO.6)_A002.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 11:24:08
分析者：朝日

ﾋﾟｰｸ情報：全ﾋﾟｰｸ

ﾋﾟｰｸ番号	保持時間	ピーク面積	成分名
1	2.79	361822	NH4+

被験物質-水区(No.6)



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
ｻﾌﾟﾚｯｻｰ：CSRS(ﾘｻｲｸﾙﾓｰﾄﾞ/電流値100mA)
ｻﾝﾌﾟﾙ注入量：25μL
検出器：電気伝導度(ﾚﾝｼﾞ100μS)

サンプル分析レポート

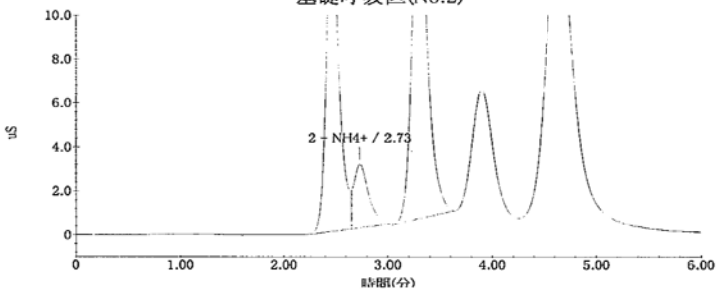
サンプル名 : 基礎呼吸区(No.2)
データファイル名 : ...¥B12011-基礎呼吸区(NO.2)_003.DXD

メソッドファイル名 : c:¥peaknet¥method¥分析条件¥b12011.met
分析日時 : 2013/03/27 10:47:04
分析者 : 朝日

ピーク情報 : 全ピーク

ピーク番号	保持時間	ピーク面積	成分名
1	2.46	1254076	
2	2.73	285496	NH4+
3	3.29	1648301	

基礎呼吸区(No.2)



分析条件

カラム : IonPac CS12A(4mmI.D. × 250mm)
移動相 : 30mmol/L-メタンスルホン酸水溶液
流量 : 1.0mL/min
サプレッサー : CSRS(リサイクルモード/電流値100mA)
サンプル注入量 : 25 μL
検出器 : 電気伝導度(レンジ 100 μS)

サンプル分析レポート

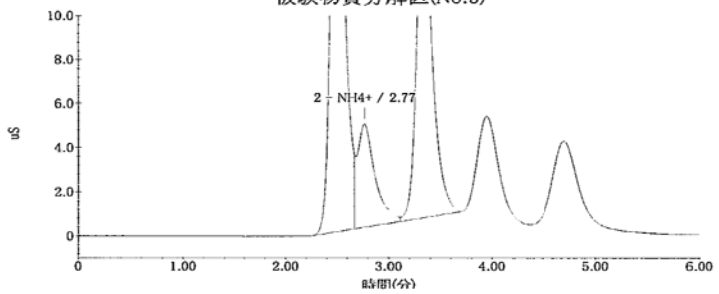
サンプル名 : 被験物質分解区(No.3)
データファイル名 : ...¥B12011-被験物質分解区(NO.3)_003.DXD

メソッドファイル名 : c:¥peaknet¥method¥分析条件¥b12011.met
分析日時 : 2013/03/27 11:30:17
分析者 : 朝日

ピーク情報 : 全ピーク

ピーク番号	保持時間	ピーク面積	成分名
1	2.49	2328814	
2	2.77	567218	NH4+
3	3.33	1600934	

被験物質分解区(No.3)



分析条件

カラム : IonPac CS12A(4mmI.D. × 250mm)
移動相 : 30mmol/L-メタンスルホン酸水溶液
流量 : 1.0mL/min
サプレッサー : CSRS(リサイクルモード/電流値100mA)
サンプル注入量 : 25 μL
検出器 : 電気伝導度(レンジ 100 μS)

サンプル分析レポート

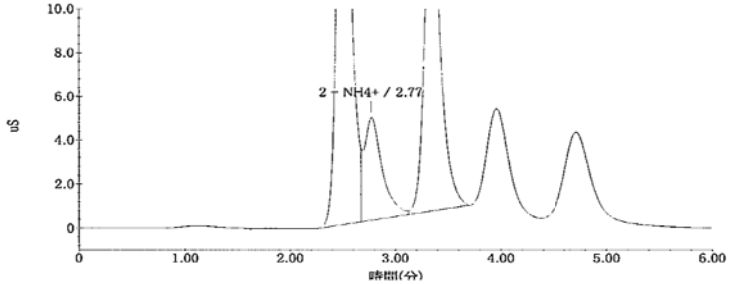
サンプル名：被験物質分解区(No.4)
データファイル名：...¥B12011-被験物質分解区(NO.4)_005.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 10:59:21
分析者：朝日

ピーク情報：全ピーク

ピーク番号	保持時間	ピーク面積	成分名
1	2.50	2324368	
2	2.77	561293	NH4+
3	3.34	1611371	

被験物質分解区(No.4)



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
サプレッサー：CSRS(リサイクルモード/電流値100mA)
サンプル注入量：25μL
検出器：電気伝導度(レンジ100μS)

サンプル分析レポート

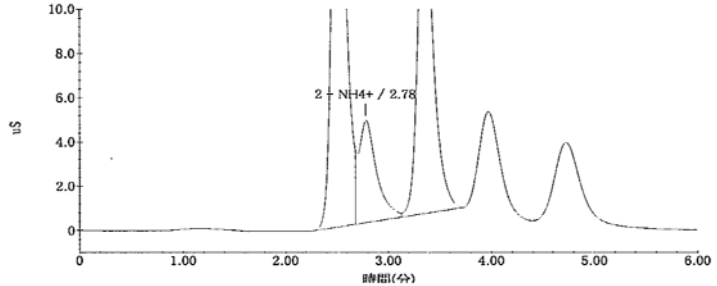
サンプル名：被験物質分解区(No.5)
データファイル名：...¥B12011-被験物質分解区(NO.5)_006.DXD

メソッドファイル名：c:¥peaknet¥method¥分析条件¥b12011.met
分析日時：2013/03/27 11:05:28
分析者：朝日

ピーク情報：全ピーク

ピーク番号	保持時間	ピーク面積	成分名
1	2.51	2337559	
2	2.78	563021	NH4+
3	3.35	1607421	

被験物質分解区(No.5)



分析条件

カラム：IonPac CS12A(4mmI.D.×250mm)
移動相：30mmol/L-メタンスルホン酸水溶液
流量：1.0mL/min
サプレッサー：CSRS(リサイクルモード/電流値100mA)
サンプル注入量：25μL
検出器：電気伝導度(レンジ100μS)

付図 2-15　ICP分析 Nb^{5+} 標準液測定結果

<<分析結果>>

分析名称 ：Nb定量_広栄ﾃｸﾉ　　担当者　：山本

試料名　：超純水　　分析日時 ：2013/03/28 13:38

<強度>

元素名	Nb
1回目	.159283
2回目	.159760
3回目	.159760
平均	.159601
R	.000477
S	.000275
CV	.172467

<<分析結果>>

分析名称 ：Nb定量_広栄ﾃｸﾉ　　担当者　：山本

試料名　：Nb 5.0μg/mL　　分析日時 ：2013/03/28 13:41

<強度>

元素名	Nb
1回目	12.6592
2回目	12.6043
3回目	12.6751
平均	12.6462
R	.070777
S	.037133
CV	.293630

<<分析結果>>

分析名称 ：Nb定量_広栄ﾃｸﾉ　　担当者　：山本

試料名　：Nb 25.0μg/mL　　分析日時 ：2013/03/28 13:43

<強度>

元素名	Nb
1回目	63.4257
2回目	63.6136
3回目	63.3282
平均	63.4558
R	.285389
S	.145061
CV	.228601

<<検量線計算結果>>

Nb　309.418 nm　分析名称：Nb定量_広栄ﾃｸﾉ

	a	b	c	d
検量線係数：	0.00000E+00	0.00000E+00	3.94581E-01	-3.04565E-02

標準偏差：.037165　相関係数：.999996　濃度単位：μg/mL

No.	試料名	濃度	強度	計算値	差	マーク
1	超純水	.000000	.159601	.032519	.032519	
2	Nb 5.0μg/mL	5.00000	12.6462	4.95949	-.040511	
3	Nb 25.0μg/mL	25.0000	63.4558	25.0080	.007992	

付図 2-16　ICP分析 Nb^{5+} 残留量及び水酸化ニオブ(V)生成量測定結果

<<分析結果>>

分析名称　：Nb定量_広栄テク1　　担当者　：山本

試料名　：被験物質分解区No.3ろ液　　分析日時　：2013/03/28 13:47

〈強度〉

元素名	Nb
1回目	.157441
2回目	.158547
3回目	.158243
平均	.158077
R	.001105
S	.000571
CV	.361243

〈濃度〉

元素名	Nb μg/mL
1回目	.031667
2回目	.032103
3回目	.031983
平均	.031918
R	.000436
S	.000225
CV	.705943

0.03 mg/L × 300 mL / 1000 = 0.01 mg

0.01 mg / 5.60 mg × 100 = 0%

<<分析結果>>

分析名称　：Nb定量_広栄テク1　　担当者　：山本

試料名　：被験物質分解区No.3ろ残　　分析日時　：2013/03/28 13:53

〈強度〉

元素名	Nb
1回目	14.7611
2回目	14.8356
3回目	14.7772
平均	14.7913
R	.074440
S	.039170
CV	.264817

〈濃度〉

元素名	Nb μg/mL
1回目	5.79401
2回目	5.82338
3回目	5.80035
平均	5.80591
R	.029373
S	.015456
CV	.266207

5.81 mg/L × 20 / 1000 × (100/2) = 5.81 mg

5.81 mg / 5.60 mg × 100 = 104%

付図 2-17　ICP分析 Nb^{5+} 残留量及び水酸化ニオブ(V)生成量測定結果

<<分析結果>>

分析名称　：Nb定量_広栄テクノ　　担当者　：山本

試料名　：被験物質分解区No.4ろ液　　分析日時　：2013/03/28 13:49

<強度>

元素名	Nb
1回目	.158286
2回目	.159197
3回目	.157961
平均	.158481
R	.001235
S	.000640
CV	.404029

<濃度>

元素名	Nb
	μg/mL
1回目	.032000
2回目	.032359
3回目	.031872
平均	.032077
R	.000487
S	.000253
CV	.787642

$0.03\,mg/L \times 300/1000 = 0.01\,mg$

$\frac{0.01\,mg}{5.60\,mg} \times 100 = 0\,\%$

<<分析結果>>

分析名称　：Nb定量_広栄テクノ　　担当者　：山本

試料名　：被験物質分解区No.4ろ残　　分析日時　：2013/03/28 13:55

<強度>

元素名	Nb
1回目	14.7167
2回目	14.7561
3回目	14.8137
平均	14.7622
R	.097022
S	.048795
CV	.330544

<濃度>

元素名	Nb
	μg/mL
1回目	5.77647
2回目	5.79201
3回目	5.81475
平均	5.79441
R	.038283
S	.019254
CV	.332280

$5.79\,mg/L \times 20/1000 \times (50/2) \times 2 = 5.79\,mg$

$\frac{5.79\,mg}{5.60\,mg} \times 100 = 103\,\%$

付図 2-18　ICP分析 Nb^{5+} 残留量及び水酸化ニオブ(V)生成量測定結果

付図 2-19　ICP分析 Nb^{5+} 残留量測定結果

<<分析結果>>

分析名称 ：Nb定量_広栄テクノ　　担当者　：山本

試料名　：被験物質-水区No.6　　分析日時 ：2013/03/28 10:26

<強度>

元素名	Nb
1回目	48.4600
2回目	48.3901
3回目	48.3375
平均	48.3958
R	.122528
S	.061469
CV	.127013

<濃度>

元素名	Nb μg/mL
1回目	18.8640
2回目	18.8366
3回目	18.8161
平均	18.8389
R	.047913
S	.024037
CV	.127590

$18.84\,mg/L \times \frac{300}{1000} = 5.65\,mg$

$\frac{5.65\,mg}{5.60\,mg} \times 100 = 101\%$

付図 3　　DOC分析測定結果

```
CAL CURVE F# 3   TC標準液 0.0mg/l
 1st STANDARD   TC    0.0mg/l VIAL: S1
        [x 1,   26μl, #WASH 4, SP  0min]
  #  AREA
  1   304
  2   311
  3   264
 - - - - -
 MN   293
 SD    25
 CV  8.65 %

      DATE  03(MAR)-27-2013  10:50
```

```
CAL CURVE F# 3   TC標準液 20.0mg/L
 2nd STANDARD   TC   20.0mg/l VIAL: S2
        [x 1,   26μl, #WASH 4, SP  0min]
  #  AREA
  1 19054
  2 19190
  3 19226
 - - - - -
 MN 19156
 SD    90
 CV  0.47 %

      DATE  03(MAR)-27-2013  10:59
```

```
CAL CURVE F# 4   IC標準液 0.0mg/L
 1st STANDARD   IC    0.0mg/l VIAL: S1
        [x 1,   33μl, #WASH 4, SP  0min]
  #  AREA
  1   249
  2   201
  3   174
 - - - - -
 MN   208
 SD    37
 CV  18.2 %

      DATE  03(MAR)-27-2013  11:04
```

```
CAL CURVE F# 4   IC標準液 20.0mg/L
 2nd STANDARD   IC   20.0mg/l VIAL: S3
        [x 1,   33μl, #WASH 4, SP  0min]
  #  AREA
  1 25043
  2 25133
  3 25085
 - - - - -
 MN 25087
 SD    45
 CV  0.17 %

      DATE  03(MAR)-27-2013  11:13
```

```
SAMPLE#  42   TC  VIAL: 42 超 純 水
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  574  0.298
   2  411  0.125
   3  486  0.204
   4  808  0.546
   5  398  0.111
 - - - - - - - - - - - - - - -
 MN   535  0.257
 SD   167  0.177
 CV  31.3 %
 COR CONC DIL  0.257 [x 1.0]
 COR CONC INJ  0.257 [x 1.0]

      DATE  03(MAR)-27-2013  11:24
```

（手書き：不採用のため削除。残存のないことを確認。130327 飛松）

```
          急性試験開始日使用水
SAMPLE#   1   TC  VIAL:  1
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1   491  0.210
   2   483  0.201
   3   530  0.251
 - - - - - - - - - - - - - - -
 MN   501  0.220
 SD    25  0.026
 CV  5.01 %
 COR CONC DIL  0.220 [x 1.0]
 COR CONC INJ  0.220 [x 1.0]

      DATE  03(MAR)-27-2013  11:30
```

```
          急性試験開始日使用水
SAMPLE#   1   IC  VIAL:  1
 [x 1,   33μl, C#  4, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1   232  0.019
   2   274  0.053
   3   232  0.019
 - - - - - - - - - - - - - - -
 MN   246  0.030
 SD    24  0.019
 CV  9.85 %
 COR CONC DIL  0.030 [x 1.0]
 COR CONC INJ  0.030 [x 1.0]

SAMPLE#   1   TOC(TC-IC)    0.190 mg/l

      DATE  03(MAR)-27-2013  11:36
```

```
SAMPLE#   2   TC  VIAL:  2 基礎呼吸区 No.2
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  1720  1.513
   2  1674  1.464
   3  1613  1.399
 - - - - - - - - - - - - - - -
 MN  1669  1.458
 SD    53  0.057
 CV  3.21 %
 COR CONC DIL  1.458 [x 1.0]
 COR CONC INJ  1.458 [x 1.0]

      DATE  03(MAR)-27-2013  11:43
```

```
SAMPLE#   2   IC  VIAL:  2 基礎呼吸区 No.2
 [x 1,   33μl, C#  4, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1   641  0.348
   2   716  0.408
   3   552  0.276
 - - - - - - - - - - - - - - -
 MN   636  0.344
 SD    82  0.066
 CV  12.9 %
 COR CONC DIL  0.344 [x 1.0]
 COR CONC INJ  0.344 [x 1.0]

SAMPLE#   2   TOC(TC-IC)    1.114 mg/l

      DATE  03(MAR)-27-2013  11:49
```

```
SAMPLE#   3   TC  VIAL:  3 被験物質分解区 No.3
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  3400  3.294
   2  3499  3.399
   3  3415  3.310
 - - - - - - - - - - - - - - -
 MN  3438  3.334
 SD    53  0.056
 CV  1.55 %
 COR CONC DIL  3.334 [x 1.0]
 COR CONC INJ  3.334 [x 1.0]

      DATE  03(MAR)-27-2013  11:56
```

```
SAMPLE#   3   IC  VIAL:  3 被験物質分解区 No.3
 [x 1,   33μl, C#  4, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  3214  2.416
   2  3275  2.465
   3  3171  2.382
 - - - - - - - - - - - - - - -
 MN  3220  2.421
 SD    52  0.042
 CV  1.62 %
 COR CONC DIL  2.421 [x 1.0]
 COR CONC INJ  2.421 [x 1.0]
SAMPLE#   3   TOC(TC-IC)    0.913 mg/l

      DATE  03(MAR)-27-2013  12:03
```

```
SAMPLE#   4   TC  VIAL:  4 被験物質分解区 No.4
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  4042  3.974
   2  4121  4.058
   3  4121  4.058
 - - - - - - - - - - - - - - -
 MN  4094  4.030
 SD    45  0.048
 CV  1.11 %
 COR CONC DIL  4.030 [x 1.0]
 COR CONC INJ  4.030 [x 1.0]

      DATE  03(MAR)-27-2013  12:10
```

```
SAMPLE#   4   IC  VIAL:  4 被験物質分解区 No.4
 [x 1,   33μl, C#  4, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  4229  3.232
   2  4246  3.246
   3  4278  3.271
 - - - - - - - - - - - - - - -
 MN  4251  3.250
 SD    24  0.020
 CV  0.58 %
 COR CONC DIL  3.250 [x 1.0]
 COR CONC INJ  3.250 [x 1.0]

SAMPLE#   4   TOC(TC-IC)    0.780 mg/l

      DATE  03(MAR)-27-2013  12:17
```

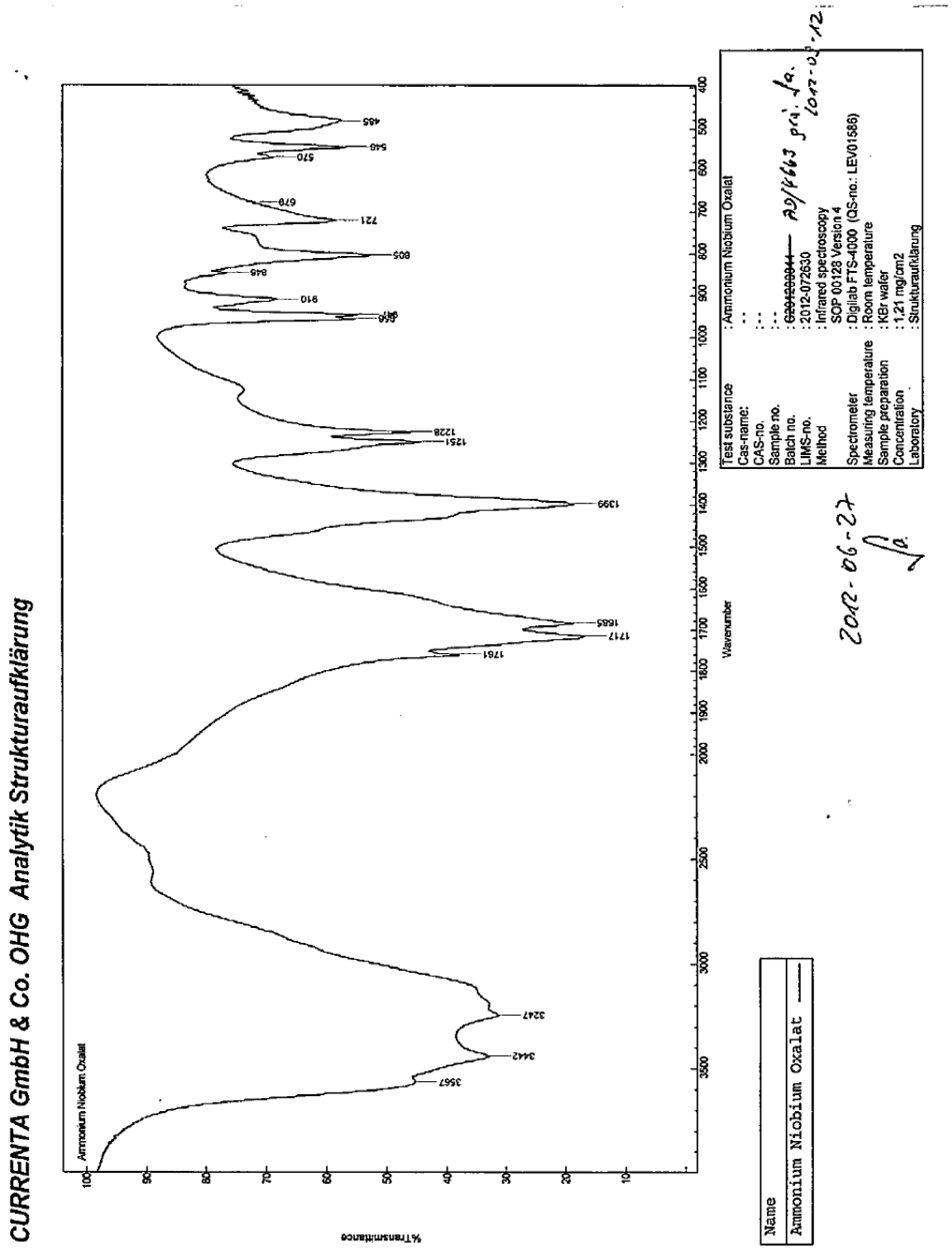
```
SAMPLE#   5   TC  VIAL:  5 被験物質分解区 No.5
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  4018  3.949
   2  4070  4.004
   3  3966  3.894
 - - - - - - - - - - - - - - -
 MN  4018  3.949
 SD    52  0.055
 CV  1.29 %
 COR CONC DIL  3.949 [x 1.0]
 COR CONC INJ  3.949 [x 1.0]

      DATE  03(MAR)-27-2013  12:24
```

```
SAMPLE#   5   IC  VIAL:  5 被験物質分解区 No.5
 [x 1,   33μl, C#  4, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1  3894  2.963
   2  4085  3.116
   3  4204  3.212
 - - - - - - - - - - - - - - -
 MN  4061  3.097
 SD   156  0.125
 CV  3.85 %
 COR CONC DIL  3.097 [x 1.0]
 COR CONC INJ  3.097 [x 1.0]

SAMPLE#   5   TOC(TC-IC)    0.852 mg/l

      DATE  03(MAR)-27-2013  12:31
```

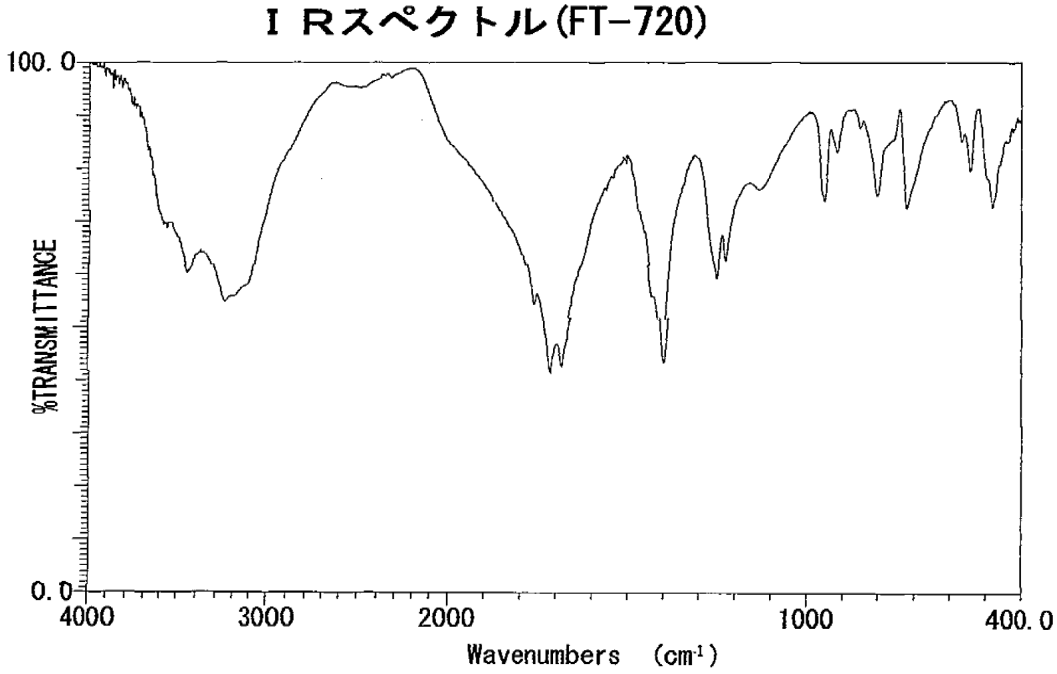
```
SAMPLE#   6   TC  VIAL:  6 被験物質一水区 No.6
 [x 1,   26μl, C#  3, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1 13692  14.20
   2 13474  13.97
   3 13647  14.15
 - - - - - - - - - - - - - - -
 MN 13604  14.11
 SD   115  0.122
 CV  0.84 %
 COR CONC DIL  14.11 [x 1.0]
 COR CONC INJ  14.11 [x 1.0]

      DATE  03(MAR)-27-2013  16:22
```

```
SAMPLE#   6   IC  VIAL:  6 被験物質一水区 No.6
 [x 1,   33μl, C#  4, #WASH 4, SP  0min]
   #  AREA mg/l       C#  μl RG
   1   226  0.014
   2   168  -0.03
   3   115  -0.07
 - - - - - - - - - - - - - - -
 MN   169  -0.03
 SD    55  0.044
 CV  32.7 %
 COR CONC DIL  -0.03 [x 1.0]
 COR CONC INJ  -0.03 [x 1.0]

SAMPLE#   6   TOC(TC-IC)    14.14 mg/l

      DATE  03(MAR)-27-2013  16:28
```

付図 4　　IRｽﾍﾟｸﾄﾙ ANO 同定(被験物質提供者取得ﾃﾞｰﾀ)



付図 5　　IRｽﾍﾟｸﾄﾙ ANO 物質確認(試験施設(2013.2.6)取得ﾃﾞｰﾀ)

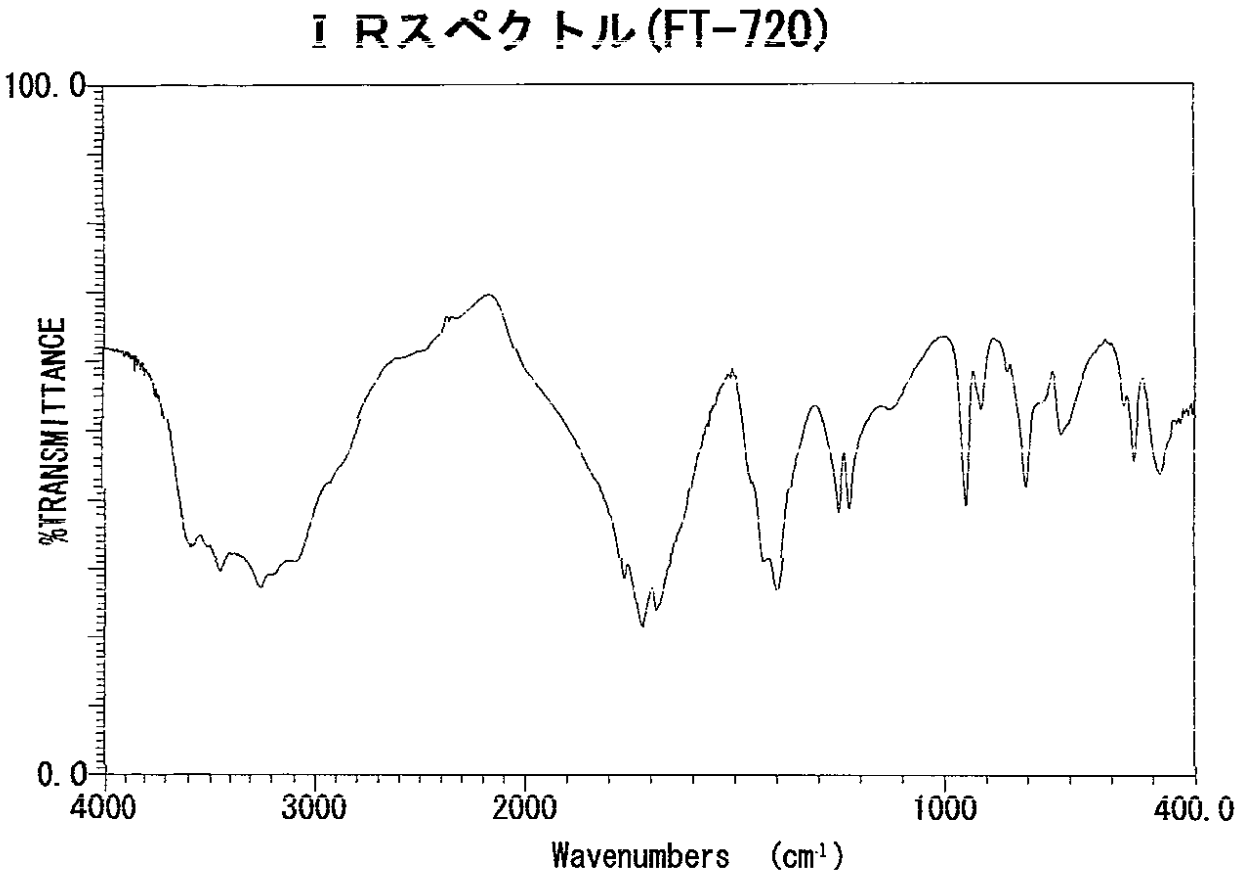


ファイル名	:	B12011-1
タイトル	:	ANO Lot No. AD/4638
測定日時	:	2013年02月06日　08時54分03秒
測定分解能	:	4 cm^{-1}
スキャン回数	:	10 回
測定ゲイン	:	2
コメント	:	試験No. B12011 物質確認 KBr-disc法

試験担当者　：　朝日　　2013 年　2 月　6日

試験責任者　：　山本　　2013 年　2 月　6　日

付図 6　　IRｽﾍﾟｸﾄﾙ ANO 安定性確認(暴露試験開始日(2013.2.27)取得ﾃﾞｰﾀ)

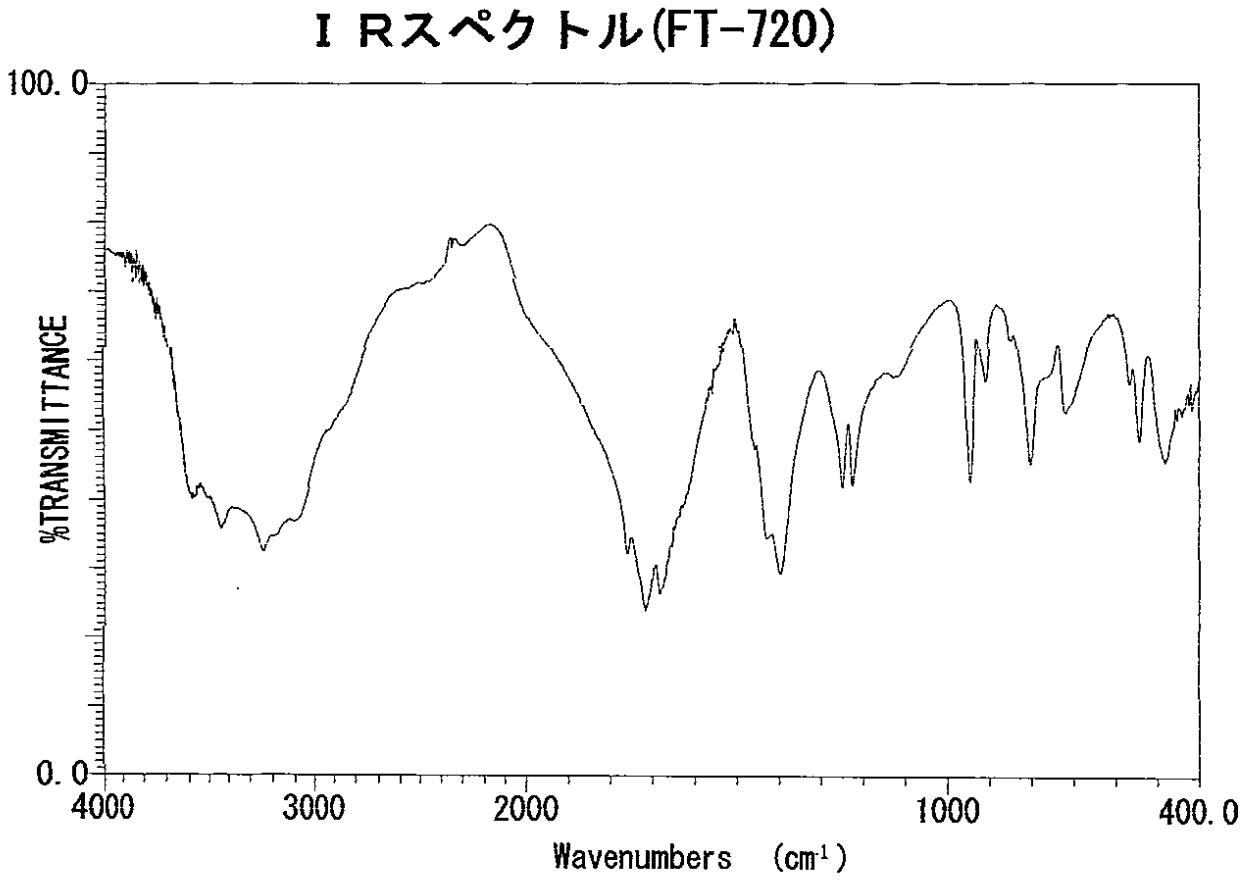


ファイル名　：B12011-2
タイトル　：ANO Lot No. AD/4638
測定日時　：2013年02月27日　13時46分38秒
測定分解能　：4 cm-1
スキャン回数　：10 回
測定ゲイン　：1
コメント　：試験No. B12011
安定性確認　KBr-disk法

試験担当者　：飛松　　2013年 2月27日

試験責任者　：　　2013 年 2月27日

付図 7　　IRｽﾍﾟｸﾄﾙ ANO 安定性確認(暴露試験終了日(2013.3.27)取得ﾃﾞｰﾀ)



ファイル名　：B12011-3
タイトル　：ANO Lot No. AD/4638
測定日時　：2013年03月27日　10時52分08秒
測定分解能　：4 cm^{-1}
スキャン回数　：10 回
測定ゲイン　：1
コメント　：試験No. B12011
安定性確認　KBr-disk法

試験担当者　：飛松　　2013年 3月27日

試験責任者　：　　2013 年 3 月 27 日

付図 8　　被験物質の物質確認及び保管条件下に於ける安定性確認表

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物質確認及び保管条件下に於ける安定性確認表

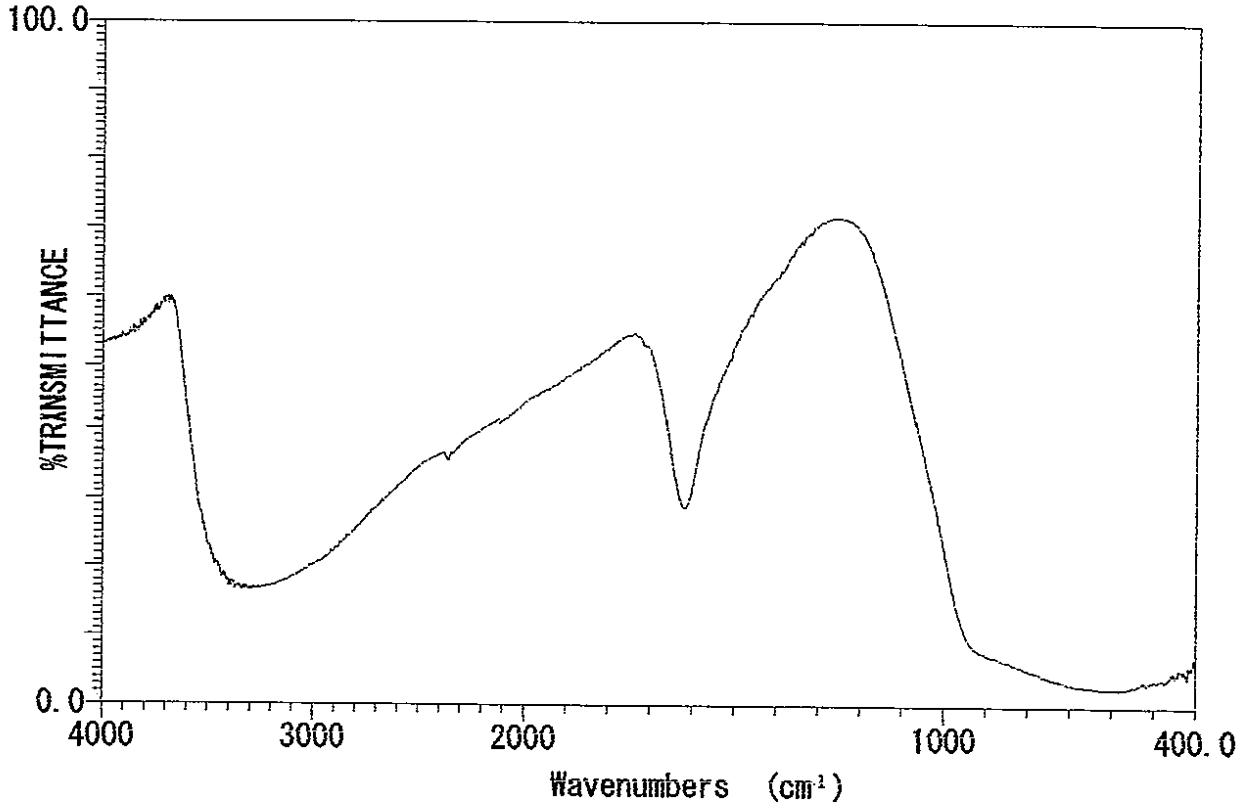
試験No.　B12011

被験物質名　Ammonium Niobium Oxalate (ANO)

	年月日	試験担当者	評価	試験責任者	備考
物質確認	130206	朝日	被験物質に相違ないことを確認した。	130206 上本	
安定性の確認	130227	飛松	安定であることを確認した。	130227 上本	暴露試験開始日
安定性の確認	130327	飛松	安定であることを確認した。	130327 上本	暴露試験終了日

付図 9　　IRｽﾍﾟｸﾄﾙ 水酸化ﾆｵﾌﾞ(Ⅴ)標準品(暴露試験終了日(2013.3.27)取得ﾃﾞｰﾀ)

ＩＲスペクトル(FT-720)



ファイル名　：B1211nb1
タイトル　：水酸化ﾆｵﾌﾞ（V）　LotNo.63515
測定日時　：2013年03月27日　13時51分53秒
測定分解能　：4 cm^{-1}
ｽｷｬﾝ回数　：10 回
ｺﾒﾝﾄ　：試験No.B12011
　　KBr-disc法

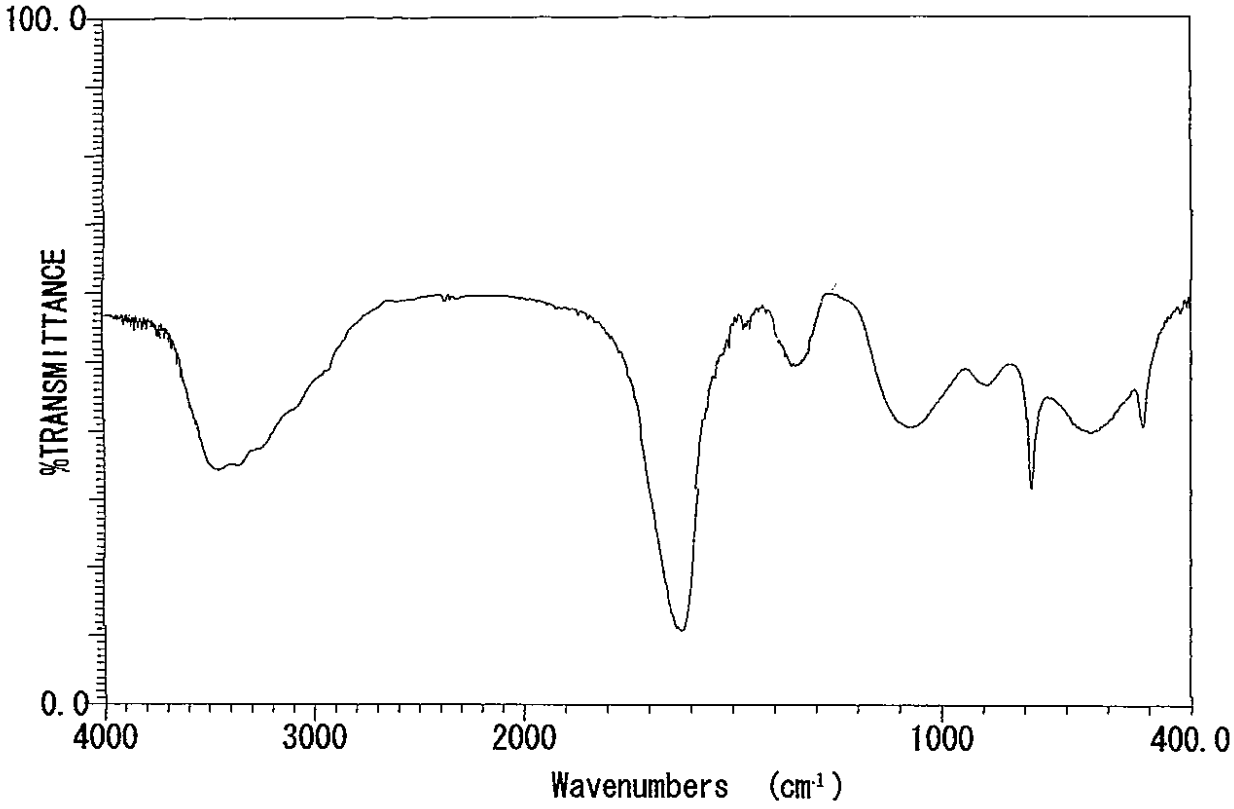
試験担当者　：　山本　2013 年 3 月 27 日

試験責任者　：　山本　2013 年 3 月 27 日

付図 10　IRｽﾍﾟｸﾄﾙ ﾆｵﾌﾞｲｵﾝの分解物である水酸化ﾆｵﾌﾞ(Ⅴ)の定性分析

(暴露試験終了日(2013.3.27)取得ﾃﾞｰﾀ)

ＩＲスペクトル(FT-720)



ファイル名　：B12011-8
タイトル　：被験物質分解区No.4
測定日時　：2013年03月27日 13時56分32秒
測定分解能　：4 cm^{-1}
スキャン回数　：10 回
測定ゲイン　：4
コメント　：試験No.B12011
KBr-disc法

試験担当者　：　　2013 年 3 月 27日

試験責任者　：　　2013 年 3 月 27 日