AR226_3302

Accession No.	C03-3620
Study No.	33620

Study Report

Analysis of Residual Solvent (Ethanol) in [redacted]
(non-GLP study)

October 14, 2003

Chemicals Evaluation and Research Institute, Japan
--Kurume Laboratory--

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Title:	Analysis of Residual Solvent (Ethanol) in [redacted] (non-GLP study)
Commissioned by:	DuPont KK 1-8-1 Shimo-meguro, Meguro-ku, Tokyo 153-0064
Testing Facility:	Kurume Laboratory Chemicals Evaluation and Research Institute, Japan (CERI) 19-14 Chuo-machi, Kurume-shi, Fukuoka Prefecture 830-0023
Purpose of Study:	To determine the level of ethanol remaining in Forafac® 1157.
Schedule:	Study began on: July 29, 2003 Test date: July 29, 2003 Study completed on: October 14, 2003
Personnel:	Coordinator: Yasuko MATSUNOBE,* Testing Section 1 Researcher: Tomohiko YOSHIDA
Approval of Study Report:	[signed] Yasuko MATSUNOBE Study Coordinator October 14, 2003

* Translator's Note: Spellings of personal names may need to be verified.

Summary

Title of Study

Analysis of Residual Solvent (Ethanol) in [redacted] (non-GLP study)

Study Conditions

(1) Concentration of sample furnished: 1,000 mg/L
(2) Analysis: Analysis of ethanol by gas chromatography (GC)

Test Results

Content of ethanol in sample furnished: 0%

1. Test Substance

Name: [redacted]

Lot No.: [redacted]

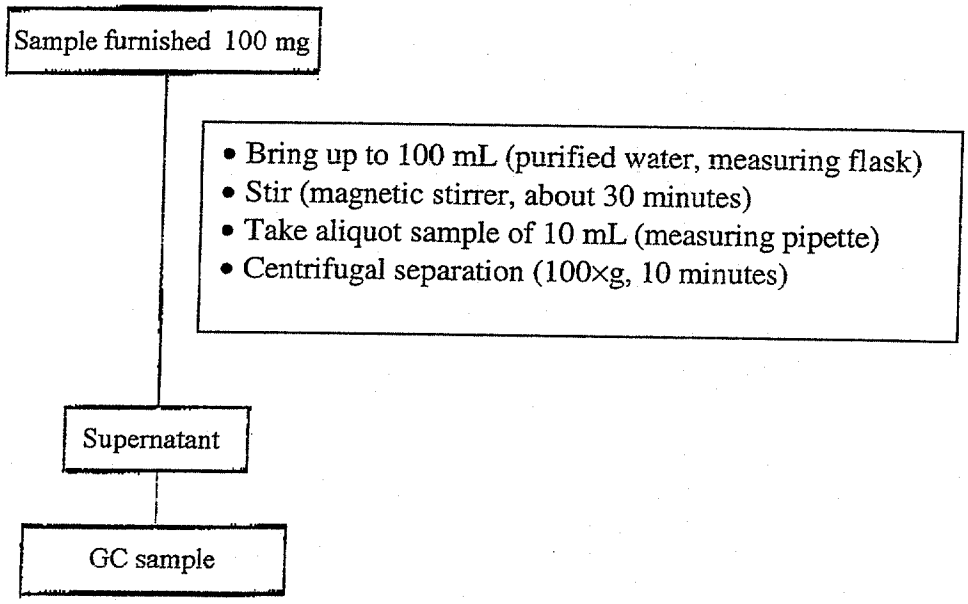
2. Execution of Study

Gas chromatography (GC) samples prepared as described in section 2.1 below were analyzed in accordance with section 2.2 on the next page, based upon which the ethanol contents of the samples furnished for testing were computed.

2.1 Preparation of Sample for Analysis

Pretreatment was carried as shown in the flow scheme below so as to prepare a GC sample for ethanol analysis.

Flow Scheme



2.2 Ethanol Analysis by Gas Chromatography

The GC sample prepared in section 2.1 above was analyzed for ethanol using the quantitative determination conditions shown below. The ethanol concentration in the GC sample was determined by comparing the peak surface area on chromatograms for a 40.0 mg/L standard solution and for the GC sample, and calculating the ratio between the two (see Fig. 2).

(1) Quantitative Determination Conditions:

Apparatus:	Gas chromatograph GC-17A (Shimadzu Corp.)
Detector:	hydrogen flame ionization detector (FID)
Column:	INNOWAX, film thickness 0.25 μm (Agilent Technologies)
Column temperature:	40°C
Sample insertion port temperature:	200°C
Carrier gas:	helium column flow rate, 1.7 mL/min
Hydrogen:	60 kPa
Air:	50 kPa
Injection rate:	2 μL
Split ratio:	5:1
Detector	
Temperature:	230°C
Sensitivity:	10°

(2) Preparation of Standard Solution:

A standard solution for determining the ethanol concentration in analysis samples was prepared as follows.

One hundred milligrams of ethanol standard product (Nakalai Tesque, Inc.; grade 1 reagent) was precisely weighed out and dissolved in purified water to form a 1,000 mg/L ethanol solution. This solution was diluted with purified water to form a 40.0 mg/L standard solution.

(3) Preparation of Calibration Curve:

Standard solutions having concentrations of 10.0, 20.0 and 40.0 mg/L were prepared in the same way as described in (2) above. These were then analyzed under the quantitative determination conditions given in (1) above, following which a calibration curve was prepared from the peak areas on each of the chromatograms thus obtained and the respective solution concentrations (see Fig. 1).

2.3 Calculation of Ethanol Content

The ethanol content was calculated using the following formula.

$$\text{Content (\%)} = \frac{A(D)}{A(A)} \times 100$$

where A (D): Amount of ethanol detected in GC sample (mg)
A (A): Amount of furnished sample used to prepare GC sample (= 100 g)

3. Results of Study

The results of analysis are given below.

	Amount of sample furnished (ng)	Amount of ethanol detected (mg)	Ethanol content (%)	Table	Fig.
GC sample	100	0	0	1	2

As is apparent from the above table, ethanol was not detected in the GC samples. Accordingly, it would appear that the sample furnished for testing was free of residual ethanol.

Sample description	A	D	E	F
Standard solution 40.0mg/L (ethanol)	57545			
Solvent blank	n.d.			
Sample for GC analysis (1000 mg/L solution of the item supplied by the sponsor)	n.d.	0	0	0

Amount of the item supplied by the sponsor added : 100 (mg)

A : Peak area (μV·sec)

B : Final volume : 10 (mL)

C : Ratio of portion used for analysis : 10/100

D : Detected amount of ethanol (mg)

$D = G \times (A / A(Standard)) \times (B / C) / 1000$

E : Percentage content of ethanol in the item supplied by the sponsor (%)

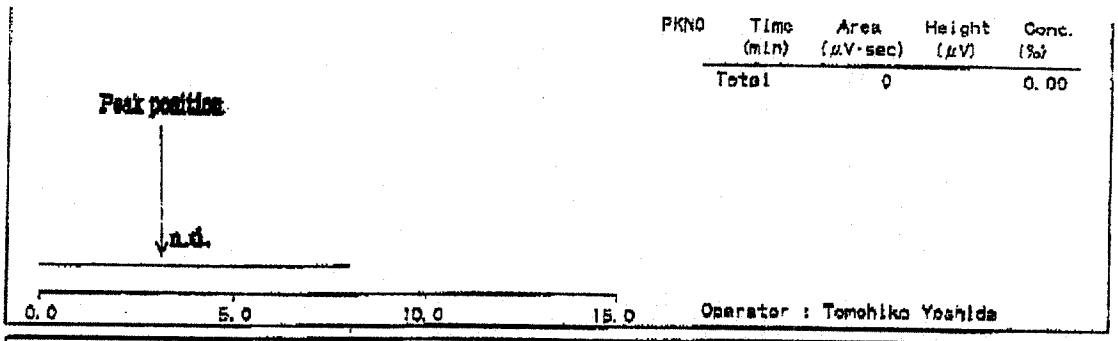
$E = D / 100 (mg) \times 100$

F : Average percentage content of ethanol in the item supplied by the sponsor (%)

G : Concentration of ethanol of standard solution : 40.0 (mg/L)

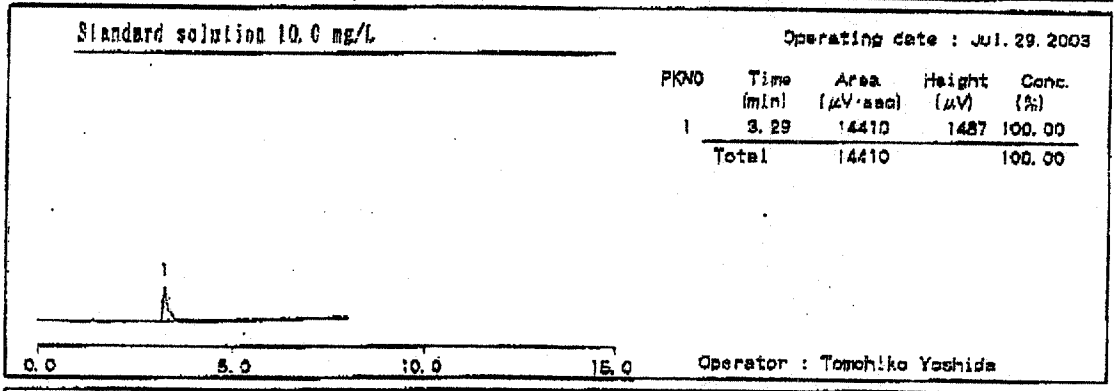
See Fig. 2

October 8, 2003 Name T. Yoshida



PKNO	Time (min)	Area (μV·sec)	Height (μV)	Conc. (%)
Total		0		0.00

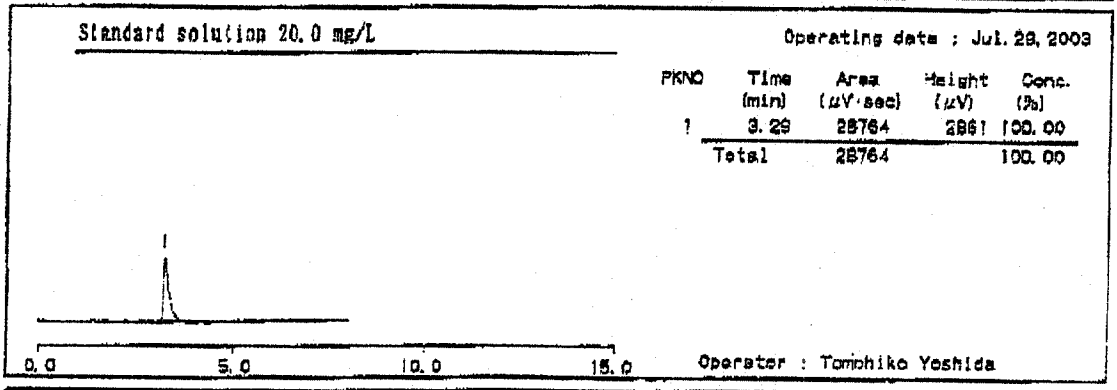
Operator : Tomohiko Yoshida



Operating date : Jul. 29. 2003

PKNO	Time (min)	Area (μV·sec)	Height (μV)	Conc. (%)
1	3.29	14410	1487	100.00
Total		14410		100.00

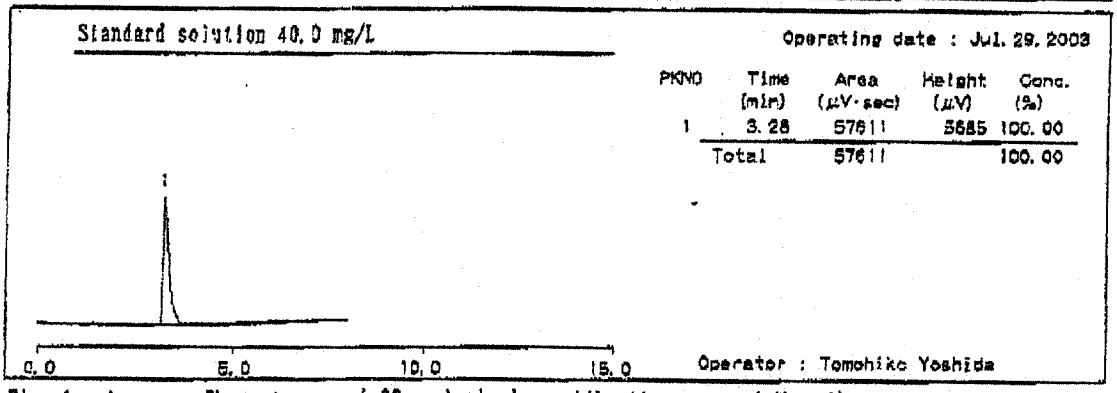
Operator : Tomohiko Yoshida



Operating date : Jul. 29. 2003

PKNO	Time (min)	Area (μV·sec)	Height (μV)	Conc. (%)
1	3.29	28764	2861	100.00
Total		28764		100.00

Operator : Tomohiko Yoshida



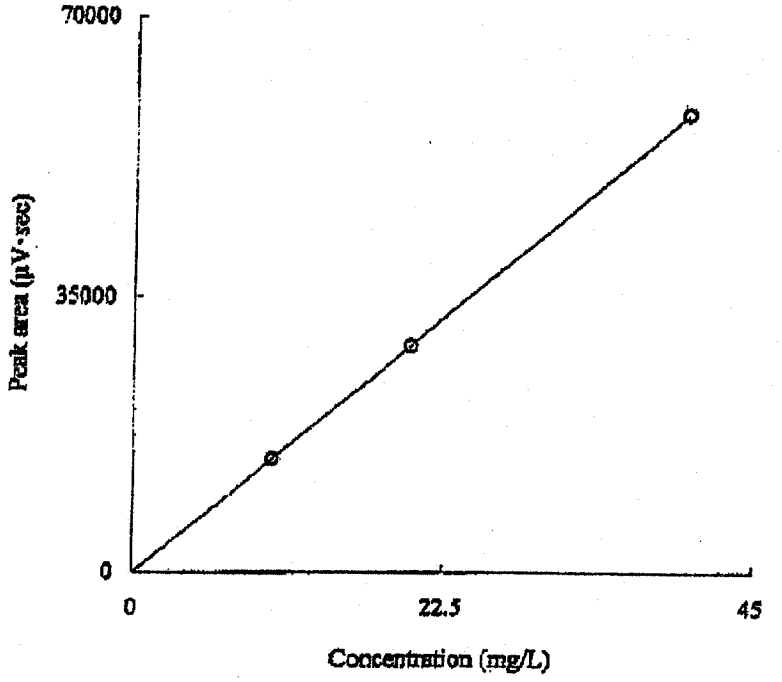
Operating date : Jul. 29. 2003

PKNO	Time (min)	Area (μV·sec)	Height (μV)	Conc. (%)
1	3.28	57611	5685	100.00
Total		57611		100.00

Operator : Tomohiko Yoshida

Fig. 1 - 1 Chromatogram of GC analysis for calibration curve (ethanol).

Date : Jul. 29. 2003 Name : T. Yoshida

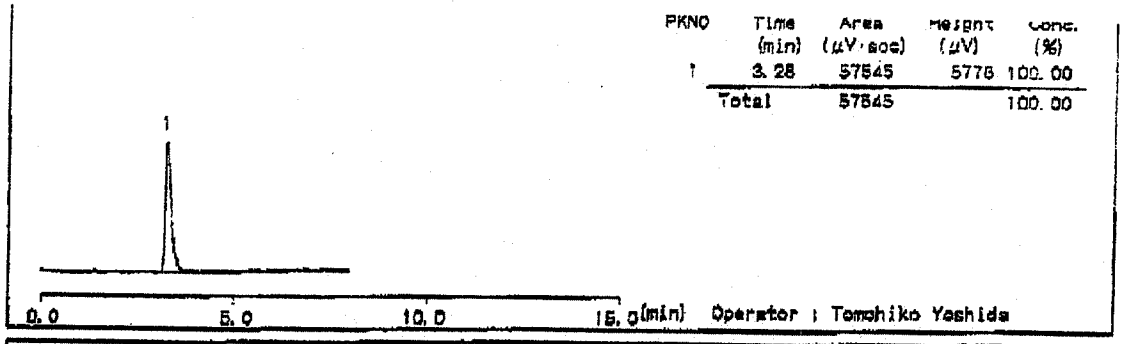


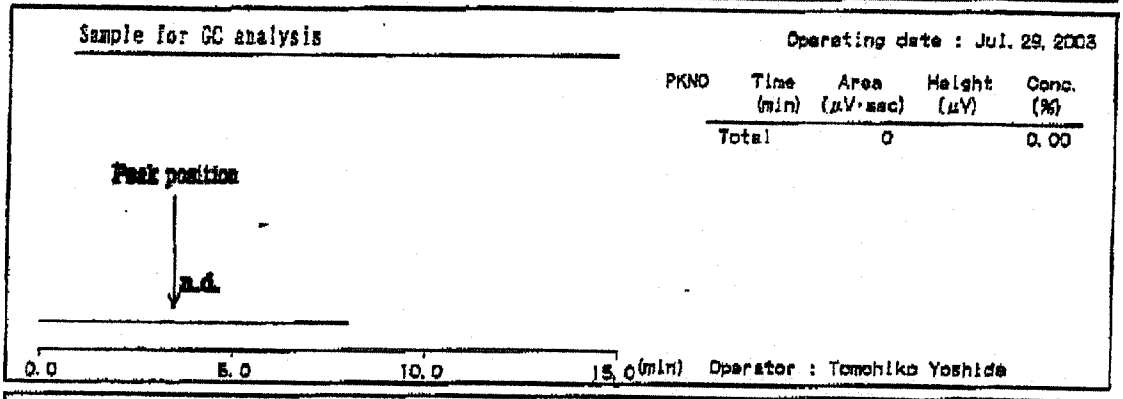
y = 1440x
r = 1.00

Concentration (mg/L)	Peak area (μV·sec)
10.0	14410
20.0	28764
40.0	57611

Fig. 1 - 2 Calibration curve of ethanol.

October 9, 2003 Name T. Yoshida





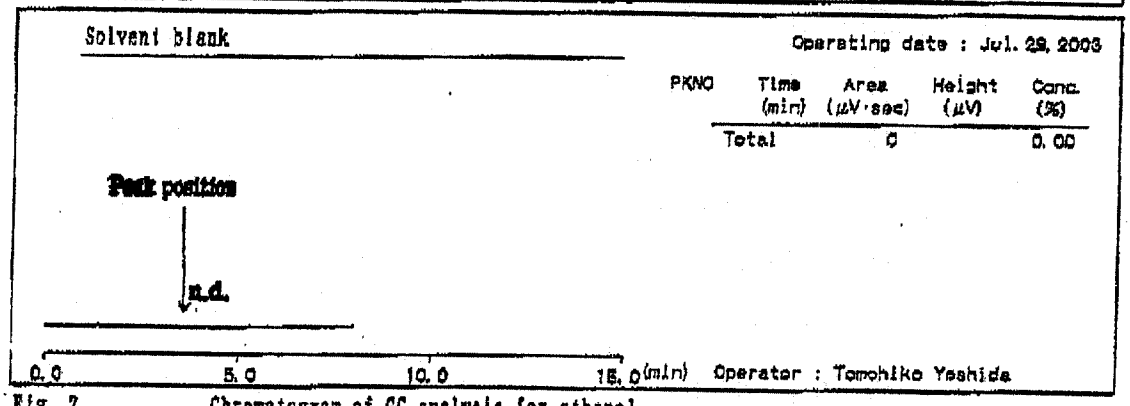


Fig. 2 Chromatogram of GC analysis for ethanol.

Date : Jul. 29, 2003 Name : T. Yoshida

Translation: Language Services
F. Metreaud
December 4, 2003