

Isolation and Identification of Volatile Compounds from a Wine Using Solid Phase Extraction, Gas Chromatography, and Gas Chromatography/Mass Spectrometry

Koji Wada[†] and Takayuki Shibamoto*

Department of Environmental Toxicology, University of California, Davis, California 95616

Recovery efficiencies of main volatile components of wine from porous polymer Porapak Q were investigated using dichloromethane, ethyl ether, and pentane as solvents. The nine wine components used for recovery tests were 2-methyl-1-propanol, 3-methyl-1-butanol, 2-phenylethanol, ethyl hexanoate, ethyl octanoate, diethyl butanedioate, hexanoic acid, octanoic acid, and decanoic acid. Dichloromethane showed the highest recovery efficiency, followed by ethyl ether and pentane. Nearly 80% recovery for ethyl hexanoate and ethyl octanoate was obtained by dichloromethane for a concentration of 10 ppm. Over 96% recovery was obtained by dichloromethane for 2-phenylethanol, diethyl butanedioate, hexanoic acid, and octanoic acid for 10 ppm. Volatile components from a commercial wine were trapped on Porapak Q and subsequently recovered using an organic solvent. The major compounds identified in the three extracts were 3-methyl-1-butanol, 2-phenylethanol, octanoic acid, monoethyl butanedioate, and hexanoic acid.

Keywords: *Gas chromatography; solid phase extraction; wine volatile chemicals*

INTRODUCTION

Numerous aroma constituents have been found in wine (Baumes et al., 1986; Herraiz et al., 1991). To determine these constituents, many methods for isolating volatiles from wine, such as the headspace collection (Salinas et al., 1994), supercritical fluid (Blanch et al., 1995), ultrasound (Cocito et al., 1995), and microextraction (Ferreira et al., 1993) methods, have been reported. In addition, a purge and trap method that involves a direct injection of the sample into a gas chromatographic column has been developed (Shimoda et al., 1993; Garcia-Jares et al., 1995; Villen et al., 1995). Etievant et al. (1986), who compared various headspace techniques using a wine sample, reported that the technique chosen was dependent on the purpose of the experiments and the number of samples to be examined.

Liquid-liquid extraction is a commonly used method to isolate volatile compounds from wine. However, solvent choice is somewhat difficult because the ethanol in wine behaves as a surfactant. All these methods, however, have one or more drawbacks, such as low extraction efficiency, low reproducibility, or high cost.

Porous polymer, which was developed as a column-packing material for gas chromatography and HPLC, has been used to recover volatiles from alcoholic beverages such as beer (Hawthorne et al., 1987) and sake (Sakamoto et al., 1993). Edwards and Beelman (1990) reported 14 aroma components in the wine volatiles recovered using Amberlite XAD-2 resin. Gunata et al. (1985) also used XAD-2 resin to isolate free and glycosidically bound aroma components from a wine sample. Gelsomini et al. (1990) obtained 45 volatile and 14 nonvolatile components of wine by using commercial C-8 reversed phase columns.

In the present study, the factors—including choice/quantity of solvent and the amount of resin—affecting

recovery efficiency of volatiles from wine using Porapak Q resin were investigated. Also, a quantitative method for measuring wine flavors using Porapak Q resin was established.

EXPERIMENTAL PROCEDURES

Materials. A standard stock solution was prepared by adding 0.1, 0.5, 1.0, and 2.0 mg each of 2-methyl-1-propanol, 3-methyl-1-butanol, 2-phenylethanol, ethyl hexanoate, ethyl octanoate, diethyl butanedioate, hexanoic acid, octanoic acid, and decanoic acid to 200 mL of 10.5% ethanol (0.5, 2.5, 5, and 10 ppm).

A wine (1 gal bottle) was purchased from a local market (Carlo Rossi Chablis, 10.5% ethanol content) and stored at 5 °C. All experiments were conducted using wine from the same bottle.

Porapak Q resin (50–80 mesh) was obtained from Supelco, Inc. (Bellefonte, PA). Prior to use in the experiment, the resin was extracted with hexane for 3 h with a Soxhlet extractor and stored in an air-tight jar at 25 °C.

Determination of the Optimum Amounts of Porapak Q Resin and Sample for Recovery of Wine Components Using Dichloromethane. In order to obtain optimum conditions for wine component recovery, the following recovery experiments were performed using a standard stock solution containing nine wine components: (1) The sample volume varied (50, 100, 200, 300, 400 mL) while the amount of Porapak Q resin (5 mL) and dichloromethane (60 mL) were constant. (2) The resin amount varied (2.5, 5.0, 10 mL) while fixed amounts of the sample (200 mL) and dichloromethane (60 mL) were used. (3) The amount of dichloromethane was the variable ranging from 15 to 75 mL (15 mL increments), while the sample (200 mL) and resin (5 mL) were constant.

Porapak Q resin was packed in a 30 cm × 2.2 cm i.d. glass column equipped with a filter. The resin was washed with 100 mL each of methanol, dichloromethane, and distilled water in a series. Then a standard stock solution was added to the column. After the wine components were trapped on the resin, the column was eluted with dichloromethane. After 5 μ L of an aqueous solution of cyclohexanol (2%) was added as a GC internal standard, the extract was dried over anhydrous sodium sulfate for 12 h. The sodium sulfate was removed and then the solution was concentrated, using a rotary flash evaporator to approximately 1 mL in volume. Further con-

[†] Present address: Department of Bioscience and Biotechnology, Faculty of Agriculture, University of The Ryukyus, Nishihara, Okinawa 903-01, Japan.

centration was conducted under a purified nitrogen stream to precisely 100 μ L volume. Relative recovery efficiencies among samples were conducted using a gas chromatographic peak area ratio between each compound and the internal standard.

The resin was regenerated by washing with 100 mL each of 1% NaOH, 1% HCl solutions, and water in a series.

Recovery Efficiency of Wine Components from Porapak Q Resin with Dichloromethane, Ethyl Ether, and Pentane. Experiments were performed by the procedures described above using 5 mL of resin, 200 mL of a standard solution, and 60 mL each of dichloromethane, ethyl ether, or pentane. The quantitative analysis of the chemicals recovered from standard stock solutions was conducted with a standard curve prepared according to the internal standard method.

Isolation and Identification of Commercial Wine Volatiles. Wine (200 mL) was added to a column packed with Porapak Q (5 mL). After the wine components were trapped on the resin, the column was eluted with 60 mL each of dichloromethane, ethyl ether, or pentane. The further experiments were conducted according to the method described above. Identification of the chromatographic peaks of the samples was made by comparing their mass spectra and gas chromatographic retention indexes to those of authentic compounds.

Quantitative Analysis of Wine Components. The quantitative analysis of the chemicals recovered from standard stock solutions was conducted with a standard curve prepared according to the internal standard method previously reported (Ettre, 1967). For the wine components, the ratios between peak area of a component and that of the internal standard were reported. A Hewlett-Packard (HP) Model 5890 gas chromatograph (GC) equipped with a 60 m \times 0.25 mm i.d. DB-WAX bonded phase, fused-silica capillary column (J & W Scientific, Folsom, CA) and a flame ionization detector (FID) was used. The linear velocity of helium carrier gas was 30 cm/s. The injector and the detector temperatures were 230 $^{\circ}$ C. The oven temperature was programmed from 50 to 200 $^{\circ}$ C at 3 $^{\circ}$ C/min and held for 40 min.

An HP 5890 series II gas chromatograph interfaced to an HP 5791 A mass-selective detector (GC/MS) was used for mass spectral identification of the GC components at MS ionization voltage of 70 eV. Column and oven conditions were as stated above.

RESULTS AND DISCUSSION

Porapak Q resin, which is an ethylvinylbenzene-divinylbenzene copolymer, has been widely used for collecting headspace gas. However, it has never been used for a wine analysis. Therefore, Porapak Q was chosen for this study. Porapak Q resin reportedly produced compounds such as alkylbenzene derivatives when it was heated to recover adsorbed chemicals (Lewis and Williams, 1980). However, there are very few reports on production of similar chemicals from Porapak Q resin when it was extracted with an organic solvent to recover adsorbed compounds. Sturaro et al. (1992), who reported the use of acetone, found *meta* and *para* isomers of ethylacetophenone and diacetylbenzene as the Porapak Q breakdown products.

When blank Porapak Q was eluted with dichloromethane, ethyl ether, or pentane, eight benzene derivatives were found in the eluates in the present study. They were 1,3-diethylbenzene, 1,4-diethylbenzene, 1,2-diethylbenzene, 1-ethyl-3-vinylbenzene, 1,3,5-triethylbenzene, naphthalene, 3,4-dimethylacetophenone, and 4-ethylacetophenone. These benzene derivatives did not interfere with the quantitative analysis of a standard stock solution.

When different amounts of the standard stock solution were used, a linear relation between recovery of esters or acids and amounts of the solution used was obtained (r^2 ranged from 0.965 to 0.989). The highest

Table 1. Results of Recovery Percent on Selected Wine Components from Porapak Q Using Dichloromethane as Eluting Solvent

standard wine compound	amount of compounds spiked (ppm)			
	0.5	2.5	5.0	10
2-methyl-1-propanol	5.0 \pm 0.1	4.3 \pm 0.1	3.7 \pm 0.1	4.0 \pm 0.1
3-methyl-1-butanol	5.1 \pm 0.1	48.3 \pm 2.0	46.3 \pm 2.1	47.1 \pm 2.5
2-phenylethanol	100.0 \pm 3.5	103.0 \pm 3.9	111.0 \pm 4.0	103.0 \pm 3.7
ethyl hexanoate	58.0 \pm 2.0	66.9 \pm 0.8	74.1 \pm 2.5	77.4 \pm 1.2
ethyl octanoate	55.0 \pm 1.4	66.8 \pm 2.2	73.0 \pm 2.3	79.1 \pm 2.2
diethyl butanedioate	102.0 \pm 2.6	101.9 \pm 2.7	110.2 \pm 3.2	102.9 \pm 2.7
hexanoic acid	82.0 \pm 2.3	91.1 \pm 2.0	99.1 \pm 2.2	97.3 \pm 2.1
octanoic acid	107.0 \pm 4.1	98.5 \pm 3.8	108.4 \pm 4.0	102.8 \pm 3.5

Table 2. Results of Recovery Percent on Selected Wine Components from Porapak Q Using Ethyl Ether as Elution Solvent

standard wine compound	amount of compounds spiked (ppm)			
	0.5	2.5	5.0	10
2-methyl-1-propanol	8.1 \pm 0.2	7.4 \pm 0.1	6.0 \pm 0.5	6.1 \pm 0.2
3-methyl-1-butanol	4.3 \pm 0.1	44.0 \pm 2.2	41.1 \pm 1.9	39.1 \pm 1.2
2-phenylethanol	66.5 \pm 2.3	76.1 \pm 3.7	81.9 \pm 2.5	73.5 \pm 2.4
ethyl hexanoate	42.7 \pm 1.9	46.4 \pm 3.1	45.9 \pm 3.3	53.2 \pm 3.2
ethyl octanoate	41.3 \pm 2.0	50.0 \pm 2.2	54.1 \pm 2.0	57.3 \pm 2.2
diethyl butanedioate	62.3 \pm 3.1	77.2 \pm 3.5	79.2 \pm 2.9	74.7 \pm 3.2
hexanoic acid	62.1 \pm 2.7	75.0 \pm 0.9	73.8 \pm 2.3	75.3 \pm 2.5
octanoic acid	72.9 \pm 2.4	54.8 \pm 2.8	64.7 \pm 2.2	62.2 \pm 2.7

Table 3. Results of Recovery Percent on Selected Wine Components from Porapak Q Using Pentane as Elution Solvent

standard wine compound	amount of compounds spiked (ppm)			
	0.5	2.5	5.0	10
2-methyl-1-propanol	0.0 \pm 0.0	0.0 \pm 0.0	0.1 \pm 0.0	0.1 \pm 0.0
3-methyl-1-butanol	13.0 \pm 1.0	16.1 \pm 1.0	16.7 \pm 1.0	13.6 \pm 1.0
2-phenylethanol	42.7 \pm 2.1	54.2 \pm 2.7	58.1 \pm 2.3	59.2 \pm 2.2
ethyl hexanoate	42.1 \pm 3.0	67.0 \pm 3.3	63.8 \pm 3.0	66.8 \pm 2.9
ethyl octanoate	38.0 \pm 2.8	48.8 \pm 2.1	67.2 \pm 2.5	76.2 \pm 2.3
diethyl butanedioate	65.0 \pm 2.5	79.5 \pm 2.2	85.1 \pm 2.3	90.0 \pm 3.4
hexanoic acid	2.0 \pm 0.0	15.3 \pm 1.2	29.7 \pm 1.8	43.5 \pm 2.0
octanoic acid	2.0 \pm 0.0	21.1 \pm 1.1	36.0 \pm 1.5	53.3 \pm 2.1

recovery was obtained from 200 mL of standard stock solution in the case of alcohols. Consequently, 200 mL of the standard stock solution was used for further experiments. In the case of different amounts of resin used, the recovery of acids did not show significant differences. The highest recovery of esters was achieved when 5 mL of resin was used. The recovery of alcohols increased with the amount of resin. However, the amount of resin breakdown products also increased when the amount of resin increased over 5 mL. Therefore, 5 mL of resin was used for further experiments. The optimum volume of dichloromethane was 60 mL, which was used for further experiments.

Tables 1–3 show the results of the recovery test on selected wine components from Porapak Q using dichloromethane, ethyl ether, and pentane as elution solvents. The values are mean \pm standard deviation ($n = 3$). The chemicals (alcohols, esters, acids) tested, were chosen from typical wine components reported previously (Baumes et al., 1986; Herraiz et al., 1991; Shimoda et al., 1993). This study was conducted to find the most appropriate solvent for recovering wine chemicals from Porapak Q. Alkyl alcohols tested revealed poor recovery with the solvents used. In particular, almost no 2-methyl-1-propanol was desorbed from Porapak Q with pentane (Table 3). Dichloromethane desorbed nearly 50% of 2-methyl-1-butanol from samples containing over 2.5 ppm (Table 1). On the other hand, dichloromethane recovered 2-phenylethanol by nearly 100% at all levels tested. All solvents showed better recovery when the concentrations of chemicals increased. The highly non-

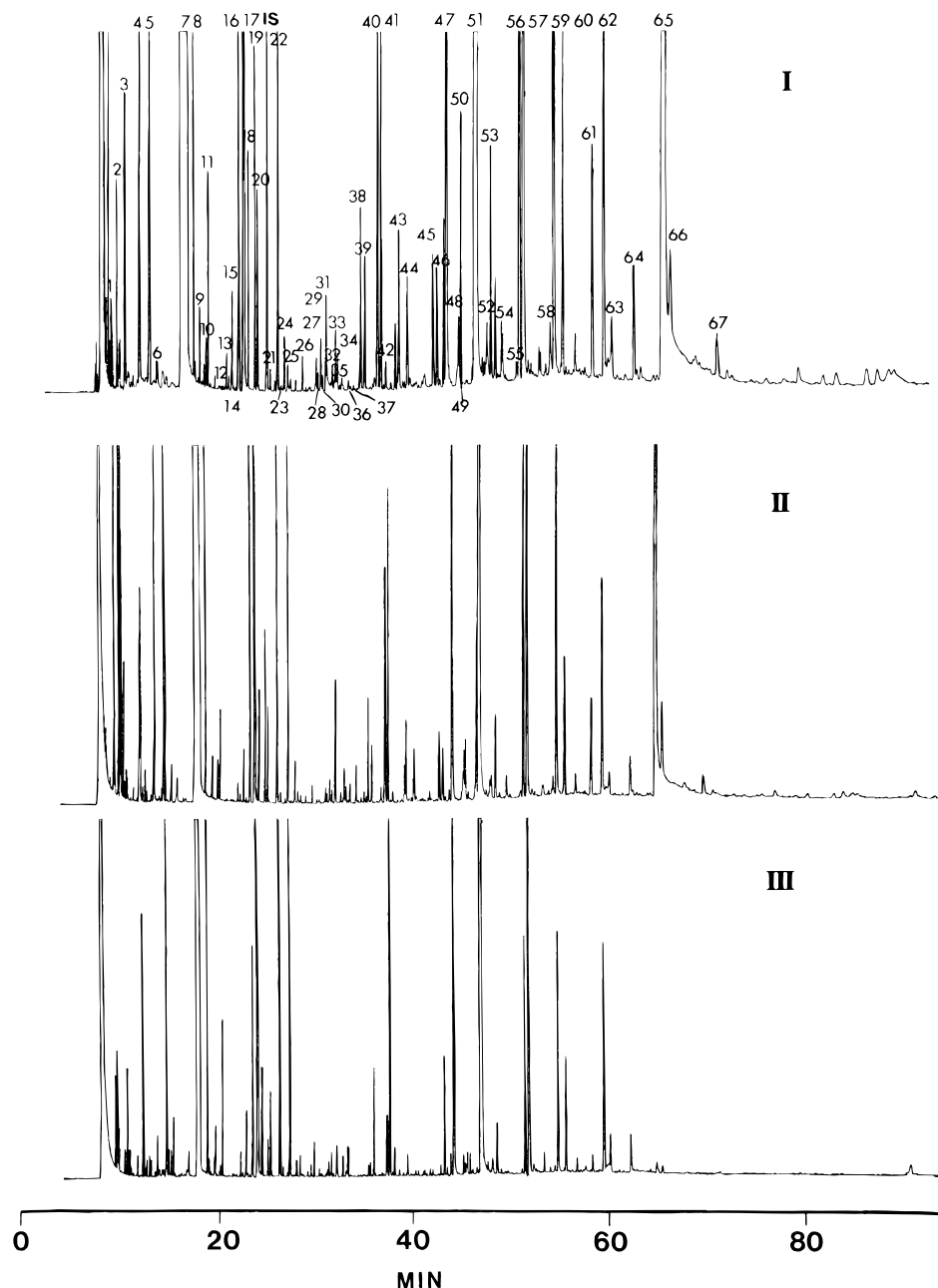


Figure 1. Gas chromatograms of volatile components from a wine sample obtained by extraction with dichloromethane (I), ethyl ether (II), and pentane (III).

polar solvent pentane exhibited unsatisfactory results. Even at a 10 ppm level of diethyl butanedioate, the highest recovery obtained by pentane was 87.1%, whereas dichloromethane gave almost 100% recovery for the same compound at the same level. Ethyl ether showed slightly better recoveries than pentane did in most cases, except in the cases of ethyl octanoate and diethyl butanedioate (Table 2). Generally, dichloromethane showed the highest recovery efficiency, followed by ethyl ether and pentane. Therefore, dichloromethane was used for additional experiments.

Figure 1 shows typical gas chromatograms of volatile components from a wine sample obtained by extraction with dichloromethane, ethyl ether, and pentane. Table 4 shows compounds identified in the above three extracts, along with their Kovats index (Kovats, 1965) and peak area ratio. It is obvious that dichloromethane extract contains the most compounds and generally in the highest concentrations. It is possible to remove most of the water and ethanol from an aqueous alcoholic

beverage to concentrate volatile organic chemicals using porous polymers such as Porapak Q, because water and ethanol have a short retention time (Jennings, 1978).

3-Methyl-1-butanol was the major component in all three wine samples (Table 4), which was consistent with a previous report (Baumes et al., 1986). The 2-phenyl-ethanol was the second largest component, followed by octanoic acid and monoethyl butanedioate in the extract obtained with dichloromethane or ethyl ether. However, only a trace amount of monoethyl butanedioate was recovered by the pentane extraction. Liquid-liquid extraction with pentane/dichloromethane (2:1) did not recover acid components well, including octanoic acid (Baumes et al., 1986). This may be due to the high water solubility of acids. On the other hand, Herraiz et al. (1991) satisfactorily trapped and recovered acid components of wine using a silica gel column.

Pentane seems to be superior to ethyl ether as a solvent to desorb alkyl esters from Porapak Q. For example, ethyl hexanoate, hexyl acetate, ethyl oc-

Table 4. Volatile Compounds Identified in Wine Samples Obtained by Extraction Using Different Organic Solvents

peak no. in Figure 1	compound	<i>I</i> ^a	peak area ratio of volatiles extracted with		
			dichloro-methane	ethyl ether	pentane
1	ethanol	959	0.05	0.14	0.08
2	ethyl butanoate	1009	1.13	<i>b</i>	<i>b</i>
3	1-propanol	1046	0.38	0.58	0.20
4	2-methyl-1-propanol ^c	1094	0.94	1.66	0.03
5	isoamyl acetate	1130	1.09	0.79	0.88
6	unknown	1153	0.05	0.07	0.05
7	3-methyl-1-butanol ^c	1236	117.92	111.72	24.53
8	ethyl hexanoate ^c	1248	0.84	0.61	0.71
9	1-pentanol	1262	0.07	0.05	0.04
10	ethyl 2-oxopropanoate	1276	0.04	0.05	0.01
11	hexyl acetate	1280	0.16	0.11	0.13
12	3-hydroxy-2-butanone	1287	<i>b</i>	<i>d</i>	<i>d</i>
13	4-methyl-1-pentanol	1324	0.03	0.02	0.02
14	ethyl heptanoate	1330	<i>b</i>	<i>b</i>	<i>b</i>
15	3-methyl-1-pentanol	1338	0.12	0.07	0.06
16	ethyl lactate	1355	1.18	0.92	0.21
17	1-hexanol	1366	6.03	5.24	3.77
18	(<i>E</i>)-3-hexen-1-ol	1374	0.20	0.14	0.09
19	3-ethoxy-1-propanol	1389	0.34	0.24	0.03
20	(<i>Z</i>)-3-hexen-1-ol	1393	0.18	0.13	0.07
21	3-methyl-2-butanol	1425	0.02	<i>b</i>	<i>b</i>
22	ethyl octanoate ^c	1443	1.01	0.60	0.74
23	furfural	1451	<i>b</i>	<i>b</i>	<i>e</i>
24	acetic acid	1459	0.05	0.06	<i>b</i>
25	1-heptanol	1467	0.03	<i>b</i>	<i>b</i>
26	2-ethyl-1-hexanol	1483	0.04	0.03	0.03
27	ethyl 3-hydroxybutanoate	1532	0.05	0.02	<i>b</i>
28	benzaldehyde	1535	0.02	<i>b</i>	<i>b</i>
29	2-methyl-4,5-dihydro-3(2H)-thiophenone	1542	0.07	0.03	0.02
30	propanoic acid	1547	<i>b</i>	0.02	<i>b</i>
31	2,3-butanediol (D,L)	1556	0.20	0.23	0.03
32	1-octanol	1569	0.05	<i>b</i>	<i>d</i>
33	2-methylpropanoic acid	1576	0.09	0.05	<i>d</i>
34	unknown	1581	0.05	0.02	0.03
35	2,3-butanediol (meso)	1591	0.03	0.03	<i>d</i>
36	1,2-propanediol	1611	<i>b</i>	0.07	<i>d</i>
37	butanoic acid	1628	<i>b</i>	<i>b</i>	<i>d</i>
38	ethyl decanoate	1637	0.23	0.18	<i>b</i>
39	γ -butyrolactone	1647	0.19	0.11	0.11
40	3-methylpentanoic acid	1679	0.81	0.56	0.09
41	diethyl butanedioate ^c	1688	0.92	0.57	0.70
42	unknown	1700	0.03	<i>b</i>	0.03
43	3-(methylthio)-1-propanol	1734	0.22	0.15	0.03
44	unknown	1754	0.19	0.10	<i>b</i>
45	unknown	1819	0.25	0.15	<i>b</i>
46	2-phenylethyl acetate	1828	0.18	0.10	<i>b</i>
47	hexanoic acid ^c	1852	7.04	4.12	1.89
48	benzyl alcohol	1879	0.15	0.07	0.03
49	unknown	1881	0.06	0.10	<i>b</i>
50	unknown	1886	0.43	0.13	<i>b</i>
51	2-phenylethanol ^c	1930	47.50	27.46	18.52
52	unknown	1960	0.13	0.10	0.03
53	(<i>E</i>)-2-hexenoic acid	1971	0.29	0.16	0.60
54	unknown	1996	0.07	0.04	<i>b</i>
55	γ -valerolactone	2049	0.03	<i>d</i>	<i>b</i>
56	diethyl hydroxy-butanedioate	2060	2.37	1.48	0.27
57	octanoic acid ^c	2072	8.71	4.93	2.78
58	unknown	2148	0.12	0.06	<i>b</i>
59	2,4-hexadienoic acid	2159	2.99	1.76	0.33
60	unknown	2182	0.57	0.32	0.14
61	3,5-diethylphenol	2256	0.46	0.28	0.03
62	decanoic acid ^c	2283	1.22	0.65	0.38
63	unknown	2300	0.13	0.06	0.05
64	unknown	2340	0.23	0.12	0.07
65	monoethyl butanedioate	2395	8.69	6.05	<i>b</i>
66	unknown	2407	0.22	0.25	<i>d</i>
67	unknown	2472	0.13	0.08	<i>d</i>

^a Kovats index on DB-WAX. ^b Peak area ratio less than 0.01.

^c Compound used for the standard stock solution. ^d Not detected.

tanoate, and diethyl butanedioate were recovered more efficiently by pentane than by ethyl ether. Therefore, it may be possible to roughly estimate the type of

compound for unknown extracts by comparing the results of the three solvent extractions demonstrated in this study. Unknowns at peak nos. 34 and 42 are likely alkyl esters.

As described above, there have been many reports on methods for isolating volatile aroma components from wine. These methods exhibited satisfactory results on isolation of volatile compounds. However, they also have some drawbacks. The solid phase extraction method described in this study demonstrates that this technique is simple and rapid for recovering volatile compounds from aqueous alcoholic samples.

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