A Nationwide Survey on Organic Solvent Components in Various Solvent Products:

Part 1. Homogeneous Products Such as Thinners, Degreasers and Reagents*

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Abstract: Organic solvent products used in the various factories were collected nationwide in Japan, and analyzed by gas chromatography. Of the 1179 samples collected, 522 samples were homogeneous (including 321 thinners, 145 degreasers and 56 reagents and others), and were analyzed by means of direct injection into a FID-gas chromatograph with two FSWCOT (OV 101 and PEG 6000) capillary columns. In thinners (n=248), the average number of solvent components per sample was 4.14 when the thinners containing gasoline were excluded. Toluene was the most popular component in thinners (present in 56%) followed by ethyl acetate, methanol, xylenes, ethylbenzene, isobutyl alcohol, ethyleneglycol monoethylether acetate, butyl acetate and methyl isobutyl ketone. Gasoline was also detected in 23%. In degreasers (n=123), the average number of solvent components

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per sample was 1.64 when the degreasers containing gasoline were not taken into account. Trichloroethylene was present in 21% of degreasers followed by tetrachloroethylene, 1, 1, 1-trichloroethane, toluene, acetone, xylenes and ethylbenzene. Gasoline was found in 14%. A small amount of 1, 4-dioxane was detected as an additive in every 1, 1, 1-trichloroethane products. With regard to reagents and others (n=53), the average number of components per sample was 1.40 if samples containing gasoline were excluded, and most (73%) of reagents and others (n=41) consisted of a single component. The potent toxic solvents such as benzene, chloroform, carbon tetrachloride, 1, 2-dichloroethane, 1, 1, 2-trichloroethane, 1, 1, 2, 2-tetrachloroethane and carbon disulfide were only rarely found in the present homogeneous products.

Key words: Degreasers—GC analyses—Organic solvent—Reagents—Thinners

INTRODUCTION

More than 400 kinds of and large quantities of organic solvents are used in industries¹⁾, and they are often mixed for use. As the current regulation in Japan requests only 29 kinds of organic solvent constituents to be labeled on the containers and other constituents of mixed organic solvents need not to be described, many workers are exposed to the mixture of organic solvents, the constituents of which are not known. For the protection of the health of the workers, however, it is important to know the constituents of the solvents. The existing reports indicate some cases of poisoning among workers²⁻⁶⁾ and thinner/glue sniffers⁷⁻⁹⁾ are due to the mixture of organic solvents. There are some reports on the constituents of the organic solvent products collected at certain local areas or used in the factories investigated¹⁰⁻¹³⁾. Such samples were collected from only a few factories or limited areas. In the present survey, organic solvent products were collected from industries in various districts of Japan. The products collected were divided into the two groups; one consisted of homogeneous organic solvent products such as thinners, degreasers and reagents, and the other of heterogeneous products like paints, inks and adhesives. For gas chromatographic analyses the homogeneous products can be directly injected into the apparatus, but the heterogeneous ones need other technique. The studies on the homogeneous products will be presented in the present paper. The results with the heterogeneous ones are to be reported in a twin paper¹⁴⁾.

MATERIALS AND METHODS

The organic solvent products, 1179 in total, were collected in 1980 from factories of various sizes and kinds in seven districts of Japan with sampling strategy to cover an entire country. Of 1179 samples, 522 were homogeneous solvent products such as thinners, degreasers and reagents (Table 1), which were the subjects of

Table 1. Collection of homogeneous organic solvent preparations by various districts in Japan.

Preparations	I	II	III	IV	V	VI	VII	Total
Thinners	27	24	53	99	35	67	16	321
Degreasers	7	20	54	35	9	16	4	145
Miscellaneous	1	19	32	2	0	2	0	56
Total	35	63	139	136	44	85	20	522

I: Hokkaido II: Tohoku III: Kanto IV: Chubu V: Kansai
 VI: Chugoku-Shikoku VII: Kyushu

the present study. After the specific gravity of each sample was measured with a picnometer (about 5 ml) at room temperature (15–22°C), two μl of each sample was automatically injected with a Shimadzu auto-injector AOC-7 into a Shimadzu GC-RIA gas chromatograph (GC) equipped with FID's and two ϕ 0.25 mm $\times$ 50 FS-WCOT capillary columns [Silicone OV 101 and polyethyleneglycol (PEG) 6000]. The GC conditions employed were as follows; supply of H_2 and air to FID's at 0.5 and 0.6 kg/cm² respectively, N_2 flow to the OV 101 column at 47 ml/min and to the PEG 6000 column at 40 ml/min respectively at 60°C. The temperature of the oven was kept at 60°C for the first 10 minutes, increased at the rate of 5°C/min, then kept at 120°C for the last 18 minutes, so that the duration of GC analysis was 40 minutes per sample. The attenuation and the range of the GC were set at 8 and 10³, respectively. The constituents of the samples were identified by comparison of retention time on the two columns with those of authentic chemicals. For identification, 132 authentic solvents were selected, of which TLVs were set by American Conference of Governmental Industrial Hygienists¹⁵⁾. Two μl of each authentic sample was injected into the GC and the retention time and the peak area were measured. The relative retention time and peak area of the authentic solvents to those of toluene were calculated. These data and specific gravity of the 132 authentic solvents were summarized in a table to compare with those of the samples. To eliminate possible day by day variation, toluene was analyzed as a standard every day when the samples were analyzed. The content of the identified constituent in a sample was calculated as follows:

$$\text{Content (w/w\%)} = \frac{\text{identified peak area}}{\text{authentic peak area}} \times \frac{\text{authentic specific gravity}}{\text{sample's specific gravity}}$$

The content of the constituent was usually calculated from the results with OV 101 column, but the constituent which could not be separated well from other constituents on OV 101 column was calculated from the results with PEG 6000 column. If the sum up of the identified constituents in a given sample was less than 95%, the analysis was repeated. When the experimental molar responses of the 44 authentic solvents were compared with the theoretical ones, it was found that the experimental results were very close to theoretical values¹⁴⁾. The reliability of

the pretest GC analysis was further confirmed by the fact that when five samples were analyzed by gas chromatography-mass spectrometry (Shimadzu GC-MASS 6020 CPU 1123), the results agreed with those by the present GC analysis.

RESULTS

The frequency of the number of solvent components per sample are shown in Fig. 1. When the products containing gasoline were excluded, the average number of solvent components per product was 4.14 ($n=248$) for thinners, 1.71 ($n=125$) for degreasers and 1.40 ($n=53$) for other products. The frequency of each organic solvent occurred in total products, and the average and the 10–90% range are summarized in Table 2. Of the 522 homogeneous solvent products, the sum up of the identified solvent components were more than 95% in 471 (90%) samples. Of the remaining 51 products, seven samples revealed two relatively large peaks in common which could not be identified under the present conditions, so that the sum up in the products was $85.2 \pm 4.4\%$ (mean $\pm$ SD). Other 24 products gave many small peaks (11 ± 4) which were difficult to be identified, and the sum was $86.3 \pm 9.2\%$ when these peaks were excluded. In the other 16 samples, the sum was $83.4 \pm 17.2\%$, and it was considered that these samples contained the chemicals which could not be detected under the present conditions. No peaks were found in other 4 samples. Two of them were indicated on the costainers as ethyleneglycol, the third one as propyleneglycol, and the last one as diethyleneglycol monoethyl ether. With regard to thinners, toluene was the most

Fig. 1. Frequency of numbers of solvents per sample.
 Solid bars: Samples containing gasoline are excluded.
 Open bars: Gasoline is counted as one solvent.

Table 2. Organic solvent components in various homogeneous organic solvent products

Organic solvent	Thinner		Degreaser		Miscellaneous	
	N (%)	M (10-90% range)	N (%)	M (10-90% range)	N (%)	M (10-90% range)
Aromatics						
Benzene	—(—)	—	2(1)	98.5	—(—)	—
Toluene	180(56)	45.1(4-68)	12(8)	48.3(3-99)	10(18)	59.7(6-99)
Xylenes	105(33)	17.9(1-41)	10(7)	45.2(11-77)	2(4)	41.0
Ethylbenzene	102(32)	16.3(1-38)	10(7)	24.3(9-47)	2(4)	11.0
Styrene	2(1)	99.0	—(—)	—	—(—)	—
Chlorinated hydrocarbons						
Dichloromethane	1(0)	5.0	2(1)	98.5	1(2)	99.0
Chloroform	—(—)	—	—(—)	—	3(5)	98.3
Carbon tetrachloride	—(—)	—	—(—)	—	1(2)	97.0
1,2-Dichloroethane	—(—)	—	1(1)	3.0	1(2)	98.0
1,1,1-Trichloroethane	3(1)	66.3	20(14)	95.8(96-99)	1(2)	95.0
1,1,2-Trichloroethane	—(—)	—	—(—)	—	1(2)	99.0
1,1,2,2-Tetrachloroethane	—(—)	—	1(1)	36.0	—(—)	—
1,1,2-Trichloro-1,2,2-trifluoroethane	—(—)	—	8(6)	95.9	—(—)	—
1,2-Dichloropropane	—(—)	—	1(1)	63.0	—(—)	—
Trichloroethylene	6(2)	52.2	31(21)	85.4(39-99)	—(—)	—
Tetrachloroethylene	2(1)	19.5	18(12)	67.3(3-99)	—(—)	—
Chlorobenzene	1(0)	98.0	—(—)	—	—(—)	—
Alcohols						
Methanol	98(31)	21.8(3-42)	20(14)	44.7(5-99)	8(14)	61.6
Ethanol	8(2)	14.4	8(6)	40.1	4(7)	72.5
Isopropyl alcohol	45(14)	25.0(2-96)	6(4)	42.5	4(7)	86.0
1-Butyl alcohol	11(3)	23.4(3-96)	—(—)	—	2(4)	98.5
2-Butyl alcohol	—(—)	—	—	—	1(2)	99.0
Isobutyl alcohol	70(22)	5.9(3-10)	3(2)	3.0	2(4)	8.0
Diacetone alcohol	8(2)	20.1	—(—)	—	—(—)	—
Esters						
Methyl acetate	—(—)	—	1(1)	20.0	—(—)	—
Ethyl acetate	122(38)	18.5(4-39)	6(4)	45.0	3(5)	49.0
Propyl acetate	5(2)	5.4	—(—)	—	—(—)	—
Isopropyl acetate	13(4)	8.0(1-14)	—(—)	—	—(—)	—
Butyl acetate	58(18)	16.8(2-25)	—(—)	—	—(—)	—
2-Butyl acetate	4(1)	3.8	—(—)	—	—(—)	—
Isobutyl acetate	36(11)	9.7(2-16)	2(1)	8.0	1(2)	20.0
Amyl acetate	1(0)	37.0	—(—)	—	1(2)	99.0
Ketones						
Acetone	28(9)	36.1(3-96)	12(8)	86.7(72-99)	4(7)	53.8
Methyl ethyl ketone	36(11)	35.5(1-97)	7(5)	44.4	4(7)	50.5
Methyl butyl ketone	—(—)	—	1(1)	99.0	—(—)	—
Methyl isobutyl ketone	50(16)	11.7(1-20)	2(1)	7.5	—(—)	—

Table 1. Continued

Organic solvent	Thinner		Degreaser		Miscellaneous	
	N (%)	M (10-90% range)	N (%)	M (10-90% range)	N (%)	M (10-90% range)
Ethers and glycol derivatives						
Tetrahydrofuran	2 (1)	6.0	—(—)	—	3 (5)	95.0
Ethyleneglycol monomethyl ether	3 (1)	50.3	—(—)	—	2 (4)	99.0
Ethyleneglycol monoethyl ether	10 (3)	12.8(5-26)	—(—)	—	2 (4)	99.0
Ethyleneglycol monoethyl ether acetate	67(21)	14.9(3-27)	2 (1)	35.5	1 (2)	21.0
Ethyleneglycol monobutyl ether	45(14)	10.2(2-13)	2 (1)	6.0	3 (5)	50.3
Diethyleneglycol monoethyl ether	—(—)	—	1 (1)	99.0	—(—)	—
1,4-Dioxane	—(—)	—	18(10)	2.7 (2- 3)	2 (4)	51.0
Alicyclic compounds						
Cyclohexanone	1 (0)	22.0	1 (1)	99.0	—(—)	—
Cyclohexane	1 (0)	21.0	—(—)	—	—(—)	—
Miscellaneous compounds						
Carbon disulfide	1 (0)	95.0	—(—)	—	—(—)	—
n-Hexane	8 (2)	4.8	2 (1)	95.5	—(—)	—
n-Heptane	—(—)	—	2 (1)	49.0	1 (2)	99.0
Dimethyl formamide	—(—)	—	1 (1)	99.0	3 (5)	98.7
Dimethyl sulfoxide	—(—)	—	—(—)	—	1 (2)	97.0
Acetonitrile	—(—)	—	1 (1)	99.0	—(—)	—
Gasoline	73(23)	77.1(29-99)	20(14)	92.8(46-99)	3 (5)	72.3

N: Numbers of the samples in which the given solvent were detected

—: Not detected

(%): [Numbers observed/Numbers analyzed] × 100

M: Arithmetic mean of contents.

10-90% range: The range in which 80% of the contents observed (i.e., excluding 10% each of the minimum and maximum) were embraced.

popular component and present in 56% of thinners, followed by ethyl acetate in 38%, xylenes in 33%, ethylbenzene in 32%, methanol in 31%, isobutyl alcohol in 22%, ethyleneglycol monoethylether acetate in 21%, butyl acetate in 18%, and methyl isobutyl ketone in 16%. The average content of toluene per thinner was 45.1% (n=180) and thus toluene was a main component in thinners. This was followed by acetone (36.1%, n=28), methyl ethyl ketone (35.5%, n=36), methanol (21.8%, n=98), xylenes (17.9%, n=105), and ethylbenzene (16.3%, n=102). It should be worth to note that the mixture of xylenes and ethylbenzene are usually used as technical xylene in industries. Isobutyl alcohol was present in the 70 thinners (22%), but its average content was only 5.9%. The 73 thinners (23%) contained gasoline; 38 of them (12%) were gasoline themselves, but the

remaining 35 (11%) were mixtures of gasoline and other solvents. The average content of gasoline per thinner was 77.1% ($n=73$). With regard to degreasers, trichloroethylene was the most popular component being present in 31 degreasers (21%), followed by 1,1,1-trichloroethane and methanol in 20 (14%), tetrachloroethylene in 18 (12%), toluene in 12 (8%), acetone in 12 (8%), and xylenes and ethylbenzene in 10 (7%). The halogenated hydrocarbons and acetone were usually single constituent of the degreaser even if a small quantity of additives might be present, while methanol, toluene and xylenes were usually in mixtures. 1,4-Dioxane was detected in 18 (10%) of degreasers and the average content was 2.7%. It was usually present as an additive in 1,1,1-trichloroethane. Twenty degreasers (14%) contained gasoline. With regard to reagents and others, three fourth of them consisted of a single component.

DISCUSSION

In the present study, the homogeneous organic solvent products were analyzed by GC in order to elucidate the components of the organic solvents currently used in industries in Japan. By analyses of the 522 homogeneous products, 5 aromatic hydrocarbons, 12 halogenated hydrocarbons, 7 alcohols, 8 esters, 4 ketones, 7 ethers including glycol derivatives, 2 alicyclic hydrocarbons and 6 other solvents were identified. Total number of the solvents identified was 51. Besides, gasoline was found in 18% of the 522 products. Among aromatic hydrocarbons, toluene was the most popular solvent being used as a main component both in thinners and in degreasers. It was followed by xylenes. Benzene was not found the homogeneous products except for two particular products of degreaser. Benzene is presumably not used in thinner or degreaser because its use is legally restricted and its insidious toxicity is well known in Japan since an outbreak of fatal intoxication cases in Osaka in 1958⁽¹⁶⁾. Among halogenated hydrocarbons, trichloroethylene and tetrachloroethylene, possible neurotoxins^(17,18) and carcinogens^(19,20), were popular solvents in degreasers. Next popular were 1,1,1-trichloroethane and 1,1,2-trichloro-1,2,2-trifluoroethane. Among esters, ethyl acetate, butyl acetate and isobutyl acetate were popular in thinners. Among ketones, acetone, methyl ethyl ketone and methyl isobutyl ketone were popular both in thinners and in degreasers. Methyl butyl ketone, a well known toxin of the peripheral nerves^(21,22), was found only in one degreaser product. Among esters including glycol derivatives, ethyleneglycol monoethylether acetate was the most popular solvent, followed by ethyleneglycol monobutylether and ethyleneglycol monoethylether. The average contents per sample were small and their volatilities are low, but they will be readily absorbed through the intact skin⁽²³⁾ and may be toxic to testes⁽²⁴⁾. Among aliphatic hydrocarbons, n-hexane, known to be neurotoxic^(2,25), was found only in 2% of thinners and degreasers. Gasoline was found in 18% of total samples, popular both in thinners and degreasers. While the gasoline components were not

analyzed in detail in the present study, the presence of benzene, a human leukemogen²⁶⁾, was positively excluded after careful examination. The unleaded fuel gasoline, however, should be used in industries with cautions, because aromatic hydrocarbons including benzene are often present at high quantities^{27,28)}. Other highly toxic solvents such as chloroform, carbon tetrachloride, 1,2-dichloroethane, 1,1,2-trichloroethane, 1,1,2,2-tetrachloroethane, and carbon disulfide were found only rarely in the present homogeneous solvent products. It is considered important from the viewpoint of occupational health that every components of organic solvents used in industry should be indicated and the popular solvents should be kept under careful control to prevent chronic poisoning.

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