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RAPID DETERMINATION OF SOLVENT COMPONENTS IN PAINT BY COMPUTER-AIDED FUSED-SILICA CAPILLARY GAS CHROMATOGRAPHY

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SUMMARY

Fused-silica capillary column gas chromatography was applied to the analysis of organic paint solvents with substantial savings of time and labour. More than 60 components in various types of paint solvents could be successfully determined on a PEG 20M stationary phase. The relative standard deviation of the amounts of the compounds determined was less than 3.0% and the analysis time per sample was 40-45 min.

INTRODUCTION

Organic paint solvents, which consist of up to 70 components with a variety of properties, often enter the environment in painting processes of industrial products and outdoor structures. Certain aromatic components of paint solvents are toxic or photochemically reactive in the ambient air and the use or emission of such hazardous compounds at painting sites should be minimized. Gas chromatography (GC) is the most commonly applied method for the analysis of these complex mixtures. Conventional packed column GC and glass capillary column GC are, however, not useful for separating all components of different types of paint solvents on a single stationary phase. Therefore, to cover all components by these methods, multiple stationary phases should be used under different operating conditions according to the properties of the components. This results in time-consuming and impractical procedures for the routine analysis of large numbers of paint samples.

Recently developed fused-silica capillary columns (FSCCs) for GC have advantages over conventional columns in terms of flexibility, inertness and ultra-high resolution¹⁻⁴. FSCCs are easier to instal in a gas chromatograph with less chance of

breakage than glass columns. The outstanding features of FSCCs with respect to inertness and resolution allow analyses on a single stationary phase of complex mixtures such as environmental pollutants⁵⁻¹⁰, essential oils^{3,7,11,12}, drugs^{8,13,14} and waxes^{8,15,16}. On the other hand, recent computer-aided gas chromatographs may provide new capabilities for the precise control of working conditions and for the rapid processing of large numbers of analytical data.

So far, few reports have been published concerning the determination of solvent components in paints by FSCC GC. In this paper, a rapid routine method for the determination of all solvent components in paints, except for triethylamine, by computer-aided GC with a PEG 20M FSCC is presented.

EXPERIMENTAL

Reagents and materials

The reagents used as standards were of special grade from Wako (Osaka, Japan) and Tokyo Kasei Kogyo (Tokyo, Japan). A 50 m × 0.2 mm I.D. PEG 20M FSCC from Hewlett-Packard (Avondale, PA, U.S.A.) was used.

Apparatus

A Hewlett-Packard 5880A gas chromatograph with an inlet system as shown in Fig. 1, a flame-ionization detector (FID) and a Basic programmed microprocessor was employed. The working conditions were as follows. The injection temperature was 180°C and the column temperature was (A) isothermal at 60°C for 4 min, then programmed from 60 to 160°C at 4°C/min and isothermal at 160°C for 6 min and (B) isothermal at 60°C for 8 min, then programmed from 60 to 160°C at 4°C/min and isothermal 160°C for 6 min; after the analysis, the column was heated at 180°C for 2 min, cooled to 40°C, then returned to the initial temperature. The FID temperature was 200°C. The carrier gas was nitrogen at a base flow-rate at 80 ml/min and a column flow-rate of 0.91 ml/min (splitting ratio 1:88). The septum purge gas was nitrogen at 2 ml/min and the make-up gas was nitrogen at 30 ml/min. The baseline change due to the programmed temperature was compensated for by the microprocessor.

A Hewlett-Packard 5995A gas chromatograph-mass spectrometer with the FSCC was used to identify unknown peaks.

Analytical procedure

A 2-g amount of a paint (or 1 g of a thinner solvent) was placed in a 20-ml

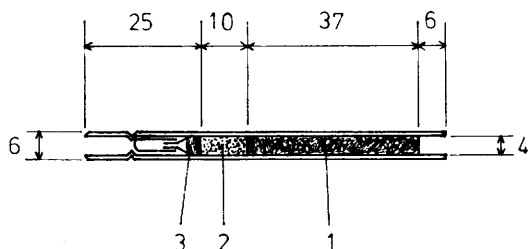


Fig. 1. Glass tube injection port. 1 = Quartz-wool; 2 = 2% OV-101 on 60-80-mesh Chromosorb W AW DMCS; 3 = quartz-wool. Dimensions in millimetres.

volumetric glass tube with a PTFE membrane-sealed cap, and 80 mg of *n*-propylcyclohexane (IS-1) and 90 mg of phenylcyclohexane (IS-2) were added as internal standards. The mixture was adjusted to 20 ml with 2-propanone and 1–2 μ l of the diluted sample was analysed by GC.

Identification and quantitation of solvent components

The calculation and the filing of the data were carried out by using the micro-processor. The relative retentions (min/min) and response factors (g/g) of individual components relative to IS-1 or IS-2 were obtained prior to the analysis of paint samples by analysing standard samples consisting of homogeneous family members. Solvent components in paints were identified by agreement of their relative retentions with those of the standards. Components that were difficult to identify only by GC, if present, were identified by GC–mass spectrometry (MS). The identified components were quantified by using the response factors and the peak areas relative to IS-1 or IS-2. For the quantitation, IS-1 was used for *o*-xylene and earlier peaks than *o*-xylene, and IS-2 for later peaks than *o*-xylene. If a sample contained compounds non-separable from IS-1, IS-2 was used for all the peaks.

RESULTS AND DISCUSSION

When paint solvents were suddenly vaporized with a pigment in the injection port in GC analysis, sols of the solvents, and/or the pigment seemed to be produced, thus clogging the capillary column or changing abruptly the splitting ratio of the carrier gas flow even during one analytical run. Use of a glass tube inlet partially packed with 2% OV-101 on 60–80-mesh Chromosorb W AW DMCS, shown in Fig. 1, was useful in filtering such sols and fine particulates in samples. The quartz-wool at the head was replaced after 50–60 injections of paint samples to remove the deposit of pigments. If the column head was obstructed, a few centimetres of the head were cut off. The resulting splitting ratio of the carrier gas was constant over a few months under the given conditions.

Table I gives the relative retentions and response factors defined as the response ratio of the component to the internal standard in this GC system, and Fig. 2 shows typical chromatograms of these standard compounds. Excellent resolution for these compounds with different polarities were achieved under the conditions used. All solvent components in various types of paints could be effectively determined on the single stationary phase. Triethylamine in water paints, however, was exceptionally difficult to determine accurately with the FSCC system because of its great adsorption on the injection port and the column tubing.

Identification of solvent components in a paint could usually be completed in 4–5 min after all of the components had been eluted, followed by immediate quantitation of the identified compounds. For petroleum solvents that contained aliphatic hydrocarbons as major constituents, only IS-2 was used because of the appearance of complex peaks around the IS-1 peak. Usually, the determination was effectively performed under temperature conditions (A). If complex peaks that were difficult to identify were eluted, analysis was carried out under temperature conditions (B) also. GC–MS was occasionally used to identify unexpected compounds in the solvent. In the GC analysis, the within-day standard deviation of the retention times of the

TABLE I

RELATIVE RETENTIONS AND RESPONSE FACTORS OF COMMONLY USED SOLVENT COMPONENTS ON A PEG 20M FSCC

Relative retention is the ratio of the retention time of a compound to that of the internal standard, IS-1 (*n*-propylcyclohexane) or IS-2 (phenylcyclohexane). Times in the parentheses indicate absolute retention times of the internal standards. Response factor is the response ratio of a compound to the internal standard (valid only for the injection port, the splitting device and the FID used here).

No.	Component	Relative retention (min/min)				Response factor (g/g)	
		Conditions A		Conditions B		IS-1	IS-2
		IS-1 (6.05 min)	IS-2 (25.10 min)	IS-1 (6.12 min)	IS-2 (27.76 min)		
1	Methanol	0.853	0.206	0.845	0.180	0.345	0.256
2	2-Propanol	0.844	0.231	0.877	0.187	0.498	0.369
3	2-Methyl-1-propanol	1.21	0.292	1.25	0.266	0.656	0.487
4	2-Pentanol	1.31	0.315	1.37	0.292	0.625	0.464
5	1-Butanol	1.40	0.366	1.49	0.317	0.668	0.496
6	Diacetone alcohol	2.45	0.589	2.82	0.601	0.634	0.471
7	3-Methoxybutanol	2.49	0.601	2.89	0.615	0.599	0.444
8	2-Ethylhexanol	3.21	0.775	3.73	0.794	1.10	0.818
9	Ethyl acetate	0.855	0.206	0.847	0.181	0.370	0.275
10	Isobutyl acetate	1.04	0.251	1.05	0.233	0.605	0.449
11	<i>n</i> -Butyl acetate	1.19	0.287	1.22	0.259	0.591	0.439
12	Methyl Cellosolve* acetate	1.91	0.460	2.17	0.462	0.393	0.292
13	Cellosolve* acetate	2.13	0.514	2.46	0.524	0.491	0.365
14	3-Methoxybutyl acetate	2.40	0.578	2.78	0.591	0.591	0.439
15	<i>n</i> -Butyl Cellosolve* acetate	3.06	0.739	3.56	0.758	0.706	0.524
16	Carbitol** acetate	4.23	1.02	4.80	1.02	0.466	0.346
17	<i>n</i> -Butyl Carbitol** acetate	5.07	1.22	5.65	1.20	0.681	0.506
18	2-Propanone	0.803	0.194	0.794	0.169	0.532	0.395
19	2-Butanone	0.868	0.209	0.861	0.184	0.582	0.433
20	4-Methyl-2-pentanone	1.03	0.248	1.03	0.220	0.670	0.497
21	Cyclohexanone	2.07	0.500	2.35	0.500	0.849	0.630
22	N,N-Dimethylformamide	2.22	0.535	2.54	0.541	0.359	0.267
23	3,3,5-Trimethyl-2-cyclohexene-1-one	3.70	0.892	4.22	0.900	1.12	0.829
24	Methyl Cellosolve*	1.51	0.365	1.64	0.349	0.328	0.244
25	Cellosolve*	1.72	0.414	1.92	0.408	0.449	0.334
26	Isopropyl Cellosolve*	1.77	0.426	1.98	0.421	0.539	0.401
27	<i>n</i> -Propyl Cellosolve*	2.10	0.514	2.44	0.520	0.557	0.413
28	<i>n</i> -Butyl Cellosolve*	2.69	0.649	3.13	0.666	0.544	0.404
29	Methyl Carbitol**	3.71	0.894	4.22	0.897	0.457	0.339
30	Carbitol**	3.86	0.930	4.41	0.939	0.594	0.441
31	<i>n</i> -Butyl Carbitol**	4.76	1.15	5.33	1.13	0.756	0.561
32	Isoamyl Carbitol**	5.14	1.24	—	—	0.783	0.581
33	<i>n</i> -Amyl Carbitol**	5.23	1.26	—	—	0.795	0.590
34	<i>n</i> -Hexane	0.740	0.179	0.732	0.156	0.713	0.530
35	<i>n</i> -Heptane	0.760	0.183	0.753	0.160	0.847	0.629
36	<i>n</i> -Octane	0.803	0.194	0.796	0.169	0.924	0.686
37	<i>n</i> -Nonane	0.899	0.214	0.882	0.188	0.967	0.740

TABLE I (continued)

No.	Component	Relative retention (min/min)				Response factor (g/g)	
		Conditions A		Conditions B		IS-1	IS-2
		IS-1 (6.05 min)	IS-2 (25.10 min)	IS-1 (6.12 min)	IS-2 (27.76 min)		
38	<i>n</i> -Decane	1.06	0.255	1.07	0.227	1.07	0.796
39	<i>n</i> -Undecane	1.36	0.329	1.45	0.309	1.19	0.881
40	<i>n</i> -Dodecane	1.81	0.436	2.04	0.435	1.24	0.922
41	<i>n</i> -Tridecane	2.36	0.569	2.73	0.582	1.31	0.975
42	Benzene	0.924	0.223	0.918	0.196	0.910	0.676
43	Toluene	1.11	0.267	1.12	0.239	0.998	0.741
44	Ethylbenzene	1.37	0.331	1.45	0.309	1.00	0.746
45	<i>p</i> -Xylene	1.40	0.337	1.48	0.316	1.04	0.771
46	<i>m</i> -Xylene	1.42	0.343	1.51	0.322	1.07	0.792
47	Isopropylbenzene	1.56	0.375	1.69	0.360	1.03	0.766
48	<i>o</i> -Xylene	1.59	0.384	1.74	0.369	1.05	0.782
49	<i>n</i> -Propylbenzene	1.71	0.413	1.89	0.403	1.17	0.886
50	<i>p</i> -Ethyltoluene	1.78	0.429	1.98	0.421	1.19	0.885
51	<i>m</i> -Ethyltoluene	1.79	0.431	1.99	0.424	1.19	0.885
52	1,3,5-Trimethylbenzene	1.88	0.452	2.11	0.448	1.21	0.899
53	<i>o</i> -Ethyltoluene	1.96	0.472	2.21	0.471	1.24	0.917
54	1,2,4-Trimethylbenzene	2.06	0.497	2.34	0.499	1.17	0.869
55	1,2,3-Trimethylbenzene	2.21	0.532	2.53	0.538	1.21	0.895
56	4-(<i>n</i> -Propyl)toluene	2.35	0.565	2.69	0.573	1.16	0.861
57	2-(<i>n</i> -Propyl)toluene	2.36	0.569	2.72	0.578	1.18	0.875
58	4-Ethyl- <i>m</i> -xylene	2.49	0.600	2.87	0.611	1.14	0.848
59	5-Isopropyl- <i>m</i> -xylene	2.52	0.608	2.92	0.622	1.19	0.866
60	1,2,4,5-Tetramethylbenzene	2.84	0.684	3.28	0.698	1.28	0.946
61	1,2,3,5-Tetramethylbenzene	2.90	0.698	3.35	0.712	1.22	0.907
62	1,2,3,4-Tetramethylbenzene	3.18	0.766	3.66	0.780	1.29	0.955
63	Tetrahydronaphthalene	3.38	0.815	3.88	0.826	1.17	0.870
64	Pentamethylbenzene	4.02	0.967	4.56	0.971	1.30	0.967
65	Naphthalene	4.47	1.08	5.04	1.07	1.32	0.973

* Cellosolve: a group of monoalkyl ethers of ethylene glycol and their derivatives.

** Carbitol: a group of monoalkyl ethers of diethylene glycol and their derivatives.

compounds was less than 0.1%, and the standard deviation of the determined values was less than 3.0% except for the amine. Once the standard data were on file, the analysis time per sample was 40–45 min.

The retention times of the compounds changed with long-term use of the columns. In particular, hydrocarbons tended to show earlier elution with time than oxo compounds. To keep the identification valid, the retention data should be refilled after 100–120 h of use by using standard samples. The response factors in the GC system changed considerably with changes in the injection temperature, the carrier gas flow-rate and splitting ratio. The response factors, which remained almost constant for long-term analysis unless the conditions were changed, were also refilled along with the retention data.

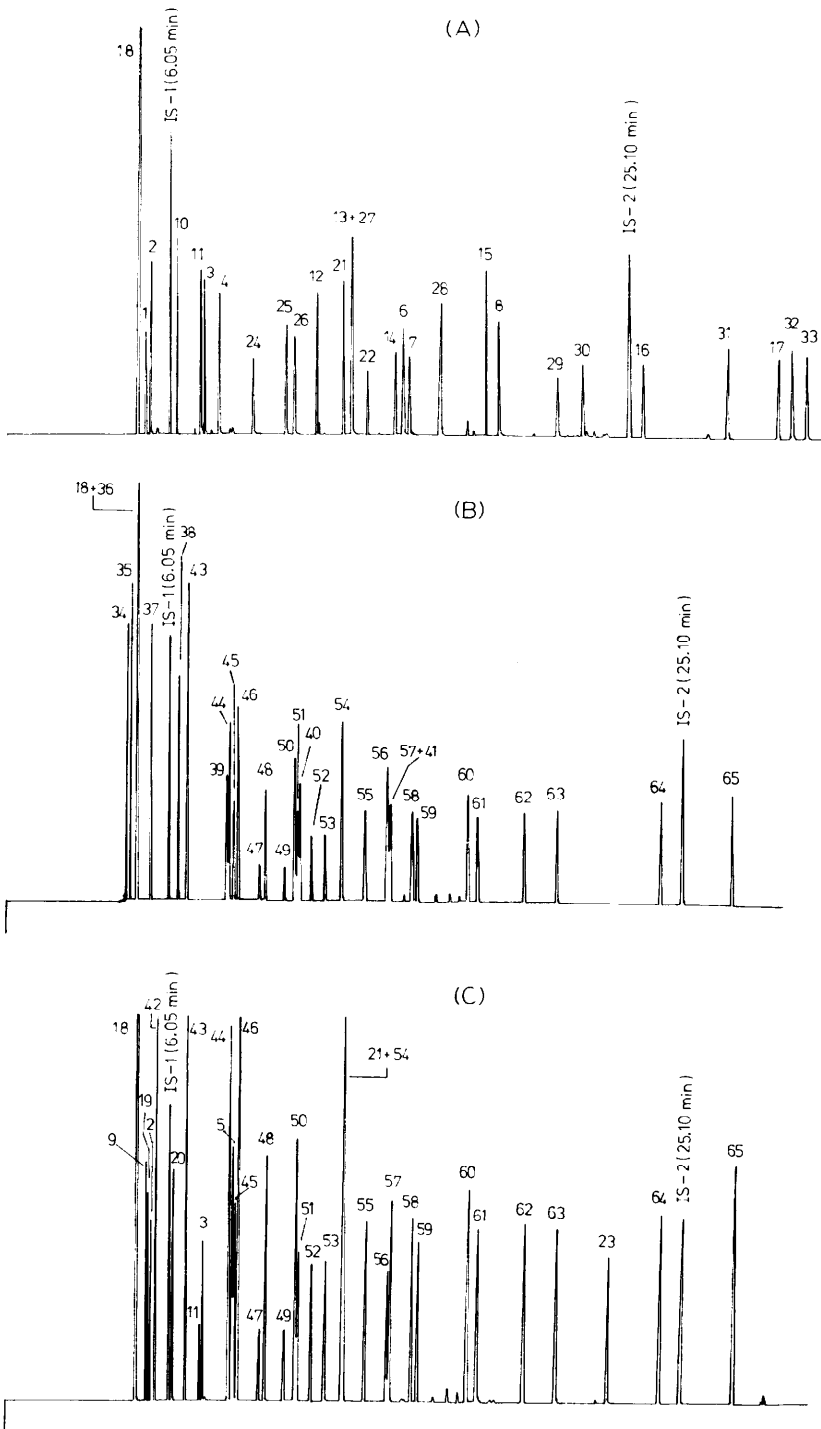


Fig. 2. Gas chromatograms of commonly used solvent components. Peak numbers: see Table I.

TABLE II

ORGANIC SOLVENT COMPONENTS IN TYPICAL SYNTHETIC RESIN PAINTS

The numbers in parentheses indicate components listed in Table I. BrC_n = branched hydrocarbons with *n* carbon atoms.

Paint	Sample No.	Components (% _v , w/w)
Acrylic resin	1	(1) 0.3, (7) 0.2, (9) 0.2, (30) 0.5, (32) 1.2, (33) 0.8
	2	(2) 0.3, (5) 1.6, (11) 3.5, (13) 9.7, (25) 5.8, (30) 1.2, (31) 2.6, (43) 0.5, (44) 1.8, (45) 0.5, (46) 1.0, (48) 0.6
Alkyd resin	1 (hard gloss)	(BrC ₉) 1.5, (37) 2.0, (BrC ₁₀) 2.6, (38) 3.8, (BrC ₁₁) 3.4, (39) 2.5, (BrC ₁₂) 0.2, (40) 0.2, (BrC ₁₃) 0.5, (41) 0.7, (43) 1.0, (44) 0.7, (45) 0.5, (46) 1.0, (48) 0.7, (49) 0.2, (50 + 51) 1.6, (52) 0.7, (53) 0.4, (54) 2.3, (55) 0.7, (58) 0.2, (59) 0.4, (60) 0.1, (62) 0.1
	2	(BrC ₉) 0.5, (37) 0.9, (BrC ₁₀) 0.7, (38) 1.3, (BrC ₁₁) 1.2, (39) 1.0, (40) 0.1, (42) 0.4, (43) 0.5, (44) 0.4, (45) 0.4, (46) 0.8, (47) 0.1, (48) 0.5, (50 + 51) 1.2, (52) 0.6, (53) 0.4, (54) 1.9, (55) 0.5, (57) 0.5, (57) 0.5, (58) 0.2, (59) 0.3, (60) 0.1, (61) 0.1, (62) 0.1
Chlorinated rubber	1	(BrC ₁₃) 0.3, (41) 0.1, (44) 7.0, (45) 4.5, (46) 10.8, (48) 5.0, (54) 0.1, (58) 0.2, (59) 0.5, (60) 0.3, (61) 0.5, (62) 0.2
	2	(44) 6.3, (45) 5.7, (46) 14.4, (47) 0.1, (48) 7.2, (49) 0.5, (50 + 51) 2.8, (52) 1.0, (53) 0.5, (54) 2.9, (55) 0.4, (56) 0.2, (57) 0.6
Clear lacquer		(9) 20.0, (43) 9.4, (44) 23.9, (45) 6.8, (46) 17.2, (48) 7.3
Electric insulating		(1) 4.1, (2) 1.3, (3) 1.5, (5) 0.2, (10) 1.7, (11) 5.6, (43) 26.7, (44) 5.6, (45) 1.7, (46) 3.5, (48) 1.7
Epoxy resin	1	(16) 0.8, (19) 1.3, (20) 5.4, (21) 2.1, (43) 5.2, (44) 6.1, (46) 5.9
	2	(2) 6.2, (5) 6.4, (13) 5.3, (19) 8.6, (21) 9.3, (43) 36.8, (44) 6.1, (45) 0.7, (46) 1.4
Hydrophilic resin	1	(1) 2.0, (2) 10.7, (5) 6.8, (12) 10.4, (13) 10.8, (21) 2.8, (25) 0.7, (43) 1.1, (44) 2.5, (45) 0.7, (46) 1.5, (48) 0.8, (1-pentanol) 2.5
	2	(1) 2.4, (2) 0.7, (5) 8.4, (12) 13.5, (13) 25.7, (21) 3.4, (31) 1.6, (44) 1.4, (45) 0.6, (46) 1.2, (48) 0.5, (1-pentanol) 3.4
Phthalate resin		(BrC ₉) 1.0, (37) 1.0, (BrC ₁₀) 2.1, (38) 2.5, (BrC ₁₁) 2.8, (39) 1.8, (BrC ₁₂) 0.6, (40) 0.2, (BrC ₁₃) 1.0, (41) 1.0, (42) 0.7, (43) 0.3, (44) 0.8, (45) 0.4, (46) 0.7, (48) 0.7, (49) 0.3, (50 + 51) 1.9, (52) 1.0, (53) 3.3, (55) 0.8, (56) 0.1, (58) 0.3, (59) 0.1, (60) 0.3, (61) 0.3, (62) 0.1, (63) 0.1
Polyester resin		(1) 0.3, (3) 2.1, (5) 0.2, (11) 0.6, (16) 0.9, (25) 7.5, (29) 0.4, (30) 0.1, (43) 0.4, (44) 1.1, (45) 0.3, (46) 0.7, (48) 0.4, (50 + 51) 1.2, (52) 0.5, (53) 0.3, (54) 2.2, (55) 0.4, (58) 0.2
Synthetic resin, ready mixed		(BrC ₉) 0.8, (37) 2.0, (BrC ₁₀) 1.3, (38) 3.5, (BrC ₁₁) 2.6, (39) 2.4, (40) 0.2, (42) 0.7, (44) 1.0, (45) 0.5, (46) 1.1, (47) 0.1, (48) 0.7, (49) 0.2, (50 + 51) 1.6, (52) 0.8, (53) 0.4, (54) 2.4, (55) 0.6, (57) 0.6, (58) 0.2, (59) 0.4, (60) 0.2, (61) 0.1, (62) 0.1
Urethane		(9) 4.8, (10) 3.3, (13) 5.3, (14) 3.0, (21) 8.3, (44) 4.1, (45) 0.9, (46) 2.0, (48) 1.0
Vinyl resin	1	(9) 17.6, (11) 9.8, (42) 0.1, (43) 22.0, (44) 4.3, (45) 1.0, (46) 2.5, (48) 1.2, (59) 0.1
	2	(5) 50.6, (20) 1.1, (21) 23.7, (43) 8.0
Water emulsion		(31) 12.0, (triethylamine) 0.3*
Water-soluble resin	1	(30) 0.91, (32) 2.0, (33) 1.3
	2	(2) 5.5, (16) 1.7, (28) 7.9, (triethylamine) 1.1*
	3	(28) 10.1, (triethylamine) 1.4*
	4	(28) 0.8, (31) 0.9, (triethylamine) 2.6*

* Determined by using a 2 m × 2 mm I.D. glass tube packed with 10% PEG 20M + 10% KOH on 80–100-mesh Chromosorb W AW DMCS with temperature programming from 60 to 140°C at 6°C/min and a nitrogen flow-rate of 50 ml/min.

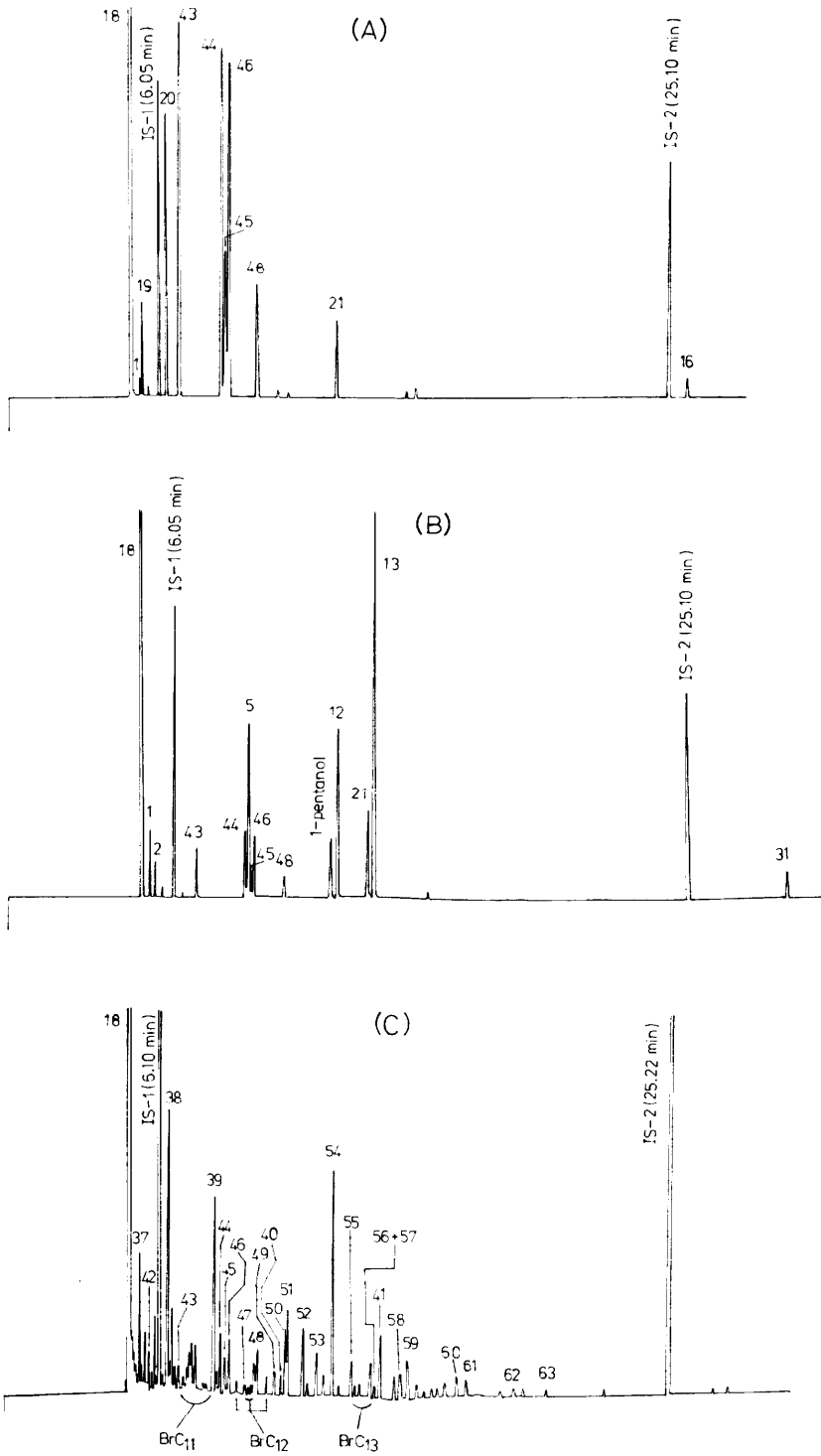


Fig. 3.



Fig. 3. Gas chromatograms of organic solvent components in typical synthetic resin paints. Peak numbers: see Table I. BrC_n = branched hydrocarbons with n carbon atoms. (A) Epoxy resin paint; (B) hydrophilic resin paint; (C) phthalate resin paint; (D) water-soluble resin paint.

Table II reports analytical data for various currently used paint solvents and Fig. 3 shows typical chromatograms. The various types of paint solvents were successfully determined with considerable savings of time and labour over conventional GC methods.

As these features indicate FSCC GC was useful in analysing routinely a large number of paint solvents for the investigation of toxic and/or photochemically reactive compounds in paint solvents. The data obtained will be helpful in minimizing the emission of such substances into the environment.

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