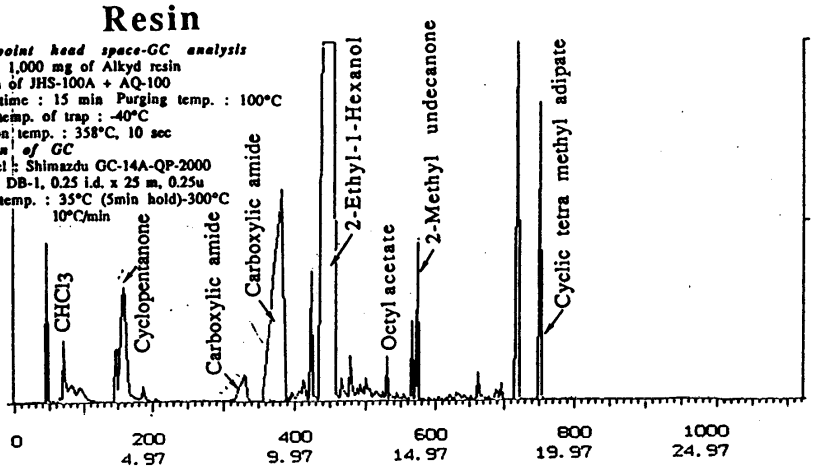
応用分析例

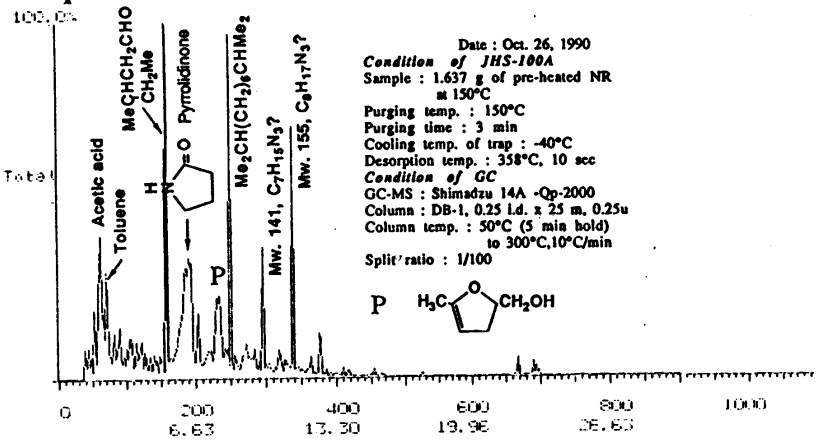
Head Space-GC/MS Analysis of Volatile Substances in Alkyd Resin

Curie point head space-GC analysis
Sample : 1,000 mg of Alkyd resin
Condition of JHS-100A + AQ-100
Purging time : 15 min Purging temp. : 100°C
Cooling temp. of trap : -40°C
Desorption temp. : 358°C, 10 sec
Condition of GC
GC model : Shimazdu GC-14A-QP-2000
Column : DB-1, 0.25 i.d. x 25 m, 0.25u
Column temp. : 35°C (5min hold)-300°C
10°C/min



塗料用のアルキッド樹脂中の揮発性物質をJHS-100に液体サンプラAQ-100を使用して分析した。残存溶媒、酸アミド、1、4ーブタンジオールのほか、副製物のアルコールエステル類を多数検出することができた。

Head space-GC-MS analysis of pre-heated NR

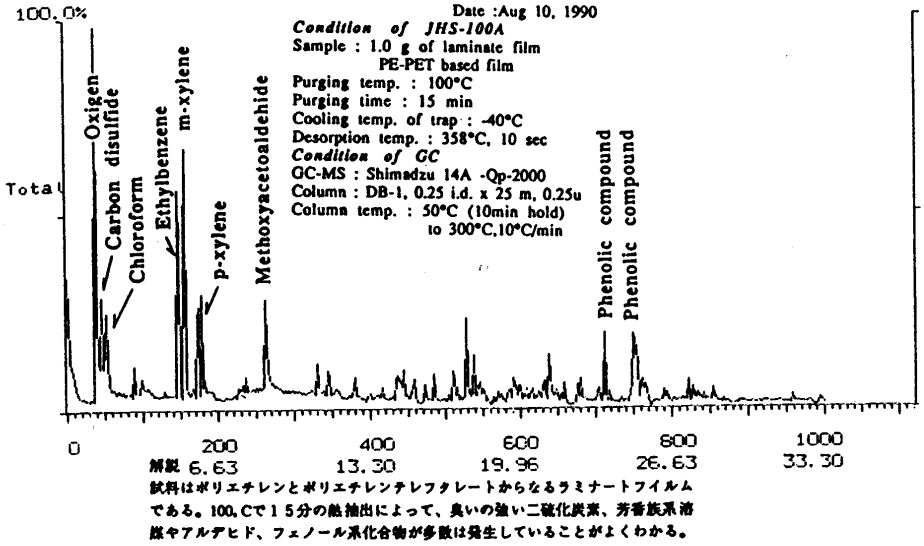


Date : Oct. 26, 1990
Condition of JHS-100A
Sample : 1.637 g of pre-heated NR at 150°C
Purging temp. : 150°C
Purging time : 3 min
Cooling temp. of trap : -40°C
Desorption temp. : 358°C, 10 sec
Condition of GC
GC-MS : Shimadzu 14A -Qp-2000
Column : DB-1, 0.25 i.d. x 25 m, 0.25u
Column temp. : 50°C (5 min hold) to 300°C,10°C/min
Split ratio : 1/100

P

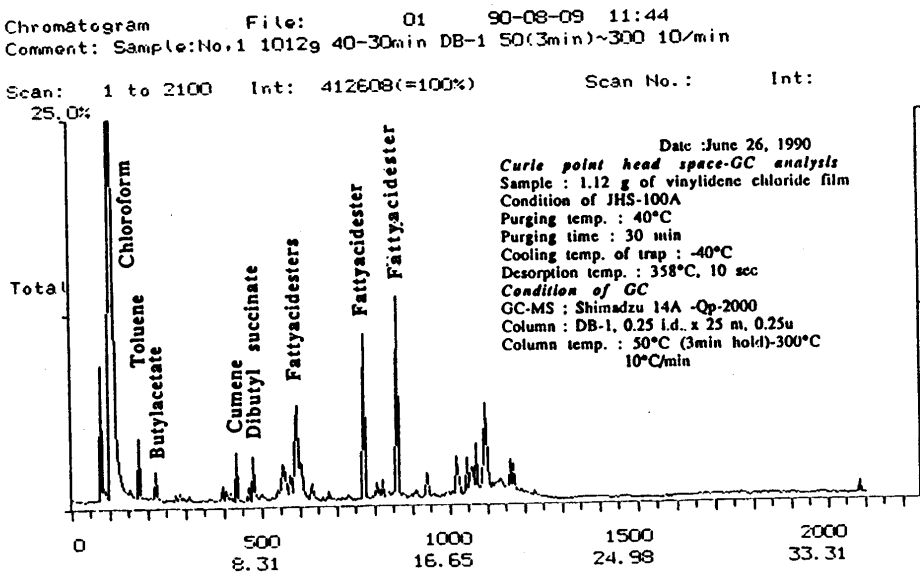
解説
パルク状の天然ゴムを粉砕するには150°Cで加熱することによって天然ゴム中に存在する蛋白質、糖類を一部分解したほうが容易に粉砕することができるようになる。この操作を行なうと、天然ゴムの臭気が著しく強くなる。天然ゴム中に残存する臭気成分を分析する目的でこの分析をおこなった。
クロマトグラムに記載されたアルデヒド類、窒素系化合物がその臭気成分と推定される。パラフィン系炭化水素類も残存していることがよくわかる。

Head space-GC-MS analysis of laminate film



試料はポリエチレンとポリエチレンテレフタレートからなるラミナートフイルムである。100.Cで15分の熱抽出によって、臭いの強い二硫化炭素、芳香族系溶媒やアルデヒド、フェノール系化合物が多数は発生していることがよくわかる。

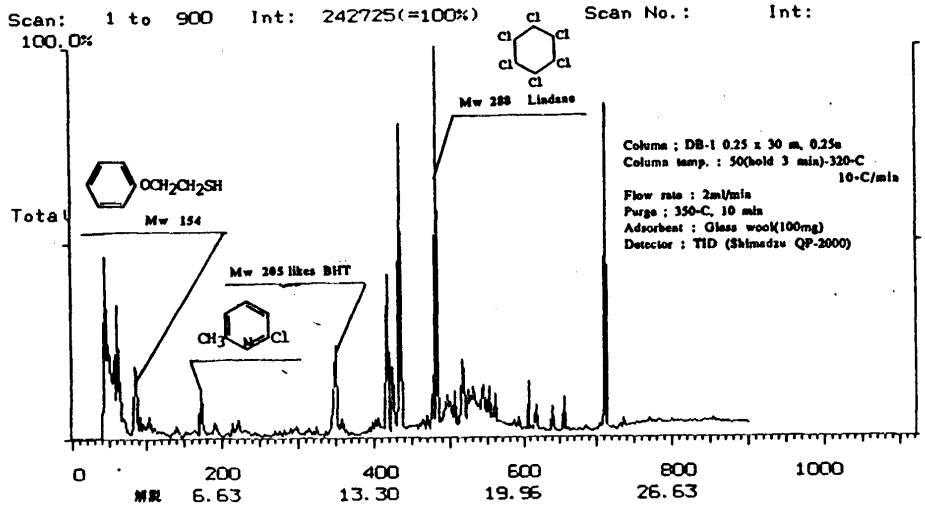
Head space-GC-MS analysis of Poly vinylidene chloride film



Polyphenylene sulfide

Chromatogram File: PPSJAI.02 90-03-02 12:50
Comment: 99.9 mg glass wool

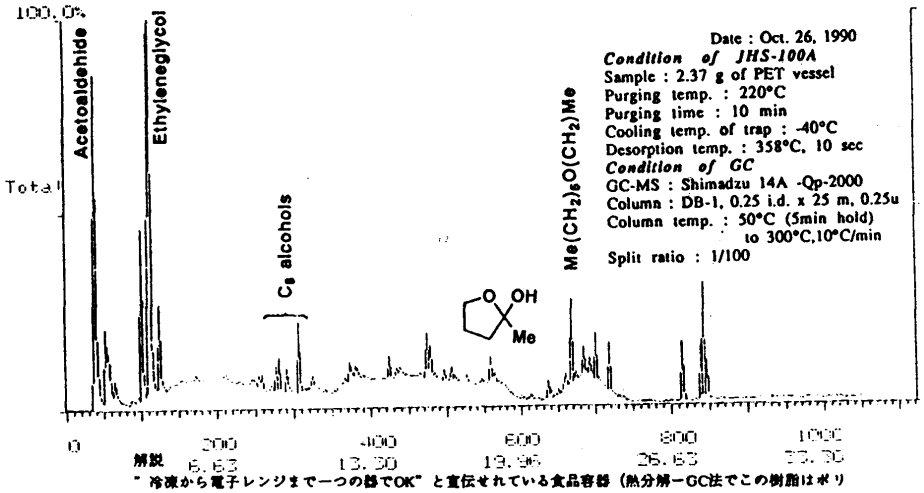
Scan: 1 to 900 Int: 242725(=100%) Scan No.: Int:



解説

ポリフェニレンサルファイド樹脂は安定な樹脂ではあるが、350°Cで10分間熱抽出を行なうと原料に含まれていたと思われる塩素系化合物を検出することができた。

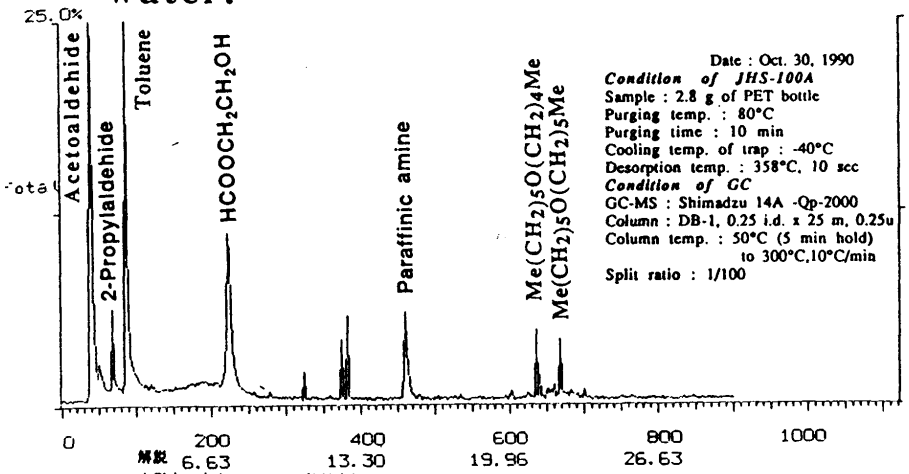
Head space-GC-MS analysis of food vessel for the microwave oven



解説

"冷凍から電子レンジまで一つの器でOK"と宣伝されている食品容器（熱分解ーGC法でこの樹脂はポリエチレン テレフタレートと判明）がある。この説明書によると220°Cまでならオーブンで加熱可能と述べられていることから、この容器をその温度で10分間加熱をおこない発生するガスの分析をおこなつた。
樹脂原料に由来するアセトアルデヒド、エチレングリコールが主成分であつた。

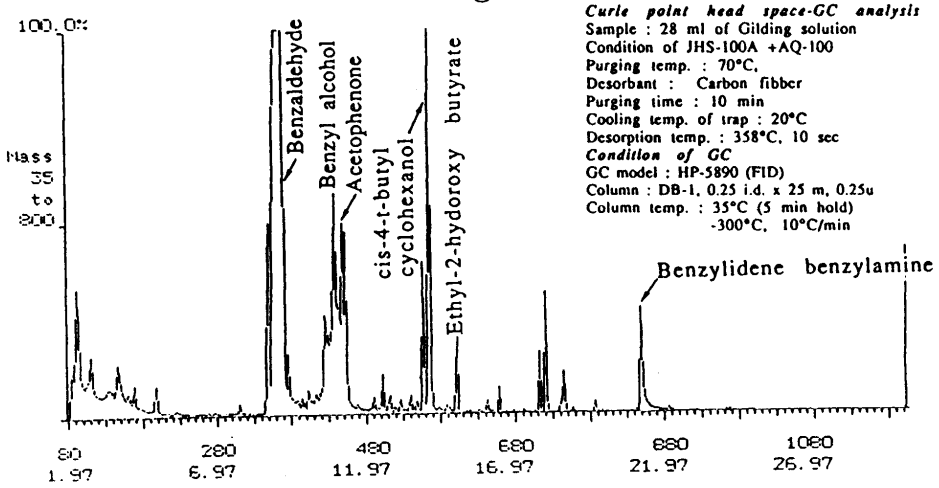
Head space-GC-MS analysis of PET bottle for the drinking water.



解説

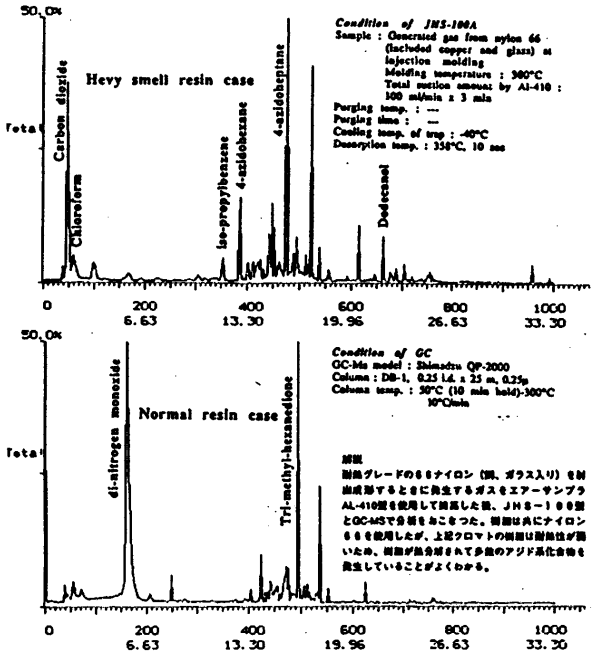
六甲山の水をパックした飲料水用の容器（熱分解－GC法でこの樹脂はポリエチレン テレフタレートと判明）を5mm x 5mm角に切断して試料とし80°Cで１０分間熱抽出をおこない発生ガスの分析をおこなった。アセトアルデヒド、トルエン、エチレングリコール脂肪酸エステル、脂肪族アミンなどを検出することができた。

Head Space-GC/MS Analysis of Volatile Organic Substances from Electric Gilding Solution.

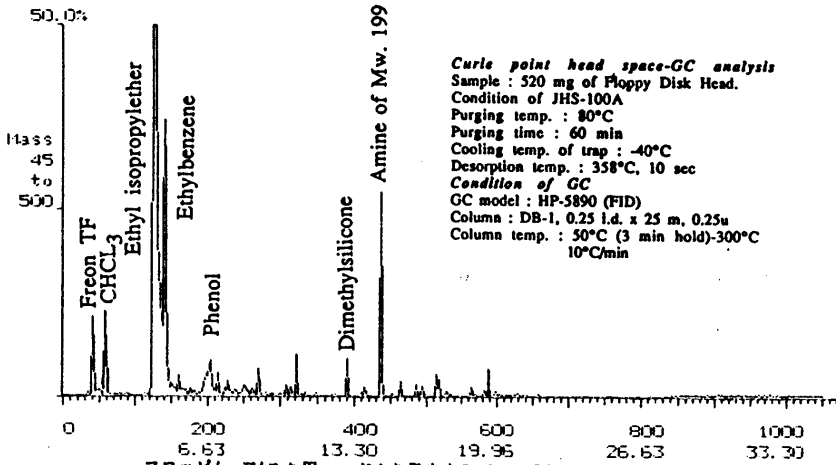


光沢ニッケル鍍金液中に含まれる有機物をJHS-100型と液体サンプラAQ-100型を使用して分析を行なった。光沢剤等有機物が、電極で起こる酸化還元反応によって、多数の有機物が生成されていることがわかる。

Generated Gas Analysis from Nylon 66 at Injection Molding by Head Space-GC/MS

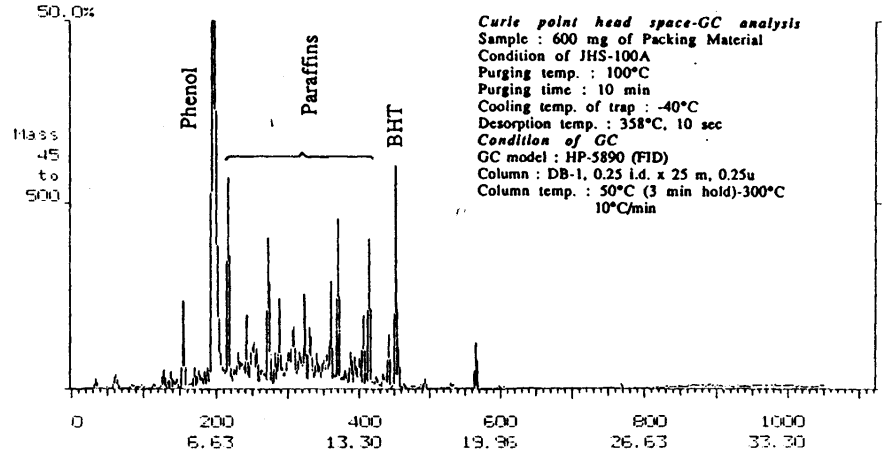


Head Space-GC/MS Analysis of Volatile Substances from Floppy Disk Head



フロッピイ ディスク用ヘッドより発生するガスを分析した。残存フロン溶媒、接着剤より発生したエチルベンゼン、フェノールのほかディメチルシリコンを検出することができた

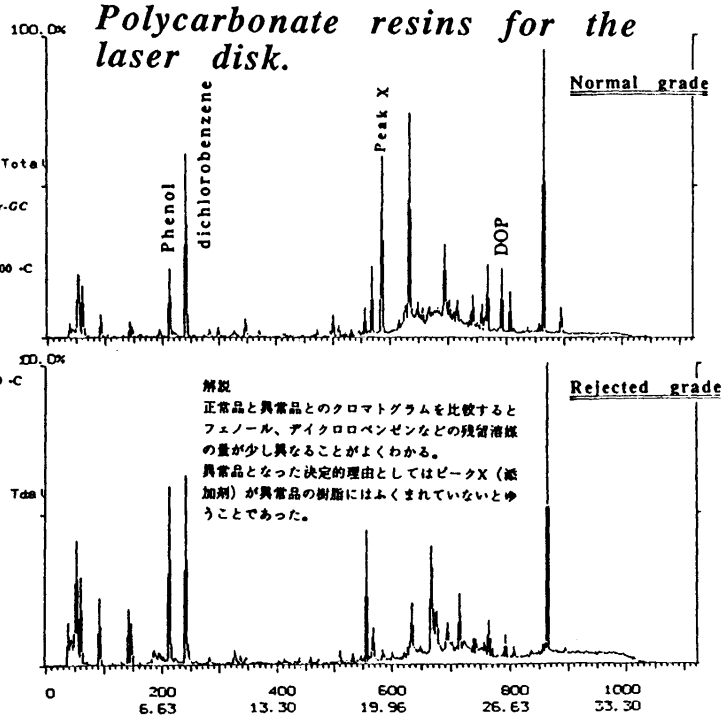
Head Space-GC/MS Analysis of Packing Material for Electronic Goods



Curie point head space-GC analysis
Sample : 600 mg of Packing Material
Condition of JHS-100A
Purging temp. : 100°C
Purging time : 10 min
Cooling temp. of trap : -40°C
Desorption temp. : 358°C, 10 sec
Condition of GC
GC model : HP-5890 (FID)
Column : DB-1, 0.25 i.d. x 25 m, 0.25u
Column temp. : 50°C (3 min hold)-300°C
10°C/min

塩化ビニール製の電子部品用の梱包剤を分析した。塩化ビニールの安定剤であるBHTのほか、出所不明のフェノールおよび数種のパラフィン系化合物が得られた。

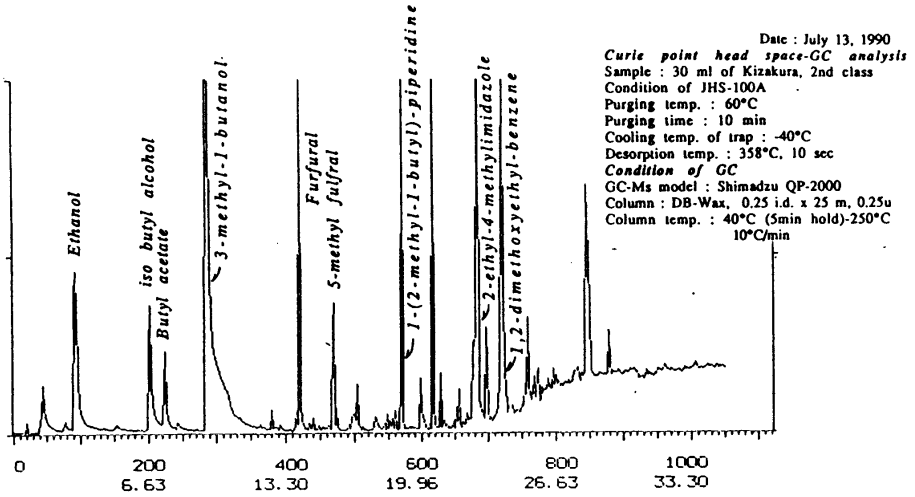
Polycarbonate resins for the laser disk.



Curie point head space sampler-GC analysis
Sample name ; Polycarbonate resin
Sample amount ; 111 mg
Pyrolysis temp. : 50 °C (3 min hold) x 300 -C
Purging temp. :250 -C, 10 min
Cooling temp. : -40-C
Needle temp.; 400 °C
GC model ; GC-14A
MS model ; Shimadzu QP-2000
Columa ; DB-1, 0.25 i.d. x 30 m
Columa temp. ; 50°C,(hld. 3 min) to 300 -C
10-C/min °C/min
Split ratio ; 1/100

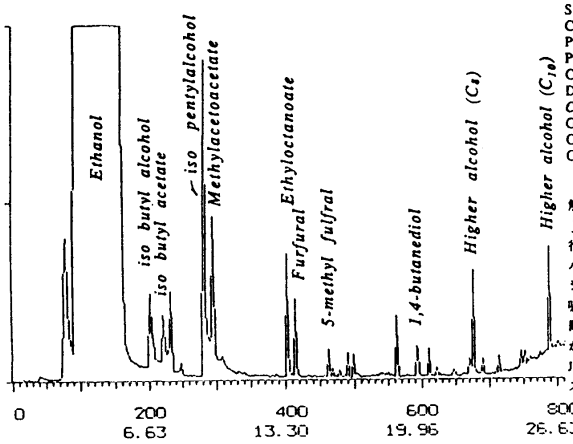
解説
正常品と異常品とのクロマトグラムを比較するとフェノール、ディクロロベンゼンなどの残留溶媒の量が少し異なることがよくわかる。
異常品となった決定的理由としてはピークX（添加剤）が異常品の樹脂にはふくまれていないとゆうことであった。

Head space-GC-MS analysis of Japanese sake.



Date : July 13, 1990
Curie point head space-GC analysis
Sample : 30 ml of Kizakura, 2nd class
Condition of JHS-100A
Purging temp. : 60°C
Purging time : 10 min
Cooling temp. of trap : -40°C
Desorption temp. : 358°C, 10 sec
Condition of GC
GC-Ms model : Shimadzu QP-2000
Column : DB-Wax, 0.25 i.d. x 25 m, 0.25u
Column temp. : 40°C (5min hold)-250°C
10°C/min

Head space-GC-MS analysis of Whisky.



Date : July 13, 1990
Curie point head space-GC analysis
Sample : 30 ml of Suntry Reserve
Condition of JHS-100A
Purging temp. : 60°C
Purging time : 10 min
Cooling temp. of trap : -40°C
Desorption temp. : 358°C, 10 sec
Condition of GC
GC-Ms model : Shimadzu QP-2000
Column : DB-Wax, 0.25 i.d. x 25 m, 0.25u
Column temp. : 40°C (5min hold)-250°C
10°C/min

解説
ＪＨＳ－１００Ａ型に液体サンプサーAQ100型を付属して酒類の分析を行なった。AQ-100に試料をセットして60.Cでパブリングを行なった。パブリング時に多量に発生する水はJHS-100A型の試料管内の塩化カルシームで除去されるが、その他の成分はテナックスを充填した吸着管に吸着される。１０分間パブリングを行なった後乾燥窒素を吸着管に３分間程度流すことによって、吸着管内に過剰に吸着したエタノールは破過が小さいことからエタノールを優先的に除去することができる。
JHS-100A型はパージアンドトゥラップ方式のため、静止型ヘッドスペースサンプラに比べ高沸点化合物まで幅広く分析できることがよくわかる。

(4) 複合分析法による最新の高分子化合物の分析

(A) キューリーポイント Py－GC 法による PET フィルム上の

超薄膜の定量分析

要旨

PET 上に塗布された起薄膜の PMMA フィルムの膜厚を Py-GC 法によって再現性良く測定することができた．1.350μの膜厚を測定することができる検量線が得られた．

Quantitative Analysis of the Ultra-thin Layer- of
PMMA on PET Film Surface by Curie-Point
Pyrolysis - Gas Chromatography

Sample

Standard samples: Ultra-thin layers with the thickness of 0, 100, 240 and 360Å on PET films(thickness of 0.12 mm), respectively.

Unknown sample: Coded "a" and "b".

Experimental

Sample size: 0.4 - 0.5 mg without pretreatment.

Pyrolysis: JAI JHP-3 Curie-point pyrolyzer with Pyrofoils of 315, 423, 500 and 590.C for 5 see. Oven and transfer pipe temperature: 210 and 200℃, respectively.

GC: HP 5890A GC system with Hitachi D-2520 Integrator. DB-1701 fused silica capillary column(J & W Sci.). 30 m x 0.25 mm i. d., 30℃(10 min) to 300℃, lO℃/min, He 50 m1/min. split 50:1.

Results and Discussion

1. As well known. the typical characteristic pyrolysate of polymethyl methacrylate(PMMA) on the pyrogram is methyl methacrylate(MMA. monomer), and pyrolysates of benzene(B), divinyl terephtalate(DET, monomer), etc. could represent the presence of polyethylene tcrephtalate(PET) in sample [l]. Considering the relative Weights of PMMA in the samples are extremely small(1/10.000 to I/3,000 on thickness for standard samples), all of the experimental.parameters, such as pyrolysis temperature, Sample size and GC separation conditions, have great influence on the quantitative identification of PMMA fraction from PET background. Therefore, it is necessary to vary and estimate the experimental parameters carefully. When, the samples are

pyrolyzed at lower temperatures(315 and 423oC). much of the polymers are not pyrolyzed even if the time is increased [2,3]. Contrariwise, pyrolysis at higher temperatures(590℃, for example)

formed much of the lower boiling point pyrolysates which could overlap the weak peak of PMMA. Therefore, pyrolysis temperature of 500℃ is recommended in this work. Rather large sample size is necessary to identify small amount of PMMA fraction. But, the large amount of sample will influence the reproducibility, because the heat is transferred more slowly in a large sample [4]. The smaller the sample size, the more homogeneous is the temperature in the sample and the straighter is the calibration graph. However, there is a reasonable sample size range in 0.4 - 0.5 mg that can be used for this study. Finally, the GC condition should be available for separation of MMA peak from other peaks especially in relative small retention time range, so that the minimum column temperature have to be set at a lower temperature. The illustration pyrogram of a standard sample pyrolyzed at 500℃ is shown in Fig.1, in which a small and significant MMA peak could be found clearly.

2. As mentioned before, PET weight fractions are incomparably larger than PMMA in the films, i.e. the relative contents of PET in the samples can look upon as a constant. Therefore, the complete separated chromatographic peaks and structural characteristic pyrolysates of PET, such as benzene(a), Styrene(S), DET etc. can be defined as the internal standards for quantitative analysis. The plots of peak area ratios of MMAIB, MMA/S and MMA/DET vs. thickness of PMMA on PET films are shown in Fig.2. Excellent liner relationships could be found in the ,.calibration curves, even for the trace of PMMA. The data also reflected that the 'selection of internal standards, especially the intensity comparable peaks, is reasonable. Therefore, the calibration data can be used for quantitative analysis of the ultra-thin layer of PMMA on PET film surface on the level of 10-4 graduate.

3. The data for unknown samples of "a" and "b" listed in Table 2. That results showed that all of the calibration curves, i.e. the internal standards, are available for quantitative microanalysis of ultra-thin layers of PMMA on PET films and giving correlative data. The measured thickness of sample "b" also indicated the sensitivity limitation of this method may be extended to 1/20,000 for PMMA-PET system.

Conclusion

Microanalysis of polymer materials is getting interested especially in high technique field recently. Lack of the information in this area made us to find new measurement for that purpose. This work implied that Curie-point pyrolysis - gas chromatography is perhaps a potential method for simple and rapid microanalysis of polymer materials without pretreatment.

Reference I

[1] S.Tsuge, H.Ohtani, Pyrolysis Gas Chromalography - Fundamental and Pyrogram Alias, Tech.Syst.. Tokyo, 1989, p.142. 310.

[2] I.Ericsson, I.Chromalog.Scl'., 16. l978, 340.

[3] I.Q.Walker. I.Chromalog.ScI'.. 15, l977. 267.

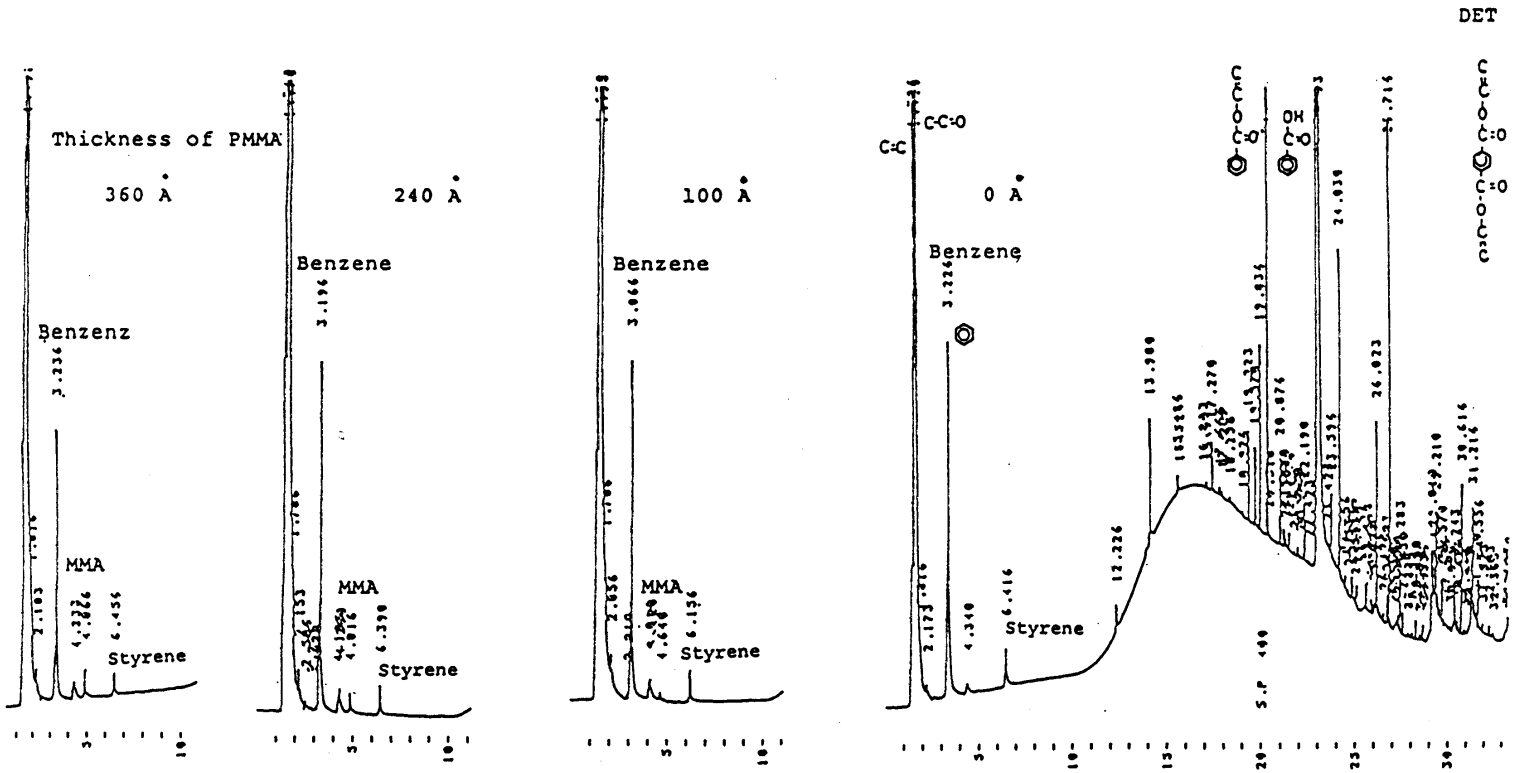
[4] I.Ericsson, J.Anal.Appl.PyroI., S. 1985, 73.

Table 1 Calibration Data

PMMA Thickness, Å	0	100	240	360
Peak Intensity, a.u.				
MMA	0	2,118	5,463	5,610
Benzene(B)	-	94,294	108,073	85,989
Styrene(S)	-	8,624	9,019	6,663
Monomer(DET)	-	105,707	104,281	74,927
Intensity Ratio				
MMA/B, x 10^2	0	2.25	5.05	6.52
MMA/S, x 10	0	2.46	6.06	8.42
MMA/DET, x 10^2	0	2.00	5.24	7.49

Table 2 Determination of Unknown Samples

Sample Code	Peak	Intensity a.u	Intensity Ratio	Thickness of PMMA, Å Experimental	Average
a	MMA	4,875			
	B	230,212	MMA/B = 0.0212	108	
	S	19656	MMA/S = 0.248	104	106
b	MMA	1,538			
	B	132,930	MMA/B = 0.0116	62	
	S	10,533	MMA/S = 0.146	60	61



Pyrograms of PET Films with Ultra-thin PMMA layers

Pyrol.Temp.: 500°C, JHP-3; Column: DB-1701, 30°C, 10 min.- 300°C, 10°C/min.

(B) キューリーポイント　ヘッドスペース　サンプラによる
フロッピイディスクより発生する揮発性成分の分析

フロツピイディスクを 7℃以下の低温下で使用するとフロッピイディスクが破損したり、磁気ヘッドを傷める事故が多くなる．それらの事故原因は、フロッピイディスクの表面上に浸析してくるオイル状物質の種類とその量と言われている．最初のデータは、事故の最も少ないフロッピイディスクの分析例で、後のデータは事故の多いフロツビイディスクの分析例である．

APPLICATION OF CURIE POINT PYROLYSIS AND HEADSPACE GC-MS IN FLOPPY DISK ANALYSIS

Floppy disk consists of PET film and double side magnetic coating layers, which are complex formulation of ferromagnetic powder(Fe304), polymeric adhesive and some kinds of small molecular additives. Once produced, it may be difficult to analyze since it usually is a multi-component and insoluble polymer system. However, PyGC-MS should be useful in the analysis of the insoluble polymer adhesive without separation from ferromagnetic material, but the presence of semi-volatile additives may interfere that process. In case such as this, combination' with headspace GC-MS could be available for floppy disk analysis.

Sample and Pretreatment

Sample: Commercial 5 1/2 floppy disk.

Pretreatment: For PyGC-MS analysis, a small piece of disk is soaked in chloroform for a few min, then scrape off the magnetic coating material from PET film in $CHCl_3$ Carefully, remove $CHCl_3$ by heating at 50°C. Non-pretreatment for headspacc analysis.

Analysis Conditions

Pyrolysis: JHP-3 Curie Point Pyrolyzer with a Pyrofoil of 590°C, pyrolysis time: 5 see, oven temp.: 200°C, interface temp.: 200°C, sample weight: 1 mg including Fe_3O_4.

Headspace: JHS-100 Headspace Sampler, purging temp.: l50°C. time.. 10 min, adsorption on Tenax, temp : -40°C, time: 10 min, desorpting temp.: 358°C by a Pyrofoil, time:.10 see, : sample weight: 150 mg including PET and Fe_3O_4. .

GC –MS: Shimadzu GCMS-QP 2000A, column: J & W Sci. DB-1 fused silica capillary column (30 m × 0.25 mm i.d.) coated with bonded methyl silicone polymer, column temp.: 40°C to 250°C, l0°C/min, carrier gas: He, 50 m l /min split: 50:1, quadrupole MS, EI, 70 ev.

Results

Fig.1-A illustrated the total ion current(TIC) pyrogram of the coating layer materials with the peak assignments.. The most characteristic degradation products, tolylene diisocyanate(TDI) and benzene mono-cyanate(BMI), exclusively corresponded to TDI-polyurethane component in the polymer materia1[1,2]. A cylic urethane(CU) peak yielded by the decomposition through an ester exchange process of polyurethane also reflected that conclusion[3]. Some alicyclic pyrolyzates, cyclopentanone(CP) and tetrahydrofuran(THF), are formed from adipic acid and butanediol portions of the polyester segments, respectively[1,2]. Summarizing all of the pyrogram characters, the coating polymeric material of the floppy disk is identified as TDI polyester-polyurethane. Otherwise, a small and wide peak of benzonic acid(BA) is implied by the degradation of residual PET film. Some higher aliphatic alcohols and acid peaks in Fig.1-A are though to arise from the additives of the material or/and that with partial degradation, which would be confirmed by using headspace GC-MS analysis.

The TIC chromatogram from headspase GC-MS of the floppy disk is shown in Fig.1-a with the peak assignments. The main peaks of cyclohexanone(CH) and toluene(T) indicated the presence of the residual solvents. There also are some higher aliphatic alcohols peaks in Fig.1-B, as the pyrogram. Therefore, they may be assumed as additive(s), for example lubricant or antistatic agent in the material. But we prefer to suggest that the original carbon numbers of the aliphatic chains are perhaps not definite, like the MS spectra showed. The reason could be attributed to the thermal cleaveges of the longer aliphatic chains, especially that with branching and unsaturated bond structures.

Conclusion

The combination of Cuire point pyrolysis and headspace GC-MS measurements is not only for the identification of the material composition of the floppy disk in this work. but also shown it would be a powerful technique for the analysis of multi-component polymer materials, especially of that including non-volatile, semi-volatile and volatile component samples.

References

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[4]N.Oguri, A.Onishi, T.Hanai, J.High Resolullon Chromatog., 14', 79 (1991).

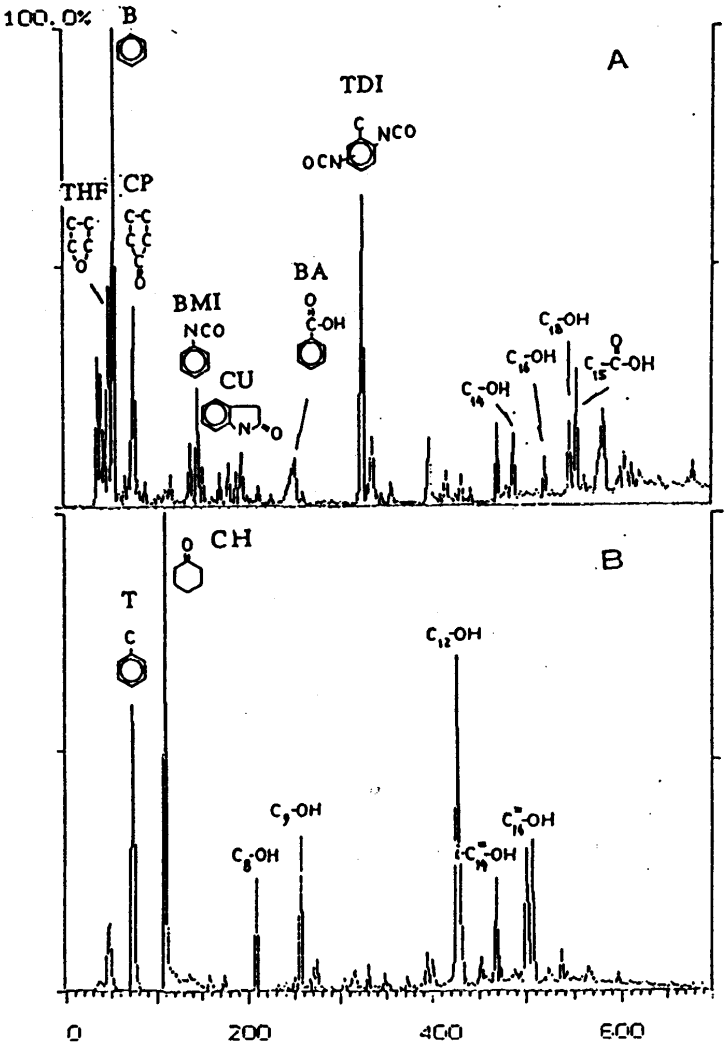

Fig.1 TIC Pyrogram(A) and Headspace Chromatogram(B) of the Floppy Disk.

References

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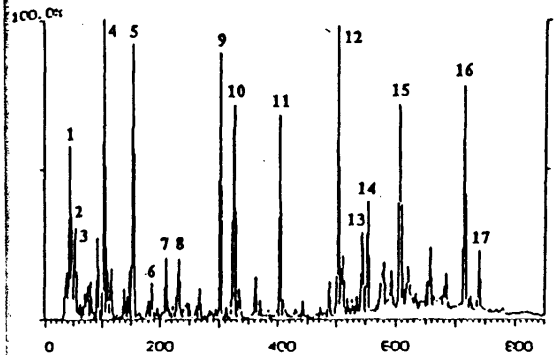


Fig.1 TIC Pyrogram of the floppy disk.

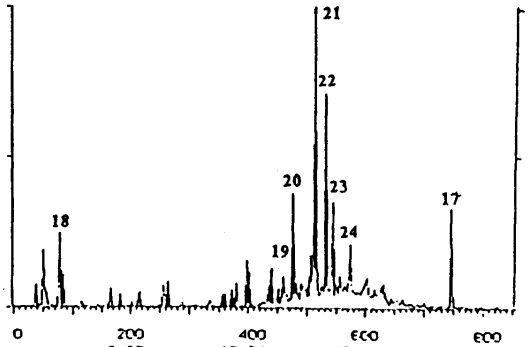
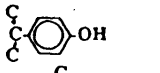
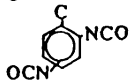
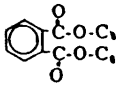
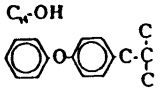
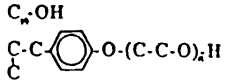
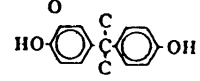
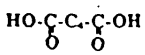


Fig.2 TIC Chromatogram from Headspace of the floppy disk.

Table 1 Peak assignments of the pyrogram & chromatogram.

1	C_3-CN	9		16	C_1-OH
2		10		17	
3		11	C_{13}-OH	18	
4	C_4-OH	12	C_{16}-OH	19	C_{15}-OH
5		13		20	C_{18}-OH
6		14	C_{17}-OH	21	
7	HO-C_4-OH	15		22	C_{14}-OH
8				23	C_{12}-OH
				24	C_{10}-OH

（C）複合分析法による熱硬化性不飽和ポリエステルの分析

試料 A（硬化剤）と試料 B（ポリマ）を分取 HPLC．Py－GC／MS．ヘッドスペースーGC／MS 及び DI Probe-MS を使って完全分析を行なった．

POLYMER ANALYSIS BY COMBINATION OF CURIE-
POINT HEATING TECHHIQUES AND PRRPARATIVE
HPLC
EXPERIMENTA L

Sample;

A set of uncured unsaturated -polyester resin separated to two parts (coded sample A and B) consists of prepolymer, reactive monomer, curing accelerator and additives.

Instruments and Expermental Conditions

Pyrolysis: JHP-3 Curie-point Pyrolyzer with a Pyrofoil of 500 or 220℃ for 5 sec. Oven and interface temperature: 200℃ and 250℃ respectively. Sample size: 0.1 mg.

Headspace: JHS-100 Curie-Point Headspace Sampler, purging at 220℃ for 1 min, adsorption on Tenax at –40℃, desorpting at 255℃ by a foil for 10 sec, sample size: 0.3 mg.

DI Probe: JDl-800 Curie-point DI Probe is inserted in a mass system up to the position where the sample cell (9 mm × 2 mm i.d., quartz) at the tip of the probe can be connected to the ion source.

GC-MS: Shimadzu GCMS QP 2000A with a J & W Sci. DB-1 fused silica capillary column, 35℃(5 min) to 300℃, 8℃/min, He: 50 m1/min, split of 50:1. Mass: El 70 ev, 250℃ HPLC: LC-908 Recycle Preparative HPLC with columns of Jaige1-lH and JaigeI-2H, $CHCI_3$ 3.8 ml/min. 40 Kg/cm2 , uv-254H and RI detectors, injection: 3 ml, 50 mg

RESULTS AND DISCUSSION

Curie-pint PyGC-MS, Py DI-MS, headspace GC-MS and preparative HPLC were Combined for the compositional analysis of the polymer The flowchart diagram of the analysis process is illustrated in Fig.1.

The TIC pyrogram of sample A at 500℃ is showed in Fig.2, in which some pyrolysates, such as hexladiene(peak 2), hexeno1(peak 3) phthalic anhydride(peak 5), mono-cyclohexenyl phthalate(peak 7) and dihexenyl phthalate(peak 11. monomer) reflected on the segments of a unsaturated polyester derived from phthalic acid and hexendio1 [1,2]. Other Pyrolysate peaks in Fig.2 may be caused by the presence of some aromatic compound(s) mixed with polyester. To confirm that. sample A was separated and collected to two fractions, F-I and F-2 as shown in Fig. 3, by recycle preparative HPLC [3], and then F-I and F-2 were identified by PyGC-MS again, respectively. The pyrogram F-1 showed all of the characters of poly(dihexenyl phthalate) giving the same conclusion as before. The sufficient decomposition of F-2 at 220℃ indicated it would be a thermally labile compound. Summarizing all of the pyrolysis features, F-2 could be assigned to benzoyl peroxide as a curing accelerator for the unsaturated polyester. as shown in Fig.4. Finally a peak 12 in Fig.2 indicated a high aliphatic alcohol presented in the material, implying that as a kind of surfactant.

Some acrylate monomer peaks, methyl acrylate(MA), methyl methacrylate(MMA) and iso-hexyl methacrylate(HMA) were identified on the pyrogram of sample B (Fig.5-a)[4]. Therefore sample a could be reasonably deduced as a copolymer of MA, MMA and HMA. Otherwise, a high aliphatic alcohol peak and a dioctyl phthalate (DOP) peak in Fig.4-a regarded as some additives, perhaps as surfactant and plasticizer in the material. respectively.

Fig.6 is the TIC pyrogram and corresponding mass spectrum of sample B pyrolyzed at 500℃, which indicated the species observed is co(MA-MMA-HMA)polymer from the monomer fragments with m/e values of 86(MA), 100(MMA) and l70(HMA). Though the conclusion is the same as that by PyGC, but it is suitable for fingerprint identification purpose. In addition, a through-put of up to one sample was within few minutes, and then rapid analysis is getting possible [5,6]

Headspace GC-MS was carried out at a purging temperature of 225°C, in which a monomer-free co(MA-HMA)polymer was analyzed first. No significant pyrolysate formed at that temperature for the purified copolymer. A headspace TIC chromatogram of sample B is shown in Fig.4-b, in which. the monomer peaks of MMA and HMA presented with obverse intensities compared to sample size. The results pointed out there are some reactive monomers dissolved in the copolymer as the crosslinking agents of the unsaturated polyester [7]

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熱硬化性不飽和ポリエステル樹脂の分析

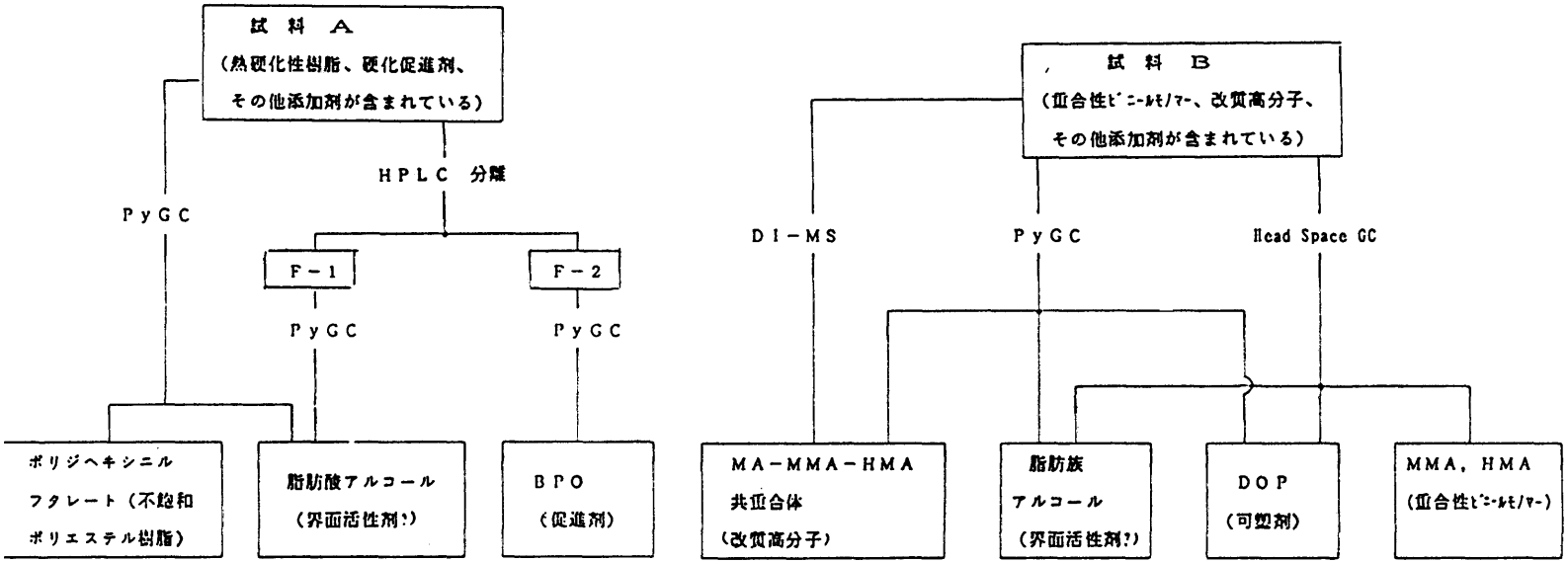
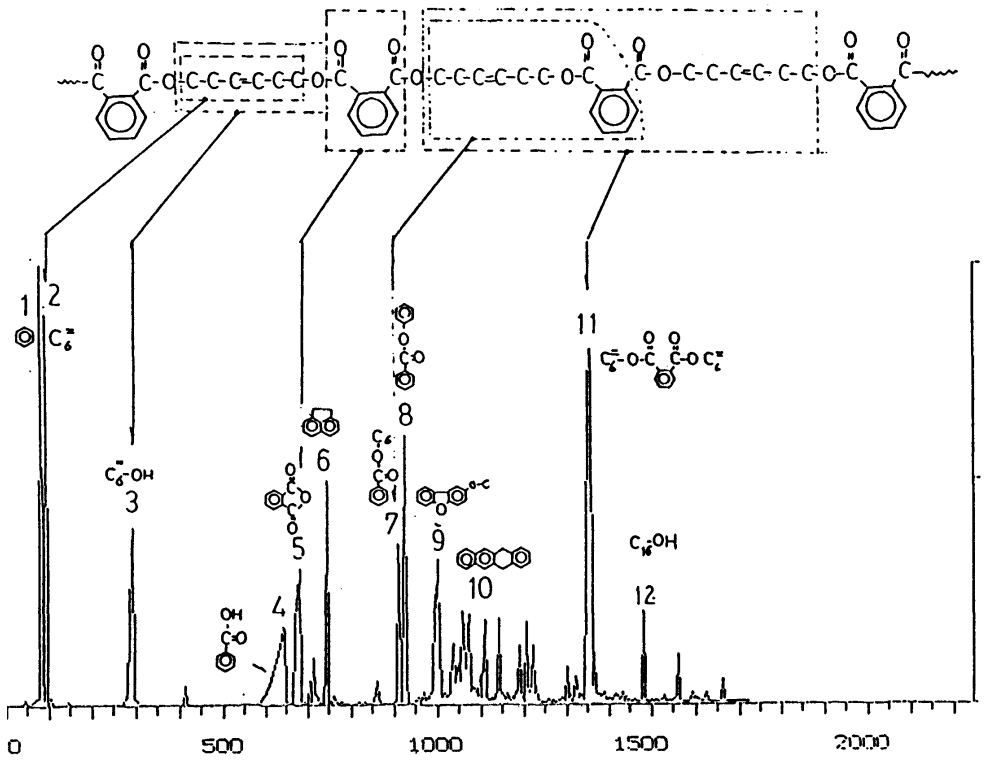


Fig.1 Flowchart Diagram of the Polymer Analysis



	1	Benzene	△	7	Cyclohexenyl phthalate
△	2	Hexadiene		8	Phenyl benzoate
△	3	Hexenol		9	Methoxydibenzofuran
	4	Benzoic acid		10	Dihydronaphthacene
△	5	Phthalic anhydride	△	11	Dihexenyl phthalate
	6	Acenaphthene	◇	12	C_{16} aliphatic alcohol

Fig.2 TIC Pyrogram. of Sample A

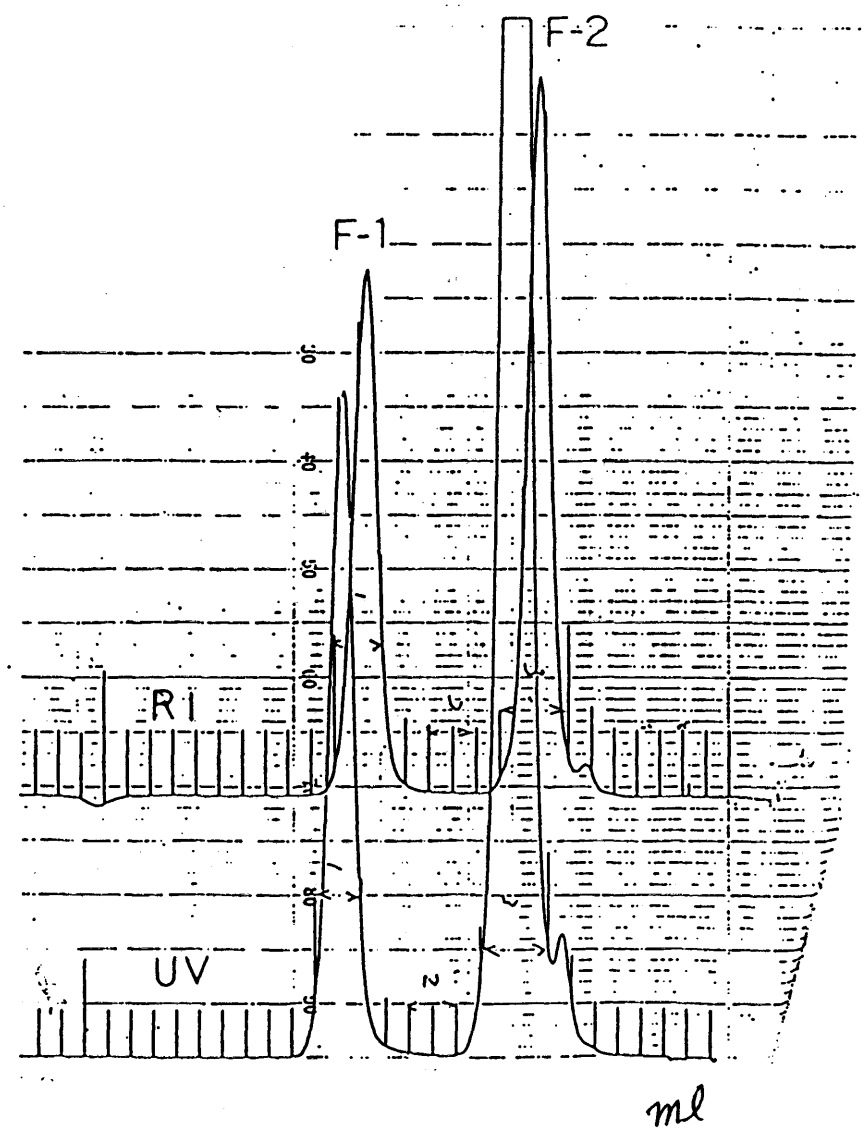
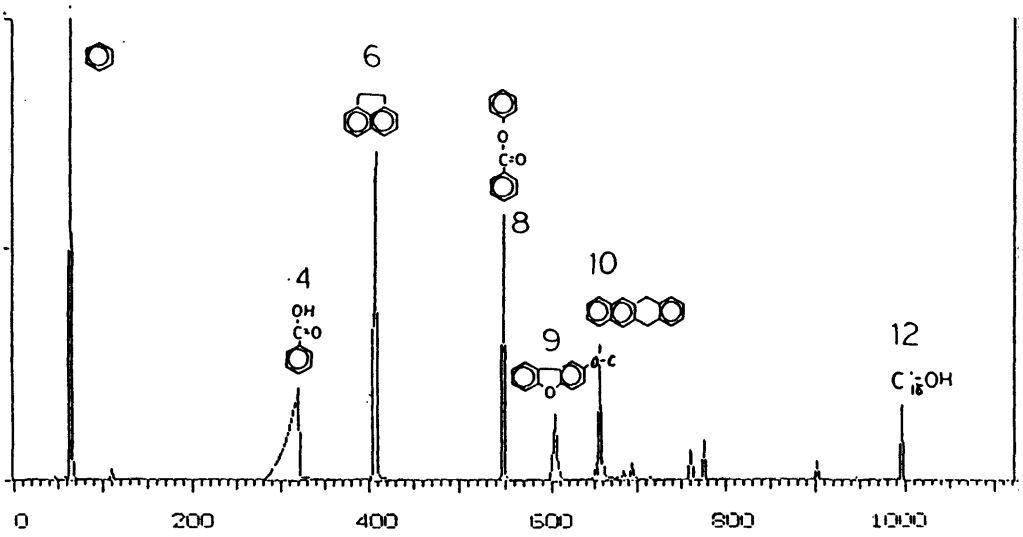
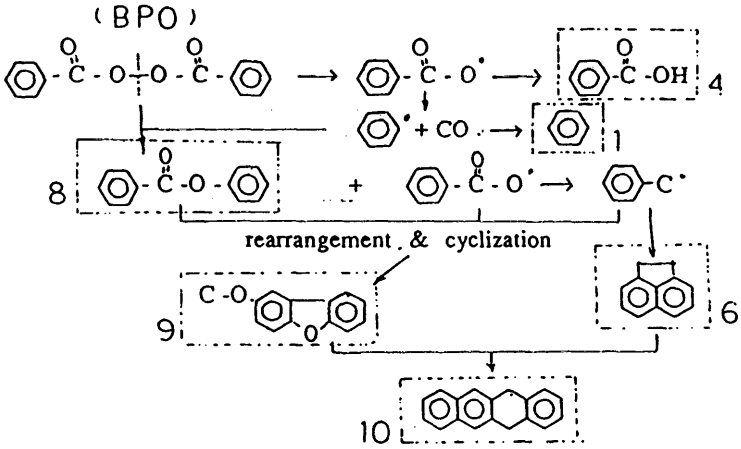


Fig.3 Recycle HPLC Chromatogram of Sample A.





1 Benzene

4 Benzoic acid

6 Acenaphthene

8 Phenyl benzoate

9 Methoxydibenzofuran

10 Dihydronaphthacene

◇ 12 C_{16} aliphatic alcohol C_{15}-C-OH

Fig.4 TIC Pyrogram of HPLC Fraction F-2

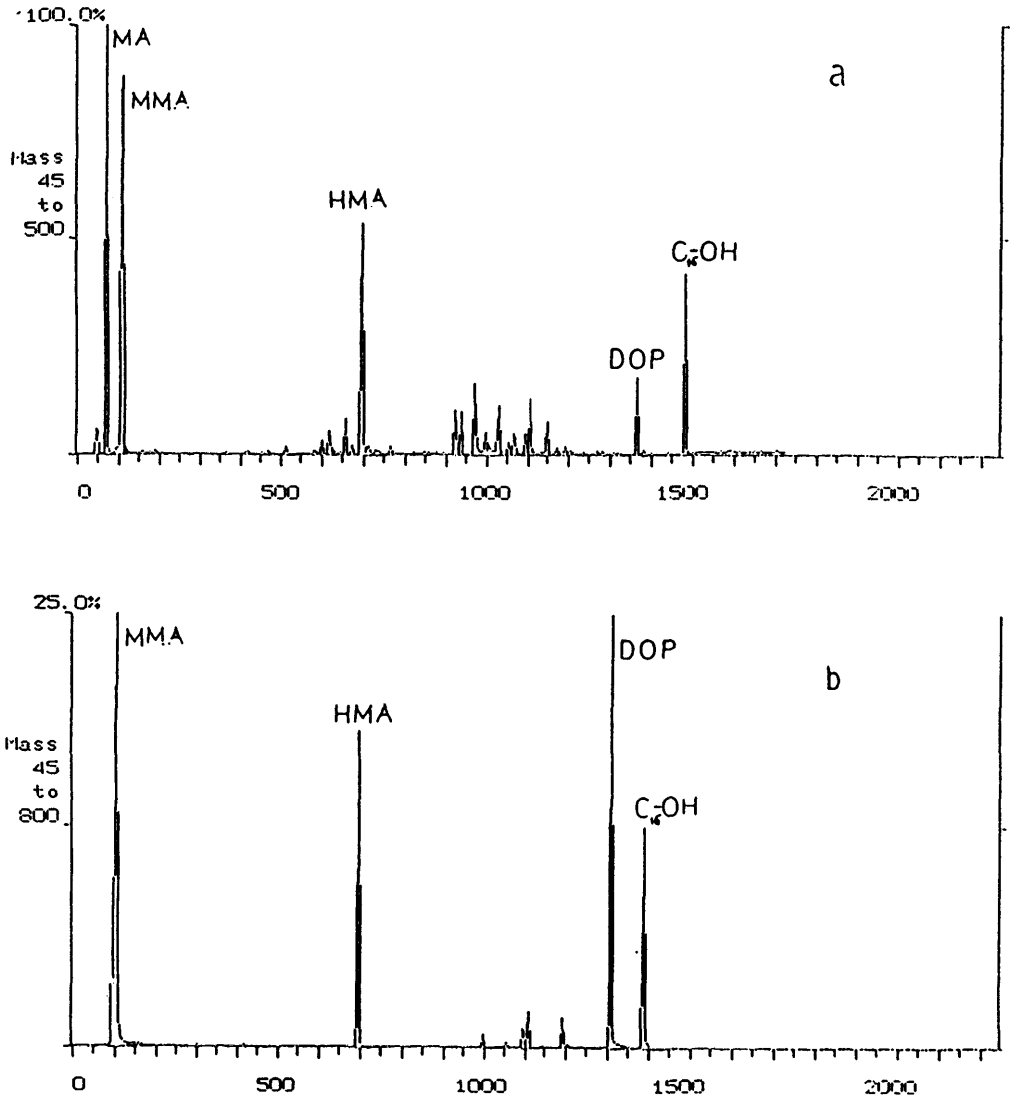


Fig. 5 TIC Pyrogram (a) and Headspace Chromatogram (b) of Sample B.

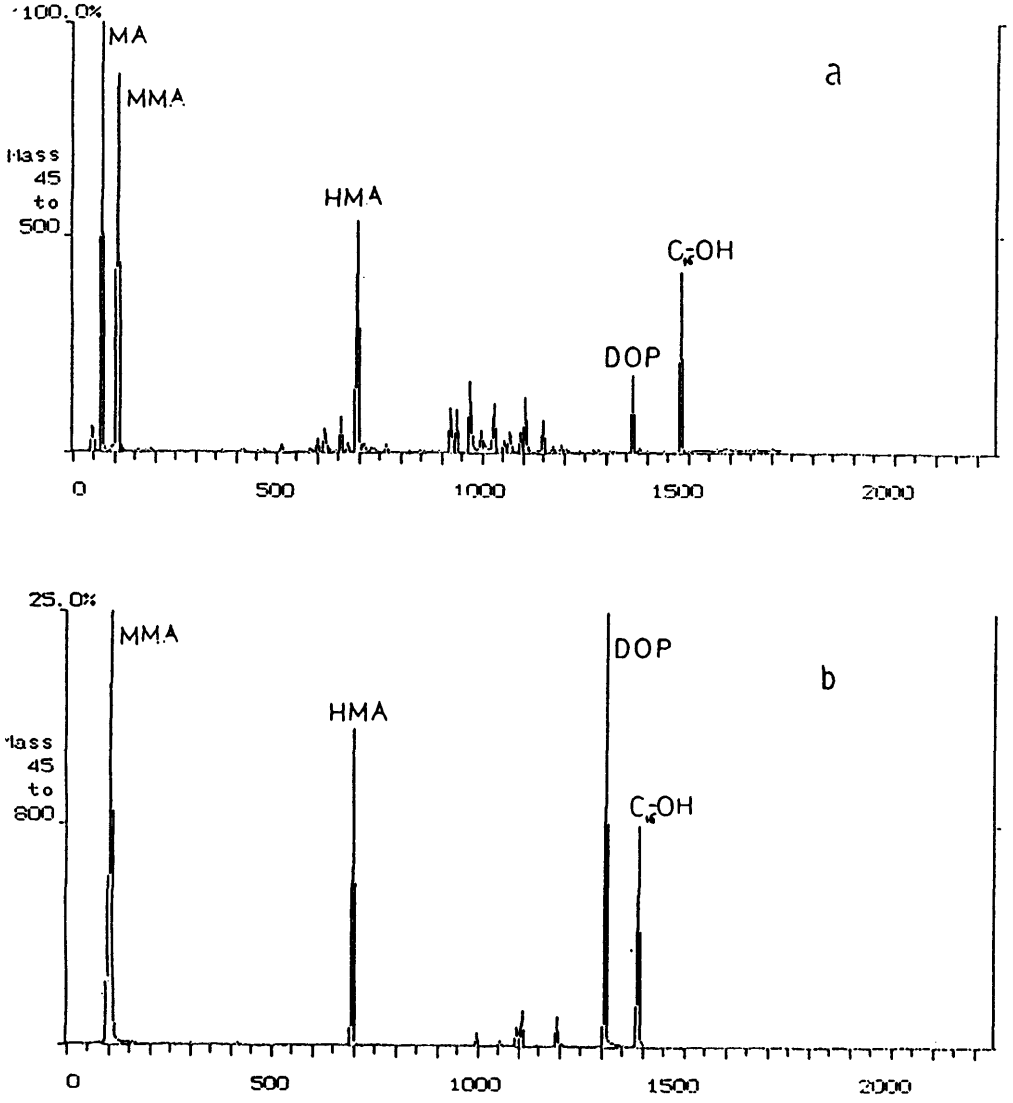


Fig. 5 TIC Pyrogram (a) and Headspace Chromatogram (b) of Sample B.