

Solving Analytical Problems

using Multi-functional Pyrolyzer®

Version 1.0

Functional materials

- Adhesives
- Dyes
- Functional polymers
- Inks
- Paints
- Pigments
- etc.

Introduction to Frontier Laboratories

In 1991, Frontier Laboratories introduced the first commercially available pyrolyzer based on a patented vertical micro-furnace. Since then, a number of innovative accessories have been developed which extend the capabilities of the system beyond simple pyrolysis. Today, the Frontier system can be configured to perform evolved gas analysis (EGA), multi-step thermal desorption and pyrolysis (TD/Py), reactive pyrolysis, and heart-cutting of individual EGA fractions...all automatically! A complete system enables the analyst to fully characterize virtually any complex sample.

The Frontier Multi-functional Pyrolyzer® is used to solve analytical problems – problems that occur in production, problems caused by product failure, problems with instrument contamination, problems with challenging sample preparation regimes, or problems that arise when faced with superior competitive products. These are just few of the areas which a Frontier Multi-functional Pyrolyzer® will prove to be the most cost effective means of responding to the everyday challenges encountered in the modern analytical laboratory.

This monograph presents several examples where the Frontier system is used to provide chemical characterization of a diverse set of sample matrices. In most instances, the “sample” is analyzed directly, which not only saves time but eliminates the inherent errors associated with conventional sample preparation techniques such as solvent extraction, derivatization, etc.

If you desire more information about any of the solutions presented or should you find yourself with a difficult analytical problem similar to these presented, your best source of information is the Frontier web site: www.frontier-lab.com.



All information subject to change without notice.

Analytical problems and Frontier-Based Solutions

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Problem: Is there an example showing the composition of adhesives?

Analysis: The EGA-MS library search is a combination of Evolved Gas Analysis, a thermal analysis technique using Double-Shot Pyrolyzer, and mass spectrometry; and is very useful as a primary searching technique for unknowns.

Result: An example shown is analysis of an adhesive with unknown composition. Shown in Fig. 1 are the EGA curve of an adhesive and its averaged spectra obtained from zones A, B, and C with the background (BG) subtracted. Peak A was considered to arise from a low boiling component by its elution temperature, and was found to be a compound shown in Fig. 1 (a) by a normal MS library search (Wiley275). Wiley275 library search was also performed on peak B, and found mainly to be of acetic acid. Table 1 shows library search results by EGA-MS LIB with F-Search performed on peak C, and various vinyl polymers were found. Because peak B contains acetic acid, the material should contain vinyl acetate. As shown in this example, EGA-MS technique and library search with EGA-MS LIB are extremely useful as a primary library search technique.

Ref: Multi-functional Pyrolyzer® Technical Note, PYA1-013E

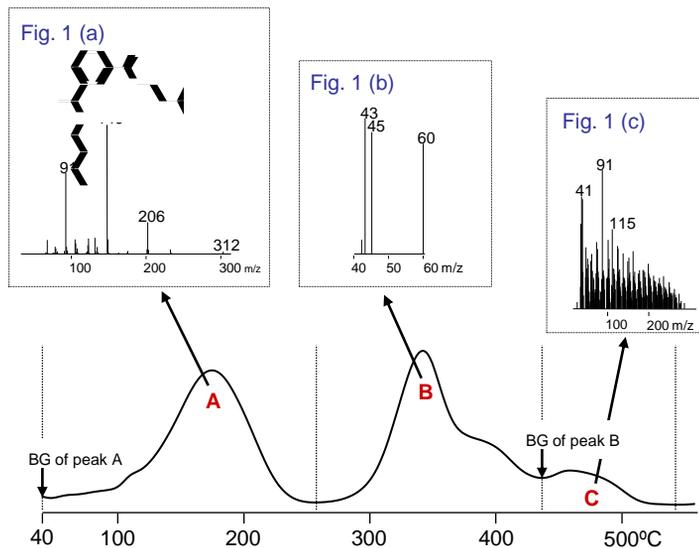


Fig. 1 EGA Curve of the Adhesive

Pyrolyzer furnace temp.: 100°C–600°C (20°C/min), Carrier gas: He 50kPa, Split ratio: ca. 1/50
 EGA capillary tube: 0.15mm id, 2.5 m (UADTM-2.5N), GC oven temp.: 300°C isothermal
 Injection temp.: 320°C, Amt. of sample : ca. 1.0mg, Detector : MS (m/z=29-400, 0.1scan/sec
 PY-GC interface temp.: 320°C (AUTO mode)

Table 1: Library Search Result of Peak C

Name	Qual.(%)
1. Poly (vinyl chloride) ; PVC:	62
2. Poly (vinyl acetate) ; PVAc	60
3. Poly (vinyl alcohol) ; PVA	43

Problem: How can polysiloxanes be quantitatively determined?

Analysis: Samples are pyrolyzed under nitrogen atmosphere. Pyrolyzates are separated and analyzed by Py-GC/MS.

Result: Fig. 1 shows typical pyrograms of dimethyl/methylphenyl substituted polysiloxanes. Table 1 lists component ratios of various siloxane copolymers obtained from pyrograms together with reference components. It is found that expected components are in a good agreement with nominal ones. This result demonstrates that component analysis of siloxane copolymers can be accomplished with a high precision by use of Py-GC/MS technique.

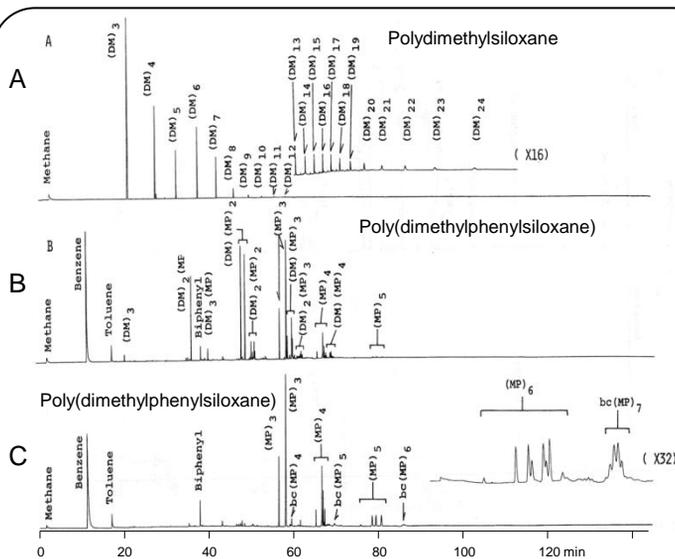


Fig. 1 Pyrograms of dimethyl/methylphenyl substituted polysiloxane

Table 1 Nominal and expected components of siloxane copolymers

Sample	Nominal component mol%	Expected component mol%
OV-3	DM:MP = 80:20	DM:MP = 82.5:17.5
OV-7	DM:MP = 60:40	DM:MP = 61.8:38.2
OV-11	DM:MP = 30:70	DM:MP = 31.1:68.9
OV-25	MP:DP = 50:50	MP:DP = 46.5:52.5
SE-52	DM:DP = 95: 5	DM:DP = 94.9: 5.1
XE-60	DM:MC = 50:50	DM:MC = 45.3:54.7

DM: dimethylsiloxane, MP: methylphenylsiloxane
DP: diphenylsiloxane, MC: 2-cyanoethylmethylsiloxane

Problem: How can the structural characterization of hydrogenated acrylonitrile-butadiene rubbers (NBRs) be done using Py-GC?

Analysis: Hydrogenated NBRs were prepared from NBR and Pd catalyst. Py-GC system had a vertical micro-furnace and was directly attached to a GC with an FID. About 70 μg each of samples was pyrolyzed at 550°C. The identifications of peaks were done by a directly coupled GC-MS.

Result: Fig. 1 shows the pyrograms of NBR samples at 550°C before and after the hydrogenation that were obtained using a capillary column with poly(dimethylsiloxane) stationary phase. Characteristic peaks in the pyrogram of N-37(0) were butadiene (BD) monomer, BD dimer, and acrylonitrile (AN) monomer; whereas those of hydrogenated NBR consisted of a series of linear mononitriles (MN(A)s) up to C_{12} , each of which consisted of a doublet corresponding to an α -olefinic MN(A) (the former) and a saturated MN(A) (the latter). Another series of mononitrile positional isomers (MN(B)s) are also observed. HC peaks of each carbon number consisted of a triplet corresponding to an α,ω -diolefin, α -olefin, and a n-alkane.

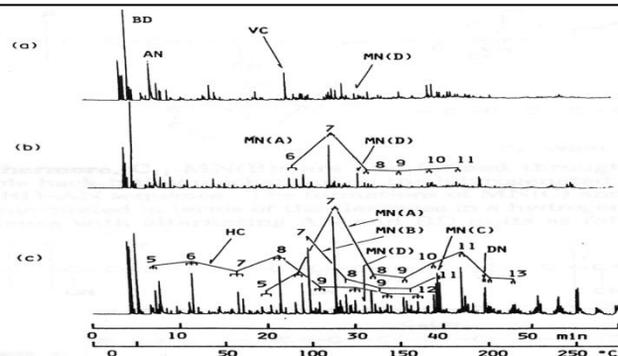


Fig. 1 Pyrograms of NBRs before and after hydrogenation at 550°C separated by a poly(dimethylsiloxane) column: (a) N-37(0); (b) N-37(44); (c) N-37(98). See Table 1 for abbreviations.

Table 1 Characteristic Degradation Products from Hydrogenated NBR

Compound class	Abbreviation	Sequence
Butadiene	BD	B
Butadiene dimer (4vinylcyclohexane)	VC	BB
Acrylonitrile	AN	A
Hydrocarbons	HC	EE EEE
Mononitriles	MN(A)	EA EEEE
	MN(B)	EA EAA
	MN(C)	EAE
	MN(D)	BA
	DN	AEA
Dinitriles	DN	AEA

B = 1,4-butadiene unit; A = acrylonitrile; E = hydrogenated 1,4-butadiene unit

Problem: Is there a simple method to analyze the chemical composition of a blended rubber sample?

Analysis: About 200 µg of a rubber mixture, composed of polybutadiene(PB)- polyisoprene (PI)- polystyrene (PS), is placed in a sample cup and pyrolyzed at 550°C.

Result: Fig.1 shows the pyrogram for the blended rubber sample. The monomers of each component, which are butadiene, isoprene and styrene, are the main pyrolyzates. The calibration curves between relative peak intensities for the specific peaks and the ratio of PB to total weight of the sample shows a fairly good linear relationship with a correlation coefficient greater than 0.99. The calibration curve for the PB composition in the blended sample is shown in Fig.2. Using this calibration curve, a fairly accurate determination of the component is possible within 3% of accuracy.

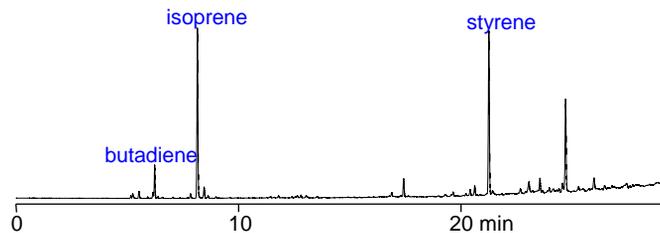


Fig.1 Pyrogram of a blended rubber

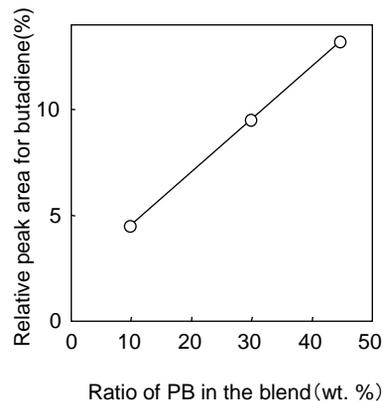


Fig.2 Calibration curve for PB in the blend

Problem: What is the best quantitative method for the analysis of a cationic polymer with a quaternary ammonium salt? Can Py-GC/MS be used?

Analysis: Polyquaternium-4 (PQ-4 in Fig. 1) was used. The EGA-MS system is comprised of a Multi-functional pyrolyzer attached to the injection port of a GC, which was connected to a MS via a deactivated stainless-steel tube. In thermal desorption-MS analysis, a separation column was used instead. Volatile components trapped at the head of the separation column by MicroJet Cryo-Trap was analyzed by GC/MS.

Result: An EGA thermogram for PQ-4 is shown in Fig. 2. As shown in the blowup of 250~300°C region, the formation of chloromethane was observed. Thus, the quantitative analysis of PQ-4 was performed using the amount of chloromethane formed. Fig. 3 shows the chromatogram of the components eluted between 100~340°C, obtained by GC/MS. At the retention time of 1.5 min, a peak due to chloromethane is markedly observed. This was repeated 8 times and the reproducibility of the peak area of chloromethane is shown in Table 1.

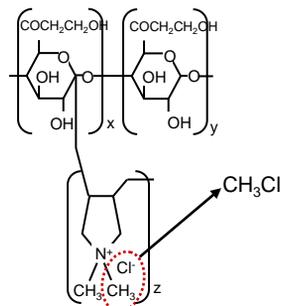


Fig. 1 Structure of PQ-4 & chloromethane formation

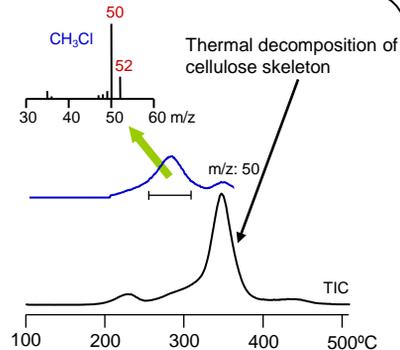


Fig. 2 EGA thermogram of PQ-4

Pyrolyzer furnace temp.: 100-500°C (20°C/min),
Carrier gas : He 50 kPa, split ratio : ca. 1/50
EGA tube : id=0.15 mm, L=2.5 m (UADTM-2.5N),
GC oven temp.: 300°C, Sample: 0.2 mg,
MS scan range : 29-600 (m/z), scan rate: 0.2 scan/sec

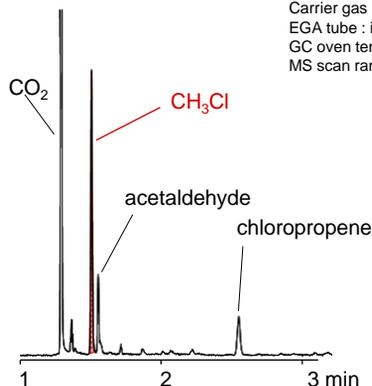


Fig. 3 Chromatogram of PQ-4 obtained by thermal desorption-GC/MS

Table 1 Reproducibility of chloromethane peak area

n	Peak area (x10 ⁵)
1	5.89
2	5.57
3	6.23
4	6.01
5	6.12
6	5.82
7	5.85
8	5.83
Average	5.92
RSD (%)	3.44

Pyrolyzer furnace temp.: 100-340°C (20°C/min, 2min hold), flow rate : 1ml/min, split ratio : 1/50
Separation column : Ultra ALLOY5+ (5% diphenyl 95% dimethylpolysiloxane, length 30m id 0.25mm, film thickness : 1.0µm), GC oven temp.: 40°C (2min hold) - 300°C (20°C/min)
Sample size : 5.64 µg, MS scan range : 29-600 (m/z), scan rate : 2 scan/sec

Problem: How can a quaternary-ammonium-cation polymer added in a polymer be analyzed qualitatively and quantitatively?

Analysis: As an example, analysis of polyquaternium-4 (PQ-4), a quaternary-ammonium salt, coated on polyethylene terephthalate (PET) sheet is described here.

Result: Fig. 1 shows a chromatogram obtained by GC/MS after cryo-trapping thermally desorbed components (100~340°C) from a PET sheet coated with PQ-4. The peak for chloromethane, or a decomposition component of PQ-4, appeared at retention time of ca. 1min. The reproducibility for the peak area of characteristic ion m/z 50 was as good as 3.46% RSD. The calibration curve created from samples of varied concentrations (1~600ppm) gave a good linearity as shown in Fig. 2.

Analytical condition:
 Separation column: Ultra ALLOY+-5 30M-1.0F
 GC oven temperature: 40°C(2min)~20°C/min~300°C

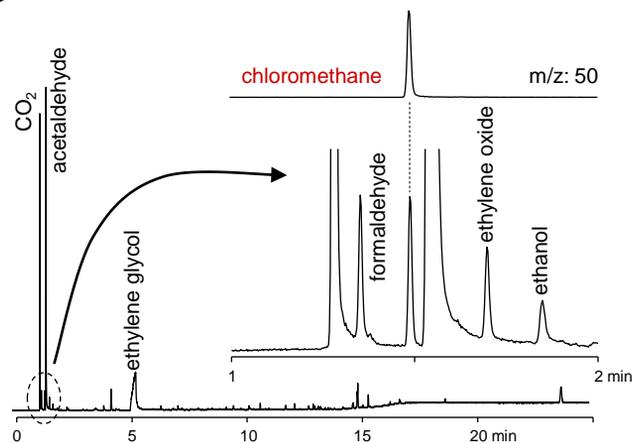


Fig. 1 Chromatogram of PET sheet coated with PQ-4 obtained by thermal desorption-GC/MS

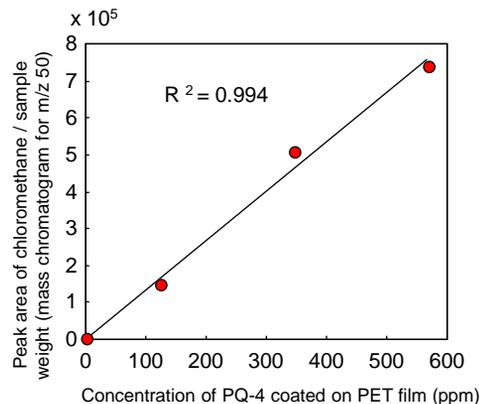


Fig. 2 Calibration curve for PQ-4 on PET film

Problem: Can black ballpoint pen inks be differentiated by Py-GC/MS? If so, please describe in detail.

Analysis: The pyrograms of 35 inks were used to create an “ink” library. Each ink was scribed onto copy paper. The ink sample was pyrolyzed at 550°C. The integration-summation (INT-SUM) mass spectrum was calculated for each pyrogram and used to construct an “ink” MS library. One arbitrarily selected ink was used as unknown, and to test the viability of the library.

Result: Pyrograms of the unknown and four inks stored in the library are shown in Figure 1. The major components of each ink are solvents such as phenoxyethanol and diethyl aniline. All elute before 7 min on the pyrogram. Inks A through D showed similar pyrograms; however, small peaks are observed after 7 min. These are dyes and additives and differ from ink to ink. It is difficult to differentiate the inks using only the INT-SUM* mass spectra (Fig. 2(a)), because of the presence of ions m/z 77, 94, and 138 which are from the phenoxyethanol. Thus, a second INT-SUM mass spectra was created from components eluting after 7 min (Fig. 2 (b)). Now, significant differences in match quality are observed and the unknown ink can be easily identified as Ink C.

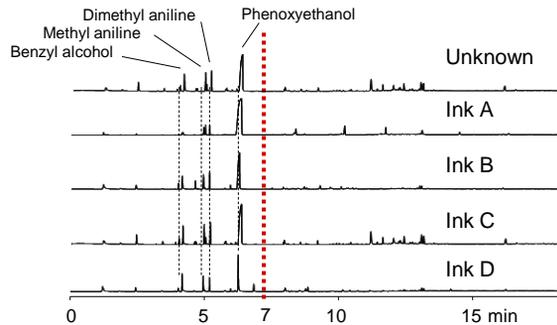
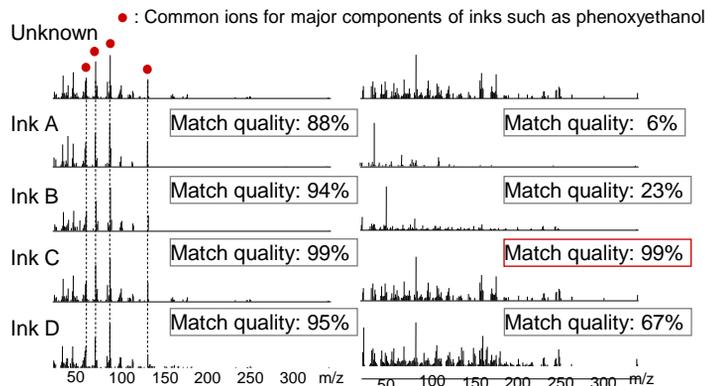


Fig. 1 Pyrograms of oil-based black ballpoint pen inks

Pyrolysis temp.: 550°C, GC oven: 40 - 300°C (20°C/min), Separation column: Ultra ALLOY*+5 (5% diphenyl 95% dimethylpolysiloxane, L=30 m, i.d.=0.25 mm, df=0.25 μ m), Carrier gas: 1 mL/min, He, Split ratio: 1/50, sample: ca. 200 μ g



(a) Mass spectra created from entire pyrogram (0-20 min) (b) Mass spectra created from pyrograms of components eluting after 7 min.

Fig. 2 Comparison of mass spectra created (a) from entire pyrogram and (b) from components eluting after 7 min.

Problem: Is there a technique to obtain information about the sequence distributions of polyacetal (PA)?

Analysis: Multi-component copolymers of PA, containing 1-9 mol% of oxyethylene units [(-OCH₂CH₂-)(E)] against the main chain oxymethylene units [(-OCH₂-)(F)], are analyzed. The PA sample combined with solid cobalt sulfate (CoSO₄•7H₂O) catalyst is frozen and pulverized. About 100 µg of the powder sample is placed in the sample cup prior to reactive pyrolysis GC/MS at 400°C.

Result: The pyrogram from the reactive pyrolysis GC/MS for the PA sample is shown in Fig.1. A series of cyclic ethers consisting of E and F units are observed. This reflects the sequence structures of E units in the original polymer chain. From the relative peak intensity of these cyclic ethers, it is possible to estimate the E unit content in the polymer sample as well as the sequence distributions.

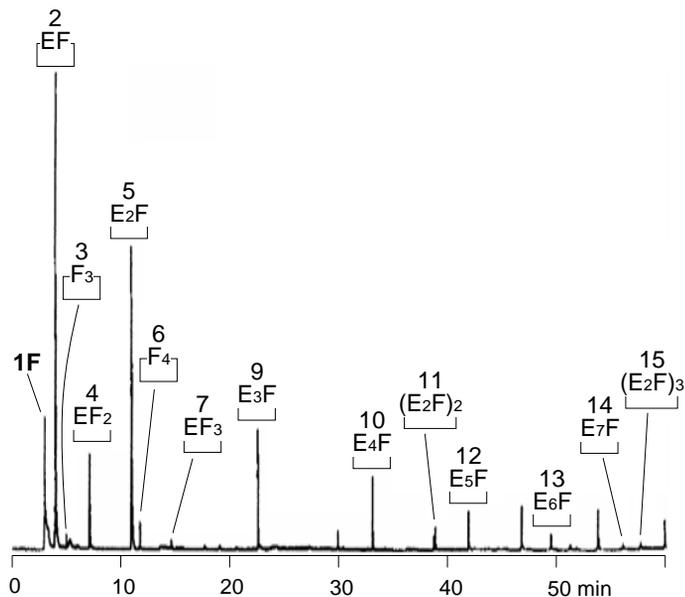


Fig. 1 Pyrogram of copolyacetal by reactive pyrolysis

Problem: How is hindered-amine light stabilizer (HALS) in polypropylene (PP) analyzed?

Analysis: Adekastab LA-68LD [MW=1900] as a high molecular weight HALS, and Irganox 1010 and irgafos 168 as antioxidants were added to a PP polymer sample. Then TMAH is added and the PP is analyzed by reactive thermal desorption GC (RTD-GC).

Result: Fig. 1 shows a typical chromatogram of a PP sample containing 10,000 ppm of HALS obtained by RTD-GC in the presence of TMAH at 300°C. On this chromatogram, the reaction products originated from piperidine (peaks 3 and 4) and spiro ring (peaks 5 and 6) moieties in the original HALS molecule were clearly observed without interferences from pyrolyzates of the substrate PP polymer backbone. The precision was as good as 5% RSD or less.

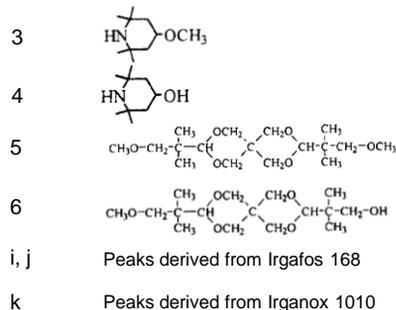
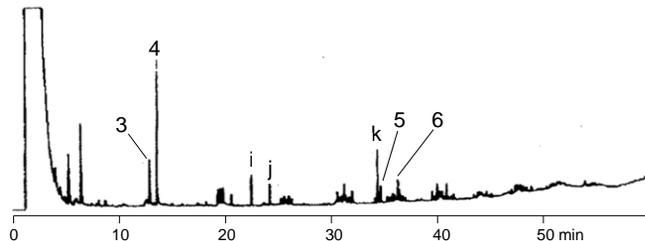
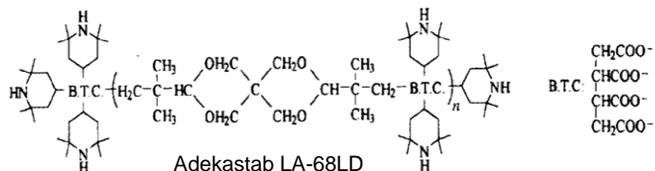
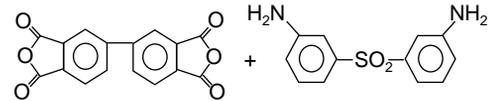


Fig. 1 Typical pyrogram of a polypropylene sample obtained by RTD-GC

Problem: Is it possible to analyze compounds out-gassing during the curing of polyimide?

Analysis: Microgram quantities of the sample containing bisphenyl tetracarboxylic acid di-anhydride (BPDA) and sulfonyl bisbenzamine (3,3-DDS) are placed in a sample cup. EGA is performed during the thermal polymerization of the mixture.?

Result: Fig.1 shows that polyimide is synthesized when the mixture is heated using a two step process. Fig.2 presents the thermograms for various out-gassing compounds formed during the preparation of polyimide. This result demonstrates that DMAA* and CO₂ are formed during the first heating process and SO₂ and aniline are generated during the second heating process.



(BPDA)

(3,3-DDS)

The first heating process
150°C(30min)

The second heating process
120°C~450°C(3°C/min)

→ Polyamic acid → Polyimide is formed
→ Out-gases are evolved

Fig.1 The synthetic process of polyimide

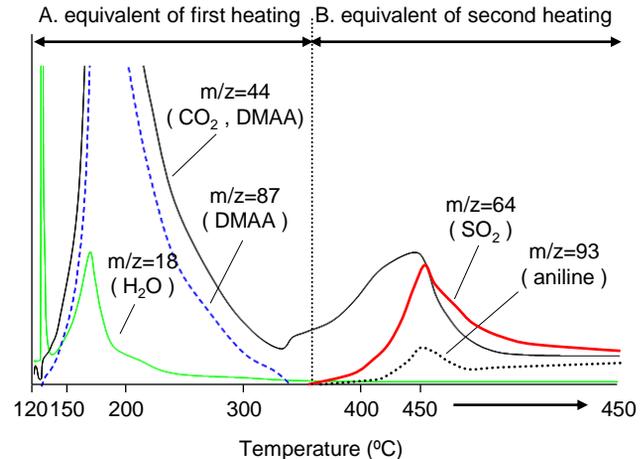


Fig. 2 Thermogram with mass chromatogram in the process of polyimide synthesis

Problem: Is there an example of thermal degradation study of flame retarded PBT using Py-GC/MS system?

Analysis: Flame-retarded polybutyleneterephthalate (PBT) consisted of PBT, brominated polycarbonate, Sb₂O₃ (80/15/5wt%). All the polymer samples were cryo-milled into a fine powder at liquid N₂ temperature. The pyrolyzer was connected to a MS via a capillary separation column. During programmed heating of the furnace (60–700°C at 10°C/min), thermal degradation products were cryo-trapped at the head of the separation column. Upon removing the trap, the GC/MS analysis was performed (35–300°C at 10°C/min).

Result: TIC chromatograms of products from (a) PBT, (b) Br-PC, (c) FR-PBT, and (d) mass chromatogram for m/z362 (SbBr₃). The chromatogram for FR-PBT in Fig. 3(c) shows components observed in both PBT and Br-PC together with additional peaks (14 and 18) due to Br-PBT derivatives. It was thus shown that the technique should be very useful to obtain information on the synergistic flame-retardancy of halogenated organic compounds/Sb₂O₃ system.

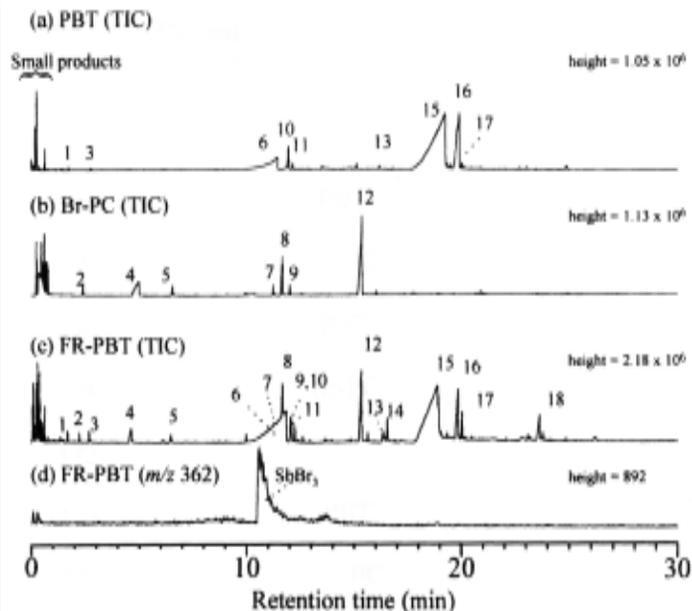


Fig. 1 Typical gas chromatograms of the degradation products from (a) PBT, (b) Br-PC, (c) FR-PBT, and (d) mass chromatogram for m/z362 (parent ion of SbBr₃).

Problem: Has characterization of chitin-based polymer hybrids been done by EGA and EGA-MS?

Analysis: Chitin-graft-poly(2-methyl-2-oxazoline) was prepared, where living poly(2-methyl-2-oxazoline) side chains (polymerization=19.6%, Mw/Mn=1.1) was selectively grafted onto free amino groups of the partially N-deacetylated chitin (acetylation=52%). For the EGA-MS system used was a pyrolyzer attached to the injector of a GC was directly coupled with a quadrupole MS via a deactivated stainless steel capillary tube.

Result: Figure 1 shows thermograms of the chitin derivative/PVA blends obtained by EGA-MS as function of temperature together with those for the chitin derivative and PVA. The TIC curve of PVA shows two-stage degradation. The thermal degradation of blends also occurs in two stages. The degradation products were identified by EGA-GC/MS. Figure 2 shows the TIC of (a) the products eluted during the first stage and (b) during the second stage for the B (60/40) blend sample. The major products such as water and various unsaturated and aromatic aldehydes may be formed during the first degradation stage through dehydration of PVA followed by scission of the resulting polyene chains (Fig. 2a). On the other hand, various degradation products originating from chitin derivatives are formed during the second degradation stage (Fig. 2b).

Ref: Multi-functional Pyrolyzer® Technical Note, PYA3-008E

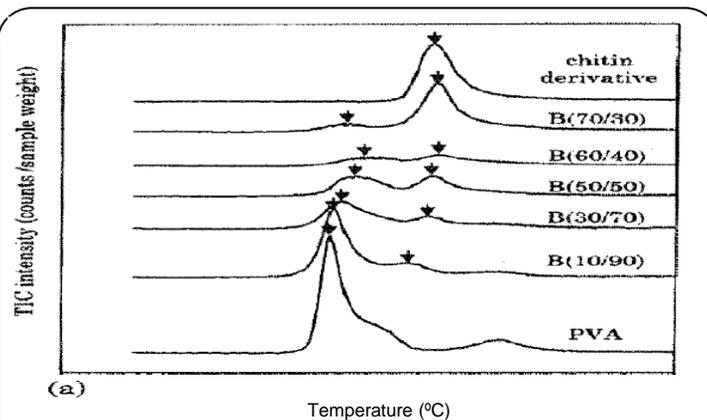


Fig. 1 Thermograms of the thermal degradation products from the chitin derivative/PVA blend samples observed in TIC by EGA-MS

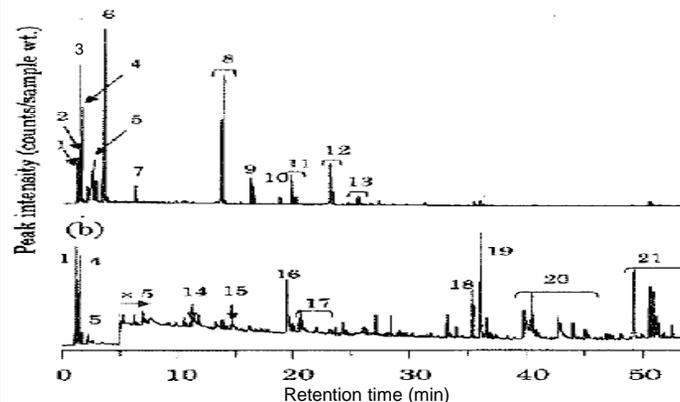


Fig. 2 TIC chromatograms of degradation products formed from the B(60/40) blend during heating ranges (a) 240-340°C and (b) 340-480°C

Problem: How can volatiles from a UV curable resin be analyzed?

Analysis: A dry film was analyzed by ultraviolet light (UV) irradiation/pyrolysis-GC/MS technique. The dry film was an acrylic UV curable resin. A small disc sample was used for this experiment. The UV irradiation was conducted in He at 60 °C for 10 min using a micro UV irradiator (UV-1047Xe). During UV irradiation, volatiles evolved from the sample was cryo-trapped at the head of a separation column. Then GC/MS analysis was conducted. Volatiles analysis under an identical condition but no UV irradiation was also made.

Result: Chromatograms of volatile components released from the resin with and without UV irradiation and their magnified view are shown in Fig. 1. Without UV irradiation, decomposed polymerization initiator was only observed, on the other hand, upon UV irradiation various organic compounds including 300 ppm of methyl methacrylate (MMA) against the original sample weight were observed. As shown here, the UV/Py-GC/MS technique allows you to analyze volatiles released from a UV curable resin during its curing process without complex treatments.

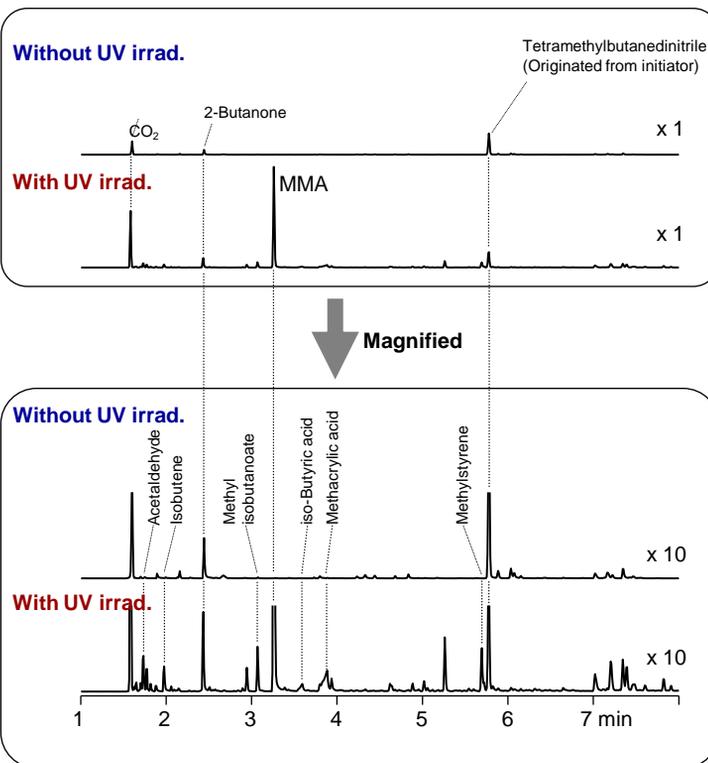


Fig. 1 Analysis of volatiles released from UV curable acrylic dry film

Micro UV irradiator: UV-1047Xe, separation column: Ultra ALLOY+1 (dimethylpolysiloxane L=30 m, id=0.25 mm, df=0.5 μ m), atmosphere gas: He, Column flow rate: 1 ml/min, split ratio: 1/10 sample size: 350 μ g (3 mm diameter disc), GC oven temp: 40 ~ 300 °C (20 °C/min)

Problem: Is there any simple way to analyze volatile degradation products released from high impact polyethylene (HIPS) during its photo-/thermal/oxidative degradation?

Analysis: Online ultraviolet(UV)/pyrolysis(Py)-GC/MS system can be used to analyze the volatiles. It has a micro UV irradiator equipped with a xenon (Xe) lamp. 10 μ l of dichloromethane solution (2 mg/ml) of HIPS was placed in a sample cup. The sample was irradiated for 1 hr at 60°C under air stream. Volatiles released were cryo-trapped at the head of a separation column. Upon UV irradiation, to drive the volatile products into the column, the sample was heated to 250°C (in He). After the cryo-trap was removed, GC/MS analysis was then started.

Result: Fig. 1 shows the chromatograms of volatiles from HIPS with and without UV irradiation. Upon 1 hour UV irradiation, volatile degradation products originated from PS were observed in addition to 2-propenal. The observation of 2-propenal indicates that a volatile degradation product was indeed generated from butadiene rubber contained in HIPS.

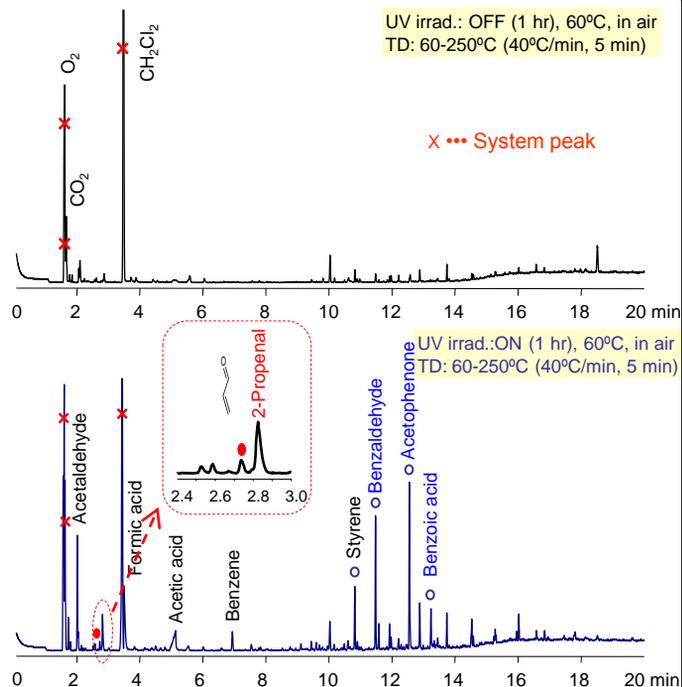


Fig. 1 Chromatograms of volatile degradation products from HIPS generated during UV irradiation obtained by UV/Py-GC/MS

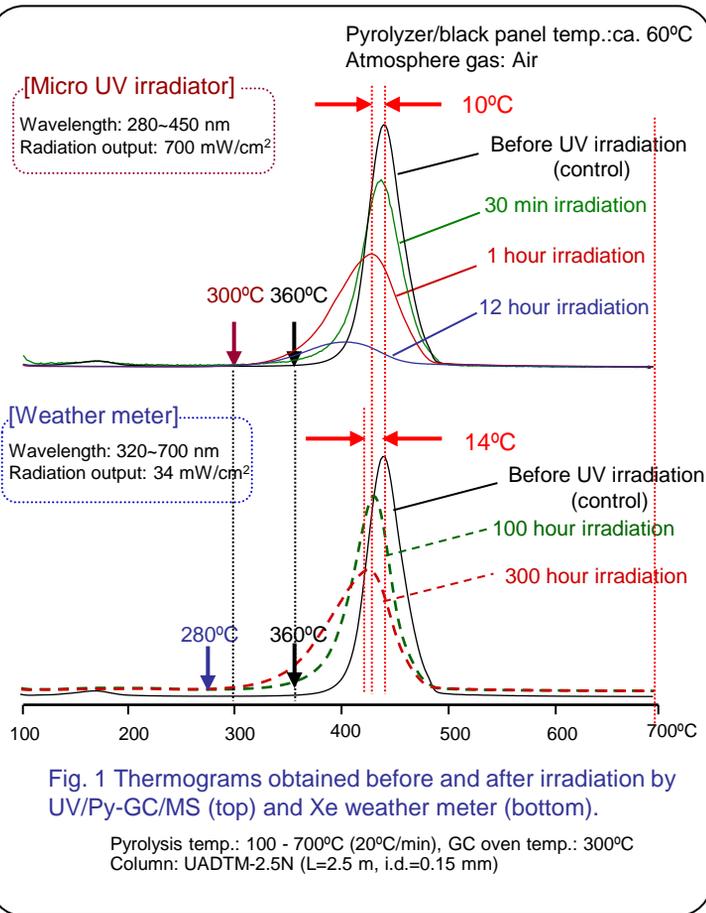
<UV irradiation> Micro UV irradiator : UV-1047Xe, irradiation: 1 hr, furnace temp.: 60°C, 10 ml/min Air, <GC/MS> Separation column: Ultra ALLOY*-1 (polydimethylsiloxane, L=30 m, i.d.=0.25 mm, df=0.5 μ m), GC oven temp.: 40°C(5 min)-240°C(20°C/min), GC injection temp.: 250°C

Problem: Have you found any correlation between micro UV irradiator (Xe lamp) and Xe weather meter in the degradation study of high impact polyethylene (HIPS)?

Analysis: The correlation between the two was investigated by evolved gas analysis (EGA)-MS. HIPS samples were irradiated for 30 min, 1 hour, and 12 hours by the micro UV irradiator at 60°C in air stream, then EGA was performed. In the Xe weather meter experiment, after irradiation of a HIPS plate for 100 or 300 hours, the surface was scraped off and analyzed by EGA.

Result: HIPS sample irradiated for 1 hour by the UV irradiator shows the peak top lowered by 10°C, and the pyrolysis onset temperature lowered from 360 to 300°C. Further, the peak became broader. Also size-exclusion chromatography revealed the molecular weight lowered from 285,000 to 240,000, indicating degradation of the polymer main chain. Also, HIPS sample irradiated for 300 hours by Xe-weather meter shows the peak top lowered by 14°C and the pyrolysis onset temperature lowered from 360 to 280°C. Since both thermograms are almost identical in shape, there is a good correlation between the two.

Ref: Multi-functional Pyrolyzer® Technical Note, PYA5-004E



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Flexible: Multiple operating modes; evolved gas analysis (EGA), thermal desorption (TD) pyrolysis (Py), Heart-Cut method, library search (polymers, additives, pyrolyzates)

Reproducible: Guaranteed

Simple operation: A single computer screen for each operating mode

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Multi-Shot Pyrolyzer EGA/PY-3030D



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