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Pyrolyzer-GC/MS를 이용한 플라스틱 및 고무 재료의 열분해 특성 분석 솔루션

2023. 02. 23 (목) PM 14:00

영인과학 마케팅팀

영인과학
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01

Py-GC/MS의 다양한 응용

고분자 화학
POLYMER CHEMISTRY

미량 첨가제
ADDITIVES

프탈레이트
PHTHALATE ESTERS

미세플라스틱
MICROPLASTICS

코팅 & 접착제
COTINGS & ADHESIVES

고무 및 탄성중합체
RUBBER & ELASTOMERS

바이오 에너지
BIOMASS & BIOENERGY

잉크 및 페인트
INK & PAINTS

종이 및 섬유
PAPER & FIBERS

과학수사
FORENSIC

* Frontier Lab website: https://www.frontier-lab.com/technical-information/technical-note/

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02

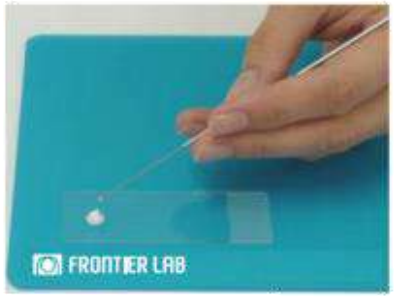
Py-GC/MS 시료 전처리

Micro Sample Collector

Collects small amount of powder or liquid sample.

Sampling needle

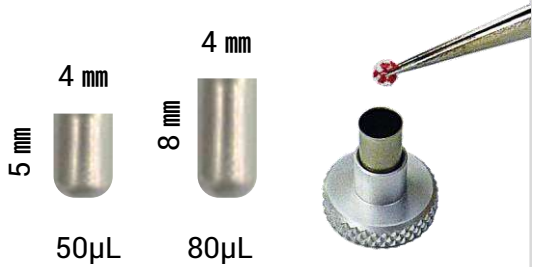
Collect samples of viscous liquids using the tip of the needle.



Double head spatula

Gather powdered sample.

Sample cups *(Eco-cup)*

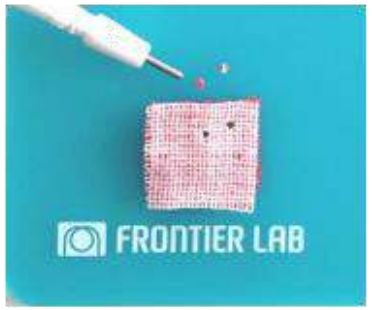


Scissors

Cut a sheet or film-like sample into smaller sizes.

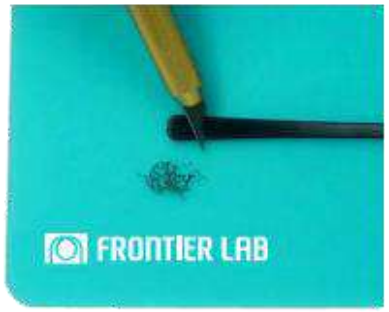
Micro-puncher

Punch out film like sample into small discs.



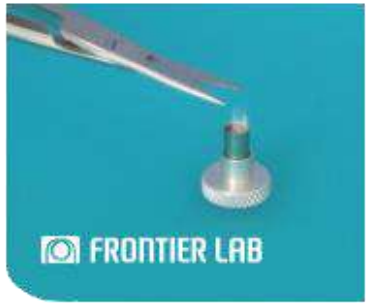
Cutter knife

Scrape off sample surface.



Precision Scissors

Cut quartz wools protruding from sample cup (Eco-cup).



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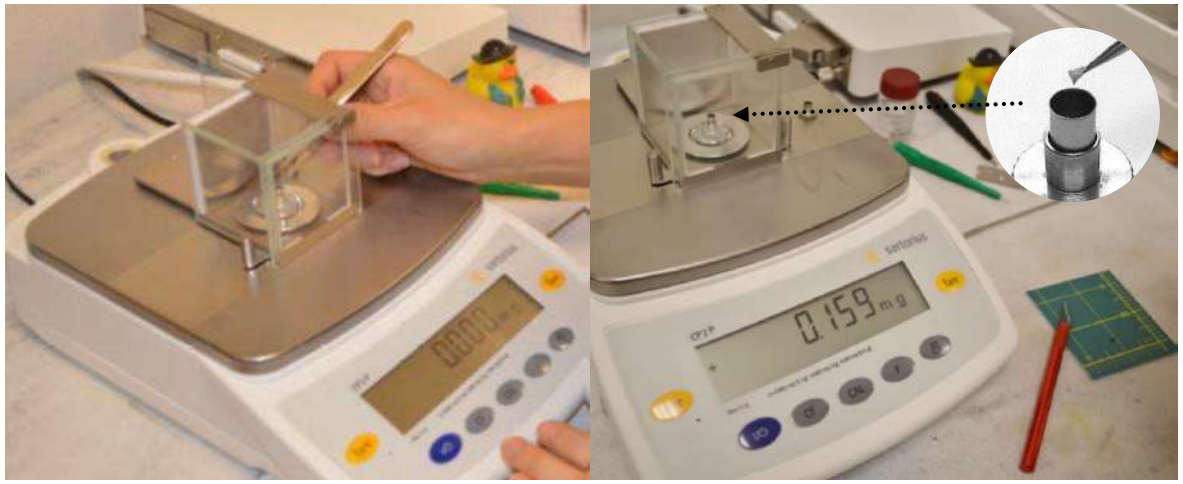
02

Py-GC/MS 시료 전처리

샘플컵 (Eco-cups)

저소음 동결 분쇄기 (IQ MILL-2070)

미량 분석 저울 (Microbalance)



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02

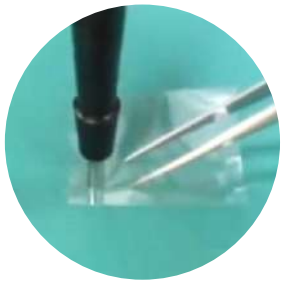
Py-GC/MS 시료 전처리

STEP 01

Sample Preparation

Solid samples
Using a cutter knife

Solid samples
Using a cryogenic mill



Film samples
Using Micro-puncher

Liquid sample
Using a micro syringe

STEP 02

Weighing Sample

Sampling
Placed in a sample cup

Weighing Samples
Using a micro balance

STEP 03

Placed the Sample on to Pyrolyzer

Inject directly
Without solvent extraction

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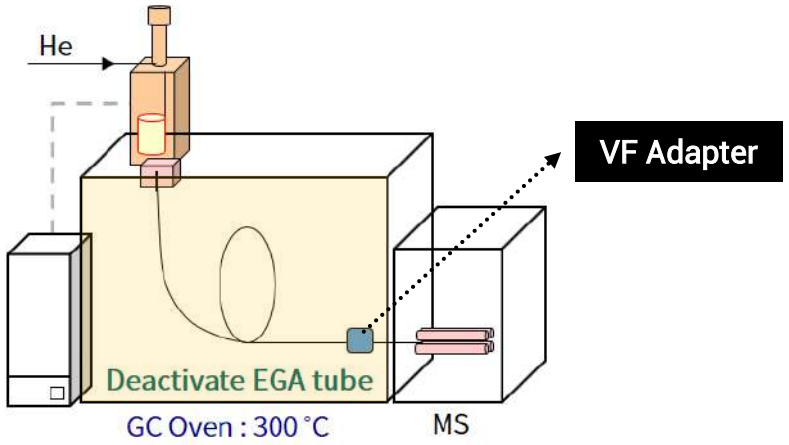
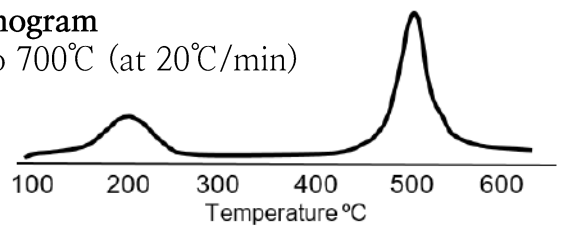
03

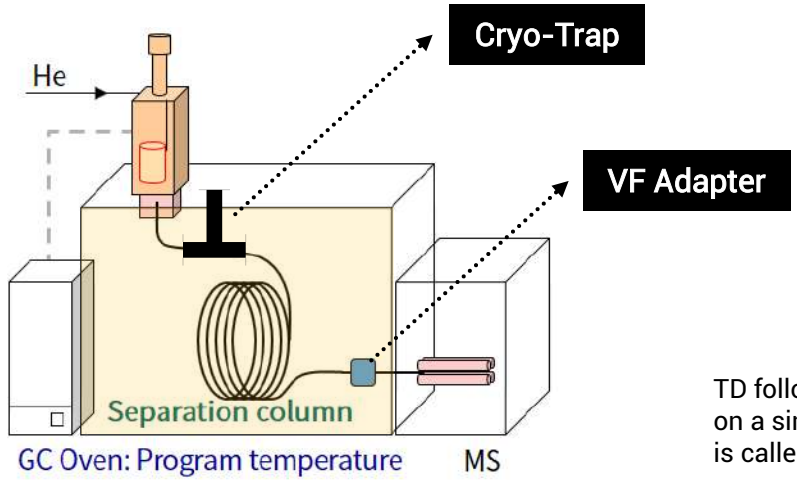
Py-GC/MS 분석 시스템
EGA-MS vs. TD/PY-GC/MS



EGA-MS

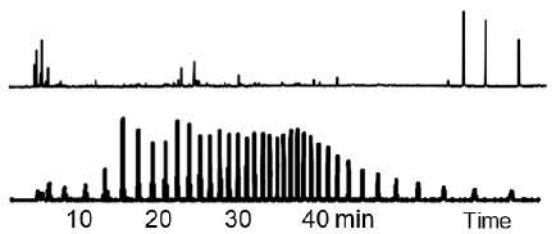
Thermogram
100 to 700℃ (at 20℃/min)





TD-GC/MS & PY-GC/MS

TD followed by PY
on a single sample
is called a Double-Shot.



TD Chromatogram
100 to 300℃ (at 20℃/min)

Pyrogram (Py-GC/MS)
600℃

- **EGA-MS**: 시료의 전반적인 열적 특성정보 확인을 위한 첫 번째 분석 단계. EGA metal tube(2.5m x 0.15mm i.d.)를 이용해 GC inlet과 MS사이에 연결.
- **TD-GC/MS 및 Py-GC/MS**: EGA Thermogram 정보를 바탕으로 분석 컬럼을 사용하여 수행되는 정밀 분석 단계.
- **Vent-free GC/MS Adapter(VFA)**: MS venting 없이 수분 이내 신속한 EGA tube 및 분석 컬럼 교환 가능

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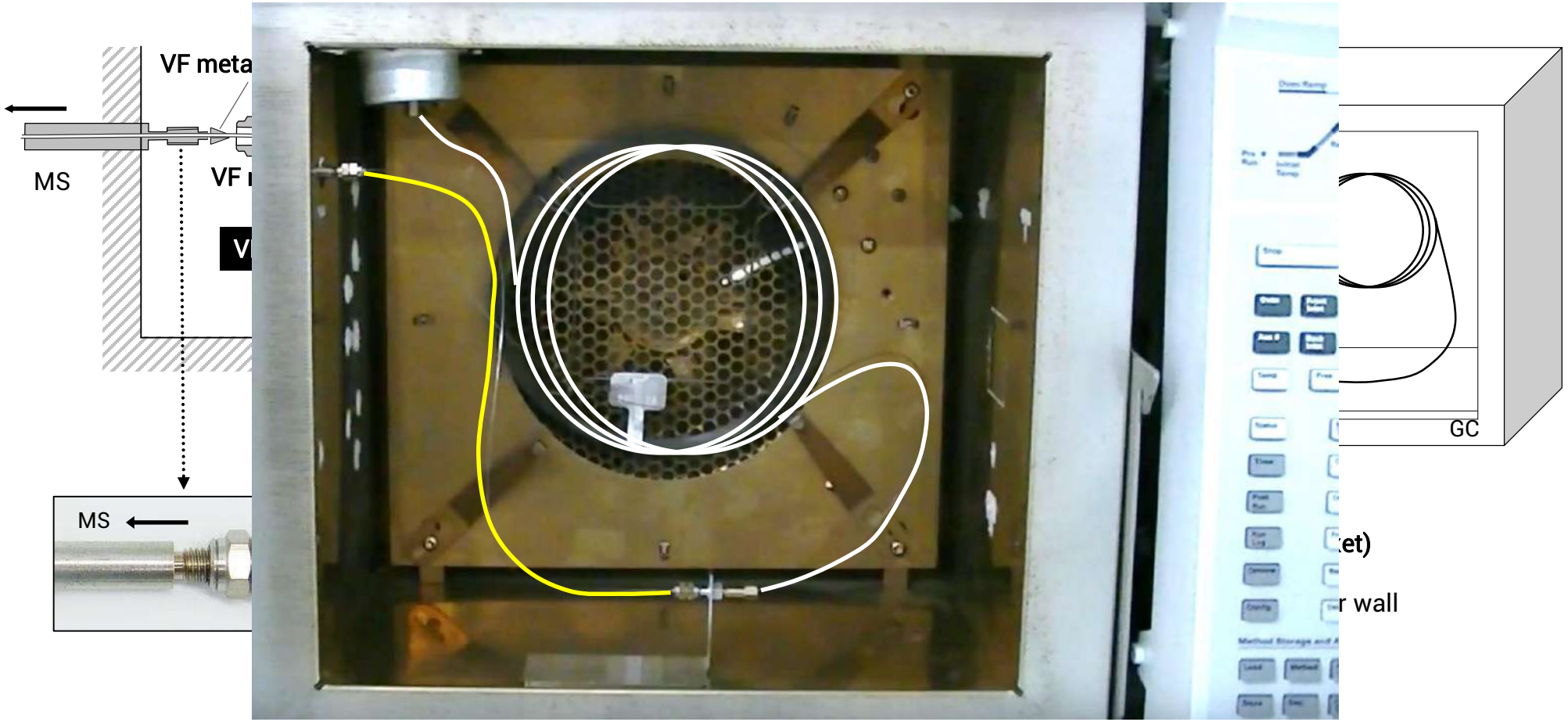
04

Vent-free GC/MS Adapter(VFA)



Vent-free GC/MS adapter(VFA)는 GC/MS 분석에 활용 가능한 유용한 인터페이스 장치입니다. 고도로 비활성화된 모세관 튜브(i.d. 0.15 mm, L 50 cm)를 분석 컬럼과 MS 검출기 사이에 연결하여 MS 검출기가 작동하는 동안에도 GC 컬럼을 신속하게 교체할 수 있습니다.

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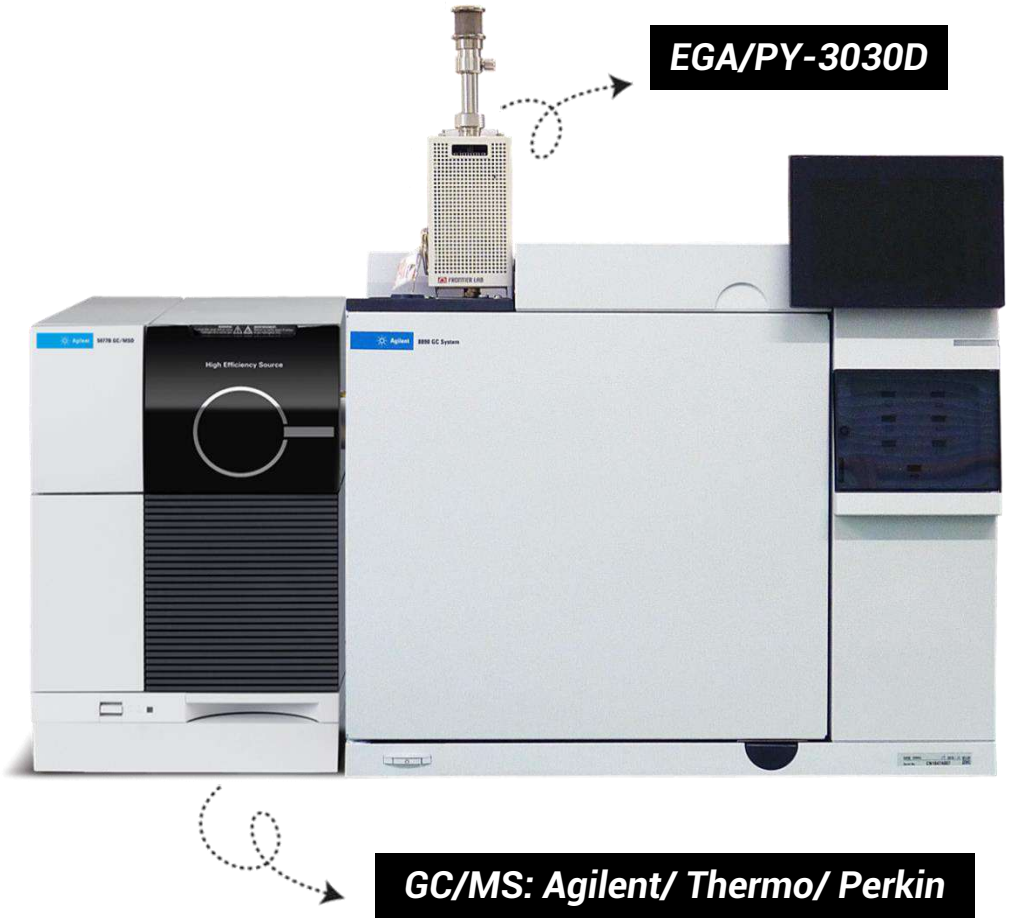
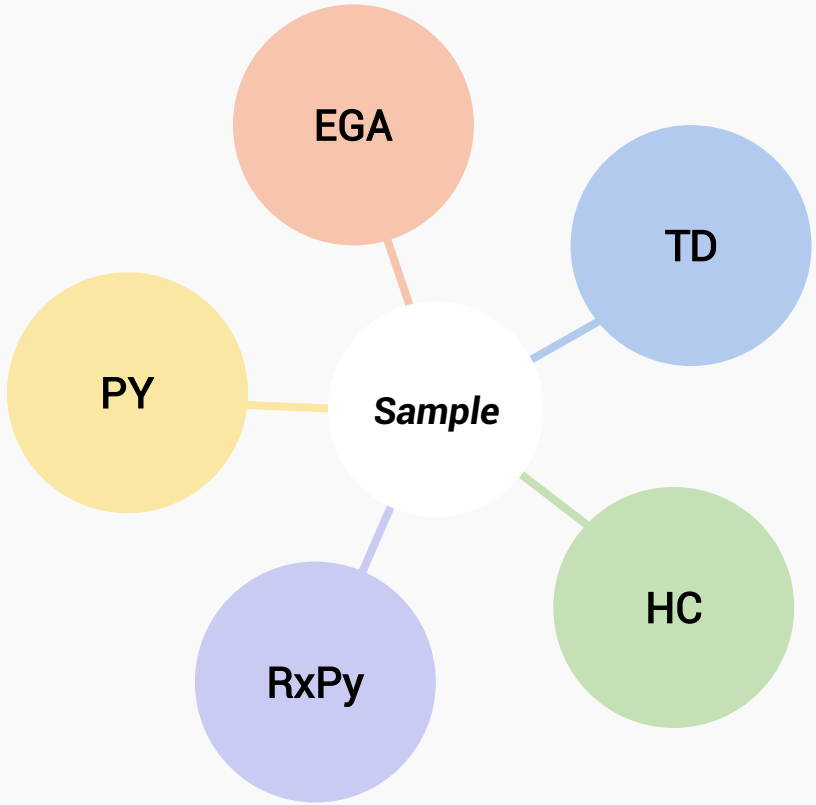
05

멀티샷 파이롤라이저 분석 기법





- **EGA**: Evolved Gas Analysis
- **TD**: Thermal Desorption
- **PY**: Pyrolysis
- **HC**: Heart-Cutting
- **RxPy**: Reactive Pyrolysis

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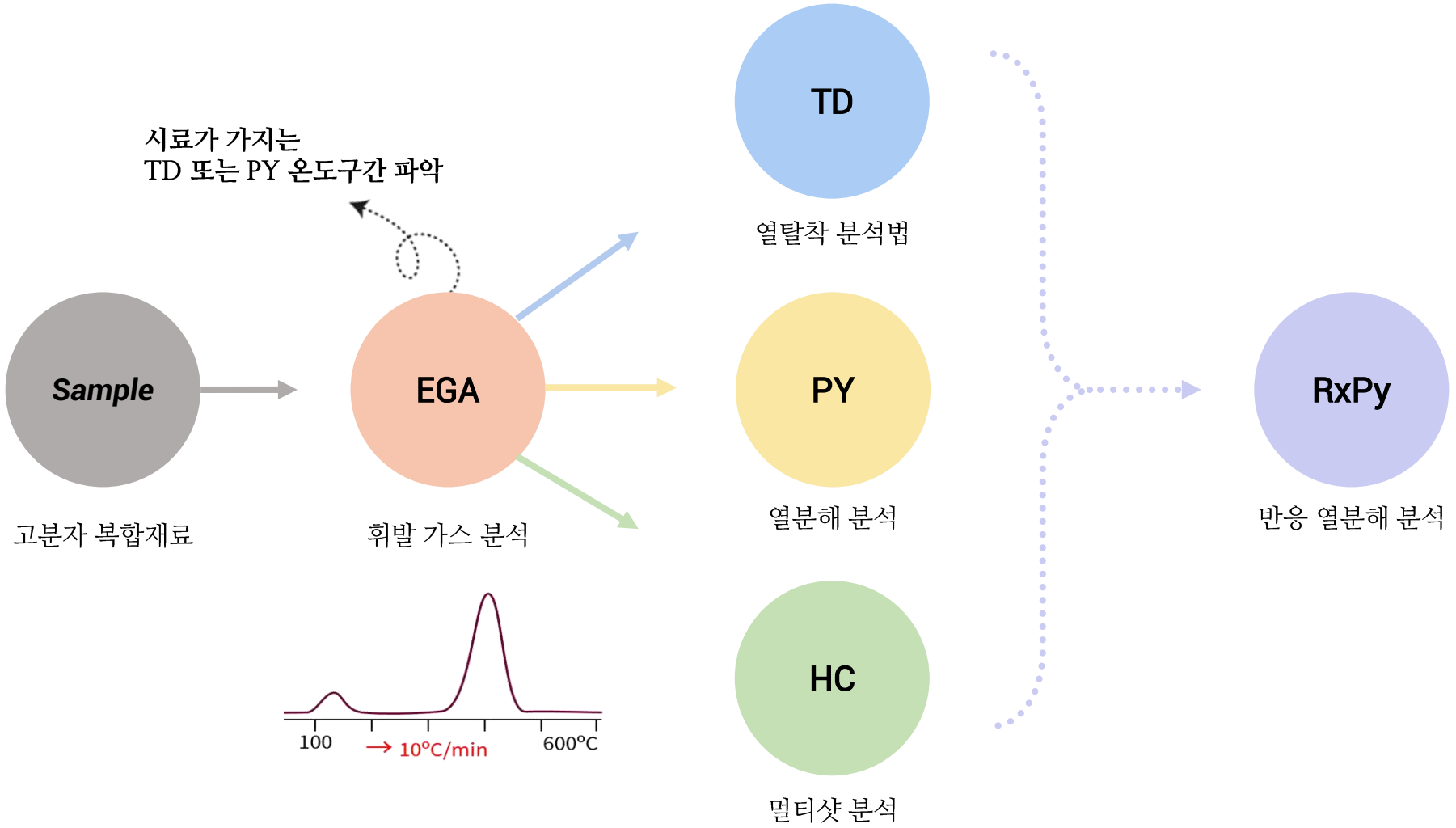
06

고분자 복합재료 특성화를 위한 Multi-Shot Pyrolyzer Method Map



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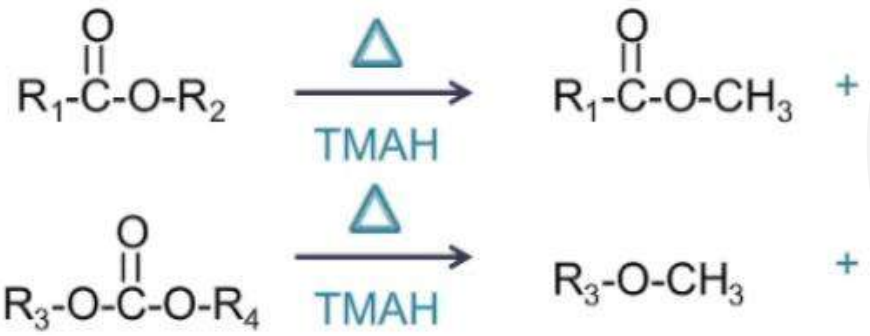
고분자 복합재료 특성화를 위한 Multi-Shot Pyrolyzer Method Map

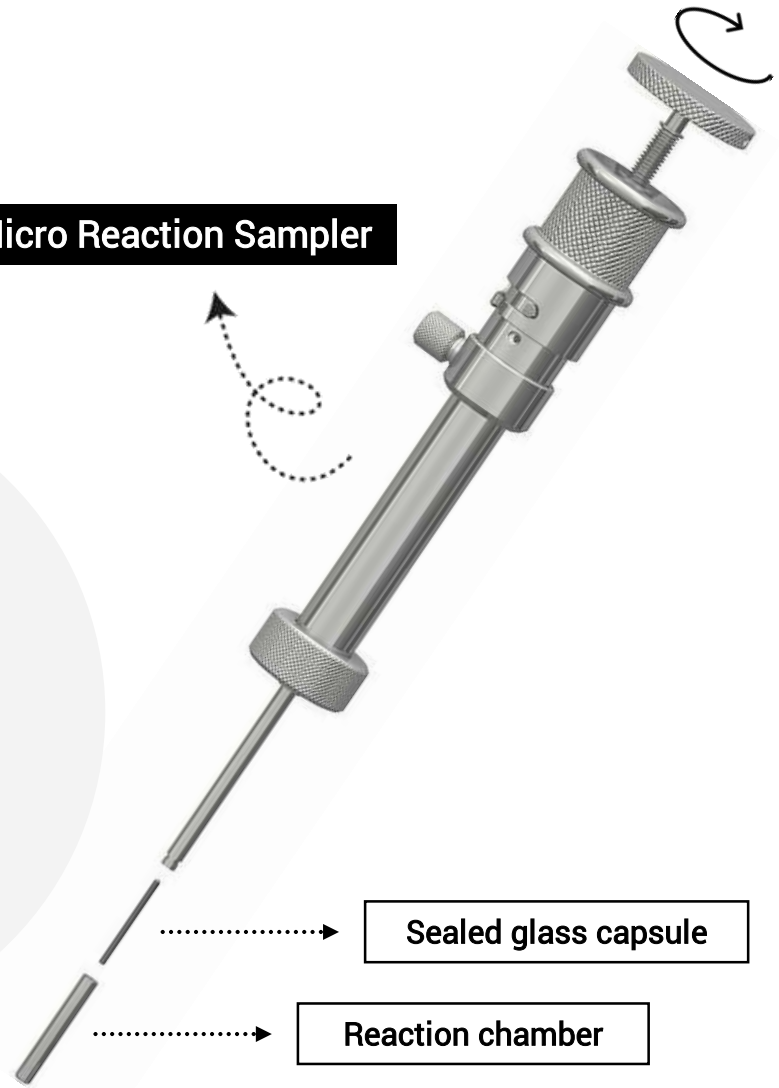
Reactive Pyrolysis (Rx Py)

RxPy

Synonym :

Thermally Assisted Hydrolysis and Methylation (THM)





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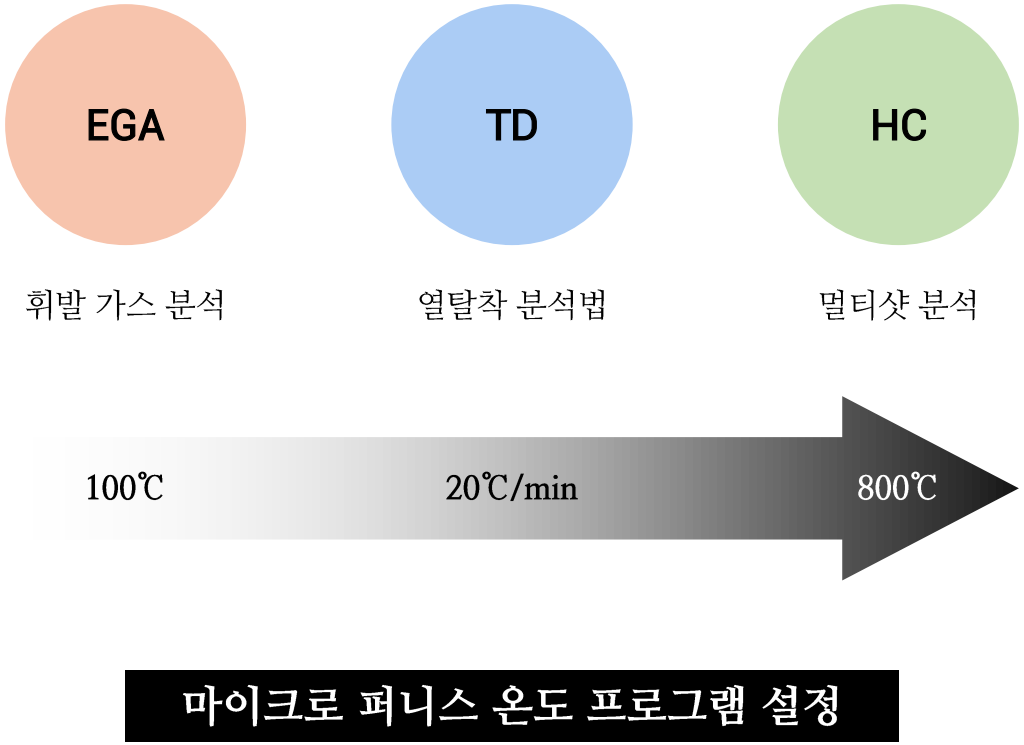
고분자 복합재료 특성화를 위한 Multi-Shot Pyrolyzer Method Map



마이크로 퍼니스 온도 프로그램 설정

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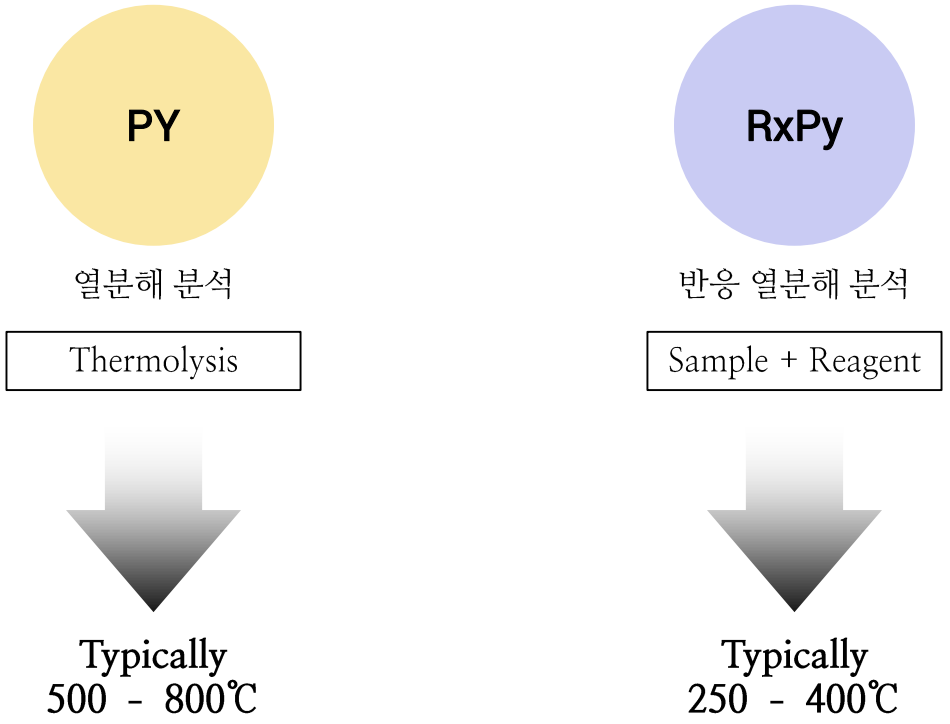
고분자 복합재료 특성화를 위한 Multi-Shot Pyrolyzer Method Map



등온 분석 (Isothermal)

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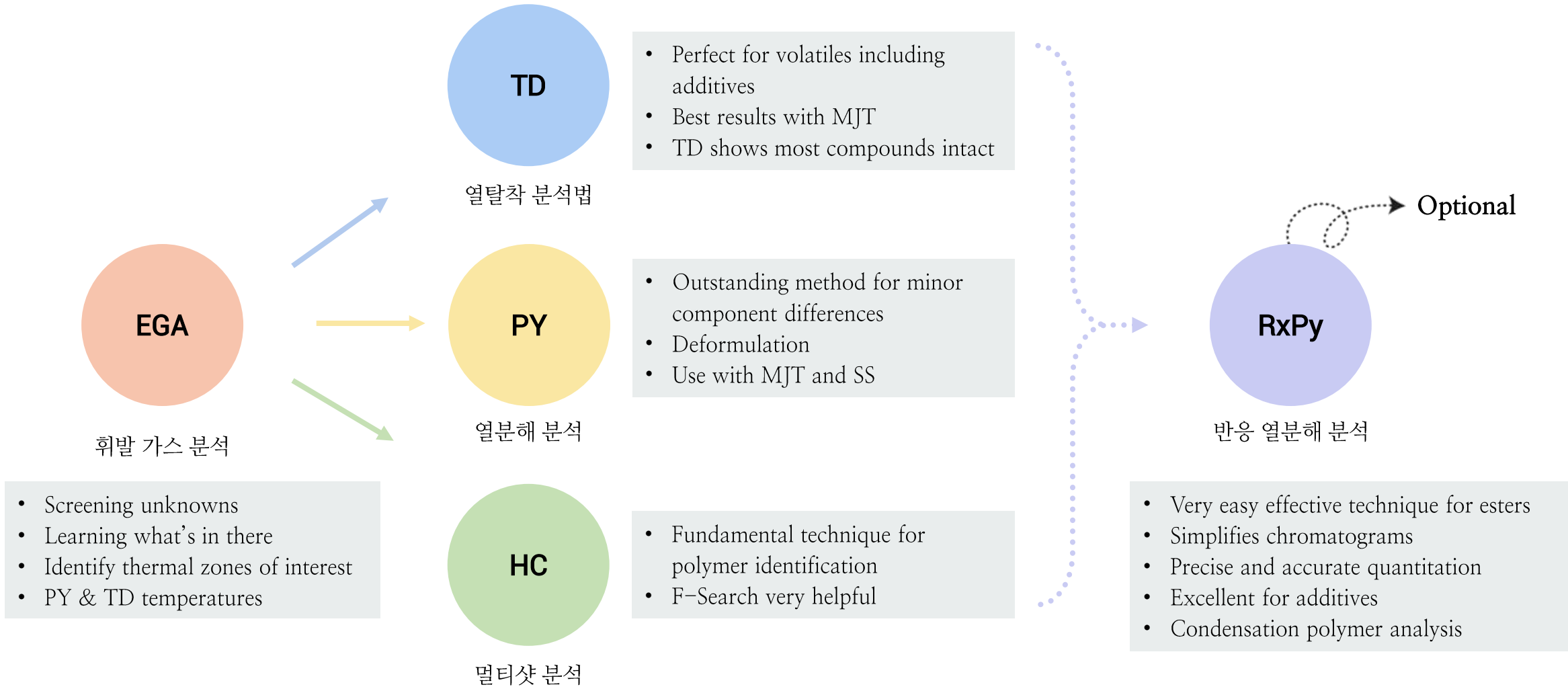
고분자 복합재료 특성화를 위한 Multi-Shot Pyrolyzer Method Map



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07

EGA/PY-3030D 멀티샷 파이롤라이저 Py-GC/MS 자동화 시스템 구성

Typical systems

- Multi-Shot Sampler (EGA/PY-3030D)
- Auto-Shot Sampler (AS-2020E)
- MicroJet Cryo-Trap (MJT-2035E)
- Ultra ALLOY metal capillary column
- Vent-free GC/MS Adapter
- F-Search system Ver. 3.7

More optional systems

- Selective Sampler (SS-2010E)
- Carrier Gas Selector (CGS-1050Ex)
- Online Micro Reaction Sampler
- Online Micro UV Irradiator (UV-1047Xe)
- F-Search MPs Ver. 2.1
- Cryogenic Mill (IQ MILL-2070)

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08

멀티샷파이롤라이저 내부 구조 및 시료 이동 경로

멀티샷 파이롤라이저 시료 주입 방법

- Manual injection by Double-Shot Sampler
- Automated injection by Auto-Shot Sampler

퍼네이스 온도 설정 (40 – 1,050℃)

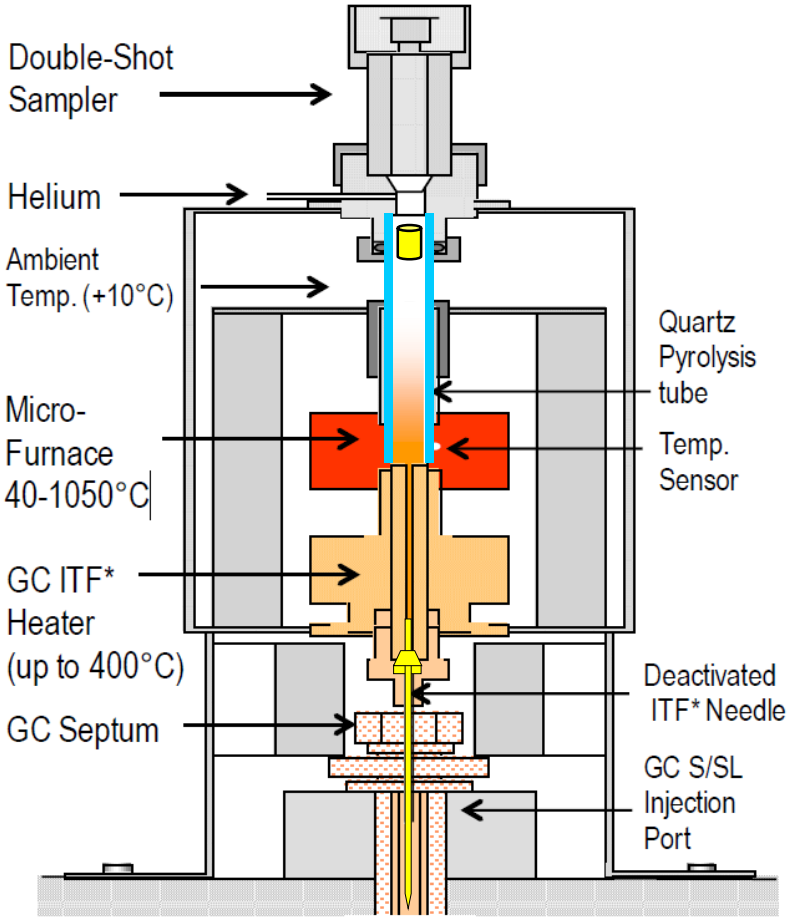
- Isothermal
- Temperature programmed

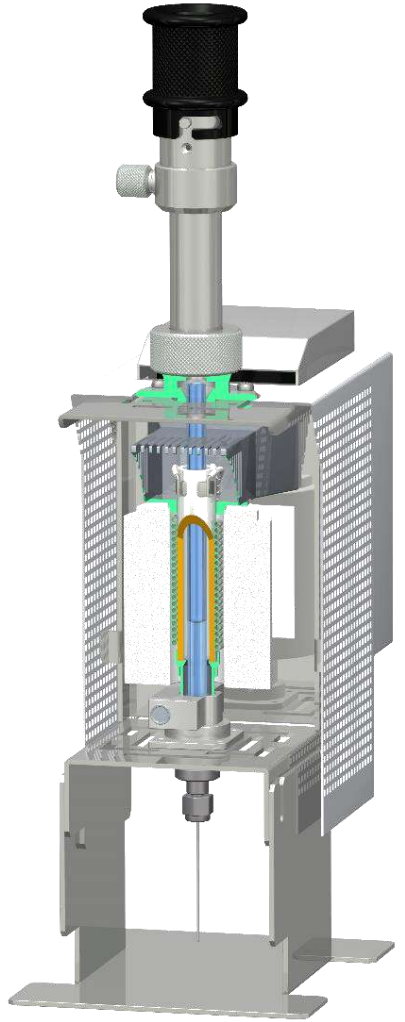
Multiple analytical functions

- Evolved Gas Analysis (EGA)
- Thermal Desorption (TD)
- Flash pyrolysis (PY or Single-Shot)
- Heart-Cutting/EGA-GC/MS (Multi-Shot)

Reactive Pyrolysis(RxPy) Method uses

- Isothermal TD + Reagent (i.e. TMAH)





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09

파이롤라이저 자동시료주입 시스템
오토샷 샘플러 AS-2020E

Carrousel of
Auto-Shot top cover

Auto-Shot Sampler

- 최대 48개 시료컵 자동 연속 주입
- 분석 재현성 향상 : RSD 〈 3% (e.g. PS pyrogram)
- Carrier gas 가압에 의한 시료컵 자동 주입 및 회수
- 모든 분석 메서드 자동화 설정
  - ✓ Double-Shot, Heart-Cut, MicroJet Cryo-Trap, Selective Sampler, etc.

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10

고분자 라이브러리 시스템
F-Search

EGA-MS Library

Polymer

Py-GC/MS Library

Polymer

MS Library

Additive

Pyrolyzates MS Library

Polymer

F-Search Engine (ver. 3.7)

1,315 polymers and 590 additives

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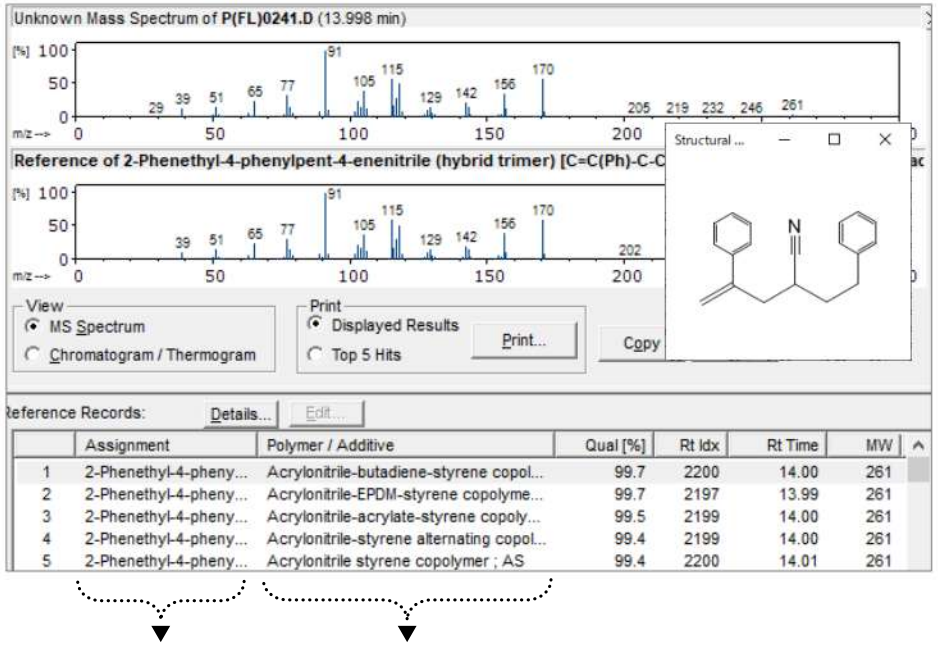
10

고분자 라이브러리 시스템
F-Search

2023 updated Ver. 3.7

Pyrolyzate-MS 라이브러리를 이용한
Polymer search 예

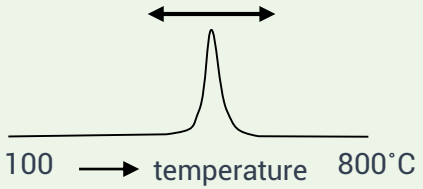


Pyrolyzate candidates

열분해를 통해 2-Phenethyl-4-phenylpent-4-enenitrile을 생성하는 Polymer candidates

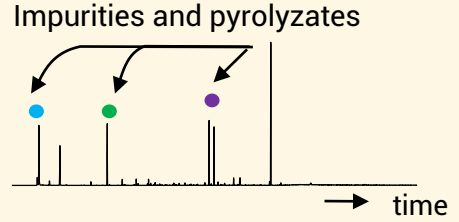
EGA-MS Library

- Thermograms in library
- Integrated sum spectra



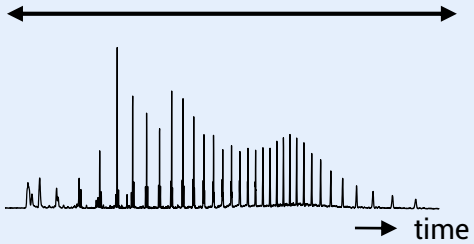
Additive-MS Library

- Individual peak spectra
- Impurities



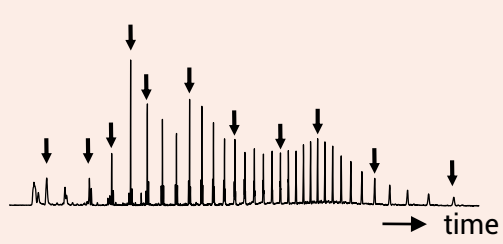
Py-GC/MS Library

- Pyrograms in library
- Integrated sum spectra



Pyrolyzates-MS Library

- Individual peak spectra
- Structures of polymers



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Application Notes 01.

Problem

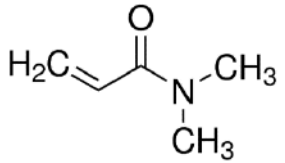
Can outgassing be determined during cutting of polyimide?

Analysis

EGA-MS Analysis

Result

BPDA+3, 3-DDS produces DMAA*, CO2, H2O in 1st stage.
CO2, SO2 and aniline in 2nd stage.



*DMAA: N,N-Dimethylacetoacetamide

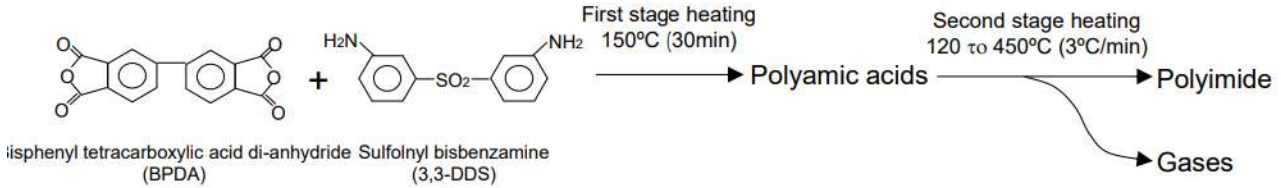


Fig. 1 Curing Process of Polyimide

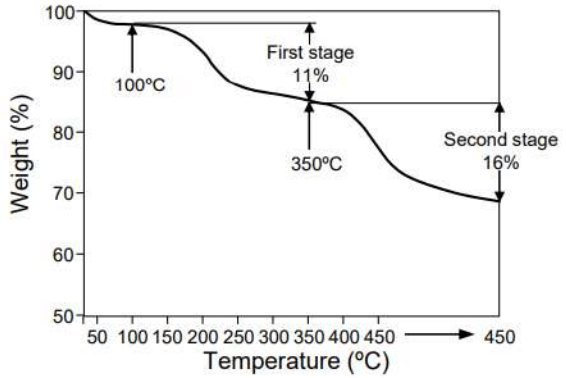


Fig. 2 TGA Curve for Polyimide Curing Process

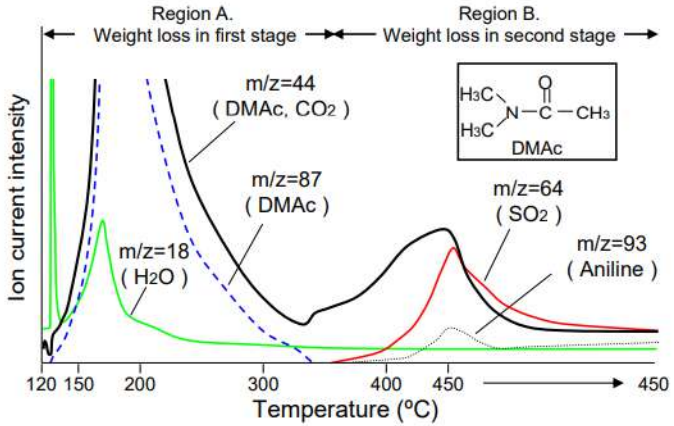


Fig. 3 EGA Curves of Polyimide Curing Process by Selected Ion Monitoring

https://www.frontier-lab.com/assets/file/technical-note/PYA3-002E.pdf

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11

Application Notes 02.

Problem

Ceramic composite material(injection molding) characterize?

Analysis

EGA-MS Analysis

Result

A: Phthalate
B: Saturated hydrocarbon
C: PBMA
D: Polystyrene
Using Wiley and F-Search MS Libraries

Fig. 1a Library Search Result for Peak C

Name	Qual
1. Poly(n-butyl methacrylate) (PBMA)	**: 72**
2. Poly(2-hydroxyethyl methacrylate) :	: 4
3. Higher methacrylate copolymer	: 2

Fig. 1b Library Search Result for Peak D

Name	Qual
1. Polystyrene (PS)	**: 90**
2. Styrene-ethylene-butadiene-styrene-block copolymer	: 78
3. Modified poly(phenylene oxide)	: 64

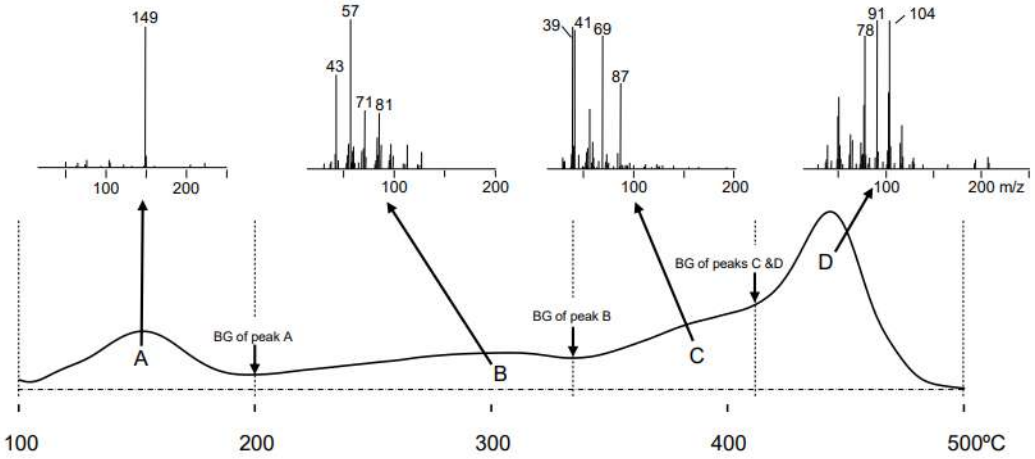

Fig. 1 EGA Curve of Ceramic Composite Material (Injection molding)
Pyrolysis furnace temp : 100°C~500°C (20C/min), Carrier gas : He 50kPa, Split ratio : ca. 1/50
EGA capillary tube : 0.15mm id, length 2.5m (UADTM-2.5N), GC oven temp : 300°C
Injection port temp : 320°C, Amount of sample : ca. 0.5mg, Detector : MS (m/z=29-400, 0.1 scan/sec)
PY-GC interface temp : 320°C (AUTO mode)

https://www.frontier-lab.com/assets/file/technical-note/PYA1-010E.pdf

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11

Application Notes 03.

Problem

Can an antioxidant in PE be separated from polymer backbone and quantitated?

Analysis

EGA, then TD(100 to 300℃),
EIC of 205 and 220 m/z for BHT
TD with column next

Result

EGA shows where BHT evolves and what TD temp is 100 to 200 ℃.
TD chromatogram separates BHT, ESTD allows quant. (This is 510 ppm BHT by FID, %RSD 〈3%)

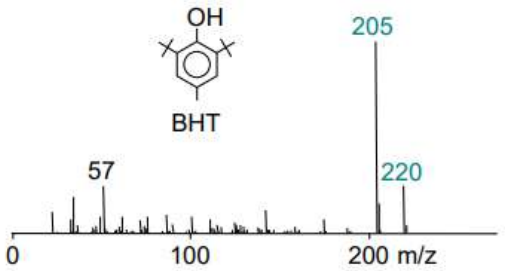

Fig. 1 Chemical structure of BHT and its mass spectrum

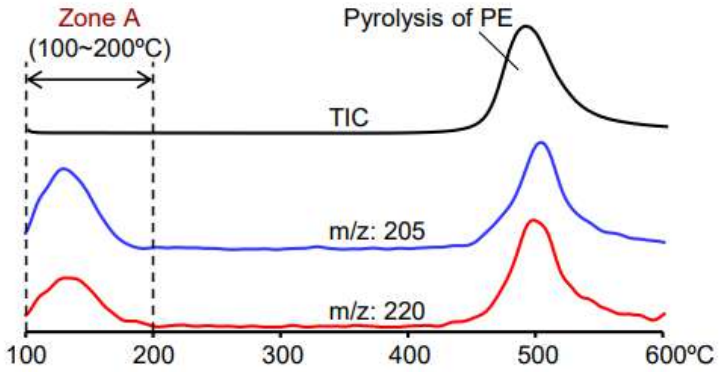

Fig. 2 Thermograms of a PE Sample
Furnace temp. : 100-600°C, (20°C/min), split ratio : 1/50
Sample size : about 0.5mg, detector : MS

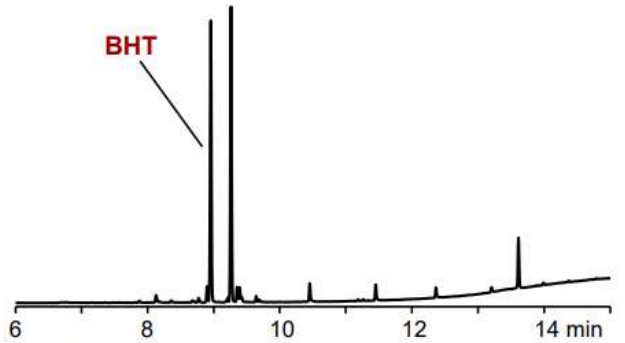

Fig. 3 Chromatogram of thermally desorbed components from a PE sample
Furnace temp.; 100-200°C (20°C/min, 3min hold), flow rate : 1 mL/min,
split ratio : 1/50, GC oven temp.: 40-320°C (20°C/min), sample size : about 3mg,
detector : FID, separation column : Ultra ALLOY-5
(5%-diphenyl 95%-dimethyl polysiloxane, L=30m, id=0.25mm, df=0.25μm)

https://www.frontier-lab.com/assets/file/technical-note/PYA1-055E.pdf

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11

Application Notes 04.

Problem

How can additives be identified in PS?

Analysis

TD-GC/MS (100 - 300℃)

Result

10 additives identified using F-Search.

- Dibromophenol
- BHT
- Tinuvin P, and 770
- Triphenyl phosphine
- Sumilizer MDP-S
- Irgafos 168
- Irganox 1076
- ADK STAB PEP-36
- Decabromo phenylether

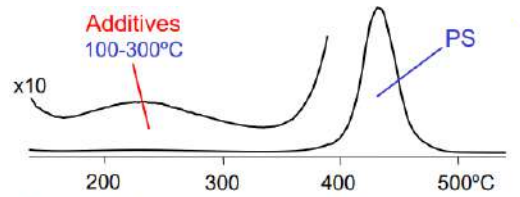

Fig.1 EGA Thermogram of PS

Py furnace temp.: 100 - 700°C (20°C/min),
GC oven temp.: 300°C,
EGA tube: UADTM-2.5N(L=2.5 m, i.d. 0.15 mm),
column flow rate: 1 mL/min He, split ratio: 1/50,
sample: 0.1 mg

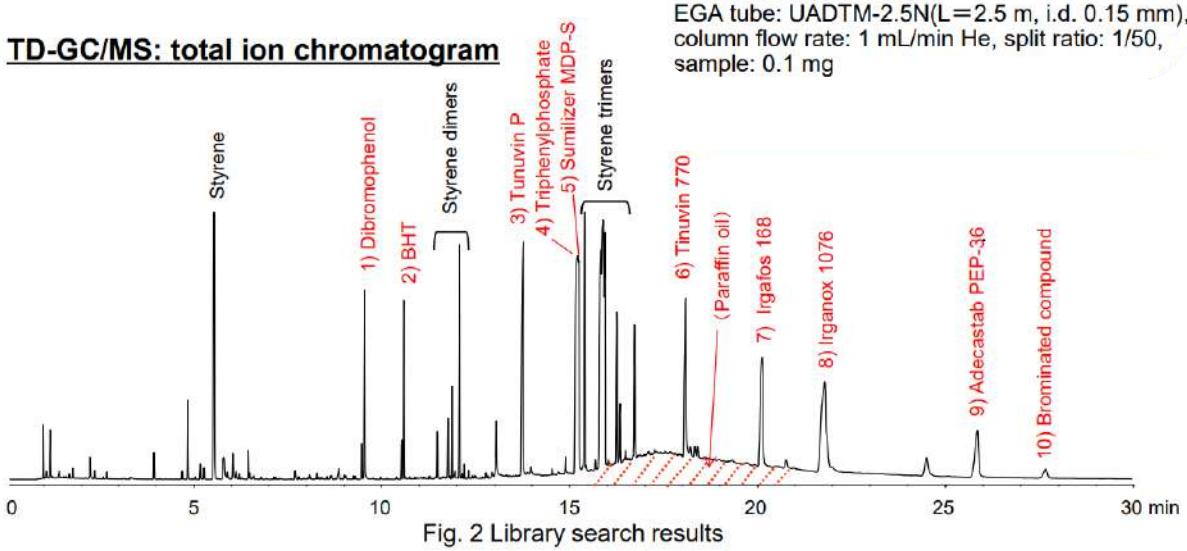

Fig. 2 Library search results

Pyrolyzer temp.: 100 - 300°C (20°C/min, 5 min), GC oven temp.: 40°C (2 min) - 320°C (20°C/min), separation column: Ultra ALLOY+-5 (5% diphenyl 95% dimethylpolysiloxane) (L=30 m, i.d.=0.25 mm, df=0.05 µm), column flow rate: 1 mL/min He, split ratio: 1/20, scan rate: 2 scans/sec, scan range: 29 - 810 (m/z), sample: 1 mg

Reference: K. Odagiri et al., 13th Polymer Analysis and Characterization (2008), II-11

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11

Application Notes 05.

Problem

Study end group subtle differences in PMMA?

Analysis

PMMA radically polymerized in 0.3% benzoyl peroxide (BPO). PY temp.: 460℃

Result

Clues to polymerization mechanisms in toluene, benzene, and no initiators are revealed in pyrograms. (a) Shows many phenyl rings not found in (b) or (c) suggesting solvent fragments incorporated into chain ends.

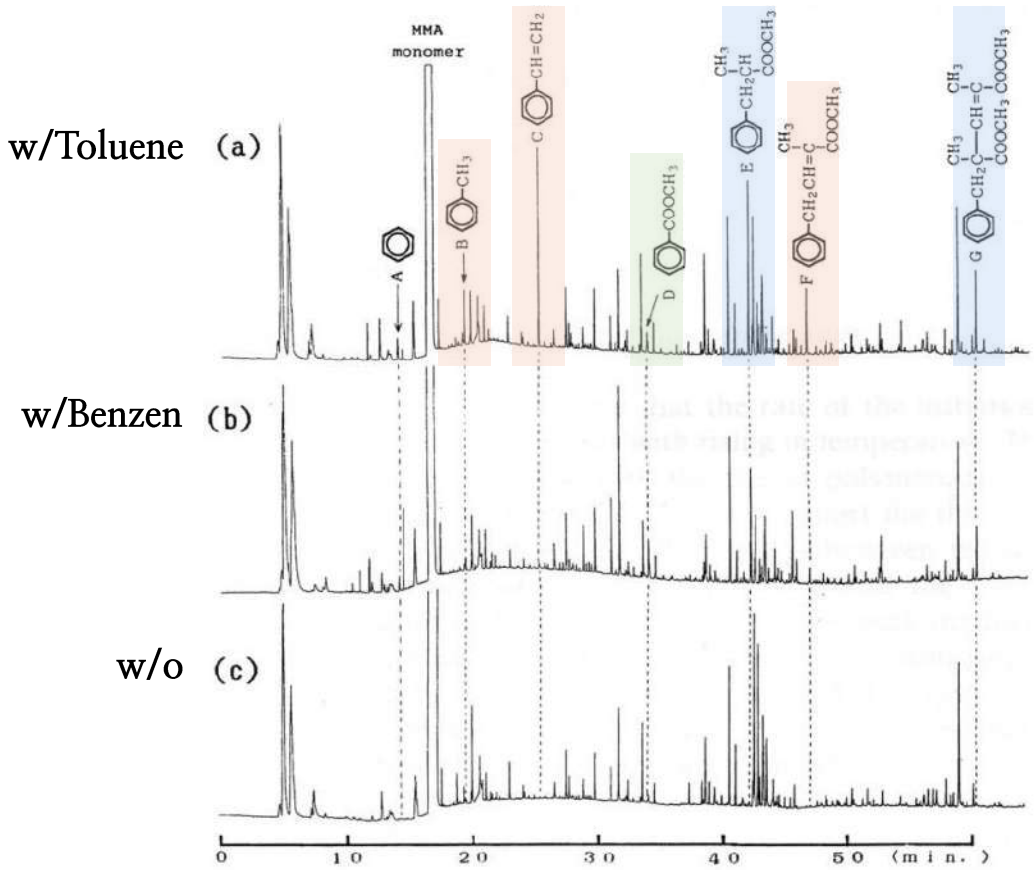

Figure 1. Pyrograms of PMMA samples at 460°C; (a) polymerized in toluene, (b) polymerized in benzene, and (c) polymerized without any initiator.

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11

Application Notes 06.

Problem

What information can be obtained from compounded rubber?

Analysis

TD followed by PY is "Double-Shot" analysis

Result

TD range: 100 to 300℃
D3 to D6 from silicon coupling agent; benzeothiazoles(vulcanization agent), antioxidant waxes, etc.

PY: 500℃ (flash pyrolysis)
Isoprene and limonene indicate high amounts natural rubber

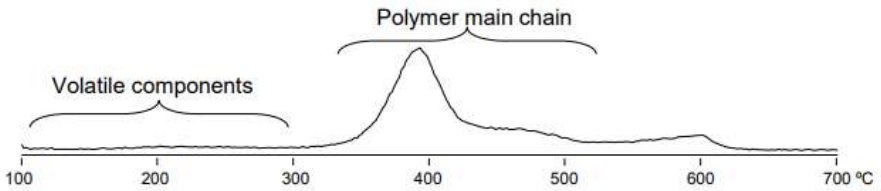

Fig. 1 Evolved Gas Curve of a Compounded Rubber

Pyrolysis temp.: 100~700 ºC (20 ºC/min), Carrier gas : He 50 kPa, Split ratio : ca. 1/20
EGA capillary tube : 0.15 mm id, 2.5 m (UADTM-2.5N), GC oven temp.: 300 ºC
Injection temp.: 320 ºC, Sample : ca. 500 µg, Detector : MS (*m/z*=29-400)

Fig.2a. Thermal Desorption Chromatogram
(100~300 ºC (20 ºC/min))

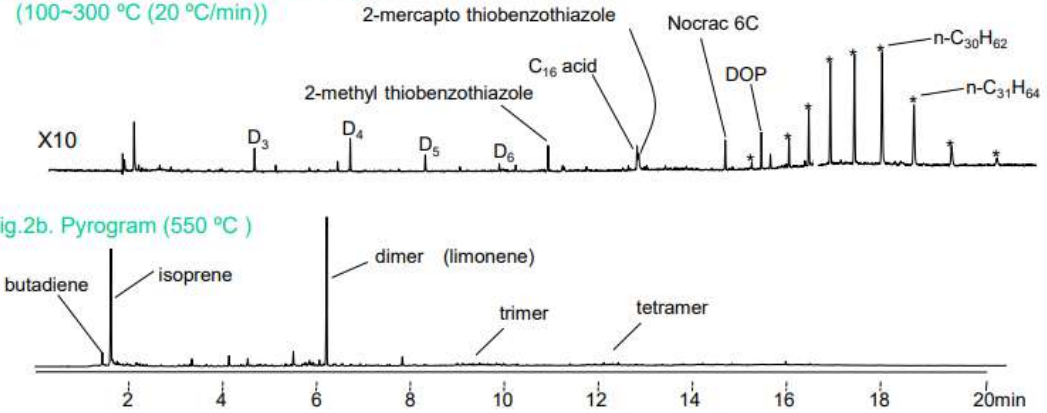

Fig. 2 GC/MS Analysis of Compounded Rubber by Double-Shot Technique

Column flow rate : 1 mL/min (fixed flow rate), Split ratio : 1/20
Separation column : Ultra ALLOY+-5 (5 % diphenyl polysiloxane), 30 m, 0.25 mm id, Film thickness : 0.25 µm
GC oven temp.: 40~300 ºC (20 ºC/min), Sample : 500 µg, Detector : MS (*m/z*=29-400, 2 scan/sec)

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11

Application Notes 07.

Problem

How can compositional analysis of thermoplastic polyurethanes(TPU)?

Analysis

PY (Single-Shot)

Result

PY: 600℃
Sample A: Adipate polyol, cyclopentanone, etc.
Sample B: ε-Caprolactone
Sample C: Pyrolyzates derived from PTMG

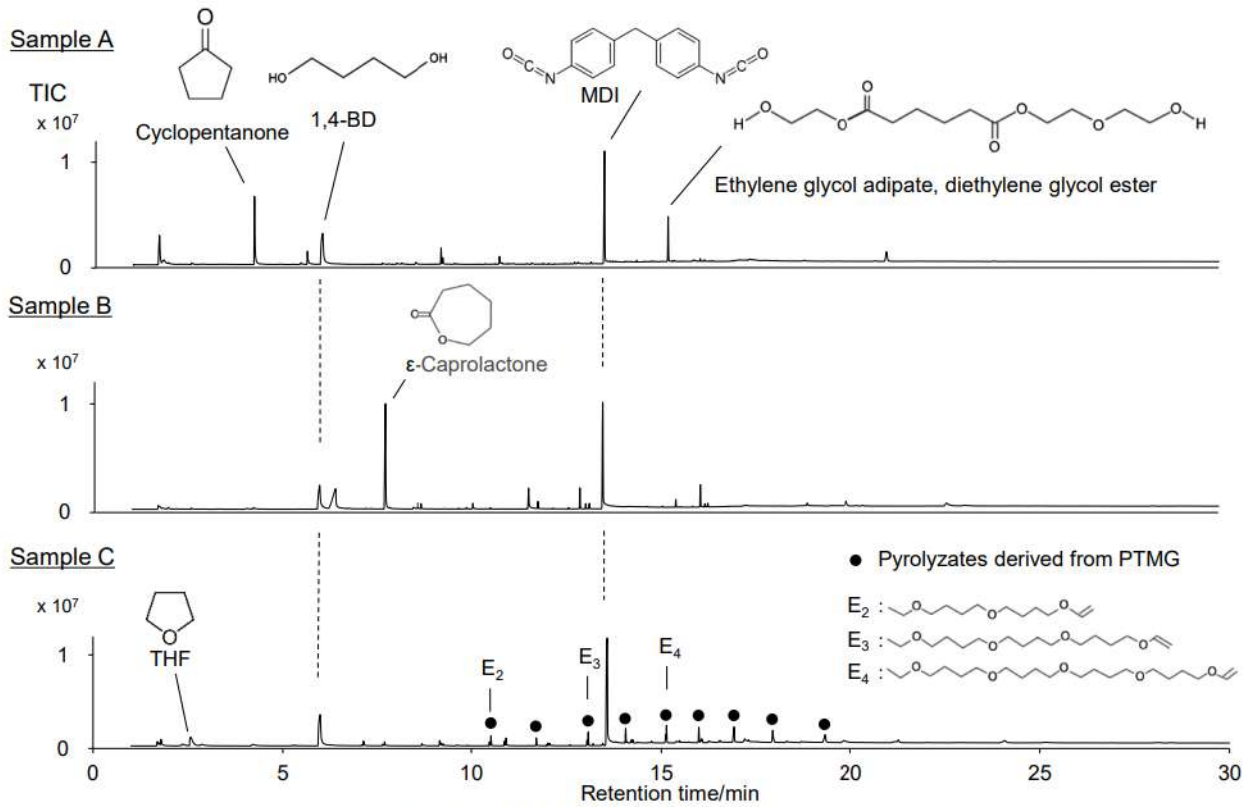

Fig.1 Pyrograms of three types of TPU samples

Pyrolyzer furnace temp.: 600 °C, GC oven temp.: 40 °C (2 min hold) – 320 °C (20 °C/min, 14 min hold), Split ratio: 1/50, Column flow rate: 1 mL/min
Separation column: UA⁺-5 (5 % diphenyl 95 % dimethylpolysiloxane, *L*=30 m, i.d.=0.25 mm, df=0.25 µm,
MS scan range: *m/z* 29 – 600, Sample amount: 0.05 mg

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11

Application Notes 08.

Problem

Can monomers of polybutylene terephthalate(PBT) be observed?

Analysis

PY and RxPy (with TMAH)

Result

PY: 500℃
It shows decomposition and decarboxylation of ester group but no monomer.

RxPy with TMAH: 400℃
It shows monomer of dimethyl derivatives of terephthalic acid and mono and dimethyl derivatives of 1,4-butanediol.

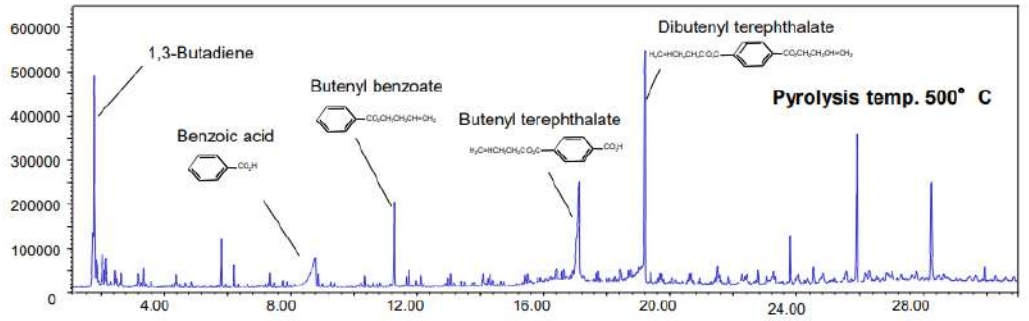

Fig. 1 Pyrogram Obtained by Flash Pyrolysis of PBT

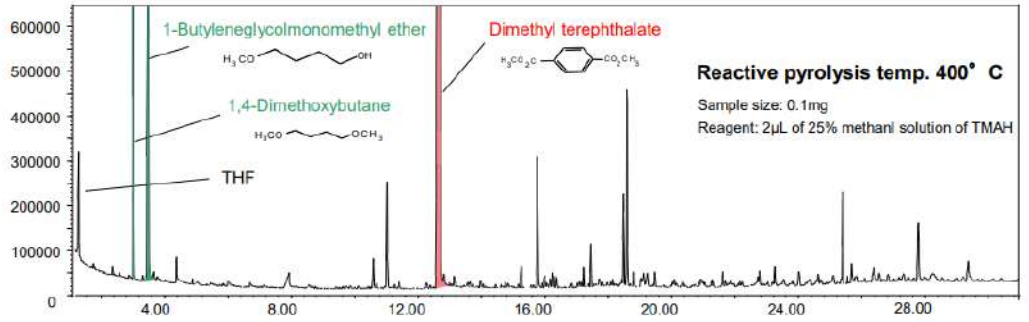

Fig. 2 Pyrogram Obtained by Reactive Pyrolysis of PBT

Analytical conditions: carrier gas: He, Injection port pressure: 103kPa, Split ratio: 1/60, Separation column: Ultra ALLOY+-5 (5% diphenyldimethylpolysiloxane)
Length: 30m, Id: 0.25mm, Film thickness: 0.25μm, GC oven temp: 38ºC~300ºC (20ºC /min), GC injection port tem: 320ºC
Material excerpted from "5. A few recent applications of Py-GC", Kiura, Wakabayashi (Mitsubishi Rayon),
2nd Pyrolysis Gas Chromatography Seminar (hosted by Frontier Lab Ltd.)

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11

Application Notes 09.

Problem

Analysis of Polyethylene Terephthalate (PET) by Reactive Pyrolysis

Analysis

RxPy (with TMAH)

Result

PY: 500℃
It shows decomposition of ester group, but no monomer.

RxPy with TMAH: 400℃
It shows PET constituent monomer of dimethyl ester of terephthalic acid, and mono and dimethyl esters of ethylene glycol

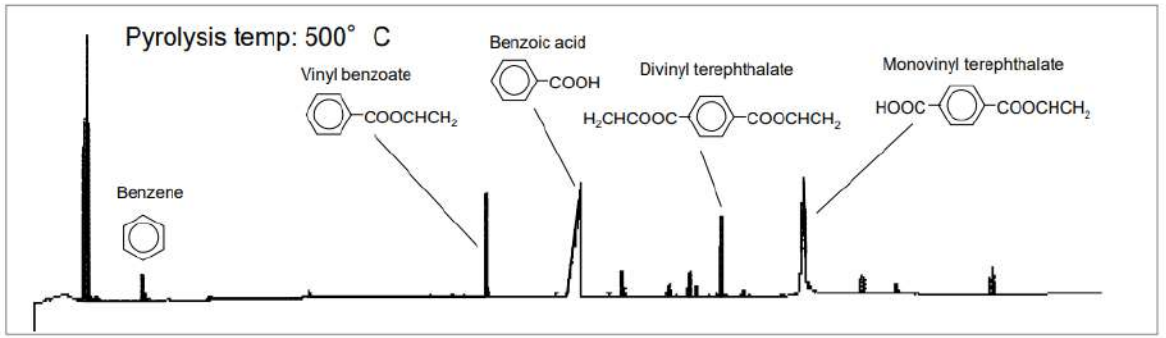

Fig. 1 Pyrogram Obtained by Flash Pyrolysis of PET

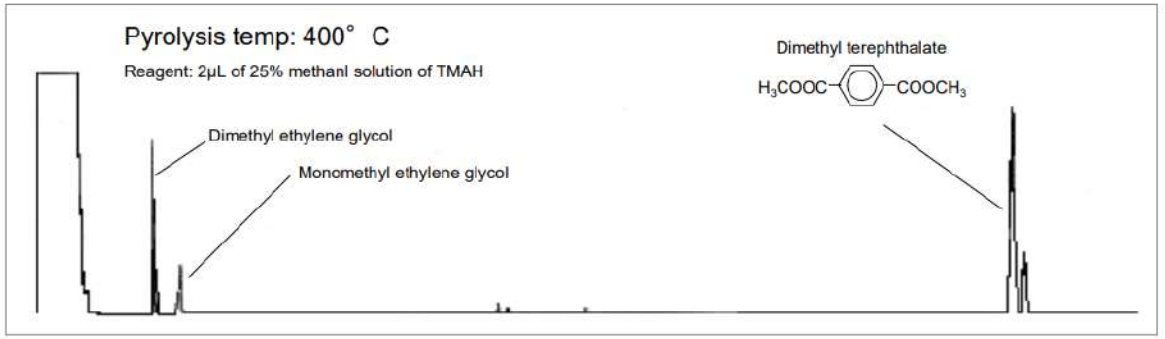

Fig. 2 Pyrogram Obtained by Reactive Pyrolysis of PET

Analytical conditions: carrier gas: He, Injection port pressure: 103kPa, Split ratio: 1/60, Separation column: Ultra ALLOY⁺-5 (5% diphenyldimethylpolysiloxane) Length: 30m, Id: 0.25mm, Film thickness: 0.25μm, GC oven temp: 38°C~300°C (20°C /min), GC injection port tem: 320°C

Material excerpted from "5. A few recent applications of Py-GC", Kiura, Wakabayashi (Mitsubishi Rayon), 2nd Pyrolysis Gas Chromatography Seminar (hosted by Frontier Lab Ltd.)

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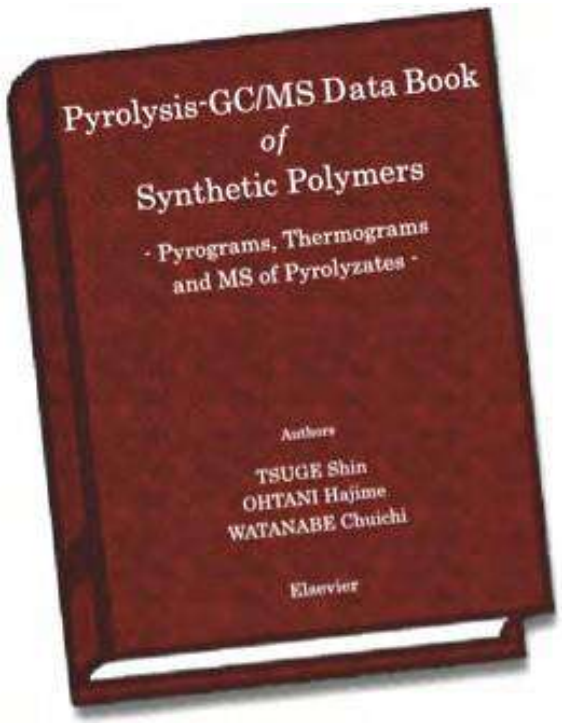
12

Pyrolysis–GC/MS Data Book of Synthetic Polymers

TSUGE Shin, Emeritus Professor of Nagoya University, Japan OHTANI Hajime,
Nagoya Institute of Technology, Japan WATANABE Chuichi, Frontier Laboratories Ltd., Japan



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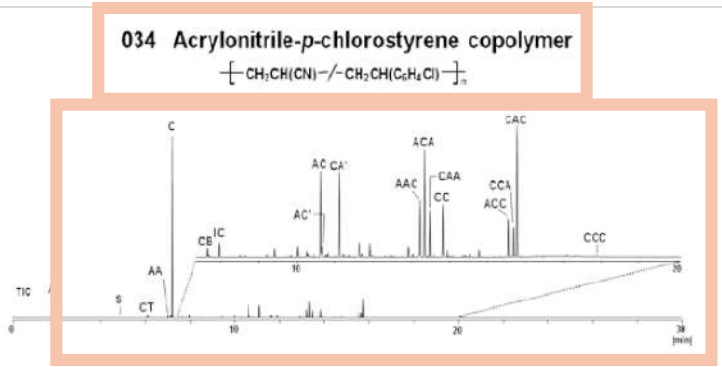
PUB DATE: October 2011
LIST PRICE: $295.00 ~
FORMAT: Hardback

판매처 | 예스24 (http://www.yes24.com/Product/Goods/5092589)

034 Acrylonitrile-*p*-chlorostyrene copolymer

$+CH_2CH(CN)-/-CH_2CH(C_6H_4Cl)+_n$

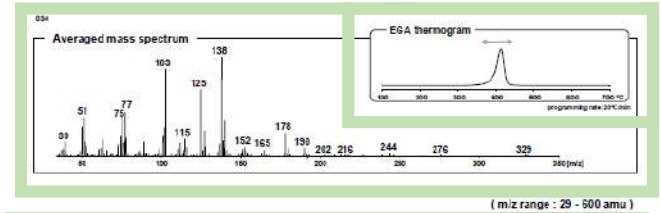


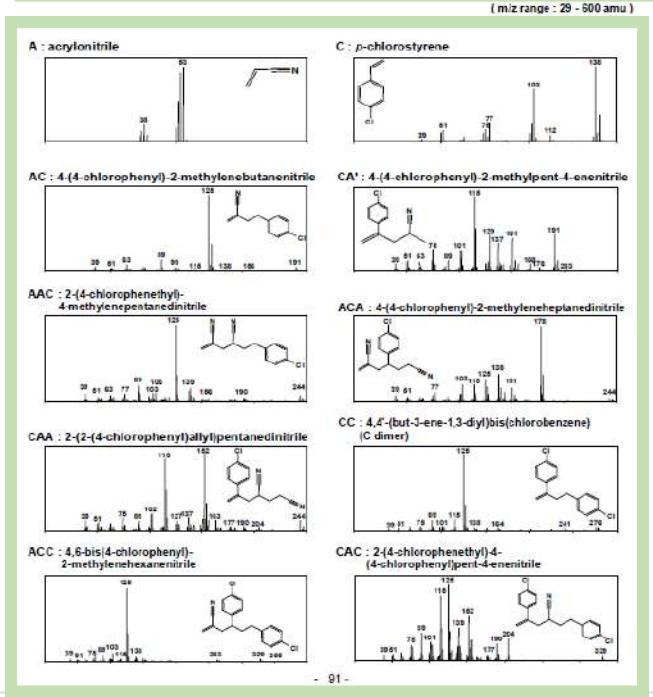
Peak Notation	Assignment of Main Peaks		Molecular Weight	Retention Index	Relative Intensity
A	acrylonitrile		53	570	2.1
S	styrene		104	889	0.1
CT	*p*-chlorotoluene		126	957	1.2
AA	C=C(CN)-C-C-CN (A dimer)		106	1056	1.1
C	*p*-chlorostyrene		138	1081	100.0
CB	*p*-chlorobenzaldehyde		140	1128	0.7
IC	4-chloroisopropenylbenzene		152	1168	1.0
AC	C=C(CN)-C-C-PhCl	(hybrid dimer)	161	1562	6.3
AC'	C(PhCl)-C-C-CN		179	1558	0.7
CA'	C=C(PhCl)-C-C(CN)-C		265	1644	7.5
AAC	C=C(CN)-C-C(CN)-C-C-PhCl	(hybrid trimer)	244	2051	4.1
ACA	C=C(CN)-C-C(PhCl)-C-C-CN		244	2075	8.2
CAA	C=C(PhCl)-C-C(CN)-C-C-CN		244	2101	3.3
CC	C=C(PhCl)-C-C-PhCl (C dimer)		276	2179	4.2
ACC	C=C(CN)-C-C(PhCl)-C-C-PhCl	(hybrid trimer)	329	2597	2.9
CCA	C=C(PhCl)-C-C(PhCl)-C-C-CN		329	2629	2.4
CAC	C=C(PhCl)-C-C(CN)-C-C-PhCl		329	2657	9.7
CCC	C=C(PhCl)-C-C(PhCl)-C-C-PhCl (C trimer)		414	3144	0.4

* bonding hydrogen is omitted : PhCl represents C_6H_4Cl (4-chlorophenyl group)

[Related Reference]

1) Okumoto, T.; Tsuge, S.; Yamamoto, Y.; Takeuchi, T. *Macromolecules* 1974, 7, 376





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상반기 세미나 일정 (예정)

일 자	장 소	주 제	응 용
2023-02-23	On-line	Pyrolysis-GC/MS를 이용한 플라스틱 및 고무의 열분해 특성 연구	Plastic, Rubber
2023-03-30	On-line	자동차 내장재 및 타이어 분석을 위한 Py-GC/MS 활용 가이드	Automobile
2023-04-20	Off-line	New GC Detector - 고감도 수분분석을 위한 새로운 GC 검출기 LUMA	Water, Pharma
2023-05-25	On-line	최신 미세플라스틱 전처리 동향 및 정성·정량 분석법	Microplsatics
2023-06-29	On-line	대기악취 채취 부터 GC/MS 분석까지! Entech 대기분석 토탈 솔루션 소개	Ambient Air, VOCs

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