C146-E184C

SHIMADZU
Excellence in Science

Frontier Laboratories Multi-functional Pyrolyzer

EGA/PY-3030D

Frontier Laboratories Multi-functional Pyrolyzer

EGA/PY-3030D

Multi-Function Pyrolyzer Supports More Efficient and Accurate Thermal Analysis

Significantly enhanced analysis capacity

High performance and high reliability

Analyze samples in any form

Auto-Shot Sampler
AS-2020E

Incorporating Pyrolysis System
GCMS-QP2050

From Double-Shot to Multi-Shot Pyrolyzer

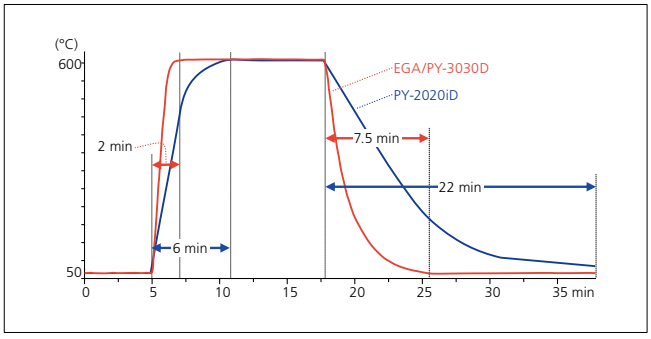
The EGA/PY-3030D Multi-Shot Pyrolyzer employs a vertical micro furnace. It is based on over 30 years' basic research into pyrolysis gas chromatography (Py–GC) by Shin Tsuge, Emeritus Professor at Nagoya University.
It is the top-class model in the series of multi-function pyrolyzers developed by Frontier Laboratories Ltd as a result of business-academia collaboration. Based on the technologies fostered during the development and upgrades of double-shot pyrolyzers, the EGA/PY-3030D achieves superior performance and ease-of-use in all aspects, from the furnace performance to details of the control software.

Pyrolysis gas chromatography (Py–GC) permits analysis of almost all sample forms, including insoluble materials and compound materials, and can also analyze trace levels of polymeric materials without the normal pretreatment. As Py–GC provides information that is difficult to obtain by other methods, it provides an extremely powerful tool for polymer characterization.

Three Features of EGA/PY-3030D

1. Significantly Enhanced Analysis Capacity
(rapid heating and cooling across a wide temperature control range)

The highly temperature-resistant hollow ceramic heater reduces the heater thermal capacity to one-sixth the previous level, permitting rapid heating and cooling across a wide temperature range from near-ambient temperature to 1050 °C.



Comparison of Heating and Cooling Rates Between 50 °C and 600 °C
for New and Previous Models

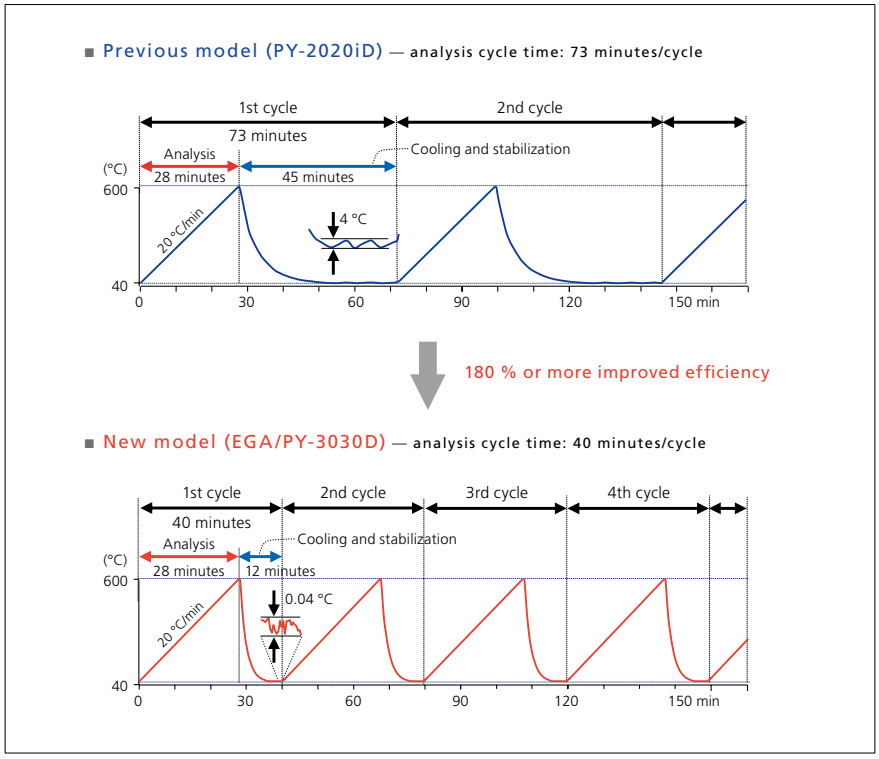
Rapid Heating and Cooling

The 600 °C/min heating rate (ten times faster than previous models) and 100 °C/min rapid cooling rate allow the set temperature to be reached rapidly.

Whereas the PY-2020iD needed six minutes to reach 600 °C, the EGA/PY-3030D gets there in just two minutes. The cooling time has also been reduced considerably from 22 minutes to 7.5 minutes.

Highly Efficient Analysis

For evolved gas analysis (EGA), the furnace repeats heating and cooling. The PY-2020iD could analyze up to 20 samples per day; in contrast the EGA/PY-3030D can handle 36 samples, an improvement in efficiency of 180 %.

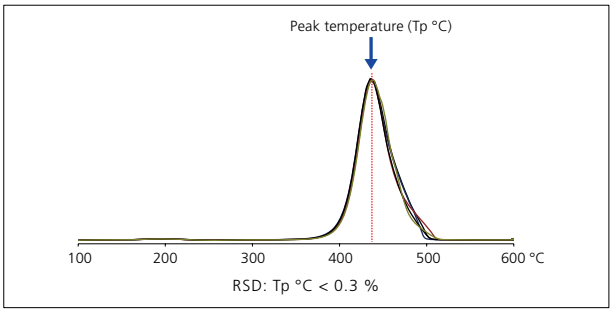


Comparison of Analysis Cycles for EGA with New and Previous Models
<Furnace heating conditions: 40 to 600 °C (20 °C/min)>

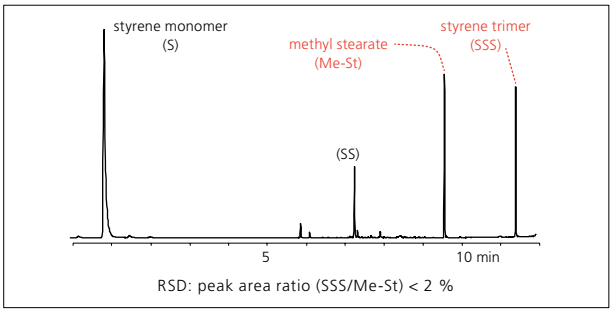
2. High Performance and High Reliability

Reproducibility of EGA Thermograms and Pyrograms

The peak temperature reproducibility in the EGA thermograms for polystyrene and the styrene trimer/methyl stearate peak area ratio reproducibility in pyrograms from single-shot analysis are measured before dispatch and the results are supplied with the instrument.



Reproducibility of Thermograms



Reproducibility of Pyrograms

3. Analyze Samples in Any Form

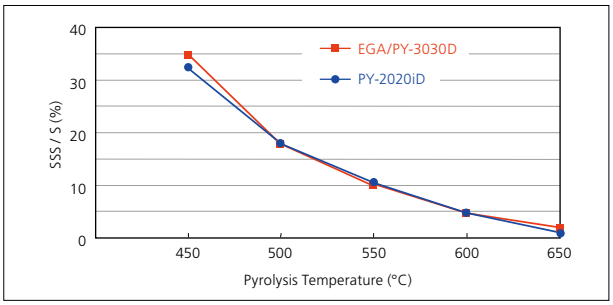
Select the optimal analysis method from the following four methods to suit the form of the sample and the aim of analysis.

Four Diverse Analytical Methods (See pages below for details.)

(1) Evolved gas analysis (EGA–MS)
(2) Single-shot analysis (Py–GC/MS)
(3) Double-shot analysis (TD/Py–GC/MS)
(4) Heart-cut analysis (Heart-Cut EGA–GC/MS)

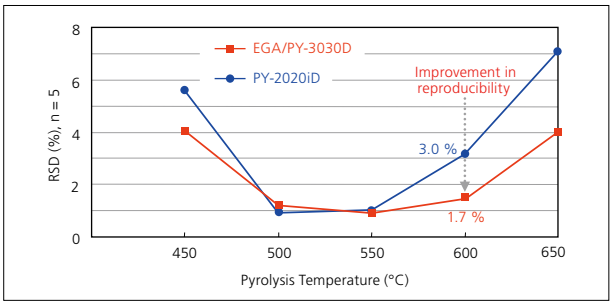
Correlation of Data with Previous Model

The multi-shot pyrolyzer offers a higher level of performance while maintaining correlation of analysis results with the conventional double-shot pyrolyzer. Existing data libraries can be used unchanged.



Correlation of Peak Area Ratio (SSS/S)

Measurements using EGA/PY-3030D and the previous PY-2020iD yield similar values for the pyrolysis temperature dependence of the ratio of styrene monomer (S) and trimer (SSS) generated by the thermal decomposition of polystyrene (PS).



Correlation of Peak Area Ratio (SSS/S) Reproducibility

The EGA/PY-3030D, with its wide range of furnace temperature uniformity and precise temperature control, enhances the pyrogram reproducibility above 600 °C.

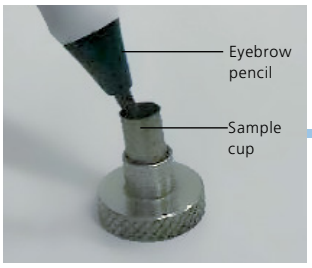
Material Characterization Using the Multiple Analytical Methods <Multiple Analyses for Unknown Samples>

Abstract: When working with challenging samples, such as an eyebrow pencil, the first step is to characterize the sample using evolved gas analysis (EGA–MS). Analysis of the EGA thermogram provides information about the thermal complexity of the sample, the nature of the polymer and the presence or absence of specific compounds of interest. EGA will help the analyst select the next step in the process.

A good example of using EGA to suggest what analysis will yield the most useful information about the sample is the characterization of a commercial eyebrow pencil.

Like many cosmetics, an eyebrow pencil is a complex mixture of compounds ranging from volatiles to polymers.

The eyebrow pencil is placed directly into the sample cup and analyzed directly; there is no sample prep.



Sampling of Eyebrow Pencil

Evolved Gas Analysis (EGA–MS)

EGA–MS requires a direct connection between the split/splitless injection port and the detector. This is done using a deactivated 0.15 mm i.d. capillary tube (EGA tube). As the sample is heated the evolved gases flow to the detector.

EGA thermogram reflects thermal properties of the entire sample

The EGA thermogram of the eyebrow pencil (on the right) has three peaks: A, B, and C, which are the volatiles in the sample, and two peaks: D and E, which originate from the polymeric portion of the sample.

F-Search displays two-dimensional multi-ion mass chromatograms

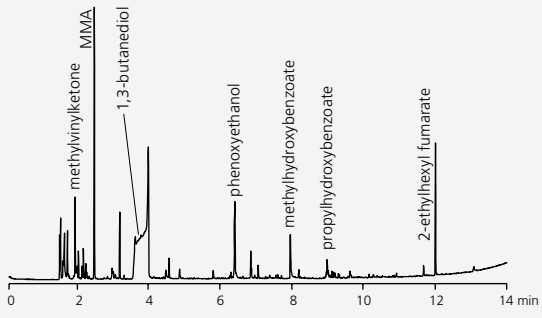
The data indicates that peaks A and B contain multiple components. Peaks D and E can be tentatively identified using F-Search and the EGA–MS library.

Single-Shot Analysis (PY–GC/MS)

The sample cup free-falls into the pyrolyzer furnace. The sample temperature goes from ambient to the pyrolysis temperate in less than 20 msec. Pyrolysis occurs instantly and the pyrolyzates are introduced into a GC separation column.

The pyrogram of the eyebrow pencil, shown below, was obtained at 550 °C, which is 50 °C higher than the temperature indicated by the EGA thermogram. The single-shot method is simple; however, the pyrogram represents the degradation of all organics in the sample which sometimes makes it difficult to interpret.

■ Peaks (A+B+C+D+E) [PY: 550 °C]



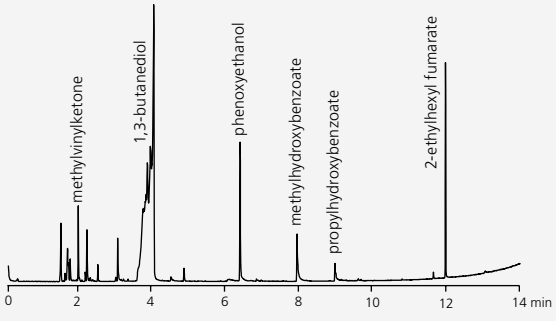
Double-Shot Analysis (TD/PY–GC/MS)

Double-shot analysis provides information about the volatile constituents and the polymeric content of the sample. The eyebrow pencil is analyzed in two steps. STEP 1 : volatiles evolving from the sample are analyzed by thermal desorption (TD)–GC/MS. This process results in a total ion chromatogram that can be used to identify the individual compounds in the sample.

STEP 1 TD–GC/MS Method

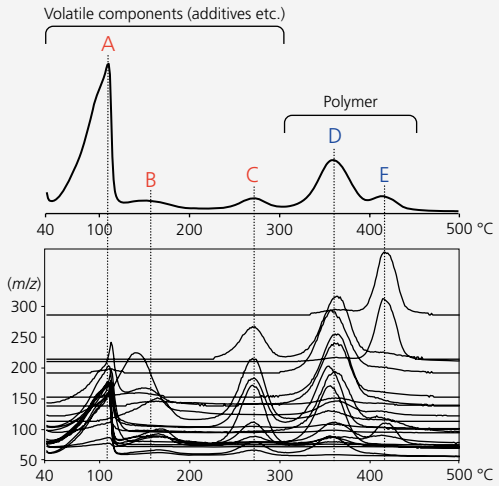
The total ion chromatogram shown below includes that portion of the sample (peaks A, B, and C) that evolves between 100 and 300 °C. A MicroJet Cryo-Trap is used to focus the individual compounds during the desorption interval at the head of the column. This maintains peak fidelity and column resolution.

■ Peaks (A+B+C) [TD: 100→300 °C]



Qualitative and quantitative analysis based on data from varied sources including F-Search and other analytical techniques

- Identification of volatiles (A, B, C):
  F-Search (EGA and additives) / NIST, Wiley library

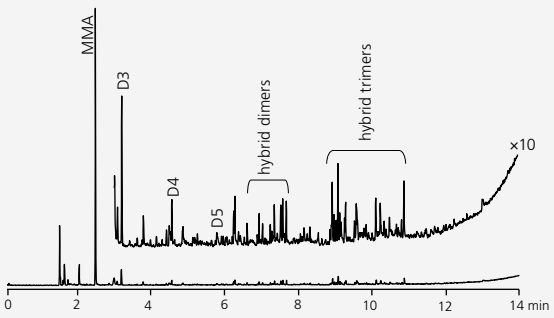


STEP 2 : the residual sample is pyrolyzed at a temperature determined by the EGA data. The pyrogram facilitates the identification of the polymeric content of the sample.

STEP 2 Py-GC/MS Method

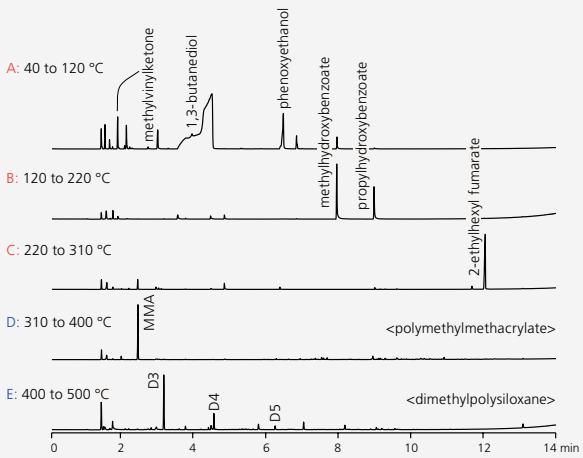
Shown below is a pyrogram of peaks D and E. Because fractions A–C have already been extracted from the sample, the peaks in the pyrogram are pyrolyzates of the polymer. The eyebrow pencil polymer contains acrylic resin and dimethylpolysiloxane.

■ **Peaks (D+E) [PY: 550 °C]**



Heart-Cut Analysis (EGA–GC/MS)

Vapors evolving from each EGA thermal zone are selectively introduced into the GC separation column and analyzed by GC/MS. Using the selective sampler and the MicroJet Cryo-Trap, up to eight thermal zones can be isolated and analyzed. Shown below are the chromatograms obtained when each of the EGA thermal zones (A–E) is analyzed sequentially. The entire method can be automated using the auto-shot sampler.



- Identification of volatiles (D, E) originated from polymer: F-Search (polymer and pyrolyzates libraries)

Ultimate Hardware Sophistication

Samplers can handle all sample forms, liquid or solid

Double-shot thermal desorption/pyrolysis sampler.

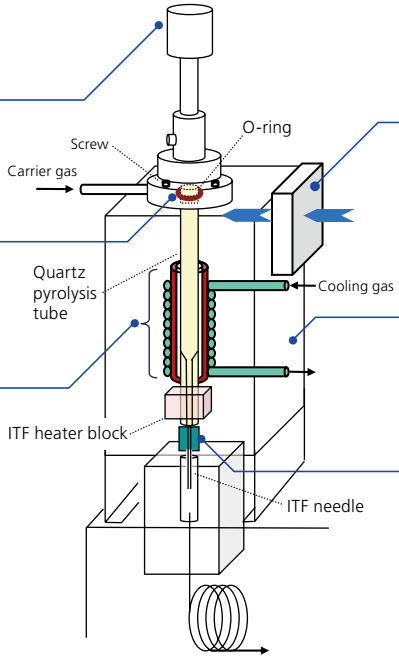
Easy maintenance

The carrier gas seal at the top of the quartz pyrolysis tube is made by simply inserting the top O-ring and tightening the three screws.

Newly developed ceramic heater furnace

The furnace adopts a newly developed ceramic heater for rapid heating and cooling as well as a temperature range up to 1050 °C. It offers a 600 °C/min heating rate, which is ten times faster than previous models, and a 100 °C/min rapid cooling rate, which is three times more efficient.

Newly Developed
Ceramic Heater



The cooling fan maintains the sample temperature at ambient temperature during stand-by

Even at a 600 °C furnace temperature, it can maintain the sample at ambient temperature at the stand-by position.

Special thermal insulation achieves excellent thermal stability

Even at a 1000 °C furnace temperature, it can maintain the external panel surface temperature at 60 °C max.

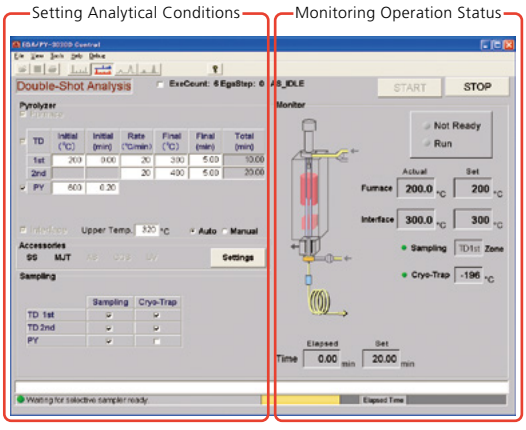
Heat-sink adapter minimizes temperature troughs

The elution of high-boiling components is possible using the combination of a septum cap and a heat-sink adapter to minimize temperature troughs at the connectors between the pyrolyzer and GC.

Simple, Easy-to-Use Control Software

Setting Analytical Conditions and Monitoring Operation Status

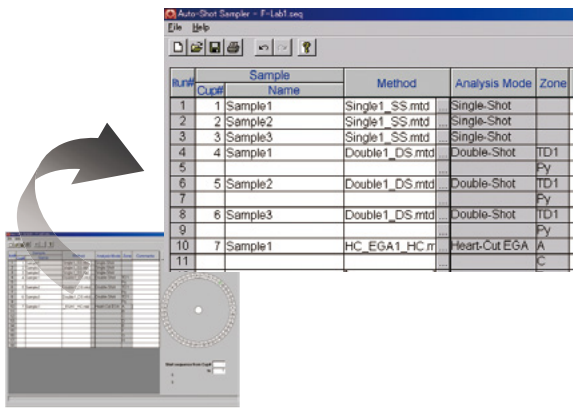
The furnace temperature conditions and peripherals are set in the left half of the window. Setup is simple, even for analyses demanding complex linking of multiple peripherals.
The operation status of all instruments can be monitored in the right half of the window.



Serial Automated Analysis with a Sequence Table

Different analytical modes can be set alternately to perform serial automated analysis of multiple samples using an Auto-Shot Sampler. This permits the measurement of a series of data by running a single sequence.

(As the separation column must be replaced with an EGA tube for evolved gas analysis, EGA must be performed using a separate sequence.)



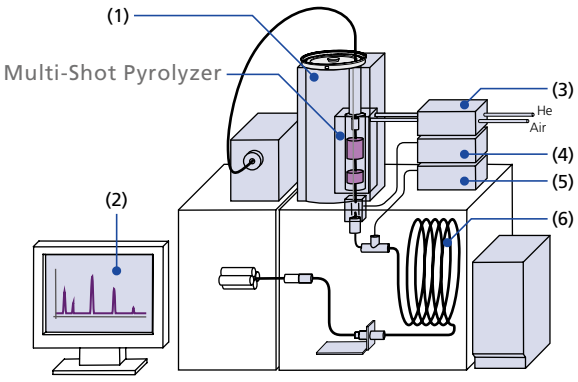
Window to Create Sequences

Peripherals

(1) Auto-Shot Sampler (AS-2020E)

Permits automated analysis of up to 48 samples, enhances reliability, and achieves dramatic labor savings.

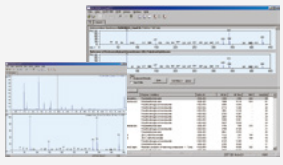
The pyrolyzer is positioned inside the Auto-Shot Sampler.



Py-GC/MS System (Multi-Shot Pyrolyzer and peripherals)

(2) F-Search System (library and analysis software)

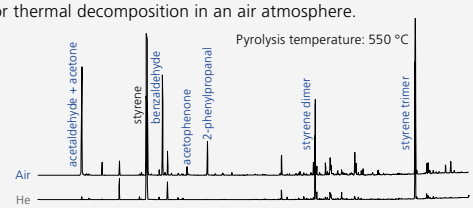
This software supports the analysis of polymers and additives using evolved gas analysis, thermal desorption GC/MS, or instantaneous pyrolysis GC/MS.



Examples of chromatograms and mass spectra search results

(3) Carrier Gas Selector (CGS-1050Ex)

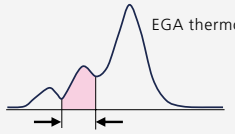
Used for thermal decomposition in an air atmosphere.



Comparison of polystyrene pyrograms measured in air and helium (He)

(4) Selective Sampler (SS-2010E)

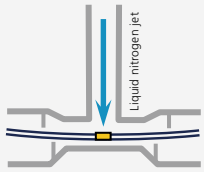
Offers automatic or manual heart-cut of required temperature fractions during the heating process and introduction into the separation column.

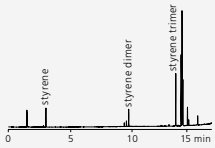


GC/MS analysis with heart-cut of required temperature fractions

(5) MicroJet Cryo-Trap (MJT-2030E)

This is a cryogenic trap for volatile components that sprays a liquid nitrogen jet around the separation column inlet. It is effective for automating analysis.

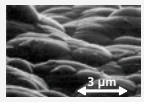




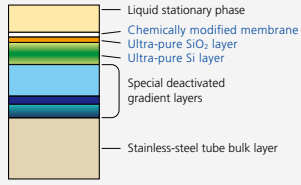
Analysis of residual oligomers in polystyrene by thermal desorption analysis using MJT-2030E

(6) Ultra ALLOY® Metal Capillary Columns

These columns are subjected to a multilayer gradient deactivation process for superb flexibility, inertness, and resistance to heating and contamination.



SEM image of deactivated tube stainless-steel inner surface



Combinations of Analysis Methods and Peripherals

		Peripheral Used for Analysis Method				
		(1) Auto-Shot Sampler	(2) F-Search System *1	(3) Carrier Gas Selector	(4) Selective Sampler	(5) MicroJet Cryo-Trap
Four Analysis Methods	Evolved gas analysis (EGA)	○	a, d	—	—	—
	Single-shot analysis	○	b, c, d	—	○	○
	Double-shot analysis	○	b, c, d	—	○	○
	Heart-cut EGA analysis	○	b, c, d	—	◎	○
Other Analysis Method	Analysis methods in air atmosphere (except EGA)	○	b, c	◎	○	○

◎: Essential; ○: Convenient or required for some analysis applications; —: Not required

*1 Recommended libraries: (a) Polymer library for EGA, (b) Polymer library for pyrograms, (c) Library for pyrolyzates, (d) Library for additives

Application Examples at 1000 °C High Temperature

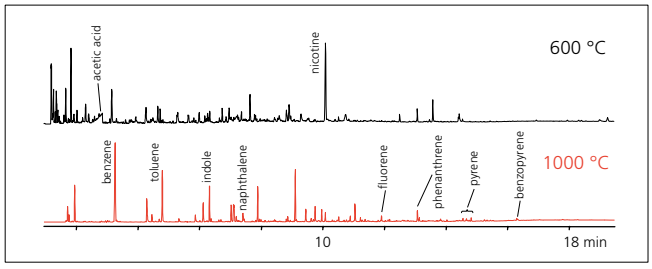
Pyrograms of tobacco leaves at 600 °C and 1000 °C measured in helium and air atmospheres

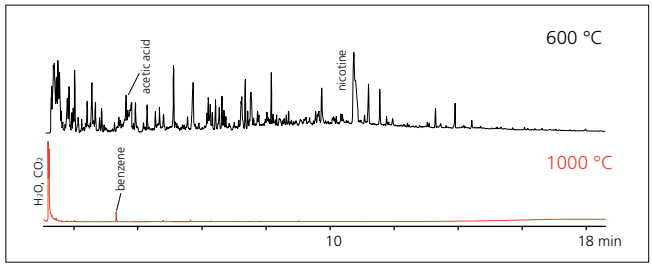
Helium (He) Atmosphere

Acetic acid and nicotine are confirmed in the 600 °C pyrogram but carcinogenic polycyclic aromatic hydrocarbons are generated at 1000 °C.

Air Atmosphere

In addition to the acetic acid and nicotine in the 600 °C pyrogram in a helium atmosphere, a striking number of oxides are observed. It is apparent that the major pyrolyzates at 1000 °C are water and carbon dioxide.





EGA/PY-3030D Specifications

1. Performance (using MS detector)

- Reproducibility of EGA thermograms — Peak temperature for polystyrene: ≤0.3 % (RSD)
- Reproducibility of pyrograms — Proportion of polystyrene pyrolyzates generated: ≤2 % (RSD)

2. Furnace and sampling unit

- Vertical micro furnace
  - Temperature control range: (Ambient temperature + 10 °C) to 1050 °C (1 °C increments)
  - Temperature stability: ±0.1 °C max.
  - Heating rate: 600 °C /min max. (1 °C /min increments)
  - Cooling method/cooling time: Forced cooling by nitrogen gas or compressed air; 10 minutes max. (800 °C to 50 °C, cooling gas pressure: 500 kPa)
- Interface unit (ITF)
  - Temperature control range/stability: 40 °C to 450 °C (1 °C increments)/ ±0.1 °C max.
- Sampler: Double-Shot Sampler, Liquid Sampler
- Inert sample cups (max. capacity): SF (50 µL), LF (80 µL)

3. Temperature controller (PC control)

- Overheat limit temperature: PY: 1100 °C, ITF: 500 °C

4. Standard accessories

Ultra ALLOY capillary column, EGA capillary tube, sample cups, control software, standard samples to check performance, etc.

5. Other

- Power requirements: 100–120 or 200–240 V AC, 50/60 Hz (400 W max.)
- Pyrolytic furnace; temperature controller: 76 (W) × 143 (D) × 215 (H) mm/ 1.6 kg, 120 (W) × 310 (D) × 310 (H) mm/ 5.4 kg
- Items provided by user
  1. Gas chromatograph: with Split/Splitless injection port
  2. Furnace cooling gas: nitrogen or air
  3. Control computer: GC control computer can be used (one USB port)

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