

# Multi-Shot Pyrolyzer<sup>®</sup>

## EGA/PY-3030D

**Flexible**  
**Versatile**  
**Reproducible**



## Why Evolved Gas Analysis?

## Why Pyrolysis?

Today, analytical pyrolysis encompasses much more than simple flash pyrolysis of polymeric materials. Virtually any material (liquid or solid) can be characterized using an array of techniques, which are designed into a modern day, Multi-Functional Pyrolysis System. Consider how these techniques can help you quickly solve the most difficult analytical challenge:

### Analytical Techniques:

- Evolved Gas Analysis (EGA)
- Pyrolysis (PY)
- Reactive Pyrolysis (RxPY)
- Multi-step Thermal Desorption (TD)
- Thermal Desorption followed by Pyrolysis (Double-Shot)
- Heart cuts based on the EGA thermogram
- UV Irradiation
- Conventional sorbent based thermal desorption
- High pressure, high temperature reaction chemistry

Examples of what can be done with a pyrolysis system, like the EGA/PY-3030D:

### Thermal Desorption-GC/MS

- Phthalates in PVC
- Residual bisphenol A in polycarbonate
- Herbicides in wood pulp
- Volatiles formed during irradiation
- Hydrocarbon profiles of various shale oils
- High temperature fuel additives
- Outgassing of electronic components
- Outgassing of medical devices

### Reactive Pyrolysis GC/MS - in less than one hour

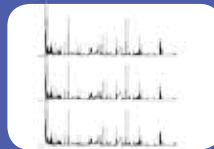
- Fatty acids in a grain
- Fatty acids in a variety of biomass materials
- Fatty acids in cosmetics

### Double-Shot GC/MS

- Additives in rubber
- Additives in paints, varnishes
- Contamination of disk drives

## What's new from Frontier Laboratories?

### Why are most laboratories integrating the Frontier Multi-Shot Pyrolyzer into their mainstream analytical protocols?



#### Guaranteed reproducibility and accuracy

Every facet of the system is designed to ensure reliability and data quality. All wetted surfaces are quartz, there is no transfer line, there is no cross contamination.



#### Versatility

The Pyrolyzer can be configured to analyze C2 vapors, C100 solids and everything in between.



#### Increase laboratory productivity

Sample prep takes less than five minutes; the low mass ceramic furnace heats and cools in record time.



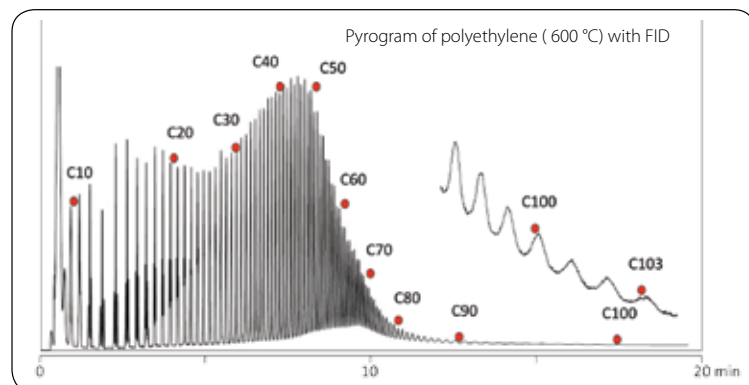
#### Analyze any sample matrix

Virtually any material (gas, liquid or solid) can be chemically characterized.



#### Tools to help understand the data

F-Search software and four MS libraries utilize MS and GC data to simplify data interpretation.



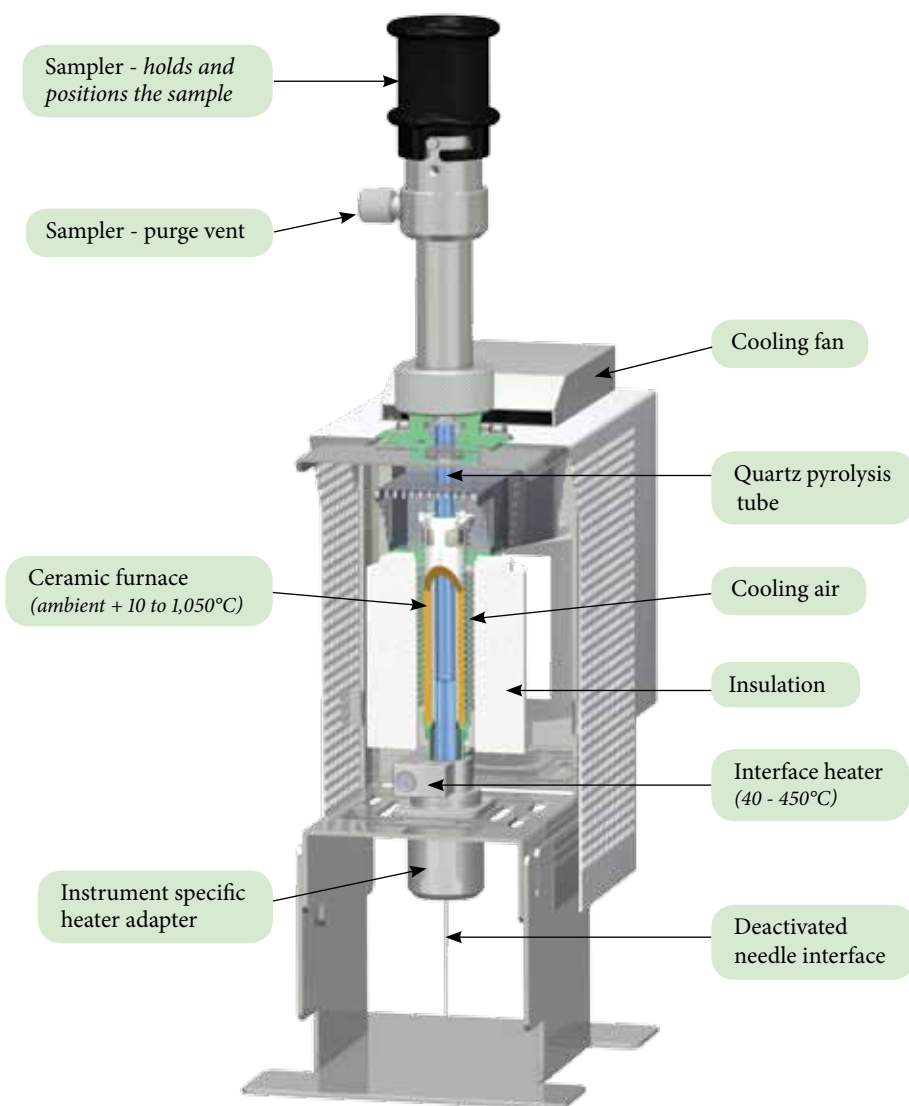
Precision and accuracy reflect the thermal homogeneity of the system thermal gradients between the 3030D and GC are eliminated by replacing the standard septum nut with one that fits snugly into the unique heat sink adapter at the bottom of the pyrolyzer. The effectiveness of this design is underscored by the C100 chromatogram.



# Pyrolyzer EGA/PY-3030D

Frontier Laboratories' new **Multi-Shot Pyrolyzer EGA/PY-3030D** is based upon the proven superiority of their patented vertical micro furnace, but everything else is new. A low mass ceramic heater heats and cools quickly. The needle interface has been re-designed to ensure thermal uniformity. The temperature control algorithm literally guarantees temperature reproducibility ( $\pm 0.1^\circ\text{C}$ ) and the operating software has a number of new features. The concept is sound, the design is simple and the engineering first rate; a two year warranty is standard — from day one!

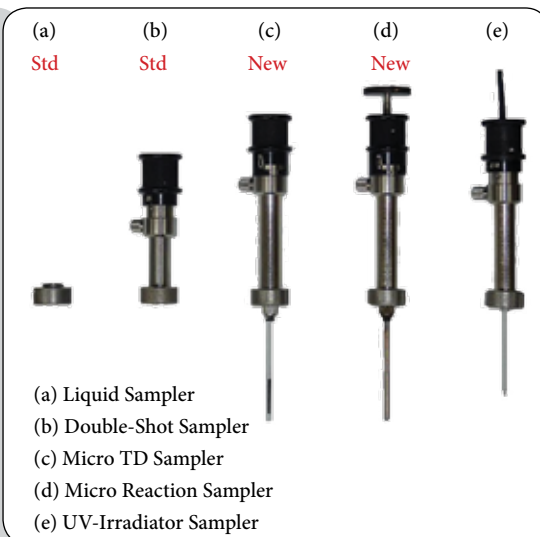
Two new samplers give the 3030D Pyrolyzer an even larger role in the analytical laboratory. Odors and other VOCs can be concentrated on conventional sorbents like Tenax and thermally desorbed using the TD Sampler. High pressure, high temperature chemistry can be investigated using the micro reactor sampler. Couple these two samplers with the many innovative accessories that can be added to the 3030D with the power of F-Search Software — you will agree that Frontier is all about analytical performance, versatility and capability.



## Versatility: One instrument, multiple analytical techniques

The Pyrolyzer EGA/PY-3030D can be configured with five different samplers (shown) and other accessories. By simply changing the sampler, the 3030D can be used to make liquid injections, desorb sorbent tubes or SPME fibers, investigate reactions at high pressure and even monitor the volatiles released as a material is UV irradiated. There is a configuration for nearly every analytical challenge: from C2 to C100, literally ethane to Shale!

For more information see [www.frontier-lab.com](http://www.frontier-lab.com)



# Material characterization using the multi-mode capability of the Frontier Pyrolyzer EGA/PY-3030D

**Abstract:** When working with challenging samples, such as an **eyeliner 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 eyeliner. Like many cosmetics, eyeliner is a complex mixture of compounds ranging from volatiles to polymers.



The **eyeliner** is placed directly into the sample cup and analyzed directly; there is **no sample prep.**

## Evolved gas analysis (EGA-MS)

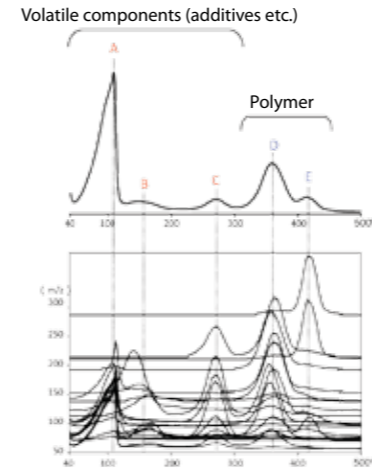
EGA-MS requires a direct connection between the split/splitless injection port and the detector. This is done using a deactivated 0.15mm i.d. capillary tube (EGA tube). As the sample is heated the evolved gases flow to the detector.

### EGA thermogram reflects thermal properties of the entire sample

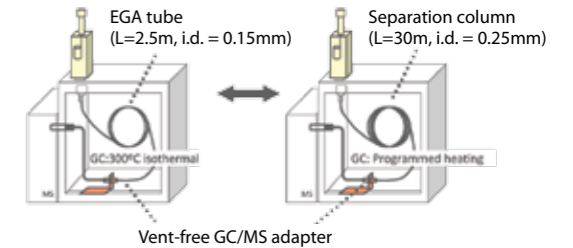
The EGA thermogram of the eyeliner (on the right) has three peaks: A, B, and C which are the volatiles in the sample and two peaks: D and E which originate from the polymeric portion of the sample.

### 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.



## Evolved gas analysis



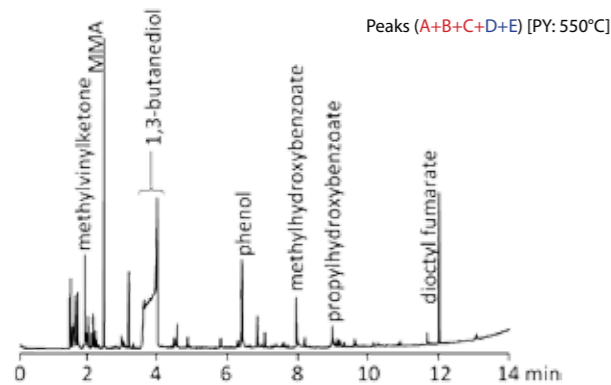
Using Vent-free adaptor, columns can be replaced without venting the MS.

Switching between EGA tube and separation column

## Single-Shot analysis (PY-GC/MS)

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

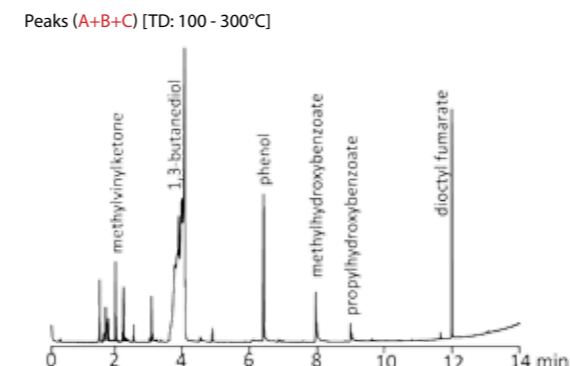
The pyrogram of the eyeliner, shown below, was obtained at 550°C, which is 50°C higher than the temperature indicated by the EGA thermogram. The single-shot method is simple, however, the pyrogram represents the degradation of all organics in the sample which sometimes makes it difficult to interpret.



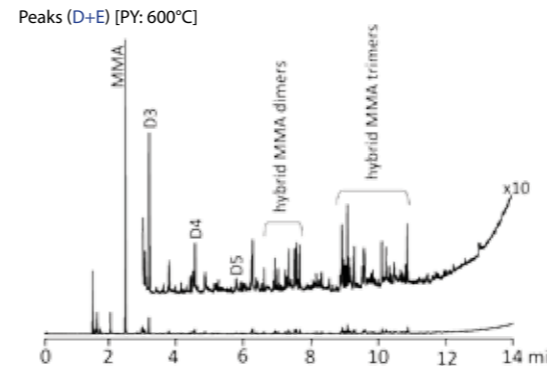
## Double-Shot analysis (TD/PY-GC/MS)

Double-shot analysis provides information about the volatile constituents and the polymeric content of the sample. The eyeliner 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 which can be used to identify the individual compounds in the sample. 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.

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.



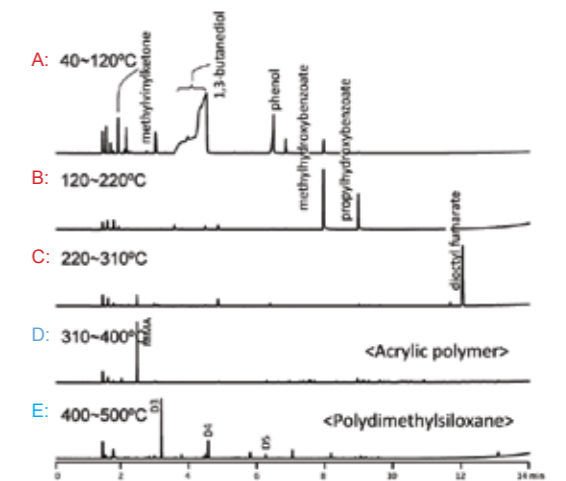
Shown below is a pyrogram of peaks D and E. Because fractions A-C have already been extracted from the sample, the peaks in the pyrogram are pyrolyzates of the polymer. The eyeliner polymer contains acrylic resin and dimethylpolysiloxane.



## 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.

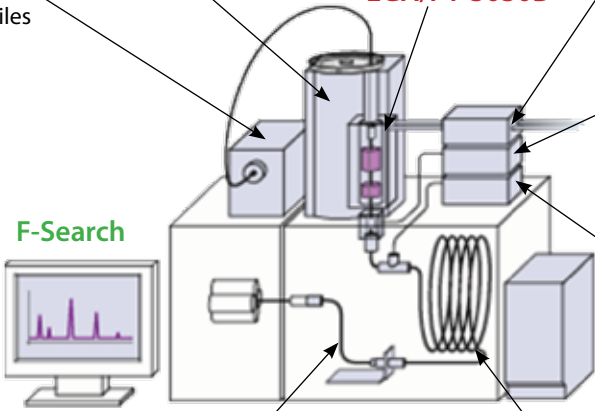
Shown below are the chromatograms obtained when each of the EGA thermal zones (A - E) is analyzed sequentially. The entire method can be automated using the auto-shot sampler.



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
- Identification of volatiles (D, E) originated from polymer: F-Search (polymer and pyrolyzates libraries)
- Identification of volatiles (A, B, C, D, E): user generated library

## 3030D Accessories for extended capability



**Micro-UV Irradiator (UV-1047Xe)**  
Any polymeric material can be exposed to UV radiation, in any atmosphere, at temperature, for virtually any length of time. Volatiles formed during the irradiation are trapped and analyzed.  
*Technical Notes: PYA5-003E, PYA5-001E*

**Auto-Shot**

**Carrier Gas Selector (CGS-1050Ex)**  
Automatically switches between two different gases. Heat sample in air, separate using helium.  
*Technical Notes: PYT-23E, 24E and PYA4-003.*

**Selective Sampler (SS-1010E)**  
Heart-cut individual EGA thermal zones. Each zone can be directed to the analytical column for analysis or automatically vented.  
*Technical Notes: PYA-012E, 019E and 023E*

**MicroJet Cryo-Trap (MJT-1035E)**  
Vapors are trapped using LN<sub>2</sub>. Special glass Tee reduces consumption of LN<sub>2</sub>. Manual, automated, and stand-alone versions.  
*Technical Note: PYT-019E*

**GC/MS Adapter (MS402180)**  
The Vent-free adapter facilitates changing columns or the change over from EGA to the analytical mode without venting the MS.

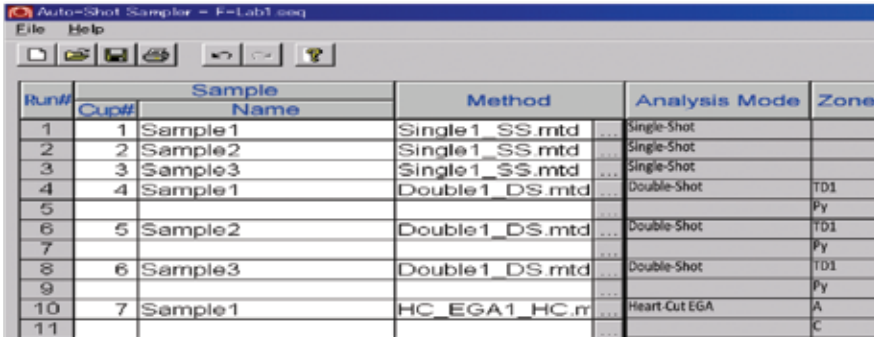
**Ultra ALLOY Metal Capillary Columns**  
Available with a number of high temperature stationary phase, column lengths, film thickness and column inner diameters. Multi-layer deactivation treatment enhances thermal stability, inertness and durability.  
*Technical Notes: UAT-002E, 003R and 004E*

**F-Search**

## Auto-Shot Sampler (AS-1020E)



Up to 48 samples can be analyzed. When performing multiple analyses on a single sample, the sample is held at near-ambient temperature between analyses. The sample path has one isolation valve and two magnetic cup positioning valves which are always at ambient temperature.



Run#	Cup#	Sample Name	Method	Analysis Mode	Zone
1	1	Sample1	Single1_SS.mtd	Single-Shot	
2	2	Sample2	Single1_SS.mtd	Single-Shot	
3	3	Sample3	Single1_SS.mtd	Single-Shot	
4	4	Sample1	Double1_DS.mtd	Double-Shot	TD1
5					Py
6	5	Sample2	Double1_DS.mtd	Double-Shot	TD1
7					Py
8	6	Sample3	Double1_DS.mtd	Double-Shot	TD1
9					Py
10	7	Sample1	HC_EGA1_HC.mtd	Heart-Cut EGA	A
11					C

The automated analysis of multiple samples requiring different analytical modes can be analyzed in a single sequence. Evolved gas analysis (EGA), which is done with a tube rather than a separating column, requires a separate sequence table.

# 3030D Accessories *for extended capability*

## F-Search Software (PY-1110E)

F-Search is used to identify individual compounds and/or polymers using a patented search algorithm and specialized Frontier MS libraries. The libraries include GC as well as MS chromatograms. Each library can be edited. In addition, custom in-house libraries can be created. Four libraries are available: polymer, pyrolyzates, additives and EGA thermograms.

The GC/MS data obtained with the instruments manufactured by Agilent Technologies, Shimadzu, and JEOL can be searched without modification. Data generated on GC/MS instruments manufactured by other companies can also be searched after converting the data file to a NetCDF (AIA).

If your system includes the NIST/EPA/NIH mass spectral library (National Institute of Standards and Technology) and its search engine software, it can be accessed directly from within F-Search.

### F-Search Software

#### Polymer Analysis

EGA library (Thermogram: 268 polymers) PY-1112E

Polymer library (Pyrogram: 268 polymers) PY-1113E

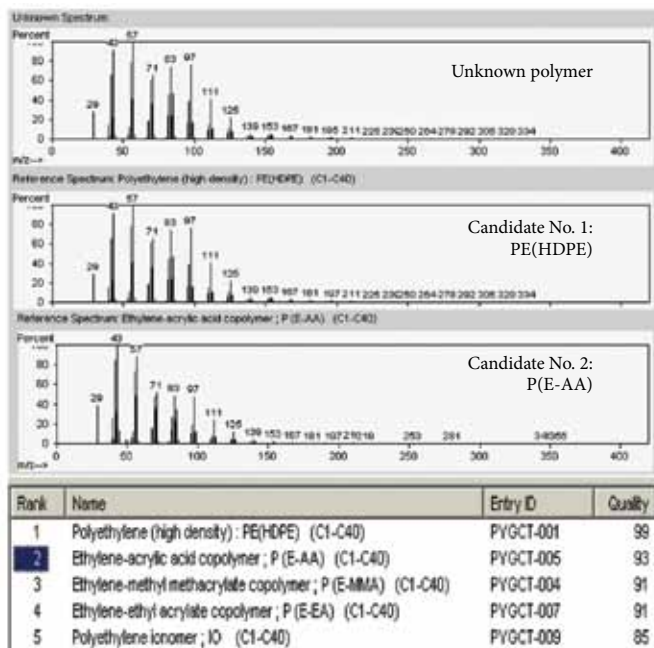
Pyrolyzates library (with chemical structures) PY-1115E

#### Additive Analysis

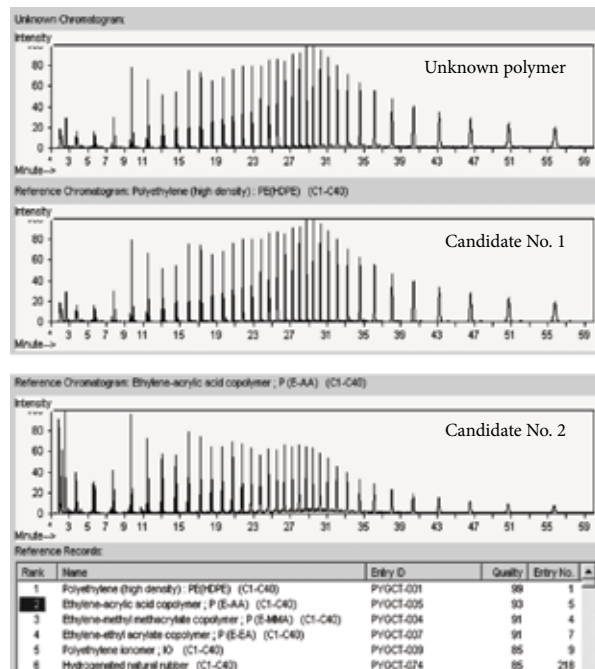
Additive library (chromatogram: 358 additives) PY-1114E

Technical Notes: PYA2-013E, 054E and PYA3-006, F-Search Product Bulletin.

The partial results for a typical library search are illustrated below.



The average MS spectrum of the unknown along with the averaged spectrum of the two best matches are shown. A table showing match quality is presented.



The pyrograms of the unknown and the two best matches are displayed for comparative purposes.



# Compatibility & Support

The EGA/PY-3030D Multi-Shot Pyrolyzer is compatible with major manufacturer's GC or GC/MS systems.

All Frontier channel partners receive periodic service training on all Frontier Lab products. This ensures that our customers receive quality support worldwide.

Visit our website to view a number of support videos including maintenance and F-Search demonstration.



[www.frontier-lab.com](http://www.frontier-lab.com)

*Information subject to change without notice.*

© 2013 Frontier Laboratories

IW130301 Ver. 3.00