



Sample-Stacking to Increase Sensitivity Using a CDS Pyroprobe

Application Note

Polymer

Author:

Karen Sam

Abstract

This application note introduces a sample-stacking technique to improve sensitivity for low concentration samples.

Introduction

CDS Analytical's DISC (Drop-In-Sample-Chamber) tube can hold a 10 μL sample volume or 10 mg sample weight with a density of 1g / cc. Typically, only a small fraction (1/100) of this volume is needed in the analysis of a pure polymer. However, if the polymer is diluted in an inorganic matrix like sand or glass fibers, as may be the case in micro-plastic and nano-plastic analysis, a higher volume of sample may be required. Cases in which over 10 mg is needed, the sample can be divided into multiple tubes and still analyzed with one GC run for an increase in sensitivity. For example, one entire 125 mm Whatman glass fiber filter deposited with nano-plastics may be cut and divided into two tubes for analysis in one GC run. This technique is referred to as sample-stacking.

A Pyroprobe 6200 with autosampler in trapping mode is used to demonstrate this technique. Two DISC tubes are pyrolyzed (sampled) sequentially to the sorbent trap while it is resting at an ambient temperature. After the pyrolyzing all the samples, the trap is desorbed while the GC run begins. In this application note, polystyrene was run in one DISC tube in trapping mode then compared to the same amount of polystyrene but divided into two tubes.

Experimental

Polystyrene was dissolved in toluene at a concentration of 10 μg / μL as a stock solution. Two microliters each were added to each of 5 glass fiber filters (Whatman GF/A) and allowed to dry. Each filter was cut in half and placed into 2 DISC tubes for the sample-stacking experiment. Two Pyroprobe methods were run in a sequence for one GC run. The first method pyrolyzed Tube 1 to the sorbent trap of the 6200 while trap remained at an ambient temperature and the GC was not started. The second method pyrolyzed the second tube to the trap at an ambient temperature, after which, the trap desorbed contents of both tubes to the GC and the Pyroprobe sent the GC Start signal.

Pyrolysis

Pyroprobe

Post-Pyro Delay: 0s

Tube 1:

DISC: 550°C 30s
 Trap Rest: 45°C
 Trap Final: 45°C 0s
 Interface: 300°C
 Transfer Line: 325°C
 Valve Oven: 300°C
 Use GC Ready: ON
 Issue GC Start: OFF

Tube 2:

DISC: 550°C 30s
 Trap Rest: 45°C
 Trap Final: 300°C 3min
 Interface: 300°C
 Transfer Line: 325°C
 Valve Oven: 300°C
 Use GC Ready: ON
 Issue GC Start: ON

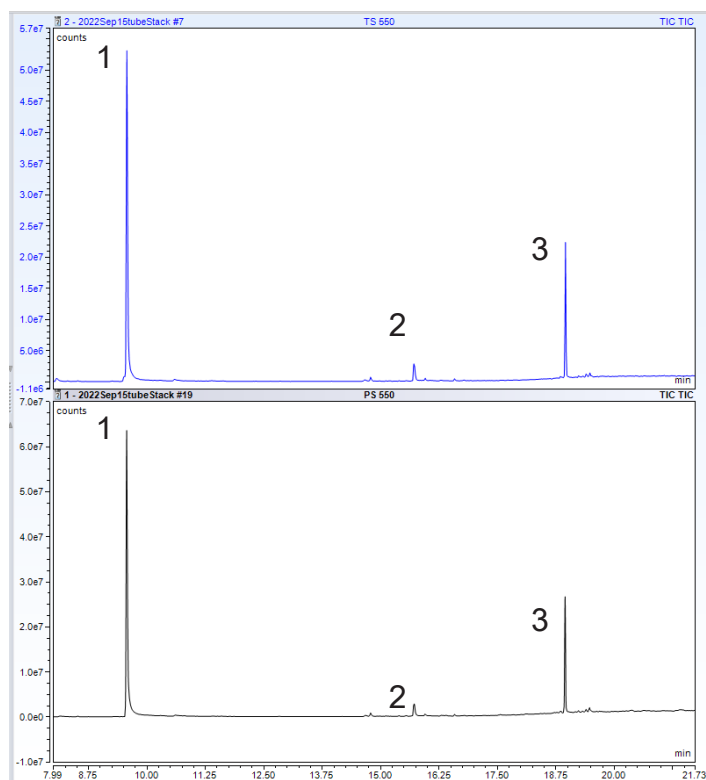


GC-MS

Column: 5% phenyl (30m x 0.25mm)
Carrier: Helium 1.25mL/min
15:1 split
Injector: 360°C
Oven: 34°C for 4 minutes
12°C/min to 320°C
hold 15min
Ion Source: 230°C
Mass Range: 35-600amu

Results & Discussion

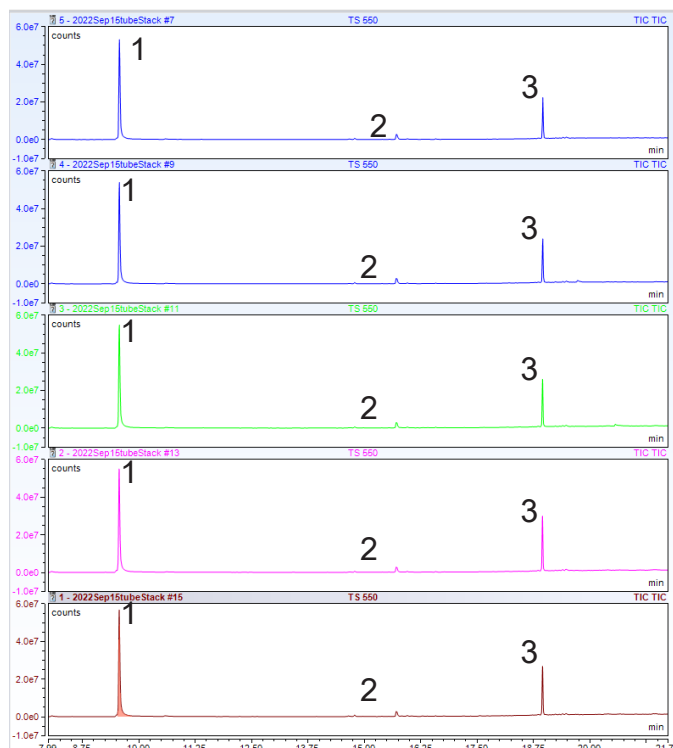
Figure 1 compares the pyrolysis of Polystyrene in one tube versus the same amount divided into two tubes using the sample stacking method. The average area of the styrene peak 5 stacked tubes was 96% of the area of the styrene peak in the single tube, indicating excellent recovery.



Peak	Identification
1	Styrene
2	Styrene Dimer
3	Styrene Trimer

Figure 1. Pyrolysis of Polystyrene (20 µg) divided into 2 DISC tubes (top), and in one DISC tube (bottom).

A styrene area RSD under 6% was acquired after five sample-stacking replicates (Table 1).



Peak	Identification
1	Styrene
2	Styrene Dimer
3	Styrene Trimer

Figure 2. Tube Stacking Replicates.

Table 1. Styrene Area Replicates.

Replicate	Styrene Area
1	1999322
2	2012680
3	2169051
4	2191710
5	2287111
RSD %	5.78

Conclusion

While pyrolysis of polymers often requires under 100 µg sample sizes, there are occasions when larger sample sizes are needed. For example, nano-plastics may be in low concentrations in an inorganic matrix such as sand, silica or ground glass fiber filters. Additionally, one entire 125 mm Whatman glass fiber filter deposited with nano-plastics may be cut and divided into two tubes for analysis in one GC run.