

## Points to Consider in Obtaining Pyrograms by Py-GC/MS in Air Circumstance [About Separation Column and Air Purge Time in MS]

**[Background]** When obtaining a pyrogram by Py-GC/MS in air, after the pyrolysis of samples the separation column and the MS must be purged with He before GC/MS analysis is started. The time which takes for purging varies on the type of GC/MS used, the kind of separation columns, and carrier gas settings. Here, the time which takes for purging the system with settings commonly used in Py-GC/MS analyses was studied.

**[Experimental]** A Double-Shot® Pyrolyzer directly coupled with the split/splitless injection port of a GC was connected to a MS (HP5791, vacuum pump capacity 60L/sec) via a capillary separation column (30m, id 0.25mm). Air was used for the atmosphere gas during pyrolysis. A Selective Sampler was attached underneath the injection port, so that the gas flowing into the separation column was able to be switched from air to He any time. The injection port pressure was set to 100kPa.

**[Results]** After air was run through the separation column for a minute, the gas flow was switched by using the selective sampler, and He gas was allowed to flow into the separation column and then into the MS ion source. Fig. 1 shows a TIC plot against the time for purging. Initially air existed in the system, therefore a elevated baseline was observed. As the air purging progressed, the baseline is gradually lowered and in about 3 min it was leveled off. This result showed GC/MS analysis must be started after 3 min of purging or more, if air is allowed to flow into the system for 1 min. In a similar manner, the relation between the air flow time and required purging time was examined and the result obtained is shown in Fig. 2. Up until 20 min of air flow time, the air flow time and purging time were in monotonously increasing relationship, however, beyond 20 min, it was found that the required purging time stayed constant at 20 min.

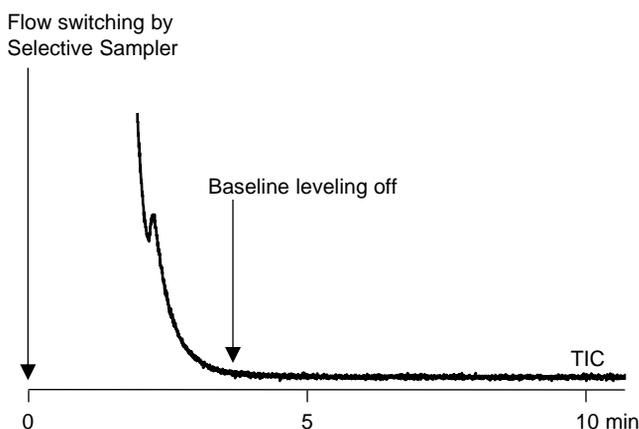


Fig. 1 Baseline shift after air was run through the column

Column inlet pressure: 100 kPa, Split ratio: ca. 1/50, Separation column: Ultra ALLOY (30 m, id 0.25 m), GC oven temp: 40°C, MS scan range: 29-100 (m/z), scan rate: 2 scans/sec

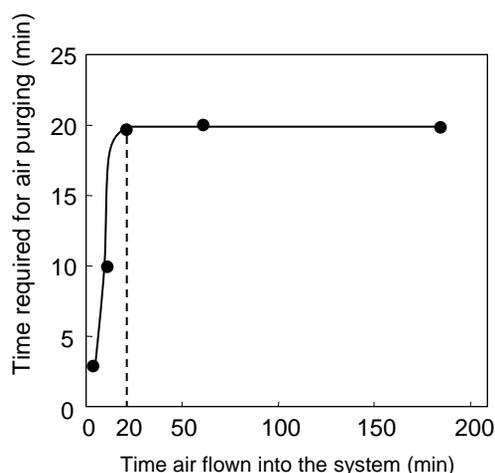


Fig. 2 Relationship between air flow time and time required for system purging

**Keywords :** Pyrolysis in air,

**Products used :** Multi-functional pyrolyzer, Carrier gas selector, Selective sampler

**Applications :** General polymer analysis, Environmental analysis, Work environmental analysis

**Related technical notes :** PYA4-001E, PYT-022E, PYT-024E

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