Procedures for Acquiring Pyrograms in Air and Its Automation

[Background] To analyze the processes of thermal oxidation/degradation of polymers, air needs to be used as the atmosphere gas in analyses using a Py-GC/MS system. In this case, when the pyrolysis or thermal desorption in air is completed, the column and MS vacuum system are purged with helium gas before the GC/MS analysis is started in order to prevent oxidative degradation of separation column and mal-functioning of MS. This, however, involves complicated procedures. Here, general procedures for obtaining pyrograms in air atmosphere and an advanced automated Py-GC/MS system are briefly described.

[Experimental] Fig. 1 shows the construction of a Py-GC/MS system used. A Double-Shot® Pyrolyzer directly coupled with a split/splitless injection port was further connected to an MS through a separation column. A carrier gas fed to the pyrolyzer was provided through a carrier gas selector, permitting air or helium to be selected. Underneath the injection port, a selective sampler was installed, allowing the sample gases from the injection port to flow into the separation column or to be exhausted. Also, at the front of the separation column, a MicroJet Cryo-Trap was installed in order to chill the portion of the column down to near -196ºC so as to cold-trap volatile organics formed.

[Results] Table 1 summarizes operational sequences of devices required when obtaining pyrograms in air in manual operation and in an automated system. In manual operations, a series of complicated operations A~C are required for an operator to perform. On the other hand, in the automated system, all the operations beyond A-2 are performed automatically. This simplifies whole operations and eliminates oxidative degradation of the separation column due to miss-operations, inadequate analysis due to incomplete trapping, or contaminations and deteriorations of the system.

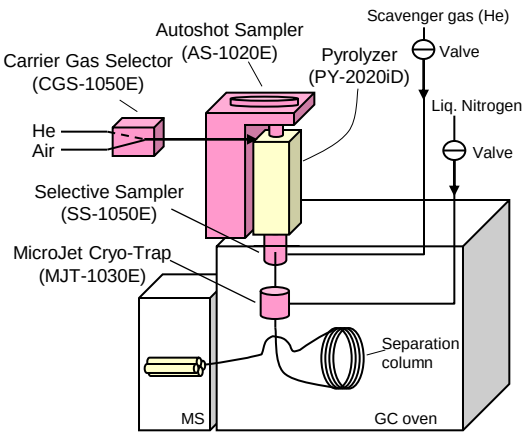

Figure 1. Automated oxidative pyrolysis and pyrogram acquisition system

Table 1. Required operations for Py-GC/MS analysis in air

Required sequence of operations		Manual operations	Automated operations
A. Prep. for sample introduction	A-1. Introduce air into pyrolysis furnace	Switch carrier gas lines	Switch gases fed to Py (CGS-1050E) Switch gases fed to column (He → Air, SS-1010E)
	A-2. Cool separation column to cold trap	Immerse head of column into liq. N_2	Start chilling column head by MJT-1030E
B. Heating sample	B-1. Heat sample in air (thermal desorption, pyrolysis)	Introduce sample into furnace	Drop sample into furnace
	B-2. Cold trap organics formed	Wait until heating time is over	Hold until operation is complete
C. GC/MS analysis	C-1. Switch gases fed to separation column (Air → He)	Switch back carrier gas lines	Switch gases fed to colum (Air → He, SS-1010E)
	C-2. Purge air from the system (several minutes to half hour)	Wait until required time is reached	Maintain state until operation is complete
	C-3. Finish cold trapping	Remove separation column from liq. N_2	Stop chilling (MJT-1030E)
	C-4. Start temp. programmed GC/MS analysis	Start GC/MS	Issue start signal to GC from Py temp. controller

Automatically repeatable

Keyword : Air atmosphere, Automation

Applications : General polymer analysis, Environmental analysis

Related technical notes : PYA4-001E, PYA4-002E, PYA4-003E, PYA4-004E, PYT-019E, PYT-023E

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