

Comparison of Free-Fall Sample Introduction versus Sliding Sample Introduction

In pyrolyzers with a heating furnace, pyrogram patterns are greatly influenced by the rate of sample introduction into the heating furnace. In order to attain a good reproducibility, the sample introduction into the furnace must be done in a shortest period of time at a constant rate in each run. The sample introduction by the action of gravitational free-fall system developed by S. Tsuge (see Fig. 1A)¹⁾ allows rapid sample introductions at constant rate. Table 1 compares the reproducibility of polystyrene pyrograms (see Fig. 2) obtained by a sliding system (manually sliding sample into furnace, see Fig. 1B) and by a free-fall system. When the sample was manually inserted into the furnace in about 1 sec and 2 sec, the reproducibilities of formation of styrene monomer and styrene trimer were 2.43% and 6.12%, respectively. On the other hand, the free-fall system was found to give an excellent reproducibility of 0.63%.

1) S.Tsuge, & T.Takeuchi, Analytical Chem., 1977, 49, 348-350

Table 1 Comparison of Reproducibility of PS Pyrograms Obtained by Sliding and Free-fall Systems

Sample Introduction	SSS/S (%)	RSD (%)
Sliding system (1sec)	11.78	2.43
Sliding system (2sec)	11.65	6.12
Free-fall system	13.85	0.63

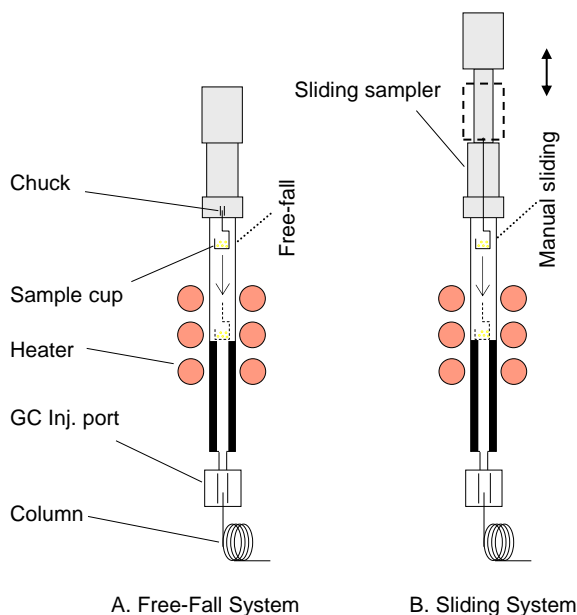


Fig. 1 Free-fall and Sliding Systems

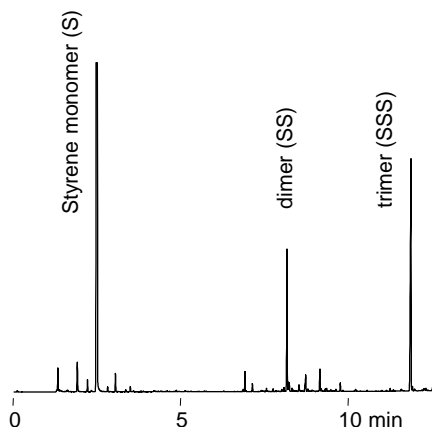


Fig. 2 Pyrogram of PS Obtained by Free-fall System

Sample : 30µg, Pyrolysis temp.: 550°C, He pressure : 140kPa, Split ratio : 1/50, Column : 5% diphenyl polysiloxan, 30m, 0.25mm id, Film thickness : 0.25µm (Ultra ALLOY⁺-5, Frontier Lab Ltd.) GC oven temp.: 70-320°C (20°C/min)

Keywords : Free-Fall System, Pyrolysis Speed, Reproducibility, Polystyrene

Products used : Multi-functional pyrolyzer, UA-5

Applications : General Polymer Analysis

Related technical notes :

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