

[Highly sensitive detection of polystyrene by on-line splitless pyrolysis-gas chromatography/mass spectrometry with cryo-trapping of pyrolyzates and forced venting of carrier gas](#)

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Abstract:

Pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS) has been extensively used as a powerful tool for polymer analysis. In Py-GC/MS measurements, GC methods have usually employed split injection conditions for qualitative and quantitative analysis, but splitless injection of pyrolyzates to GC/MS has been reported to obtain higher sensitivity for small amount of analytes, since split ratio is inversely related to the amount of analyte entering the detection systems. However, the slower total carrier gas flow rate of splitless injection, which is necessary to keep the column flow rate at around a few mL/min, can increase residence time in the pyrolysis chamber, which can cause unfavorable secondary reactions of pyrolyzates such as decomposition and recombination in the pyrolyzer. In this study, we developed a new on-line splitless Py-GC/MS method called the Forced-Venting-and-Trap-Splitless (F-Splitless) method, based on cryo-trapping of pyrolyzates and forced venting of carrier gas under high carrier gas flow rate. Calibration curves measured by the F-Splitless method for polystyrene (PS) using the styrene monomer and trimer as key compounds show a good linearity over the concentration range from 0.01 ng to 100 ng. Limit of detection (LOD) and limit of quantification (LOQ) were 17 pg and 56 pg for the styrene monomer (S) based on the criterion of LOD: $3\sigma/a$ and LOQ: $10\sigma/a$, where a is the slope of the calibration curve and σ is the standard deviation of the peak area of the minimum concentration, using the selected ion monitoring (SIM) mode. At a trace concentration level of PS, contamination from the environment was recognized. However, 100 pg of PS showed a signal to noise ratio (S/N) of 570 for S in SIM mode and 1.8 pg of PS could be quantified according to the criterion of LOQ based on signal to noise ratio (S/N), i.e., $S/N = 10$.

* Excerpted from online journal website (Click the title)

Frontier Labs Products used:

Multi-Shot Pyrolyzer (EGA/PY-3030D), Auto-Shot Sampler (AS-1020E), Ultra ALLOY+-5, Vent-free GC/MS adapter, MFS-2015E, MJT-1030E, F-Search