Evolved gas analysis-mass spectrometry in an oxidative atmosphere using a temperature-programmable furnace-type pyrolyzer

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Abstract:

Evolved gas analysis-mass spectrometry (EGA-MS) has been used to characterize the effect of heating a material in an oxidative or non-oxidative environment. Measurements in an oxidative environment are usually done by using the mixture of air and helium (He) as the carrier gas; however, ionization efficiency of evolved gases in a mass detector (MSD) decreases in such an environment, resulting in a corresponding decrease in sensitivity. The drop in sensitivity can be reduced by diluting the air in the carrier gas with He after evolving gases from a sample and before the evolved gases flow to the MSD. This study reports on a simple modification of the gas inlet system so that experiments requiring oxidative conditions in an air atmosphere can be routinely performed without loss of the MSD sensitivity. The impact of using this modification on the data is demonstrated using polystyrene (PS) as a test sample. EGA curves were acquired in two flow configurations: (1) adding He to the carrier gas immediately after the furnace, which reduces the concentration of nitrogen and oxygen in the carrier gas; thus, minimizing the quenching of the ionization and the associated decrease in signal-to-noise ratio (S/N) and (2) the standard configuration which simply uses a mixture of He and air as the carrier gas. Data obtained using the modified method had a much high S/N than that obtained using a gas mixture (He and air or He and oxygen) as a carrier gas. The RSD ($n = 5$) values for the peak area and the peak temperature of the EGA curve of PS were 1.6 and 0.60 % respectively, and these values were comparable with those obtained using a He carrier gas. The modified gas inlet system was also applied to the analysis of isotactic polypropylene.

Frontier Labs Products used:

Multi-Shot Pyrolyzer (EGA/PY-303D), UADTM-2.5 N, Vent-free GC/MS adapter