PYROLYSIS GC-MS

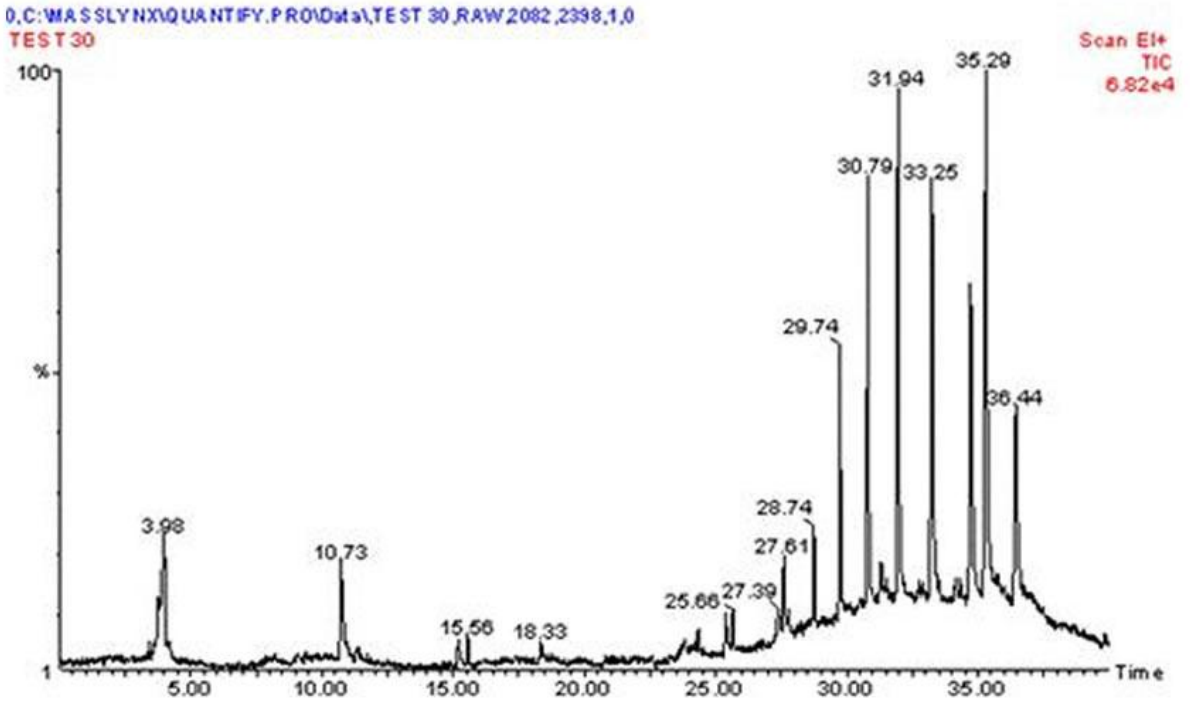
CDS TEMPERATURE PROGRAMMABLE PYROPROBE WITH TRIO 2000 GC-MS

In order to understand the decomposition of a polymer or the mode of action of a fire retardant, it is normally necessary to understand the chemistry behind the real-scale observations. This is a simple controlled temperature pyrolysis experiment, which allows pyrolysis products to be analysed by Gas Chromatography (GC), GC-MS, or using a different cell, infrared spectroscopy. In this way both gases and condensable material can be analysed.

PYROLYSER

The Pyroprobe has the ability to perform reactant gas pyrolysis, headspace trapping of volatiles on a built-in analytical trap, and thermal desorption analysis from sorbent tubes. It can pyrolyze the sample and send it directly to the gas chromatograph (configuration 2), slowly heat the sample and trap the volatiles on the analytical trap prior to analysis (configuration 1), or perform pyrolysis using a reactant gas, such as air, oxygen, or hydrogen (configuration 1). The analytical trap can be replaced with a sorbent tube and analysed by thermal desorption.

When using the trap of the 5200, carrier gas from the probe zone goes through the trap during pyrolysis, and the collected products are transferred to the GC by desorbing the trap. This permits slow heating rates with collection of the pyrolyzate and the use of a reactant gas that is not introduced into the carrier gas stream and thus the GC. The Pyroprobe can be heated in multiple steps during a sample analysis.



The pyrolysis probe allows different heating rates and atmospheres to be used from 1°C min-1 to 1000°C s-1. This technique is ideal for identification of the widest range of species present in a pyrolysis gas over a present temperature range. In the example below, a copolymer of acrylonitrile and diethyl(acryloyloxy-1-ethyl) phosphonate (DEAEP) was decomposed in nitrogen at 10°C min-1 and the volatiles were collected between 250-300°C. One of the active flame retardant species resulting from the decomposition of the DEAEP comonomer is diethylphosphite, seen here on the GC trace, and identified from its mass spectrum by matching with the NIST library.

Pyrolysis GC-MS complements TGA-FTIR as it provides snapshot identifying all evolved species over a particular temperature range. TGA FTIR shows the variation of the spectrum as a function of temperature, once identified; the temperature evolution profile of a species may be monitored.