GAS
MESS- UND ANALYSENGERÄTE

Pyrolysis Application Short Message No. A002

Pyrolysis GC/MS of synthetic resins

Resins are solid, hard up to soft, organic non crystalline products with more or less wide molecular weight distribution. A distinction is drawn between natural and synthetic resins. The major group are the synthetic resins which were produced by polyaddition, polymerization or polycondensation of monomers.
Typical synthetic resins are epoxy resin, formaldehyde resins for example melamine formaldehyde resin and urea-formaldehyde resin and polyester resin. Epoxy resins were produced by addition of epichlorohydrin and bisphenol A. The application area are predominantly high quality components, coating and adhesion. Melamine formaldehyde resins were produced by reaction of melamine and formaldehyde to hexahydroxy-methylene melamine and following condensation by longer reaction time and higher temperature. Areas of application for this resin are: binder in paper laminate and lacquer and sockets. Polyester resins (saturated and unsaturated) are made by reacting of glycols (for example propylene and diethylene glycols) with dicarboxylic acid (for example maleic and phthalic acids).

These resins are typical exponent of duromere, synthetic resins with high net structure which are not solve in solvents and not ductiled by higher temperature.

As a result of this characteristics is the analytical investigation with pyrolysis coupled with GC or GC/MS a important tool.

The measurement of epoxy resin shows the following gas chromatogram (Figure 1). The sample was placed into a tube shaped ferromagnetic sample carrier with the Curie point temperature of 764°C. By this temperature the sample was pyrolysed for 10 seconds. The volatile fragments of the resin which was build during the pyrolysis process were analysed by the gas chromatography- mass spectrometry. The pyrolyzate shows the characteristic fragments of the epoxy resin (for example: bisphenol A and compounds with one or two epoxide groups). Additional was found a signal of the reactive diluent styrene.

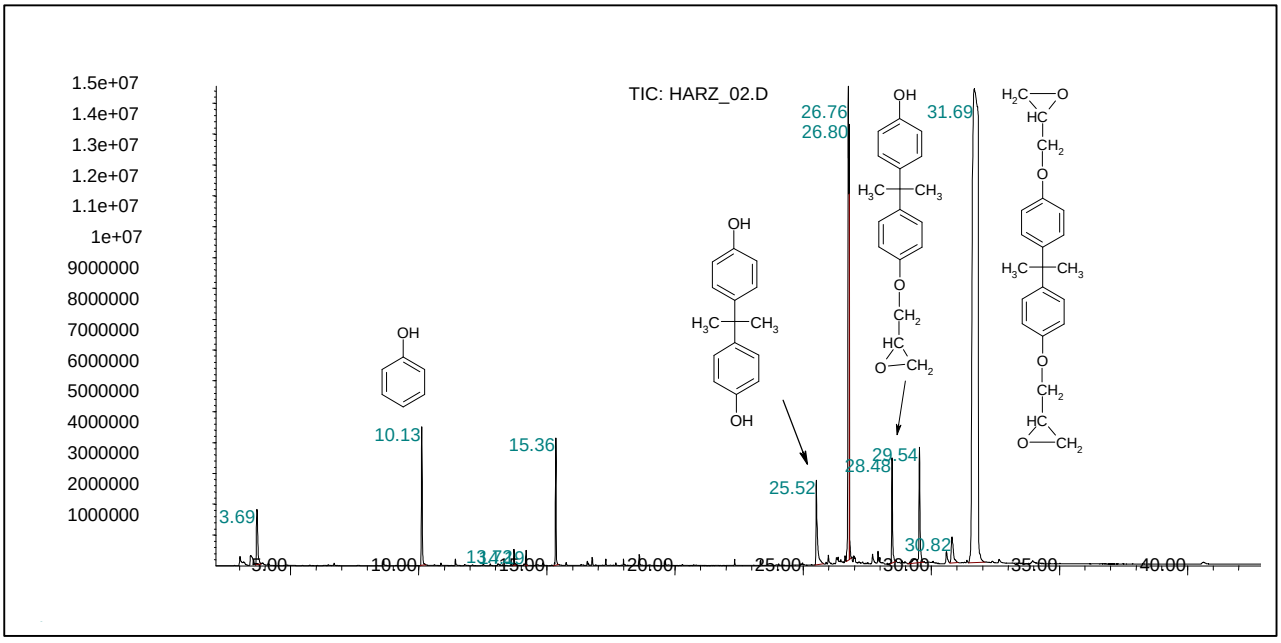

Figure 1: Sample of epoxy resin, pyrolysis temperature 764° C, pyrolysis time 10 sec

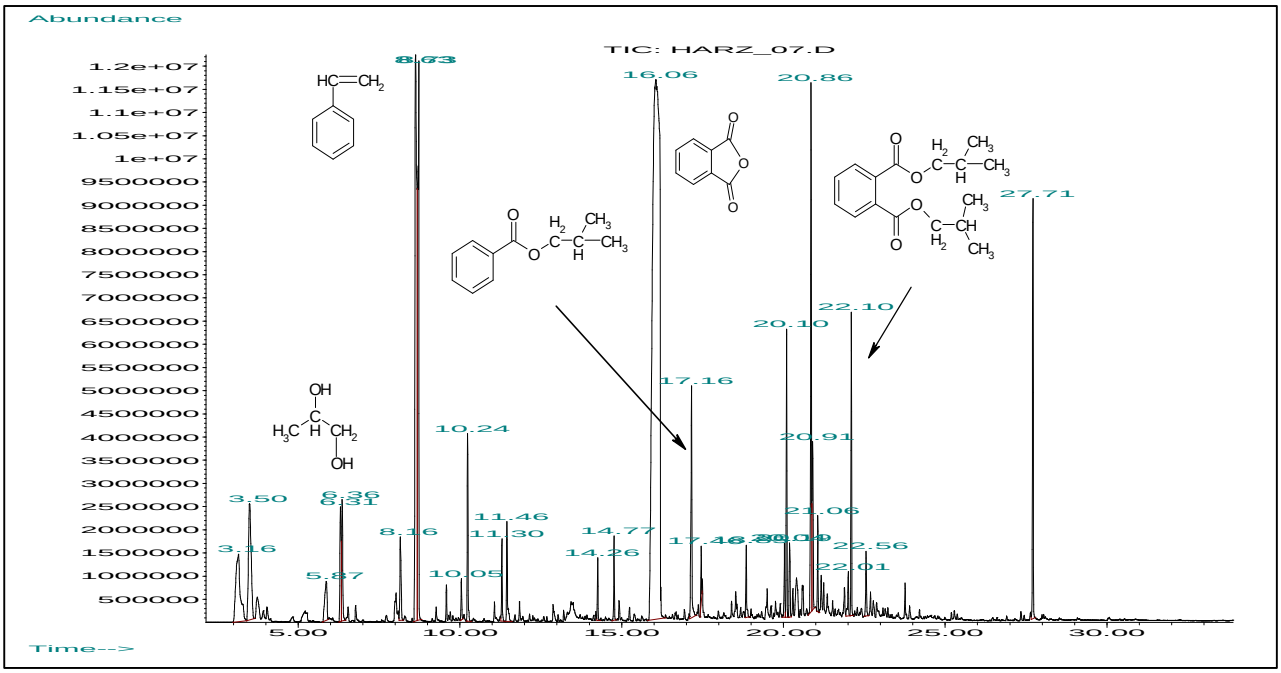

Figure 2: Sample of unsaturated polyester resin, pyrolysis temperature 650°C, pyrolysis time 10 sec

Figure 2 shows the spectra of unsaturated polyester resin. For this resin was used a pyrolysis temperature of 670°C and a pyrolysis time of 10 seconds. In this spectra were found signals of the raw material propylene glycol and phthalic anhydride, fragments of the resin [Benzoic acid, 2-methylpropylester and 1,2-Benzene dicarboxylic acid bis (2-methylpropyl) ester] and of the reactive diluent styrene.

Instrumentation

Pyrolysis:

- Pyrolyser Pyromat, Autosampler with 24 positions
- Pyrolysis temperature of 650°C and 764°C
- Pyrolysis head and interface temperature 180°C and 200°C
- Pyrolysis time 10 second

Gas chromatography

- HP GC 6890
- Initial temperature: 50°C for 2 minutes
- Ramp rate: 10°C/min. to 315°C for 16 minutes
- Column: 50m x 0.22mm BPX5
- Carrier gas: Helium
- Split: 20:1
- Helium flow 1 ml/min

Mass spectrometry

- HP GC/MS 5973
- Transfer line 280°C
- Mass range 12 – 550 amu

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