

Self-built low-cost pyrolyzer using Curie-Point temperature coupled to a gas chromatograph-ion mobility spectrometer

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Summary:

Pyrolysis is a well-known decomposition technique under inert gas for the analysis of liquids and solids by analyzing the volatile decomposition products. This work presents the design of a low-cost pyrolyzer based on the Curie-Point method. Using a 1 mm diameter Fe50Ni50 wire, the pyrolyzer heats up the wire carrying the sample to 520 °C within 3.71 s, verified by an optical infrared thermometer. For demonstration, this pyrolyzer was coupled to a gas chromatograph-ion mobility spectrometer (GC-IMS) and tested with Actimel yoghurt samples.

Keywords: Pyrolysis, Gas chromatography, Ion mobility spectrometry

Background, Motivation and Objectives

For onsite quantification of volatile compounds in more complex backgrounds, a mobile gas chromatograph-ion mobility spectrometer (GC-IMS) is a good choice, as it provides decent separation power and low limits of detection in the pptv-range while the instrumental effort is comparatively low. However, low- or non-volatiles need to be transferred into the gas phase before analysis by GC-IMS, e.g., by thermal desorption. Another well-known method is pyrolysis.

Pyrolysis is defined as the thermal conversion process or decomposition of a sample in the absence of oxygen at temperatures exceeding 250 °C. The exclusion of oxygen prevents sample combustion. Over time, three methods of heating the sample until chemical bonds break and more volatile decomposition products are released have been established: Inductively (including the Curie-Point method) and resistively heated filaments as well as furnace pyrolyzers.

The Curie-Point method is explained in detail by Sobeih et al. [1]. It uses ferromagnetic wires inductively heated by a time-varying magnetic field. These wires are self-regulating at their specific Curie temperature, and thus allow for fast temperature ramping to their Curie temperature without any external temperature control.

As commercially available pyrolyzers are large in size and therefore not suited for mobile applications, we present a simple, miniaturized and low-cost setup.

Design

The Curie-Point method has been chosen due to the low instrumental complexity and the excellent reproducibility as a consequence of the used physical effect for end temperature control.

The Sectional view of the pyrolysis unit is visible in Figure 1. A 1 mm diameter ferromagnetic wire is used to minimize thermal mass. A glass liner from Agilent (5190-2292) flushed with nitrogen surrounds this wire. Around the glass liner is a single-wound copper coil with an inner diameter of 10 mm with a total of 15.5 windings. The coil has a total inductance of 600 nH.

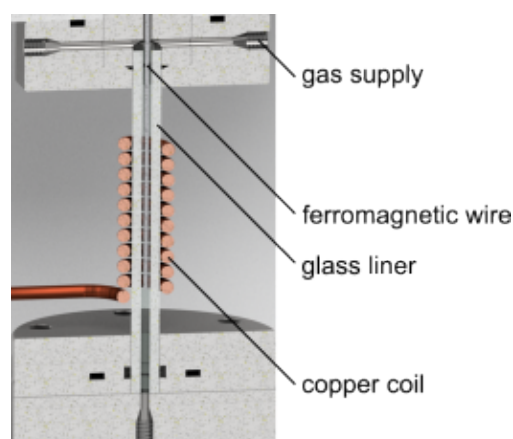


Figure 1 Sectional view of the pyrolysis unit structure

The generated pyrolysis products are transferred through a heated transfer line into the subsequent GC-IMS within pure nitrogen. The IMS is an ultra-fast polarity switching PCB-IMS with dual drift tubes for simultaneous recording of

positive and negative ions, as described by Hitzemann et al. [2].

The magnetic field in the coil is generated by a full bridge inverter with external frequency generation. A voltage-controlled oscillator allows for an adjustable frequency from 340 Hz to 1 MHz to control the full bridge inverter, which is fed by a supply voltage of up to 42 V.

Results

The rise time of the wire temperature and the temperature stability at Curie temperature were measured through the glass wall of the glass liner and a small gap between the windings of the coil using an optical infrared thermometer from Optris (CTlaser 3MH1 with CF2 lens giving a measurement spot of ≤ 0.5 mm and fast temperature recording rate of 1 kHz). The rise time is defined as the time required for heating from 10% to 90% of the Curie temperature beginning at ambient temperature. Unfortunately, the temperature measurement range of the used infrared sensor starts at 150 °C, which is above the 10% of the Curie temperature of the used wire, so that the rise time was estimated by using a linear regression fit function derived from the measured temperature ramp in the range from 150 °C to 467 °C. The heating process is considered to be completed when the slope of the temperature ramp falls below 10^{-3} K/s. The end temperature is calculated as the mean value of the following thousand measuring points. Figure 1 shows a measured heating curve of a 1 mm diameter and 60 mm long Fe50Ni50 wire. The final temperature of 520 °C matches the specified Curie temperature of 520 °C. The full bridge inverter is operated at 500 kHz and supplied with 20 V giving 60 W of heating power. The temperature rise can be estimated to 3.7 s with a linear temperature ramp of 118.6 K/s from ambient temperature to 90% of the final temperature.

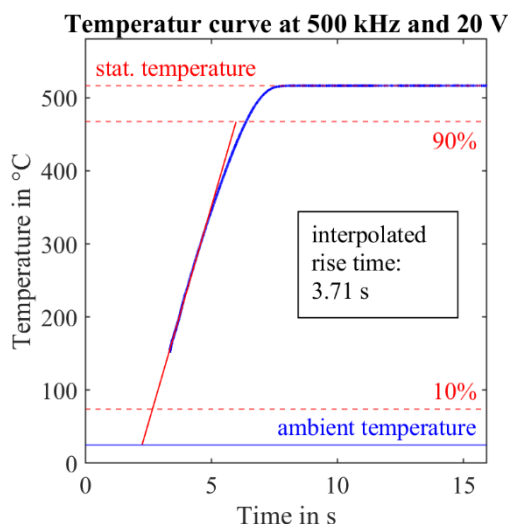


Figure 2 Heating curve of the Fe50Ni50 wire. The end temperature of 520 °C matches the specified Curie

temperature of 520 °C. The full bridge inverter is operated at 500 kHz and supplied with 20 V.

For demonstration of general feasibility, the developed pyrolyzer is coupled to a GC-IMS. Figure 2 shows the topographic plot of the measuring results when analyzing an Actimel yoghurt sample. Therefore, the previously described wire was simply dipped into yogurt and was directly transferred into the pyrolysis unit.

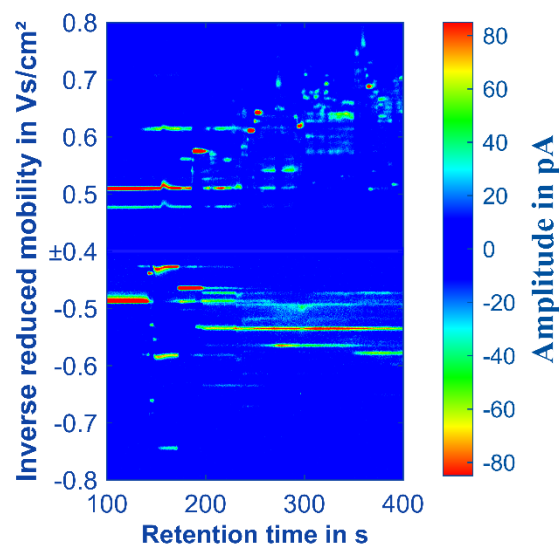


Figure 3 Topographic plot of an Actimel yogurt sample analyzed with Pyrolysis-GC-IMS.

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