A novel storage interface for TGA-GC/MS analysis

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Introduction

GC-MS is a powerful technique to analyze gases evolved during a TGA experiment when many different decomposition products may be released simultaneously. In this situation the identification of the evolved gases by MS or FTIR can be extremely difficult. By GC/MS, the different compounds will be separated by GC and then identified by MS. However, this requires that the gas samples representing the evolved gas composition at a certain temperature can be injected into the GC/MS. We present a novel TGA-GC/MS interface which can store up to 16 gas samples collected during a TGA experiment. The collection temperatures can be specified by the user. The collected gas samples will be analyzed after the TGA experiment by GC/MS. The analysis parameters both for the GC and the MS can be set for each stored gas sample individually. We present one application example illustrating the benefit of GC/MS in combination with this novel interface.

Example nitrile butadiene rubber NBR

Contrary to the manufacturer's specifications, synthetic rubber such as nitrile butadiene rubber (NBR) is often contaminated with natural rubber (NR). This is a problem because NR is a strong allergen and triggers allergic reactions with many people. The experiments presented here describe the investigations performed to identify the components of an allegedly pure NBR sample. DSC and dynamic mechanical measurements show clearly two glass transitions. This indicated the presence of an elastomer blend. TGA-GC/MS analysis was therefore performed to identify the unknown component responsible for this.

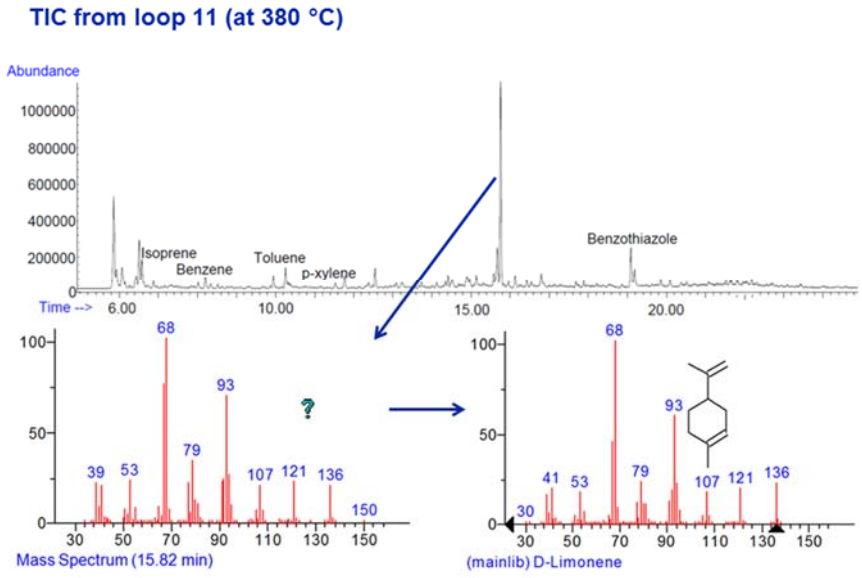

Figure 1: Total ion chromatogram (TIC) for loop 11, the MS at 15.82 min and its identification as D-Limonene.

Evaluation of the MS analysis results showed that the main component of the elastomer was NBR (2-propene nitrile and 2-methyl-2-propene nitrile as typical decomposition products of NBR).

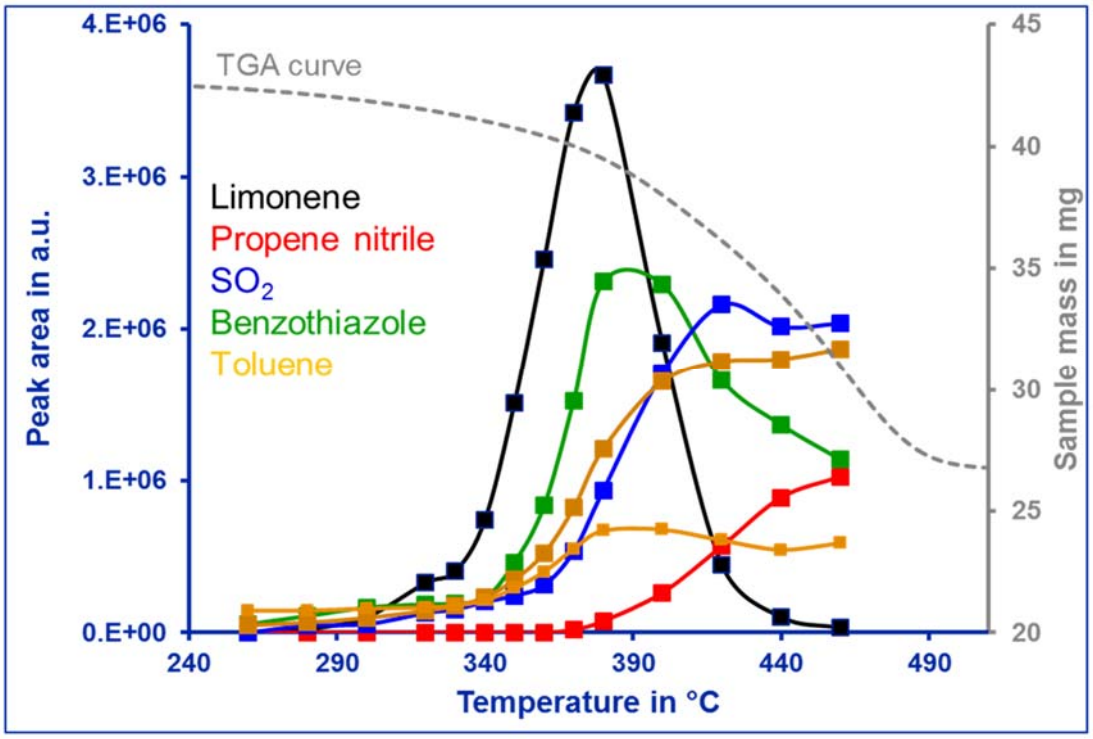

Figure 2: Emission profile of the main specific compounds.

Further main substances in the gas mixtures were limonene, isoprene and xylene (Fig. 1). Figure 2 shows the emission profiles of different mean identified decomposition products.

Limonene is a cyclic monoterpene with ten carbon atoms and is a typical decomposition product of NR. The presence of limonene eliminates the possibility of synthetic isoprene rubber as an impurity in the elastomer sample. The second component of the elastomer was therefore clearly identified as NR. This result would not have been possible by DSC analysis on its own.