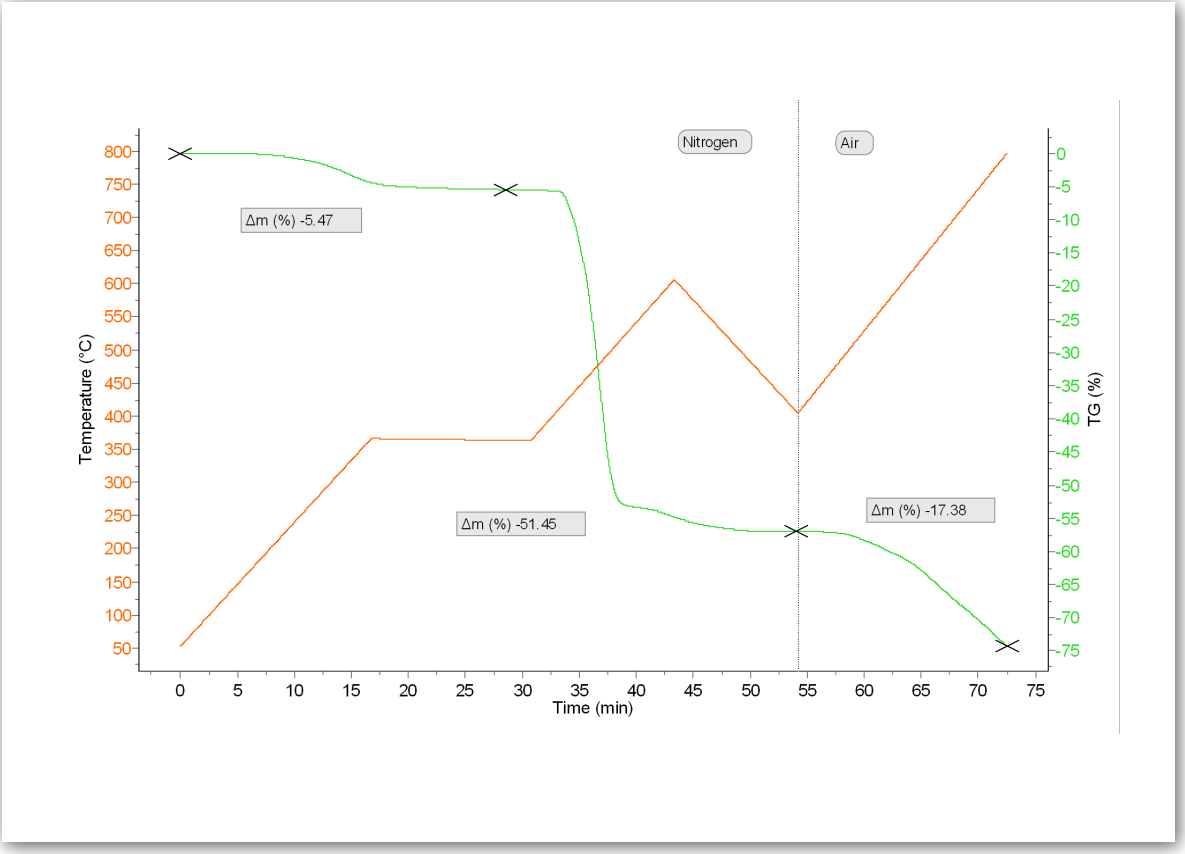
Compositional analysis of rubber by TGA

INTRODUCTION

TGA is frequently used to determine the content of polymers, rubbers, elastomers and related materials with regards to plasticizers/oil, polymer content, carbon black filler content, white filler content, and residual content. Indeed, the measurement of weight loss over specific temperature ranges and atmosphere program provides an indication of the composition of the sample, including volatiles and inert filler, as well as indications of thermal stability.



EXPERIMENT

The compositional analysis is directly obtained from the thermogravimetric test but in order to separate the rubber components, the test has to be performed firstly under nitrogen to decompose the organic matter and then under oxygen to burn the carbon content.
The following experimental procedure was used in the present example:

- A small piece (35 mg) of the rubber sample was placed into an alumina crucible
- A nitrogen flow rate (30 ml/min) was applied
- The sample was heated from 50°C to 350°C at 20°C/min
- The temperature was stabilized at 350°C during 15 minutes
- The sample was heated from 350°C to 600°C at 20°C/min
- The sample was cooled down to 400°C at 20°C/min
- Nitrogen flow was changed to air (30 ml/min) and the sample was heated up to 800°C at 20°C/min

RESULTS AND CONCLUSION

In a compositional analysis test, the mass losses observed are used to determine the amounts of organics (oil, polymer), carbon black and ash (filler) in a rubber compound. For the investigated rubber material the following results were obtained:

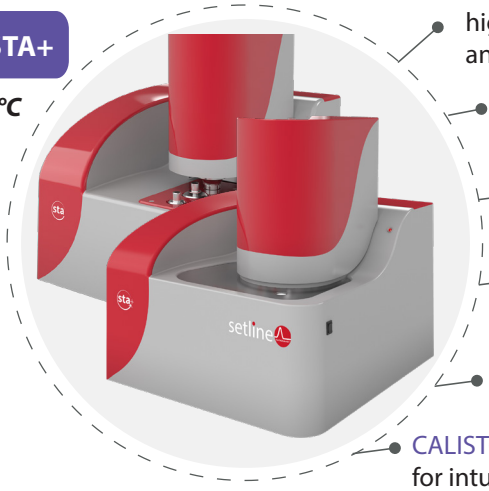
- from 50°C to 350°C under nitrogen, the mass loss is due to the decomposition of plasticizer, oil and wax content : 5.47%
- from 350°C to 600°C under nitrogen, the mass loss is due to the decomposition of the elastomer content : 51.45%
- from 400°C to 800°C under air, the mass loss is due to the combustion of the carbon black content : 17.38%
- The remaining mass at the maximum temperature is the amount of ash (containing fillers) in the samples: 25.38%

Setline STA is very well adapted for the investigation of the decomposition of polymeric materials and especially for their compositional analyses. For such a test, Setline STA provides a very flexible heating program for the definition of the different heating and cooling sequences, together with a very efficient and automated gas switching device.

INSTRUMENT

SETLINE STA / STA+

Ambient to 1100°C



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