

THERMAL SOLUTIONS

Rapid Determination of Carbon Black in Elastomers

PROBLEM

The addition of carbon black to an elastomer can have a positive effect upon its mechanical, electrical and physical properties. However, since carbon black is far more expensive than the elastomer master batch, a balance is usually sought between product performance and profitability during this addition. Hence, a test to rapidly determine the carbon black content of elastomer formulations is desirable. Furthermore, the shorter the analysis time, the sooner the data can be used to adjust raw material components to the blender.

SOLUTION

Thermogravimetric analysis (TGA), a thermal analysis technique which measures weight changes in a material as a function of temperature, provides a rapid method for quantifying the gross composition of polymer formulations. By heating elastomers in the TGA under nitrogen to 600°C, and then switch-

ing to oxygen at 800°C it is possible to determine the amounts of oils, polymer, carbon black and inorganic filler present. In the example elastomer chosen here, no fillers were found to be present in the initial TGA scan, and hence the TGA procedure for determining carbon black shown in Figure 1 was further simplified to heating the elastomer at 100°C/minute to 600°C in nitrogen and holding isothermal for five minutes. The residue was a direct indication of the carbon black content. Table 1 shows the precision obtained.

The TGA evaluation in this case took about 10 minutes with cool down between runs of around 5 minutes. The productivity and simplicity of this approach can be further improved by the use of a TGA autosampler and autoanalysis software respectively. Figure 2 shows the tabular output available from the autoanalysis software.

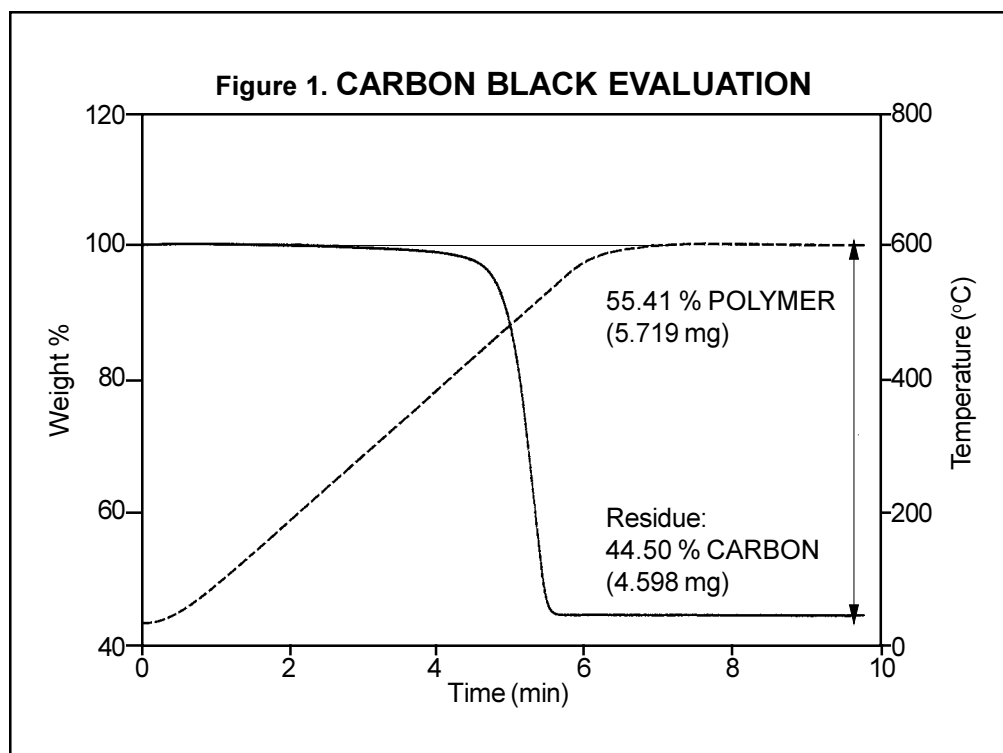


Table 1. TYPICAL ANALYSIS RESULTS

Sample No.	Polymer Content %	Carbon Black Content %
1	55.41	44.60
2	55.44	44.56
3	55.51	44.50
4	55.43	44.56
5	55.48	44.52
6	55.41	44.57
7	55.47	44.53
8	55.44	44.56
9	55.39	44.61
10	55.47	44.53
Average	55.45	44.55
Std. Deviation	0.037	0.035

Figure 2. TGA RESULTS PRINTOUT

TGA Autoanalysis

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File: C:JT992,111

Run Date:

Program: TGA-Auto V1.0D

Run Number: 807

Autoanalyzed with: C:AUTO.CMD

TA Instruments 2000 Thermal Analysis -- TGA 1000 xC

Sample: MB1890-A-0 1

Size: 10.3110 mg

Calibration Constant: 1.0000

Operator: MIKE

Method: 100/MIN TO 600xC ISO 5MN

Comment: 100 xC/MIN to 600xC, NITROGEN ATMOSPHERE

Transition	Start min	Onset min	Midpoint min	End min	Stop min	Weight mg	%
POLYMER	0.00				9.65	5.713	55.41
CARBON			Residue at		9.65	4.598	44.60

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