Crosslinked Polyethylene

SpeedExtractor E-916:
Determination of the crosslinked polymer content in Polyethylene samples using Pressurized Solvent Extraction (PSE)

A quick and easy method for the determination of the crosslinked polymer content in Polyethylene (PE) samples is introduced.

1. Introduction

During the production of polyethylene (PE) the determination of the crosslinked polymer content is part of the quality control process. The PE will be extracted by Pressurized Solvent Extraction (PSE) using the SpeedExtractor E-916, and the crosslinked content will be determined gravimetrically.

To determine the crosslinked content the residual sample will be used, not the extract. The non crosslinked content in the polymer will be separated from the sample using extraction.

2. Experimental

Equipment: SpeedExtractor E-916

Samples: 3 Polyethylene samples; crosslinked content: 98%; 96 % and 70 %.

Determination: For the extraction glass fiber extraction thimbles will be used. Prior to extraction a blank value for the thimbles has to be determined.

Weigh 200 mg sample into a dried glass fiber extraction thimble and cover it with glass wool. Load the sample into an extraction cell and start the extraction using the SpeedExtractor E-916 (Table 1).

Table 1: Method for extraction with the SpeedExtractor E-916

Parameter	Value
Temperature	160 °C
Pressure	100 bar
Solvent	Xylene 100 %
Cell	40 mL
Collection bottle	240 mL
Number of cycles	3
Heat-up Hold Discharge	1 min 10 min 2 min
Flush with solvent	1 min
Flush with gas	2 min
Extraction time	1 h

After the extraction is complete the extraction thimble is removed from the extraction cell using tweezers and the glass wool is replaced. The extraction thimble with the residual sample is dried in a vacuum drying oven for 2 h at 50 °C and 25 mbar and cooled down to ambient temperature in a desiccator. The weight of the residue is determined.

3. Results

The blank value of the extraction thimbles was determined to be 0.0069 g (rsd: 3.0 %). This value was used for the calculations of the crosslinked contents.

Table 2: Results for sample 1; expected value: 98 %

Sample 1	m_{sample} [g]	$m_{thimble,\ before}$ [g]	$m_{thimble,\ after}$ [g]	%Residue
1	0.2550	2.1389	2.3855	99.4
2	0.2067	2.0868	2.2855	99.5
3	0.2299	2.1523	2.3727	98.9
4	0.2585	2.2816	2.5302	98.8
mean				**99.1**
rsd				**0.34**

Table 3: Results for sample 2; expected value: 96 %

Sample 2	m_{sample} [g]	$m_{thimble,\ before}$ [g]	$m_{thimble,\ after}$ [g]	%Residue
1	0.2486	2.2382	2.4714	96.6
2	0.2484	2.0811	2.3149	96.9
3	0.2470	0.1593	2.3920	97.0
4	0.2666	2.0398	2.2908	96.7
mean				**96.8**
rsd				**0.19**

Table 4: Results for sample 3; expected value: 70 %

Sample 3	m_{sample} [g]	$m_{thimble,\ before}$ [g]	$m_{thimble,\ after}$ [g]	%Residue
1	0.3036	2.2481	2.4620	72.7
2	0.2521	2.1248	2.3014	72.8
3	0.2282	1.9996	2.1588	72.8
4	0.2192	2.0737	2.2263	72.8
mean				**72.8**
rsd				**0.04**

4. Conclusion

The crosslinked polymer content of PE samples can be determined using a PSE method on the SpeedExtractor E-916. The results obtained are comparable to the expected values.

5. References

Operation Manual for the SpeedExtractor E-916

For more detailed information and safety considerations please refer to the Application Note no. 158/2014.