

Fat in Polymer

SpeedExtractor E-916, Multivapor™ P-6:
Determination of the fat content in Polymer samples using the SpeedExtractor E-916



1. Introduction

A quick and easy method for the determination of the fat content in polymer samples is introduced. The samples are extracted simultaneously under high pressure and elevated temperature with the SpeedExtractor E-916 using a pressurized solvent extraction (PSE) method. After parallel extraction the solvent is evaporated to dryness using the 6 position Multivapor™ P-6 evaporator. The fat content is determined gravimetrically.

2. Equipment

- SpeedExtractor E-916
- Multivapor™ P-6 with vacuum pump V-700 and vacuum controller V-855
- Analytical balance (accuracy +/- 0.1 mg)
- Drying oven

3. Chemicals and Materials

Chemicals:

- Isopropanol ACS, Fluka (59300)
- Acetone ENVISOLV for analysis of dioxin, furan and PCB, Fluka (31062)

Materials:

- Cellulose extraction thimbles, BUCHI (11055334)

Samples:

2 Polymer samples (similar to natural rubber); expected fat content: 27 %

4. Procedure

The determination of the fat content includes the following steps:

- Sample preparation
- Extraction with the SpeedExtractor E-916
- Evaporation of the solvent with the Multivapor™ P-6

4.1. Sample preparation

1. Cut the samples into small pieces (approx. 1 cm) with a knife
2. Weigh 2 g samples into the extraction thimbles

4.2. Extraction with SpeedExtractor E-916

3. Choose the extraction method on the SpeedExtractor E-916 (Table 1)
4. Preheat the SpeedExtractor to the temperature in the method without the extraction cells present. Ensure the heating block is in the middle position and the protecting shield is closed
5. Dry 240 mL collection bottles for 30 min at 102 °C in a drying oven then allow the collection bottles to cool down to ambient temperature until a constant weight is obtained.
6. Prepare the 40 mL extraction cells: place a glass fiber filter (11055932) and metal frit in the bottom of the extraction cell and close with a plug (053209)
7. Load the extraction thimble into the extraction cell
8. Close the top of the extraction cell with a filter paper (049572) using a plunger (053037)
9. Insert 240 mL collection bottles into the collection unit
10. When the temperature is reached, insert the extraction cells using a gripper (053030) and start the extraction

Table 1: Extraction method for the SpeedExtractor E-916

Parameter	Value
Temperature	120 °C
Pressure	100 bar
Solvent	Acetone 50%, Isopropanol 50%
Extraction cell	40 mL
Collection bottle	240 mL
Number of cycles	6
Heat up	1 min
Hold	10 min
Discharge	5 min
Vial change	After cycle 4
Flush with solvent	2 min
Flush with gas	10 min
Extraction time	2 h 20 min

4.3. Evaporation of the solvent using the Multivapor™ P-6

- Evaporate the solvent to dryness using the parallel evaporator Multivapor™ P-6 (Table 2).

Table 2: Parameters for the Multivapor™ P-6

Parameter	
Bath temperature	60 °C
Rotation	7
Max. cooling temperature	20 °C
Step 1 : Acetone	Vacuum : 556 mbar for 10 min
Step 2 : Isopropanol	Vacuum : 136 mbar for 10 min

- Dry 240 mL collection bottles for 30 min at 102 °C in a drying oven and allow the collection bottles to cool down to ambient temperature until a constant weight is obtained.

5. Calculation

The fat content of the sample is calculated in percent (1).

$$\% \text{ Fat} = \frac{(m_{\text{Total}} - m_{\text{Collection bottle}})}{m_{\text{Sample}}} \cdot 100\% \quad (1)$$

- % Fat : fat content of the sample in percent
 m_{Total} : weight of the collection bottle + weight of the extract [g]
 $m_{\text{Collection bottle}}$: weight of the collection bottle [g]
 m_{Sample} : weight of the sample [g]

6. Result

Table 3: Results for sample 1; expected fat content: 27 %

	m_{Sample} [g]	$m_{\text{Collection bottle}}$ [g]	m_{Total} [g]	% Fat
Cycles 1-4	2.1481	148.5874	149.1014	23.93
Cycles 5+6	2.1481	148.2664	148.3064	1.86
Sum of all cycles				25.79

Table 4: Results for sample 2; expected fat content: 27 %

	m _{Sample} [g]	m _{Collection bottle} [g]	m _{Total} [g]	% Fat
Cycles 1-4	2.1781	147.8850	148.3826	22.85
Cycles 5+6	2.1781	150.4935	150.5517	2.67
Sum of all cycles				25.52

7. Remarks

7.1. Sample preparation

Usually the samples for pressurized solvent extraction (PSE) are mixed with a dispersing agent to the dry sample and to increase the sample surface. This sample was not homogenized, just cut into small pieces. Experiments have shown that mixing with a dispersing agent negatively affects this application. To receive sufficient extraction efficiency the sample should also be rolled.

7.2. Temperature and extraction thimbles

The properties of the polymer samples depend on the temperature used. Therefore prior to extraction the temperature stability of the samples was tested. The samples were placed in a drying oven for 30 min at 120 °C. The samples became sticky, but not molten. The use of extraction thimbles is recommended to avoid the sample sticking to the extraction cell.

7.3. Solvent

The extraction was carried out with 100 % acetone, however the efficiency was much lower when compared to an extraction using 50 % acetone and 50 % isopropanol.

7.4. Number of cycles and hold time

The determination of the fat content has shown that the extraction was not efficient using 4 extraction cycles. Two additional cycles were necessary to reach a complete extraction.

8. References

Operation Manual for the SpeedExtractor E-916
Operation Manual for the Multivapor™ P-6
Operation Manual for the Vacuum pump V-700 and Vacuum controller V-855