



# Application Note

No. 232/2016

## Solvent Extractables from Rubber

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SpeedExtractor E-916, Multivapor P-6:

Determination of Solvent Extractables from Rubber using the SpeedExtractor E-916



## 1 Introduction

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Extractables are chemical compounds that migrate from rubber or plastic material under forced conditions (high temperature, solvents). Extractables from rubber samples are determined following the norm ISO 1407. This norm requires a Soxhlet extraction with an extraction time of 16 h with at least 5 cycles per hour.

This Application Notes presents an effective method for the determination of extractables from rubber samples reducing the extraction time using pressurized solvent extraction (PSE). The PSE method is carried out on the SpeedExtractor E-916 under elevated temperature and pressure. In comparison to the norm ISO 1407 the extraction time can be significantly reduced.

## 2 Equipment

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- SpeedExtractor E-916
- Multivapor™ P-6
- Analytical balance (accuracy +/- 0.1 mg)
- Drying oven

## 3 Chemicals and Materials

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Chemicals and consumables:

- Acetone, puriss. p. a. Fluka (00570)
- Extraction paper thimbles, for 40 mL cells, BUCHI (11055334)

For safe handling please pay attention to all corresponding MSDS.

Samples:

- Rubber samples;
- Sample A: expected value: 18 - 22 %
- Sample B: expected value: 19 - 24 %

The samples were already homogenized; so no further homogenization was necessary.

## 4 Procedure

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The determination of extractables from rubber samples includes the following steps:

- Sample preparation
- Extraction of the samples
- Evaporation of the solvent
- Weighing of the extracts
- Calculation of extractable content



#### 4.1 Preparation of the cells

1. Weigh in approx. 0.5 g of sample into a paper thimble, that fits the 40 mL extraction cells (the samples were not mixed with a dispersing agent)
2. Place the thimble into a SpeedExtractor extraction cell

#### 4.2 Extraction of the samples with the SpeedExtractor E-916

3. Extract the samples using the method given in Table 1.

Table 1: Extraction parameters for SpeedExtractor E-916

Parameter	Value
Temperature	100° C
Pressure	100 bar
Solvent	Acetone 100%
Cells	40 mL
Vials	240 mL
Cycles	3
Heat-up	1 min
Hold	10 min
Discharge	3 min
Flush with solvent	2 min
Flush with gas	5 min

#### 4.3 Evaporation of the solvent with the Multivapor™ P-6

4. Evaporation the solvent of the extracts using the Multivapor™ P-6 with the parameters shown in Table 2.

Table 2: Parameters for solvent evaporation

Parameter	Value
Bath temperature	45 °C
Rotation	7
Pressure	500 mbar

After the evaporation of the solvent with the Multivapor™ P-6 the collection bottles contained the extract.

5. Dry the collection bottles in a drying oven to constant weight at 102 °C
6. Let them cool down to ambient temperature for at least 1 h in a desiccator (The cooling time of the collection bottles should be the same before and after the extraction).
7. Record the exact weight

#### 4.3 Calculation

The results were calculated as percentage of extract.

$$\% \text{ Extract} = \frac{(m_{\text{total}} - m_{\text{collection bottle}})}{m_{\text{sample}}} \bullet 100\% \quad (1)$$

%Extract: : percentage of extract in the sample  
 $m_{\text{total}}$  : collection bottle + extract [g]  
 $m_{\text{collection bottle}}$  : collection bottle [g]  
 $m_{\text{sample}}$  : sample weight [g]



## 5 Results

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The amount of extractable contents in sample A and sample B are presented in Tables 3-4. The results are within the expected range.

Table 3: Result for sample A (expected extract 18-22 %)

Sample A	m <sub>sample</sub>	m <sub>collection bottle</sub>	m <sub>total</sub>	% Extract
P1	0.5333	152.7283	152.8365	20.29
P2	0.6547	149.0583	149.1951	20.90
P3	0.5134	153.2947	153.4007	21.87
<b>Average [%]</b>	-	-	-	<b>20.61</b>
<b>sd</b>	-	-	-	<b>0.30</b>
<b>rsd [%]</b>	-	-	-	<b>1.48</b>

Table 4: Result of for sample B (expected extract: 19-24%)

Sample B	m <sub>sample</sub>	m <sub>collection bottle</sub>	m <sub>total</sub>	% Extract
P4	0.5859	146.4764	146.5865	18.79
P5	0.6023	149.2818	149.3959	18.94
P6	0.6598	148.4589	148.5828	18.78
<b>Average [%]</b>	-	-	-	<b>18.84</b>
<b>sd</b>	-	-	-	<b>0.09</b>
<b>rsd [%]</b>	-	-	-	<b>0.49</b>

## 6 Remarks

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It is possible that polymer samples extracted using a pressurized solvent extraction get sticky or even molten at elevated temperatures and cause blockages in the system. In order to avoid this, the samples are placed into paper thimbles. Therefore it is necessary to carry out the extraction with 40 mL extraction cells.

Using only 0.5 g of sample, the paper thimbles are almost empty. The samples in this note did not become sticky, nor were they molten. In this case, the paper thimbles are not required.

## 7 Conclusion

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The norm ISO 1407 requires a Soxhlet extraction time of 12-16 h with at least 5 cycles per hour.

Rubber samples can be extracted with the SpeedExtractor E-916. With the SpeedExtractor E-916 the time to results is significantly reduced (1 h) when compared to Soxhlet extraction used in the norm ISO 1407 (12-16 h).

## 8 References

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[1] ISO norm 1407: Rubber - Determination of solvent extract