



# Application Note

## No. 766/2021

### Determination of acetone-soluble matter in pulp

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UniversalExtractor E-800

Extraction acetone-soluble matter in pulp using the UniversalExtractor E-800 according to ISO 14453:2014



## 1. Introduction

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A simple and reliable procedure for the determination of the acetone-soluble matter in pulp is introduced.

The pulp and paper industry, with an approximate production volume of 416 million metric tons in 2019 [1], is one of the largest industries worldwide. Pulp is the major raw materials to produce paper products and consists of lignocellulosic or man-made fibrous material from wood, fiber crops, waste paper, or rags [2,3]. The extractives, low or medium molecular weight components outside the cell wall, influence the physical properties of the material and are soluble in specific solvents such as acetone [4]. The acetone-soluble matter includes fatty acids, resin, fatty alcohols, sterols, di- and triglycerides, steryl esters and waxes [5], and is one of the specifications for the pulp quality.

In the presented application, the sample is extracted with the UniversalExtractor E-800 using the Soxhlet method. The total acetone-soluble matter content is determined gravimetrically. The procedure complies with the official method of ISO 14453:2014.

## 2. Equipment

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- UniversalExtractor E-800 with chamber heater
- Recirculating Chiller F-308
- Analytical balance (accuracy  $\pm 0.1$  mg)
- Drying oven / Vacuum drying oven
- IDEAL 2035 paper cutter

## 3. Chemicals, Reagents and Materials

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- Acetone, for analysis ACS, ISO, Reag. Ph Eur (Sigma-Aldrich, Order No. 1.00014.2511)
- Glass sample tube with frit, long (BÜCHI, Order No. 11067815)

For a safe handling please follow the guideline mentioned in the corresponding MSDS! Sample:

- Pulp sheet, expected content: 0.1%, specification: <0.2%

The sample was cut into 1 cm x 1 cm squares using an IDEAL 2035 paper cutter.

## 4. Procedure

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The determination of acetone-soluble matter content includes the following steps:

- Determination of the acetone-soluble residues
- Determination of the dry matter
- Calculation of the acetone-soluble matter

### 4.1 Acetone-soluble residues

#### Sample preparation

1. Weigh 15 g sample in the sample tube with frit, long.

#### Extraction with the UniversalExtractor E-800

2. Always use dry and clean beakers. Dry them for at least 30 min at 102 °C in a drying oven. Let them cool down to ambient temperature in a desiccator for 1 h. Record the exact weight prior to extraction.

3. Place the glass sample tube with frit containing the sample into the extraction chamber and adjust the level sensor to the sample's height
4. Fill the solvent into the beakers and place them on their corresponding heating plate.
5. Close the protection shield and lower the rack. Alternatively, fill the solvent through the condensers after lowering the rack. Activate the occupied positions and open the cooling water tap or switch on the connected chiller.
6. Start the Soxhlet extraction according to the parameters listed in Table 1.

Table 1: Parameters for the UniversalExtractor E-800

Parameter	Value
Extraction method	Soxhlet
Solvent	Acetone
Solvent volume	150 mL <sup>1</sup>
Extraction:	
Time	240 min
Heating level	11
Rinse:	
Time	5 min
Heating level	11
Dry:	
Step 1	Analyte protection <input checked="" type="checkbox"/> 0 min
Heating level	9
Step 2	Analyte protection <input type="checkbox"/> 2 min
Heating level	9
Step 3	Analyte protection <input type="checkbox"/> 3 min
Heating level	0



Figure 1-2: Sample tube filled with sample (left) and placed into the extraction chamber (right)

In 4 hours extraction time, 35 and 34 cycles were performed, respectively.

#### 4.2 Drying of the extract

Dry the beakers containing the extract in a drying oven at 105 °C until constant weight. Let the beakers cool down to ambient temperature for at least 1 h in a desiccator and record the weight.

Make sure that the cooling down time of the beakers in the desiccator is the same before and after extraction. Differences in beakers temperature falsify the results.



Figure 1-5: Pulp sample during extraction (left), after drying (middle) in E-800 and after drying in drying oven (right)

<sup>1</sup> It is necessary to use 150 mL of solvent due to the large volume but low density of these samples.

### 4.3 Dry matter content

#### Sample preparation

1. Always use dry and clean beakers. Alternatively, beaker glasses can be used. Dry them for at least 30 min at 102 °C in a drying oven. Let them cool down to ambient temperature in a desiccator for 1 h. Record the exact weight.
2. Weigh 20-25 g of sample into the beakers.

#### Drying

3. Dry the samples in a drying oven for 3 hours at 102 °C to constant weight.
4. Let the beakers cool down to ambient temperature for 1 h in a desiccator and record the weight. The cooling time of the beakers should be the same then before the extraction. The exact weight was recorded.

## 5. Calculation

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The acetone-soluble matter content of the samples was calculated in percent.

$$X = \frac{a-b}{c} * 100 \quad (1)$$

X: content acetone-soluble matter, %  
a: mass of the extraction residues [g]  
b: mass of the blank residue [g]  
c: mass of the oven-dry sample [g]

$$a = m_{total} - m_{beaker} \quad (2)$$

$m_{total}$ : weight beaker + extract [g]  
 $m_{beaker}$ : weight beaker [g]

$$b = m_{total,blank} - m_{beaker,blank} \quad (3)$$

$$c = m_{sample} * \left( 1 - \frac{m_{total,dm} - m_{beaker,dm}}{m_{sample,dm}} \right) \quad (4)$$

$m_{total,dm}$ : weight beaker + dried sample [g]  
 $m_{beaker,dm}$ : weight beaker [g]  
 $m_{sample,dm}$ : weight sample before drying [g]  
 $m_{sample}$ : weight of extracted sample [g]

## 6. Results

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The detailed results of the extractions are shown in Tables 2 and 3.

Table 2: Results for the acetone-soluble residues, determined in duplicate with UniversalExtractor E-800

Sample	m <sub>Sample</sub> [g]	m <sub>beaker</sub> [g]	m <sub>total</sub> [g]	Extract [%]	Mean value [%]	rsd [%]
Pulp	15.1412	111.2475	111.2667	0.11	<b>0.11</b>	2.00
	15.1597	111.3394	111.3591	0.11		
Blank	0	110.9104	110.9129			

Table 3: Results for the dry matter, determined in duplicate with UniversalExtractor E-800

Sample	m <sub>Sample, dm</sub> [g]	m <sub>beaker, dm</sub> [g]	m <sub>total, dm</sub> [g]	Dry matter [%]	Mean value [%]	rsd [%]
Pulp	23.9558	108.6367	130.7946	7.50	<b>7.46</b>	0.91
	22.9938	109.7938	131.084	7.41		
Blank	0	110.6326	110.6324			

Considering the dry matter contents, an acetone-soluble matter content of 0.12% was determined.

## 7. Conclusion

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The determination of acetone-soluble content in pulp samples by use of the UniversalExtractor E-800 provides reliable and reproducible results. The results correspond well with the expected values with low relative standard deviations (rsd).

## 8. References

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- [1] <https://pgpaper.com/wp-content/uploads/2018/07/Final-The-Global-Paper-Industry-Today-2018.pdf>, 27.08.2021.
- [2] <https://www.pulpandpaper-technology.com/articles/pulp-and-paper-manufacturing-process-in-the-paper-industry>, 27.08.2021.
- [3] Bajpai, P. Biermann's Handbook of Pulp and Paper: Raw Material and Pulp Making. Elsevier, 2018.
- [4] Colodette J. L., A comparison of methods for eucalypt wood removal extractives, 5th International Colloquium on Eucalyptus Pulp, 2011.
- [5] ISO 14453: Pulps - Determination of acetone-soluble matter, 2014.

Extraction Reports App  
Operation Manual of the UniversalExtractor E-800