
Oil Determination in Insulating Wool according to the Randall method

Reference: British Standard Specification **BS 2972:1989** Method of test for inorganic thermal insulating material

Tested with **VELP Scientifica SER 148/6 Solvent Extractor** (Code F30300242)



Introduction

Substantial quantities of heat energy are wasted daily in houses and buildings because of under insulated and uninsulated surfaces.

Mineral wool and fiber glass are currently used as thermal insulation materials for energy efficient buildings, thanks to their low thermal conductivity values. Thermal insulators help in reducing heat loss, achieving enormous cost savings, and enhancing environmental sustainability.

In the insulating material production process, oil is used for the dust binding and to improve the water repellent properties. Hence, the quality control of mineral wool production includes a check to its oil content.

Determination of Oil Content in Inorganic Thermal Insulating Material

Randall method is a modification of the standard Soxhlet extraction: in Randall, the test portion is in contact with pure hot solvent, ensuring a fast solubilization with a considerable reduction of the extraction time (approx. 90 minutes).

Three extraction phases are necessary with SER 148 (see the following picture):



First, the extraction is made by immersion of the sample in the boiling solvent. This step is followed by a rinsing phase with hot solvent. Then, all the solvent evaporates and it is recovered in a condenser. The calculation of the total fat content follows gravimetrically after drying the extract.

Sample

Mineral insulating wool Expected oil content: < 1%

Chemicals and Equipment Required

- Grinder
- Analytical balance
- Extraction thimbles (33x80 mm) (Code CM0111148)
- Glass extraction cups (Code A00000142)
- Viton seals (Code A00000061)
- Petroleum Ether 40 - 60 °C as solvent

Sample Preparation

The mineral insulating wool sample has been homogenized by grinding for some seconds up to powder consistence.

Extraction Thimbles Preparation

Weigh 8 g of sample with accuracy of 0.1 mg (M_{sample}), directly in each 6 cellulose thimbles, using the Thimble weighing cup (Code A00001146). Fix every thimble with one magnetic extractions thimbles holders (Code A00001142): it is necessary to transfer directly the sample to SER 148.

Glass Extraction Cups Preparation

Keep the empty glass extraction cups in an oven (105 °C for 1 hour).

Cool them in a desiccator until constant weight of the tare (M_{tare}).

Extraction Procedure with SER 148

Before starting the extraction. Select one of the programs (1-29) and set the following parameters:

- Temperature: 110 °C
- Immersion Time: 45 minutes
- Washing Time: 30 minutes
- Recovery Time 10 minutes

Introduce the glass cups containing the solvent (Petroleum Ether, 100 ml) in the extraction equipment. Close the extraction unit, and press START button to activate the cooling water flow and the heating. Immerse the thimbles into the boiling solvent by placing the slider into “*Immersion*” position. After 45 minutes press ENTER and extract the thimbles out of solvent, by placing the slider into “*Washing*”.

After 30 minutes of reflux washing press ENTER and close the knob, let the solvent evaporate and recover it by condensation.

- To reduce the recovery time, turn on the air pump pressing AIR.
- Prevent the complete solvent evaporation (few ml are required).

Dry the cups in an oven (1 hour at 105 °C); then let them cool to room temperature in a dessiccator and record the accurate weight (M_{tot}).

Typical Results on Mineral insulating wool

The results are gravimetrically determined, by using the formula:

$$\text{Fat \%} = (M_{tot} - M_{tare}) \times 100 / (M_{sample})$$

Where:

M_{sample} = sample weight (g)

M_{tare} = weight of the empty cup (g)

M_{tot} = weight of the cup containing the fat residues (g)

M_{tare}	M_{tot}	Oil (g)	M_{sample}	Oil %
127.2891	127.3020	0.0129	8.0310	0.16
127.8918	127.9044	0.0126	8.0302	0.16
126.5618	126.5756	0.0138	8.0254	0.17
128.8790	128.8920	0.0130	8.0063	0.16
127.1813	127.1946	0.0133	8.0352	0.17
127.9248	127.9384	0.0136	8.0276	0.17
			Average ± SD%	0.16 ± 0.01
			RSD% *	3.42

Expected oil content: < 1%

* RSD% = (Standard Deviation x 100) / Average

Conclusion

The obtained results are reliable and reproducible in accordance with the expected values, with a low standard deviation, that means high repeatability of the results, considering the low level of fat content.

SER 148 Solvent Extractor is ideal for the fat content determination in insulating materials.

Benefits of Randall method by using SER 148 are:

- up to 5 times faster than Soxhlet (hot solvent vs. cold solvent)
- low solvent consumption (high solvent recovery)
- limited cost per analysis
- full operator safety in compliance with IP55
- worldwide official method
- the use of a evaporator is not needed, because the solvent recovery is performed directly on SER148 unit.