

Oil Determination in Recycled PET according to the Randall method

Reference: Velp Internal Procedure

Tested with VELP Scientifica SER 158/6 Solvent AutoExtractor (Code S303A0380)



Introduction

Polyethylene terephthalate (PET) and recycled PET (rPET) materials are known for their durability and versatility and are accordingly analyzed for many reasons, for example to discover if the material has been previously recycled, and if so, how many times, to confirm its Molecular Weight, and to discover its purity, for example regarding presence of heavy metals.

Solvent extraction is one of the analytical methods commonly used to determine various compounds present on a known substrate matrix specifically; the present application has been studied to determine glue and organic substances in weight from rPET flakes.

Oil Determination in rPET

Hot solvent extraction process with SER 158 Series can be summed up in 5 steps, for a fully unattended operation:



During [IMMERSION] the sample is immersed in boiling solvent. Then the [REMOVING] step automatically lowers the level of the solvent to below the extraction thimble. During [WASHING] the condensed solvent flows over the sample and through the thimble to complete the extraction process. The fourth step involves solvent [RECOVERY]. Approximately 90% of the solvent used is collected in the internal recovery tank. The final step is the [COOLING] of the extraction cups containing the extracted matter. The cups are raised to prevent burning. The extraction cups containing the extract are placed in a drying oven, cooled in a desiccator and weighed for the extract percentage calculation.

Sample

PET Oil labeled value: 100-300 ppm

Chemicals and Equipment Required

- Analytical balance (0.1 mg sensibility)
- Extraction thimbles (40x80 mm) (Code A00000296)
- Glass extraction cups L Ø 65x120mm (Code A00000302)
- Viton seals
- Petroleum Ether 40 – 60 °C as solvent

Sample Preparation

About 150g of rPET flakes were sifted in order to remove all dust. The sieved sample is dried in an oven at 105°C for 12h. Fix the Extraction thimbles with the Extraction thimbles holders (Code A00000292). Then, put 20 g of sample (*Sample*) directly in the VELP extraction thimbles using the Thimble weighing cup (Code A00000310).

Position the extraction thimbles in the extraction cups.

Glass Extraction Cups Preparation

Position the empty extraction cups in a drying oven (105 °C) for 1 hour and cool them in a desiccator. Connect the optional VELP barcode scanner (Barcode scanner with USB socket code: A00000364 or Wireless barcode scanner Code: A00000365) to SER 158.

Select Analysis/Details, scan the extraction cup and weigh the tare (*Tare*).

The extraction cups containing the extraction thimble can now be placed on the ultra-fast heating plate of SER 158.

Extraction Procedure with SER 158

On the ControlPad select “Analysis”, and then Create a Custom Method including the following parameters:

- Immersion Time: 30 minutes
- Removing Time: 40 minutes
- Washing Time: 10 minutes
- Recovery Time 10 minutes
- Cooling Time: 4 minutes
- n-Hexane, 200 ml

Close the safety guard and add the solvent using the automatic solvent dispensing system SolventXpress™ to minimize exposure to the solvent ensuring operator safety.

Press START to begin the extraction process.

At the end of analysis position the extraction cups containing the extract in a drying oven (1 hour at 105 °C), cool them in a desiccator to room temperature.

In Results menu select the extraction cups batch ID analyzed, press calculate, scan the extraction cups with barcode reader and weigh (*Total*).

Results on rPET

Analysis results are calculated automatically and stored in the ControlPad when entering the weights into the software (manually or automatically through a balance). The extract percentage calculation is performed by using the following formulas:

$$\text{Extract (g)} = (\text{Total} - \text{Tare})$$

$$\text{Extract (\%)} = \text{Extract} \times 100 / (\text{Sample})$$

Where:

Sample = sample weight (g)

Tare = weight of the empty extraction cup (g)

Total = weight of the extraction cup + extract (g)

Sample (g)	Tare (g)	Total (g)	Extract (%)
20.4255	143.9636	143.9696	0.020%
20.5575	140.7315	140.7376	0.020%
20.0279	144.0568	144.0619	0.015%
20.5465	140.7318	140.7369	0.015%
20.5284	143.9632	143.9687	0.017%
20.2864	143.9634	143.9687	0.016%
		Average ± SD%	0.017 ± 0.002

Oil Labeled Value: 100-300 ppm (0.01-0.03%)

Conclusions

The results obtained are reliable and reproducible in accordance with the expected values, especially considering the low amount of Oil in this matrix. Therefore, SER 158 Solvent Extractor is ideal for the Oil Determination in Recycled PET.

Benefits of hot solvent extraction (Randall) by using SER 158 Automatic Solvent Extractor:

- up to 5 times faster than Soxhlet (hot solvent vs. cold solvent)
- low solvent consumption (high solvent recovery, approximately 90%) - limited cost per analysis
- no exposure to solvent
- worldwide official method
- full traceability with automatic result calculation and on-board archive
- Cloud connectivity to VELP Ermes ensuring software updates, real time monitoring and control, notifications and enhanced service support.