



Ash from Petroleum Products by Microwave

UOP Method 1016-17

Scope

This test method covers the determination of ash in the range 0.01 to about 50.0 mass-%, in petroleum products ranging from distillate and residual fuels to crude oils and waxes to pitches and asphalts using a microwave ashing oven.

References

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The sample is weighed into a crucible (ceramic, platinum, nickel, or quartz) and reduced to ash by heating in a microwave ashing oven at 775°C, cooled and weighed. Size and type of crucible is determined by the amount of sample to be weighed. The ramping stages of the heating profile are determined by the type of sample, with liquids requiring a longer ramp time than materials that are solid at room temperature.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Pyro XL microwave ashing system, Milestone, Cat. No. 47096, including analytical balance capable of direct communication with the Milestone controller and weighing up to 220g +/- 0.1mg

Crucible, quartz, 10ml, Milestone, Cat. No. 40224

Crucible, ceramic, 100ml, Milestone, Cat. No. CRU100

Crucible, ceramic, 50ml, Milestone, Cat. No. CRU50

Crucible, ceramic, 25ml, Milestone, Cat. No. CRU25

Procedure

The analyst is expected to be familiar with general laboratory practices, and the equipment being used. Dispose of used reagents, materials, and samples in an environmentally safe manner in accordance with local regulations.

Set Operating Parameters

Install the analyzer according to the instructions in the manufacturer's operating manual. The conditions listed in Table 1 and Table 2 apply specifically to the Milestone PyroXL microwave

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. SAFETY DATA SHEETS (SDS) OR EXPERIMENTAL SAFETY DATA SHEETS (ESDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

ashing system. If other instrumentation is used, the conditions may be different depending on the wattage, exhaust velocity, and chamber dimensions.

Table 1
PyroXL Settings for Samples Liquid at Room Temp

<u>Step</u>	<u>Watts</u>	<u>Time</u>	<u>Temp °C</u>
Ramp	1000	00:05:00	90
Hold	1000	00:30:00	90
Ramp	1200	01:00:00	775
Hold	1200	01:00:00	775
Exhaust	0	00:45:00	0

Table 2
PyroXL Settings for Samples Solid at Room Temp

<u>Step</u>	<u>Watts</u>	<u>Time</u>	<u>Temp °C</u>
Ramp	1000	00:05:00	90
Hold	1000	00:20:00	90
Ramp	1200	01:00:00	775
Hold	1200	02:00:00	775
Exhaust	0	00:45:00	0

Determine Sample and Crucible Size

Sample size and crucible size and type are determined based on the estimated concentration of ash. Liquid samples will require larger crucibles to allow larger sample weights. See Table 3.

Table 3
Determining Sample Weight and Crucible Size

<u>Sample Type</u>	<u>Estimated %Ash</u>	<u>Sample Weight</u>	<u>Crucible Size</u>
Liquid at room temp	0.01 to 5.00	10 to 20 g	50 to 100 ml
Liquid at room temp	>5.00	5 to 10 g	25 to 50 ml
Solid at room temp	0.01 to 5.00	5 to 10 g	25 ml
Solid at room temp	>5.00	1 to 5 g	10 to 25 ml

Sample Analysis

Analyze samples and quality control materials as follows:

1. Weigh clean, dry crucible and record weight to 0.0001 g.
2. Add appropriate amount of sample to the crucible and record weight; see Table 3.
 - Sample must be homogenous so stirring with a spatula may be required. Samples that are solid at room temperature or viscous may require heating in order to thoroughly mix, which can be done in an oven of appropriate temperature.
3. Place crucibles with samples into microwave ashing oven chamber, close oven door and start ashing program.
 - Program the microwave ashing oven according to manufacturer instructions for the sample type. Table 1 and Table 2 list parameters for the Milestone PyroXL microwave ashing system.
4. After program is complete carefully remove the crucibles and reweigh, recording the weight.
 - If crucibles are warm allow to cool to ambient temperature inside the ashing oven, or use appropriate heat resistant gloves or tongs and place crucibles in a desiccator. Reweigh after reaching ambient temperature.
5. Calculate ash concentration; see *Calculations*.
6. Clean crucibles and discard ash residue.

Calculations

Calculate the mass of the ash in the original sample as follows:

$$\text{Ash, mass \%} = \left(\frac{A - C}{S - C} \right) * 100$$

where:

A = weight of crucible and ash residue, g

C = weight of crucible, g

S = weight of crucible and sample, g

For values from 0.01 mass-% to 10.0 mass-%, report to two decimal places. For values 10.0 mass-% and higher, report to one decimal place.

Precision

Precision statements were determined using UOP Method 999, "Precision Statements in UOP Methods," from precision data obtained using an autosampler.

Repeatability and Site Precision

A nested design was carried out for determining ash on three different samples by two analysts, with each analyst performing analyses on two separate days, performing two analyses each day for a total of 24 analyses. Using a stepwise analysis of variance procedure, the within-day estimated standard deviations (esd) were calculated at the concentration means listed in Table 2. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 2 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 2 with 95% confidence.

Table 4
Repeatability and Site Precision, mass-%

	<u>mean</u>	Repeatability		Site Precision	
		<u>Within Day esd</u>	<u>Allowable Difference</u>	<u>Within Lab esd</u>	<u>Allowable Difference</u>
Asphaltenes	41.13	0.395	1.29	0.433	1.41
Pitch	9.25	0.110	0.36	0.133	0.44
Residual Fuel Oil	1.20	0.082	0.27	0.097	0.32

The data in Table 4 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Time for Analysis

The elapsed time for this method is 4 to 5 hours. Labor time varies based on the need to heat and mix the samples.

Suggested Suppliers

Milestone Inc., 25 Controls Drive Shelton, CT 06484, USA, 1-866-995-5100,
www.milestonesci.com