

LOSS ON IGNITION OF CATALYST AT 900°C

UOP Method 275-98

SCOPE

This method is for determining the loss on ignition (LOI) of fresh and regenerated catalysts or catalyst supports of various commercial shapes and sizes, including fines, using an ignition temperature of 900°C. The method is quantitative down to 0.1 mass-%. The method is not water specific and other volatile components such as chlorine or fluorine are also included in the LOI. The LOI of spent catalyst and spent catalyst fines containing carbon or coke cannot be determined by this method. Mass gains are possible with catalysts that contain reduced metals.

OUTLINE OF METHOD

A representative sample is weighed into a crucible and heated at 900°C for six hours. After cooling, the sample is reweighed and the mass loss is calculated as mass-percent loss on ignition.

APPARATUS

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

Balance, readability 0.1-mg

Crucible, Inconel, low-form, 45-mL capacity, with cover, B-J Scientific Products, Cat. Nos. 30-45LF and 30-45C, respectively (optional)

Crucible, porcelain, low, wide-form, 30-mL capacity, with cover, Fisher Scientific, Cat. Nos. 07-955E and 07-970G, respectively

Desiccator, 250-mm ID, and porcelain plate, Fisher Scientific, Cat. Nos. 08-631B and 08-636F, respectively

Furnace, muffle, capable of operation at $900 \pm 15^\circ\text{C}$, Fisher Scientific, Cat. No. 10-549-166

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. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).
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Gloves, lint-free nylon, Fisher Scientific, Cat. No. 11-393-2B

Hot plate, variable temperature to 250°C, Fisher Scientific, Cat. No. 11-495-27

Thermometer, surface, with temperature probe, Omega Engineering, Cat. Nos. HH81 and 88108K, respectively

Tongs, crucible, 530-mm length, Fisher Scientific, Cat. No. 15-207

Tongs, crucible, 230-mm length, Fisher Scientific, Cat. No. 15-186

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified.

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

Desiccant, indicating Drierite, 8-mesh, Fisher Scientific, Cat. No. 07-578-3A

Grease, high-vacuum, Fisher Scientific, Cat. No. 14-635-5D

PROCEDURE

For general purposes, such as base metal determination, a single LOI determination is sufficient. For determination of noble metal on catalyst, duplicate LOIs are generally recommended. See the appropriate method for the catalyst being analyzed to determine the LOI requirements before proceeding.

In all cases, the LOI must be performed with the sample in the appropriate state. For Lot analysis, the sample used to obtain the LOI must be in the same state as the sample when shipped, whole pill (sphere), whole extrudate, or powder; not ground. For noble metal analysis, since the noble metals are determined on ground sample, the LOI must also be determined on ground sample. Check the appropriate method, standard operating procedure, work instruction or specification to avoid errors.

For analyses where the LOI is used to correct the analysis results to a dry basis, always weigh the sample for LOI at the same time the sample is weighed for the elemental analysis. Where duplicate LOIs are stipulated, report the average of the two LOI values. If the two values differ by more than 0.3 mass-% absolute, rerun both the LOI(s) and the associated elemental analysis, since the sample may have gained or lost moisture during the interim.

1. Obtain a representative portion of the catalyst or catalyst support to be analyzed.
 - Refer to UOP Method 918 for sampling procedures.
2. Ignite a porcelain crucible and cover by heating for 20 minutes in a muffle furnace at 900°C.
 - The crucible cover needs to be heated only once per day.
 - An Inconel crucible and cover is also satisfactory as an alternative.
 - The muffle furnace should be calibrated periodically.

3. Remove the crucible and cover from the furnace with tongs and place them in a desiccator containing a desiccant.
 - Discretion must be used regarding the number of crucibles placed in the desiccator at one time since an overheated desiccator may shatter.
 - When the top of the desiccant bed turns pink, stir to redistribute.
 - The desiccant should be replaced when it is one-third spent, as indicated by the color change.
4. Allow the crucible and cover to cool to ambient temperature and then weigh the crucible to the nearest 0.1 mg.
 - The crucible cover remains in the desiccator.
 - The cooling time will vary depending on the number of crucibles being cooled.
 - The crucible must be handled with small tongs or lint-free gloves– not with bare hands.
5. Weigh approximately 2 g of the sample to the nearest 0.1 mg into the ignited crucible immediately after completing Step 4.
 - Most catalysts are hygroscopic and should not be unnecessarily exposed to air.
6. Preheat the crucible containing the sample on a hot plate, having a surface temperature of 150 to 200°C, for approximately 20 minutes.
 - Fresh, whole-pill samples known to contain low volatiles do not require this preheating step.
 - A separate muffle furnace at a temperature of approximately 400°C may be substituted for the hot plate.
7. Transfer the crucible containing the sample to the 900°C muffle furnace with crucible tongs.
8. Allow the sample to remain in the furnace at the 900°C temperature for six hours.
 - When multiple crucibles are placed in the furnace, additional time may be required for the furnace to return to 900°C.
9. Remove the crucible from the furnace with crucible tongs, place it in the desiccator and cover it with a crucible cover.
10. Allow the crucible and cover to cool to ambient temperature.
 - The cooling time will vary depending on the number of crucibles being cooled.
11. Weigh the uncovered crucible and contents to the nearest 0.1 mg.
 - The crucible must be handled with small tongs or lint-free gloves– not with bare hands.

CALCULATIONS

Calculate the LOI at 900°C to two decimal places when the value is below 5 mass-%, or to one decimal place when value is above 5 mass-%, using the formula:

$$\text{LOI, mass - \%} = 100 \frac{A - C}{A - B}$$

where:

- A** = mass of ignited crucible and sample, before heat, g
B = mass of ignited crucible, g
C = mass of ignited crucible and sample, after heating, g
100 = factor to convert g/g to mass-%

REPORT

Report the LOI value at 900°C.

State the mass-% gain in cases where oxidation has occurred.

In the case of duplicates, report only the average of the two determinations.

PRECISION

The data in the Table were generated using UOP Method 888.

Repeatability

Based on two tests performed by each of two analysts on each of two different days (8 tests per lab) and using data collected from each of three laboratories, the within-laboratory estimated standard deviations (esd) were calculated at the LOI levels shown in the Table. Two tests performed in one laboratory by different analysts on different days should not differ by more than the allowable differences shown (95% probability).

Reproducibility

The between laboratory estimated standard deviations (esd) were calculated for the LOI levels shown in the Table. Two tests performed in different laboratories by different analysts, on different days, should not differ by more than the allowable differences shown (95% probability).

Table					
Fresh Catalyst	LOI Level, mass-%	Repeatability		Reproducibility	
		Within-Lab esd, mass-%	Allowable Difference, mass-%	Between-Lab esd, mass-%	Allowable Difference, mass-%
Whole-pill	1.63	0.068	0.20	0.129	0.51
Whole-pill	4.77	0.118	0.34	0.118	0.46
Ground	1.99	0.103	0.30	0.241	0.95
Whole-pill, bimetallic	1.62	0.077	0.23	0.108	0.42
Whole-pill, hygroscopic	3.95	0.079	0.23	0.099	0.39

TIME FOR ANALYSIS

The elapsed time for one test is 7.8 hours. The labor requirement is 0.5 hour.

REFERENCES

UOP Method 888, "Precision Statements in UOP Methods"

UOP Method 918, "Collection and Grinding of Adsorbents and Catalysts for Analysis"

SUGGESTED SUPPLIERS

B-J Scientific Products, Inc., 173 Queen Ave., SE, Albany, OR 97321 (503-976-8383)

Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219 (412-562-8300)

Omega Engineering, Inc., P.O. Box 4047, Stamford, CT 06907 (203-359-1660)