

LOSS ON IGNITION OF CATALYSTS AT 500 C

UOP Method 412-87

SCOPE

This method is for determining the loss on ignition of fresh or used catalysts at the specific temperature of 500 C.

OUTLINE OF METHOD

A representative, weighed sample is heated at 500 C to constant weight. The sample is reweighed and the loss in weight calculated as percent loss on ignition.

APPARATUS

Balance, readability 0.1-mg

Crucible, wide form, porcelain, Coors, Size 0, 17-mL capacity, Sargent-Welch Scientific, Cat. No. S-23665-F, or equivalent

Crucible cover, porcelain, Coors, 45-mm ID, Sargent-Welch Scientific, Cat. No. S-23720-G, or equivalent

Desiccator, Sargent-Welch Scientific, Cat. No. S-25005-E, or equivalent

Furnace, box and controller, electric, capable of maintaining 500 ± 10 C, Sargent-Welch Scientific, Cat. Nos. S-37005-BZ and S-37105, respectively, or equivalent

Hot plate, capable of maintaining 200 C, Sargent-Welch Scientific, Cat. No. S-41220-A, or equivalent

Oven, gravity convection, capable of maintaining 200 C, Sargent-Welch Scientific, Cat. No. S-64605-A, or equivalent

Thermometer, bimetallic, surface, Sargent-Welch Scientific, Cat. No. S-81442, or equivalent

Tongs, crucible, nickel-chromium, riveted, Sargent-Welch Scientific, Cat. No. S-82165, or equivalent

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).

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. References to water mean deionized or distilled water.

Calcium sulfate, anhydrous, indicating, Drierite, 8-mesh, Sargent-Welch Scientific, Cat. No. SC-11989, or equivalent

PROCEDURE

Preheat a crucible in a furnace at 500 ± 10 C to constant weight. Remove the crucible with crucible tongs and place it in a desiccator. Allow the crucible to cool and obtain a tare weight to the nearest 0.1 mg.

Loss on ignition should be run on either whole pill or ground catalyst depending on the type of sample required for other related analysis. Weigh a sample of approximately 2 g to the nearest 0.1 mg.

Preheat the crucible and contents by placing it on a hot plate for one hour at a temperature of 150 ± 50 C. This heating period removes volatile matter that could otherwise cause agitation and loss of catalyst at elevated temperatures.

Remove the crucible from the hot plate with crucible tongs and place it in the furnace at 500 ± 10 C. Meanwhile, place the crucible cover in a drying oven at 200 C.

After one hour, transfer the crucible to a desiccator and immediately place the preheated cover on the crucible. Allow the crucible to cool for approximately 15 minutes, then rapidly weigh the uncovered crucible and contents to the nearest 0.1 mg. Since prolonged weighing time can cause errors, it is necessary to weigh all samples quickly when doing multiple analyses.

Repeat the heating at 500 ± 10 C for successive 30-minute periods until the final weight remains constant. A one-hour ignition period is usually sufficient to effect constant weight.

CALCULATIONS

Calculate the loss on ignition from the formula:

$$\text{Loss on ignition at 500 C, mass-\%} = 100 \frac{W_i - W_f}{W_i}$$

where:

W_f = final sample weight after ignition at 500 C, g

W_i = initial sample weight taken for analysis, g

100 = factor to convert to percent

PRECAUTIONS

Hygroscopic samples should have as little exposure to the atmosphere as possible. Sample under a nitrogen blanket if absolutely necessary.

PRECISION

Based on 6 replicate determinations, the estimated standard deviation (esd) was calculated for loss on ignition at the levels shown in the table. Duplicate results by the same operator should not differ by more than the allowable difference shown (95% probability).

Level, mass-%	esd, mass-%	Allowable Difference, mass-%
1.1	0.05	0.1
2.5	0.06	0.2
6.1	0.15	0.4

TIME FOR ANALYSIS

The elapsed time for one analysis is 2.8 hours. The labor requirement is 0.4 hour.

SUGGESTED SUPPLIER

Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077