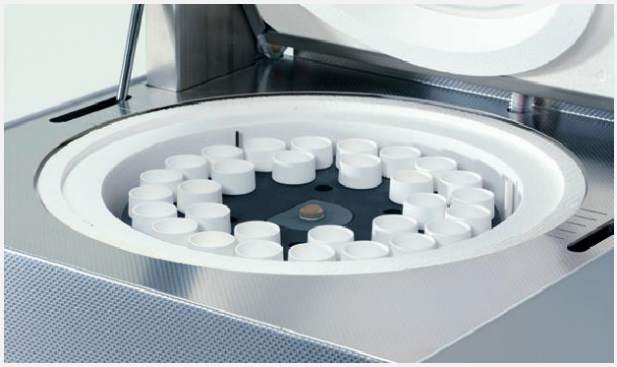
Thermogravimetry for the Determination of the Loss on Ignition on Petrochemical Catalysts

prepASH 340 Series

Burning | High Temperature | Improved Safety | Weighing Samples



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1. Abstract

The Loss on Ignition (LOI) is an important analytical parameter in the assay of petrochemical catalysts. The LOI of the "as is" sample has an impact on the net lot size. The LOI on the laboratory sample has an important influence on the final laboratory result, expressed on the calcined material.

This paper will show the excellent repeatability and precision of a Precisa prepASH129 automatic drying and ashing furnace for the determination of the LOI in petrochemical catalysts.

Homogeneity tests on original material show that this type of product is very homogeneous, so the total sample size for LOI on raw material can be reduced to quantities that can be handled in the prepASH129 furnace.

Repeatability tests on the laboratory samples show that the use of this furnace provides high quality results for less labor time.

The prepASH129 furnace is robust, easy to learn and provides a visual presentation of the process occurring in the sample, ensuring complete calcining.

Umicore will implement the use of this furnace for the "as is" sample and for the laboratory sample in the near future.

2. Introduction

2.1. LOI on a petrochemical catalyst

Catalysts are used to facilitate chemical reactions or to eliminate atmospheric emission of pollutants

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in the petrochemical industry. Many of these catalysts contain Pt and / or Pd on an alumina and silica carrier. Depending on the application, these catalysts can have a different form such as beads, pellets, small rods,.. Due to the processes involved, these materials are contaminated with volatile organics and moisture at the end of lifetime.

During the sampling process, the lot is weighed, and a representative sample is produced for the lot "as is", including the volatile organics and moisture. As grinding and milling of this final sample will have an important impact on the concentration of these organics and moisture, a loss on ignition is performed on an "as is" sample (LOI on "as is" sample). This sample should be drawn as soon as possible to the weighing of the lot. It is not used for precious metal assaying but allows calculating the metal content to a reproducible calcined lot size.

A part of the final sample is milled and ground to obtain a quality laboratory sample for metal assaying. During this process, the concentration of the volatile components of the sample will certainly change. This implies also that a new LOI on the laboratory sample has to be determined in order to be able to calculate the laboratory results to the same calcined state (LOI on the laboratory sample). As the lot size is recalculated to the calcined state, it is clear that the accuracy and precision of the LOI analysis on the "as is" sample is of utmost importance.

As the laboratory results are also recalculated to the calcined state, it is obvious that the LOI on the laboratory sample is of equal importance and is as important as the accuracy of the assay procedure applied to determine the Precious Metal content.

As both the calcined lot size and the laboratory result are used to calculate the total amount of metal present in the lot, it is essential that the calcined state of the "as is" sample and of the laboratory sample are identical. Therefore, is to be preferred that the same procedure is used.

2.2. The prepASH129 furnace

The PrepASH129 furnace, built by the Swiss company Precisa, is a thermogravimetric analysis instrument and combines a high temperature furnace, capable of heating to 1000°C, with a built-in balance system.

Fig.1 Front and inside view of the prepASH129 furnace

The balance is installed below the furnace and the weight is transferred from the hot furnace to the outside by a ceramic rod. The main applications for the furnace today are moisture and ash value determinations in coal and food industry.

In one working cycle, up to 29 samples and a reference crucible are ashed or calcined fully automatically. Depending on the density of the material, up to approximately 50g can be added to each crucible. These 25ml crucibles are made of Cordierite (a Magnesium Aluminium Silicate) and have a specific shape to fit into the carrousel. Lowering the central axis of the furnace positions one crucible on top of the lift, which is in contact with the balance. Sequentially the crucibles are weighed throughout the complete program.

Weight changes as a function of temperature is monitored and stored. Heating can be programmed in several steps with different ramp settings to increase the temperature as required. Up to 3 flags can be set to store the weight loss.

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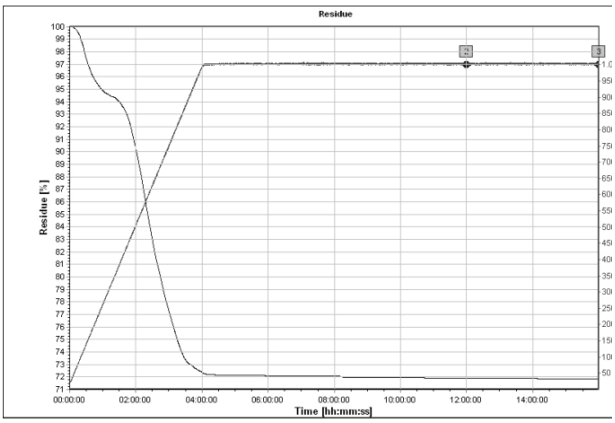

Fig. 2 Residual weight and temperature (°C) versus time (Hrs) for one sample in the prepASH129 furnace

These results can be exported to an external computer or LIMS system. A reference crucible corrects for buoyancy effects caused by the heat inside the furnace. The furnace can be flushed during heating with dry air, nitrogen, or oxygen. For our application, we flush with 3 liter/min of dry air. The instrument is equipped with a touch screen with VGA resolution. Weight loss versus temperature is shown during the process. The complete TGA curves can be transferred to a computer for storage and evaluation. Opening and closing of the furnace, changing samples, programming the method is done by the instructions shown on the touch screen.

3. Loss on Ignition on the "as is" sample

The Umicore LOI sample of 1.5 Kg is blended for 10 minutes with a Turbula mixer and is divided in 16 equal portions of about 90 gram by a rotary splitter. These sample bags are immediately sealed to avoid changes in the amount of volatile components during storage of the spare LOI samples.

Six portions are taken for the LOI determination (§3.1.2.).

Precision of the LOI has to be very good as imprecision induces uncertainty on the calcined lot size.

The calcined state of the "as is" material has to be the same as the calcined state of the laboratory sample. If not, the calculation of the metal content of the lot is inaccurate.

The LOI is determined by heating the samples to a preset temperature and time. At Umicore we generally apply 1000°C. The time the sample remains at the final temperature must be sufficient to remove all volatile temperature must be sufficient to remove all volatile components. If this time is too short and a constant weight is not achieved, factors including the packing of the sample (thin versus thick layer) and the physical aspect (powder, beads, rods,..) can influence the LOI. At Umicore, the sample is kept at 1000°C for 12 hrs.

There is a risk for unwanted loss of material by putting the samples directly in the calcination furnace at the final temperature. This may lead to a vigorous decomposition of the volatile components. Therefore, we put the "as is" sample for 3 hrs at 200°C and then raise the temperature with a ramp of 300°C/hr from 200°C to 1000°C (approximately 3 hrs).

Accuracy of the LOI method is very important. One must avoid systematic errors, e.g. by moisture uptake during cooling after the calcining step.

3.1. Actual Umicore procedure

3.1.1. Equipment

- Programmable muffle furnace (temperature calibrated), capable of maintaining a temperature of 1000°C
- Analytical balances
- Ceramic dishes, diameter 130 mm
- Stainless steel trays, 410 mm x 300 mm
- A cabinet with permanent nitrogen flush. The cabinet used at Umicore consists of small individual storage boxes (2 trays per box), so the individual doors can be closed immediately after the calcined sample is

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introduced. In this way we can protect each sample from ambient air while loading the rest of the samples into the cabinet. ·

- Tongs, spatula, brushes...

3.1.2. Procedure

- Take 6 sample bags from the "as is" sample.
- Tare 6 ceramic dishes and register the weight.
- Transfer the content of one sample bag (approx. 90 g) to one dish and weigh immediately to 10mg precision. (initial weight m1)
- Place the ceramic dish in the muffle furnace at 200°C.
- Take the ceramic dishes out of the hot furnace, place each one in a stainless-steel tray and put this dish + tray in a cabinet with continuous dry nitrogen purge to prevent moisture uptake during the cooling of the sample. This cooling takes approx. 30 minutes.
- Remove the ceramic dishes from the cabinet and weigh immediately. (final weight m2)
- Calculate the LOI according to the formula

$$LOI\ (\%) = (m1 - m2)*100 / m1$$

3.2. Proposed procedure with the Precisa PrepASH129 furnace

The basic principle, drying to constant weight at 1000°C, is maintained with the PrepASH129 furnace. Due to the continuous weighing of the sample, it is possible to monitor the weight loss throughout the whole process.

Because of the limited capacity of each individual crucible in the PrepASH129 system, the content of one sample bag per lot will be divided over different individual crucibles.

In §3.3.3 we will show that analyzing only one bag provides sufficient information and accuracy compared to the present method on 6 bags.

3.2.1. Equipment

- A furnace with incorporated weighing system,
- e.g. Precisa PrepASH129 or equivalent, capable of heating and weighing up to 10g samples at temperatures up to 1000°C.
- Analytical balance, incorporated in the furnace system.
- Crucibles specific for the furnace (shape and size) made of C410 Cordierite.
- Cotton gloves, tongs, spatula, brushes...

The PrepASH129 furnace program in our laboratory:

- Heat from room temperature to 1000°C over a period of 4 hrs.
- Monitor weight loss on continuous base. The exact weight loss is registered after 8 hrs and after 12 hrs at 1000°C

3.2.2. Procedure

- Put the Cordierite crucibles on a quartz dish and ignite at 1000°C. This can also be done in the PrepASH129, but this reduces the availability of the furnace for LOI determinations.
- Remove the crucibles from the furnace and put these in a desiccator. Let cool to room temperature.
- Place the crucibles in the PrepASH129 and auto-zero the internal balance (push-button instruction).
- Open a sealed sample bag containing the "as is" sample.
- Take the crucibles one by one out of the carrousel and fill with the catalyst material to approximately 4/5th of the volume.
- Place the crucible immediately back to the same position in the carrousel. Weighing is done automatically. Enter the sample identity.
- Fill the next crucible from the same sample bag until the complete content is transferred to the crucibles. Depending on the density and packing of the "as is" sample, this requires 6 to 12 crucibles.
- Put $CaCO_3$ or $CaC_2O_4.H_2O$ into the last crucible (position 29). The thermal decomposition to CaO will be used as QC of

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the process.

- Start the program of the PrepASH129 furnace.
- Normally, after 12 hrs constant weight is achieved. Most samples are already completely calcined after 8 hrs. This can be verified after the process by comparing the LOI after 8 hrs and after 12 hrs at 1000°C. These results should be within 0.2% of the final LOI after 12 hrs. If not, the thermogram of the decomposition process has to be checked to assure that all organics are burned off after 12 hrs.
- The LOI for the "as is" sample is the average value of the individual LOI results determined over the different samples per bag.

3.3. Results

3.3.1. Quality control

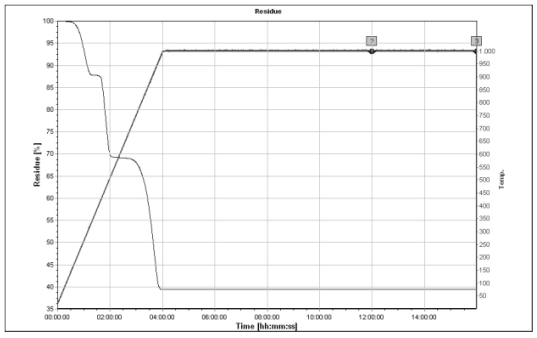

Fig. 3 The thermogram recorded for Calcium oxalate monohydrate with the prepASH129

Accuracy of the weighing process:

Thermogravimetry is a technique in which the mass of a substance and / or its reaction product is measured as a function of temperature whilst the substance is subject

Calcium oxalate monohydrate decomposes in different steps to calcium oxide.

$CaC_2O_4.H_2O$ -> CaC_2O_4 + $H_2O\uparrow$ - 12.3% referred to $CaC_2O_4.H_2O$

CaC_2O_4 -> $CaCO_3$ + $CO\uparrow$ - 31.5% referred to $CaC_2O_4.H_2O$

$CaCO_3$ -> CaO + $CO_2\uparrow$ - 61.6% referred to $CaC_2O_4.H_2O$

or

$CaCO_3$ -> CaO + $CO_2\uparrow$ - 44.0% referred to $CaCO_3$

The final decomposition starts at about 500°C with a maximum rate at about 900°C. Calcium oxalate monohydrate or calcium carbonate can therefore be used to monitor the process in the PrepASH129 furnace.Hereto we add e.g. anhydrous $CaCO_3$ to the last crucible and we transfer the results for the LOI in a SPC chart to monitor accuracy of the weighing process in the furnace.

Accuracy of the temperature:

- To monitor the temperature, the furnace is equipped with a thermocouple. The accuracy is checked with each annual service.
- On weekly basis we check the temperature by adding Ag grains (MP 962°C) and Au grains (MP 1064°C) to 2 different crucibles. At 1000°C, the Ag grains should melt together while the Au grains may not. After the test, the Ag and Au can easily be removed from the crucibles and can be recuperated. This is a rather basic approach but is sufficient to monitor important deviations of the temperature of the furnace.
- On daily basis the decomposition curves of $CaCO_3$ or $CaC_2O_4.H_2O$ give sufficient information to recognize important anomalies.

3.3.2. Comparison of the precision and accuracy

To show that the LOI sample is accurately distributed over the 16 sample bags with the rotary splitter, we have selected 5 Pt catalysts with different physical aspects. Lot 1 & 5: beads, lot 2: beads and fines, lot 2 & 3: extrudates. Results are shown in §3.3.2.1.

To compare accuracy and precision of the actual method and the method with the PrepASH129, a

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comparison is made over the same 5 Pt catalysts.

The results are given in §3.3.2.2.

3.3.2.1. Comparison between the individual sample bags.

"As is" samples are subdivided from the 1.5 Kg sample with a rotary splitter. One may expect that each sample bag is representative due to this procedure. To show this, we have analyzed 4 complete sample bags per individual lot, each bag was subdivided over several crucibles. The results are presented in table 1.

Lot 1	bag 1	bag2	bag 3	bag 4
crucible 1	6.65	6.30	5.64	5.69
crucible 2	6.33	5.89	6.98	6.40
crucible 3	6.30	6.46	6.65	6.62
crucible 4	6.41	6.28	6.62	6.60
crucible 5	6.26	6.48	6.90	6.37
crucible 6	6.27	6.73	6.30	6.48
crucible 7	6.51	6.60	6.16	6.91
crucible 8	6.54	6.68	6.28	6.31
crucible 9	5.76	6.35	6.43	5.22
average / bag	6.34	6.42	6.44	6.29
std dev / bag	0.25	0.26	0.41	0.52

Lot 2	bag 1	bag2	bag 3	bag 4
crucible 1	6.68	6.49	6.66	6.71
crucible 2	6.51	6.54	6.67	6.56
crucible 3	6.57	6.65	6.43	6.68
crucible 4	6.73	6.60	6.63	6.47
crucible 5	6.63	6.62	6.67	6.58
crucible 6	6.67	6.61	6.79	6.73
crucible 7	6.64	6.75	6.69	6.65
crucible 8	6.68	6.70	6.64	6.68
crucible 9	6.83	6.80	6.85	6.67
crucible 10	6.67	6.65	7.01	7.00
average / bag	6.66	6.64	6.70	6.67
std dev / bag	0.09	0.09	0.15	0.14

Lot 3	bag 1	bag2	bag 3	bag 4
crucible 1	2.60	2.63	2.55	2.61
crucible 2	2.57	2.60	2.55	2.62
crucible 3	2.60	2.61	2.57	2.68
crucible 4	2.60	2.59	2.55	2.69
crucible 5	2.61	2.62	2.55	2.65
crucible 6	2.56	2.61	2.55	2.65
crucible 7	2.60	2.63	2.55	2.65
crucible 8	2.63	2.63	2.56	2.66
average / bag	2.60	2.61	2.55	2.65
std dev / bag	0.02	0.02	0.01	0.02

Lot 4	bag 1	bag2	bag 3	bag 4
crucible 1	16.37	16.47	16.30	16.43
crucible 2	16.43	16.62	16.55	16.41
crucible 3	16.51	16.68	16.40	16.47
crucible 4	16.65	16.48	16.33	16.50
crucible 5	16.54	16.48	16.50	16.48
crucible 6	16.50	16.76	16.53	16.62
crucible 7	16.50	16.56	16.69	16.58
crucible 8	16.46	16.58	16.43	16.54
average / bag	16.50	16.58	16.47	16.51
std dev / bag	0.08	0.10	0.13	0.07

Lot 5	bag 1	bag2	bag 3	bag 4
crucible 1	8.97	9.05	9.24	8.95
crucible 2	9.20	8.89	8.92	9.22
crucible 3	8.99	9.03	8.70	9.32
crucible 4	9.03	9.02	8.77	9.14
crucible 5	8.92	8.98	8.92	9.02
crucible 6	9.02	9.06	8.86	8.91
crucible 7	9.08	9.13	8.77	9.01
crucible 8	9.11	9.25	8.97	9.30
crucible 9	8.99	9.08	8.78	8.93
crucible 10	8.74	8.96	9.16	9.10
average / bag	9.00	9.04	8.91	9.09
std dev / bag	0.12	0.10	0.18	0.15

Table 1. Individual results of the different crucibles per bag per sample with the prepASH129

We have performed a paired t-test between the sample bags per sample with the largest difference in mean value per lot. (table 2)

Lot ID	min LOI %	max LOI %	max-min LOI %	significant? paired t-test
lot 1	6.29	6.44	0.15	No
lot 2	6.64	6.70	0.06	No
lot 3	2.55	2.65	0.10	Yes
lot 4	16.47	16.58	0.11	No
lot 5	8.91	9.09	0.18	No

Table 2. Paired t-test between the bags with the largest differences per lot

We can conclude that the difference between the individual sample bags is statistically not significant, except for lot 3. In all cases, the difference is very small in absolute value.

Due to the individual results obtained for the different crucibles out of one bag, individual outliers can be recognized and if necessary, the LOI can be repeated on a new sealed bag the day after.

3.3.2.2. Comparison of the accuracy for the results obtained by the actual method and the PrepASH129 furnace.

To compare the results of the actual method and the method with the PrepASH129 furnace, we have compared the results per bag obtained by the 2 methods. These results are presented in table 3.

	lot 1		lot 2		lot 3		lot 4		lot 5	
	Actual	Prep129	Actual	Prep129	Actual	Prep129	Actual	Prep129	Actual	Prep129
	6.23	6.34	6.65	6.66	2.45	2.60	16.34	16.50	8.04	9.00
	6.09	6.42	6.61	6.64	2.49	2.61	16.27	16.58	8.18	9.04
	6.28	6.44	6.59	6.70	2.51	2.55	16.36	16.47	8.03	8.91
	6.15	6.29	6.59	6.67	2.58	2.65	16.31	16.51	8.18	9.09
	6.30		6.60		2.53		16.26		8.19	
	6.37		6.59		2.57		16.23		8.06	
	6.24	6.37	6.61	6.67	2.52	2.60	16.30	16.52	8.11	9.01
	0.10	0.07	0.02	0.02	0.05	0.04	0.05	0.05	0.08	0.08
Actual - Prepash129	-0.14		-0.06		-0.08		-0.22		-0.90	

Table 3. Comparison of the results obtained by the actual method and the prepASH129

According to the t-test, all these differences are significant. The PrepASH129 furnace gives systematically higher LOI compared to the actual method. Most of the differences are very small, but in some cases the difference cannot be neglected (lot 5). This can be explained by the fact that, despite all measures taken to avoid moisture uptake during cooling to room temperature, the sample does take up "moisture" during the actual method.

Verification by the PrepASH129 of the moisture on the calcined material of lot 5 (calcined by the actual method) confirms that the material has increased approximately 1% in weight during the cooling step.

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3.4. Comparison of labor time

The procedure with the PrepASH129 is less time consuming compared to the actual method. The difference is about 20 minutes per lot.

4. Loss on Ignition on the laboratory sample

To determine the precious metals content of the lot, an accurate assaying procedure is obviously very important. Umicore applies a fire assay lead collection procedure in combination with ICP.

For analyses where the LOI is used to calculate the results to a calcined state, it is essential that the samples for LOI and for fire assay be weighed at the same time. Hereto 2 bags with laboratory sample are opened and the content is equilibrated over 24h with the ambient air. Then, at the same time, sample is taken for fire assay lead collection and for the LOI determination. Finally, the fire assay results obtained are recalculated to calcined state with the LOI obtained on this lab sample.

The formula is

ppm (calcined material) = ppm (stabilized material) / (1 - LOI (%)) / 100)

In this formula, the result for the LOI plays an important role. The LOI result has to be in agreement with 3 criteria:

- Precision of the LOI has to be as good or better than the fire assay procedure to retain precision for the final result.
- Accuracy of the LOI method is very important. One must avoid systematic errors, e.g. by moisture uptake during cooling. Small errors on the LOI become more important as the absolute value of the LOI increases, because the LOI is in the denominator of the formula.
- The laboratory calcined state has to be the same as the calcined state of the "as is" sample, otherwise the calculation of the metal content of the lot is inaccurate. This implies that the use of the same equipment for determining the LOI on both the "as is" and the laboratory sample is to be preferred.

The LOI is determined by heating the samples to a preset temperature, the same temperature used for the "as is" sample. There is a risk for unwanted loss of material by putting the samples directly in the calcination furnace at the final temperature. We use a ramp setting of approximately 300°C/hr from room temperature to 1000°C.

Just as for the "as is" sample, the time the sample remains at the final temperature must be sufficient to remove all volatile components. The sample is kept at 1000°C for 12 hrs.

4.1. Actual Umicore procedure

The procedure applied at Umicore today is similar to the method described in ASTM procedure 954-3, developed by UOP (ref 1).

4.1.1. Equipment

- Programmable muffle furnace (temperature calibrated), capable of maintaining a temperature of 1000°C
- Analytical balances, vacuum pump, dessicator with safety shields
- Alundum crucibles, e.g. diameter 40mm , volume 30 ml
- Ceramic dishes, e.g. diameter 160 mm, height 60 mm
- Quartz dishes, e.g. diameter 100 mm; height 20 mm
- Weighing bottles with lid, e.g. diameter 50 mm
- Cotton gloves, tongs, spatula, brushes...

4.1.2. Procedure

Our laboratory receives 2 sample bags with prepared laboratory sample from the sampling department. All analyses below are performed on each sample bag.

Both samples are processed throughout our

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laboratory as 2 independent samples (different chemists, different furnaces, different ICP units and ICP standard sets). Only at the end results are combined. Herewith you find a condensed method description of the actual procedure. All handling of the crucibles are done with lint-free gloves. We do not use desiccant in the desiccator, as the catalyst may be more hygroscopic than the desiccant itself.

Therefore, we use vacuum to avoid moisture

uptake during cooling in the desiccator.

- Spread the sample in a ceramic dish and let it stabilize in ambient air for 24 Hrs (draft-free). Mix the sample regularly.
- Place the alundum crucibles on a quartz dish and ignite at 1000°C. Remove from the furnace and put the crucibles in a desiccator.
- Place the clean and dry weighing bottles with lid in a desiccator.
- Remove the empty alundum crucibles and the weighing bottles with lid from the desiccator and weight together (1 crucible + 1 weighing bottle with lid = 1 set) to 0.1 mg.
- Weigh approximately 4.0000g to the nearest 0.1mg into the alundum crucibles. Weigh at the same time for the fire assay procedure.
- Place the alundum crucibles in the furnace on a quartz dish and raise the temperature to 1000°C over 3 hrs. Leave the samples in the furnace for 12 hrs at 1000°C.
- Take the alundum crucibles from the hot furnace and place a.s.a.p. into the desiccator. Do not put too many samples in one desiccator as an overheated desiccator may shatter.
- Remove air from the desiccator and let the samples cool down.
- Let dry air flow into the desiccator.
- Open the desiccator and place the alundum crucibles immediately into the corresponding weighing bottles. Close the lid.
- Weigh the crucible + corresponding weighing bottle with lid to 0.1 mg.
- Calculate the LOI:

$$LOI\ (\%) = (m2-m3) / (m2-m1) * 100\ \%$$

m1 = weight of the empty crucible + weighing bottle & lid

m2 = weight of the empty crucible + weighing bottle & lid + sample before calcination

m3 = weight of the empty crucible + weighing bottle & lid + sample after calcination

4.2. Procedure with the Precisa PrepASH129 furnace

The basic principle, drying to constant weight at 1000°C

4.2.1. Equipment

- The same instrument is used as for the "as is" sample.

4.2.2. Procedure

We will continue working on 2 sample bags as this is part of our standard laboratory quality procedure. The procedure is similar to the procedure for the "as is" sample. Differences are listed below.

- Spread the sample in a ceramic dish and let it stabilize in ambient air for 24Hrs (draft-free). Mix the sample regularly.
- Fill with the stabilized catalyst material to approximately half of the volume of the Cordierite crucible. This corresponds with 4g to 8g, depending on the density of the material.
- Place the crucible immediately back to the same position in the carrousel, Weighing is done automatically. Enter the sample identity.
- Proceed until all samples are introduced into the furnace.
- Put $CaCO_3$ or $CaC_2O_4.H_2O$ into the last crucible (position 29). The thermal

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decomposition to CaO will be used as QC of the process.

- The LOI is transferred to the LIMS system of the laboratory.

4.3. Results

4.3.1. Quality control

Accuracy of the weighing process and the temperature is evaluated in the same way as for the "as is" sample (§3.2.2)

4.3.2. Precision of the PrepASH129 on a laboratory sample

To determine the precision of the LOI by the PrepASH129, we have calcined the laboratory sample of the former 5 lots after stabilizing in ambient air. Remember that this LOI result may not be compared with the LOI of the "as is" sample, as it underwent several steps in the sampling department and was stabilized for 24 hrs in the laboratory prior to the LOI analysis. All 4 results were obtained in one furnace run.

	lot 1	lot 2	lot 3	lot 4	lot 5
crucible 1	10.35	20.05	9.45	17.84	9.52
crucible 2	10.37	20.09	9.44	17.83	9.51
crucible 3	10.37	20.08	9.45	17.83	9.56
crucible 4	10.41	20.08	9.45	17.82	9.52
average	10.38	20.08	9.45	17.83	9.53
std dev	0.02	0.02	0.00	0.01	0.02

Table 3 LOI on the laboratory sample with the prepASH129

These results show that the precision of the LOI, determined with the PrepASH129 is excellent.

4.3.3. Repeatability of the PrepASH129 furnace

It is also important to check the day-by-day repeatability of the PrepASH129 furnace. Hereto we kept about 100g of 2 stabilized laboratory samples in a closed container and we ran these samples over 5 different batch runs (i.e. different days). We have selected 2 lots, one with an LOI of about 8%, and one with a rather high LOI of about 28%. The results are presented in table 4.

These results show the excellent repeatability of the PrepASH129 furnace.

lot 6			
day	crucible	result	average/day
1	1	7.69	7.66
	2	7.64	
2	1	7.61	7.61
	2	7.61	
3	1	7.67	7.65
	2	7.64	
4	1	7.64	7.64
	2	7.63	
5	1	7.63	7.65
	2	7.66	
min			7.61
max			7.66
difference			0.05

lot 7			
day	crucible	result	average/day
1	1	28.21	28.19
	2	28.17	
2	1	28.20	28.20
	2	28.20	
3	1	28.21	28.22
	2	28.23	
4	1	28.26	28.24
	2	28.22	
5	1	28.25	28.25
	2	28.25	
min			28.19
max			28.25
difference			0.06

Table 4. Repeatability of the prepASH129 furnace for 2 lots. (Lot 6, Lot 7 = beads)

4.4. Comparison of labor time

The labor time for an LOI analysis in duplicate is reduced by 30 minutes due to the efficient and easy concept of the LOI furnace.

5. General conclusion

Concerning the "as is" sample, we can conclude that:

- one sample bag is sufficient for the LOI determination of the lot
- the LOI by the PrepASH129 is slightly higher compared to the actual method. This can be explained by the moisture uptake while cooling to room temperature before weighing.
- the procedure with the PrepASH129 is less time consuming compared to the actual method.

Concerning the laboratory sample, we can conclude that the PrepASH129 is an excellent tool providing us accurate, precise, and repeatable LOI figures for less labor time.

For both the "as is" and the laboratory sample, the prepASH129 furnace is robust, easy to learn and presents a visual interpretation of the processes occurring in the sample, ensuring the complete calcining of the sample.

Taken all this into account, Umicore will implement the use of this furnace for the "as is" sample and for the laboratory sample in the near future.

Ref. 1: UOP 954-03 Loss on Ignition (LOI) for Fresh, Regenerated, Used, and Spent Catalysts, Catalyst Supports, and Adsorbents (ASTM)