

Loss on ignition analysis with prepASH 340 Series for catalysts

The Loss on Ignition (LOI) is an important analytical parameter in the assay of petrochemical catalysts.

Loss on ignition with specified ignition temperature and time is done for fresh, regenerated, used, and spent catalysts, catalyst supports, and molecular sieves of various shapes and sizes, including fines. Time and temperatures are depending on composition of the catalysts. The method is not water specific; any components that volatilize at the selected ignition temperature, for example, carbon, chlorine, and fluorine, are also included in the LOI value. Some components, for example sulfates, may volatilize fully or partially depending on ignition time and temperature. Mass gains are possible with catalysts that contain reduced metals.

Automation of loss on ignition brings efficiency, quality and security into the laboratory. The step of cooling down the samples before weighing after ashing is skipped. That is a huge advantage over the manual method not only in time saving but mainly in quality. The cooling down is a very critical part in the LOI determination.

Reference customer  **umicore**
materials for a better life

Umicore and Hydrocarbon Engineering Umicore Belgium: Precious metal recovery Two prepASH129 and one prepASH229 in use. Different methods are in use (temperatures up to 1000 °C for 14 h)

Recycling of precious metals from petrochemical catalysts

The oil refining, petrochemical and chemical industry uses precious metal containing industrial catalysts. Catalysts are used to facilitate chemical reactions or to eliminate atmospheric emission of pollutants 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.

These contamination are determined as LOI. The LOI allows the calculating the metal content to a reproducible calcined (ed) lot size.

Contractual handling of spent industrial catalysts

While the supplier remains the owner of the returnable precious metals contained in the incoming spent catalyst, the first step upon arrival involves weighing and sampling in the presence of the supplier's representative. Later, once both parties have completed their own assays, they agree on a date on which they will exchange them. This ultimately leads to a final 'settlement' following agreed splitting rules, in some cases often having had recourse to an independent umpire laboratory.



Precisa APPLICATION

Processing of spent industrial catalysts

Historically, Umicore Precious Metals processes the larger part of available spent palladium bearing hydrocracking catalysts in the world, characterised by an insoluble carrier (most often Al₂O₃-SiO₂ zeolites) and a **relatively high loss on ignition** (typically 10 - 20%, sometimes as high as 35 - 40%), with or without base metal HDS (hydro-desulphurisation) catalyst and/or inert support balls. In the latter case, a creative modification to the weighing and sampling procedure is provided for. **The result of LOI is consequently important for sampling and treatment of sample and lot.**

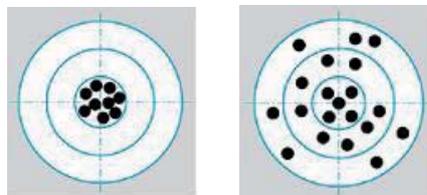
Reproducibility and Accuracy, Measured with an prepASH® 129 for analysis of petrochemical catalyst

"Permission granted for publication by Umicore and Hydrocarbon Engineering. Umicore (Belgium) is a specialty materials group with activities centered on four business areas: Advanced Materials, Precious Metals Products and Catalysts, Precious Metals Services and Zinc Specialties. Hydrocarbon Engineering is a scientific journal published by Palladian Publications Ltd (England)".

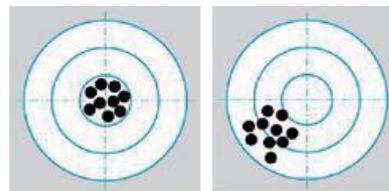
The Loss on Ignition (LOI) is an important analytical parameter in the assay of petrochemical catalysts. The LOI of the "as is" sample has an impact on the net lot size. The LOI on the laboratory sample has an important influence on the final laboratory result, expressed on the calcined material. This paper will show the excellent repeatability and precision of a Precisa prepASH® 129 automatic drying and ashing furnace for the determination of the LOI in petrochemical catalysts.

Reproducibility: These results show that the precision of the LOI, determined with the prepASH129 is excellent.

	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	0.03	0.02	0.00	0.01	0.02



Accuracy:



Calcium oxalate monohydrate decomposes in different steps to calcium oxide.
 $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O} \rightarrow \text{CaC}_2\text{O}_4 + \text{H}_2\text{O}$: 12.3% referred to $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$
 $\text{CaC}_2\text{O}_4 \rightarrow \text{CaCO}_3 + \text{CO}$: 31.5% referred to $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$
 $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$: **61.6% ref. to $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$**
prepASH: LOI 38.4%

The thermogram recorded for Calcium oxalate monohydrate with the prepASH® 129.

Concerning the laboratory sample, we can conclude that the prepASH® 129/229 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 prepASH 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 has implemented the use of this furnace for the "as is" sample and for the laboratory sample.

Precisa APPLICATION



prepASH – optimal solution to determine ash

Reduced time and effort. prepASH is a fully automatic drying and ashing equipment, so no multiple weighing back after time consuming cooling down in the dessicator but automatic calculation of results. Working in groups of similar samples in a single run will rise efficiency and optimise time of analysis.

Improved safety and efficiency. No more dangerous analysis with the open flame. With prepASH analyses can be done in time slots unused or hardly ever used so far, e.g. at night.

Increased quality. Up to 20% of each ash determination has to be re-analysed because of faulty/undefined results. prepASH is highly repeatable and reliable!

Detailed analysis reports. Due to the permanent recording of measurements during the entire process and the automatic saving of the final results, all data are retrievable at any moment.

Working Steps of moisture and ash determination

Standard Method with oven	vs.	prepASH
Heating out crucibles for constant weight before	Dry matter	Possibility to pre-define a "heating out"
Measuring tare of crucible one by one		AUTOMATICAL PROCEDURE
Sampling		Sampling
Weighing + documentation of each crucible		AUTOMATICAL + entering the sample
Samples in drying oven + START		START PROGRAM
Removing samples from oven + cool down		RESULTS (moisture)
Back weighing Samples, calculation (moisture)	Ash	RESULTS (ash)
Pre-ashing with rapid incinerator or hot plate		
Samples in muffle furnace		
Removing samples + cooling down in exsiccator		
Calculation and documentation (ash)		
Back weighing for stable results (repeat?)		