

Services and Techniques

At EAG Laboratories in Toulouse, France, we provide our clients with a local scientifically sound solution with fast turnaround times, including 72h, 48h and 24h upon receipt of the sample. We know how to solve your scientific problems.

Elemental Analysis by Glow Discharge Mass Spectrometry - GDMS

Best Quality Services

To stay at the highest technological level and to provide you with the best services, we seek to constantly develop our instrumentation and analytical services. Consequently, we added two state-of-the-art Nu Astrum instruments (figure 1) to our GDMS capacities. This instrument allows us to increase the number of samples on one hand, and to perform analyses quickly, to continue serving you and keeping you satisfied.

Figure 1: GDMS instrument - Nu Astrum

Principle

To keep it simple, GDMS relies on the creation of a low-pressure Argon plasma that allows the sputtering and the ionization of the material of interest. An Ultra-Pure Argon stream is introduced in the analytical cell where the sample

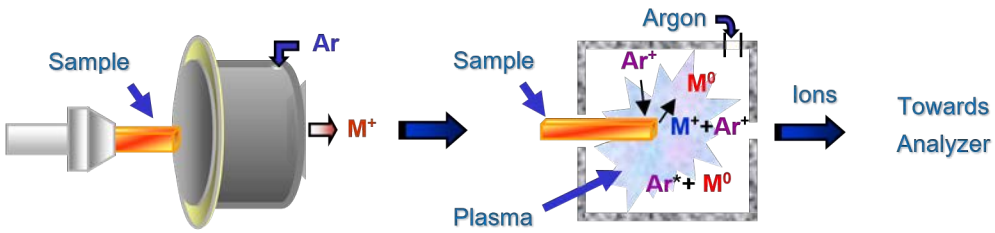

Figure 2: Simplified scheme of the GDMS principle

has been first inserted. A 1 kV direct current voltage is applied between the cell body and the sample in order to ignite the plasma. This Argon plasma bombards the surface of the sample. The sputtered species are then atomized, ionized, and extracted from the cell to fly through the High-Resolution (HR) analyzer system.

The HR analyzer is a double focused mass spectrometer (magnetic and electrostatic) and separates the ions depending on their mass on charge ratio m/z.

Detection system is a combination of different detectors which allows the sequential determination of majors, minors, trace, and ultra-trace contents, down to the ppb level.

Figure 3: GDMS instrument - Element GD

Sample Shape

GDMS can be useful on all kind of inorganic samples, conductive, semi-conductive and even non-conductive. The different configurations we can adapt on our instruments allow every problem to be answered.

Solid Conductive Samples

The most straightforward way to analyze these samples is to introduce them directly in the instrument. As is, if the analytical surface is smooth and flat, and measures at least 20 x 20mm. Under pin form, if the sample can be cut at the following size: 20 x 2 x 2 mm. The analysis requires a preliminary mechanical preparation and a chemical etching to remove every potential contamination brought by the mechanical preparation.

Element	Concentration (ppmwt)	Element	Concentration (ppmwt)
Li	0.001	Ag	0.005
Be	0.001	Cd	0.01
B	0.005	In	0.005
F	0.05	Sn	0.005
Na	0.005	Sb	0.005
Mg	0.005	Te	0.05
Al	0.005	I	0.05
Si	0.001	Cs	0.005
P	0.005	Ba	0.005
S	0.05	La	0.005
Cl	0.01	Ce	0.005
K	0.01	Pr	0.005
Ca	0.01	Nd	0.005
Sc	0.001	Sm	0.005
Ti	0.005	Eu	0.005
V	0.001	Gd	0.005
Cr	0.005	Tb	0.005
Mn	0.005	Dy	0.005
Fe	0.005	Ho	0.005
Co	0.005	Er	0.005
Ni	0.005	Tm	0.005
Cu	Matrix	Yb	0.005
Zn	0.01	Lu	0.005
Ga	0.05	Hf	0.005
Ge	0.05	Ta	1
As	0.005	W	0.005
Se	0.01	Re	0.005
Br	0.005	Os	0.005
Rb	0.005	Ir	0.005
Sr	0.005	Pt	0.005
Y	0.005	Au	0.01
Zr	0.005	Hg	0.01
Nb	0.005	Tl	0.001
Mo	0.005	Pb	0.005
Ru	0.005	Bi	0.005
Rh	10	Th	0.0001
Pd	0.005	U	0.0001

Figure 4: Table of typical detection limits for a Copper sample analyzed by GDMS

Powders and Non-Conductive Samples

The analysis of powders, or samples with a low conductivity, needs to be performed with the use of a high purity conductive binder (usually 7N grade Tantalum or Indium). This binder holds the sample mechanically and is also used as a secondary electrode to allow the ignition of the plasma.

Applications, Accuracy, and Uncertainty

GDMS can analyze the elemental trace concentrations of the whole periodic table (Hydrogen and noble gases taken apart). High Resolution allows us to surely solve most of the existing interferences, giving the ability to accurately measure percent to ultra-trace (ppbwt) contents with a relative instrumental precision of about 20%.

COFRAC Accreditation - ISO 17025 Norm Certification for HR-GDMS Analyses

Our quality system and our continuous improvement policy has led our laboratory to be accredited by the French Accreditation Committee. We're pleased to announce that this accreditation has recently been extended until 2022. Our HR-GDMS service has been accredited for most of the materials needed to be usually analyzed, as for example allied steels, Nickel alloys, Titanium alloys, Aluminum alloys or Copper samples.

The accreditation scope is available on the COFRAC website (www.cofrac.fr) with the accreditation number 1-1993.

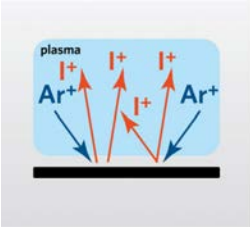

Figure 5: Simplified scheme of GDMS measurements

Interstitial Gas Analysis - IGA

We are internally equipped with three types of IGA instruments, covering specific needs in the determination of interstitial gases H, N/O and C/ S, and completing the range of the results provided by GDMS and ICP-OES/MS. These instruments allow us to provide you with a complete evaluation of the whole periodic table, from Hydrogen to Uranium.

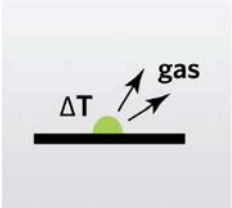

Figure 6: Simplified scheme of IGA measurements

Analysis of O, N & H Contents by Inert Gas Fusion

Inert gas fusion (using Argon or Helium) of a 1-gram sample is performed in a graphite crucible, deposited in an oven. The concentrations of the H_2, N_2 and O_2 gases that are released during the fusion are then determined using either a Thermal Conductivity Detector for H_2 and N_2, or an Infrared detector for O_2. At least three replicates per sample are analyzed, leading to a more representative evaluation of the whole sample, in terms of homogeneity.

Figure 7: LECO ONH836 Analyzer

Analysis of C and S Contents by Combustion

This time, combustion using pure Oxygen of a 1-gram sample is performed in a ceramic-made

crucible. Concentrations of the gases released in the oven, CO_2 and SO_2, are then measured using an infrared detector. At least three replicates per sample are also performed in order to have a more representative idea of the homogeneity of the sample.

Figure 8: LECO CS844 Analyzer

Applications and Limits of Detection (LOD)

All inorganic materials are theoretically suitable for IGA measurements. We can then consider the analysis of metallic matrices (Si, Cu, Ni, Co), alloys (Nickel superalloys), solid solutions (steels, shape memory alloys NiTi), refractory materials (SiC, Ta, W), oxides (Al_2O_3), nitrides (Si_3N_4), and many more.

Element	Utimate Limit of Detection (ppmwt)
H	0.1
C	1
N	1
O	1
S	1

Figure 9: Table of Ultimate Limits of Detection obtained on 7N purity Copper sample

ISO 17025 Accreditation for H, N, O, C and S Analyses

Our quality system and our continuous improvement policy has led our laboratory to be accredited by the French Accreditation Committee. We're pleased to announce that this accreditation has recently been extended until 2022.

Our IGA service received this accreditation for most commonly analyzed materials, like copper, steels, nickel alloys, titanium alloys and aluminum alloys. The accreditation scope is available on the COFRAC website (www.cofrac.fr) with the accreditation number 1-1993.

Example of Analyses in the Scope of Accreditation

Element	Concentrations in the scope (ppmwt)
C	100 to 4000
N	30 to 300
O	10 to 150
S	5 to 250
H	0.5 to 10

Figure 10: Table of elemental concentrations in the ISO 17025 accreditation scope

A High Standard Quality of Analysis

Our analytical methods are developed internally, while keeping American Society for Testing and Materials (ASTM) standards as models and examples.

- ASTM E-1447: H in Ti and Ti alloys
- ASTM E-1019: C, S, N & O in steels, Fe, Ni, Co and similar alloys

A daily quality control is performed with high metrological quality Reference Materials (CRM, primary standards) chosen from recognized providers like EURONORM-MRC, JSS, or BAM.

Thermogravimetric and Differential Thermal Analyses – TGA/DTA

Thermogravimetric Analysis, also known as TGA, monitors the evolution of a sample's mass as a function of temperature and time, under controlled atmosphere. TGA is very useful when precise information is needed concerning the determination of volatile elements or molecules, thermal stability, degradation characteristics and kinetics, or even behavior during sintering.

Figure 11: Simplified principle of a TGA/DTA measurement

For Differential Thermal Analysis, or DTA, the goal is to monitor the difference of temperature between the sample of interest and an inert reference, difference resulting from internal thermal events within the material. These internal events can be the result of a change of the chemical composition, or the crystalline structure of the studied material.

A new instrument has been acquired, Setaram LABSYS evo. It adds the possibility to monitor phenomenon from Room Temperature to 1600°C on organic, metallic or even ceramic materials. Atmosphere can be designed to fit your specific needs.

Figure 12: SETARAM LABSYS evo

All solid samples can be analyzed, without any preliminary preparation of the sample, including powders, chunks, flakes, and similar. The analysis can either be qualitative or quantitative.

We are ready to study your demand and adapt our existing procedures to fit your particular needs, to provide you with answers to your own problems. This can include:

- Composition study, deformulation
- Melting points, Lost on Ignition (LOI)
- Study of chemical reactions (hydrogenation; reaction kinetics; pyrolysis; sintering; …)
- Monitoring of degradation, study of thermal stability
- Comparison between different batches, failure analysis, and so much more

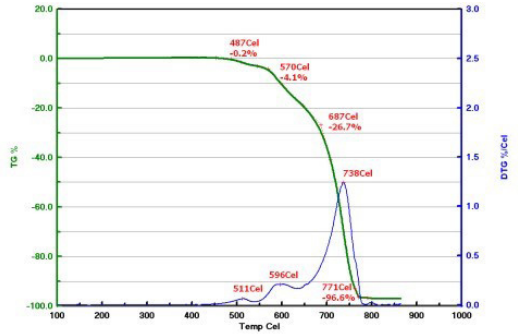

Figure 13: Results obtained by TGA/DTA

Analysis of Major Elements by Inductively Coupled Plasma Optical Emission Spectroscopy – ICP-OES

GDMS and IGA measurements are completed in our facility by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), in order to accurately measure the major concentrations in the material of interest. ICPOES is a multi-element technique, relying on wet chemistry, as the samples need to be introduced in the plasma in the liquid form.

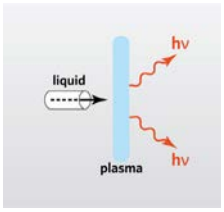

Figure 14: Simplified scheme of ICPOES measurements

We recently acquired a brand-new instrument, Analytik Jena PQ9000, to perform these analyses.

Figure 15: Analytik Jena PQ9000 instrument

It is mandatory to first mechanically prepare the sample. Our laboratory is equipped with instruments and tools designed to mechanically prepare all types of samples. It is then possible to transform solid samples into small chunks, or sample special areas of interest. The samples are then digested using specific acid mixtures, that are internally developed as a function of the material needed to be analyzed. Once again, specific equipment, such as hot plates, microwaves or even high-pressure bombs in an oven, is used to digest the most difficult matrices.

Instrument Check and External Calibration Curves

Analysis of the samples is performed after a daily instrumental check, against a list of criteria, as for example signal intensity, stability and oxides level.

The instrument is then calibrated to measure its response as a function of the analyte's concentration. Elemental standard solutions, traceable to NIST, are used to gravimetrically prepare calibration curves, allowing an increased accuracy.

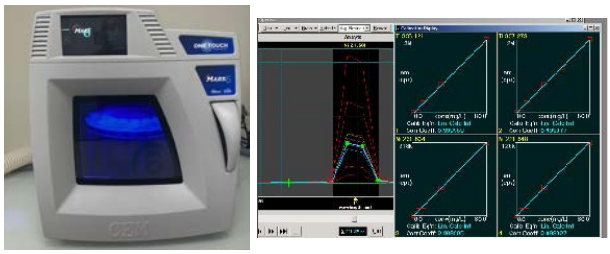

Figure 16: Microwave-assisted digestion system (left) and instrumental calibration curves (right)

Applications and Strengths

Our ICPOES instrument is dedicated to the analysis of major elements in organic or inorganic materials. Our laboratory is able to deal with metallic alloys, superalloys, oxide powders, and many more. Our experienced team can adapt a solution to your particular sample, if your sample request more than a straightforward digestion or analysis.

ICPOES enables the determination of concentrations associated to instrumental uncertainties of about 5% for concentrations above 1 wt%, and about 10% for concentrations below 1 wt%.

Our internal procedures include the sampling of 3 different replicates (generally between 0.1 and 0.5g) from the initial sample. The average of the 3 measurements is reported, with the standard deviation of these replicates. This information can be used to understand the homogeneity of the sample if the standard deviation is low, or inhomogeneity if the standard deviation is higher.

Element	Concentration (in wt%)	Specifications (in wt%)
Si	0.381+/-0.006	< 0.75
Cr	19.50+/-0.08	18-20
Mn	1.76+/-0.01	2 max
Fe	Balance	Balance
Ni	9.71+/-0.03	8-12

Figure 17: Example of a steel sample: ICPOES results and specifications

The example presented in Figure 17 shows that the ICPOES measurements are fulfilling client's expectations, even for the tightest specifications. A closer look on the standard deviations show a low dispersion of the results coming from the 3 replicates, traducing the homogeneity of the analyzed sample.

Contact Us

For more information concerning the techniques presented in this document, or the complementary techniques provided by our Eindhoven, Netherlands location or any of our US sites, please feel free to contact us, using the following phone number and email address. We'll be pleased to find a dedicated solution for you.

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