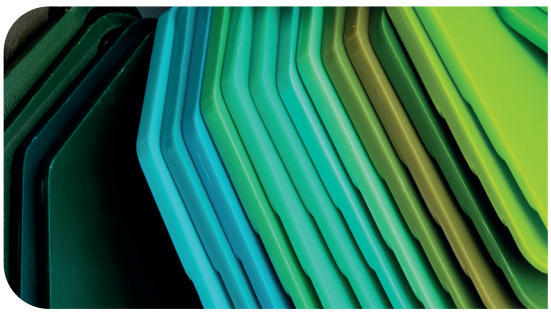
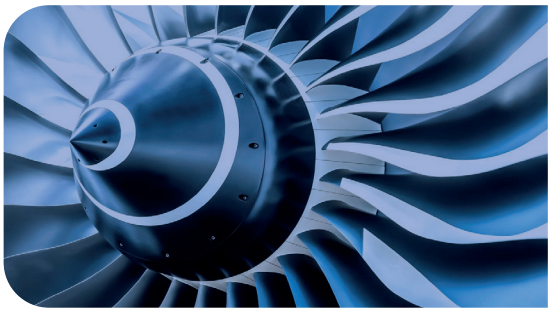
SRA INSTRUMENTS
ANALYTICAL SOLUTIONS

HYPHENATED TECHNOLOGIES FOR MATERIALS DEFORMULATION





The need of deeply investigate material characterization can be referred to many different markets from rubbers, plastics, resins, pharmaceuticals, adhesives to packaging and it is crucial in order to capture all the hidden information of an unknown sample. **Thermal Gravimetric Analysis** (**TGA**) is a powerful deformulation tool common in material analysis with which, thanks to a temperature ramp, the sample is breakdown and tracked through weight loss as components vaporize. While providing quantitative information through weight loss, this process does not give insights into the chemical identity of the off-gassing materials. Coupling, or hyphenating, different instruments can uncover results that are not possible to reach with individual techniques. The power of the analysis therefore becomes greater than the sum of its parts.

SRA Instruments developed different possible solutions dedicated to the **Evolved Gas Analysis** (**EGA**) based on coupling different instrumentation to a TGA.

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LUNIA TGA-MicroGC

- **LUNIA TGA MicroGC** is a fast organic and inorganic gas analyzer on its own.

  It's a modular device made by 1 to 3 analytical modules working simultaneously and offering separation of compounds from a gas mixture within 3 minutes.

  Each module is a complete chromatograph with micro-injector, capillary column and universal thermal conductivity detector (µTCD).

  The MicroGC sequence of analysis is started by the TGA at the beginning of the thermal degradation.

  Then the compounds generated from evolved gas are separated and detected every 2 or 3 minutes in a concentration range from few ppm to 100 % with a very good linearity.

TECHNICAL SPECIFICATION:

- Configurable MicroGC with up to 3 modules;
- Less than 3 minutes of analysis time;
- Heated interface with a software programmable valve;
- Selectable alarms related to concentrations, trends or comparison.

SOLIA TGA-MicroGC/MS Interface

- **SOLIA** is a **SRA MicroGC/MS** solution ready to be coupled to a TGA. The heated transfer line is designed to analyze the evolved gases. The configuration includes heated membrane filter that protects the analytical module injector from heavy compounds and residues.

  The µTCD of each MicroGC module can be coupled to an Agilent quadrupolar mass spectrometer which makes possible an easy identification of each separated compound. The identification is simply made using mass spectrum comparison with the NIST database.

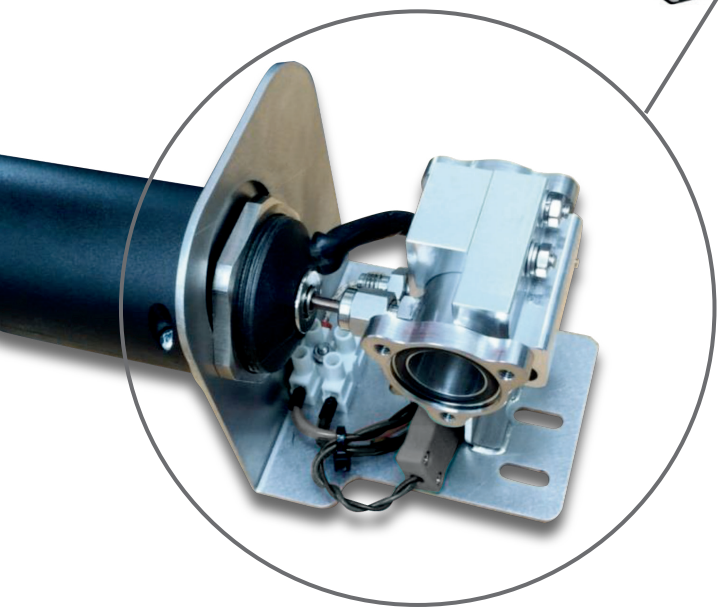
  The TGA/MicroGC coupling is the perfect solution for fast qualitative and quantitative analysis of evolved gas from a polymer or solid sample.

TECHNICAL SPECIFICATION:

- MicroGC/MS coupling;
- Automatic valve selection of the channel coupled to MSD before or during the sequence;
- Configurable MicroGC with up to 3 modules;
- Less than 3 minutes of analysis time;
- Heated interface with a software programmable valve.

ZEFIRUS TGA-FTIR Interface

- The combination of a **Thermogravimetric Analyzer** (**TGA**) with an **Infrared Spectrometer** (**TG-IR**) is the most common type of **Evolved Gas Analysis** (**EGA**) in use today. Coupling the TGA to a Fourier Transform Infrared (FTIR) spectrometer adds considerable utility to the method.



The vapors released from the material pass through a gas cell inserted in the FTIR, where the spectrum is obtained. Spectral searching provides the last link of vapors identification and thanks to the ability to detect functional groups, FTIR analysis allows greater understanding of the processes seen in the TGA.

TECHNICAL SPECIFICATION:

- Active flow technology: real quantitative analysis with no FTIR signals overlapping;
- Heated gas cell and SilcoSteel® transferlines up to 350 °C;
- Zero gravity cell design: no more fouling or dirtiness phenomena;
- Customized adapter for every TGA model;
- SRA Kinetic software directly controls Agilent Cary 630 FTIR: time resolution analysis and live quantitative data;
- Automatic triggering.

What is the Active Flow Principle?

PV = nRT

- The SRA interface we designed is based on the idea of active flow sampling with which, thanks to a pump and a mass-flow controller, the amount of the gases mass entering the gas cell is always constant during all the experiment, independently from gas density.

In other words, the pump modulates its action considering the gas expansion respect to the temperature of the TGA furnace. In this way the FTIR data will always match the TGA graph during all run time of the experiment avoiding dilution factor due to the gases expansion.

- **Active Flow Technology** is common to Maximus and Zefirus SRA interfaces.

TGA-GC/MS INTERFACE

- Thermogravimetry combined with gas chromatography and mass spectrometry (TGA-GC/MS) can be one choice for the qualitative and quantitative analysis of evolved gases.

During the thermal degradation cycle of the materials, the composition of the evolved gas changes too fast for a GC/MS analysis in its standard configuration.

With IST Interface, the chromatographic separation time is no longer a limitation for the detailed study of complex thermal analysis profiles.

IST1 Interface

- The **IST1 interface** allows to store 1 loop of the gaseous effluent from the TGA and to automatically inject it into the GC/MS. The IST1 is designed to be coupled to any TGA and GC/MS models. IST1 can be upgraded from 1 to 16 loops.

TECHNICAL SPECIFICATION:

- 1 storage loop;
- Transfer lines temperature up to 250 ºC;
- Loop volume: 250 µL in standard, customised volumes on request;
- Multisystem compatibility;
- Fully automated;
- Lines and loops treatment: Sulfinert.

IST16 Interface

- **IST16 interface** allows to sequentially store in 16 loops several fractions of the gaseous effluent from the TGA and to automatically inject these samples into the GC/MS. The gases, evolved during the cycle of the TGA, are transmitted to the storage interface and then to the GC via heated and temperature controlled micro-volume transfer lines and IST16 is designed to be coupled to any TGA and GC/MS models. The IST16 is supplied with a dedicated interface.

TECHNICAL SPECIFICATION:

- 16 storage loops;
- Transfer lines temperature: 250 ºC;
- Loop volume: 250 µL in standard, customised volumes on request;
- Multisystem compatibility;
- Fully automated;
- Lines and loops treatment: Sulfinert.

WITH IST16 INTERFACE IT IS POSSIBLE TO WORK

Collecting-revolving

- charge different loops following a time table filled by operator and then inject in the GC.

Continuous single-loop

- charge one single loop and direct injection in the GC.

MAXIMUS TGA-FTIR-GC/MS Interface

- Hyphenating **TG-IR-GC/MS** is the most powerful and complete approach to material deformulation. The specialist will be able to capture many different information from each instrument. This will lead to characterize deeply an unknown mixture to determine its primary components and identify additives or contaminants. This information may be needed, for example, to evaluate a competitor's product or to determine compliance with regulations. Due to thermal degradation of the sample, the gases produced in the TGA furnace are transferred to the FTIR gas cell thanks to a hot interface. Here a spectral analysis takes place. Then hot gases will be further flowed to GC for chromatographic separation and to the MS detector for unambiguous identification. The GC/MS injection block was designed to avoid capillary cloggings, carry over and cold spots which could affect the analysis. The system allows real-time analysis thanks to the GC instrument which can separate and detect very low levels of material in complex and unknown mixtures.

- The system is configured for Active Flow Principle, ensuring the same mass of gases flowing in the FTIR cell and GC-valve over all time of the thermal experiment.

TECHNICAL SPECIFICATION:

- Active flow technology: real quantitative analysis with no FTIR signals overlapping;
- Heated gas cell and SilcoSteel® transferlines up to 350 °C;
- FTIR Zero gravity cell design: no more fouling or dirtiness phenomena;
- Customized adapter for every TGA model and automatic triggering;
- Triggered GC injection valve heated up to 350 °C;
- SRA Kinetic software directly controls Agilent Cary 630 FTIR: time resolution analysis and live quantitative data.

TWO WAYS OF OPERATION:

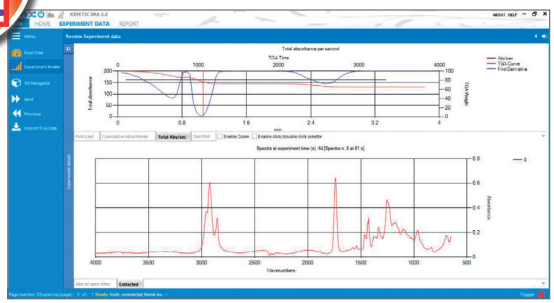
TGA-FTIR-MS
time base mode:

- No-more carry over: the sample is introduced into the MS in a non-continuous way; it is possible to set an injection sequence for the introduction of the sole fractions of interest, thus limiting the risks of system fouling (loop wash, loop load).

TGA-FTIR-GC/MS
separation mode:

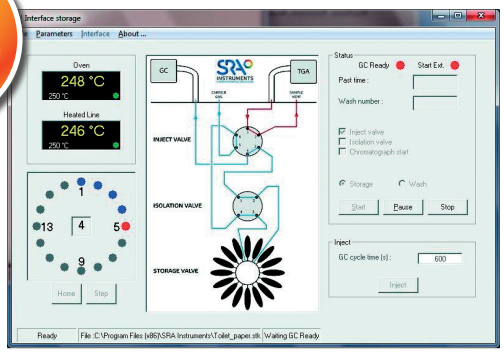
- The gas sample is transferred to the injection block only during the TGA analysis. It is possible to set the introduction of the sample fractions at constant time intervals or at variable time intervals following the progress of the TGA experiment. The gases evolved into TGA are transferred to FTIR and then directly to the GC/MS system for the selective determination of the various compounds.

SRA Kinetic Software



- The **SRA Kinetic software** is a user-friendly software developed to manage real time monitoring analysis for the Agilent Cary 630 FTIR. The acquisition parameters are saved in a single method, and spectra are saved in a single file for post-processing. During the experiment the specialist can display two overlaid spectra and monitors the trend of two spectral areas of interest with the possibility to have a 3D view of spectra over time. SRA Kinetic is compatible with TGA triggered acquisitions and let to have real-time quantification of an interested peak.

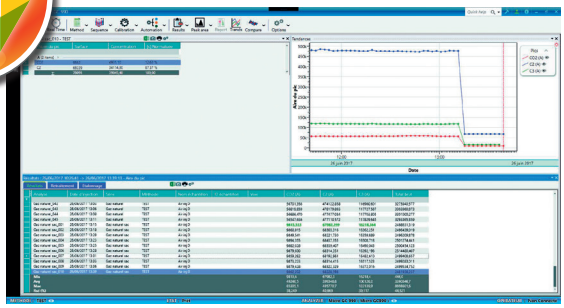
SRA IST Software



- The **SRA IST software** allows to edit the storage sequence, save the methods, view the status of the instrument and automatically manage the start of the GC analyses.

  The gases evolved during the cycle of the TGA are transmitted to the storage interface and then to the GC via heated and temperature controlled micro-volume transfer lines.

SOPRANE Software



- Developed by **SRA, SOPRANE II** performs the complete management of an analysis, from the sampling to the transmission of the results. A powerful graphical environment provides effectiveness and ease of use: it is possible to define a method and a sequence of analysis, to follow the trends for specific compounds during a TGA analysis. Soprane manages the mass spectrometer and its Agilent Masshunter software and compiles all results in a same report.

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