Thermal Analysis Excellence

TGA-IST16-GC/MS 1

STAR^e System

Innovative Technology

Versatile Modularity

Swiss Quality

TGA-IST16-GC/MS System

for Unmatched Performance

METTLER TOLEDO

TGA-IST16-GC/MS System
Unsurpassed Detailed Insight

Thermogravimetric analysis combined with gas chromatography and mass spectrometry (TGA-GC/MS) is nowadays the method of choice for qualitative and quantitative evolved gas analysis (EGA). This combination of methods allows thermal effects to be correlated with information about the molecular nature, structure and composition of materials.

METTLER TOLEDO goes a step further with the introduction of the IST16, a 16-channel storage interface. The TGA measurement can be divided into 16 segments. This allows the decomposition gases evolved from the TGA to be clearly assigned to individual steps in the TGA curve.

Features and benefits of the METTLER TOLEDO TGA-IST16-GC/MS 1:

- **Gas analysis by GC/MS** – Determination of the molecular composition and information about the molecular structure of the sample
- **IST16 interface with 16 storage loops** – allows a single TGA measurement to be divided into up to 16 separate GC/MS experiments
- **Independent TGA and GC/MS experiments** – GC/MS analysis does not require the TGA measure-ment to be stopped and so saves time
- **Automatic operation** – straightforward and efficient
- **Different modes** – selection of the best mode of operation (storage mode or continuous mode)
- **Direct coupling of the TGA to the GC/MS** – cost-effective solution for a single GC/MS analysis

The IST16 interface enables the time at which thermal effects occur to be correlated with the results of the mass spectrometric analysis.

The TGA-IST16-GC/MS system is the ideal solution for the comprehensive qualitative and quantitative molecular analysis of gaseous decomposition products.

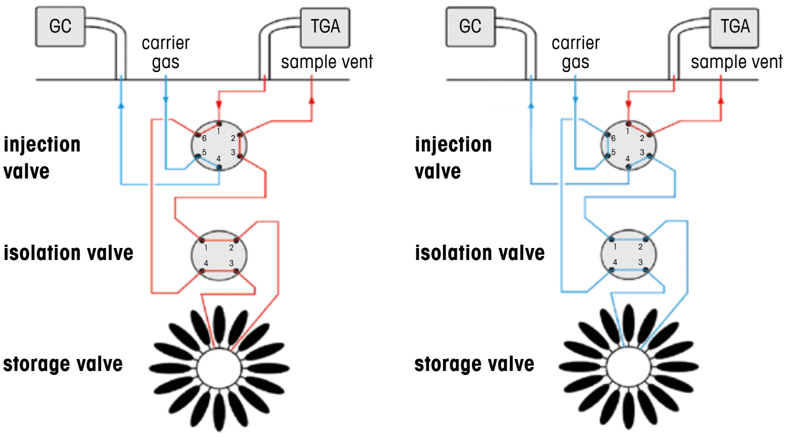
IST16 Storage Interface
Complete Molecular Identification

The unique IST16 storage interface can be operated in several different modes:

Storage mode
The IST16 allows the user to collect and store up to 16 gas samples (volume 250 µL) from the TGA at freely selectable times for subsequent processing in the GC/MS. The gas samples are automatically sequentially analyzed by GC/MS. Each gas sample can be analyzed differently.

Continuous mode (with or without IST16)
In the continuous mode, only one storage loop is used. Gas samples are collected from the TGA at defined time intervals and transferred to the GC injector. This can lead to mixing of the injected gas sample with the previous gas sample.

Heated transfer connections
The transfer lines to and from the storage interface are made of stainless steel capillary tubing and are heated to prevent the gases from condensing. The IST16 interface can be heated up to 300 °C.



Software for the IST16 storage interface
The IST16 software together with the STARe software controls every step of the experiment.

- An individual GC/MS method can be defined for each storage loop. Sequential processing of the stored samples (storage mode)
- Sequential gas transfer to the GC column (continuous mode)
- Automatic washing of the connection lines and storage loops after the experiment
- The software also allows operation of a TGA directly coupled to a GC/MS

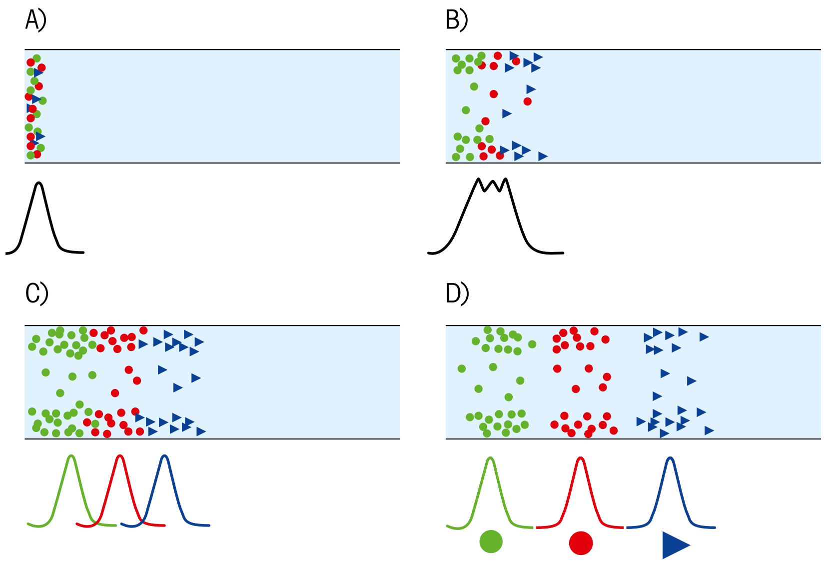
GC/MS System
Schematic Diagram of the System

A GC/MS system consists of a gas chromatograph directly coupled to a mass spectrometer. The gas sample for GC analysis is transferred from the IST16 to the GC via a heated connection line.

Gas chromatographic separation
Gas analysis begins with the separation of the components of the gas mixture by gas chromatography. The volatile compounds are transported through a meter-long column by a carrier gas (the mobile phase). The column is filled with a solid adsorbent or a liquid distributed as a thin film on a support material (the stationary phase).

During their passage through the column, the molecules are continually exchanged between the two phases. Depending on their relative affinity for the stationary phase, the individual compounds reach the end of the column at different times.

Separation of compounds ●, ●, ▶



Mass spectrometer
The mass spectrometer consists of:
- Ion source
- Analyzer
- Detector

In the quadrupole mass spectrometer used here, the ions are formed by electron impact ionization (EI) in the ion source. This is the usual method for smaller gas molecules. The analyzer consists of two pairs of parallel rod electrodes located between the ion source and the detector. The voltages applied to the rods are set so that only ions of particular mass-to-charge ratios are able to pass through the rods and reach the detector.

Scanning a particular mass-to-charge range produces a mass spectrum. Summation of the intensity of all the ions yields the so-called total ion chromatogram (TIC), which indicates the amount of gas leaving the GC column at any time.

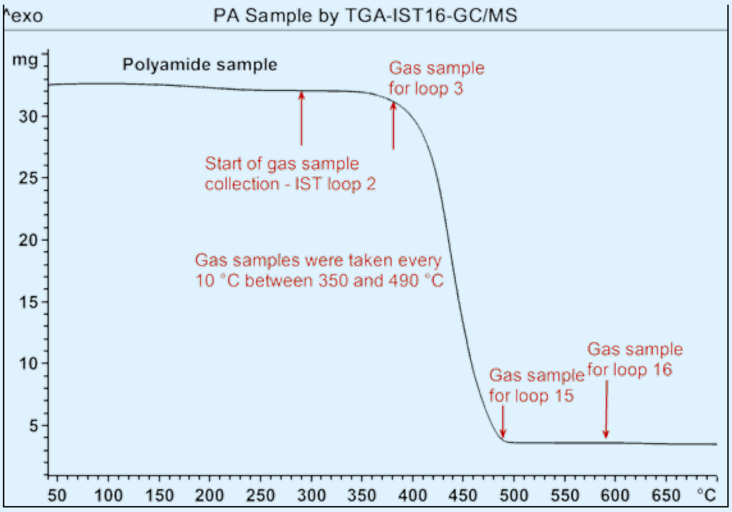
Wide Application Range
Complete Emission Gas Analysis

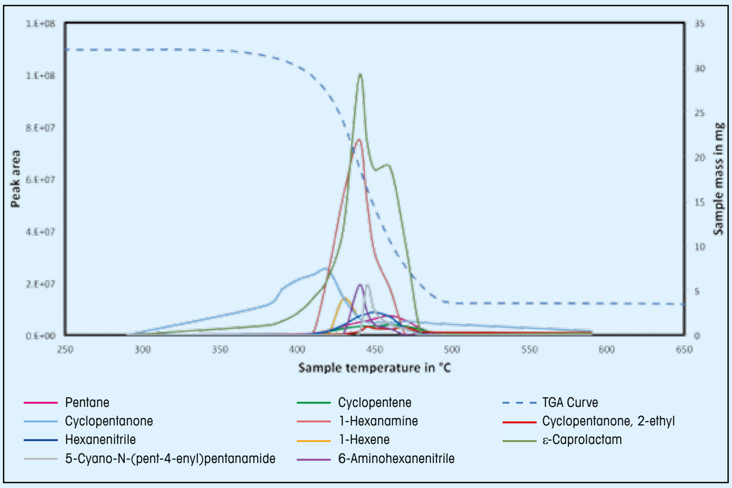
The TGA-IST16-GC/MS is the ideal instrument extension for the characterization of materials by thermogravimetric analysis. The combined system provides valuable information, whether it is used in quality control or for industrial and academic research.

Industry	Application
Automobile	Compositional analysis, volatile compounds, additives and fillers
Chemical industry	Decomposition products, isotopes, chemical reactions
Fats and oils	Decomposition products, identification of substances
Paints	Volatile compounds, chemical reactions, decomposition products
Elastomers (rubber)	Compositional analysis, volatile compounds, additives, identification of mixtures
Plastics (thermosets, coatings, adhesives, thermoplastics, packaging)	Plasticizers, decomposition products, additives and fillers, stability, identification of mixtures
Academic research/science	Decomposition products, stability, additives, auxiliary agents, isotopes, chemical reactions
Foodstuffs	Volatile compounds, stability, decomposition products, food additives
Pharmaceutical substances	Solvents, stability, degradation of substances
Biomass	Identification of pyrolysis products
Petrochemical substances	Identification of pyrolysis gases

Identification at the Molecular Level

Down to the Last Detail



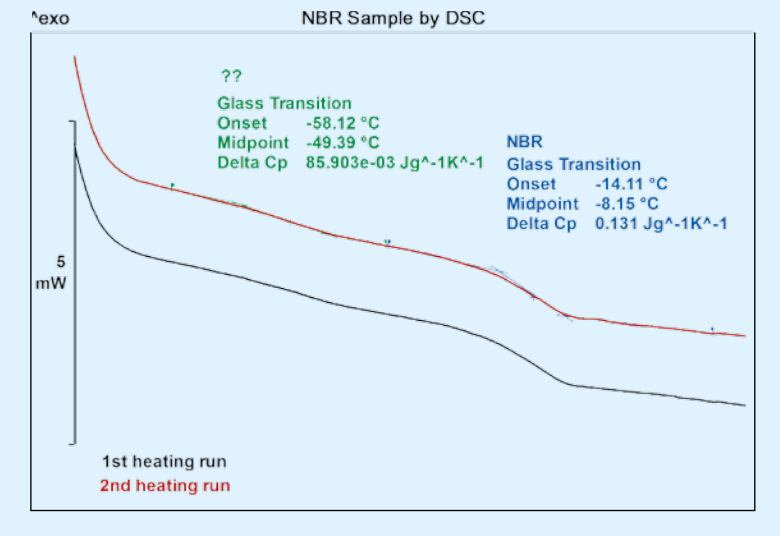


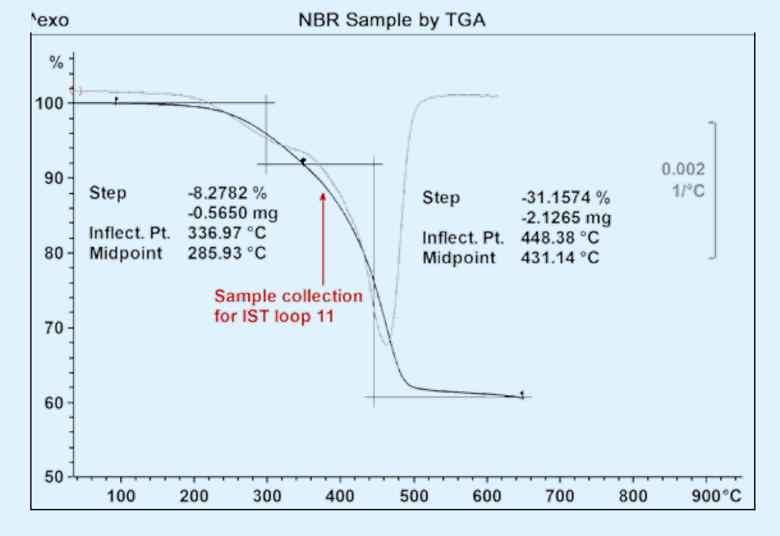
Characterization of a polyamide sample

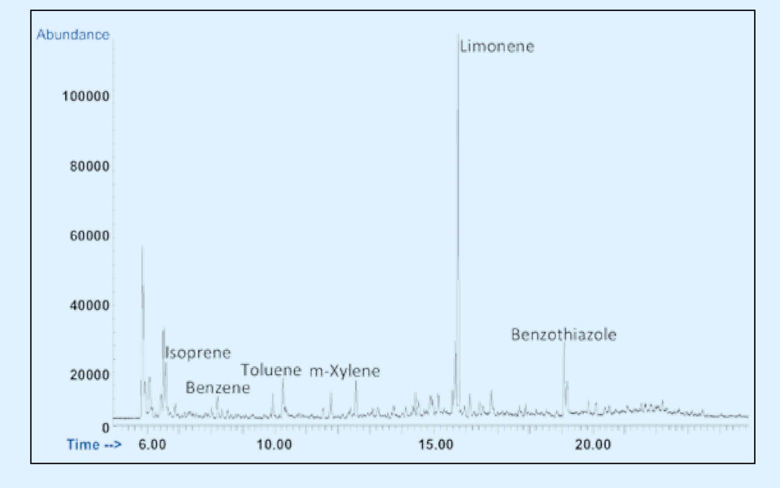
Nowadays, the incoming quality control of materials is of decisive importance for their further processing. The following example shows how the constituents of a sample of polyamide of unknown composition were determined by TGA-IST16-GC/MS. The main compounds detected were cyclopentanone, 1-hexanamine and ε-caprolactam.

Cyclopentanone and 1-hexanamine are typical decomposition products of PA 66, which is produced from hexamethylendiamine and adipic acid in a polycondensation reaction. ε-Caprolactam is a characteristic decomposition product of PA 6, which is produced in a ring-opening reaction of ε-caprolactam.

We can therefore conclude that the sample consists of a mixture of PA 6 und PA 66.







Detection of natural rubber in an allegedly pure NBR sample

Contrary to the manufacturer's specifications, synthetic rubber such as nitrile butadiene rubber (NBR, see the NBR glove in the photo) is often contaminated with natural rubber (NR). This is a problem because NR is a strong allergen and triggers allergic reactions with many people.

The experiments presented here describe the investigations performed to identify the components of an allegedly pure NBR sample. DSC measurements detected two glass transitions. This indicated the presence of an elastomer blend. The glass transition at –8 °C corresponds to NBR. The origin of the other glass transition at about –50 °C is unclear. TGA-GC/MS analysis was therefore performed to identify the unknown component responsible for this. Samples of gas were taken from the TGA at different temperatures and stored in the IST interface. The gas samples were then individually analyzed by GC/MS.

Evaluation of the MS analysis results showed that the main component of the elastomer was NBR (2-propene nitrile and 2-methyl-2-propene nitrile as typical decomposition products of NBR). Further main substances in the gas mixtures were limonene, isoprene and xylene. Limonene is a cyclic monoterpene with ten carbon atoms and is a typical decomposition product of NR. The presence of limonene eliminates the possibility of synthetic isoprene rubber as an impurity in the elastomer sample.

The second component of the elastomer was therefore clearly identified as NR. This result would not have been possible by conventional thermal analysis alone without GC/MS.

Leader in Laboratory Solutions

METTLER TOLEDO manufactures a wide array of balances and solutions for routine applications, research and development, and quality control. This is supported by innovative accessories, competent assistance and extensive applications know-how.

Microbalances, Analytical and Precision Balances
Innovation, reliability and quality have made METTLER TOLEDO famous for its balances. These qualities still determine balance standards today. A comprehensive range of accessories is available to solve every weighing task.

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The Excellence family of titrators provides highly automated state-of-the-art solutions for a wide range of routine to complex titrations and Karl Fischer moisture content determination.

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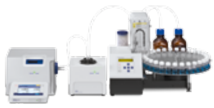
METTLER TOLEDO offers a wide range of bench and portable meters and electrodes for the determination of pH, conductivity, dissolved oxygen and ion concentration. There is a solution for every application, whether in the field, in production or in the laboratory.



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LiquiPhysics Excellence density meters and refractometers, MP melting point and DP dropping point systems offer incomparable ease of use including One Click™ operation.

Materials Characterization
The METTLER TOLEDO Thermal Analysis Excellence includes DSC, Flash DSC, TGA, TMA, DMA and microscope hot-stage systems and offers tailor-made solutions for research and development, quality control, and all kinds of academic and industrial applications.

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