



Thermal Analysis-GC-MS Coupling

Evolved Gas Analysis with Chromatographic Pre-Separation

STA/TGA-GC-MS Coupling

Ideal Design of Thermal Analyzers for Coupling

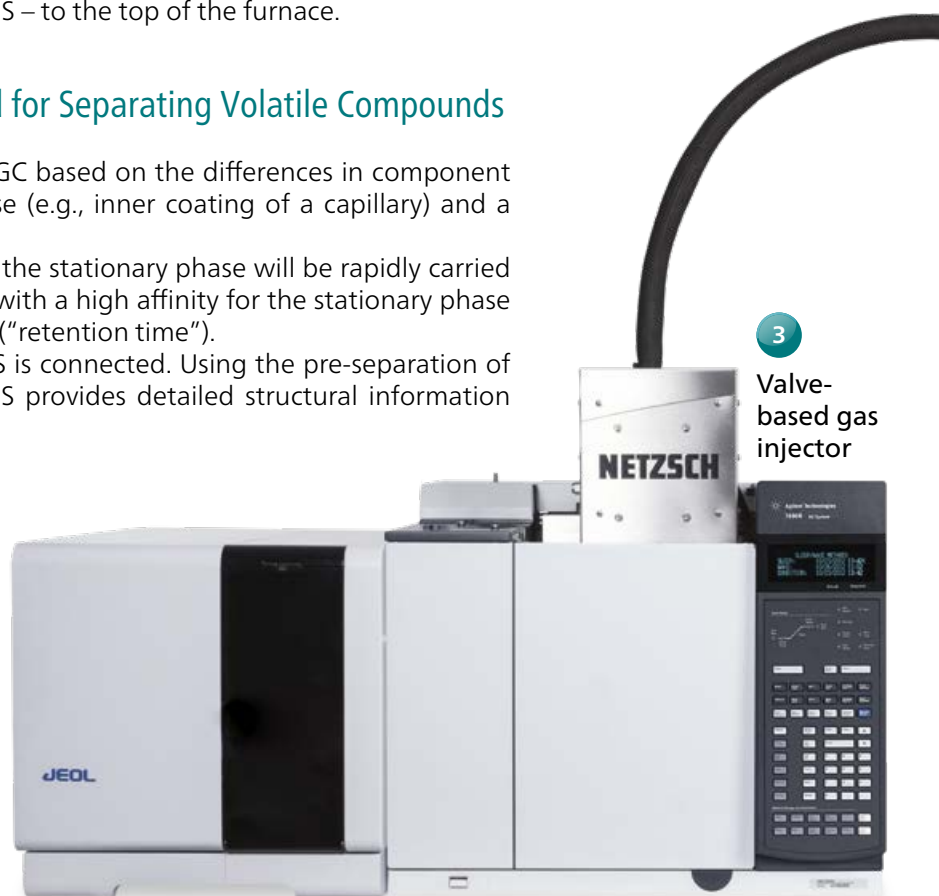
NETZSCH thermobalances (TGAs) and Simultaneous Thermal Analysis (STA) systems are arranged vertically with the sample above the balance, allowing the gases to flow in the natural upward direction. This design is ideal for coupling gas analysis instruments – such as MS, FT-IR or GC-MS – to the top of the furnace.

GC-MS – High-Resolution Method for Separating Volatile Compounds

The gas mixtures are separated in the GC based on the differences in component distribution between a stationary phase (e.g., inner coating of a capillary) and a mobile phase (e.g., He).

Gas components with a low affinity for the stationary phase will be rapidly carried away by the purge gas, whereas gases with a high affinity for the stationary phase will follow with a significant time delay (“retention time”).

At the outlet of the GC column, the MS is connected. Using the pre-separation of the gases by the GC as a basis, the MS provides detailed structural information about the compounds released.



1 Mass spectrometer

2 Gas chromatograph

3 Valve-based gas injector

1 Mass Spectrometer (MS)

- State-of-the-art MS
 - 1.5 u to 1022 u
 - $R > 2000$ (m/z 614)
 - High-speed sampling rate up to 22.222 u/s
- Tool-free servicing, e.g., simplified ion source maintenance without any need for tools
- Multiple ionization modes (Optionally available EI, PI, CI, etc.)
- Stand-alone MS measurements

2 Gas Chromatograph (GC)

- Fast change of column without venting the MS
- Multiple columns available for specific applications
- Split, splitless, pulsed split
- GC furnace up to 450°C

3 NETZSCH Box – Valve

- Double loop system for
- Software integrated in
- Special insulation design (300°C) to prevent col
- Independent injection available for additional (e.g., automatic liquid
- Possible to bypass the

Gas chromatography comes into play when MS has reached its limitations in determining the properties of complex gas mixtures.

GC-MS Information

- Separation of complex gas mixtures
- Gas detection and identification
- Compositional analysis
- Solid-gas reaction
- Decomposition products
- Pyrolysis gases
- Combustion products
- Flue gas identification
- Identification of additives (e.g., plasticizers)

TGA/DSC Information*

- Dehydration
- Binder burn-out
- Decomposition
- Pyrolysis
- Combustion
- Oxidation
- Evaporation
- Compositional analysis
- Melting/Crystallization
- Solid-solid transitions
- Glass transition
- Specific heat capacity

* For more information, please see STA Jupiter brochures.



5 PERSEUS STA 449 F1 Jupiter
TGA/DSC with integrated compact FT-IR system

*EXCELLENCE IS AN
UNENDING JOURNEY*

3 Valve-Based Gas Injector

for short injection intervals
in Proteus
design for constant temperature
and spots
system → standard injectors
uses
(sampler, ALS)
column for direct MS coupling

4 Transfer System

- Heated adapter system up to 300°C
- Glass-lined steel pipe (max. 300°C) allows for forced flow by pumping through the sampling loop

5 Customizable Thermal Analyzer (TGA/STA)

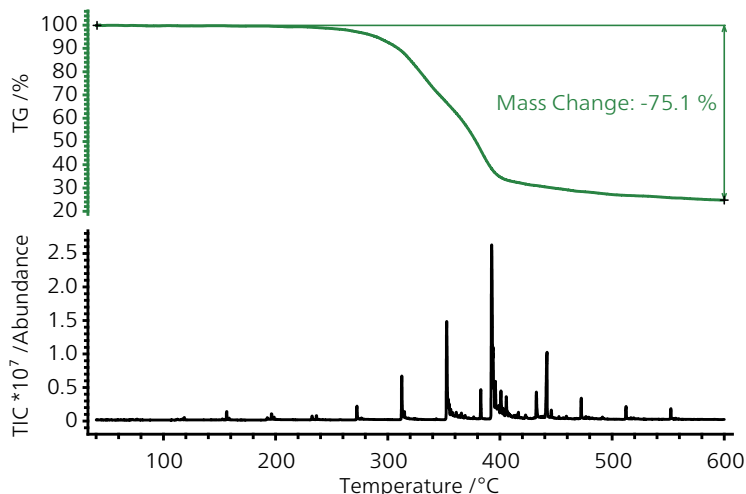
- Variable furnaces for a broad temperature range
- Various sensors
- Automatic sample changer with capacities of between 20 or 192 sample pans, depending on instrument
- Bypass system for excess gas
- Simultaneous coupling of TGA/DSC-GC-MS-FT-IR



Pyrolysis is the most effective way to convert biomass to bio-char, liquid bio oil, syngas, chemicals and other useful products. GC-MS coupled with thermal analysis is an elegant means of creating optimized temperature programs for pyrolysis and studying pyrolysis products.

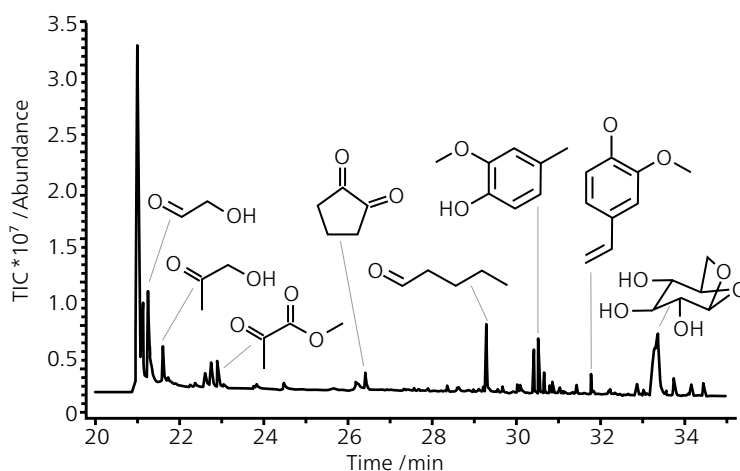
Biomass Pyrolysis

In this example, cherry wood (3.1 mg) was pyrolyzed at a heating rate of 20 K/min in a helium atmosphere. The GC-MS measurement was running in the quasi-continuous mode. Between 250°C and 450°C, the TGA signal shows a mass-loss step of 75.1%. The resulting complex gas mixture was separated by the GC column and analyzed by the MS. Continuous injections of gas samples to the GC (every 2 minutes) gave an overview of the composition of the pyrolysis gas produced.



Wood: Temperature-dependent mass changes (TGA) and total ion chromatogram (TIC) measured in the quasi-continuous mode of the STA 449 F1 Jupiter-GC-MS.

To identify the components released, a single injection of gas was applied at the point of the highest mass-loss rate (385°C) by repeating the measurement in the event-controlled mode. The resulting total ion chromatogram (TIC) with peaks over the entire range of retention times can be easily evaluated by the library search report. Exemplary database hits are shown.



Total ion chromatogram (TIC) measured in the event-controlled mode; sample gas mixture taken at 385°C.