

TG-DTA/Photoionization mass spectrometer

Thermo Mass Photo



1. Introduction

Thermo Mass Photo is a thermogravimetry-differential thermal analysis-mass spectrometry (TG-DTA-MS) instrument and a main product in the lineup of Rigaku's evolved gas analytical systems. It consists of thermogravimetry-differential thermal analysis (TG-DTA) and photoionization mass spectrometry (PIMS) equipped with a skimmer-type interface. Weight change, endothermic or exothermic phenomena, and evolved gases can be analyzed simultaneously. Therefore, Thermo Mass Photo is considered a promising analytical tool for fundamental research, qualification control, and development of new materials.

Thermo Mass Photo has been optimized as the "all-in-one" package consisting of TG-DTA and MS providing a small footprint, safety and easy-maintenance. The software supports an easy operation with a guidance function. A complicated multi-steps reaction accompanied with various evolved gases can be

investigated by the combination of controlled-rate TG (CRTG) and PIMS. In addition, the compact automatic sample changer called Smart Loader can contribute to the throughput enhancement.

2. Features

2.1. Skimmer-type interface

In TG-MS, the interface plays an important role in the effective transfer of the evolved gases. Furthermore, it adjusts the pressure difference between the TG (in ambient pressure) and the MS (in vacuum). The conventional TG-MS has adopted a capillary-type interface. However, a capillary-type interface sometimes gives rise to the re-condensation and transformation of evolved gases.

Thermo Mass Photo has adopted the skimmer-type interface to overcome this problem. The skimmer-type interface consists of two concentric ceramic tubes with

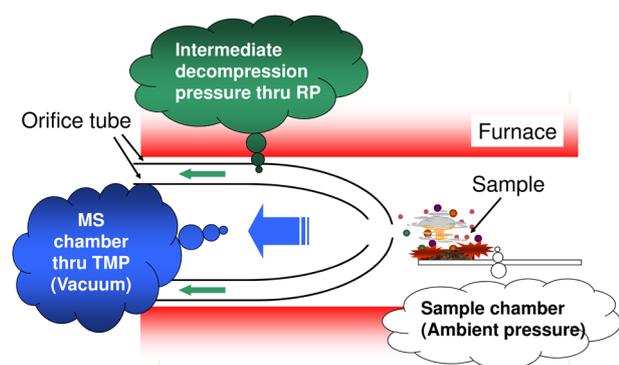


Fig. 1. Mechanism of skimmer-type interface.

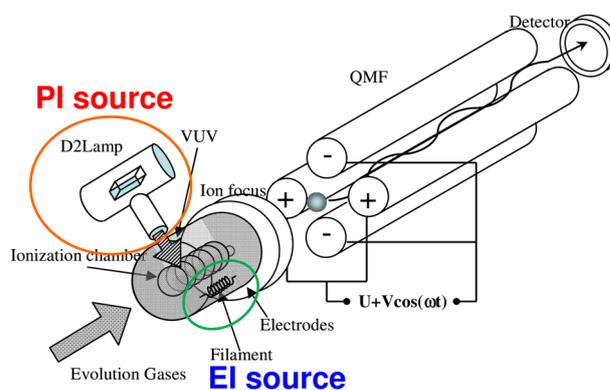


Fig. 2. Schematic view of quadrupole mass spectrometer with EI and PI sources.

tiny orifices that functions as a divergent nozzle coupling with a differential pressure reduction as shown in Fig. 1. The skimmer-type interface provides evolved gas analysis in real-time because the interface-length connecting between the TG furnace and MS chamber is minimized. In addition, this interface can be thermally programmed under the same environment as the samples because it is located inside the furnace. These features inhibit re-condensation and transformation of evolved gases, and realize the investigation of reactive and low-volatile evolved gases.

2.2. Photoionization

MS analyzers in conventional TG-MS have utilized electron ionization (EI). A gaseous molecule is ionized by colliding with an accelerated electron with 70 eV, which generates molecular ions and several fragment ions. The pattern of the fragment ions reflects the molecular structure. EI can be applied to the ionization of most gas species with high sensitivity. However, the EIMS cannot discriminate each gaseous molecule due to the overlapping of fragment ions when multi-component gases are evolved simultaneously.

Thermo Mass Photo allows the selection of photoionization (PI) as well as EI. In PI, gaseous molecules are ionized by irradiation of vacuum ultraviolet (VUV) light with 10.2 eV. PI is one of the soft ionization methods which provide only molecular ions without fragment ions. PI is appropriate well for the detection of the various gaseous molecules evolved simultaneously in real-time, such as thermal behaviors for mixtures of organic compounds and thermal decomposition of polymers.

As shown in Fig. 2, Thermo Mass Photo is equipped with a VUV light source for PI as well as a filament for EI. Users can select EI or PI on the software without exchanging the hardware.

2.3. Smart Loader

Thermo Mass Photo can be equipped with Smart Loader as shown in Fig. 3. Smart Loader can load up to 24 measurement samples. Furthermore, users can select 3 reference samples and change them in

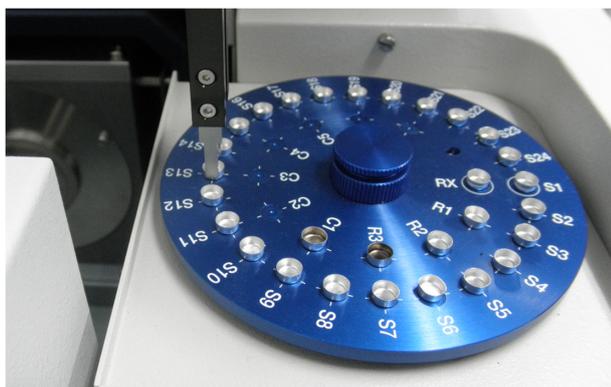


Fig. 3. Smart Loader tray which can load up to 24 measurement samples.

each measurement. The simple and user-friendly programming window allows users to set temperature programs, MS conditions, exchange-temperatures, and MS stabilization times in each measurement. Continuous measurements can be interrupted to perform other measurements.

2.4. 3D Display and Analysis Software

3D Display and Analysis Software supports the analysis and evaluation for combined information in TG-DTA and MS. This software visualizes the easy-to-see 3-dimensional diagram from the obtained matrix data shown in Fig. 4(a). In addition, users can freely select a mass spectrum at an arbitrary temperature or an MS ion thermogram of an arbitrary mass number (m/z), which shows the profile of the evolved gas as a function of the temperature. As shown in Fig. 4(b), the mass spectrum is directly linked to NIST-MS library search software and the qualitative analysis can be automatically performed.

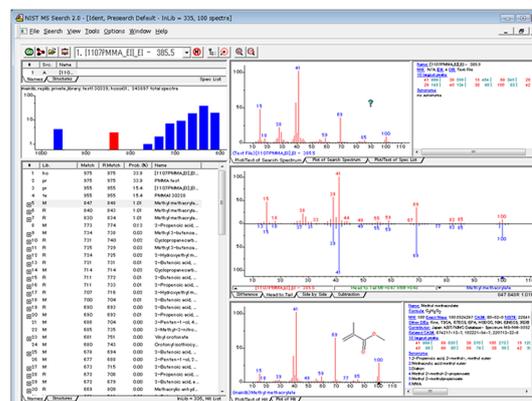
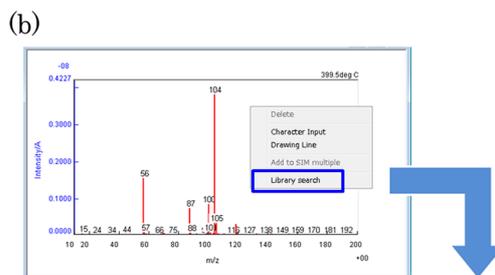
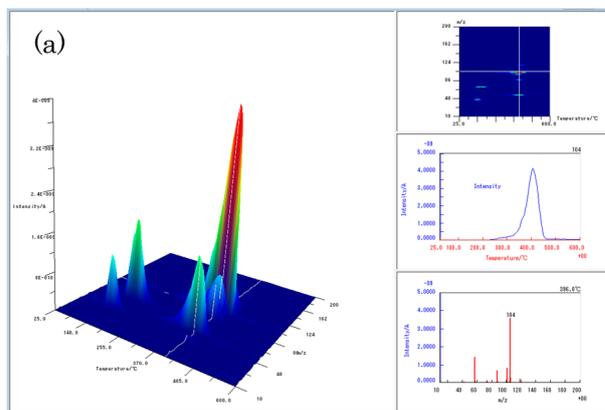


Fig. 4. (a) 3D main window. (b) Direct linkage to NIST-MS library search software.

Table 1. Specification.

		Standard model	Infrared furnace model
Thermobalance		Horizontal differential TG-DTA	
Measurement Temp. Range		Ambient –1000°C	Ambient –950°C
Max. Heating Rate		20°C/min	100°C/min
Interface		Skimmer-type interface	
Mass spectrometer	MS analyzing system	Quadrupole mass spectrometer	
	Available mass numbers (m/z)	1–410	
	Ionization	EI or PI	
	Measurement mode	TIC Scan, SIM (Max. 16 channels), partial pressure modes (10 types)	
Atmosphere (flow)		He or 20% O ₂ /He	
Smart Loader		Measurement sample 24 pieces, reference sample 3 pieces	

3. Summary

Thermo Mass Photo can provide the real-time profiling of evolved gases. Therefore various applications are available, such as analysis of hydrogen absorbing alloys, catalytic reaction, thermal decomposition of organic molecules and polymers, and stability test of pharmaceutical.