

# On-line measurement of heat-generated gas by TPD-TOFMS

## Application to functional groups, reaction analysis, etc.

We have developed a TPD-TOFMS system, which consists of a high-resolution time-of-flight mass spectrometer (TOFMS) connected to a temperature-controllable heating furnace. It is possible to separate and quantify components (e.g., CO and N<sub>2</sub>) that were difficult to separate with the mass resolution of conventional equipment, and to accurately estimate compositional equations from precise mass numbers. This system is also useful for obtaining information on functional groups in materials and for chemical reaction analysis.

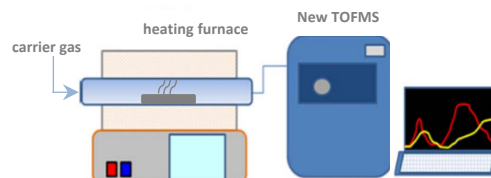
### TPD-TOFMS\*

\* Temperature Programmed Desorption-Time of Flight Mass Spectrometry

The gas generated during heating is detected in real time by a mass spectrometer. In addition to qualitative and quantitative measurements of the gas, the temperature dependence of the generation rate can also be determined.

- ◎ Large samples can be handled (~10x10x20mm)
- ◎ Simultaneous detection of inorganic and organic gases

➔ By using a high-resolution TOFMS as the detector (MS)  
More detailed qualitative and quantitative analysis of the generated gas is possible.

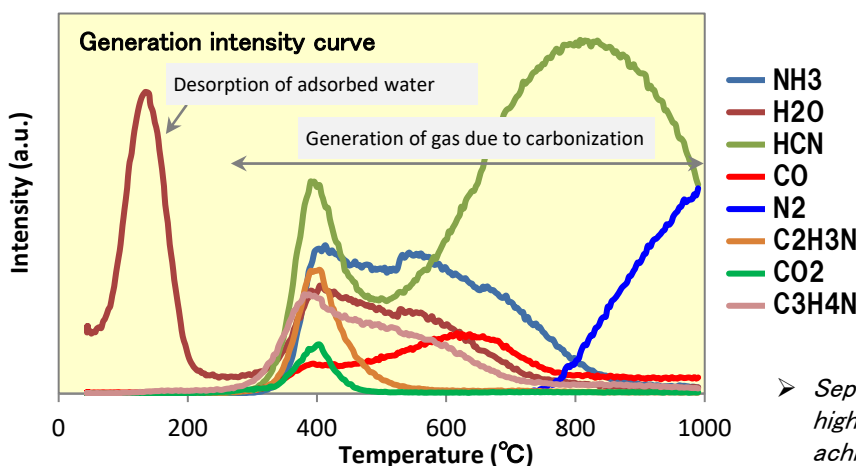


### Measurement of polyacrylonitrile (PAN) after heat treatment

Measured PAN heat-treated under air

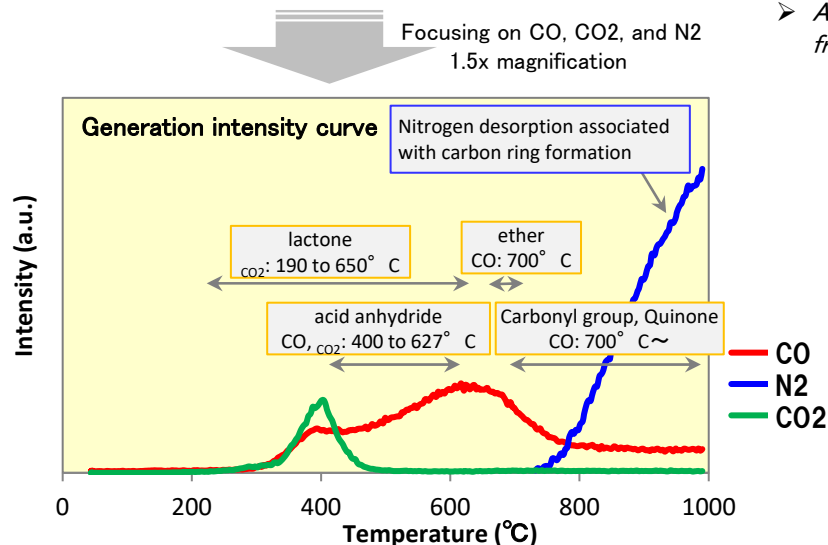
Heating conditions: Room temperature  $\xrightarrow{20^\circ\text{C}/\text{min}}$  1000°C, Helium atmosphere

Estimated amount of generation (wt%)

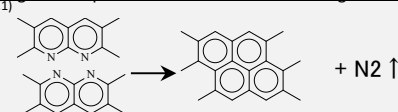


$m/z$ 17.027 NH <sub>3</sub>	0.49
$m/z$ 18.010 H <sub>2</sub> O	0.55
$m/z$ 27.011 HCN	2.6
$m/z$ 27.995 CO	0.42
$m/z$ 28.006 N <sub>2</sub>	0.83
$m/z$ 41.026 C <sub>2</sub> H <sub>3</sub> N	0.14
$m/z$ 43.989 CO <sub>2</sub>	0.55
$m/z$ 54.034 C <sub>3</sub> H <sub>4</sub> N	0.054

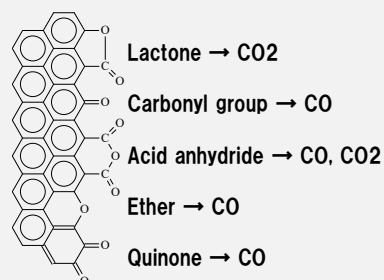
- Separation and quantification of CO and N<sub>2</sub> with high mass resolution, which was difficult to achieve with conventional equipment
- Accurate estimation of each composition formula from precise mass number



Nitrogen desorption associated with carbon ring formation (Ref.1)



Thermal decomposition of functional groups (Ref.2)



➤ The presence of the above functional groups is suggested.

➤ Estimates of functional groups can be analyzed from the amount of gas generated.

(Ref.1) K. Nukada etc., Kobunshi, 23, No.267, 445-463 (1974). (Ref.2) H. Takagi, TANSO 2009, No.237, 67-71.