Real time monitoring for gas phase H/D exchange reaction in H_2O and D_2O by Using a Compact High-Resolution Multi-Turn Mass Spectrometer

AN-MS-002

Introduction

The InfiTOF™ is a compact time-of-flight mass spectrometer which has unique multi-turn ion optics.

The InfiTOF has features blow.

1. Able to detect small elements such as Hydrogen atom (H).
2. Allow resolving power selection based on the chosen path length for the ions.
3. High resolving power achieve to separate close proximity ions such as carbon monoxide (CO) and nitrogen (N_2).
4. Able to measure long term with high m/z stability (Fluctuation < 100ppm/hour), high sensitivity and high mass accuracy.
5. Easily transportable.

The InfiTOF is suitable for real time gas reaction monitoring. In this report, we showed real time monitoring for H/D exchange reaction of light water (H_2O) and heavy water (D_2O) which is one of the simplest chemical reaction.

Experimental

Mass spectra were acquired by using the InfiTOF compact multi-turn high-resolution mass spectrometer. High-resolution mass spectra were acquired in multi-segment mode (150 turns) to give a resolving power of approximately 30,000 (FWHM) at m/z 18. Additionally, the ionizing electron voltage was set to 15 eV. Schematic drawing of experimental equipment shown in Fig.1. Between the gas chromatograph and the mass spectrometer, Vial of volume 22 mL was set up, and the two intervals as GC – vial and vial – MS were connected through a deactivated fused-silica capillary tubes 3m x 0.25mm and 30cm x 0.1mm respectively. GC Helium gas flow rate was set up 1mL/min.

H_2O and D_2O were injected 1µL each in GC inlet at a one minute time lag. Introduced gases were through in the vial, and the gases were mixed in the vial were introduced into the mass spectrometer.

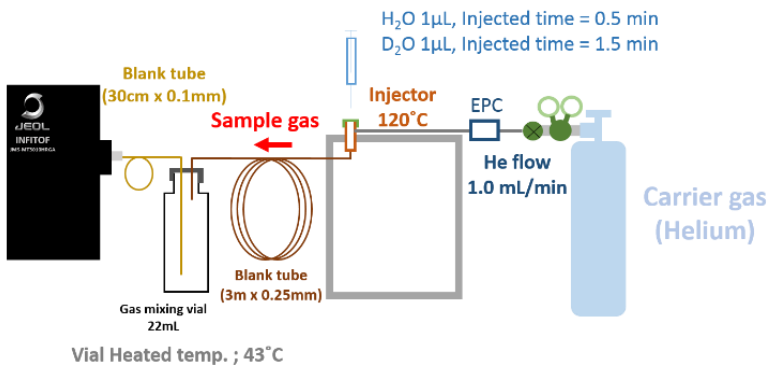

Figure 1. Schematic drawing of experimental equipment.

Table 1 Measurement conditions

Sample	H_2O (W5-4, Fisher scientific, purified by EASYpure RF, Barnstead™) D_2O(DLM-6-1000, Cambridge Isotope Laboratories, Inc.)
Mixing vial volume	22mL
Vial temperature	43˚C
Injection volume	1μL each
Injected timing	H_2O ; Injected 30 second after started to measure D_2O ; Injected 90 second after started to measure
MS	JMS-MT3010HRGA InfiTOF ™
Resolving power	Approx. 30000 (m/z 18)
Ionization Voltage	15 eV
Recording interval	1 sec/spectrum
Detector Voltage	2700V
Measurement mode	Extended mode
m/z range	m/z 17 – 22
GC	Agilent 7890B
Column (GC to Vial side)	Diactivated fused silica capillary tube (3m x 0.25mm)
Column (Vial to MS side)	Diactivated fused silica capillary tube (30cm x 0.1mm)
He flow rate	1 mL/min

Results

The extracted ion chromatogram in Fig.2 shows that each ions behavior is different. These ions were detected for 1 hour after injecting light water. H/D exchange reaction was occurred instantaneously when injecting heavy water seems to be detected $HDO^{+\cdot}$ and $D_2O^{+\cdot}$.

The mass spectra after light water injection and after light water and heavy water injection respectively are shown in Fig. 3. The mass spectrum that injected only light water shows $H_2O^{+\cdot}$ and H_3O^+ were detected mainly. The mass spectrum after both water injections showed the ions which mass deference is only 0.0015 u such as $HDO^{+\cdot}$ /H_3O^+ and $D_2O^{+\cdot}$ /H_2DO^+ were detected respectively.

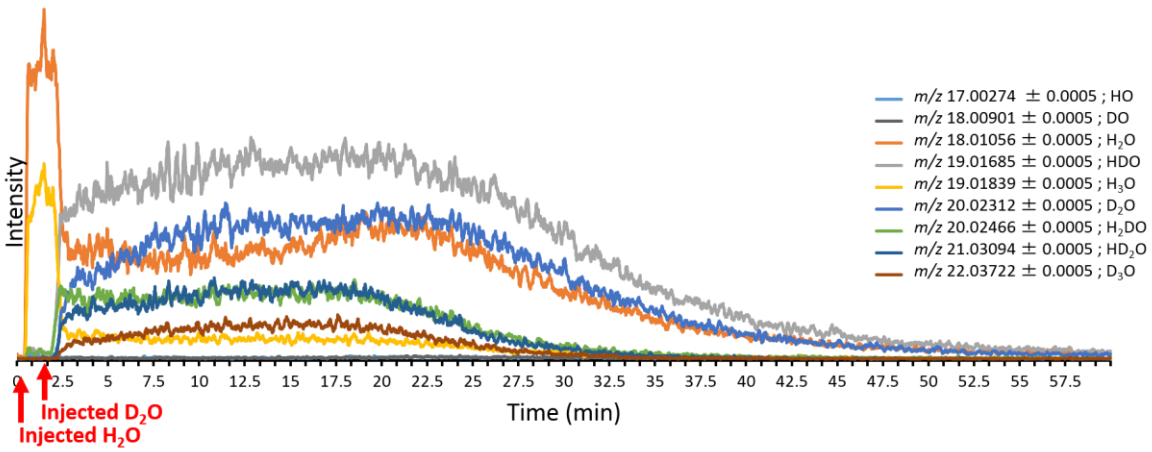

Figure 2. Extracted ion chromatogram.

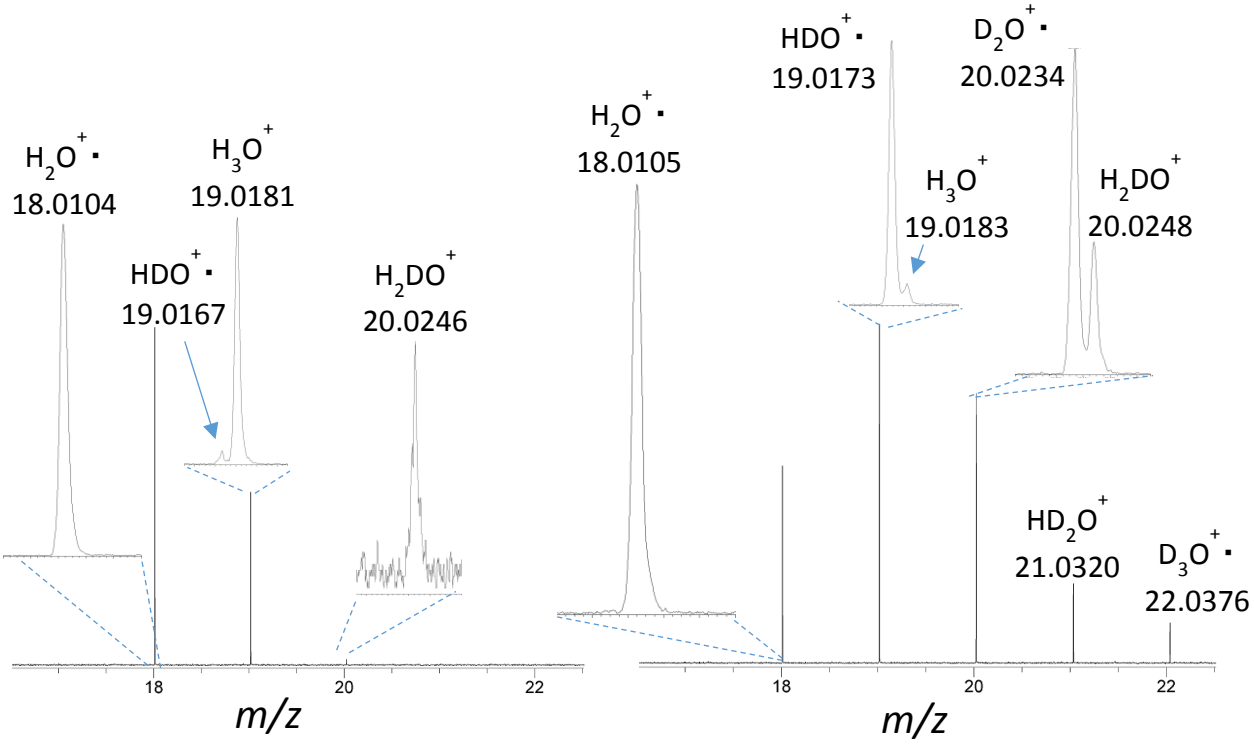

Figure 3. Mass spectra (Left; After injected only H_2O, Right; After injected H_2O and D_2O).

Conclusion

InfiTOF can separate and identify to close proximity ions such as HDO/H_3O and D_2O/H_2DO by high mass resolving power. Therefore the InfiTOF is suitable system for absolute H_2O/D_2O ratio analysis.