



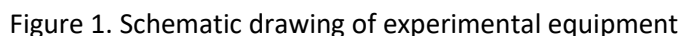
Real-Time Monitoring for Gas Phase H/D Exchange Reaction of H₂O and D₂O by Using a Compact High-Resolution Multi-Turn Mass Spectrometer

The InfiTOF™ is a compact high resolution time-of-flight mass spectrometer (TOF-MS) that has a unique multi-turn ion optics system capable of providing a variable-length ion-flight path (up to 200m) in a very compact analyzer (20cm x 20cm). The InfiTOF is able to:

- The InfiTOF enables “In-situ” high-resolution monitoring of real-time gas phase reactions. In this work, we show the real-time monitoring of a simple H/D exchange reaction of light water (H_2O) and heavy water (D_2O).

Mass spectra were acquired by using the InfiTOF with a multi-segment mode setting of 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. Figure 1 shows a schematic drawing of the experimental equipment. A 22mL mixing vial was set up between the gas chromatograph and the mass spectrometer. A deactivated fused-silica capillary tube (3m x 0.25mm) was used for the GC – vial connection, and a deactivated fused-silica capillary tube (30cm x 0.1mm) was used for the vial – MS connection. The GC helium gas flow rate was set to 1mL/min.

H₂O and D₂O were injected 1 μL each into the GC inlet at a one minute interval apart from each other. The gases mixed in the vial thus promoting the H/D exchange reaction. The resulting mixture then flowed from the mixing vial into the mass spectrometer.



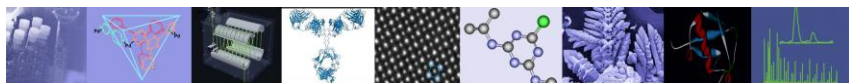


Table 1. Measurement conditions

Sample	H ₂ O (W5-4, Fisher scientific, purified by EASYpure RF, Barnstead™)
	D ₂ O(DLM-6-1000, Cambridge Isotope Laboratories, Inc.)
Mixing vial volume	22mL
Vial temperature	43°C
Injection volume	1μL each
Injected timing	H ₂ O ; Injected 30 second after start of measurement
	D ₂ O ; Injected 90 second after start of measurement
MS	JMS-MT3010HRGA InfitOF TM
Resolving power	Approx. 30,000 (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)	Deactivated fused silica capillary tube (3m x 0.25mm)
Column (Vial to MS)	Deactivated fused silica capillary tube (30cm x 0.1mm)
He flow rate	1 mL/min

Results

Figure 2 shows the extracted ion chromatograms for each component of interest in the reaction, including fragment ions. All ions were monitored for 1 hour after the light water (H₂O) was injected into the system. The H/D exchange reaction started occurring almost immediately after the injection of the heavy water, as shown by the simultaneous appearance of HDO⁺ and D₂O⁺ shortly after the D₂O injection.

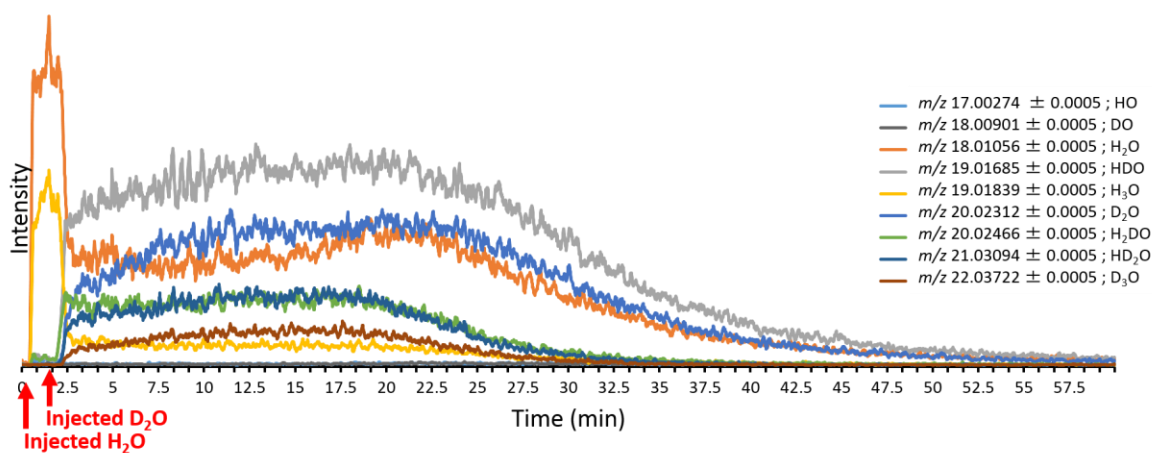


Figure 2. Extracted ion chromatograms.

Figure 3 shows the mass spectrum observed after the light water injection (left), and the mass spectrum observed after the heavy water injection (right). The mass spectrum for the light water injection was dominated by H₂O⁺ and H₃O⁺ ions while the mass spectrum after both water injections showed stronger signals for HDO⁺ /H₃O⁺ and D₂O⁺ /H₂DO⁺ ions, which have mass differences of only 0.0015 u.

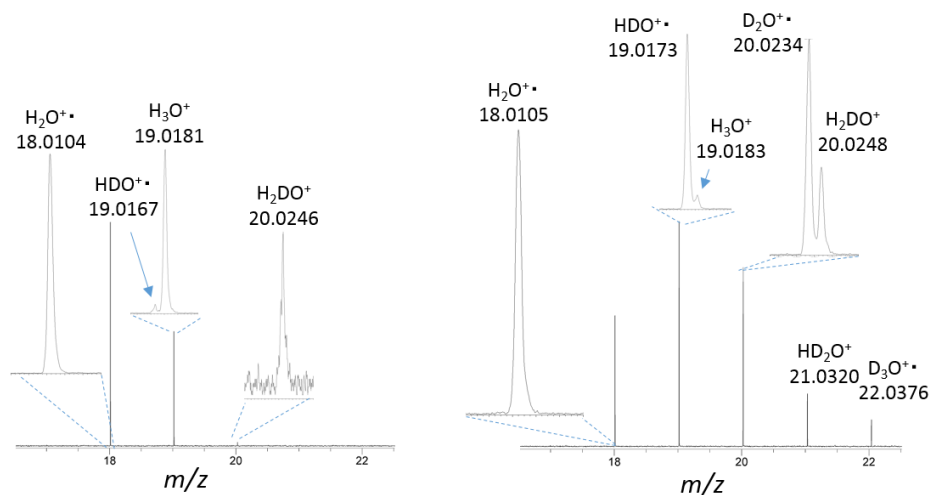
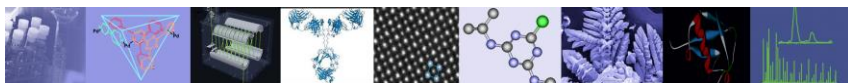


Figure 3. H/D exchange mass spectra – Left: After H₂O injection; Right: After D₂O injection.

Conclusion

The InfiTOF can achieve mass separation and accurate mass identification of similar mass ions such as HDO/H₃O and D₂O/H₂DO by high mass resolving power. Therefore, the InfiTOF is a suitable system for absolute H₂O/D₂O ratio analysis.

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