

**Supporting Information**

**Electrospray Ionization-Ion Mobility Spectrometry-High Resolution Tandem Mass Spectrometry with Collision Induced Charge Stripping for Analysis of Highly Multiply Charged Intact Polymers**

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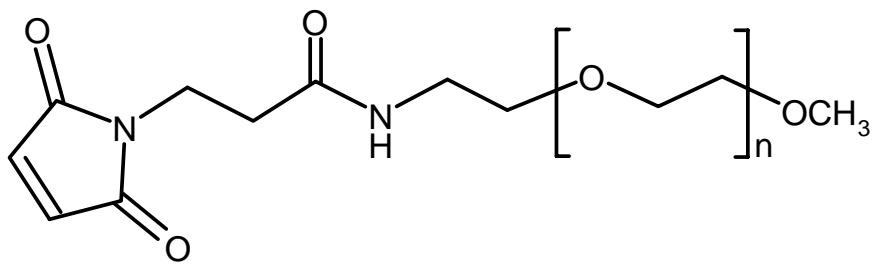
Figure S3: Effect of CICS voltage on analysis of PEO in ESI-CICS-IMS-TOFMS.

Figure S4: Analytical results of mixture of PEOs by ESI-QMS-CICS-IMS-TOFMS.

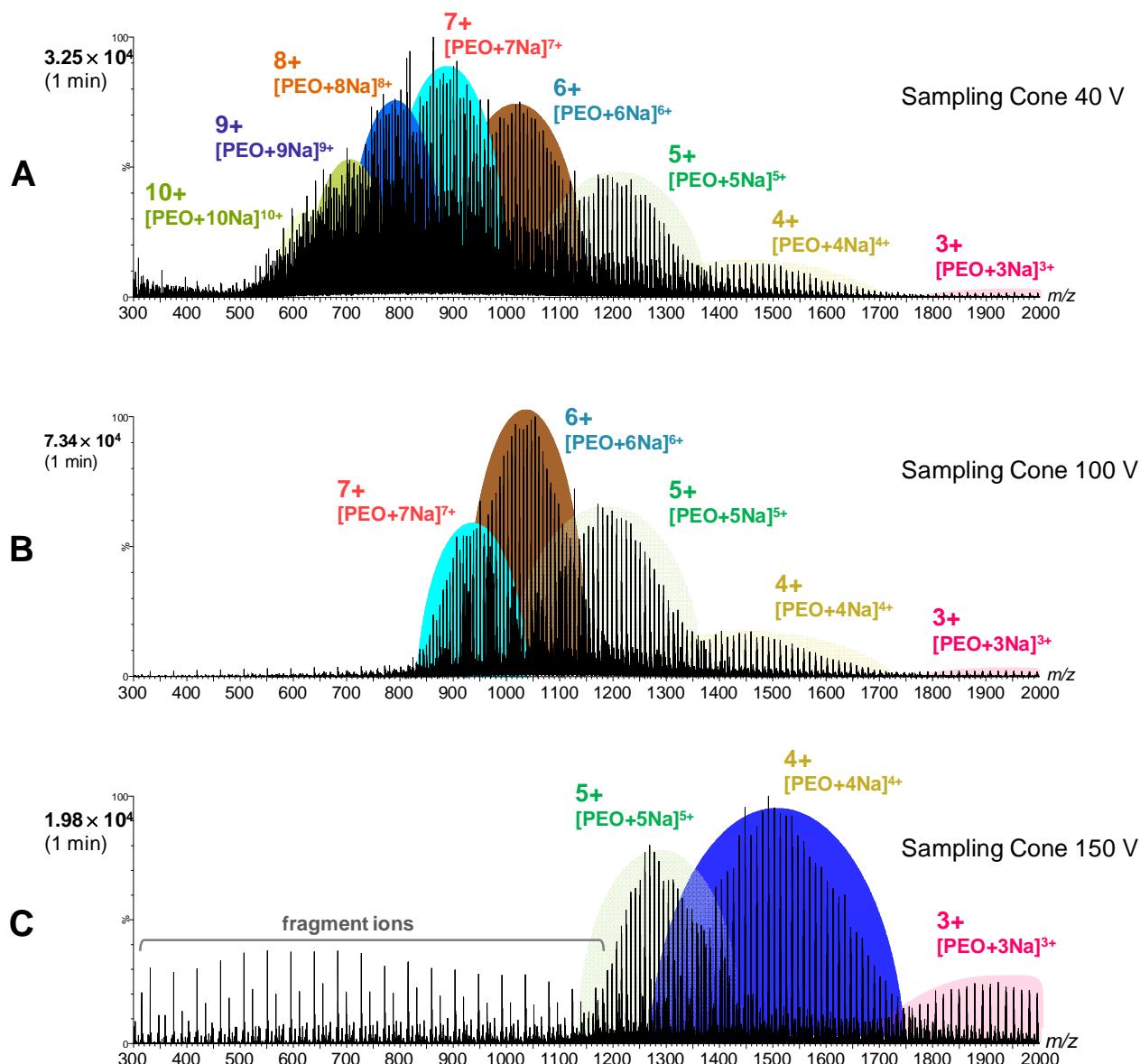
Figure S5: Power spectra of mass spectra.

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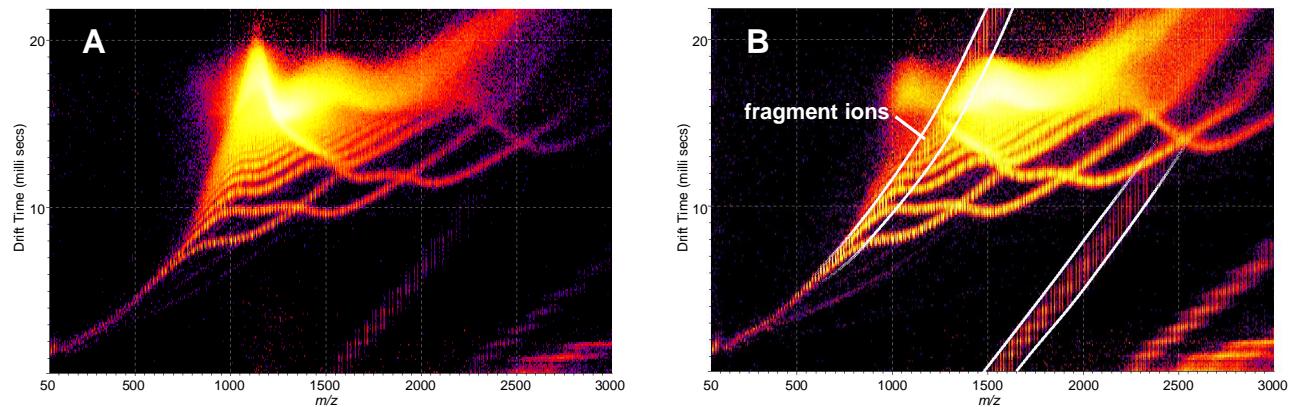
Figure S7: Analytical results of mPEO-Mal by ESI- IMS-TOFMS.



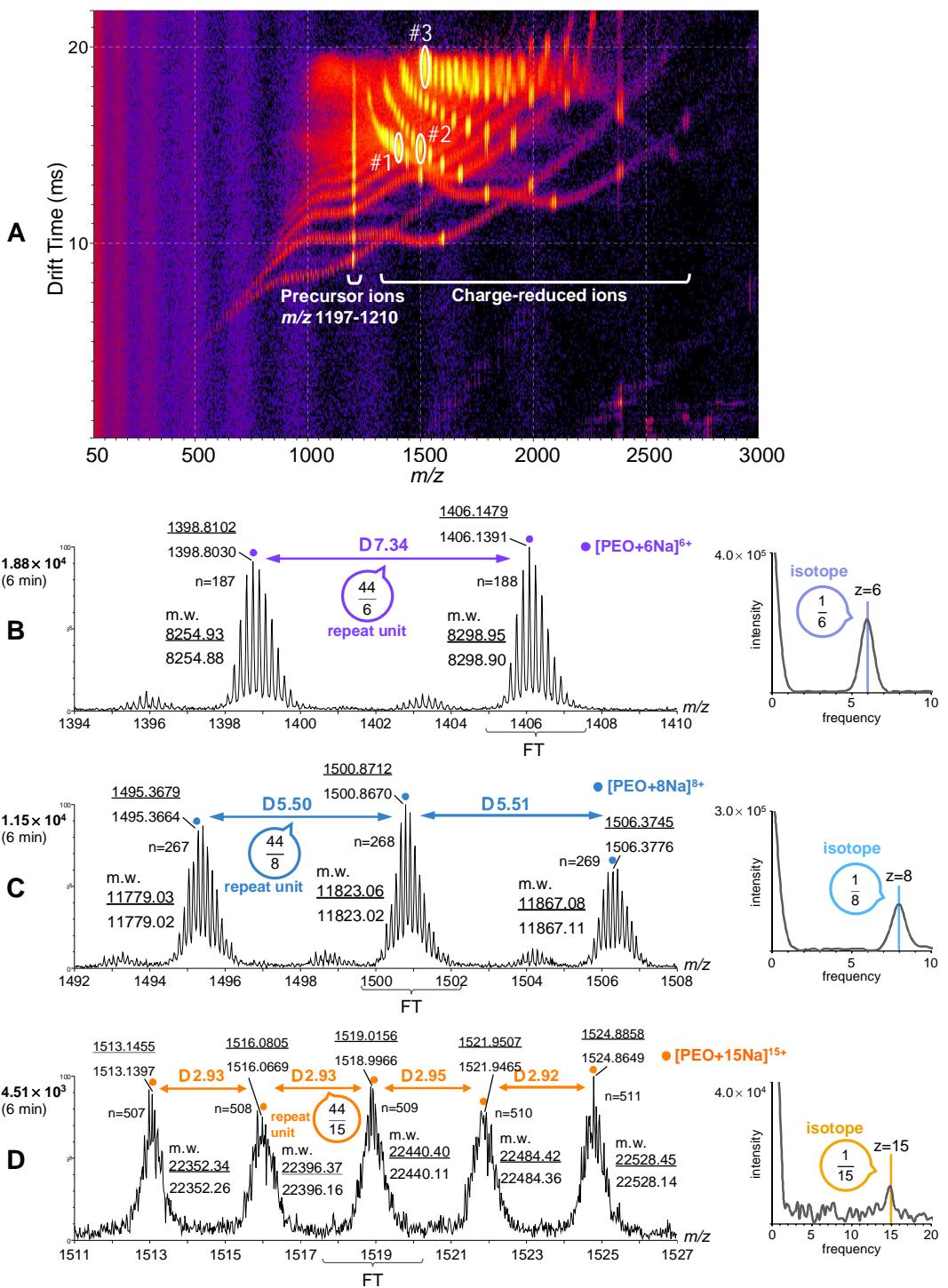
**Figure S1.** Structure of mPEO-Mal.



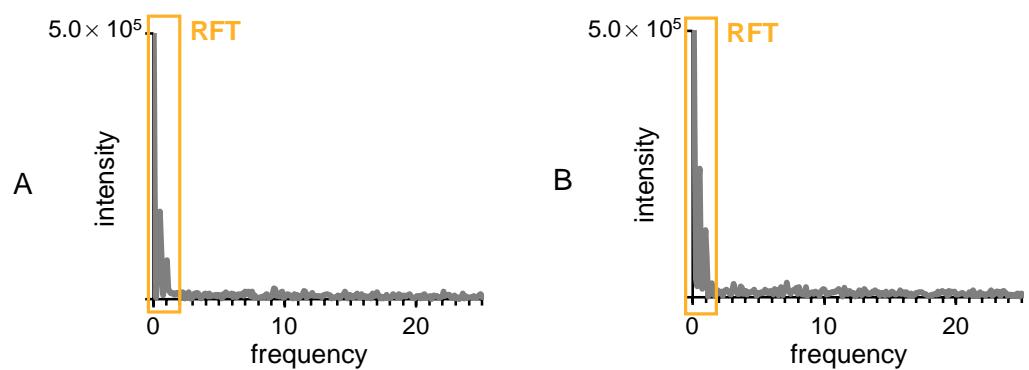
**Figure S2.** Effect of cone voltage on analysis of PEO (average molecular weight: 6 k) in ESI-TOFMS. Cone voltage: (A) 40 V, (B) 100 V, and (C) 150 V. The number of  $\text{Na}^+$  additions are identified by mass differences in of both isotope peaks and repeated units.



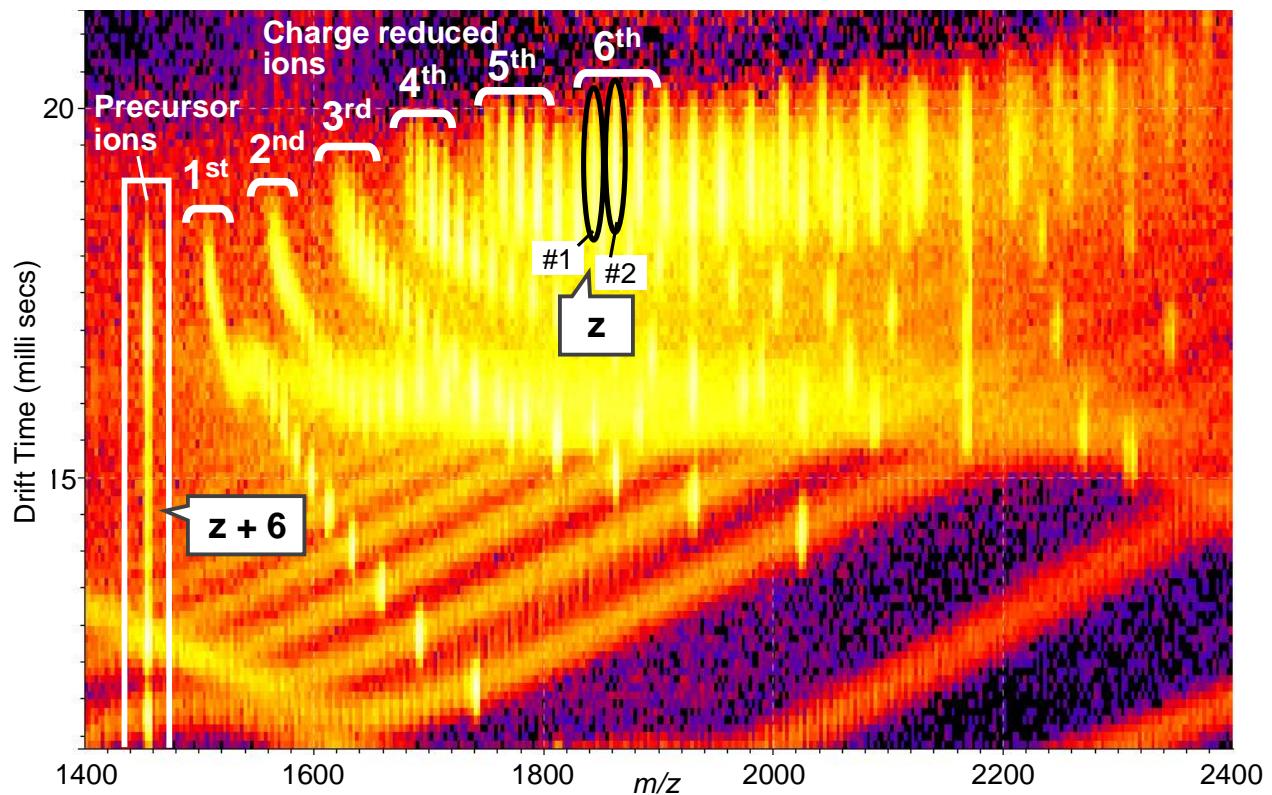
**Figure S3.** Effect of CICS voltage on analysis of PEO (average molecular weight: 20 k) in ESI-CICS-IMS-TOFMS. Cone voltage: (A) 120 V, and (B) 160 V. Bias voltage: (A) 120 V, and (B) 120 V. In (B), the fragmentation (CID) of PEOs are observed.



**Figure S4.** Analytical results of mixture of PEOs (average molecular weight: 6 k, 10k, and 20 k) by ESI-QMS-CICS-IMS-TOFMS. (A) 2D heat map of the  $m/z$  value and drift time; (B), (C), and (D) the extracted mass spectra of zones #1, #2, and #3, respectively (left), and the corresponding power spectra obtained by FT (right). The region for FT is indicated in the mass spectrum. Other conditions are the same as shown in Figure 1.



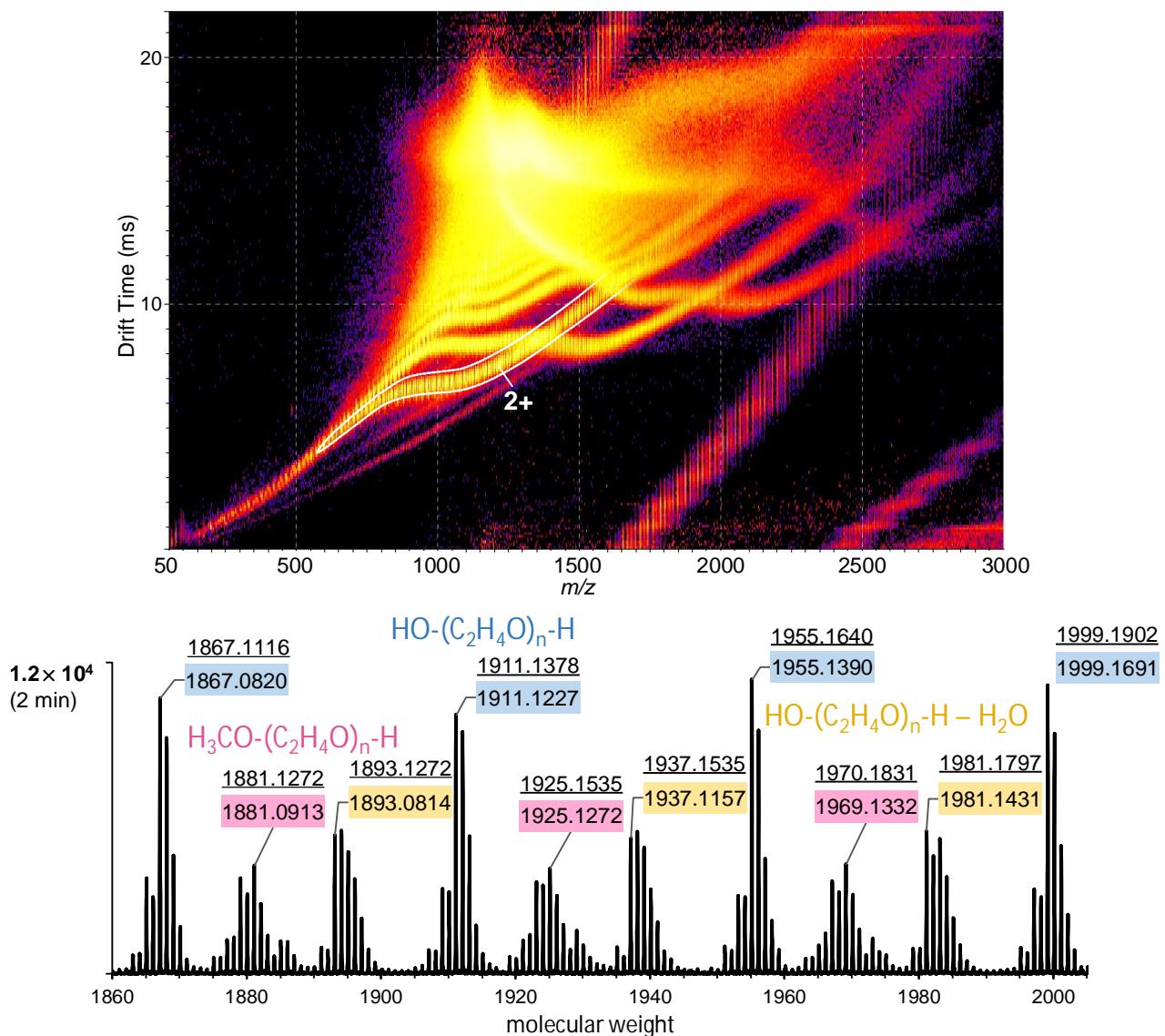
**Figure S5.** Power spectra of mass spectra (left) in (A) Figure 6B and (B) Figure 6C.



$$\left\{ \begin{array}{l} \frac{m.w_1 + 23(z_1 + 6)}{z_1 + 6} = 1450 \\ \frac{m.w_1 + 23z_1}{z_1} = 1838 \end{array} \right. \quad \underline{\quad z_1 = 22.07 \quad}$$

$$\left\{ \begin{array}{l} \frac{m.w_2 + 23(z_2 + 6)}{z_2 + 6} = 1450 \\ \frac{m.w_2 + 23z_2}{z_2} = 1857 \end{array} \right. \quad \underline{\quad z_2 = 21.04 \quad}$$

**Figure S6.** Estimation of charge number of mPEO-Mal (average molecular weight: 40 k) by solving simultaneous equations. The reduced charge number by CICS is estimated by the 2D heat map. The mass number (1450 for precursor, 1838/1857 for charge reduced ions) were measured in the extracted mass spectra.



**Figure S7.** Analytical results of mPEO-Mal (average molecular weight: 40 k) by ESI-IMS-TOFMS. (A) 2D heat map of the  $m/z$  value and drift time; (B) the extracted mass spectrum for the zone (2+).