Electronic Supplementary Material (ESI) for Journal of Analytical Atomic Spectrometry

Methodological artefacts in the XANES analysis of hexacoordinated pentavalent arsenic

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The electronic supplementary information includes a description of the mass spectrometric experiment with the As(V)-glycerol compound, along with the results, and the XANES spectra obtained during the pH stability experiment of the As(V)-glycerol experiment.

Mass Spectrometric Analysis

The As(V)-glycerol compound was dissolved in acetonitrile (12 mg in 2.4 mL to obtain ~5mg/mL) and directly infused to a Waters Micromass ZQ mass spectrometer, capable of electrospray (ESI) or atmospheric pressure chemical ionization (APCI). The compound was analyzed in ESI negative mode using the following parameters: capillary voltage 3.00 kV, cone voltage -55.56 V, extractor voltage -4.03 V, source temperature 100°C, desolvation temperature 200°C, desolvation flow rate 250 L/h, cone flow rate 66 L/h. A mass spectrum in ESI positive mode was also obtained with the following parameters: capillary voltage 3.00 kV, cone voltage 36.00 V, extractor voltage 3.00 V, source temperature 90°C, desolvation temperature 250°C, desolvation flow rate 250 L/h, cone flow rate 59 L/h.

Negative ions ($[M]^-$) were observed in ESI negative mode (Figures S1a and S1b) that corresponded to the molecular mass of As(V)-glycerol (75 for As + 3 × 90.33 for glycerol - 1 = m/z 345). Figure S2 shows the observed molecular ion $[M]^-$ was in good agreement with the theoretical mass distribution generated by MassLynx software.

The results from positive ESI are shown in Figure S3, with peaks at m/z 115.5, m/z 207.5 and m/z 279.6 corresponding to [glycerol+Na]+, [2glycerol+Na]+ and [3glycerol-3HO2+K]+ respectively. In the spectrum, a series of unknown polymer pattern peaks with a repeat unit of 85 were also observed.

Also included are:

Figure S1

Figure S2

Figure S3

Figure S4

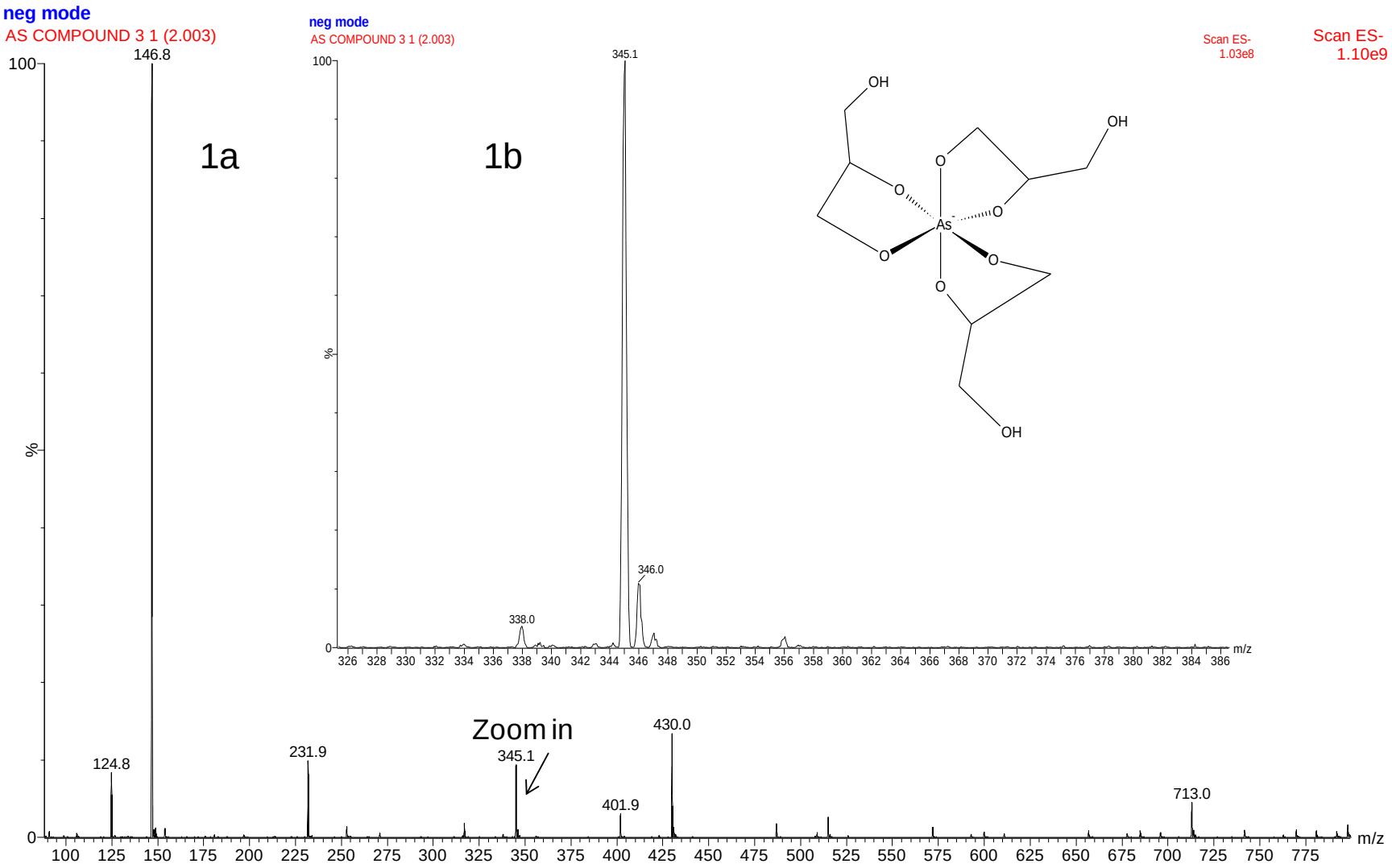

Figure S1a and 1b (inset). Negative ESI of As(V)-glycerol compound, with $[M]^-$ peak at m/z 345.

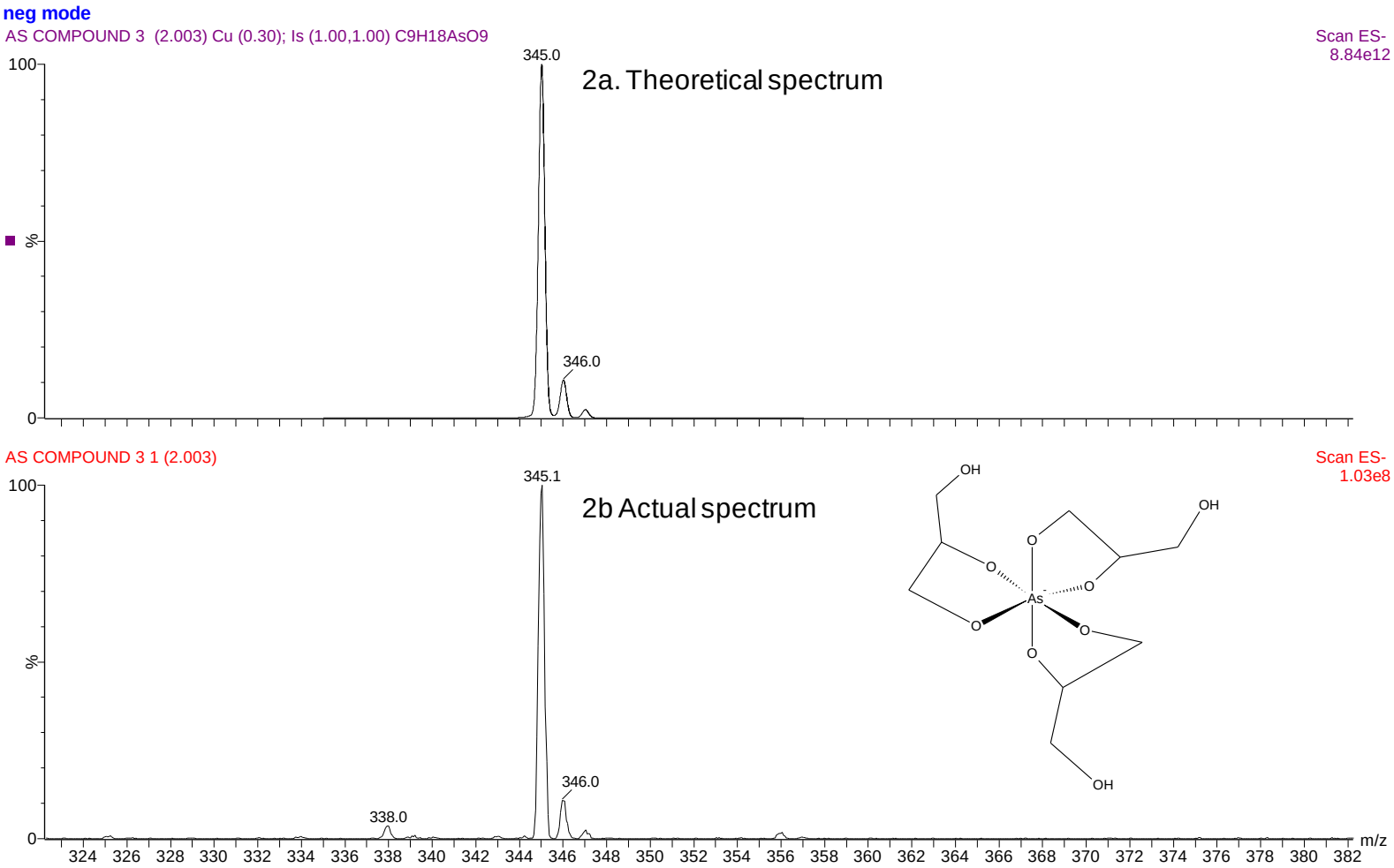

Figure S2a and 2b. Negative ESI mass spectrum of As(V) glycerol compound compared with theoretical ESI mass spectrum generated by MassLynx software.

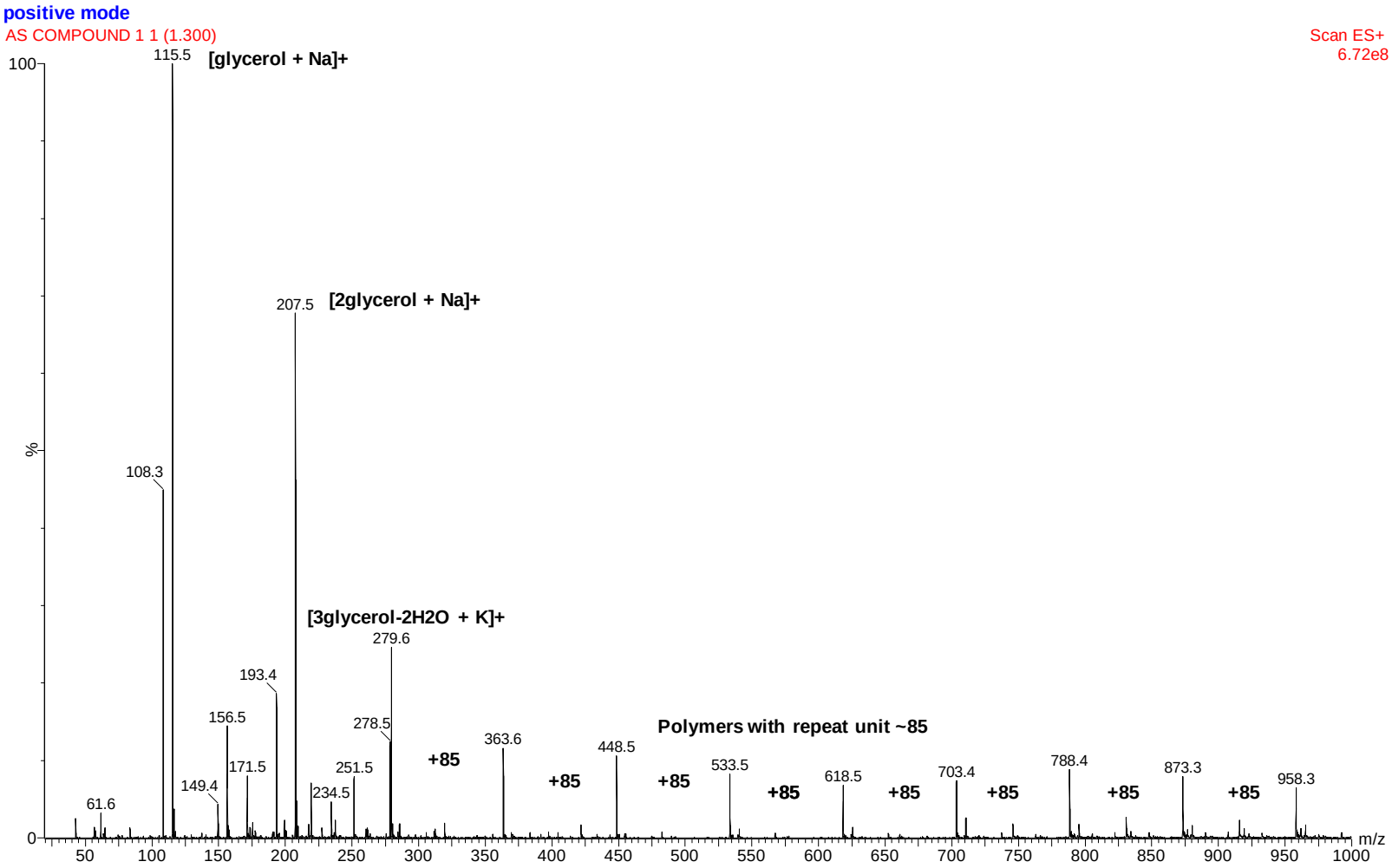

Figure S3. Positive ESI mass spectrum of As(V) glycerol compound.

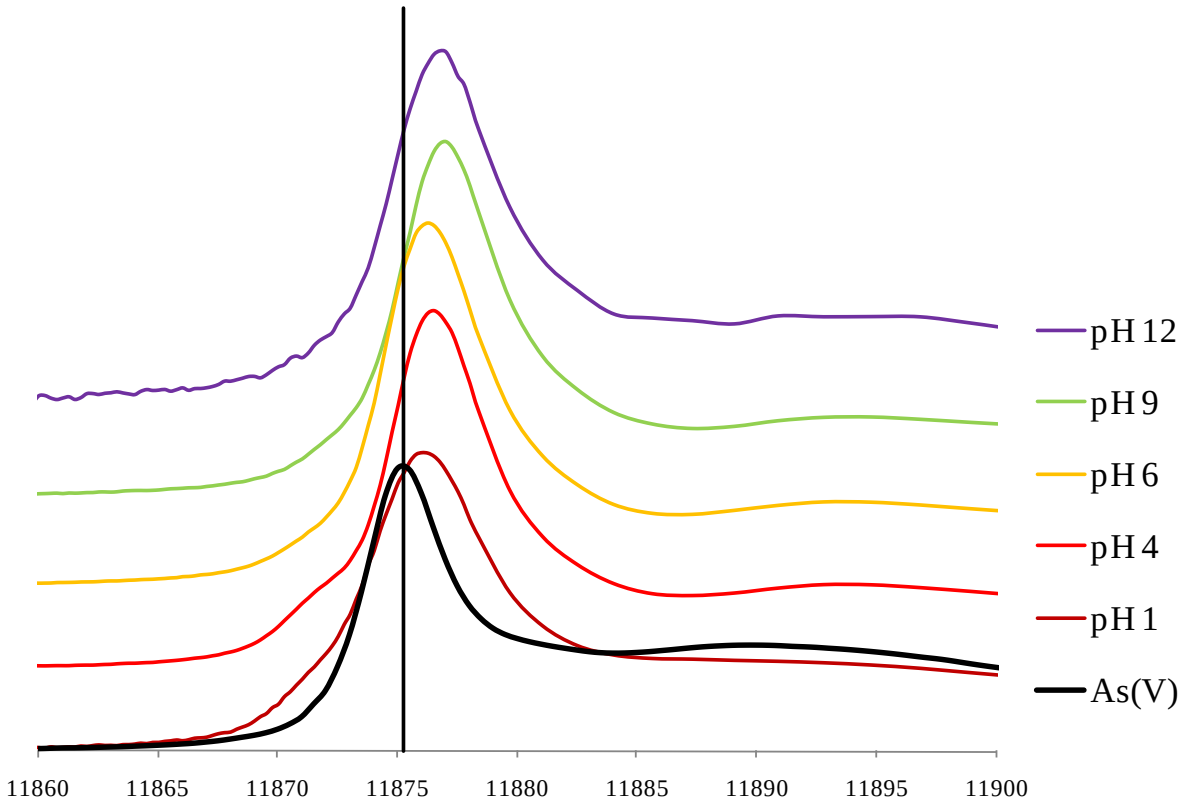

Figure S4. XANES spectra of As(V)-glycerol compound analyzed at different pHs.