Improved Isolation Procedures for Okadaic Acid Group Toxins from Shellfish (*Mytilus edulis*) and Microalgae (*Prorocentrum lima*)

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Contents

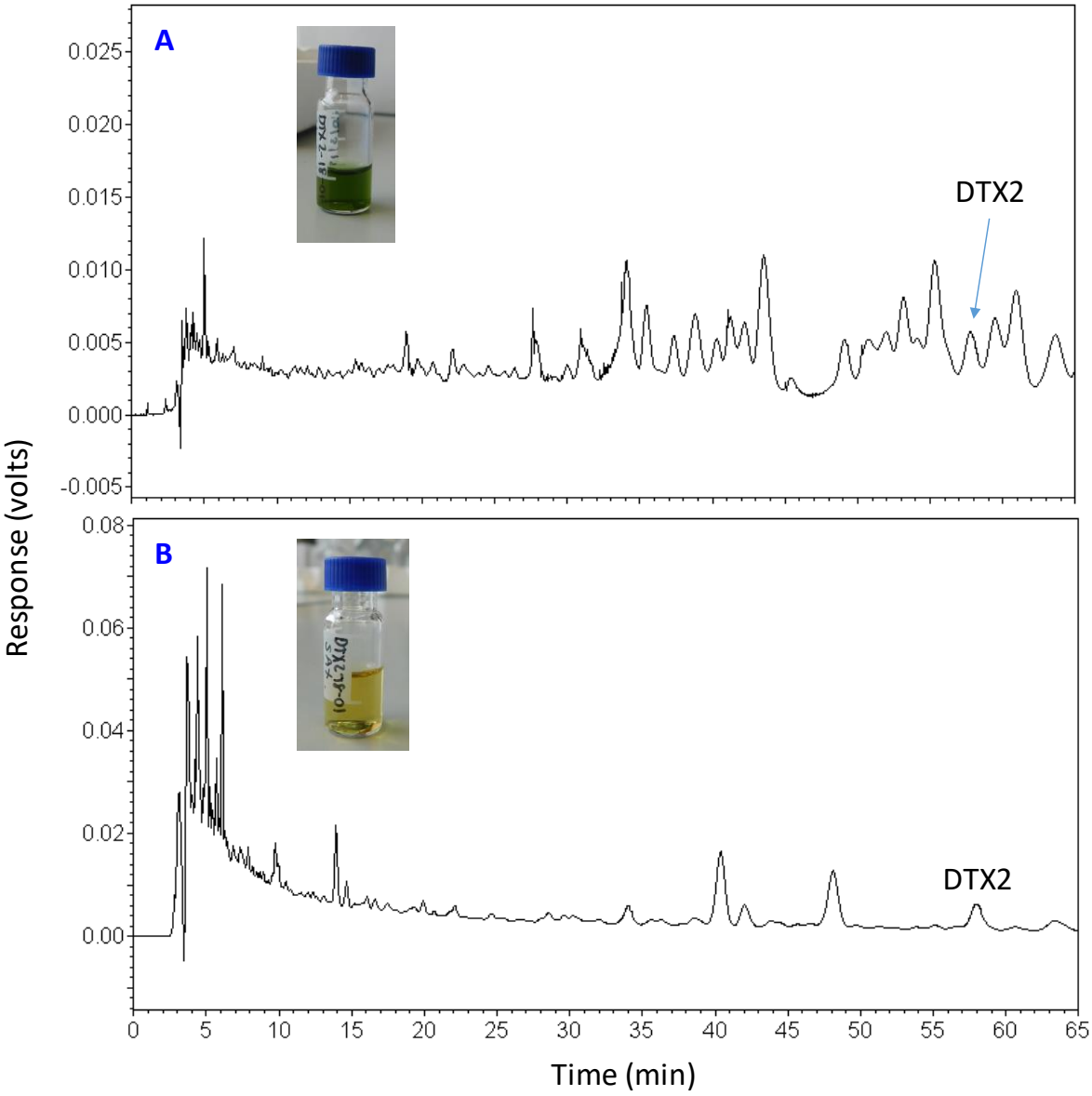

Figure S1. LC-UV (210 nm) chromatography showing difference in clean up between A) alumina (1 g loaded onto 30 g alumina) and B) SAX chromatography (3.4 g loaded onto 11.5 g SAX).

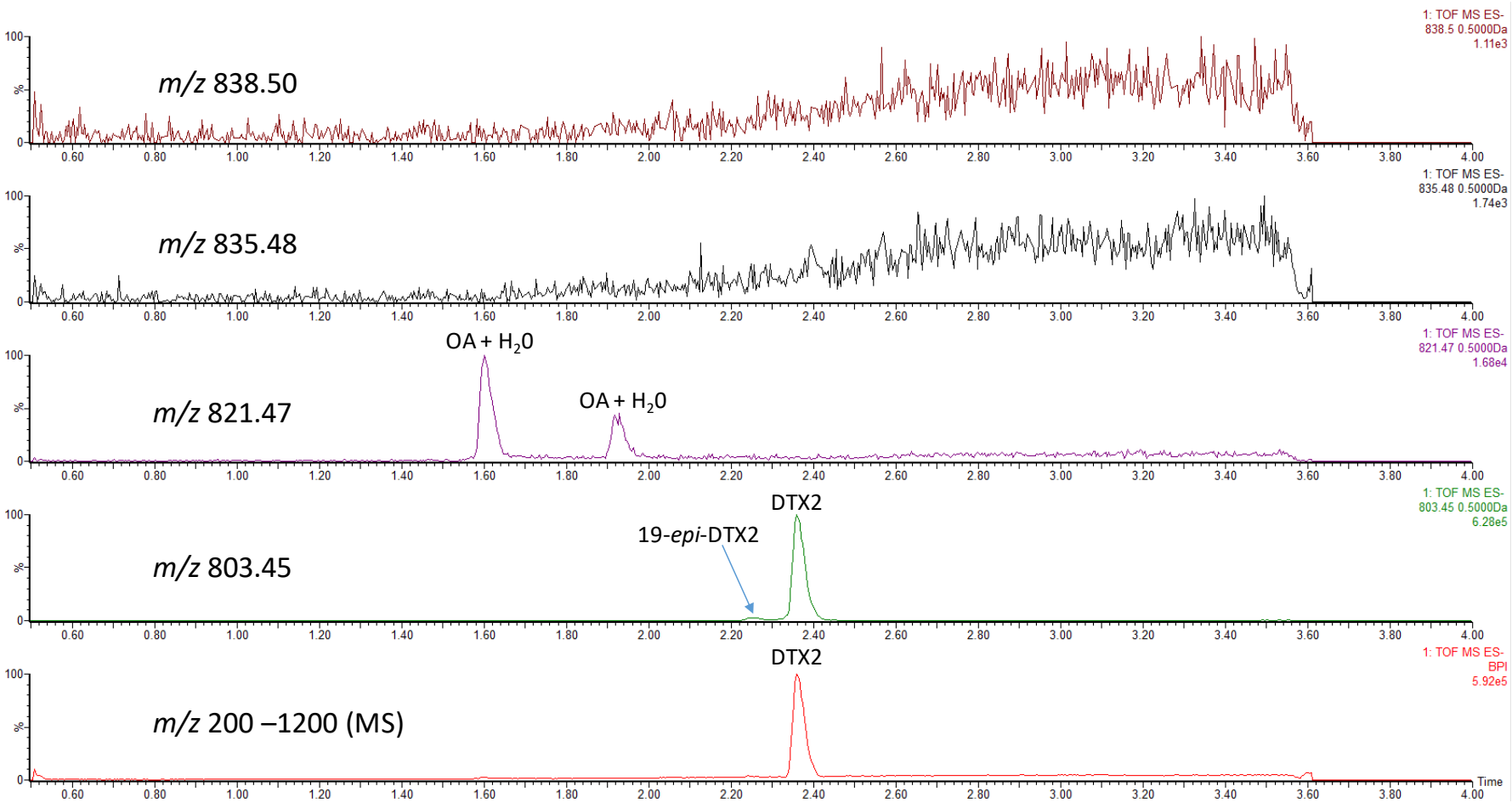

Figure S2. Purity analysis of DTX2 by LC-HRMS (acidic mobile phase, section 3.5.1).

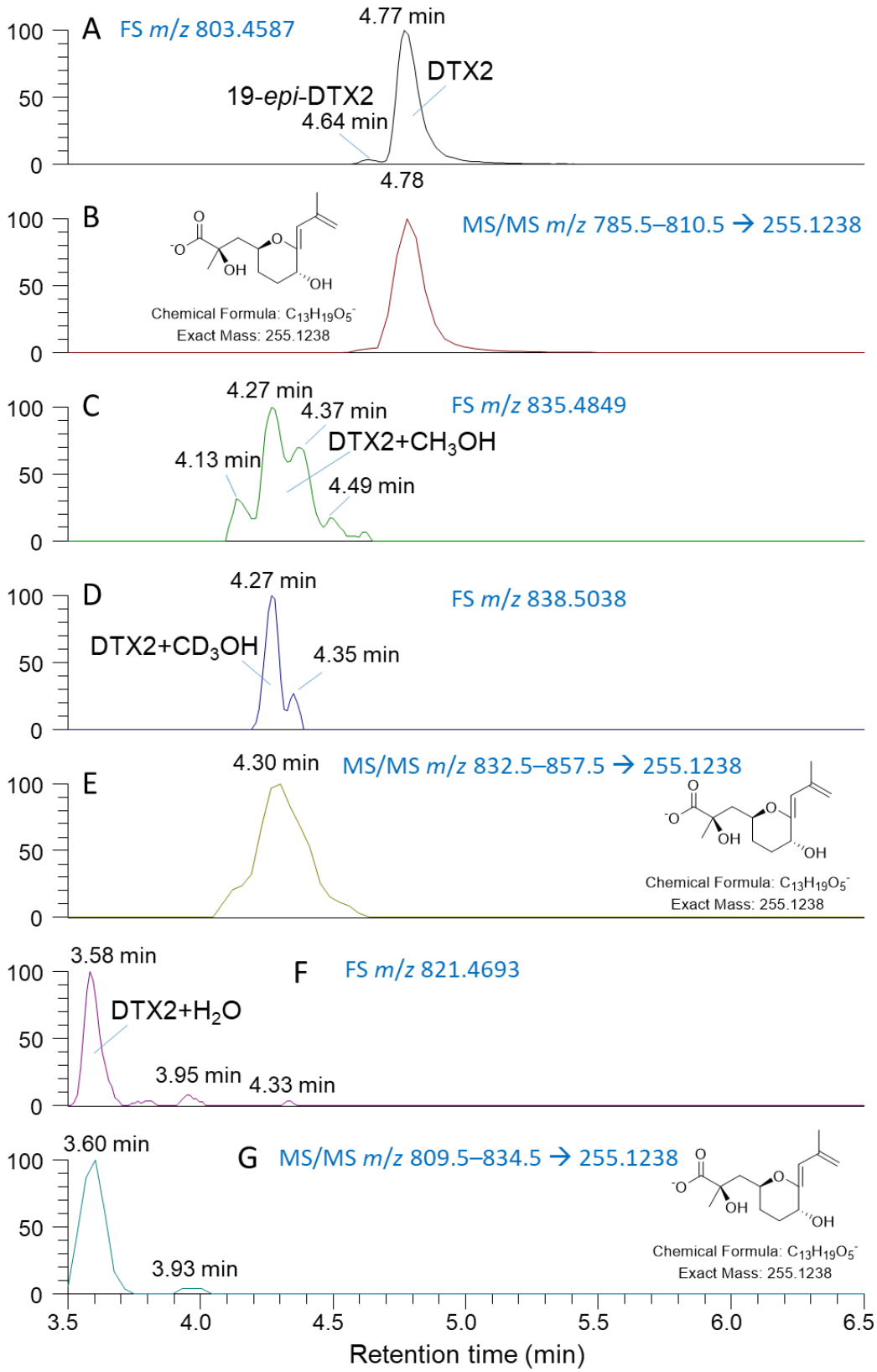

Figure S3. LC-HRMS/MS (neutral mobile phase, section 3.5.2) chromatograms of the purified DTX2 after NMR analysis, showing full scan (FS) chromatograms A, C, D and F extracted at the exact *m/z* values for DTX2, DTX2 + MeOH, DTX2 + CD_3OH, and DTX2 + H_2O (± 5 ppm), respectively, above the chromatograms of the corresponding DIA windows B, E and G extracted for product ions at *m/z* 255.1238 (± 5 ppm).

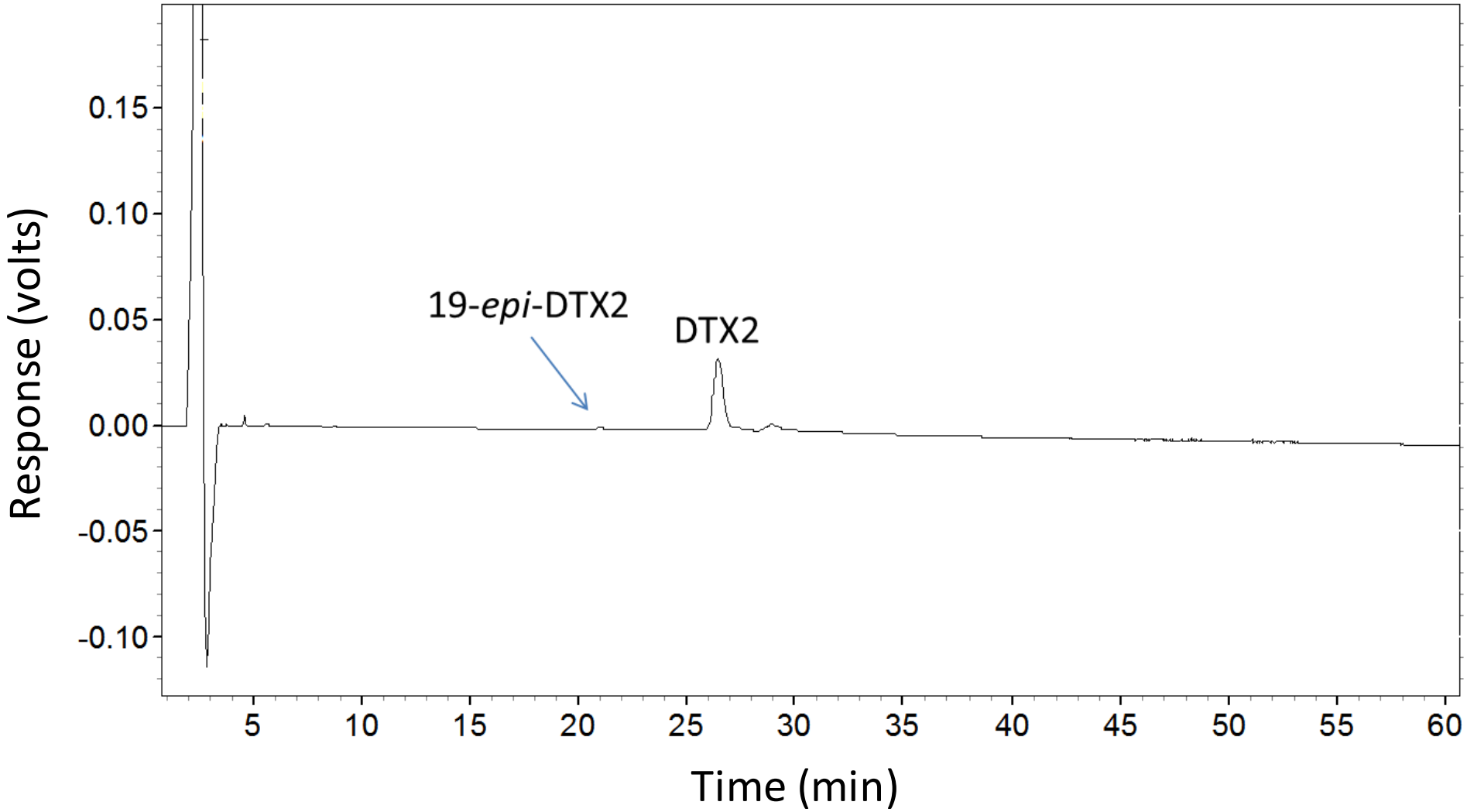

Figure S4. Purity analysis of DTX2 by LC-UV (210 nm, section 3.6).

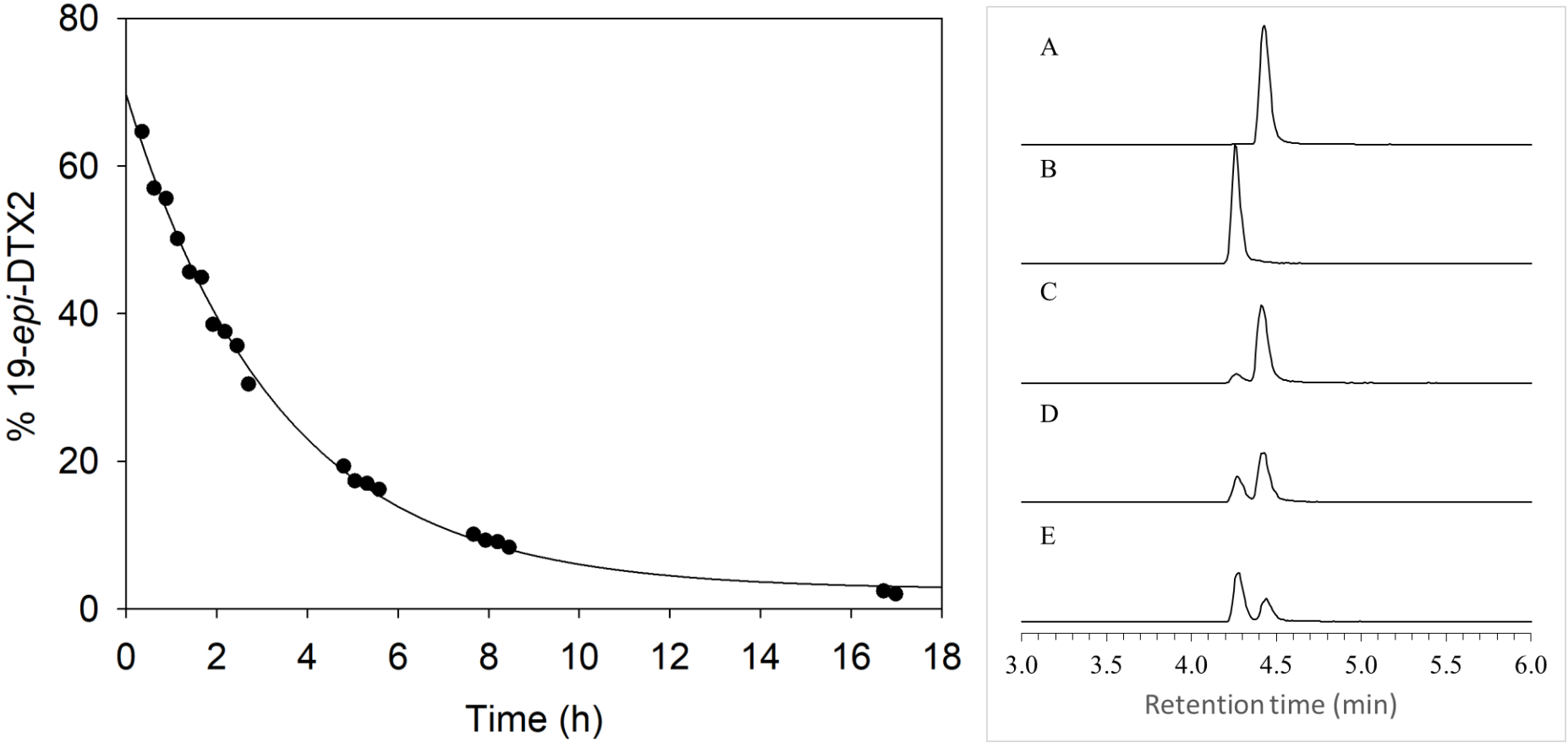

Figure S5. Left, percentage of 19-*epi*-DTX2 (as a percentage of the sum of 19-*epi*-DTX2 and DTX2) remaining versus time during incubation at 10 ℃ in MeOH containing ~1% formic acid. The fitted line is a 3-parameter exponential decay curve with $t_{½}$ 2.3 h (SE ±0.1) and final equilibrium concentration 2.6% (SE ±0.7). Right, LC–MS/MS chromatograms (*m*/*z* 803.5→255.1) of: A) NRC-CRM-DTX2b; B) purified 19-*epi*-DTX2, and; C–E) 19-*epi*-DTX2 incubated for 7.68, 2.70, and 0.35 h in MeOH containing ~1% formic acid, which formed part of the kinetic analysis shown in the graph on the left.

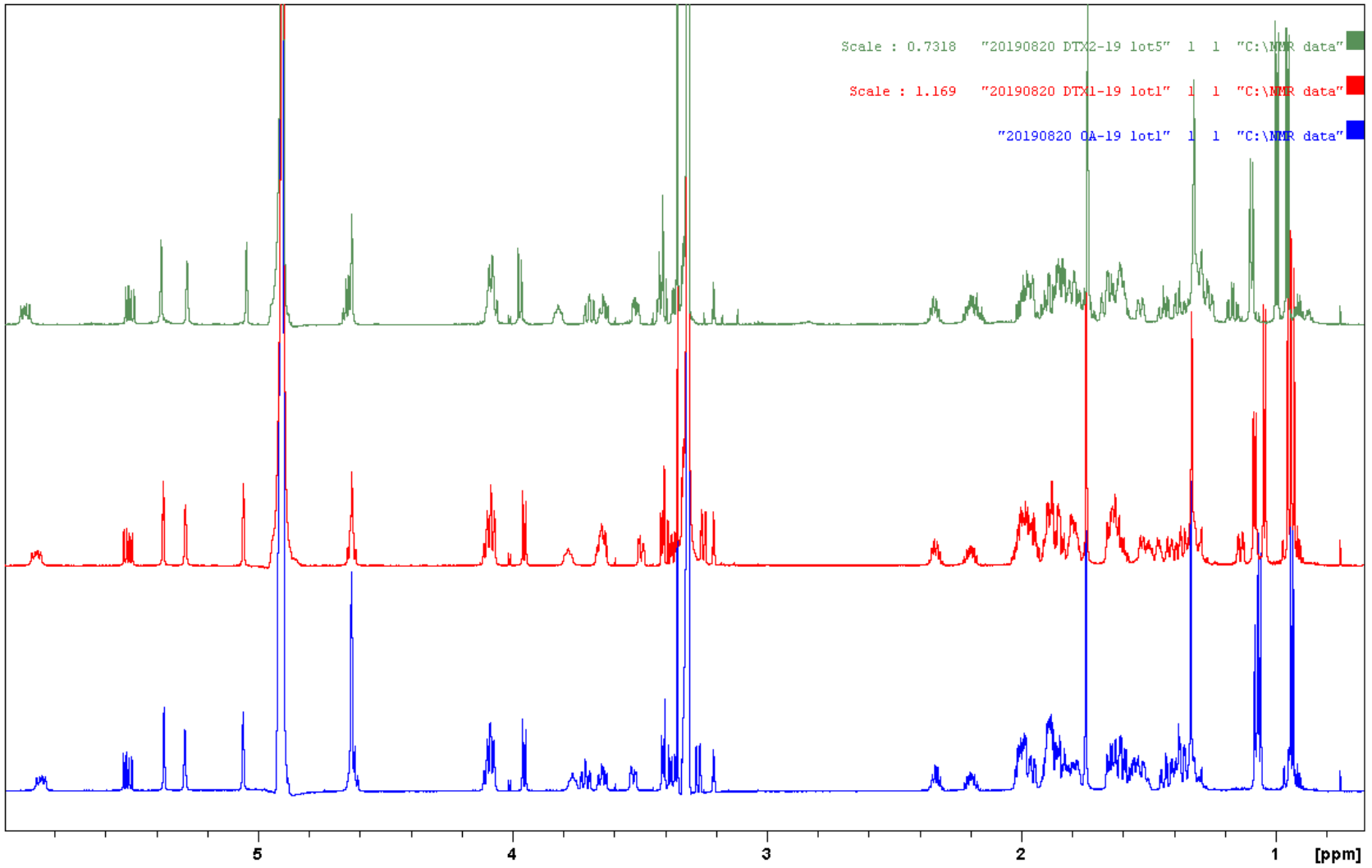

Figure S6. ^{1}H NMR spectra (0.65–6.00 ppm) of: top, DTX2 (green); middle, DTX1 (red), and; OA (blue) in CD_3OD. Spectral intensities are scaled so that spectra have the same peak heights for the olefinic resonances at 5.0–5.6 ppm.

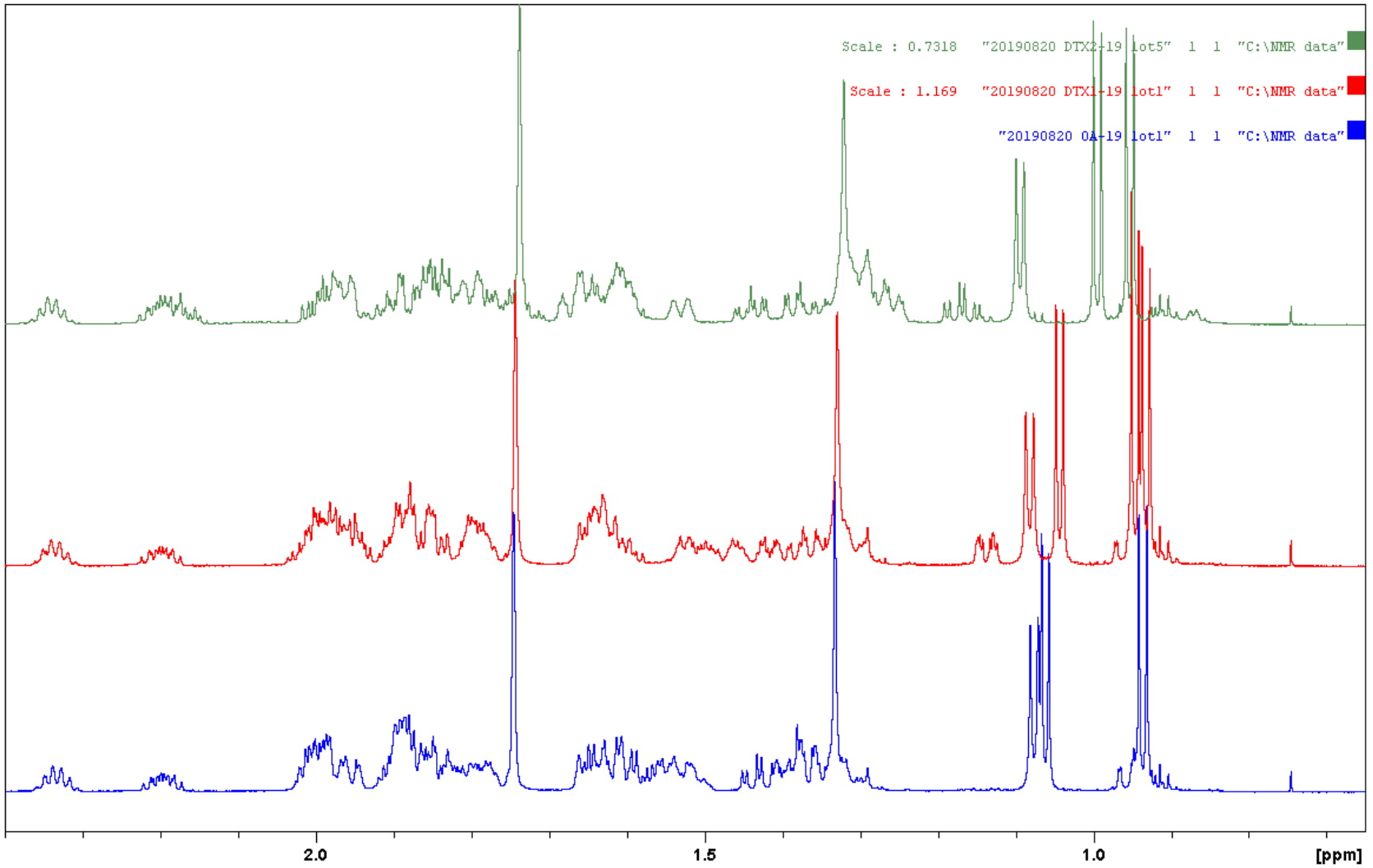

Figure S7. Expansion (0.65–2.40 ppm) of the ^{1}H NMR spectra shown in Figure S6. Top, DTX2 (green); middle, DTX1 (red), and; OA (blue) in CD_3OD.

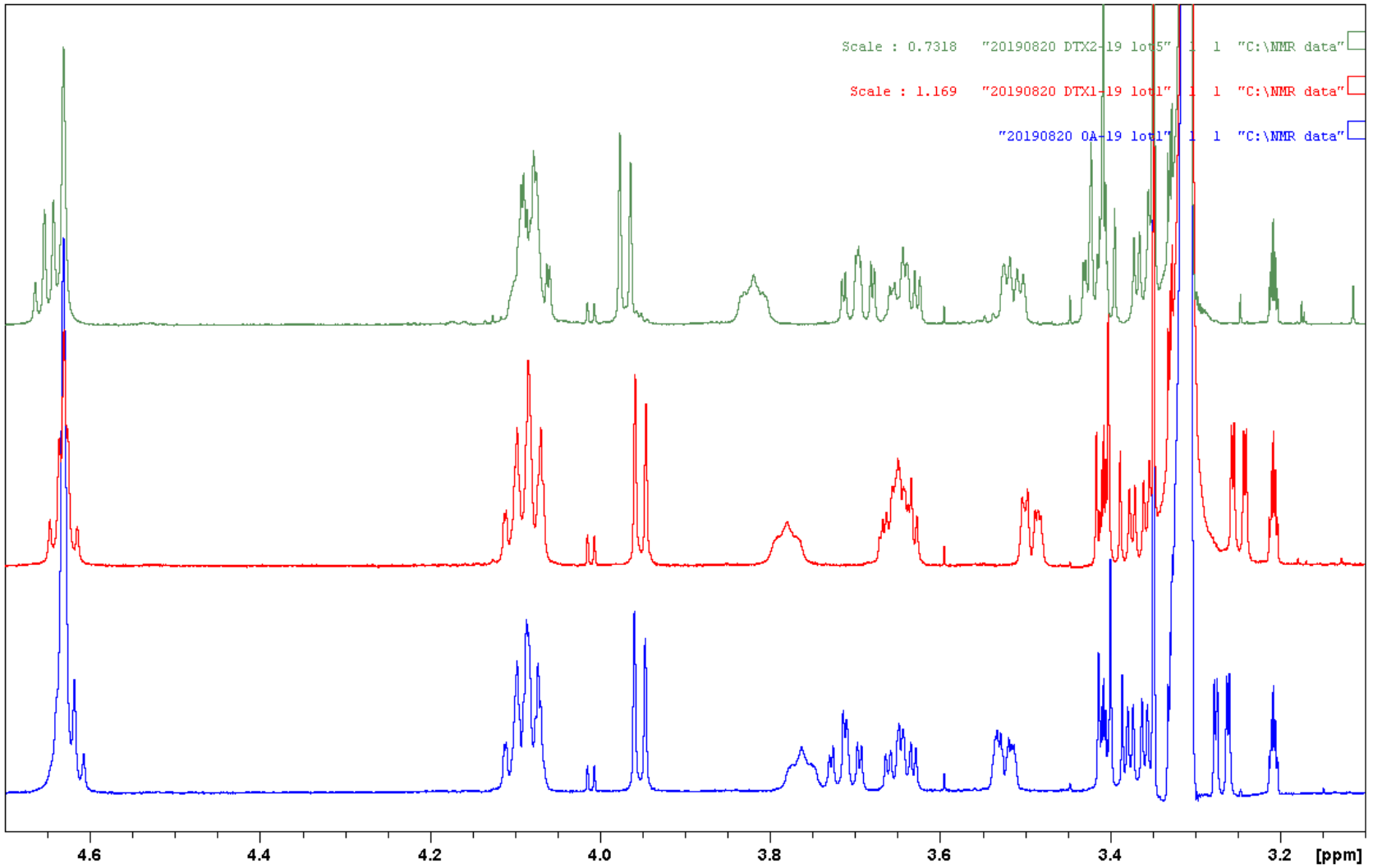

Figure S8. Expansion (3.10–4.70 ppm) of the ^{1}H NMR spectra shown in Figure S6. Top, DTX2 (green); middle, DTX1 (red), and; OA (blue) in CD_3OD.

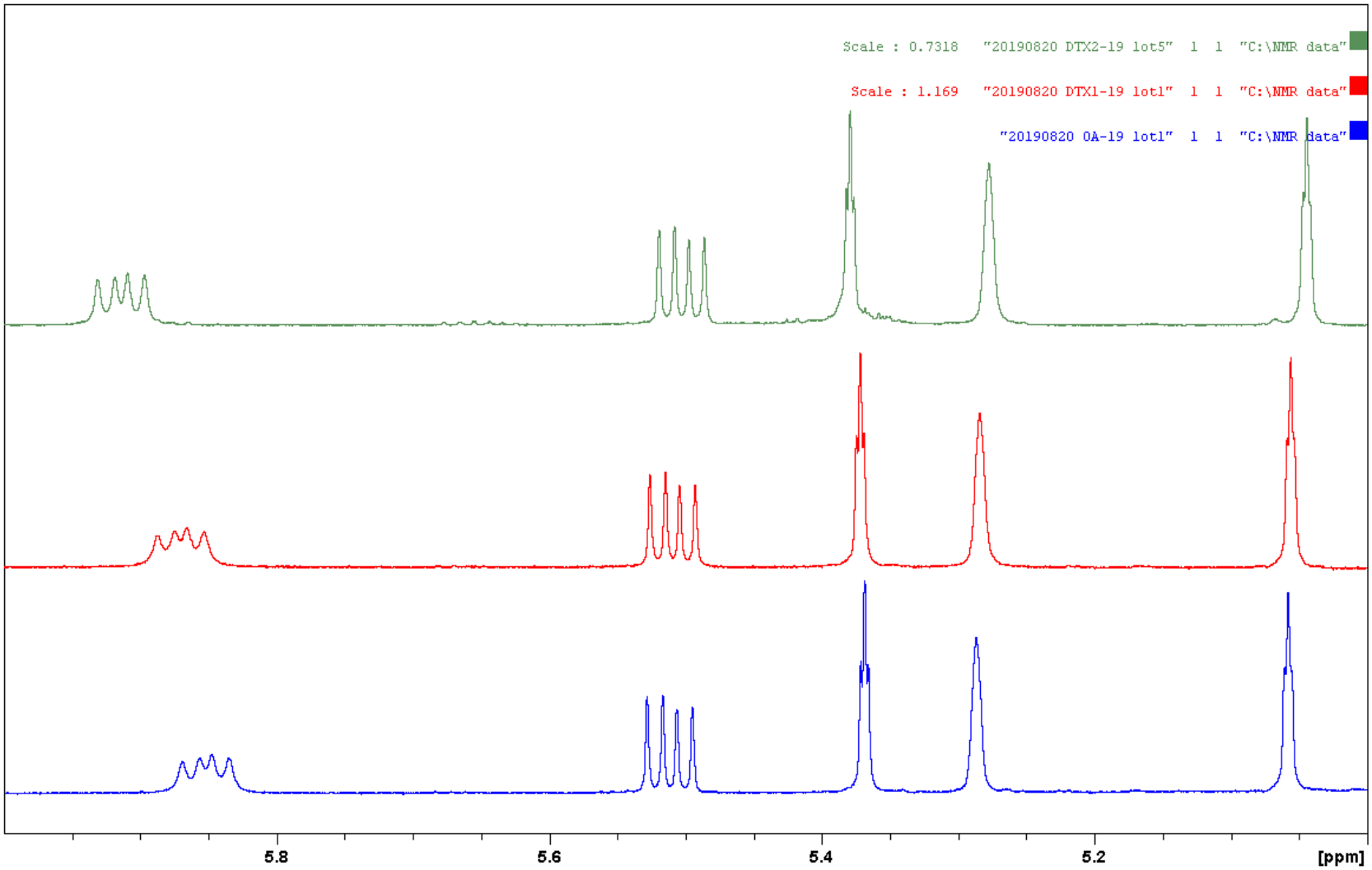

Figure S9. Expansion (5.00–6.00 ppm) of the ^{1}H NMR spectra shown in Figure S6. Top, DTX2 (green); middle, DTX1 (red), and; OA (blue) in CD_3OD.

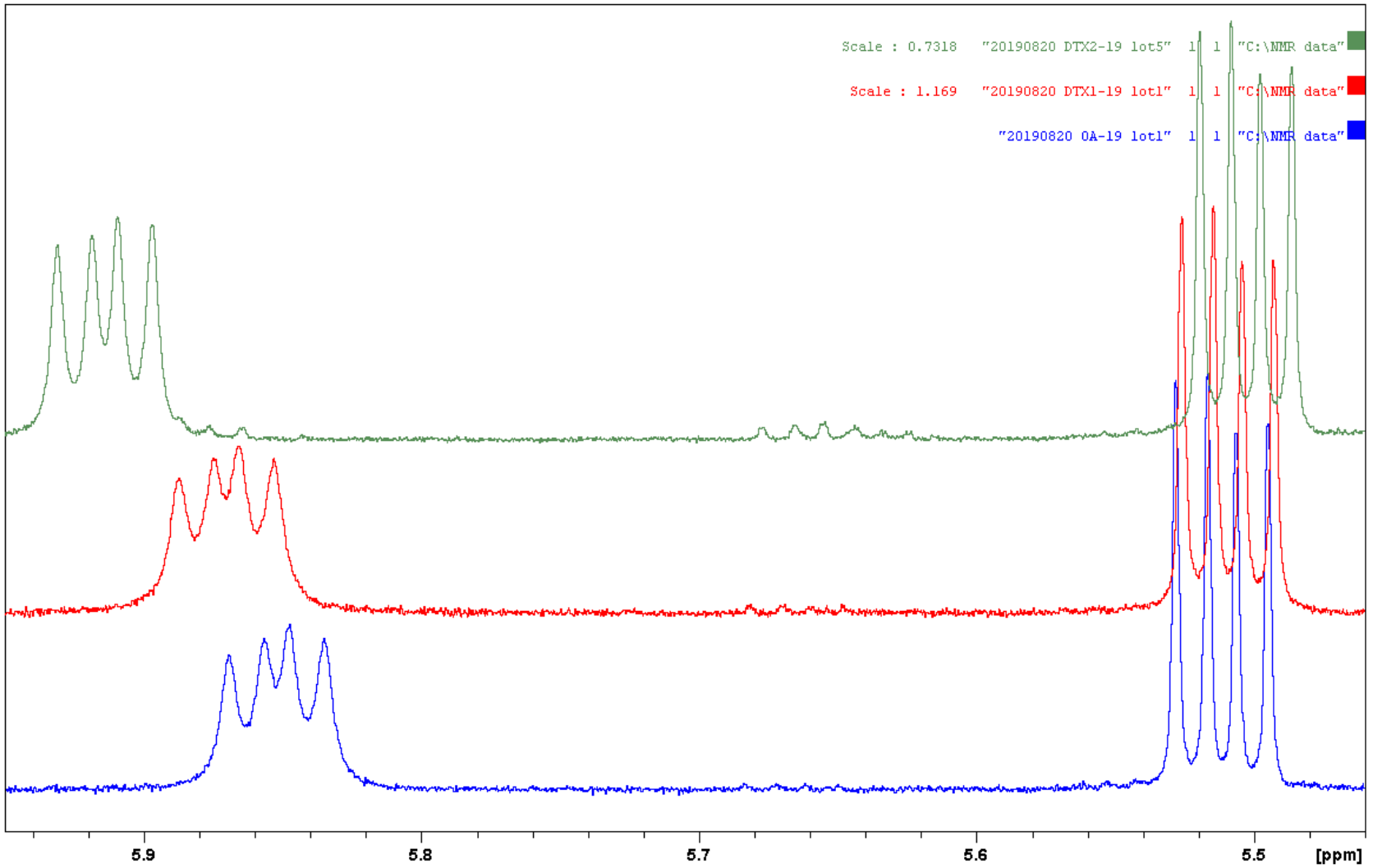

Figure S10. Expansion (5.40–5.95 ppm) of the ^{1}H NMR spectra shown in Figure S6. Top, DTX2 (green); middle, DTX1 (red), and; OA (blue) in CD_3OD. Signs of signals from possible isomers and analogues are visible at ~5.66 ppm, particularly in DTX2 (~5%). Other minor resonances at 5.88 ppm (DTX2) and at 5.55 may also be due to structurally-related impurities.

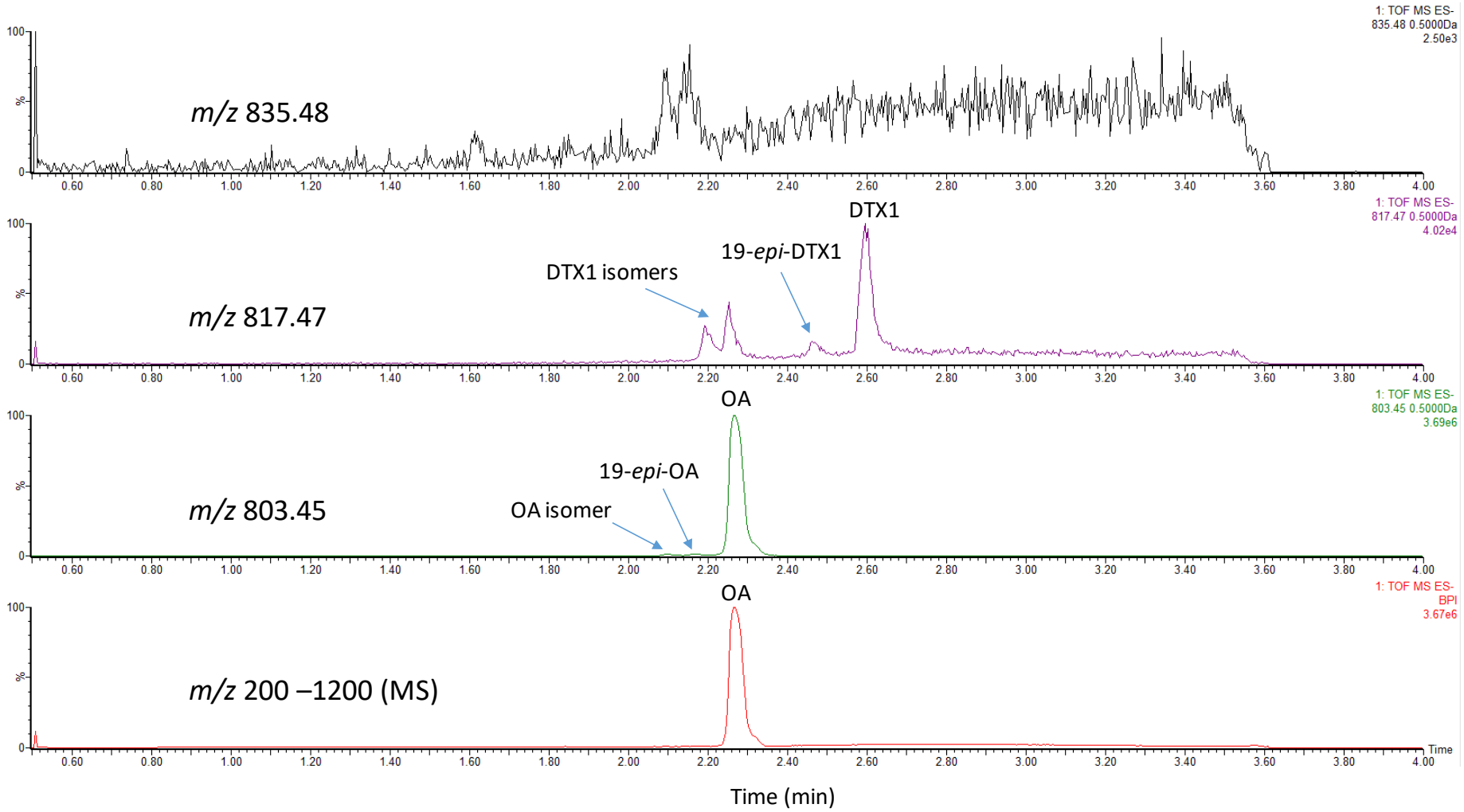

Figure S11. Purity analysis of OA by LC-HRMS (acidic mobile phase, section 3.5.1).

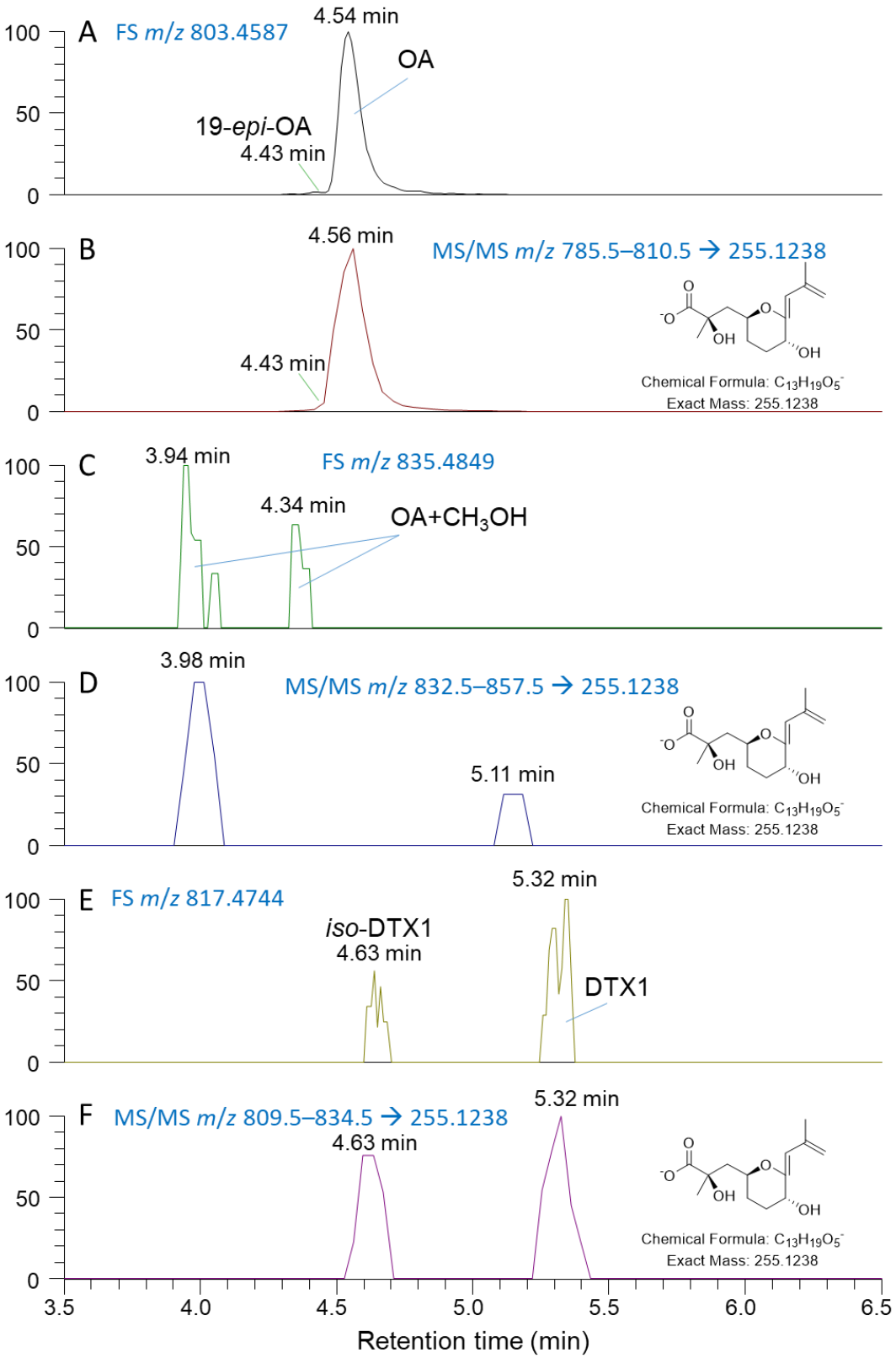

Figure S12. LC-HRMS/MS (using neutral mobile phase, section 3.5.2) chromatograms of the purified OA after NMR analysis, showing full scan (FS) chromatograms A, C, and E extracted at the exact *m/z* values for OA, OA + MeOH, and DTX1/*iso*-DTX1 (± 5 ppm), respectively, above the chromatograms of the corresponding DIA windows B, D and F extracted for product ions at *m/z* 255.1238 (± 5 ppm).

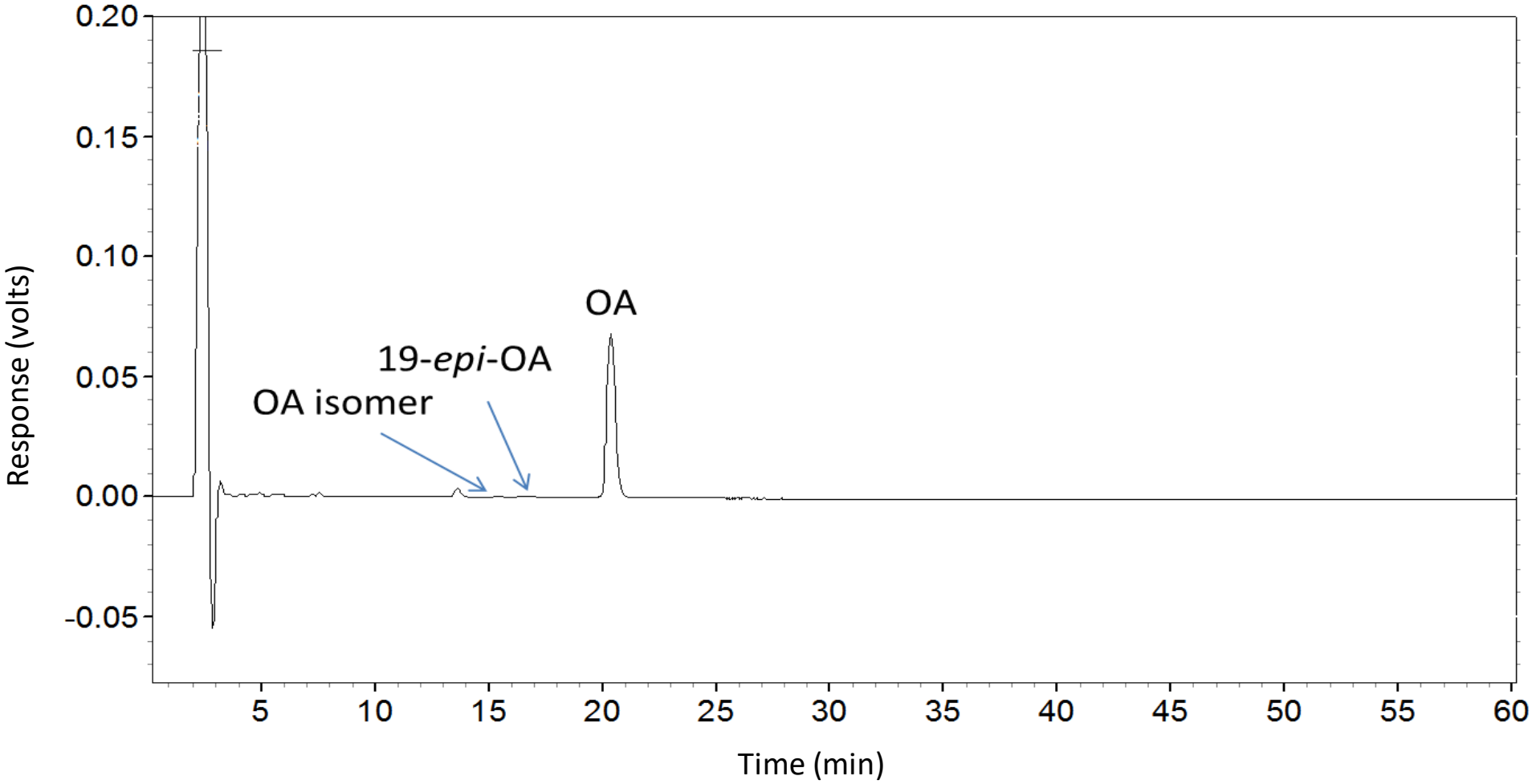

Figure S13. Purity analysis of OA by LC-UV (210 nm, section 3.6).

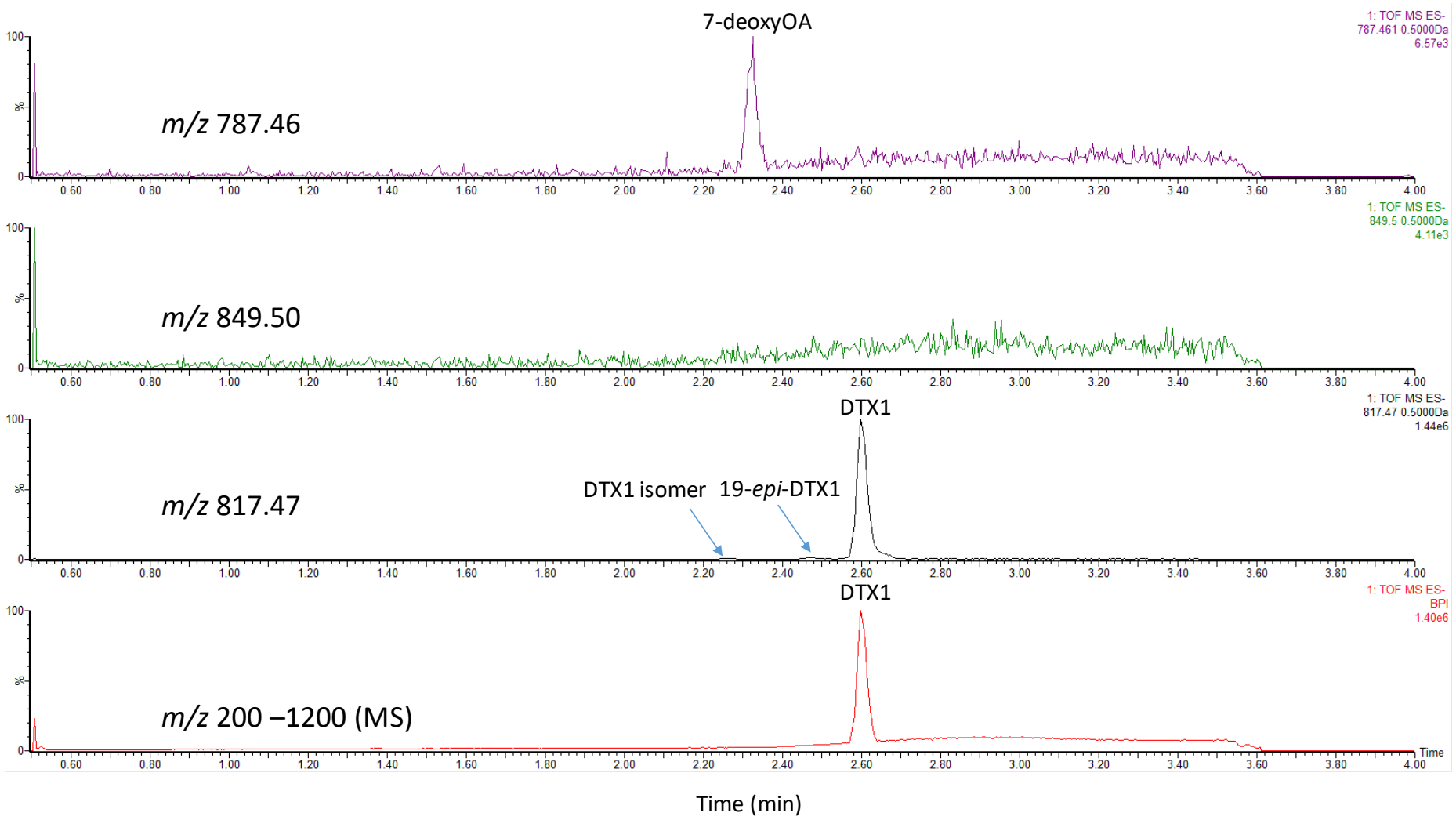

Figure S14. Purity analysis of DTX1 by LC-HRMS (acidic mobile phase, section 3.5.1).

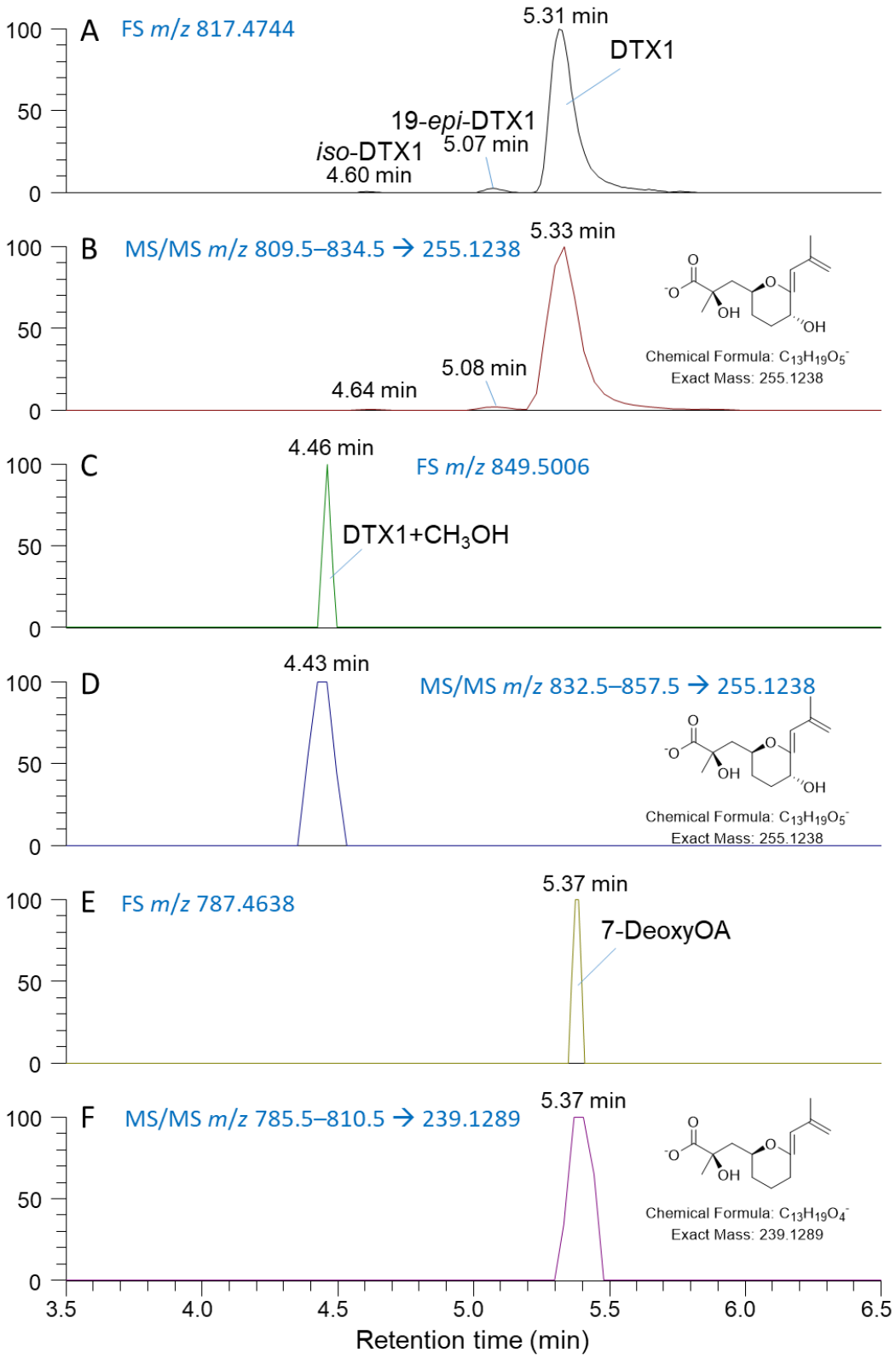

Figure S15. LC-HRMS/MS (neutral mobile phase, section 3.5.2) chromatograms of the purified DTX1 after NMR analysis, showing full scan (FS) chromatograms A, C, and E extracted at the exact *m/z* values for DTX1, DTX1 + MeOH, and 7-deoxyOA (±5 ppm), respectively, above the chromatograms of the corresponding DIA windows B, and D extracted for product ions at *m/z* 255.1238 and F extracted for *m/z* 239.1289 (±5 ppm).

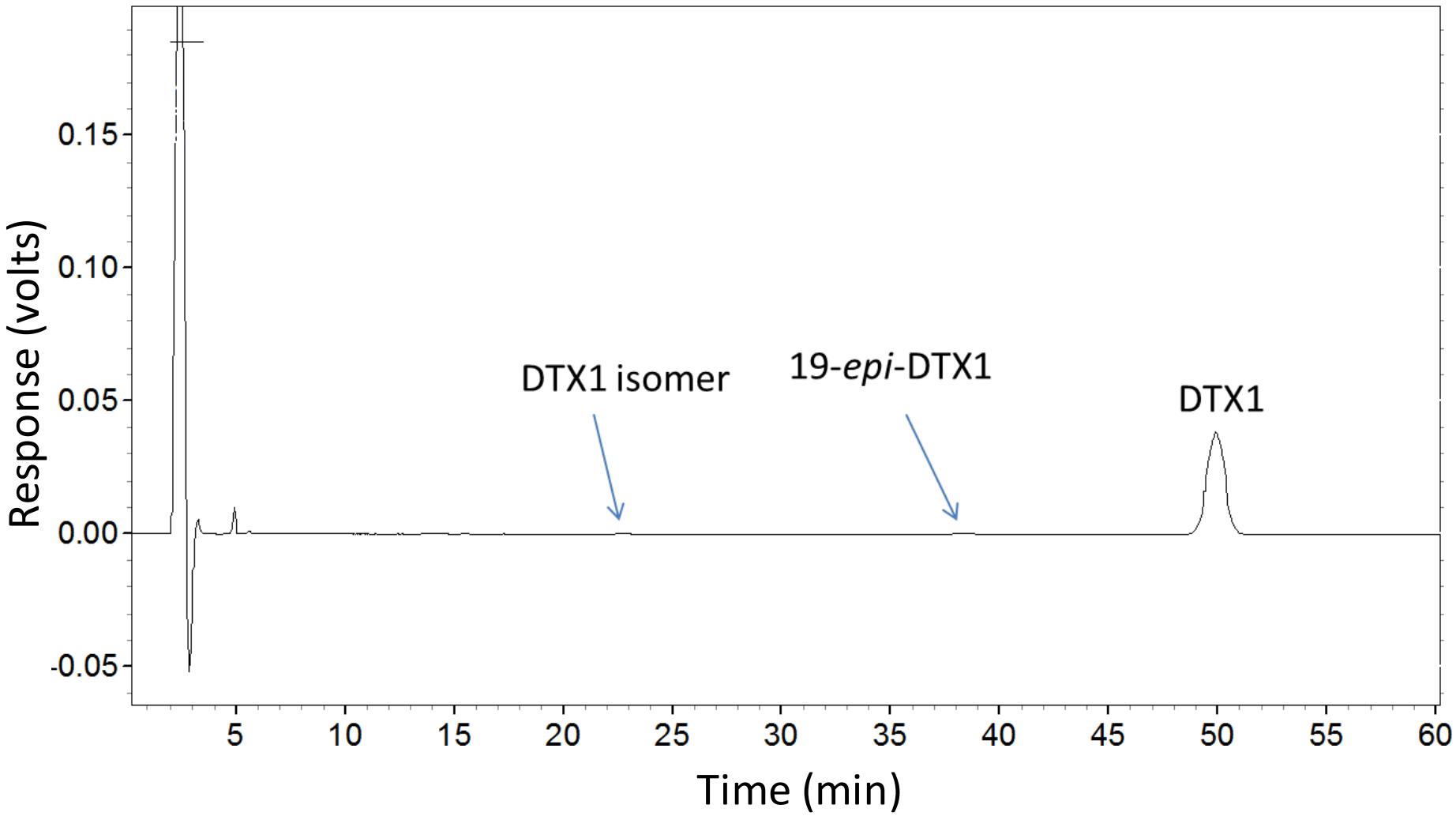

Figure S16. Purity analysis of DTX1 by LC-UV (210 nm, section 3.6).