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Supporting Information

Synthesis of β-arylbutenolides mediated by $BF_3.OMe_2$

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MATERIALS AND INSTRUMENTATION

Tetronic acid was purchased from Alfa Aesar and the other chemicals and solvents were purchased from Sigma-Aldrich and were used as received without further purification. Microwave irradiation was carried out in a Reactor Discover Reflux (CEM corporation, 300 W) in open vessel. All experiments were monitored by thin layer chromatography using Merck silica gel 60 F_{254} plates. The products were visualized with 356 and 254 nm UV lamps. The ^{1}H and ^{13}C NMR spectra were determined on a Bruker DRX 400 spectrometer in DMSO-d_6. Melting points were measured with a Buchi B-540 melting point apparatus and are uncorrected. The high-resolution mass spectral data were obtained using a Bruker Daltonics - micrOTOF-Q, fitted with an ESI operating in the positive ion mode.

General Procedure for the synthesis of β-arylbutenolides derivatives

A mixture of tetronic acid (1.0 mmol), phenol (1.0 mmol) or aryl ether, and $BF_3.OMe_2$ (2.0 eq) in $CHCl_3$ (3 mL) was taken in a reaction flask equipped with a small magnetic stirring bar and the reflux condenser. The mixture was irradiated in a microwave reactor at a power of 220 W. The reaction was monitored by TLC, and after consumption of the starting materials was cooled to room temperature. The solvent was removed from the vacuum and the precipitated crude product was washed with a mixture of hexane-ethyl acetate (8:2). When sesamol was used as phenol, the mixture was maintained at room temperature under stirring for 24 h. The proceeding after the reaction was the same described above.

Spectral Data of Compounds

4-(2-hydroxy-4,5-dimethoxyphenyl)furan-2(5H)-one (3a)

Yield: 92% (0.2149 g). Mp 236-237 ºC.

^{1}H NMR (DMSO-d_6, 400 MHz): δ 10.3 (s, OH), 6.94 (s, 1H), 6.46 (s, 1H), 6.40 (s, 1H), 5.21 (s, 2H), 3.67 (s, 3H), 3.64 (s, 3H).

^{13}C NMR (DMSO-d_6, 125 MHz): δ 174.4, 162.6, 152.8, 152.7, 142.1, 111.3, 109.5, 107.5, 100.4, 72.4, 56.3, 55.4.

HRMS (ESI^+): m/z $[M+H]^+$ calcd for $C_{12}H_{12}O_5Na$: 259.0581; found: 259.0572.

4-(6-hydroxybenzo[d][1,3]dioxol-5-yl)furan-2(5H)-one (3b)

Yield: 85% (0.1885g). Mp 228-230 ºC.

^{1}H NMR (DMSO-d_6, 400 MHz): δ 10.63 (s, OH), 7.14 (s, 1H), 6.54 (s, 1H), 6.46 (s, 1H), 6,03 (s, 2H), 5.26 (s, 2H).

^{13}C NMR (DMSO-d_6, 125 MHz): δ 174.4, 162.5, 154.0, 150.8, 140.7, 109.8, 108.3, 106.4, 101.7, 97.6, 72.5.

HRMS (ESI$^+$): m/z $[M+H]^+$ calcd for $C_{11}H_8O_5Na$: 243.0268; found: 243.0263.

4-(2,4-dihydroxyphenyl)furan-2(5H)-one (3c)

Yield: 95% (0.1825 g). Mp 241-242 ºC.

^{1}H NMR (DMSO-d_6, 400 MHz): δ 10.56 (s, OH), 10.09 (s, OH), 7.34 (d, 1H, J = 8.6 Hz), 6.40 (d, 1H, J = 2.6 Hz), 6.37 (d, 1H, J = 1.3 Hz), 6.33 (dd, 1H, J = 2.2 Hz and J = 8.6 Hz), 5.26 (brs, 2H).

^{13}C NMR (DMSO-d_6, 125 MHz): δ 174.6, 162.4, 161.6, 159.0, 130.0, 109.8, 108.8, 107.8, 102.6, 72.5.

HRMS (ESI$^+$): m/z $[M+H]^+$ calcd for $C_{10}H_8O_4Na$: 215.0319; found: 215.0313.

4-(2,4-dimethoxyphenyl)furan-2(5H)-one (3d)

White solid Mp 205-207 ºC. Yield: 0.0202 g (92%)

^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.52 (d, 1H, J = 8.66 Hz), 6.69 (d, 1H, J = 2.33 Hz), 6,64 (dd, 1 H, J = 8.65 Hz, J = 2.33 Hz), 6.50-6.46 (m, 1H), 5.29 (d, 2H, J = 1.55 Hz), 3.90 (s, 3H), 3.84 (s, 3H).

^{13}C NMR (DMSO-d_6, 125 MHz): δ 174.3, 163.3, 161.1, 160.0, 130.0, 111.2, 111.0, 106.2, 98.6, 72.1, 55.8, 55.6.

HRMS (ESI$^+$): m/z $[M+Na]^+$ calcd for $C_{12}H_{12}O_4Na$: 243.0633; found: 243.0632

NMR (^{1}H and ^{13}C) and HRMS Spectra of compounds 3a –3d

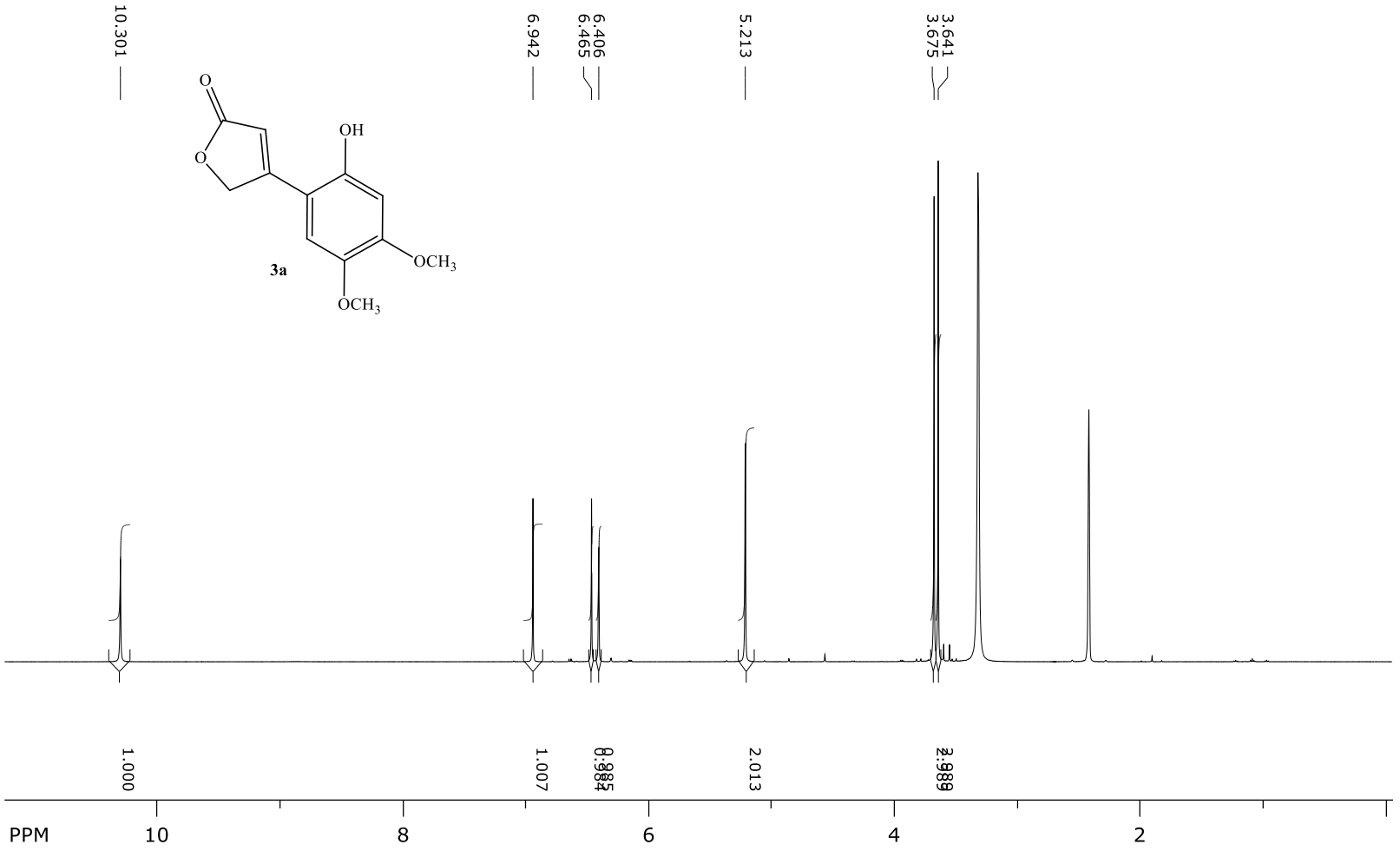

Figure 1S- ^{1}H NMR Spectrum of compound **3a** in DMSO-d6

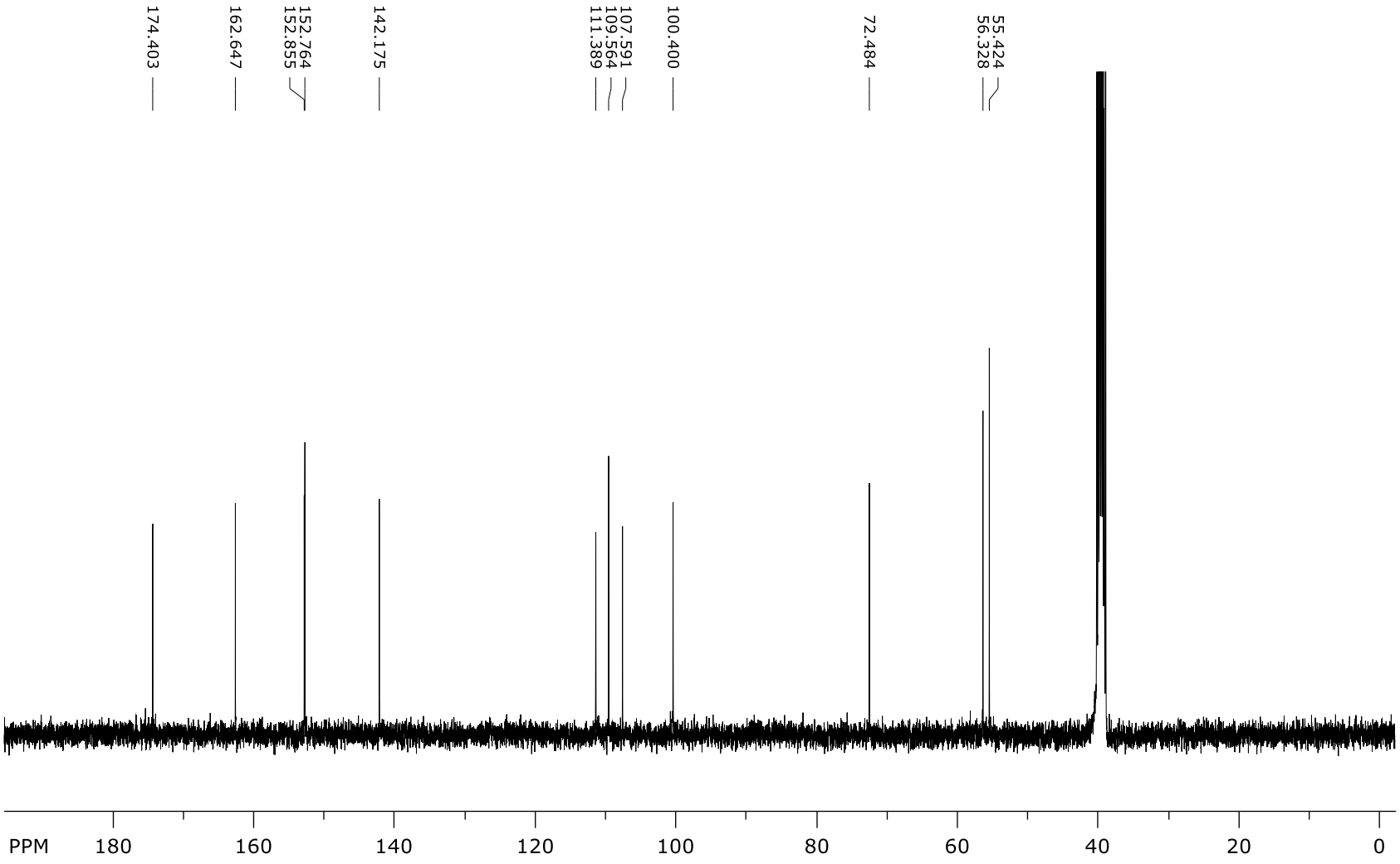

Figure 2S- ^{13}C NMR Spectrum of compound **3a** in DMSO-d6

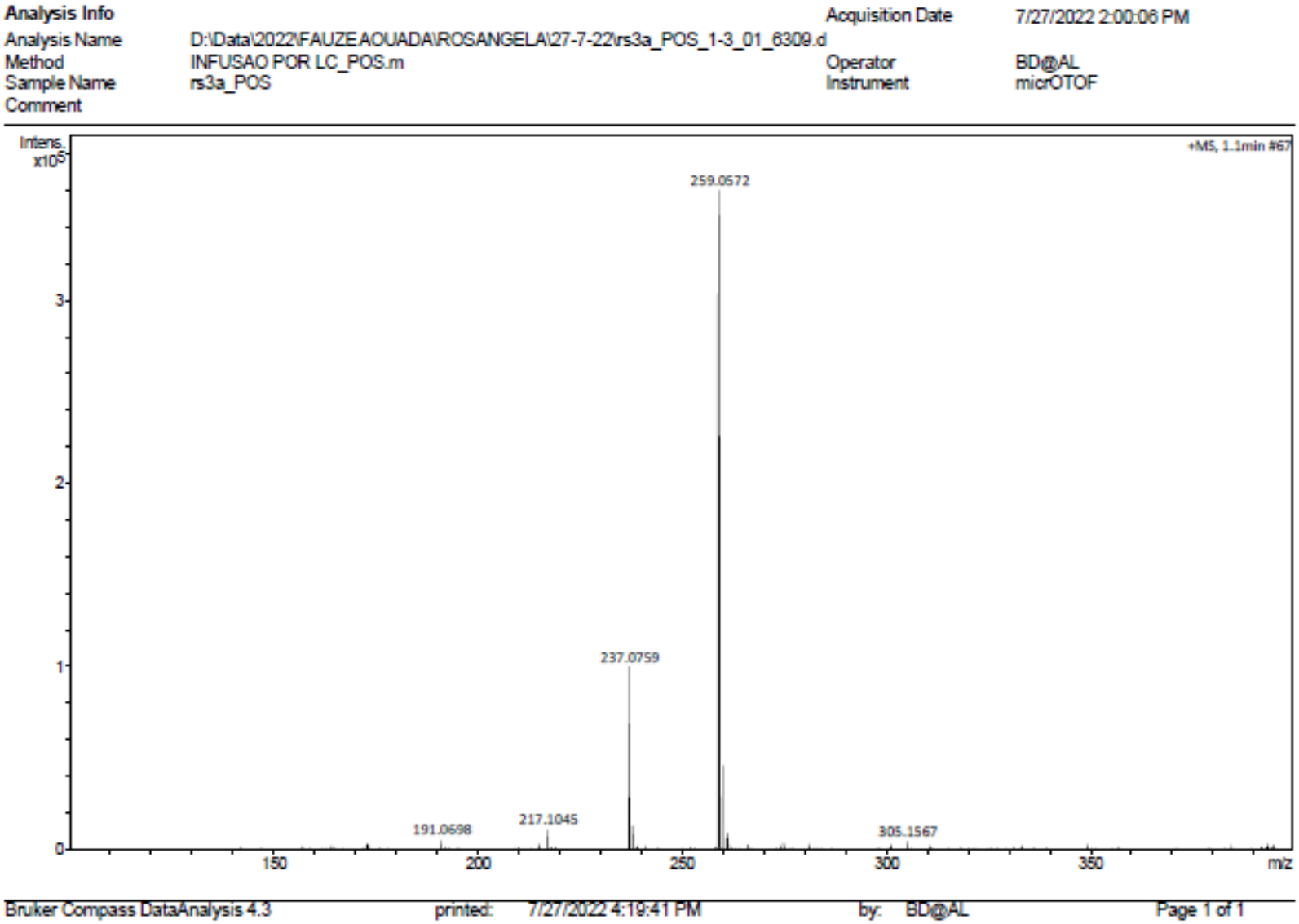

Figure 3S- HRMS spectrum of compound **3a**

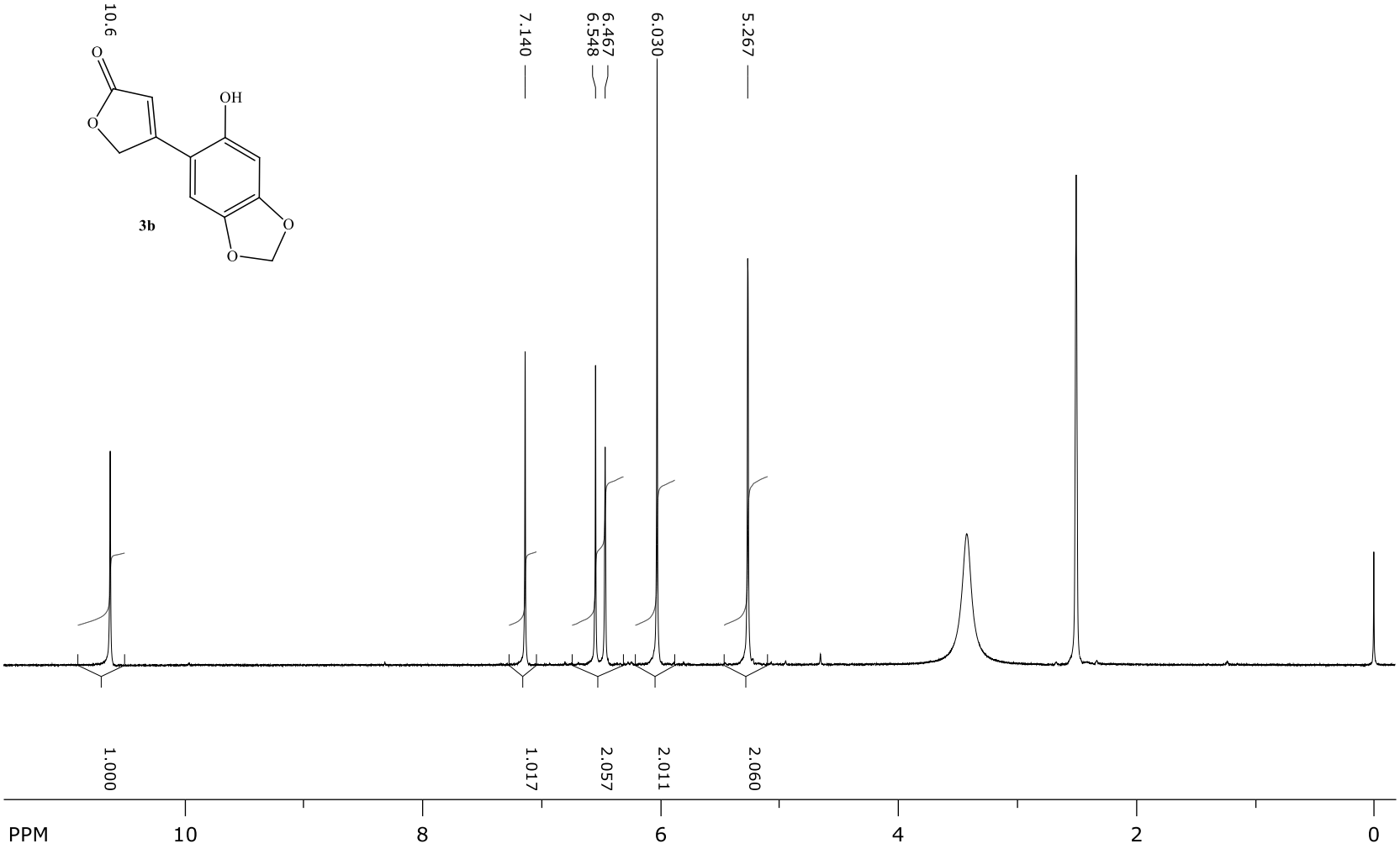

Figure 4S- ^{1}H NMR Spectrum of compound **3b** in DMSO-d6

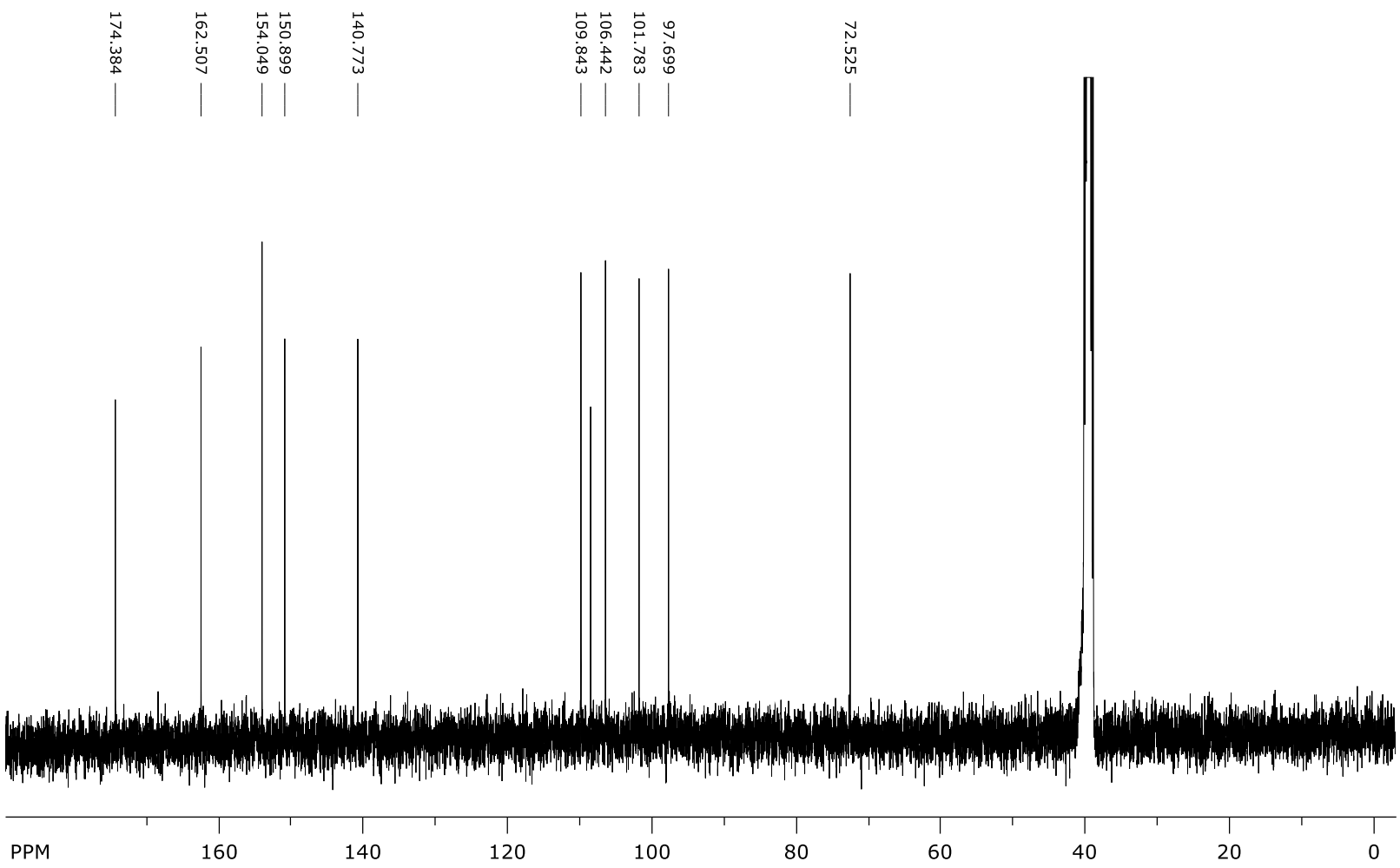

Figure 5S- ^{13}C NMR Spectrum of compound **3b** in DMSO-d6

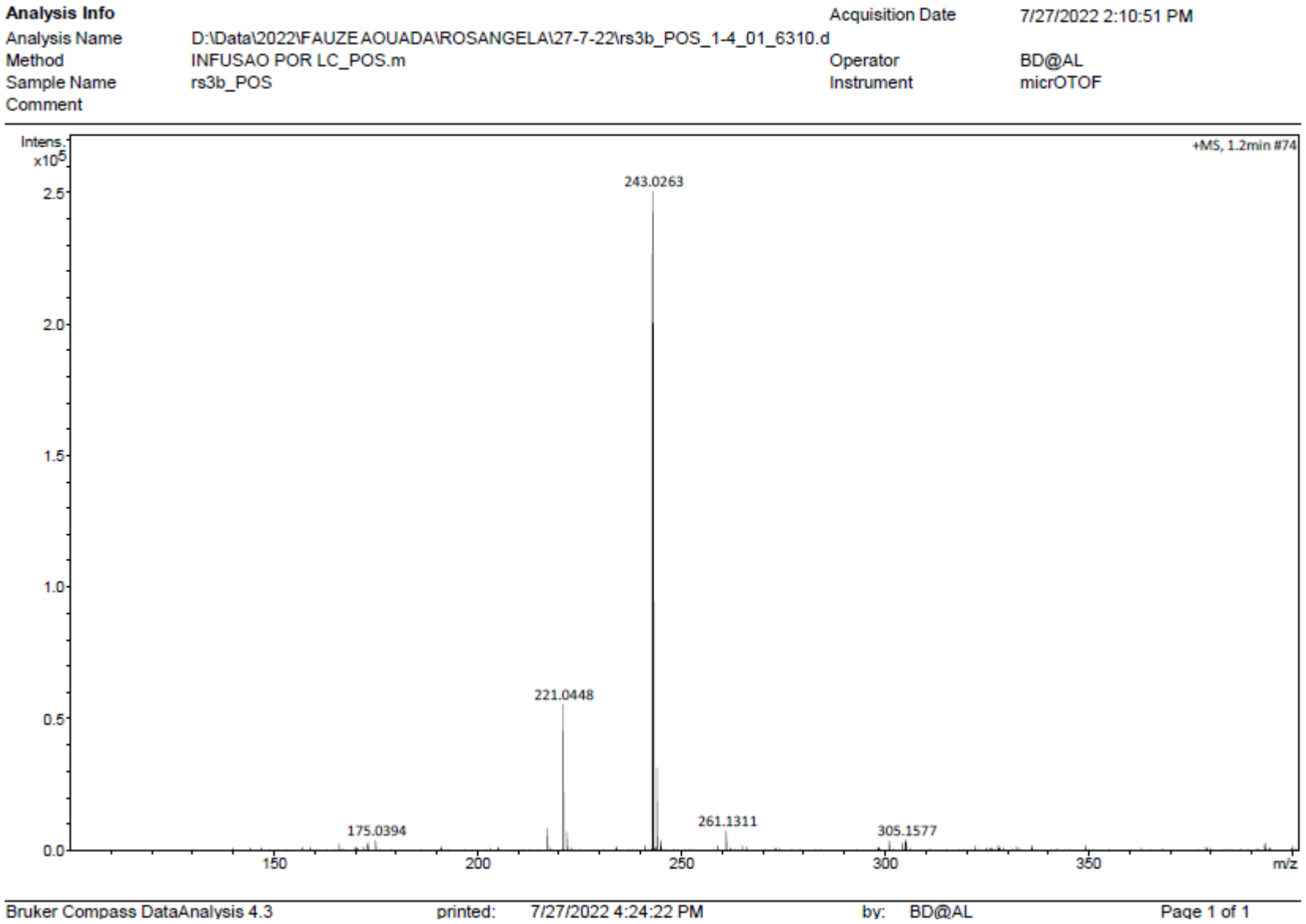

Figure 6S. HRMS spectrum of compound **3b**

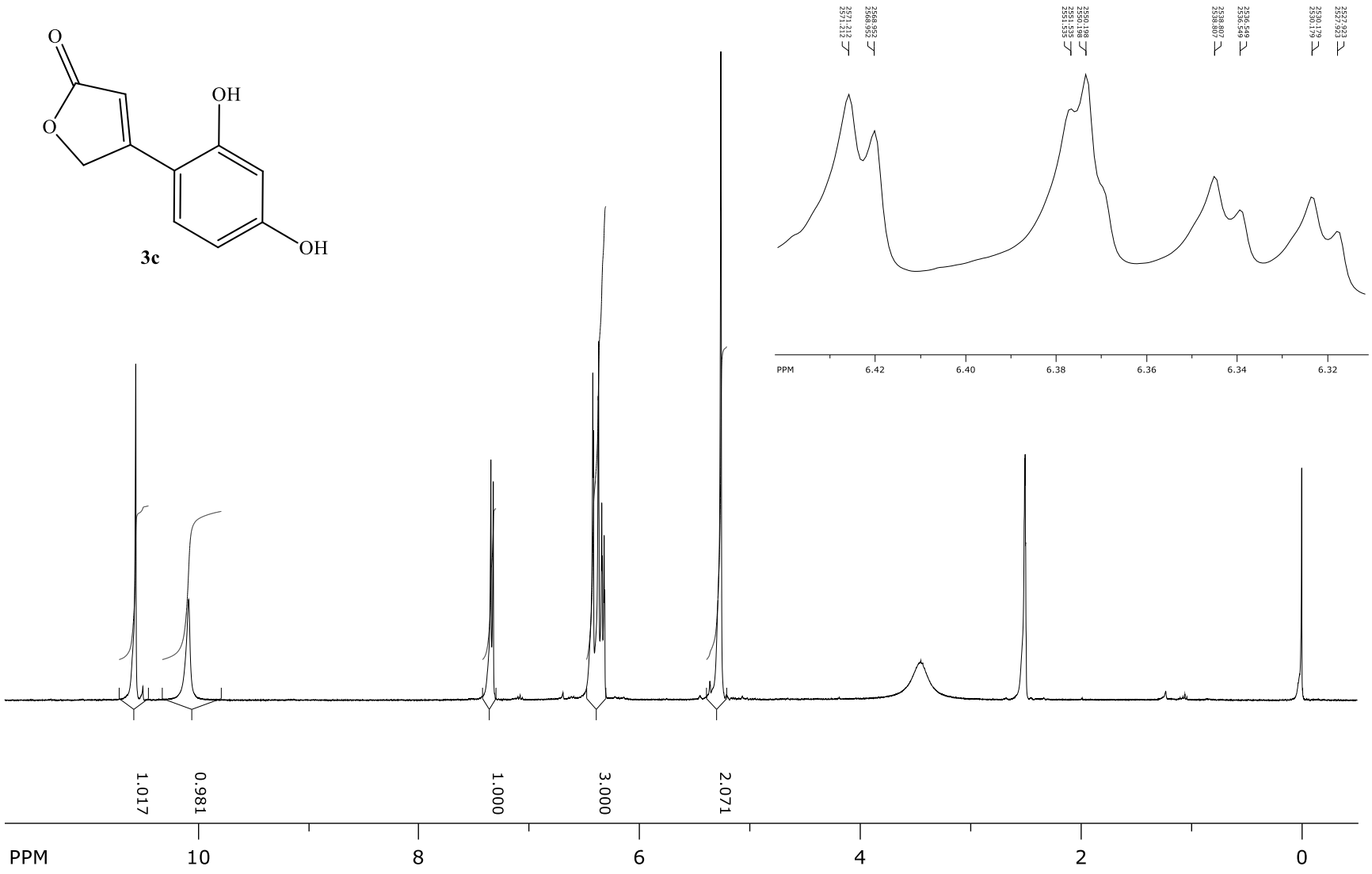

Figure 7S- ^{1}H NMR Spectrum of compound **3c** in DMSO-d6

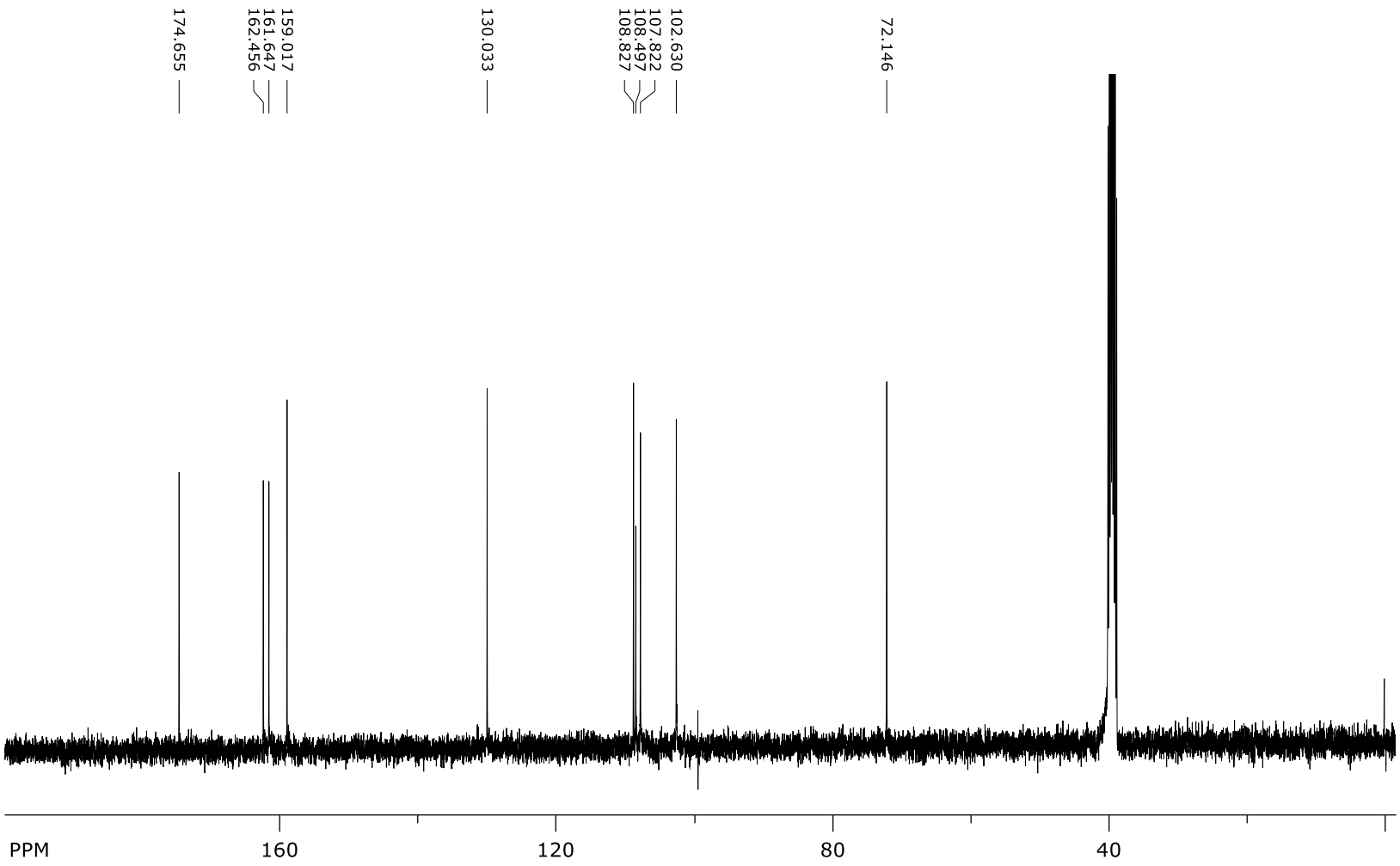

Figure 8S- ^{13}C NMR Spectrum of compound **3c** in DMSO-d6

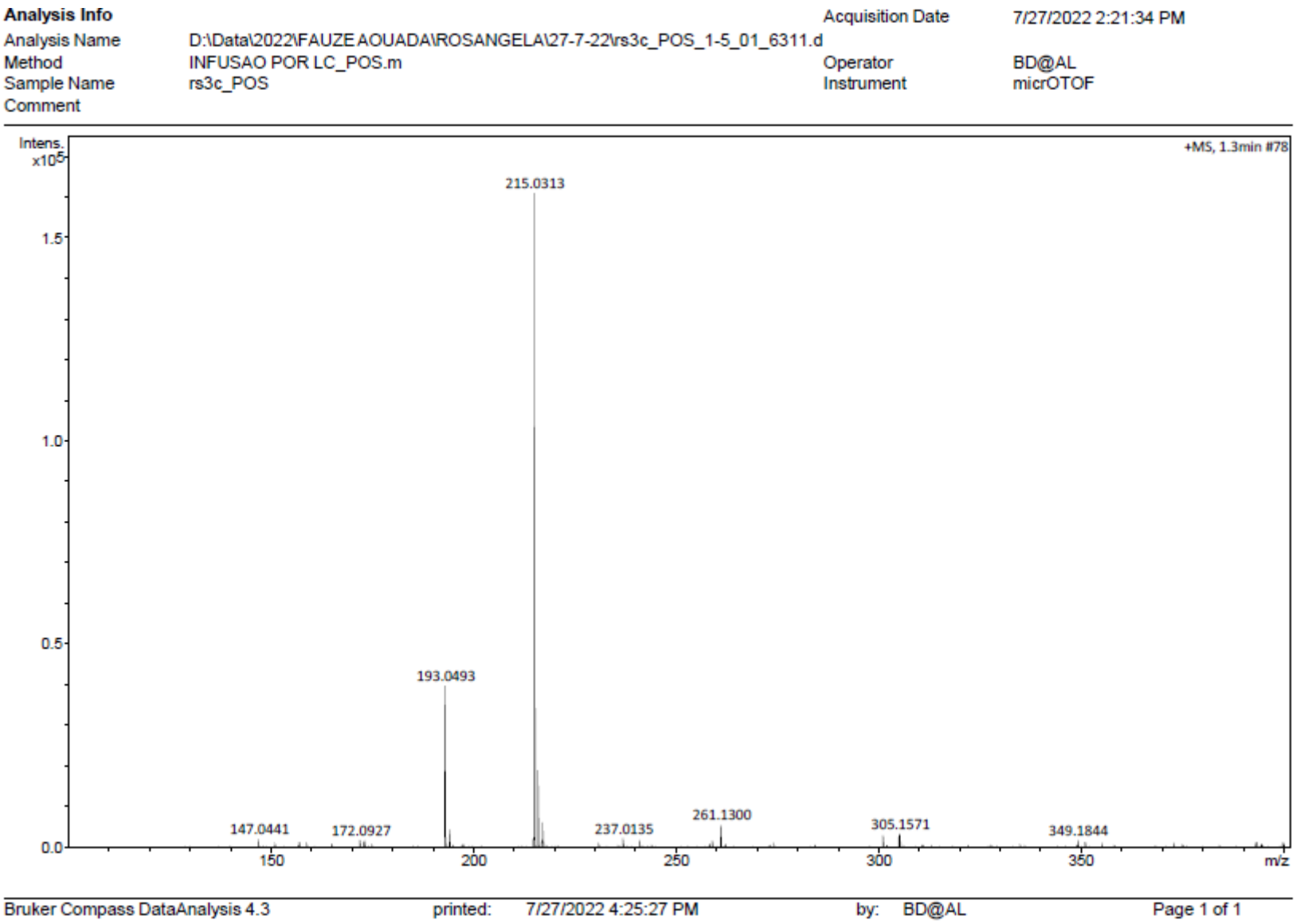

Figure 9S. HRMS spectrum of compound **3c**

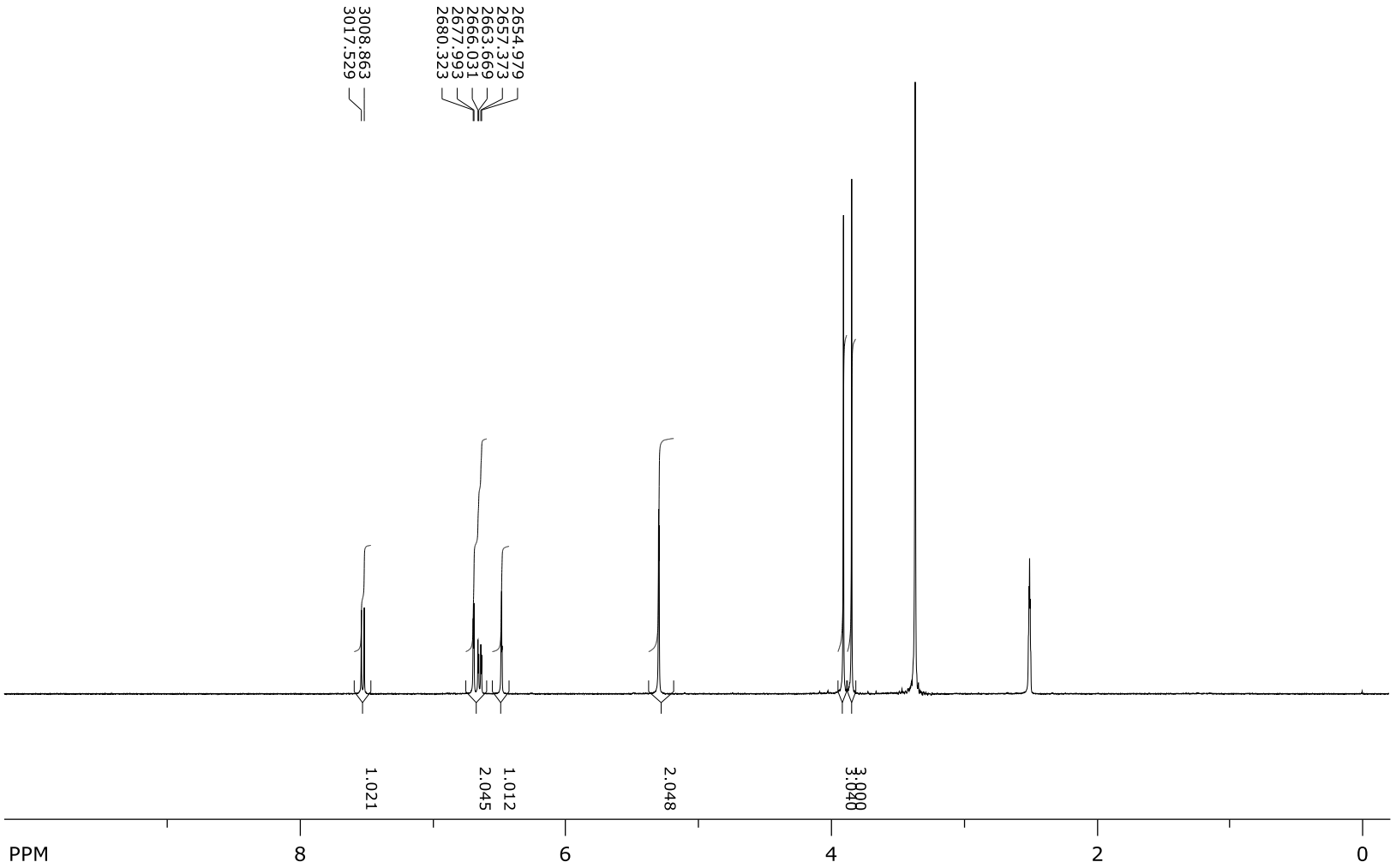

Figure 10S- ^{1}H NMR Spectrum of compound **3d** in DMSO-d6

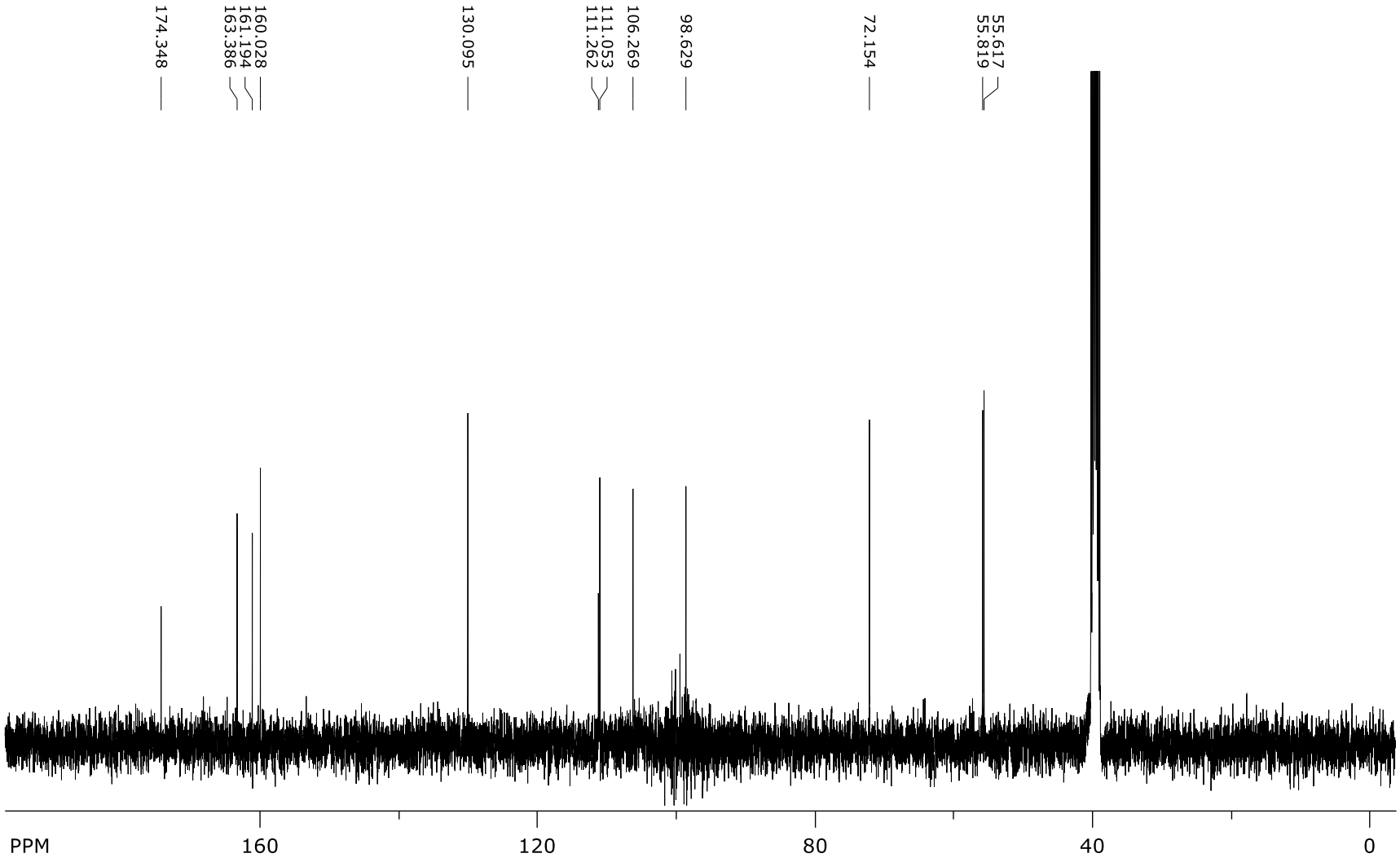

Figure 11S- ^{13}C NMR Spectrum of compound **3d** in DMSO-d6

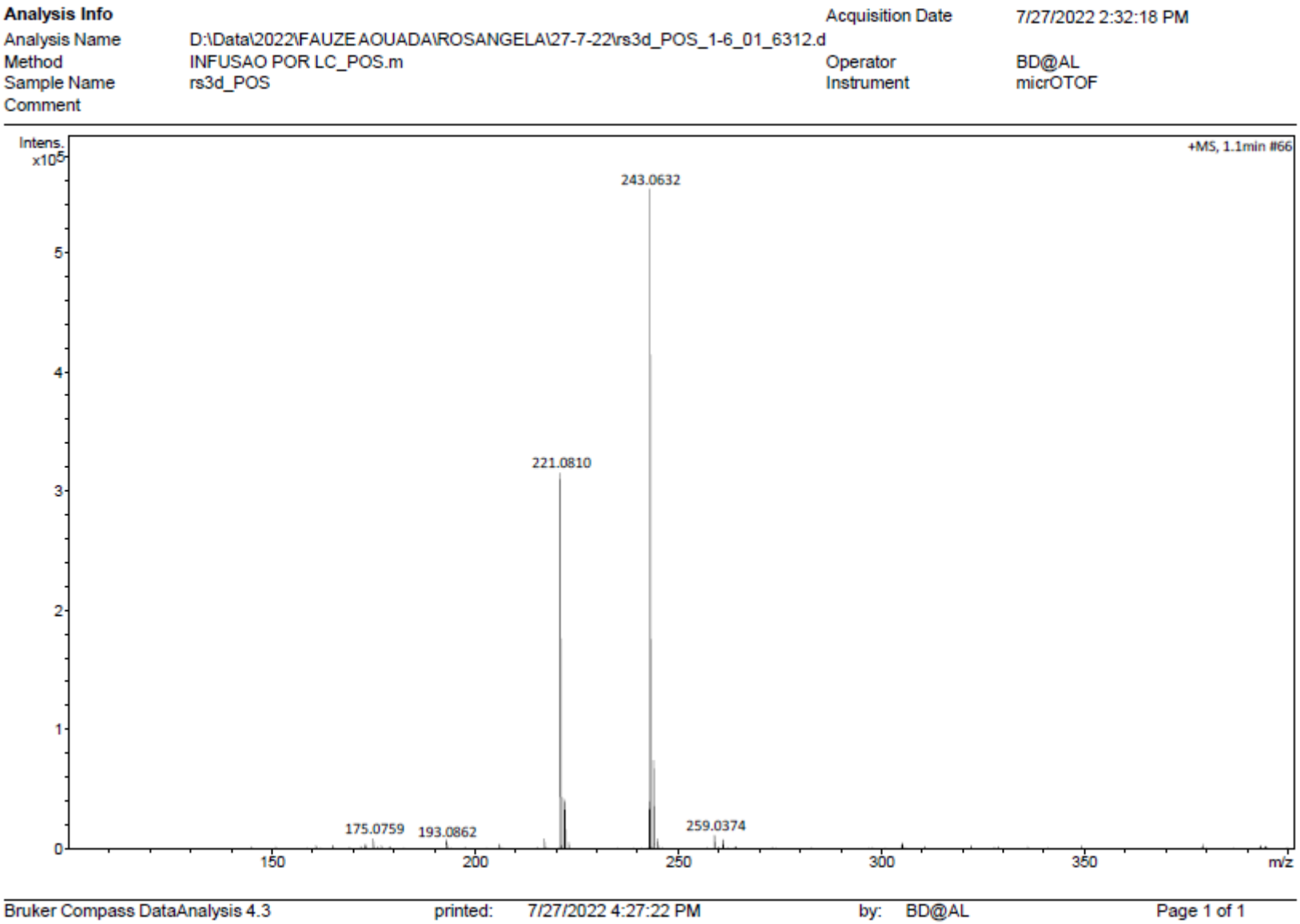

Figure 12S. HRMS spectrum of compound **3d**