Electronic Supporting Information

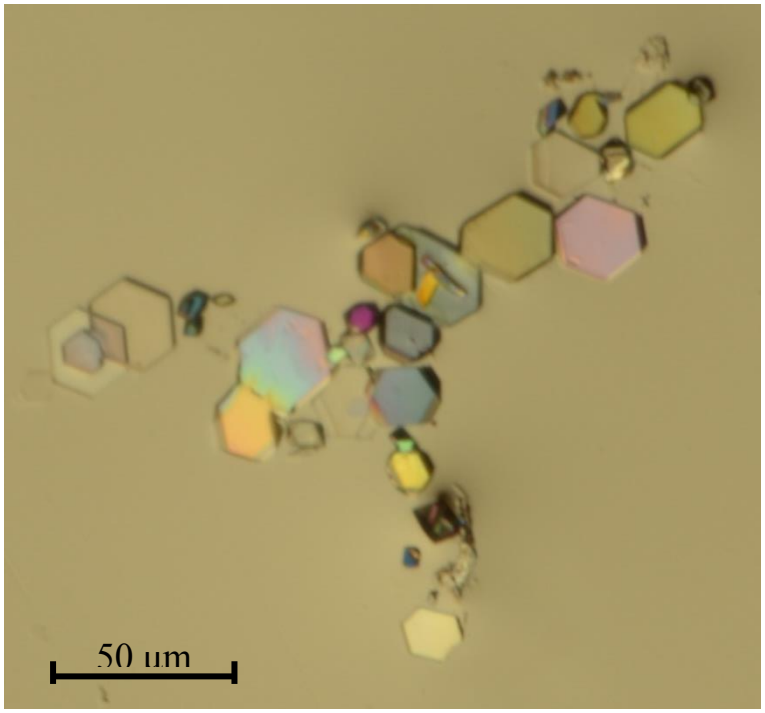

Figure S1. Photograph of microcrystalline material composing of **BU6**-2(**A1-**).

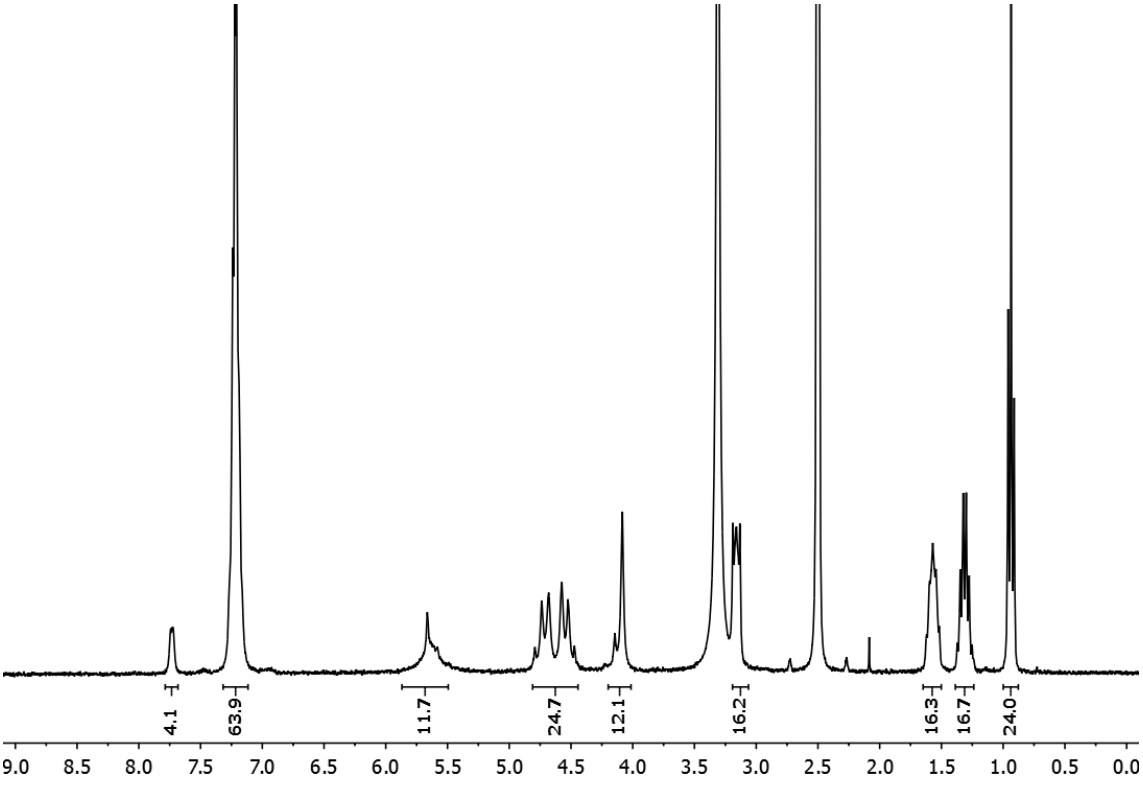

Figure S2. ^{1}H NMR spectra (300 MHz, [D6]DMSO, 30°C) of the solution of **BU6**-2($\mathbf{A_1}^-$) crystals.

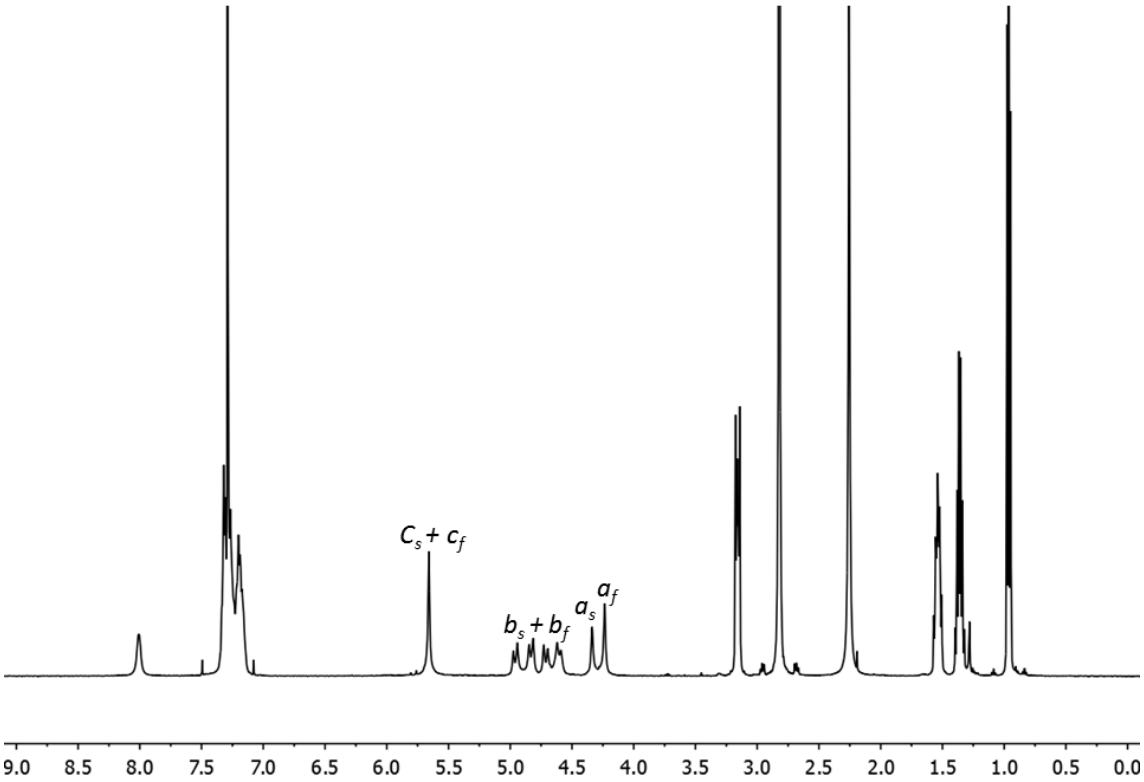

Figure S3. ^{1}H NMR spectra (300 MHz, $CDCl_3$, 30°C) solution remained after crystallization of **BU6**–2(**A1**$^-$) complex.

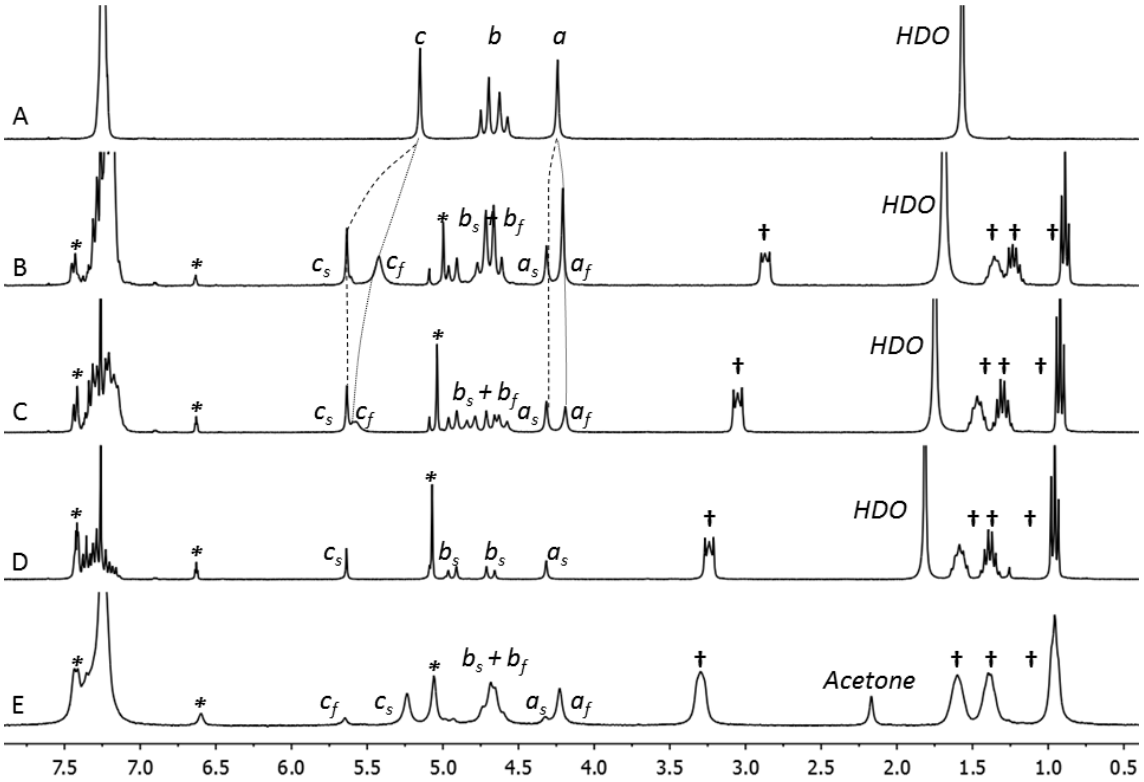

Figure S4. Full range ^{1}H NMR spectra (300 MHz, $CDCl_3$, 30°C) shown in **Figure 3** in the paper. * Signal of $\mathbf{A_2}^-$, † signal of TBA**.**

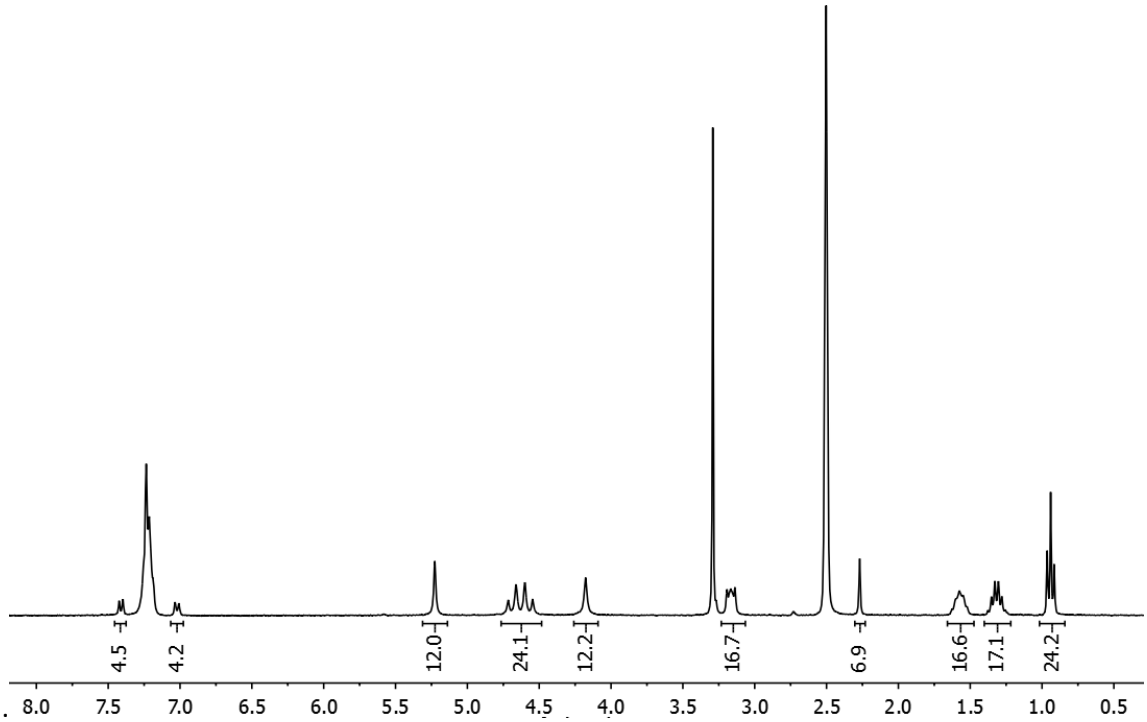

Figure S5. ^{1}H NMR spectra (300 MHz, [D6]DMSO, 30°C) of dissolved **BU6**-2(**A_3^-**) crystals.

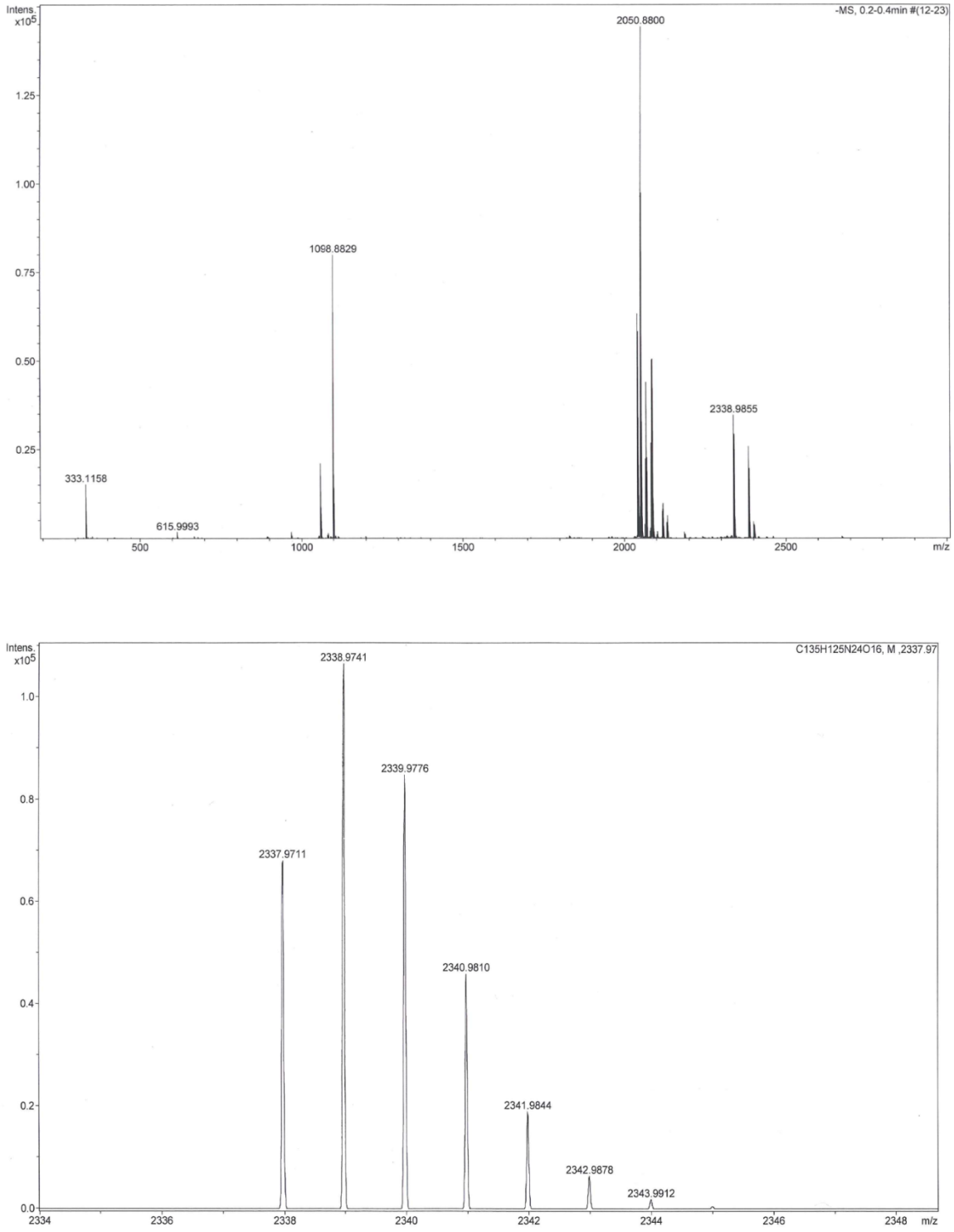

Figure S6. ESI-TOF MS spectra (negative mode) of **BU6** and $\mathbf{A_2}^-$. HRMS (ESI-TOF-): m/z calcd. for $[C_{135}H_{125}N_{24}O_{16}+H]^-$: 2338.9795, found: 2338.9741.

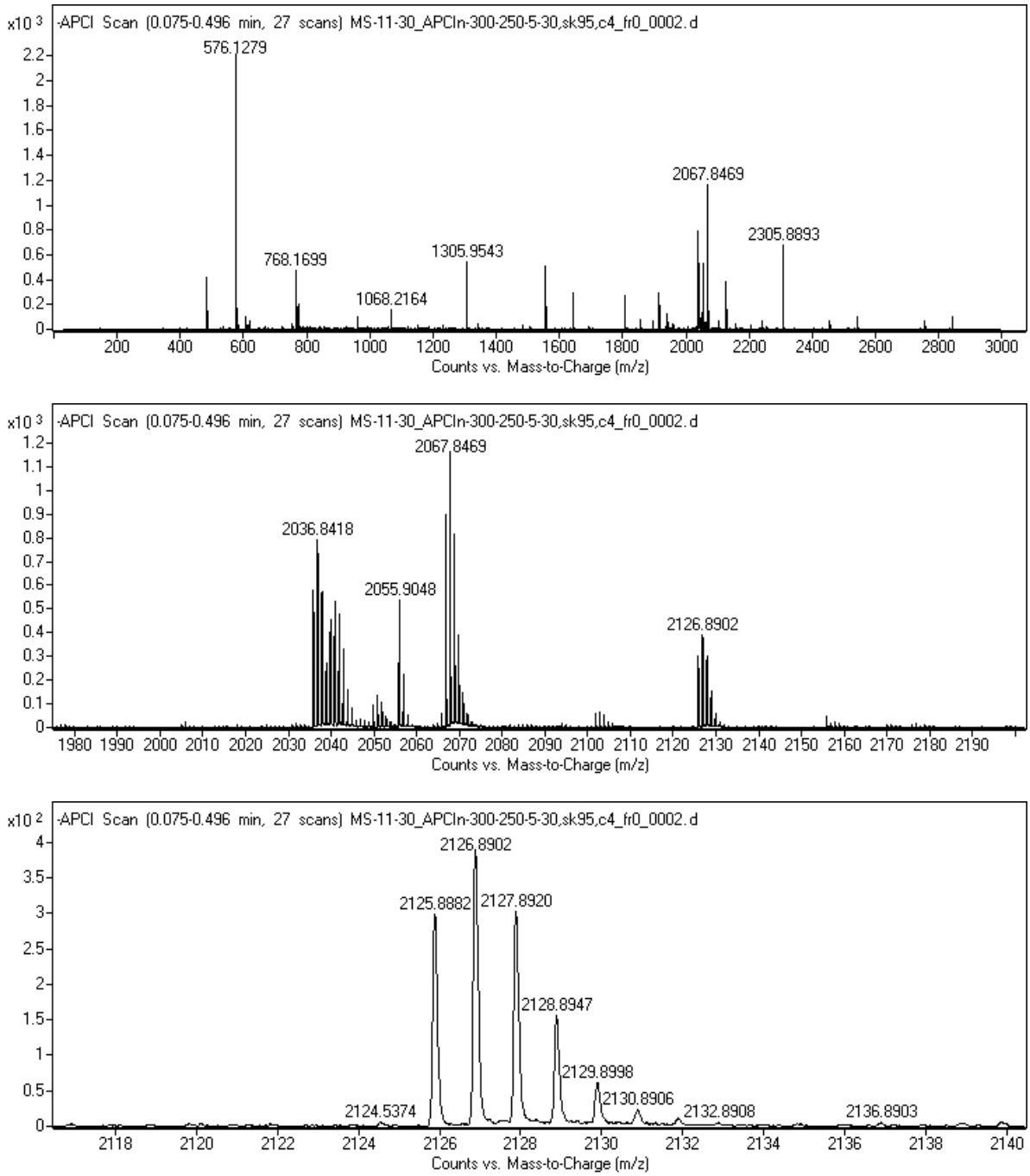

Figure S7. APCI MS spectra (negative mode) of **BU6** and $\mathbf{A_1}^-$. HRMS (APCI-): m/z calcd. for $[C_{121}H_{113}N_{24}O_{14}+H]^-$: 2126.8471, found: 2126.8902.

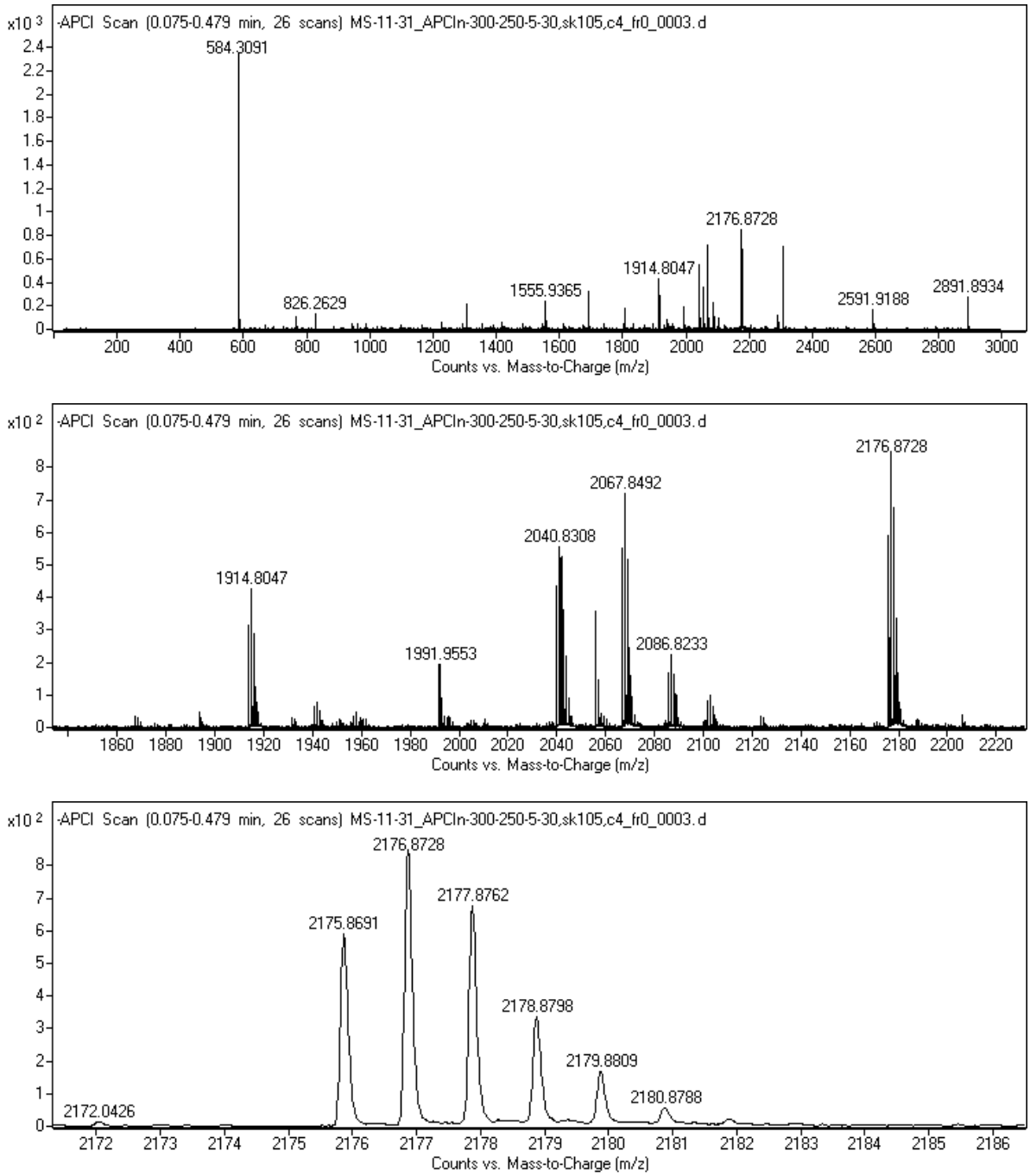

Figure S8. APCI MS spectra (negative mode) of **BU6** and **A3**$^{-}$. HRMS (APCI-): m/z calcd. for $[C_{121}H_{115}N_{24}O_{15}S+H]^{-}$: 2176.8784, found: 2176.8728.

Crystallographic data for **BU**6·2(**A1**$^{-}$TBA^{+})·2Et_2O·2H_2O ($C_{168}H_{214}N_{26}O_{20}$), M_r = 2917.65, crystal dimensions 0.40 x 0.40 x 0.30 mm, space group $P2_1/n$, a = 17.7270(4) Å, b = 20.2092(5) Å, c = 22.9150(6) Å, β = 107.271(3)°, V = 7839.1(3) Å^3, Z = 2, ρ_{calcd} = 1.236 Mg/m^3, μ = 0.082 mm^{-1}. X-ray intensity data were measured at 120 K on a Kuma KM-4 CCD diffractometer using Mo-Kα (λ = 0.71073 Å) radiation, and the structure was solved using direct method; 79854 reflections collected, 13765 unique reflections (R_{int} = 0.0509), data/restraints/parameters 13765/71/1005, final R indices (I>2σ(I)) *R*1 = 0.0433 and *wR*2 = 0.0955, $\Delta\rho_{max}$ /$\Delta\rho_{min}$ = 0.657/-0.420 e. Å^{-3}. CCDC 953267 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Crystallographic data for **BU6**·2(**A3**$^{-}$TBA^{+})·10$CHCl_3$·H_2O ($C_{170}H_{208}Cl_{30}N_{26}O_{20}S_2$), M_r = 4045.23, crystal dimensions 0.20 x 0.25 x 0.35 mm, space group $P2_1/n$, a = 21.105(2) Å, b = 19.2496(19) Å, c = 25.274(3) Å, β = 93.277(5)°, V = 10251(2) Å^3, Z = 2, ρ_{calcd} = 1.311 Mg/m^3, μ = 0.480 mm^{-1}. X-ray intensity data were measured at 120 K on a on a Rigaku MicroMax-007 HF rotating anode four-circle diffractometer using Mo-Kα (λ = 0.71075 Å) radiation, and the structure was solved using direct method; 141993 reflections collected, 18024 unique reflections (R_{int} = 0.0564), data/restraints/parameters 18024/103/1197, final R indices (I>2σ(I)) R1 = 0.1106 and wR2 = 0.3198, Δρmax /Δρmin = 2.324/-1.223 e. Å^{-3}. CCDC 965535 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.