

Supporting Information

Sulfur(VI) Fluoride Exchange Chemistry in Solid-Phase Synthesis of Compound Arrays: Discovery of Histone Deacetylase Inhibitors

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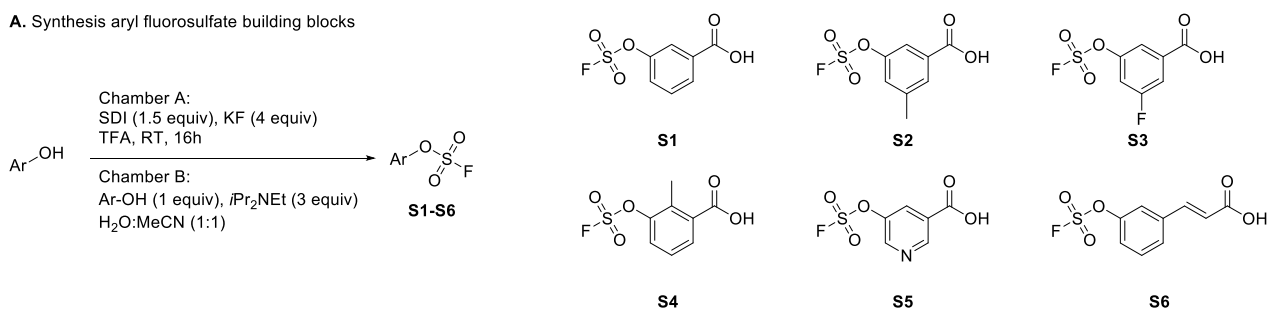
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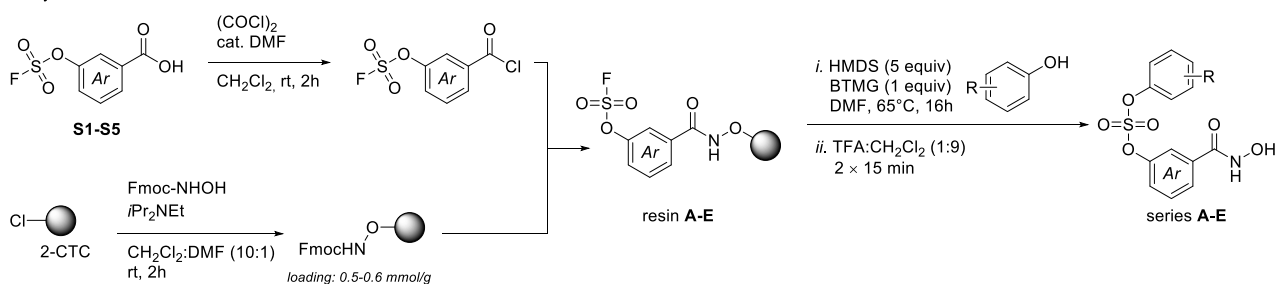
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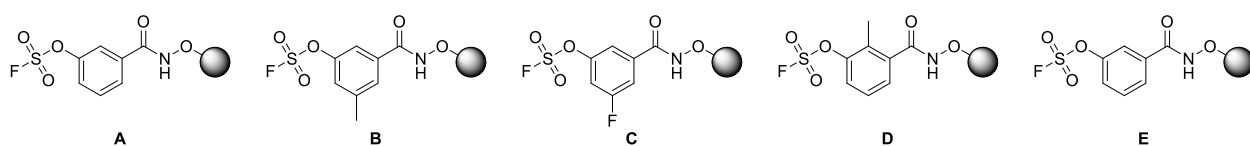
A. Synthesis aryl fluorosulfate building blocks



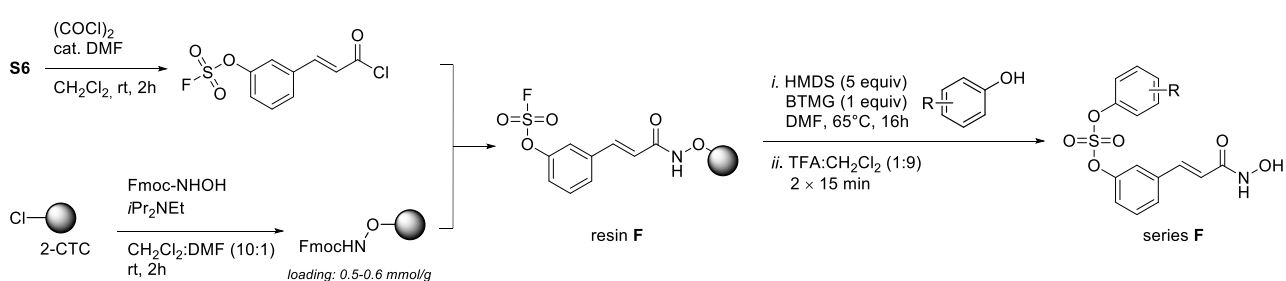
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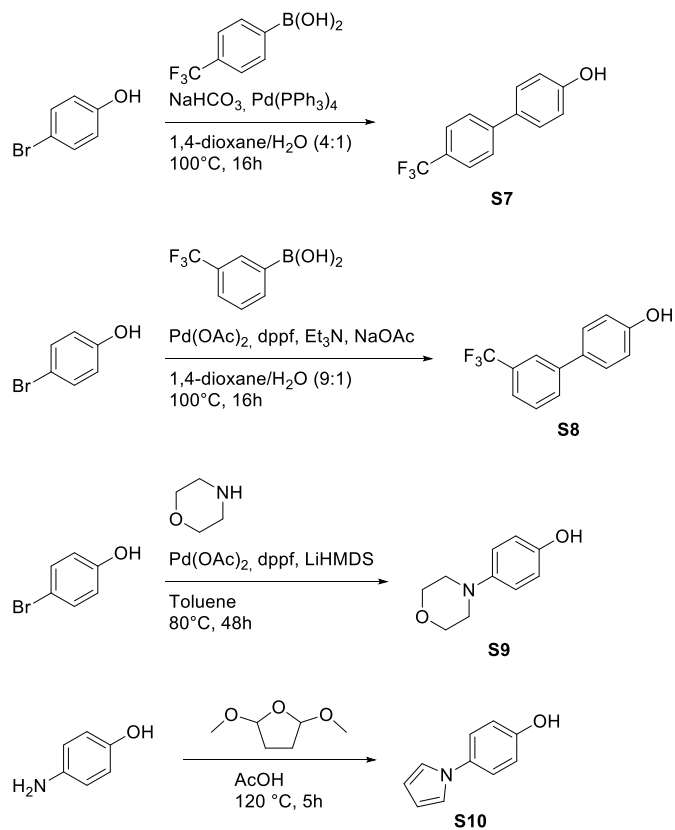
C. Structures of resins A-E



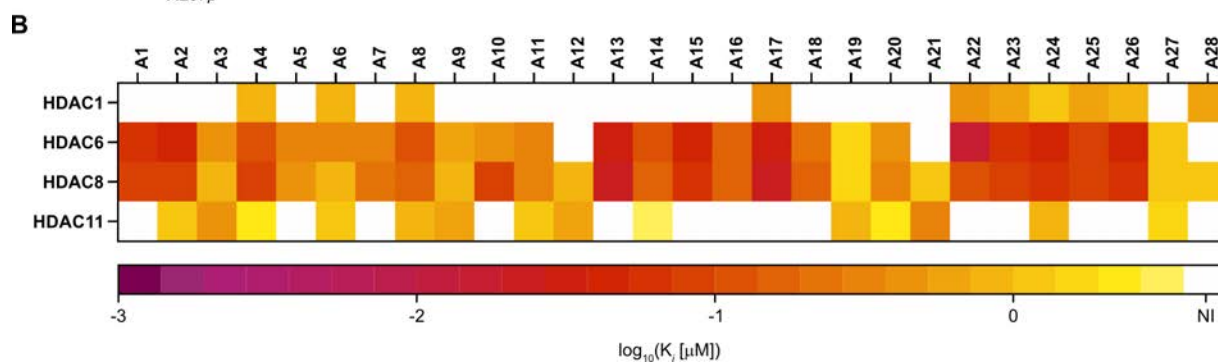
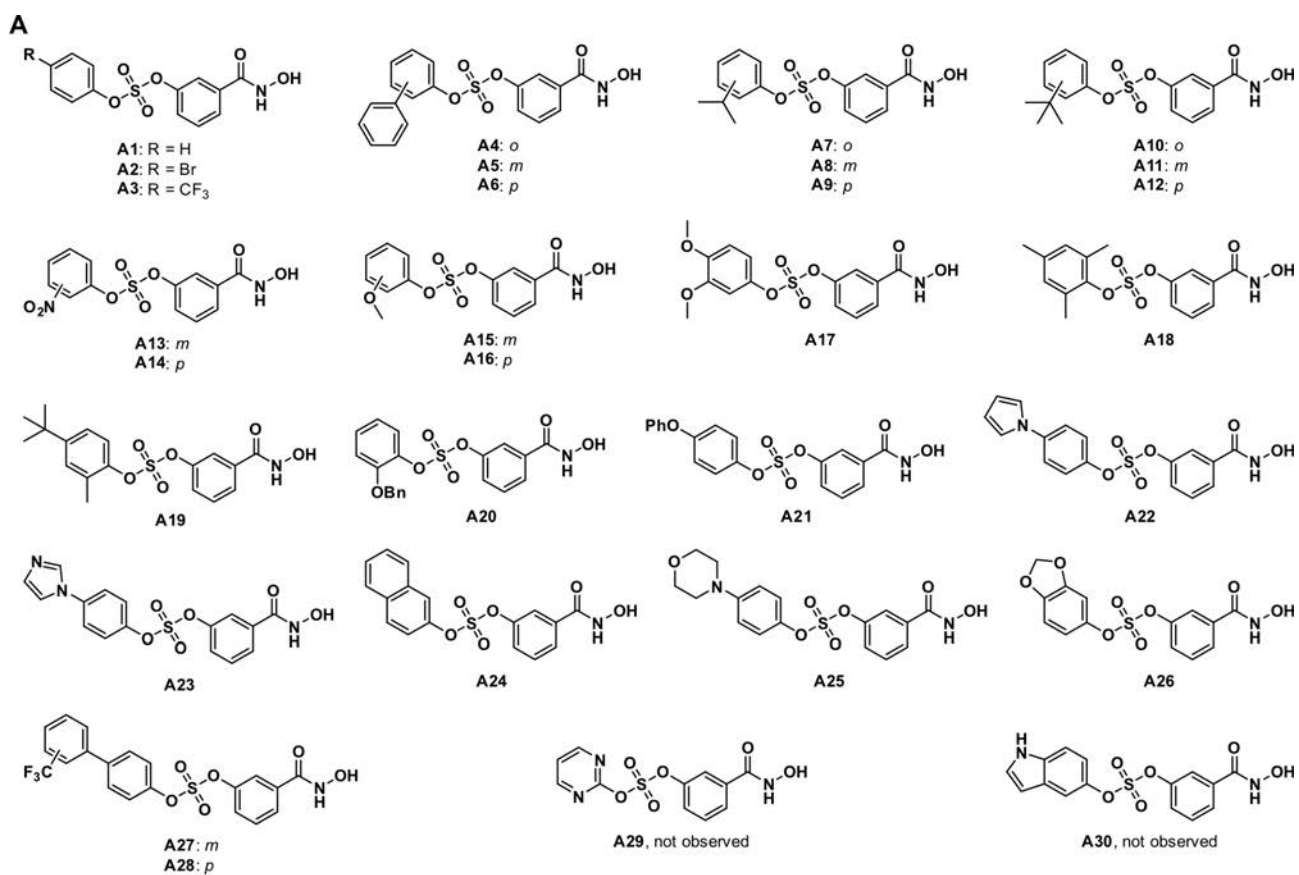
D. Synthesis of resin F and series F



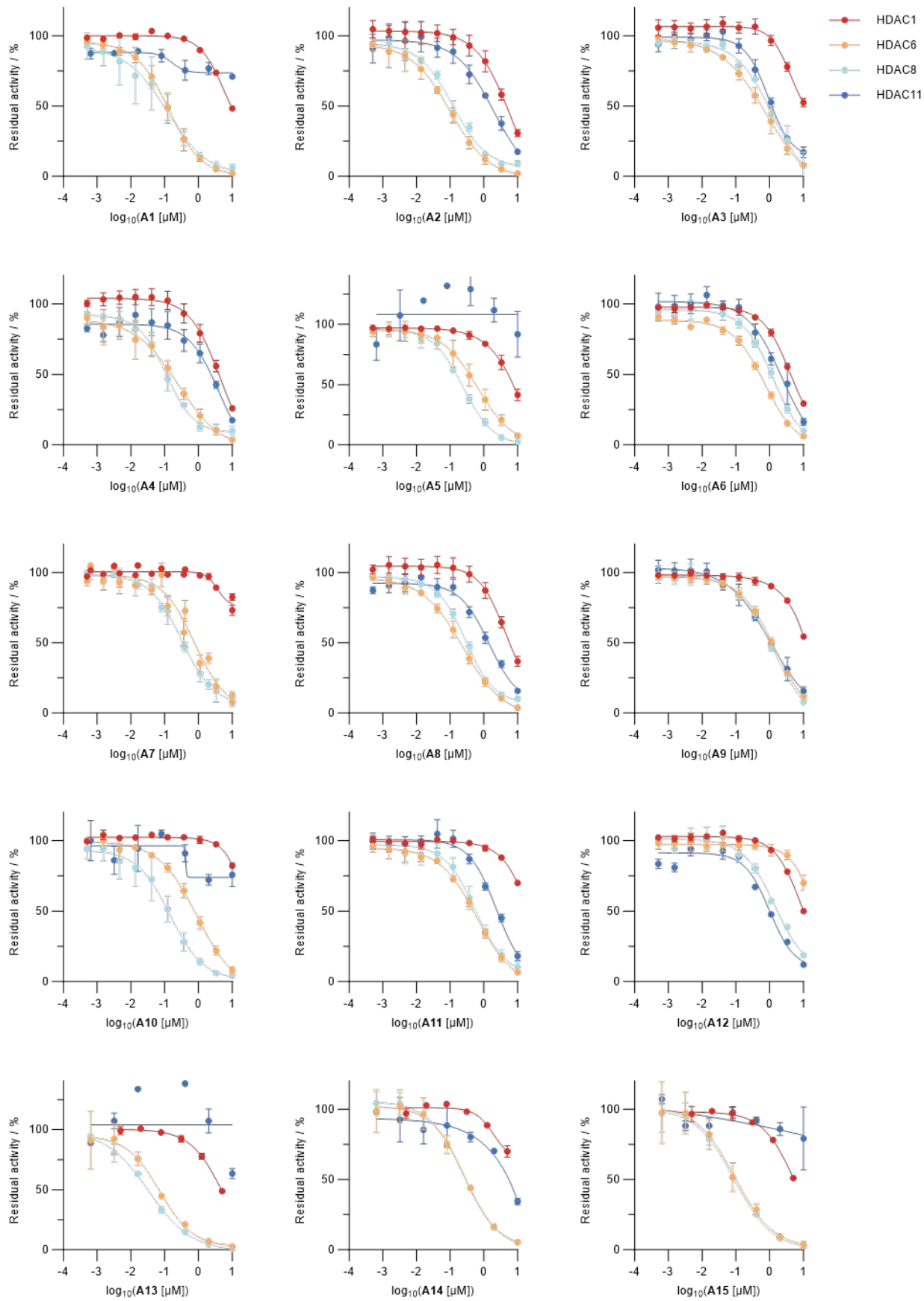
Supplementary Fig S1. A) Synthesis of aryl fluorosulfate building blocks **S1-S6**. B) Preparation of resins **A-E** and synthesis of series **A-E**. C) Structures of resins **A-E**. D) Preparation of resin **F** and synthesis of series **F**.



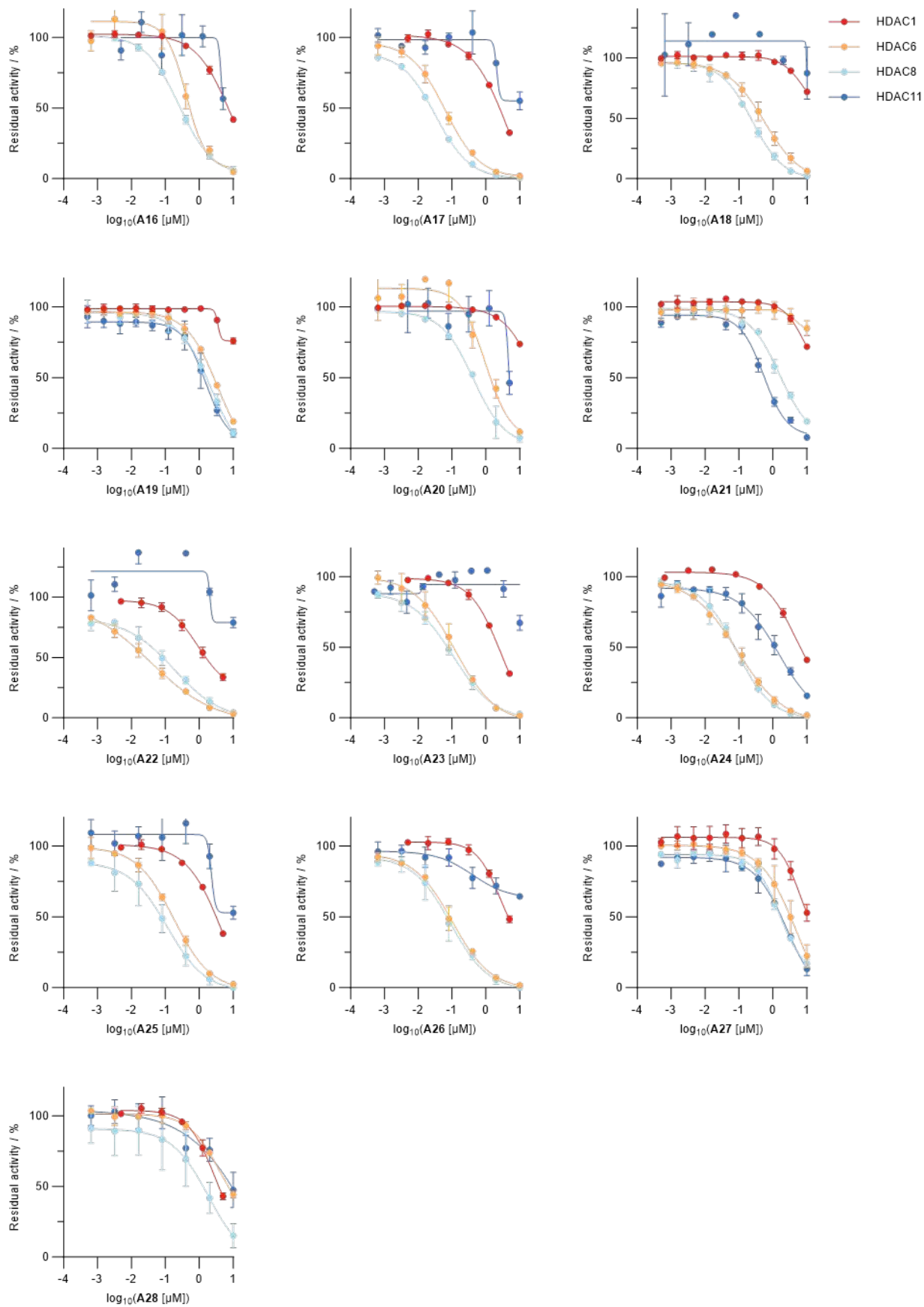
Supplementary Fig S2. Synthesis of phenols S7, S8, S9, and S10.



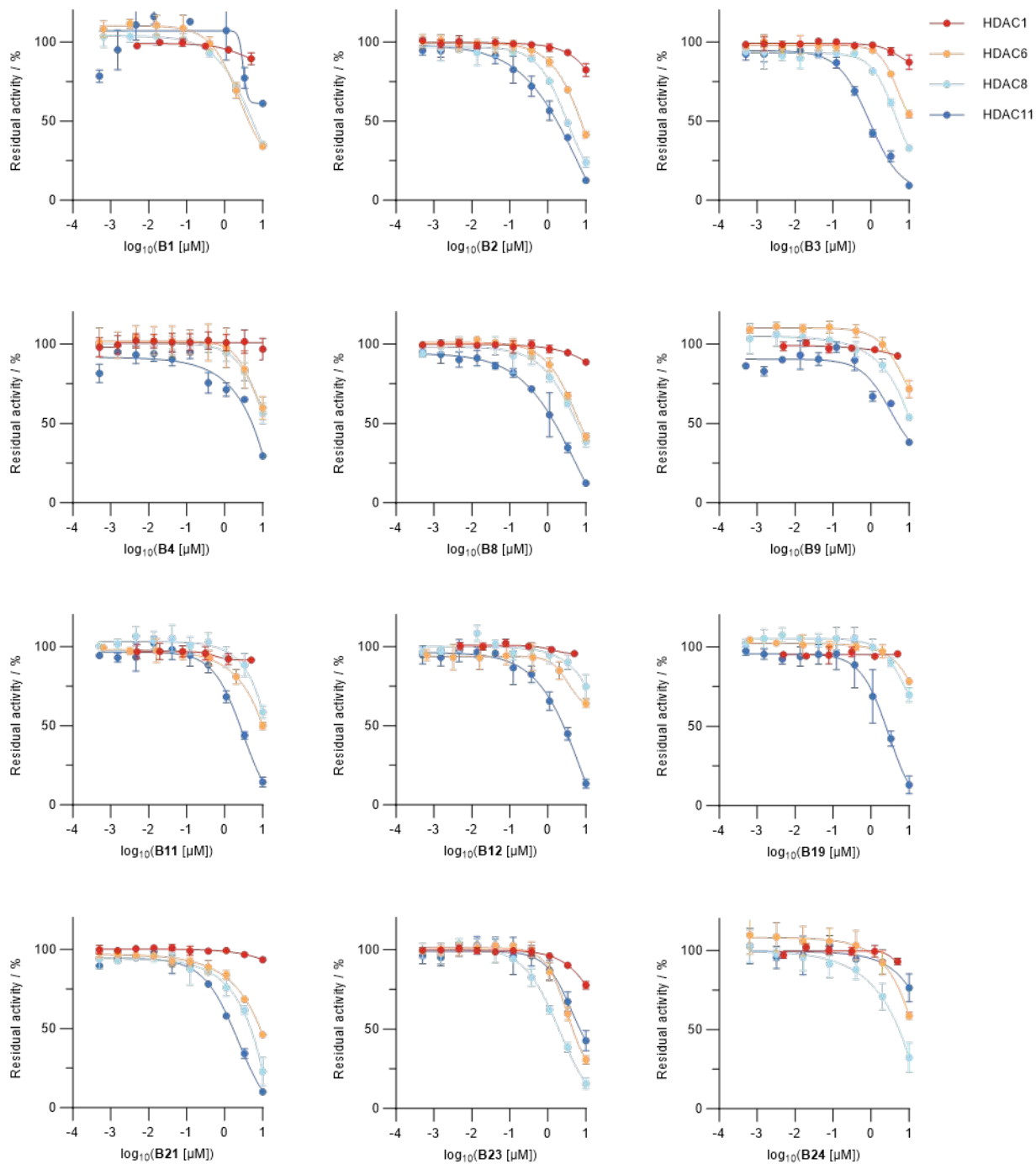
Supplementary Fig S3. Structures of compound series **A** and heatmap representing the obtained K_i values.



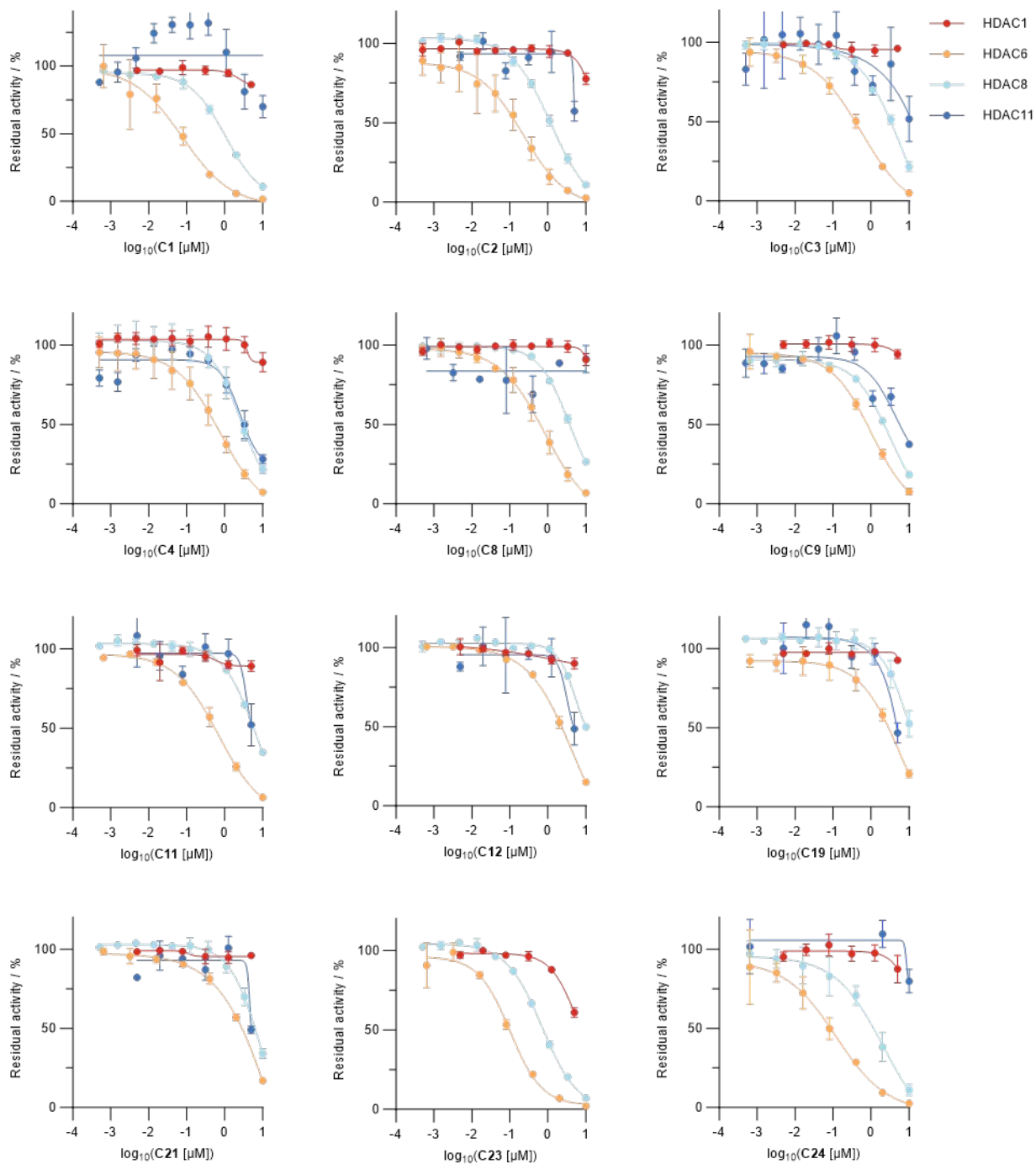
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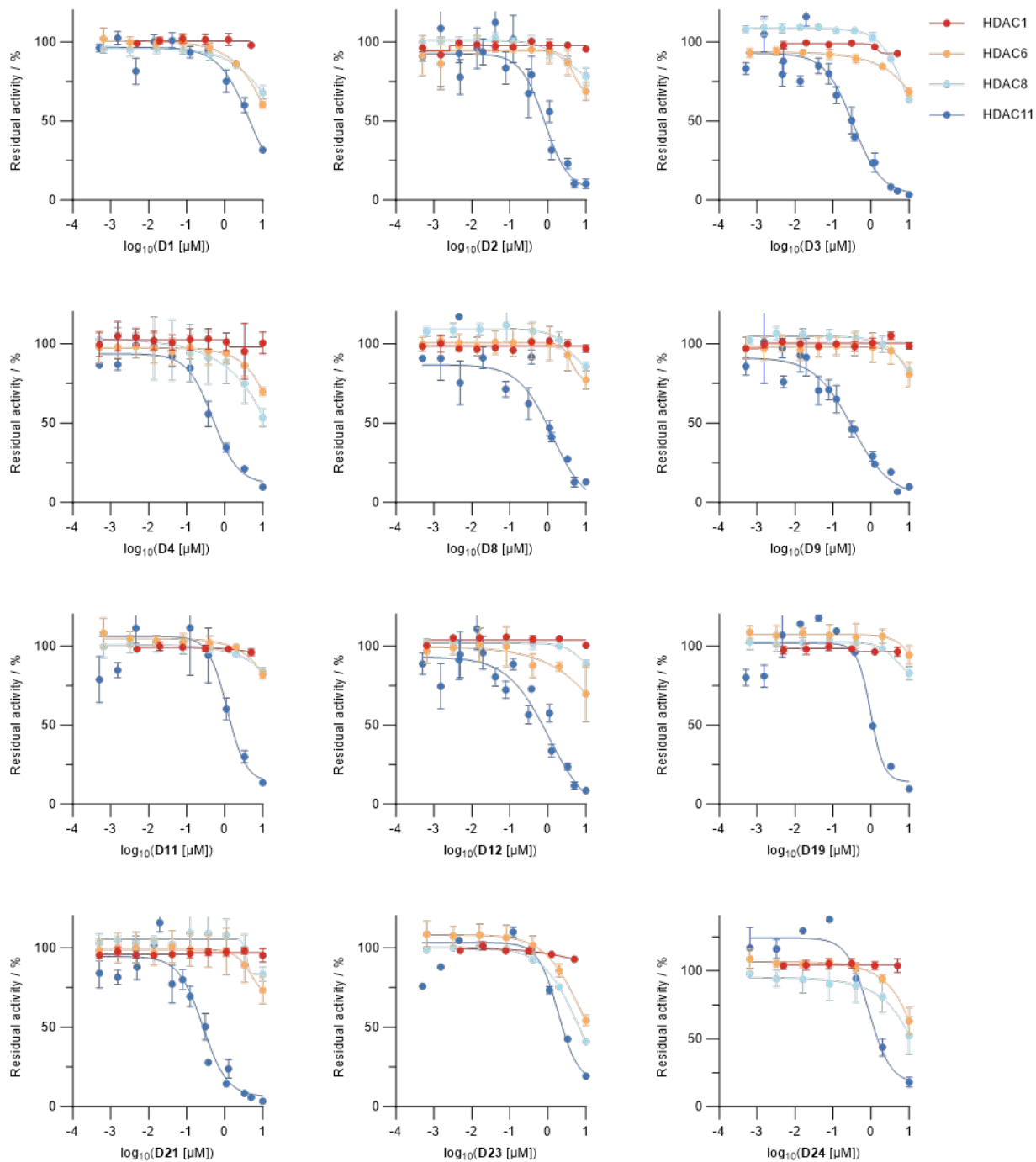
Supplementary Fig S4 continued. Full dose–response curves of compound series A.



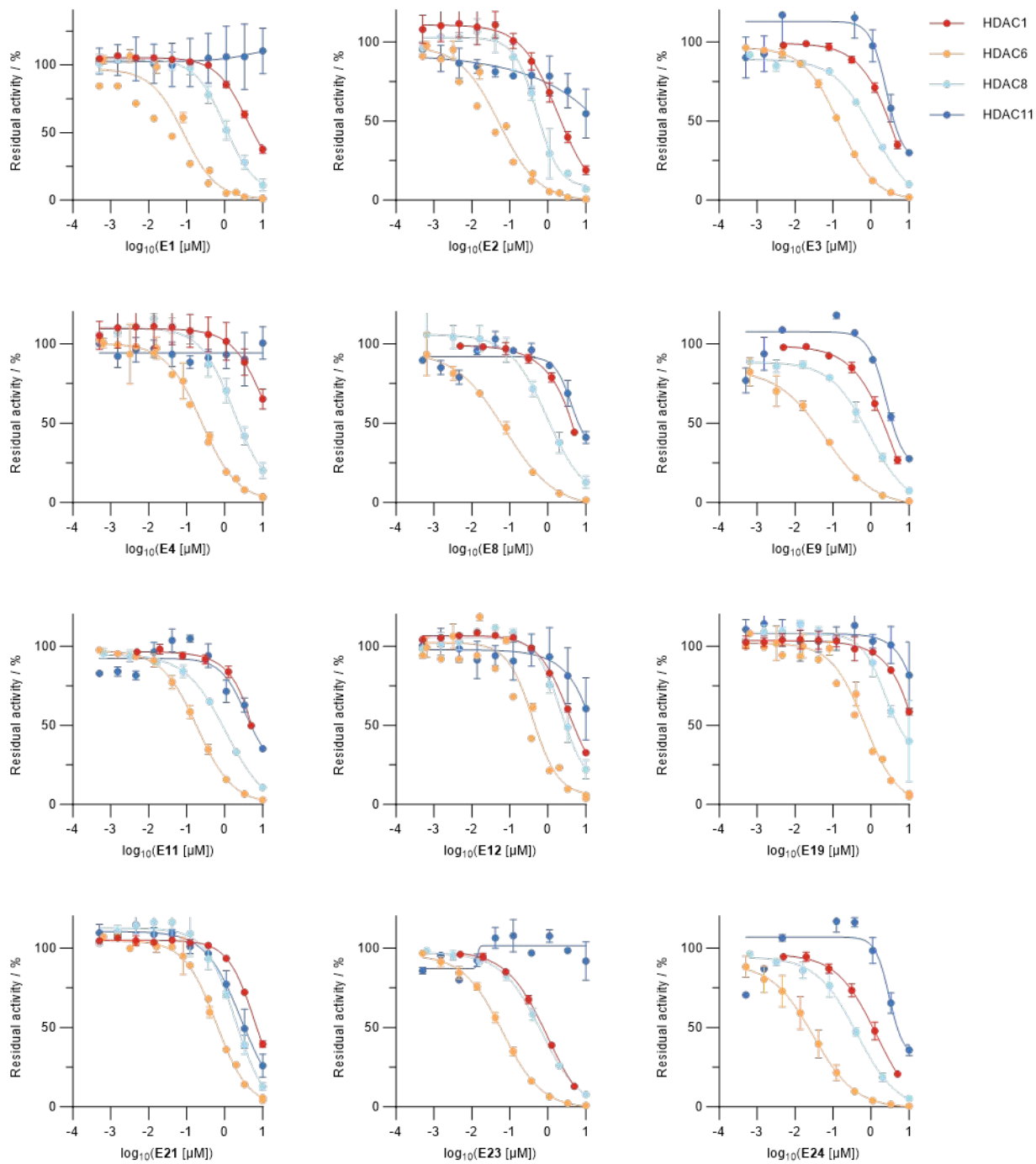
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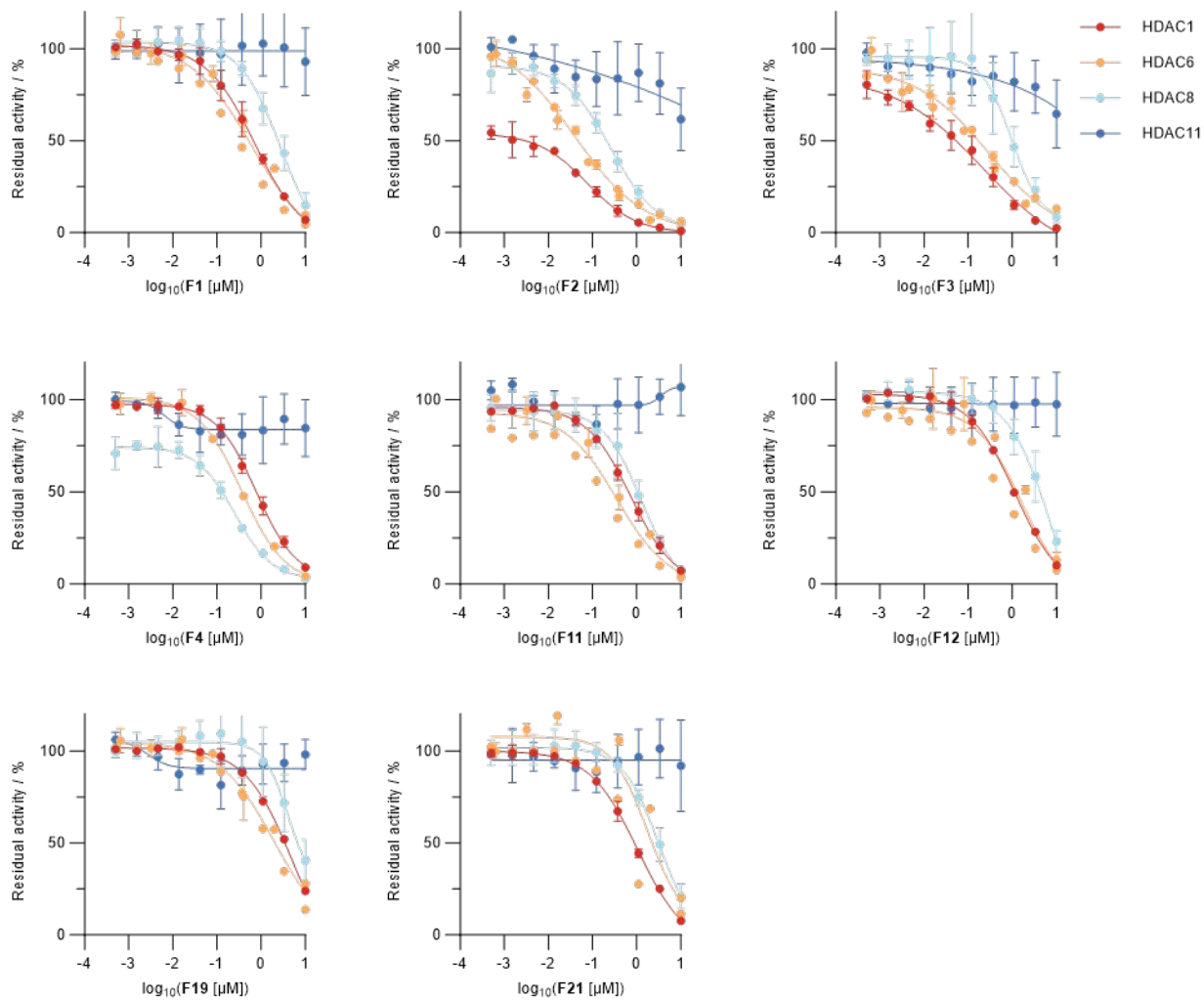
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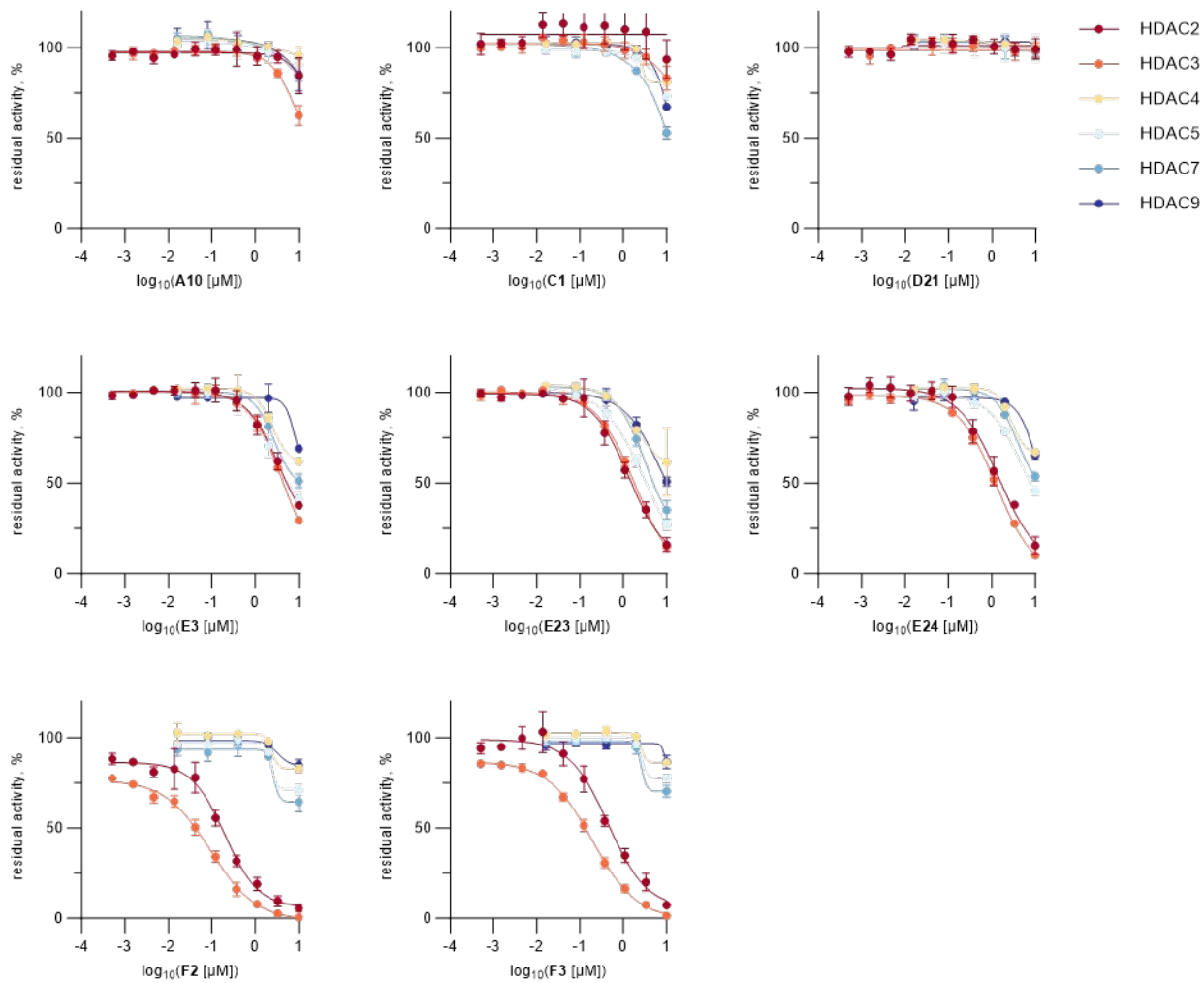
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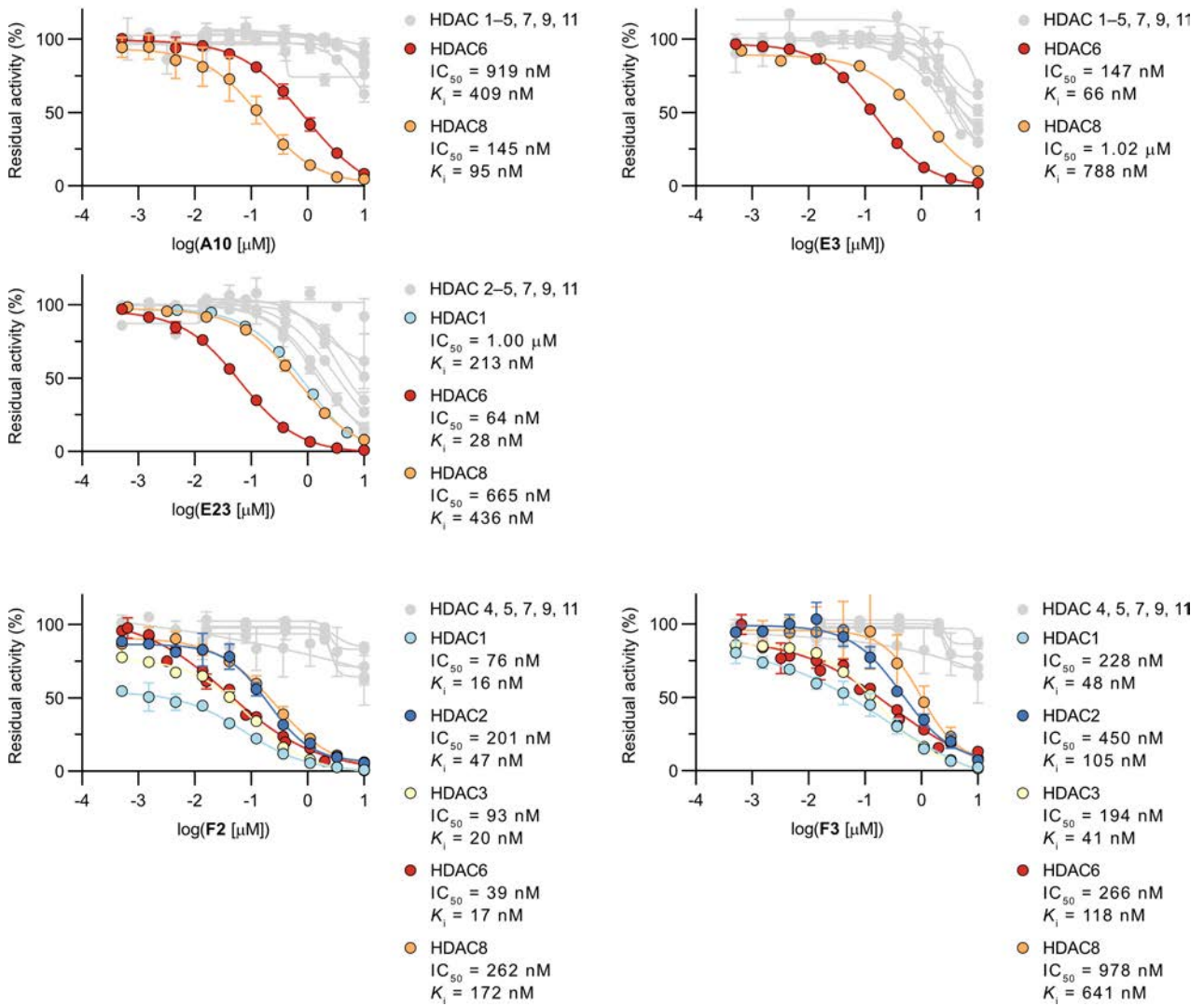
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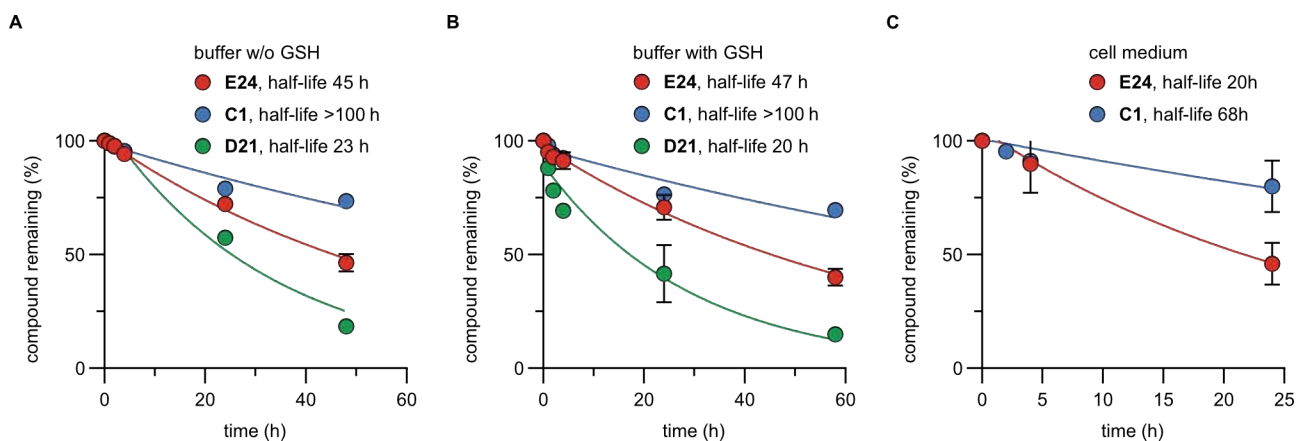
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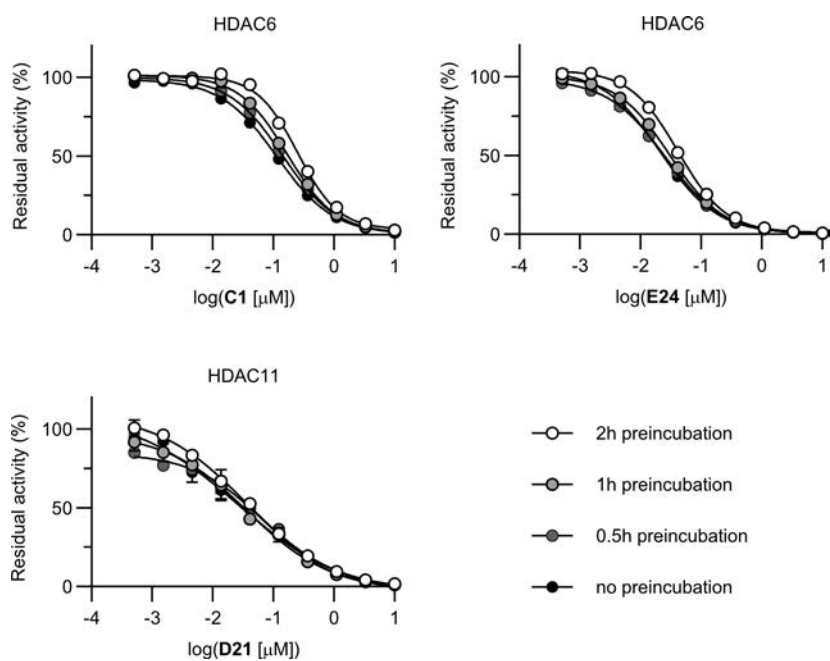
Supplementary Fig S10. Full dose–response curves of compounds **A10**, **C1**, **D21**, **E3**, **E23**, **E24**, **F2**, and **F3** against HDACs 2, 3, 4, 5, 7, and 9.



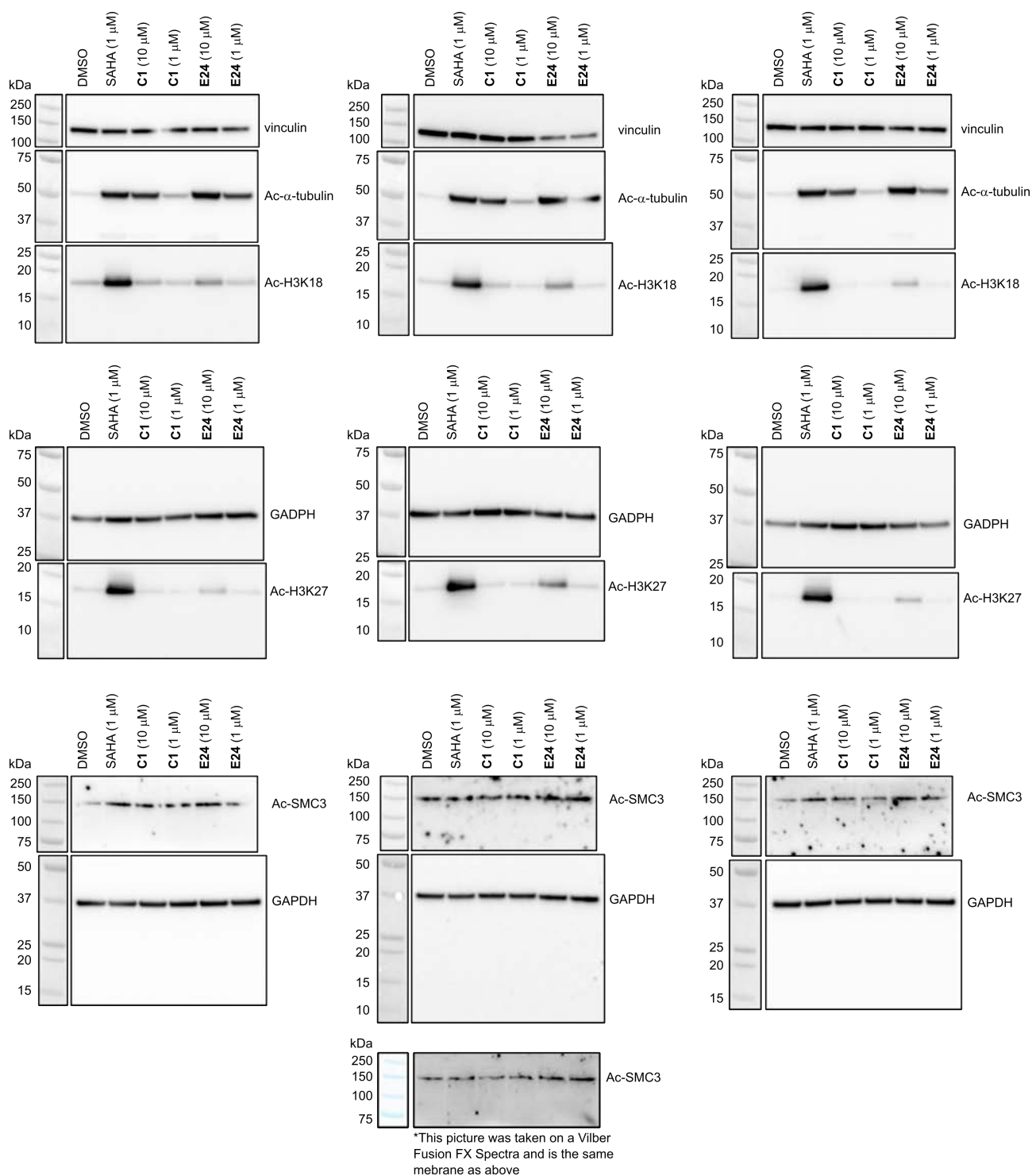
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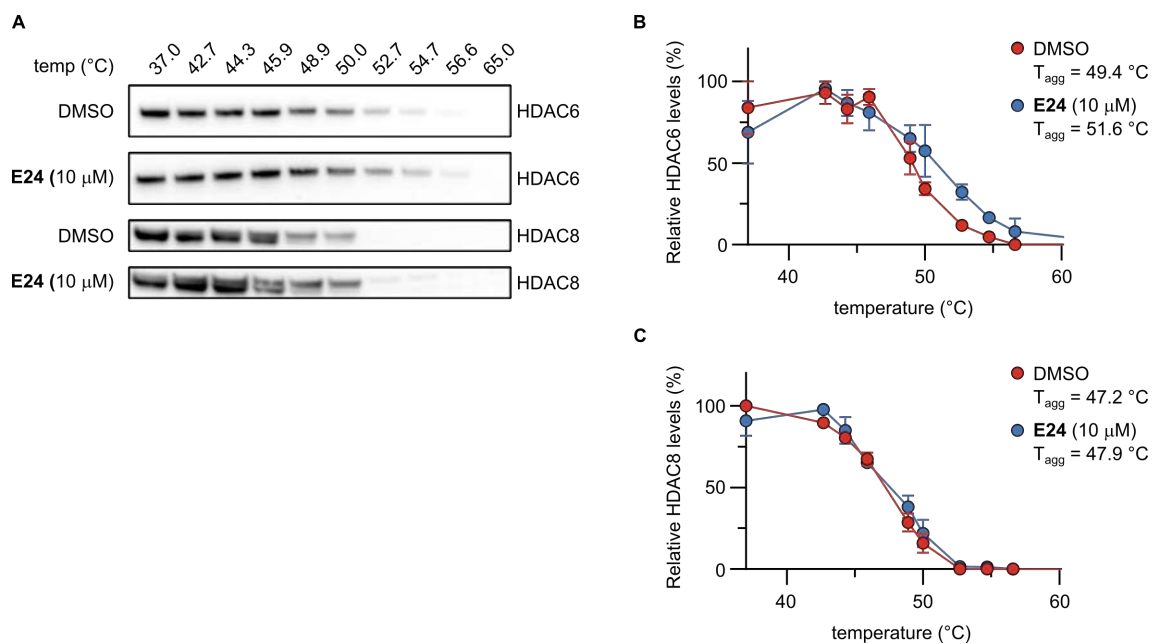
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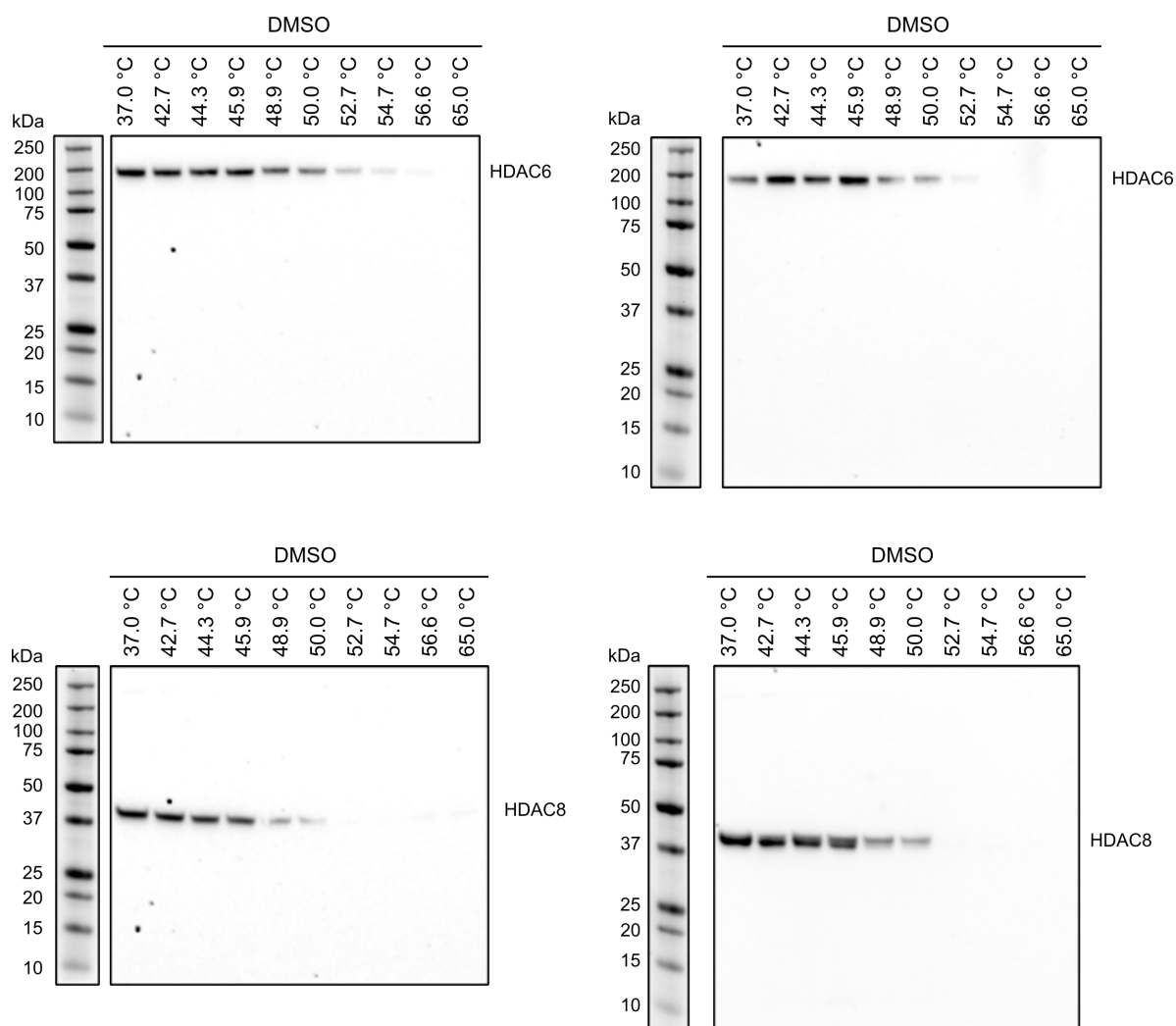
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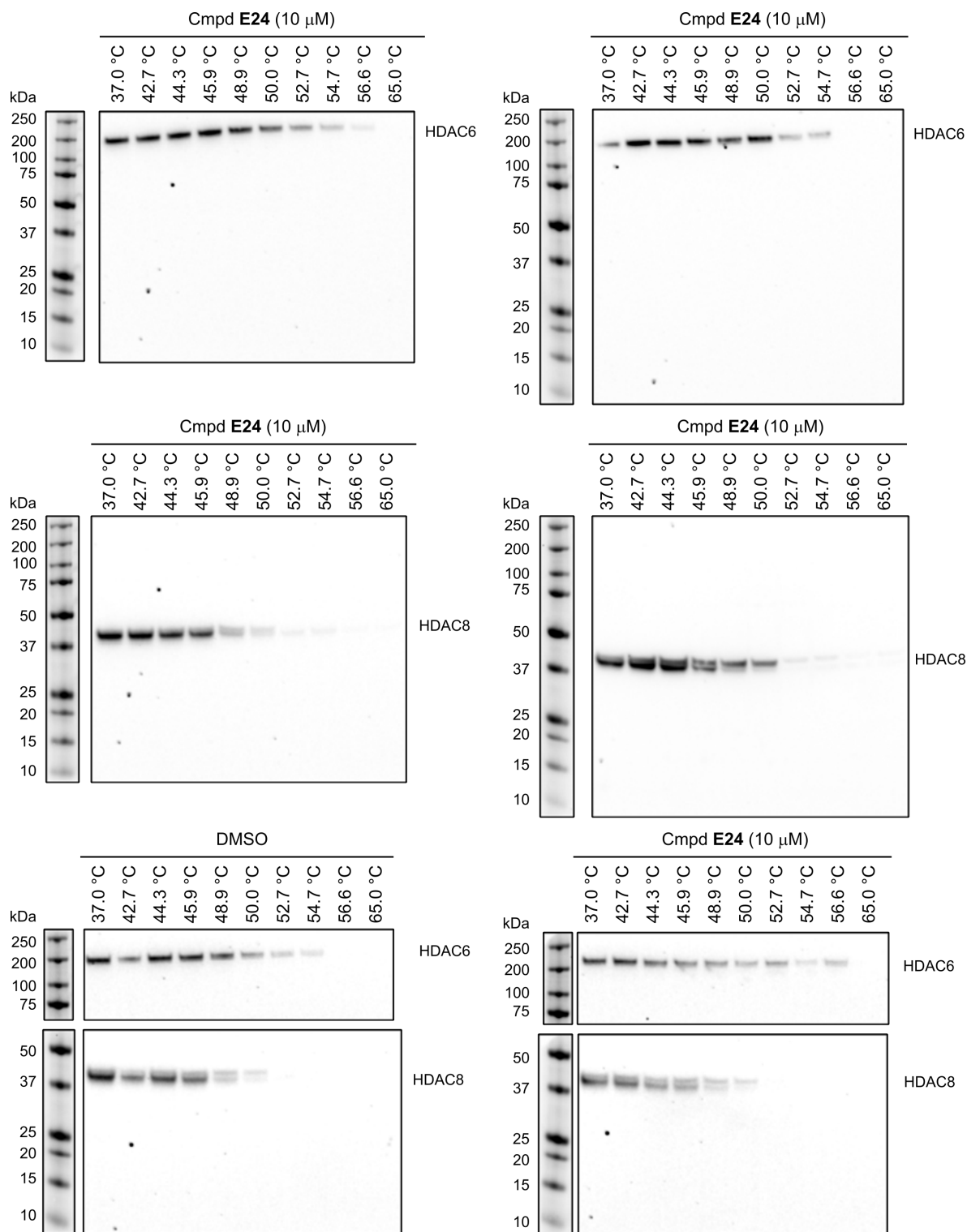
Supplementary Fig S14. Full immunoblots for all replicates ($n = 3$) of the acetylation experiment. Immunoblots for the acetylation of α -tubulin, SMC3, H3K18, and H3K27 after treatment with inhibitor **C1** (1 or 10 μM), **E24** (1 or 10 μM), SAHA or vehicle (1% DMSO) for 5 h.



Supplementary Fig S15. Cellular target engagement of **E24**. A) Representative immunoblots for the thermal shift of HDAC6 and HDAC8 in HEK293T cells after 2 h of treatment with **E24** (10 μM) or DMSO (1%) and following heat treatment (from 37–65 °C). B) Plots of the quantified data and calculated T_{agg} values (n = 3; see Supplementary Figure S16 for full blots and replicates). Images were quantified in ImageJ and T_{agg} values were calculated by fitting data to a dose–response curve with variable slope: $\text{relative HDAC level} = \text{Bottom} + (\text{Top} - \text{Bottom}) \times (1 + (T_{\text{agg}} \times \text{temp}^{-1})^h)^{-1}$. Note, we did not achieve statistical significance on the ΔT_{agg} value, which may have required additional replicates.



Supplementary Fig S16. Full immunoblots for all replicates of the CETSA experiment. Immunoblots for the thermal shift of HDAC6 and 8 in HEK293T cells after treatment with inhibitor **E24** (10 μ M) or vehicle (1% DMSO) at temperatures ranging from 37-65 °C.



Supplementary Fig S16 continued. Full immunoblots for all replicates of the CETSA experiment. Immunoblots for the thermal shift of HDAC6 and 8 in HEK293T cells after treatment with inhibitor **E24** (10 μM) or vehicle (1% DMSO) at temperatures ranging from 37-65 °C.

Table S1. IC₅₀ values and estimated K_i values against indicated HDAC enzymes.

Compound	HDAC1 LGKac		HDAC6 LGKac		HDAC8 LGKtfa		HDAC11 ETDKmyr	
	IC ₅₀ (μM) or inhibition	K _i (μM)	IC ₅₀ (μM) or inhibition	K _i (μM)	IC ₅₀ (μM) or inhibition	K _i (μM)	IC ₅₀ (μM) or inhibition	K _i (μM)
A1	52 % [10 μM]		0.132	0.059	0.118	0.077	29 % [10 μM]	
A2	6.297	1.339	0.099	0.044	0.122	0.080	1.907	1.056
A3	47 % [10 μM]		0.842	0.374	1.221	0.800	0.923	0.511
A4	4.449	0.946	0.253	0.113	0.111	0.073	4.003	2.216
A5	59 % [10 μM]		0.665	0.295	0.718	0.470	8 % [10 μM]	
A6	4.140	0.880	0.716	0.318	1.261	0.826	2.342	1.296
A7	22 % [10 μM]		0.755	0.335	0.317	0.208	9 % [500 nM]	
A8	4.340	0.923	0.240	0.107	0.290	0.190	1.433	0.793
A9	46 % [10 μM]		1.409	0.626	1.266	0.829	1.069	0.592
A10	18 % [10 μM]		0.919	0.409	0.145	0.095	24 % [10 μM]	
A11	30 % [10 μM]		0.620	0.276	0.557	0.365	2.393	1.325
A12	50 % [10 μM]		30 % [10 μM]		1.405	0.921	0.995	0.551
A13	51 % [5 μM]		0.072	0.032	0.034	0.022	37 % [10 μM]	
A14	30 % [5 μM]		0.269	0.119	0.259	0.169	6.090	3.371
A15	49% [5 μM]		0.092	0.041	0.088	0.058	21 % [10 μM]	
A16	8.338	1.773	0.413	0.183	0.255	0.167	43 % [5 μM]	
A17	2.372	0.504	0.063	0.028	0.033	0.022	45 % [10 μM]	
A18	28 % [10 μM]		0.557	0.248	0.244	0.160	13 % [10 μM]	
A19	24 % [10 μM]		3.577	1.590	2.255	1.477	1.556	0.861
A20	26 % [10 μM]		0.997	0.443	0.427	0.280	4.821	2.669
A21	28 % [10 μM]		15 % [10 μM]		1.593	1.044	0.538	0.298
A22	1.792	0.381	0.037	0.017	0.181	0.118	21 % [10 μM]	
A23	2.734	0.581	0.121	0.054	0.110	0.072	33 % [10 μM]	
A24	4.793	1.019	0.091	0.040	0.083	0.055	1.490	0.825
A25	3.023	0.643	0.178	0.079	0.114	0.075	47 % [10 μM]	
A26	4.384		0.102	0.045	0.091	0.060	35 % [10 μM]	
A27	47 % [10 μM]		3.100	1.378	1.581	1.036	2.567	1.421
A28	2.844	0.605	7.118	3.164	1.876	1.229	52 % [10 μM]	
B1	10 % [5 μM]		3.828	1.701	6.088	3.989	39 % [10 μM]	
B2	18 % [10 μM]		7.341	3.263	3.176	2.081	1.619	0.896
B3	13 % [10 μM]		45 % [10 μM]		4.517	2.959	0.910	0.504
B4	NI [10 μM]		40 % [10 μM]		13.480	8.832	5.772	3.195
B8	11 % [10 μM]		58 % [10 μM]		6.259	4.101	1.651	0.914
B9	7 % [5 μM]		28 % [10 μM]		46 % [10 μM]		6.939	3.841
B11	8 % [5 μM]		50 % [10 μM]		41 % [10 μM]		3.065	1.697
B12	NI [5 μM]		36 % [10 μM]		25 % [10 μM]		2.495	1.381
B19	NI [5 μM]		22 % [10 μM]		30 % [10 μM]		2.915	1.614
B21	6 % [10 μM]		54 % [10 μM]		4.707	3.084	2.287	1.266
B23	22 % [10 μM]		4.753	2.112	2.046	1.340	57 % [10 μM]	
B24	7 % [5 μM]		41 % [10 μM]		5.026	3.293	24 % [10 μM]	

IC₅₀ or values of %-inhibition, and calculated K_i of compounds against HDAC isoforms 1, 6, 8, and 11. Assays were performed as described in biochemical methods.

Table S1 continued. IC₅₀ values and estimated K_i values against indicated HDAC enzymes. -

Compound	HDAC1 LGKac		HDAC6 LGKac		HDAC8 LGKtfa		HDAC11 ETDKmyr	
	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)
C1	14 % [5 μM]		0.077	0.034	1.066	0.698	30 % [10 μM]	
C2	22 % [10 μM]		0.225	0.100	1.208	0.791	43 % [5 μM]	
C3	12 % [5 μM]		0.564	0.251	3.130	2.051	48 % [10 μM]	
C4	11 % [10 μM]		0.746	0.332	2.620	1.717	4.409	2.441
C8	9 % [10 μM]		0.832	0.370	3.545	2.323	9 % [10 μM]	
C9	6 % [5 μM]		1.026	0.456	3.115	2.041	6.904	3.822
C11	11 % [5 μM]		0.696	0.309	6.957	4.558	48 % [5 μM]	
C12	10 % [5 μM]		2.099	0.933	9.502	6.225	51 % [5 μM]	
C19	7 % [5 μM]		3.214	1.428	10.300	6.748	53 % [10 μM]	
C21	NI [5 μM]		2.648	1.177	5.845	3.829	51 % [5 μM]	
C23	39 % [5 μM]		0.101	0.045	0.706	0.463	NI [5 μM]	
C24	13 % [5 μM]		0.119	0.053	1.264	0.828	20 % [10 μM]	
D1	NI [5 μM]		40% [10 μM]		32 % [10 μM]		4.507	2.495
D2	NI [10 μM]		31 % [10 μM]		22 % [10 μM]		0.826	0.457
D3	7 % [5 μM]		31 % [10 μM]		37 % [10 μM]		0.338	0.187
D4	NI [10 μM]		30 % [10 μM]		47 % [10 μM]		0.491	0.272
D8	NI [10 μM]		23 % [10 μM]		14 % [10 μM]		1.290	0.714
D9	NI [10 μM]		19 % [10 μM]		17 % [10 μM]		0.321	0.178
D11	NI [5 μM]		18 % [10 μM]		16 % [10 μM]		1.167	0.646
D12	NI [10 μM]		30 % [10 μM]		11 % [10 μM]		1.050	0.581
D19	NI [5 μM]		5 % [10 μM]		17 % [10 μM]		0.979	0.542
D21	NI [10 μM]		27 % [10 μM]		17 % [10 μM]		0.269	0.149
D23	7 % [5 μM]		46 % [10 μM]		6.204	4.065	1.865	1.032
D24	NI [5 μM]		37 % [10 μM]		48 % [10 μM]		0.827	0.458
E1	3.975	0.845	0.085	0.038	1.017	0.666	NI [10 μM]	
E2	1.805	0.384	0.053	0.024	0.516	0.338	45 % [10 μM]	
E3	2.922	0.621	0.147	0.066	1.203	0.788	3.966	2.195
E4	35 % [10 μM]		0.249	0.110	1.708	1.119	NI [10 μM]	
E8	4.253	0.904	0.074	0.033	0.960	0.629	59 % [10 μM]	
E9	2.059	0.438	0.063	0.028	0.894	0.586	3.764	2.084
E11	50 % [5 μM]		0.190	0.085	1.083	0.710	6.234	3.451
E12	3.580	0.761	0.405	0.180	2.216	1.452	39 % [10 μM]	
E19	41 % [10 μM]		0.688	0.306	5.001	3.277	18 % [10 μM]	
E21	6.263	1.332	0.603	0.268	1.881	1.232	2.695	1.492
E23	1.002	0.213	0.064	0.028	0.665	0.436	8 % [10 μM]	
E24	1.416	0.301	0.030	0.014	0.422	0.276	5.404	2.992
F1	0.719	0.153	0.614	0.273	2.744	1.798	7 % [10 μM]	
F2	0.076	0.016	0.039	0.017	0.262	0.172	38 % [10 μM]	
F3	0.228	0.048	0.266	0.118	0.978	0.641	35 % [10 μM]	
F4	0.778	0.165	0.243	0.108	0.251	0.164	15 % [10 μM]	
F11	0.735	0.156	0.358	0.159	1.269	0.831	NI [10 μM]	
F12	1.151	0.245	1.658	0.737	3.701	2.425	NI [10 μM]	
F19	3.186	0.677	1.960	0.871	6.608	4.329	NI [10 μM]	
F21	1.045	0.222	1.216	0.540	3.141	2.058	8 % [10 μM]	

IC₅₀ or values of %-inhibition, and calculated K_i values for compounds against HDAC isoforms 1, 6, 8, and 11. Assays were performed as described in biochemical methods.

Table S2. Additional selectivity data for compounds **A10**, **C1**, **D21**, **E3**, **E23**, **E24**, **F2**, and **F3**

Compound	HDAC2		HDAC3		HDAC4	
	LGKac		LGKac		LGKtfa	
	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)
A10	15 % [10 μM]		37 % [10 μM]		NI [10 μM]	
C1	6 % [10 μM]		17 % [10 μM]		19 % [10 μM]	
D21	NI [10 μM]		NI [10 μM]		NI [10 μM]	
E3	5.607	1.310	4.257	0.905	38 % [10 μM]	
E23	1.366	0.319	1.906	0.405	38 % [10 μM]	
E24	1.456	0.340	1.284	0.273	33 % [10 μM]	
F2	0.201	0.047	0.093	0.020	17 % [10 μM]	
F3	0.450	0.105	0.194	0.041	14 % [10 μM]	

Compound	HDAC5		HDAC7		HDAC9	
	LGKtfa		LGKtfa		LGKtfa	
	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)	IC ₅₀ (μM) or % inhibition	K _i (μM)
A10	10 % [10 μM]		16 % [10 μM]		15 % [10 μM]	
C1	27 % [10 μM]		47 % [10 μM]		33 % [10 μM]	
D21	NI [10 μM]		NI [10 μM]		NI [10 μM]	
E3	5.966	1.966	49 % [10 μM]		31 % [10 μM]	
E23	3.560	1.173	5.070	1.679	49 % [10 μM]	
E24	8.201	2.703	46 % [10 μM]		35 % [10 μM]	
F2	29 % [10 μM]		36 % [10 μM]		15 % [10 μM]	
F3	22 % [10 μM]		30 % [10 μM]		13 % [10 μM]	

IC₅₀ or values of %-inhibition, and calculated K_i values for compounds against HDAC isoforms 2, 3, 4, 5, 7, and 9. Assays were performed as described in biochemical methods.

Biochemical Methods

HDAC Inhibition Assays

Assays were performed as previously described and were performed in 96-well plates (Fischer Scientific, cat. #: 3686) in HEPES buffer (50 mM HEPES, 100 mM KCl, 0.001% Tween-20 (v/v), 0.2 mM TCEP, pH 7.4 at 25 °C; 25 µL/well final volume) added bovine serum albumin (BSA; Sigma-Aldrich #A3059; 0.5 mg/mL for HDAC1, HDAC6, HDAC8 and 0.05 mg/mL for HDAC11). Inhibitors (3- or 5-fold dilution series starting from concentrations at either 5 or 10 µM) were incubated with enzyme (HDAC1 [5 nM], HDAC6 [1 nM], HDAC8 [0.25 nM], HDAC11 [0.5 nM]) and substrates (HDAC1, 2, 3 and 6: Ac-Leu-Gly-Lys(Ac)-AMC (20 µM), HDAC4, 5, 7, 8 and 9: Ac-Leu-Gly-Lys(TFA)-AMC (20 µM for HDAC4, 120 µM for HDAC5, 40 µM for HDAC7, 100 µM for HDAC8 and 80 µM for HDAC9), HDAC11: Ac-Glu-Thr-Asp-Lys(Myrr)-AMC (50 µM) were incubated for 30 min at 37 °C (AMC = 7-amino-4-methylcoumarin) before a solution of trypsin (Sigma-Aldrich #T8003; 25 µL, 0.4 mg/mL; final concentration 0.2 mg/mL) was subsequently added, and the assay was allowed to develop for 15 min at room temperature. Then fluorescence was recorded on a platereader (FLUOstar Omega) with excitation at 360 nm and detecting emission at 460 nm. Data was analysed to afford residual enzymatic activity relative to control wells and, assuming fast-on/fast-off mechanism, IC₅₀ values were obtained by fitting the results to the dose-response equation with a variable Hill-slope (Eq. 2). Inhibition K_i values were calculated from the Cheng-Prusoff equation (Eq. 3) and reported substrate K_M-values (HDAC1: K_M = 5.4 µM¹, HDAC2: K_M = 6.1 µM¹, HDAC3: K_M = 5.4 µM¹, HDAC4: K_M = 10.3 µM², HDAC5: K_M = 59 µM², HDAC6 K_M = 16 µM², HDAC7: K_M = 19.8 µM², HDAC8 K_M = 190 µM², HDAC9: K_M = 37 µM², HDAC11 K_M = 62 µM³). Assays were performed at least twice in technical duplicates, and data was analysed using GraphPad Prism. Enzyme source: HDAC1: BPS Bioscience #50051 (Full length with C-terminal His6 and FLAG tag); HDAC2: BPS Bioscience #50052 (Full length with C-terminal FLAG-tag); HDAC3: BPS Bioscience #50003 (Full length with C-terminal HIS-tag in complex with human NCOR2 395-489 with C-terminal GST-tag); HDAC4: BPS Bioscience #50004 (Catalytic domain (627-1084) with N-terminal GST-tag and C-terminal His-tag); HDAC5: BPS Bioscience #50005 (Catalytic domain (656-1122) with C-terminal His-tag); HDAC6: BPS Bioscience #50056 (Full length with C-terminal FLAG-tag); HDAC7: BPS Bioscience #50007 (Catalytic domain (518-end) with N-terminal GST-tag); HDAC8: BPS Bioscience #50008 (Full length with C-terminal His-tag); HDAC9: BPS Bioscience #50009 (Catalytic domain (604-1066) with C-terminal His-tag); HDAC11: BPS Bioscience #50021 (Full length untagged).

$$v_i = v_{\text{bottom}} + \frac{v_{\text{top}} - v_{\text{bottom}}}{1 + 10^{(\log \text{IC}_{50} - \log [I])h}} \quad \text{Eq. 2}$$

$$K_i = \frac{\text{IC}_{50}}{1 + \frac{[S]}{K_M}} \quad \text{Eq. 3}$$

HDAC Inhibition Assays with Preincubation

Inhibitors (**C1**, **E24** and **D21**, 10 µM–0.52 nM, 3-fold dilutions, 10 µL) were incubated with enzyme (HDAC6 for **C1** and **E24** at 2.5 nM; HDAC11 for **D21** at 1.0 nM, 10 µL) for 0, 0.5, 1 or 2 h at 37 °C followed by addition of a substrate (Ac-Leu-Gly-Lys(Ac)-AMC at 20 µM for **C1** and **E24**; Ac-Glu-Thr-Asp-Lys(Myrr)-AMC at 50 µM for **D21**, 5 µL). Total reaction volume was 25 µL and all concentrations are reported for the 25 µL reaction volume. The reactions were performed in HEPES buffer described above (0.5 mg/mL BSA for HDAC6 and 0.05 mg/mL for HDAC11). Following a 30 min incubation, a trypsin solution (0.4 mg/mL, 25 µL) was added and the plate was left standing at room temperature for 15 min before fluorescence recording. All experiments were performed twice with internal replicate wells.

Cellular assays

Cell culture

HEK293T (ATCC, CRL-1573) cells were cultured at 37 °C under a humidified 5% CO₂ atmosphere in Dulbecco's modified Eagle's medium (DMEM, Thermo Scientific, 11965118) supplemented with 10% (v/v) fetal bovine serum (FBS, Thermo Fisher Scientific, 26140079), 1% penicillin-streptomycin (Sigma-Aldrich, P4333). Cells were sub-cultured every 2–4 days.

Cellular Thermal Shift Assay (CETSA)

Cellular thermal shifts were measured as previously described.⁴⁻⁵ In brief HEK293T cells were plated in 10 cm² dishes and incubated to 80–90% confluency, where medium was replaced with culture medium containing **E24** (10 μM in 1% DMSO), or vehicle (1% DMSO). The cells were treated for 2 h before media was removed by aspiration and cells were collected in PBS (2 mL/treatment) by scraping, and pelleted by centrifugation (300 g, 5 min). Pellets were resuspended in PBS (2 mL) and pelleted by centrifugation before the washed pellets were suspended in PBS supplemented with cOmplete EDTA-free protease inhibitor cocktail (Sigma-Aldrich, COEDTAF-RO, 550 μL/cell treatment). Cell suspensions were aliquoted into PCR tubes (50 μL) and heated at temperatures ranging from 37.0 °C to 65.0 °C for 3 min followed by 3 min at 25 °C in a thermal cycler (Eppendorf Mastercycler Nexus Thermal Cycler). The cellular suspensions were then lysed by three freeze/thaw cycles, snap-freezing in a dry-ice/acetone bath followed by thawing at 25 °C in the thermal cycle. The suspensions were subjected to centrifugation (20,000 g, 20 min) at 4 °C and the supernatants were collected as whole-cell lysate and mixed with NuPAGE LDS sample buffer (ThermoFisher, NP0007) and sample reducing agent (ThermoFisher, NP0004) followed by heating to 95 °C for 10 min. Samples were then resolved by gel electrophoresis (SDS-PAGE) in NuPAGE gels (12 wells, 4–12% Bis-Tris, 1.0 mm, ThermoFisher, NP0322BOX) with MES running buffer (ThermoFisher, NP000202). Proteins were transferred to PVDF membranes (ThermoFisher, IB24001) using the iBlot2 system and blocked with 5% skim milk in tris-buffered saline with 0.1% tween-20 (TBST) at 25 °C for 1 h. Membranes were then washed with TBST (3×5 min), incubated with primary antibody (Anti-HDAC6, Cell Signaling Technology, #7558, 1:1000 or Anti-HDAC8, Cell Signaling Technology, #66042, 1:1000) in TBST with 5% BSA (4 °C, overnight), washed with TBST (3×5 min), incubated with HRP-conjugated secondary antibody (Anti-rabbit IgG HRP-linked antibody, Cell Signaling Technology, CST-7074S, 1:10,000) in TBST with 2% skim milk (25 °C, 1 h), and washed with TBST (3×5 min) and TBS (1×5 min). Membranes were visualized using enhanced chemiluminescent reagents (Pierce ECL Western blotting substrate, ThermoFisher, 32106) on a syngene PXi4 image analysis system. Protein marker used: Precision Plus Protein All Blue Standard (BioRAD, 161-0373).

Protein acetylation assays

Protein acetylation levels were determined as previously described.⁵ In brief HEK293T cells were seeded in 6-well plates and grown to 70–80% confluency, before medium was replaced by culture medium containing **C1** or **E24** (1 or 10 μM) or SAHA (1 μM) or vehicle (DMSO). The cells were treated for 5 h at 37 °C after which the media was aspirated and the cells were washed with PBS (2 x 1 mL) and lysed with 100 μL of radioimmunoprecipitation assay (RIPA) buffer (150 mM NaCl, 50 mM Tris, 1% Triton, 0.5% sodium deoxycholate, 0.1% SDS, pH 8, complemented with cOmplete EDTA-free protease inhibitor cocktail (Sigma-Aldrich, COEDTAF-RO). Plates were stored at -20 °C for >20 min and the cell lysates were then collected by scraping, sonicated with a Bandelin Sonopuls mini20 (2 s on, 2 s off, 80% amplitude, 1 min) and centrifuged (14 000 g, 10 min, 4 °C). Supernatants were collected and their concentrations were determined by the bicinchoninic acid assay (Sigma-Aldrich; #BCA1). The concentration of the lysates were adjusted with PBS to 2 μg/μL and mixed with NuPAGE LDS sample buffer (ThermoFisher, NP0007) and sample reducing agent (ThermoFisher, NP0004) followed by heating to 95 °C for 10 min. Samples (20 μg) were then resolved by gel electrophoresis (SDS-PAGE) in NuPAGE gels (10 wells, 4–12% Bis-Tris, 1.0 mm, ThermoFisher, NW04120BOX) with MES running buffer (ThermoFisher, NP000202). Proteins were transferred to PVDF membranes (ThermoFisher, IB24001) using the iBlot2 system and blocked with 5% BSA in tris-buffered saline with 0.1% tween-20 (TBST) at 25 °C for 1 h. Membranes were then incubated

with primary antibody: Vinculin (E1E9V) XP (Cell Signaling Technology, #13901, 1:1000), acetyl-histone H3 (Lys18) (D8Z5H, Cell Signaling Technology, #13998, 1:500), acetyl-histone H3 (K27) (Cell Signaling Technology, #4353, 1:500), acetyl- α -tubulin (Lys40) (Cell Signaling Technology, #5335, 1:500), Acetyl-SMC3 (Lys105/106) (MABE1073, Sigma-Aldrich, 1:250) or GAPDH (14C10, Cell Signaling Technology, #2118, 1:1000) in TBST with 5% BSA (4 °C, overnight), washed with TBST (3×5 min), incubated with HRPconjugated secondary antibody (Anti-rabbit IgG HRP-linked antibody, Cell Signaling Technology, CST-7074S, 1:10,000 or Anti-Mouse IgG HRP-linked antibody, Cell Signaling Technology, CST-7076S, 1:10,000) in TBST with 2% skim milk (25 °C, 1 h), and washed with TBST (3×5 min) and TBS (1×5 min). Membranes were visualized using enhanced chemiluminescent reagents (Pierce ECL Western blotting substrate, ThermoFisher, 32106) on a syngene PXi4 image analysis system. Protein marker used: Precision Plus Protein All Blue Standard (BioRAD, 161-0373).

Chemical Stability Assays

Inhibitors (50 μ M) were suspended in assay buffer (50 mM HEPES, 100 mM KCl, 0.001% Tween-20 (v/v), 0.2 mM TCEP, pH 7.4 at 25 °C), with or without glutathione (2 mM) or medium (unsupplemented DMEM) and incubated at 37 °C. Aliquots were taken at 0, 1, 2, 4, and 24 h and measured by UPLC using an Agilent 1260 Infinity II system equipped with a reverse phase C18 column (Poroshell, #695575-302, 3 x 100 mm) using a binary buffer system consisting of H₂O–MeCN–TFA (A, 95:5:0.1; B, 5:95:0.1) at 1.2 mL/min. The chemical stability was calculated based on the area under curve (AUC) (215 nm), relative to the time point 0 h.

Chemistry

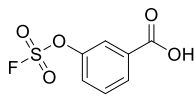
Materials and Methods

All reagents and solvents were of analytical grade and used without further purification as obtained from commercial suppliers. Anhydrous solvents were obtained from a PureSolv-system or acquired as anhydrous from vendors and stored in sealed bottles. Reactions were conducted under an atmosphere of nitrogen whenever anhydrous solvents were used. Reactions were monitored by thin-layer chromatography (TLC) using silica gel coated plates (analytical SiO₂-60, F-254) and by LC-MS. TLC plates were visualized under UV light or by dipping into a solution of potassium permanganate (10 g/L), potassium carbonate (67 g/L) and sodium hydroxide (0.83 g/L) in water. Evaporation of solvents was carried out under reduced pressure at a temperature below or equal to 40 °C. LC–MS analyses were performed on a Phenomenex Kinetex column (1.7 μ m, 50×2.10 mm) using a Waters Acquity ultra high-performance liquid chromatography (UPLC) system. Gradient A with eluent I (0.1% HCOOH in H₂O) and eluent II (0.1% HCOOH in MeCN) rising linearly from 0% to 95% of II during $t = 0.00$ –5.20 min was applied at a flow rate of 0.6 mL/min. Preparative reverse-phase HPLC purification was performed on a C8 or C18 Phenomenex Luna column (5 μ m, 100 Å, 250×21.2 mm) using an Agilent 1260 LC system equipped with a diode array UV detector and an evaporative light scattering detector (ELSD). Gradient B with eluent III (H₂O/MeCN/TFA, 95:5:0.1, v:v) and eluent IV (0.1% TFA in MeCN) rising linearly from 0% to 95% of IV during $t = 20$ –35 min at a flow rate of 20 mL/min was applied. Analytical HPLC was performed on a C18 Infinity Poroshell 120 column (2.7 μ m, 100×3.0 mm) using an Agilent 1260 Infinity II series system equipped with a diode array UV detector. Analytical HPLC was performed using gradient B during either $t = 1$ –11 min or $t = 1$ –16 min was applied at a flow rate of 1.2 mL/min. High-resolution mass spectrometry (HRMS) measurements were recorded either on a maXis G3 quadrupole time-of-flight (TOF) mass spectrometer (Bruker Daltonics, Bremen, Germany) equipped with an electrospray ionization (ESI) source or on an Agilent 1290 UHPLC equipped with a diode array detector and coupled to Agilent 6550 QTOF mass spectrometer operated in positive electrospray or on a Bruker Solarix WR by either matrix assisted laser desorption/ionization, or ESI. Nuclear magnetic resonance (NMR) spectra were

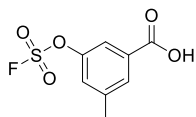
recorded either on a Bruker Avance III HD equipped with a cryogenically cooled probe (^1H NMR and ^{13}C NMR recorded at 600 and 151 MHz, respectively) or a Bruker Avance III (^1H NMR, ^{13}C NMR and ^{19}F NMR recorded at 400, 101 and 377 MHz, respectively). All spectra were recorded at 298 K. Chemical shifts are reported in ppm relative to deuterated solvent as internal standard (δ_{H} DMSO- d_6 2.50 ppm; δ_{C} DMSO- d_6 39.52 ppm; δ_{H} CDCl_3 7.26 ppm; δ_{C} CDCl_3 77.16 ppm δ_{H} MeOH- d_4 3.31; δ_{C} MeOH- d_4 49.00). Assignments of NMR spectra are based on 2D correlation spectroscopy (COSY, HSQC, and HMBC spectra). The concentrations of the compound stock solutions were determined by quantitative NMR (qNMR) using maleic acid (2H at 6.27 in DMSO- d_6) as an internal standard. Subsequently, the stock solutions were diluted to 5 mM, which were used in the assays.

Compound Synthesis and Characterization Data

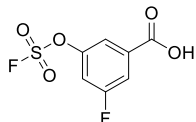
3-((Fluorosulfonyl)oxy)benzoic acid (S1). Using general procedure 1 compound **S1** was synthesized from 3-hydroxybenzoic acid (138 mg, 1.00 mmol) and isolated as a white powder (220 mg, quantitative yield) without further purifications. ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.56 (s, 1H, COOH), 8.10–8.06 (m, 1H, CHCH₂COOH), 8.05–8.02 (m, 1H, CCHC), 7.93–7.86 (m, 1H, CHCH₂CO), 7.79–7.69 (m, 1H, CHCH₂CH). ¹³C NMR (101 MHz, DMSO) δ 165.6, 149.5, 134.3, 131.3, 129.9, 125.3, 121.5. ¹⁹F NMR (376 MHz, DMSO) δ 39.0. In accordance with previously reported data.⁶ CAS-RN: 1796596-42-3.



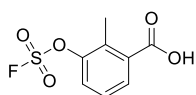
3-((Fluorosulfonyl)oxy)-5-methylbenzoic acid (S2). Using general procedure 1 compound **S2** was synthesized from 3-hydroxy-5-methylbenzoic acid (152 mg, 1.00 mmol) and isolated as a white powder (232 mg, 99%) after purification by flash column chromatography (0-50% EtOAc in heptane + 0.1% AcOH). ¹H NMR (600 MHz, DMSO) δ 13.50 (s, 1H), 7.90 (tt, *J* = 1.5, 0.8 Hz, 1H), 7.82 (t, *J* = 1.9 Hz, 1H), 7.72 (t, *J* = 2.2 Hz, 1H), 2.45 (s, 3H). ¹³C NMR (151 MHz, DMSO) δ 166.2, 149.9, 142.4, 133.8, 130.9, 126.1, 119.1, 21.0. HRMS *m/z* 232.9925 ([*M*-H]⁻, C₈H₆O₅SF₂⁻, calcd. 232.9925). In accordance with previously reported data.⁷ CAS-RN: 2866335-05-7.



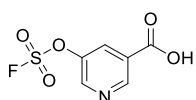
3-Fluoro-5-((fluorosulfonyl)oxy)benzoic acid (S3). Using general procedure 1 compound **S3** was synthesized from 3-fluoro-5-hydroxybenzoic acid (156 mg, 1.00 mmol) and isolated as a white powder (160 mg, 67%) after purification by flash column chromatography (0-5% MeOH in CH₂Cl₂ + 0.25% AcOH). TLC (10% MeOH in CH₂Cl₂ and 0.25% AcOH): *R*_f = 0.79. ¹H NMR (600 MHz, DMSO) δ 13.88 (br. s, 1H), 8.04 (dt, *J* = 8.5, 2.4 Hz, 1H), 7.95–7.92 (m, 1H), 7.86 (ddd, *J* = 8.6, 2.5, 1.3 Hz, 1H). ¹³C NMR (151 MHz, DMSO) δ 164.56 (d, *J* = 3.1 Hz), 162.00 (d, *J* = 250.4 Hz), 149.49 (d, *J* = 11.5 Hz), 135.12 (d, *J* = 8.3 Hz), 118.17 (d, *J* = 3.5 Hz), 117.11 (d, *J* = 22.7 Hz), 114.22 (d, *J* = 26.6 Hz). ¹⁹F NMR (376 MHz, DMSO) δ 39.7, -107.3. HRMS *m/z* 236.9675 ([*M*-H]⁻, C₇H₄O₅SF₂⁻, calcd. 236.9674). CAS-RN: 2475192-21-1.



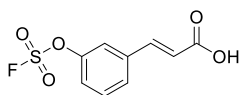
3-((fluorosulfonyl)oxy)-2-methylbenzoic acid (S4). Using general procedure 1 compound **S4** was synthesized from 3-hydroxy-2-methylbenzoic acid (152 mg, 1.00 mmol) and isolated as a white powder (164 mg, 70%) after purification by flash column chromatography (0-5% MeOH in CH₂Cl₂ + 0.25% AcOH). TLC (10% MeOH in CH₂Cl₂ and 0.25% AcOH): *R*_f = 0.75. ¹H NMR (600 MHz, DMSO) δ 13.46–13.42 (br. s, 1H), 7.91 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.76 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.54–7.49 (m, 1H), 2.50 (s, 3H, overlaps with solvent peak). ¹³C NMR (151 MHz, DMSO) δ 167.4, 149.0, 134.3, 130.9, 130.5, 127.9, 124.4, 13.1. ¹⁹F NMR (376 MHz, DMSO) δ 40.45. HRMS *m/z* 232.9927 ([*M*-H]⁻, C₈H₆O₅SF₂⁻, calcd. 232.9925).



5-((Fluorosulfonyl)oxy)nicotinic acid (S5): Using general procedure 1 compound **S5** was synthesized from 5-hydroxynicotinic acid (139.1 mg, 1.00 mmol) and isolated as a white powder (226 mg, 100%) after purification by flash column chromatography (0-5% MeOH in CH₂Cl₂ + 0.25% AcOH). ¹H NMR (400 MHz, DMSO) δ 14.00 (s, 1H), 9.18 (d, *J* = 1.7 Hz, 1H), 9.14 (d, *J* = 2.7 Hz, 1H), 8.53 (ddd, *J* = 2.6, 1.7, 0.7 Hz, 1H). ¹³C NMR (101 MHz, DMSO) δ 164.6, 150.5, 146.6, 146.4, 130.0, 128.7. ¹⁹F NMR (376 MHz, DMSO) δ 40.1. In accordance with previously reported data.⁷ CAS-RN: 2408958-14-3.



(E)-3-(3-((Fluorosulfonyl)oxy)phenyl)acrylic acid (S6): Using general procedure 1 compound **S6** was synthesized from (*E*)-3-(3-hydroxyphenyl)acrylic acid (823 mg, 5.00 mmol) and isolated as a white powder (882 mg, 70%) after purification by flash column chromatography (0-5% MeOH in CH₂Cl₂ + 0.25% AcOH). ¹H NMR (600 MHz, DMSO) δ 12.57 (s, 1H), 8.02 (d, *J* = 2.0 Hz, 1H), 7.85 (td, *J* = 5.8, 3.1 Hz, 1H), 7.66–7.60 (m, 3H), 6.70 (d, *J* = 16.0 Hz, 1H). ¹³C NMR (151 MHz, DMSO) δ 167.2, 150.1, 141.6, 137.3, 131.3, 128.9, 122.3, 122.0, 120.4. ¹⁹F NMR (376 MHz, DMSO) δ 39.1.



Resin A: Using GP2 starting from S1 and general procedure 3, resin A was synthesized with a loading of 0.52 mmol/g.

Resin B: Using GP2 starting from S2 and general procedure 3, resin B was synthesized with a loading of 0.70 mmol/g.

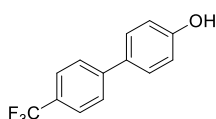
Resin C: Using GP2 starting from S3 and general procedure 3, resin C was synthesized with a loading of 0.54 mmol/g.

Resin D: Using GP2 starting from S4 and general procedure 3, resin D was synthesized with a loading of 0.59 mmol/g.

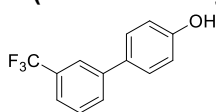
Resin E: Using GP2 starting from S5 and general procedure 3, resin E was synthesized with a loading of 0.52 mmol/g.

Resin F: Using GP2 starting from S6 and general procedure 3, resin F was synthesized with a loading of 0.61 mmol/g.

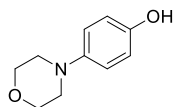
4'-(Trifluoromethyl)-[1,1'-biphenyl]-4-ol (S7): 4-Bromophenol (100 mg, 0.578 mmol), (4-(trifluoromethyl)phenyl)boronic acid (329 mg, 1.73 mmol) and sodium carbonate (368 mg, 3.47 mmol) was suspended in a mixture of 1,4-dioxane and water (4:1, 2.8 mL) and degassed with nitrogen for 10 min before Pd(PPh₃)₄ (20 mg, 0.017 mmol) was added and the solution was degassed for another 10 min before it was heated to 100 °C overnight. After cooling to room temperature, the solution was diluted with EtOAc (5 mL) and filtered through celite. The filtrate was partitioned between H₂O and EtOAc and the organic layer was dried over Na₂SO₄ before volatiles were removed under reduced pressure. Purification by flash column chromatography (0-40 % EtOAc in heptane) afforded the title compound **S7** (41 mg, 30 %) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.35 (d, *J* = 7.8 Hz, 1H, residual boronic acid), 7.79 (d, *J* = 7.8 Hz, 1H, residual boronic acid), 7.73–7.58 (m, 4H), 7.54–7.45 (m, 2H), 6.98–6.88 (m, 2H). ¹⁹F NMR (376 MHz, CDCl₃) δ -62.3, -63.2. CAS-RN: 10355-13-2.



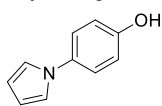
3'-(Trifluoromethyl)-[1,1'-biphenyl]-4-ol (S8): 4-bromophenol (100 mg, 0.578 mmol), (3-(trifluoromethyl)phenyl)boronic acid (165 mg, 0.867 mmol), Pd(OAc)₂ (6.5 mg, 0.028 mmol), dppf (32 mg, 0.058 mmol), Et₃N (121 μL, 0.867 mmol) and sodium acetate (47 mg, 0.578 mmol) were all suspended in a mixture of 1,4-dioxane and water (9:1, 2.9 mL) which was then degassed with nitrogen for 20 min before it was heated to 100 °C overnight. After cooling to room temperature, the solution was diluted with EtOAc (5 mL) and filtered through celite. The filtrate was partitioned between H₂O and EtOAc and the organic layer was dried over Na₂SO₄ before volatiles were removed under reduced pressure. Purification by flash column chromatography (0-40 % EtOAc in heptane) afforded the title compound **S8** (32 mg, 23 %) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.78 (tt, *J* = 1.8, 0.9 Hz, 1H), 7.71 (dt, *J* = 7.2, 1.8 Hz, 1H), 7.59–7.47 (m, 4H), 7.00–6.90 (m, 2H). ¹⁹F NMR (376 MHz, CDCl₃) δ -62.6. CAS-RN: 191724-12-6.



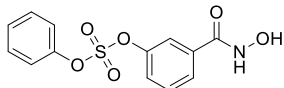
4-Morpholinophenol (S9): 4-bromophenol (173 mg, 1.00 mmol), Pd(OAc)₂ (4.5 mg, 0.02 mmol) and dppf (22 mg, 0.04 mmol) was added to a schlenk flask and evacuated three times followed by flushing with nitrogen. Toluene (5 mL) was added along with morpholine (95 μL, 1.1 mmol) and LiHMDS (1M in THF, 2.4 mL) before the reaction was heated to 80 °C for two days. The reaction was diluted with EtOAc and concentrated onto silica, before it was purified by flash column chromatography (0-55 % EtOAc in heptane) to afford the title compound **S9** (40 mg, 22%) as a yellow solid. ¹H NMR (600 MHz, MeOD) δ 6.89–6.83 (m, 2H), 6.75–6.69 (m, 2H), 3.83–3.79 (m, 4H), 3.01–2.97 (m, 4H). ¹³C NMR (151 MHz, MeOD) δ 153.0, 146.0, 119.7, 116.7, 68.0, 52.7. CAS-RN: 6291-23-2.



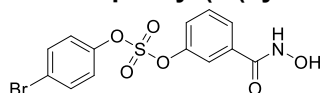
4-(1H-Pyrrol-1-yl)phenol (S10): 4-aminophenol (1.00 g, 9.16 mmol) was suspended in AcOH (25 mL) and heated to reflux before 2,5-dimethoxytetrahydrofuran (1.42 mL, 11.0 mmol) was added and the reaction was refluxed for 5 hours. After cooling to room temperature, the reaction mixture was poured into ice cold H₂O (400 mL) and precipitate was collected by filtration. The filtrate was purified by vacuum column chromatography (Isocratic with CH₂Cl₂) to afford the title compound (560 mg, 38%) as an off-white solid. ¹H NMR (600 MHz, DMSO) δ 9.47 (s, 1H), 7.34–7.30 (m, 2H), 7.16 (t, *J* = 2.2 Hz, 2H), 6.85–6.79 (m, 2H), 6.19 (t, *J* = 2.2 Hz, 2H). ¹³C NMR (151 MHz, DMSO) δ 155.2, 132.3, 121.2, 119.1, 115.9, 109.5. CAS-RN: 23351-09-9.



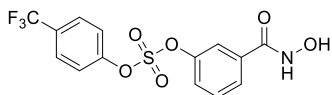
3-(Hydroxycarbamoyl)phenyl phenyl sulfate (A1): Using general procedure 4 on a 20 μmol scale using resin A and phenol, compound **A1** (1.5 mg, 24 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.22–9.19 (m, 1H), 7.86–7.73 (m, 2H), 7.67–7.52 (m, 4H), 7.50–7.41 (m, 3H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.3, 149.9, 135.1, 130.8, 130.6, 128.3, 126.4, 123.8, 121.0, 119.5, 116.9. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.673 min (98%, UV_{215.4}). HRMS m/z 310.0378 ($[\text{M}+\text{H}]^+$, C₁₃H₁₃NO₆S⁺, calcd. 310.0380).



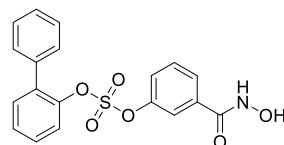
4-Bromophenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A2): Using general procedure 4 on a 20 μmol scale using resin A and 4-bromophenol, compound **A2** (1.2 mg, 15 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.21 (s, 1H), 7.86–7.82 (m, 1H), 7.80–7.78 (m, 1H), 7.77–7.74 (m, 2H), 7.68–7.61 (m, 2H), 7.49–7.43 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 149.7, 148.9, 135.1, 133.5, 130.9, 126.5, 123.8, 123.4, 120.8, 119.4. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation via the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.482 min (>98%, UV_{215.4}). HRMS m/z 387.9496 ($[\text{M}+\text{H}]^+$, C₁₃H₁₁NO₆SBr⁺, calcd. 387.9485).



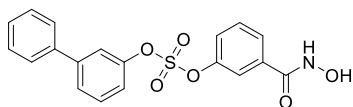
3-(Hydroxycarbamoyl)phenyl (4-(trifluoromethyl)phenyl) sulfate (A3): Using general procedure 4 on a 20 μmol scale using resin A and 4-(trifluoromethyl)phenol, compound **A3** (0.4 mg, 5 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.45 (s, 1H), 7.96 (dd, J = 7.8, 1.4 Hz, 2H), 7.86–7.82 (m, 1H), 7.80 (s, 1H), 7.75–7.70 (m, 2H), 7.68–7.63 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 152.3, 149.7, 135.2, 131.0, 128.7 (q, J = 32.5 Hz), 128.2 (q, J = 3.6 Hz), 126.7, 123.9, 123.6 (q, J = 271.8 Hz), 122.2, 119.5. ^{19}F NMR (376 MHz, DMSO) δ -60.97, -73.41 (CF₃, TFA). Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.688 min (95%, UV_{215.4}). HRMS m/z 378.0254 ($[\text{M}+\text{H}]^+$, C₁₄H₁₂F₃NO₆S⁺, calcd. 378.0253).



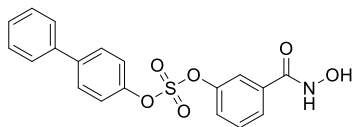
[1,1'-Biphenyl]-2-yl (3-(hydroxycarbamoyl)phenyl) sulfate (A4): Using general procedure 4 on a 20 μmol scale using resin A and [1,1'-biphenyl]-2-ol, compound **A4** (3.8 mg, 49 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.40 (s, 1H), 9.19 (s, 1H), 7.78 (dt, J = 7.8, 1.3 Hz, 1H), 7.67–7.35 (m, 12H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 149.6, 146.8, 135.7, 134.5, 132.0, 130.7, 129.6, 129.0, 128.5, 128.0, 126.3, 123.6, 121.4, 119.4, 116.9, 116.4. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.902 min (97%, UV_{215.4}). HRMS m/z 386.0706 ($[\text{M}+\text{H}]^+$, C₁₉H₁₆NO₆S⁺, calcd. 386.0693).



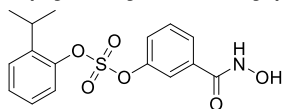
[1,1'-Biphenyl]-3-yl (3-(hydroxycarbamoyl)phenyl) sulfate (A5): Using general procedure 4 on a 20 μmol scale using resin A and [1,1'-biphenyl]-3-ol, compound **A5** (0.9 mg, 12 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.45 (s, 1H), 7.87–7.80 (m, 2H), 7.80–7.75 (m, 1H), 7.72–7.61 (m, 6H), 7.53–7.45 (m, 3H), 7.45–7.40 (m, 1H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 150.4, 149.8, 142.7, 138.2, 135.1, 131.1, 130.9, 129.2, 129.1, 128.8, 128.4, 126.9, 126.5, 123.9, 121.5, 119.8, 119.6, 119.1. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.210 min (90%, UV_{215.4}). HRMS m/z 386.0696 ($[\text{M}+\text{H}]^+$, C₁₉H₁₇NO₆S⁺, calcd. 386.0693).



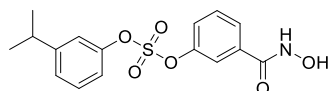
[1,1'-Biphenyl]-4-yl (3-(hydroxycarbamoyl)phenyl) sulfate (A6): Using general procedure 4 on a 20 μmol scale using resin A and [1,1'-biphenyl]-4-ol, compound **A6** (0.9 mg, 12 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.45 (s, 1H), 9.21 (s, 1H), 7.87–7.79 (m, 4H), 7.72–7.63 (m, 4H), 7.57–7.45 (m, 4H), 7.44–7.39 (m, 1H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 149.8, 149.3, 140.2, 138.6, 135.1, 130.9, 129.1, 128.8, 128.0, 126.9, 126.4, 123.8, 121.5, 119.5. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.264 min (95%, UV_{215.4}). HRMS m/z 386.0693 ($[\text{M}+\text{H}]^+$, C₁₉H₁₇NO₆S⁺, calcd. 386.0693).



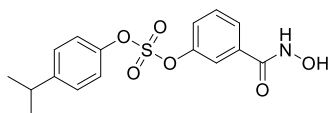
3-(Hydroxycarbamoyl)phenyl (2-isopropylphenyl) sulfate (A7). Using general procedure 4 on a 20 μmol scale using resin A and 2-isopropylphenol, compound **A7** (1 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.20 (s, 1H), 7.88–7.75 (m, 2H), 7.68–7.56 (m, 2H), 7.52–7.45 (m, 1H), 7.43–7.32 (m, 3H), 3.12 (dhept, $J = 13.6, 6.8$ Hz, 1H), 1.14 (dd, $J = 6.9, 4.2$ Hz, 6H). ^{13}C NMR (151 MHz, DMSO) δ 147.3, 140.3, 130.8, 128.45, 128.36, 127.9, 127.9, 127.7, 126.5, 124.0, 120.5, 119.7, 26.5, 22.7. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 8.787 min (96%, UV_{215.4}). HRMS m/z 374.0668 ($[\text{M}+\text{Na}]^+$, C₁₆H₁₇NO₆SNa⁺, calcd. 374.0669).



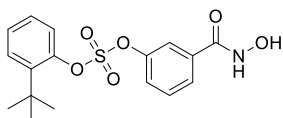
3-(Hydroxycarbamoyl)phenyl (3-isopropylphenyl) sulfate (A8): Using general procedure 4 on a 20 μmol scale using resin A and 3-isopropylphenol, compound **A8** (1.0 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.20 (s, 1H), 7.83 (dt, $J = 7.7, 1.3$ Hz, 1H), 7.80–7.74 (m, 1H), 7.67–7.62 (m, 1H), 7.62–7.55 (m, 1H), 7.45 (dt, $J = 9.9, 7.9$ Hz, 1H), 7.37–7.29 (m, 1H), 7.29–7.21 (m, 2H), 3.01–2.87 (m, 1H), 1.18 (dd, $J = 16.9, 6.9$ Hz, 6H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.3, 158.8, 151.5, 150.0, 149.8, 135.1, 130.8, 130.4, 126.2, 123.8, 119.5, 118.8, 118.2, 33.2, 23.5. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.028 min (>98%, UV_{215.4}). HRMS m/z 352.0861 ($[\text{M}+\text{H}]^+$, C₁₆H₁₈NO₆S⁺, calcd. 352.0849).



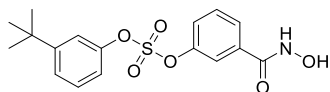
3-(Hydroxycarbamoyl)phenyl (4-isopropylphenyl) sulfate (A9). Using general procedure 4 on a 20 μmol scale using resin A and 4-isopropylphenol, compound **A9** (2 mg, 28 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.43 (s, 1H), 9.20 (s, 1H), 7.83 (dt, $J = 7.5, 1.4$ Hz, 1H), 7.78 (t, $J = 2.0$ Hz, 1H), 7.66–7.56 (m, 2H), 7.43–7.38 (m, 2H), 7.38–7.33 (m, 2H), 3.00–2.89 (m, 1H), 1.22–1.18 (m, 6H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 149.7, 148.5, 147.9, 135.1, 130.8, 128.3, 126.3, 123.8, 120.8, 120.7, 119.5, 32.9, 23.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.144 min (97%, UV_{215.4}). HRMS m/z 352.0852 ($[\text{M}+\text{H}]^+$, C₁₆H₁₈NO₆S⁺, calcd. 352.0849).



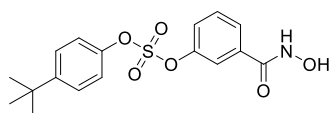
2-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A10): Using general procedure 4 on a 20 μmol scale using resin A and 2-(tert-butyl)phenol, compound **A10** (1.5 mg, 21 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.20 (s, 1H), 7.86–7.74 (m, 2H), 7.69–7.55 (m, 3H), 7.55–7.32 (m, 3H), 1.33 (d, $J = 5.3$ Hz, 9H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.3, 149.6, 140.2, 135.1, 130.8, 128.4, 128.3, 127.5, 126.5, 123.9, 119.9, 119.7, 117.1, 34.4, 30.0. Analytical UPLC gradient 40–70% eluent II in eluent I over 10 min (C18), t_{R} 7.210 min (97%, UV_{215.4}). HRMS m/z 366.1007 ($[\text{M}+\text{H}]^+$, C₁₇H₂₁NO₆S⁺, calcd. 366.1006).



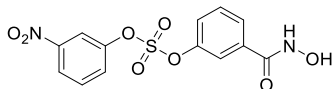
3-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A11). Using general procedure 4 on a 20 μmol scale using resin A and 3-(tert-butyl)phenol, compound **A11** (2 mg, 27 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.43 (s, 1H), 9.20 (br. s, 1H), 7.83 (dt, $J = 7.7, 1.3$ Hz, 1H), 7.78 (dd, $J = 2.5, 1.5$ Hz, 1H), 7.64 (t, $J = 8.0$ Hz, 1H), 7.59 (ddd, $J = 8.3, 2.6, 1.1$ Hz, 1H), 7.50–7.42 (m, 2H), 7.31 (ddd, $J = 2.3, 1.5, 0.7$ Hz, 1H), 7.28 (dt, $J = 6.6, 2.4$ Hz, 1H), 1.27 (s, 9H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 153.9, 149.9, 149.8, 135.1, 130.8, 130.1, 126.3, 125.1, 123.7, 119.4, 117.9, 117.8, 34.7, 30.8. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.466 min (>98%, UV_{215.4}). HRMS m/z 366.1006 ($[\text{M}+\text{H}]^+$, C₁₇H₂₀NO₆S⁺, calcd. 366.1006).



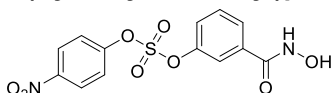
4-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A12). Using general procedure 4 on a 20 μmol scale using resin A and 4-(tert-butyl)phenol, compound **A12** (2 mg, 27 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.44 (s, 1H), 9.20 (s, 1H), 7.83 (dt, $J = 7.5, 1.4$ Hz, 1H), 7.78 (t, $J = 2.0$ Hz, 1H), 7.66–7.59 (m, 2H), 7.58–7.52 (m, 2H), 7.39–7.34 (m, 2H), 1.30 (s, 9H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.3, 150.8, 149.7, 147.7, 135.1, 130.8, 127.3, 126.4, 123.8, 120.5, 119.5, 34.5, 31.0. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.636 min (>98%, UV_{215.4}). HRMS m/z 366.1006 ($[\text{M}+\text{H}]^+$, C₁₇H₂₀NO₆S⁺, calcd. 366.1006).



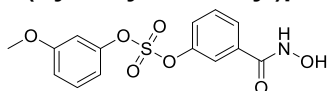
3-(Hydroxycarbamoyl)phenyl (3-nitrophenyl) sulfate (A13): Using general procedure 4 on a 20 μmol scale using resin A and 3-nitrophenol, compound **A13** (1.0 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.44 (s, 1H), 9.23–9.20 (m, 1H), 8.32 (dddd, $J = 15.4, 8.3, 2.2, 0.9$ Hz, 1H), 8.26 (dt, $J = 6.2, 2.3$ Hz, 1H), 7.99 (dddd, $J = 12.6, 8.3, 2.4, 0.9$ Hz, 1H), 7.90–7.75 (m, 3H), 7.70–7.63 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 162.2 (from HMBC), 149.6, 149.4, 148.6, 132.1, 131.0, 127.9, 126.7, 123.9, 123.2, 119.5, 116.6. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.774 min (>98%, UV_{215.4}). HRMS m/z 377.0062 ($[\text{M}+\text{Na}]^+$, $\text{C}_{13}\text{H}_{10}\text{N}_2\text{O}_8\text{SNa}^+$, calcd. 377.0050).



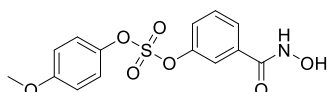
3-(Hydroxycarbamoyl)phenyl (4-nitrophenyl) sulfate (A14): Using general procedure 4 on a 20 μmol scale using resin A and 4-nitrophenol, compound **A14** (1.0 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.22 (s, 1H), 8.45–8.37 (m, 2H), 7.87–7.75 (m, 4H), 7.69–7.64 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 162.2, 153.5, 149.6, 146.5, 135.2, 130.9, 126.7, 126.3, 123.8, 122.4, 119.5. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.834 min (>98%, UV_{215.4}). HRMS m/z 355.0229 ($[\text{M}+\text{H}]^+$, $\text{C}_{26}\text{H}_{21}\text{N}_4\text{O}_{16}\text{S}_2^+$, calcd. 355.0230).



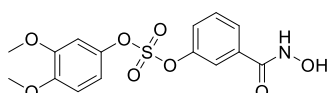
3-(Hydroxycarbamoyl)phenyl (3-methoxyphenyl) sulfate (A15): Using general procedure 4 on a 20 μmol scale using resin A and 3-methoxyphenol, compound **A15** (2.4 mg, 35 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.44 (s, 1H), 9.20 (s, 1H), 7.83 (dt, $J = 7.5, 1.4$ Hz, 1H), 7.78 (t, $J = 2.0$ Hz, 1H), 7.67–7.57 (m, 2H), 7.49–7.42 (m, 1H), 7.07–6.99 (m, 2H), 6.96 (dt, $J = 13.2, 2.4$ Hz, 1H), 3.79 (s, 3H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.3, 160.5, 150.6, 149.7, 131.0, 130.8, 126.4, 123.8, 119.5, 113.9, 112.7, 107.0, 55.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.908 min (>98%, UV_{215.4}). HRMS m/z 340.0496 ($[\text{M}+\text{H}]^+$, $\text{C}_{14}\text{H}_{14}\text{NO}_7\text{S}^+$, calcd. 340.0485).



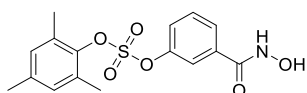
3-(Hydroxycarbamoyl)phenyl (4-methoxyphenyl) sulfate (A16): Using general procedure 4 on a 20 μmol scale using resin A and 4-methoxyphenol, compound **A16** (1.1 mg, 16 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.21 (s, 1H), 7.82 (dt, $J = 7.7, 1.3$ Hz, 1H), 7.79–7.73 (m, 1H), 7.64 (t, $J = 8.0$ Hz, 1H), 7.59 (ddd, $J = 8.3, 2.6, 1.2$ Hz, 1H), 7.41–7.32 (m, 2H), 7.09–7.02 (m, 2H), 3.79 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 162.4, 158.5, 149.8, 143.2, 135.1, 130.8, 126.3, 123.8, 122.3, 119.5, 115.3, 55.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.840 min (>98%, UV_{215.4}). HRMS m/z 340.0497 ($[\text{M}+\text{H}]^+$, $\text{C}_{14}\text{H}_{14}\text{NO}_7\text{S}^+$, calcd. 340.0485).



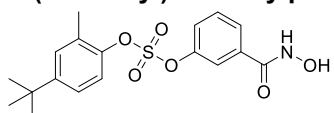
3,4-Dimethoxyphenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A17): Using general procedure 4 on a 20 μmol scale using resin A and 3,4-dimethoxyphenol, compound **A17** (3.1 mg, 42 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.43 (s, 1H), 9.20 (s, 1H), 7.82 (dt, $J = 7.6, 1.4$ Hz, 1H), 7.80–7.74 (m, 1H), 7.67–7.55 (m, 2H), 7.08–7.02 (m, 1H), 7.01–6.94 (m, 2H), 3.79 (s, 3H), 3.76 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 149.7, 149.5, 148.2, 143.2, 135.1, 130.8, 126.2, 119.5, 112.4, 111.9, 105.5, 102.1, 55.9, 55.9. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.532 min (97%, UV_{215.4}). HRMS m/z 370.0604 ($[\text{M}+\text{H}]^+$, $\text{C}_{15}\text{H}_{16}\text{NO}_8\text{S}^+$, calcd. 370.0591).



3-(Hydroxycarbamoyl)phenyl mesityl sulfate (A18): Using general procedure 4 on a 20 μmol scale using resin A and mesitol, compound **A18** (0.5 mg, 7 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.45 (s, 1H), 7.86–7.80 (m, 2H), 7.67–7.62 (m, 2H), 7.01 (s, 2H), 2.24 (d, $J = 6.2$ Hz, 9H). ^{13}C NMR (151 MHz, DMSO) δ 162.4, 150.7, 149.7, 146.5, 135.0, 130.8, 129.6, 129.1, 126.4, 124.7, 124.0, 120.2, 119.6, 34.4, 31.1, 15.9. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.021 min (95%, UV_{215.4}). HRMS m/z 352.0849 ($[\text{M}+\text{H}]^+$, $\text{C}_{16}\text{H}_{19}\text{NO}_6\text{S}^+$, calcd. 352.0849).



4-(tert-Butyl)-2-methylphenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A19): Using general procedure 4 on a 20 μmol scale using resin A and 4-(tert-butyl)-2-methylphenol, compound **A19** (0.8 mg, 11 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 7.86–7.78 (m, 2H), 7.67–7.58 (m, 2H),



7.42 (d, $J = 2.5$ Hz, 1H), 7.35 (dd, $J = 8.7, 2.6$ Hz, 1H), 7.28 (d, $J = 8.6$ Hz, 1H), 2.24 (s, 3H), 1.28 (s, 9H). ^{13}C NMR (151 MHz, DMSO) δ 162.4, 150.7, 149.7, 146.5, 135.0, 130.8, 129.6, 129.1, 126.4, 124.7, 124.0, 120.2, 119.6, 34.4, 31.1, 15.9. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.782 min (98%, UV_{215.4}). HRMS m/z 380.1164 ($[\text{M}+\text{H}]^+$, $\text{C}_{18}\text{H}_{23}\text{NO}_6\text{S}^+$, calcd. 380.1162).

2-(Benzyloxy)phenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A20): Using general procedure 4 on a 20 μmol scale using resin A and 2-(benzyloxy)phenol, compound **A20** (1.6 mg, 19 %) was isolated as a colorless fluffy powder after lyophilization. ^1H of major rotamer NMR (600 MHz, DMSO) δ 11.42 (s, 1H), 9.19 (s, 1H), 7.85–7.81 (m, 1H), 7.79 (dt, $J = 7.6, 1.3$ Hz, 1H), 7.59 (ddd, $J = 8.3, 2.5, 1.1$ Hz, 1H), 7.55 (t, $J = 7.9$ Hz, 1H), 7.43–7.28 (m, 8H), 7.03 (dddd, $J = 11.5, 8.1, 7.3, 1.6$ Hz, 1H), 5.22 (d, $J = 4.0$ Hz, 2H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 150.4, 150.3, 139.3, 136.7, 135.4, 131.0, 129.6, 128.9, 128.9, 128.4, 127.9, 126.7, 124.4, 123.0, 121.6, 115.7, 70.4. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.015 min (>98%, UV_{215.4}). HRMS m/z 416.0813 ($[\text{M}+\text{H}]^+$, $\text{C}_{20}\text{H}_{18}\text{NO}_7\text{S}^+$, calcd. 416.0798).

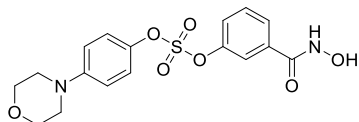
3-(Hydroxycarbamoyl)phenyl (4-phenoxyphenyl) sulfate (A21): Using general procedure 4 on a 20 μmol scale using resin A and 4-phenoxyphenol, compound **A21** (2 mg, 25 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.21 (s, 1H), 7.83 (dt, $J = 7.5, 1.4$ Hz, 1H), 7.77 (t, $J = 2.0$ Hz, 1H), 7.67–7.60 (m, 2H), 7.50–7.46 (m, 2H), 7.46–7.41 (m, 2H), 7.20 (tt, $J = 7.4, 1.1$ Hz, 1H), 7.15–7.11 (m, 2H), 7.10–7.06 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 156.3, 155.9, 149.8, 144.9, 135.1, 130.8, 130.28, 130.25, 126.4, 124.2, 123.8, 122.9, 122.8, 119.7, 119.5, 119.2. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.437 min (>98%, UV_{215.4}). HRMS m/z 402.0642 ($[\text{M}+\text{H}]^+$, $\text{C}_{19}\text{H}_{16}\text{NO}_7\text{S}^+$, calcd. 402.0642).

4-(1H-Pyrrol-1-yl)phenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A22): Using general procedure 4 on a 20 μmol scale using resin A and 4-(1H-pyrrol-1-yl)phenol, compound **A22** (3.1 mg, 41 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.44 (s, 1H), 9.20 (s, 1H), 7.86–7.79 (m, 2H), 7.78–7.71 (m, 2H), 7.70–7.61 (m, 2H), 7.57–7.49 (m, 2H), 7.44–7.37 (m, 2H), 6.32–6.26 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 149.8, 146.7, 139.4, 135.1, 130.9, 126.5, 123.8, 122.4, 120.9, 119.3, 116.4, 111.0. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.703 min (>98%, UV_{215.4}). HRMS m/z 375.0658 ($[\text{M}+\text{H}]^+$, $\text{C}_{17}\text{H}_{15}\text{N}_2\text{O}_6\text{S}^+$, calcd. 375.0645).

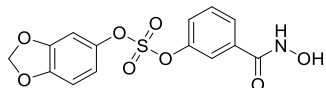
4-(1H-Imidazol-1-yl)phenyl (3-(hydroxycarbamoyl)phenyl) sulfate (A23): Using general procedure 4 on a 20 μmol scale using resin A and 4-(1H-imidazol-1-yl)phenol, compound **A23** (5.0 mg, 67 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.46 (s, 1H), 9.50 (dt, $J = 16.0, 1.5$ Hz, 1H), 8.23 (t, $J = 1.7$ Hz, 1H), 8.00–7.93 (m, 2H), 7.85 (pd, $J = 4.8, 1.5$ Hz, 1H), 7.81 (td, $J = 2.3, 1.1$ Hz, 2H), 7.79–7.73 (m, 3H), 7.69–7.58 (m, 2H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 158.8, 149.7, 149.5, 135.3, 134.7, 130.9, 126.5, 124.3, 123.8, 122.9, 120.7, 119.5, 116.9. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 3.936 min (>98%, UV_{215.4}). HRMS m/z 376.0611 ($[\text{M}+\text{H}]^+$, $\text{C}_{16}\text{H}_{14}\text{N}_3\text{O}_6\text{S}^+$, calcd. 376.0597).

3-(Hydroxycarbamoyl)phenyl naphthalen-2-yl sulfate (A24): Using general procedure 4 on a 40 μmol scale using resin A and naphthalen-2-ol, compound **A24** (1.6 mg, 11 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.45 (s, 1H), 9.21 (s, 1H), 8.12 (d, $J = 9.0$ Hz, 1H), 8.08–8.03 (m, 3H), 7.88–7.81 (m, 2H), 7.69–7.55 (m, 5H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.3, 149.8, 147.4, 135.1, 133.1, 131.8, 130.9, 130.8, 128.0, 127.9, 127.5, 127.1, 126.4, 123.9, 119.7, 119.6, 118.6. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.956 min (98%, UV_{215.4}). HRMS m/z 360.0552 ($[\text{M}+\text{H}]^+$, $\text{C}_{17}\text{H}_{14}\text{NO}_6\text{S}^+$, calcd. 360.0536).

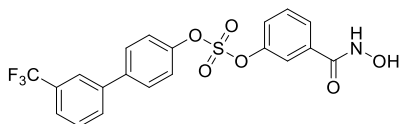
3-(Hydroxycarbamoyl)phenyl (4-morpholinophenyl) sulfate (A25): Using general procedure 4 on a 20 μmol scale using resin A and 4-morpholinophenol, compound **A25** (1.0 mg, 11 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 7.82 (dt, $J = 7.7, 1.3$ Hz, 1H), 7.77 (dd, $J = 2.5, 1.5$ Hz, 1H), 7.64 (t, $J = 8.0$ Hz, 1H), 7.58 (ddd, $J = 8.3, 2.5, 1.1$ Hz, 1H), 7.31–7.25 (m, 2H), 7.06–7.00 (m, 2H), 3.76–3.70 (m, 4H), 3.16–3.10 (m, 4H). ^{13}C NMR (151 MHz, DMSO) δ 150.5, 149.8, 142.2, 135.0, 130.8, 126.2, 123.7, 121.6, 119.4, 115.9, 66.0, 48.1. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.502 min (>98%, UV_{215.4}). 395.0922 ([M+H]⁺, C₁₇H₁₉N₂O₇S⁺, calcd. 395.0907).



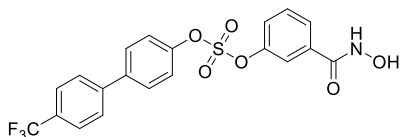
Benzo[d][1,3]dioxol-5-yl (3-(hydroxycarbamoyl)phenyl) sulfate (A26): Using general procedure 4 on a 20 μmol scale using resin A and benzo[d][1,3]dioxol-5-ol, compound **A26** (1.8 mg, 25 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.43 (s, 1H), 9.20 (s, 1H), 7.82 (dt, $J = 7.6, 1.4$ Hz, 1H), 7.79–7.73 (m, 1H), 7.67–7.58 (m, 2H), 7.13–7.07 (m, 1H), 7.02 (d, $J = 8.5$ Hz, 1H), 6.95–6.88 (m, 1H), 6.14 (s, 2H). ^{13}C NMR (151 MHz, DMSO) δ 149.7, 148.2, 146.8, 143.8, 135.0, 130.8, 126.3, 123.8, 119.5, 114.1, 108.4, 103.2, 102.5. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.703 min (>98%, UV_{215.4}). HRMS m/z 354.0291 ([M+H]⁺, C₁₄H₁₂NO₈S⁺, calcd. 354.0278).



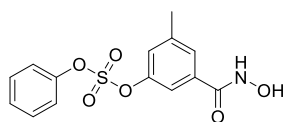
3-(Hydroxycarbamoyl)phenyl (3'-(trifluoromethyl)-[1,1'-biphenyl]-4-yl) sulfate (A27): Using general procedure 4 on a 20 μmol scale using resin A and 3'-(trifluoromethyl)-[1,1'-biphenyl]-4-ol, compound **A27** (3.8 mg, 32 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.45 (s, 1H), 9.21 (s, 1H), 8.04–7.98 (m, 2H), 7.96–7.88 (m, 2H), 7.88–7.80 (m, 2H), 7.80–7.71 (m, 2H), 7.66 (dd, $J = 4.6, 1.6$ Hz, 2H), 7.62–7.53 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 162.3, 149.8, 149.7, 139.7, 138.5, 135.1, 131.1, 130.9, 130.2, 129.86 (q, $J = 31.8$ Hz), 129.3, 126.5, 124.60 (d, $J = 3.8$ Hz), 123.82 (q, $J = 272.6$ Hz), 123.8, 123.44 (q, $J = 3.9$ Hz), 121.6, 119.5. ^{19}F NMR (376 MHz, DMSO) δ -61.0, -73.6 (CF₃, TFA). Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.932 min (>98%, UV_{215.4}). 454.0585 ([M+H]⁺, C₂₀H₁₅NO₆F₃S⁺, calcd. 454.0566).



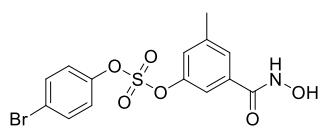
3-(Hydroxycarbamoyl)phenyl (4'-(trifluoromethyl)-[1,1'-biphenyl]-4-yl) sulfate (A28): Using general procedure 4 on a 20 μmol scale using resin A and 4'-(trifluoromethyl)-[1,1'-biphenyl]-4-ol, compound **A28** (4.5 mg, 30 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.46 (s, 1H), 9.22 (s, 1H), 7.97–7.88 (m, 4H), 7.88–7.80 (m, 4H), 7.70–7.63 (m, 2H), 7.63–7.55 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 162.8, 150.4, 150.2, 143.1, 139.0, 135.6, 131.4, 129.8, 128.83 (q, $J = 31.9$ Hz), 128.3, 126.9, 126.36 (q, $J = 3.8$ Hz), 124.48 (d, $J = 272.2$ Hz), 124.3, 122.2, 120.0. ^{19}F NMR (376 MHz, DMSO) δ -61.0, -73.5 (CF₃, TFA). Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.998 min (>98%, UV_{215.4}). HRMS m/z 454.0585 ([M+H]⁺, C₂₀H₁₅NO₆F₃S⁺, calcd. 454.0566).



3-(Hydroxycarbamoyl)-5-methylphenyl phenyl sulfate (B1): Using general procedure 4 on a 20 μmol scale using resin B and phenol, compound **B1** (3.4 mg, 53 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.37 (s, 1H), 9.17 (s, 1H), 7.67 (q, $J = 1.2$ Hz, 1H), 7.60–7.52 (m, 3H), 7.50–7.40 (m, 4H), 2.39 (d, $J = 12.7$ Hz, 3H). ^{13}C NMR (151 MHz, DMSO) δ 162.4, 158.9, 149.9, 149.6, 141.0, 134.7, 130.6, 128.2, 124.0, 121.1, 116.5, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 7.572 min (97%, UV_{215.4}). HRMS m/z 324.0549 ([M+H]⁺, C₁₄H₁₄NO₆S⁺, calcd. 324.0536).

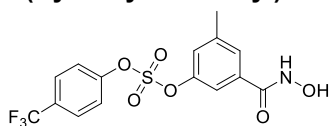


4-Bromophenyl (3-(hydroxycarbamoyl)-5-methylphenyl) sulfate (B2): Using general procedure 4 on a 20 μmol scale using resin B and 4-bromophenol, compound **B2** (1 mg, 12 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.37 (s, 1H), 9.17 (s, 1H), 7.79–7.72 (m, 2H), 7.69–7.65 (m, 1H), 7.58 (ddd, $J = 2.3, 1.5, 0.7$ Hz, 1H), 7.48–7.41 (m, 3H), 2.41 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 162.4, 149.6, 148.9, 141.1, 134.7, 133.4, 127.2, 124.0, 123.4,

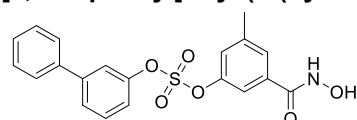


120.8, 116.4, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_R 8.793 min (>98%, UV_{215.4}). HRMS m/z 401.9642 ([M+H]⁺, C₁₄H₁₃NO₆SBr⁺, calcd. 401.9641).

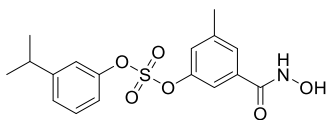
3-(Hydroxycarbamoyl)-5-methylphenyl (4-(trifluoromethyl)phenyl) sulfate (B3). Using general procedure 4 on a 20 μ mol scale using resin B and 4-(trifluoromethyl)phenol, compound **B3** (0.7 mg, 8 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.38 (s, 1H), 9.17 (s, 1H), 8.00–7.93 (m, 2H), 7.76–7.71 (m, 2H), 7.70–7.67 (m, 1H), 7.60 (t, J = 2.0 Hz, 1H), 7.47 (d, J = 2.1 Hz, 1H), 2.41 (s, 3H). ¹³C NMR (151 MHz, DMSO) δ 162.8, 152.7, 150.0, 141.6, 135.3, 129.2, 129.0, 128.6 (q, J = 3.8 Hz), 127.8, 125.0, 124.5, 123.2, 122.7, 116.9, 21.2. ¹⁹F NMR (376 MHz, DMSO) δ -60.97, -73.53 (CF₃, TFA). Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.025 min (>98%, UV_{215.4}). HRMS m/z 392.0414 ([M+H]⁺, C₁₅H₁₃NO₆F₃S⁺, calcd. 392.0410).



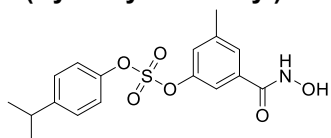
[1,1'-Biphenyl]-3-yl (3-(hydroxycarbamoyl)-5-methylphenyl) sulfate (B4): Using general procedure 4 on a 40 μ mol scale using resin B and [1,1'-biphenyl]-3-ol, compound **B4** (1.9 mg, 12f %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.33 (s, 1H), 7.65–7.59 (m, 2H), 7.60–7.50 (m, 4H), 7.47–7.36 (m, 7H), 7.18–7.16 (m, 1H), 2.34 (d, J = 10.5 Hz, 3H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 158.9, 149.5, 146.8, 140.8, 135.7, 134.5, 131.9, 129.5, 129.0, 128.4, 128.0, 127.0, 123.7, 121.4, 116.4, 114.0, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.195 min (91%, UV_{215.4}). HRMS m/z 400.0867 ([M+H]⁺, C₂₀H₁₈NO₆S⁺, calcd. 400.0849).



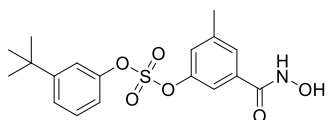
3-(Hydroxycarbamoyl)-5-methylphenyl (3-isopropylphenyl) sulfate (B8). Using general procedure 4 on a 20 μ mol scale using resin B and 2-isopropylphenol, compound **B8** (0.8 mg, 11 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.37 (s, 1H), 9.16 (s, 1H), 7.68–7.64 (m, 1H), 7.60–7.56 (m, 1H), 7.47 (t, J = 7.9 Hz, 1H), 7.39 (s, 1H), 7.35 (ddt, J = 7.7, 1.5, 0.7 Hz, 1H), 7.27 (ddd, J = 8.2, 2.6, 1.0 Hz, 1H), 7.24 (t, J = 2.1 Hz, 1H), 2.96 (hept, J = 7.0 Hz, 1H), 2.40 (d, J = 0.8 Hz, 3H), 1.20 (d, J = 6.9 Hz, 6H). ¹³C NMR (151 MHz, DMSO) δ 162.4, 151.5, 150.0, 149.7, 141, 134.7, 130.3, 127.0, 126.2, 124.0, 118.8, 118.3, 116.5, 33.1, 23.5, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.376 min (>98%, UV_{215.4}). HRMS m/z 366.1004 ([M+H]⁺, C₁₇H₂₀NO₆S⁺, calcd. 366.1006).



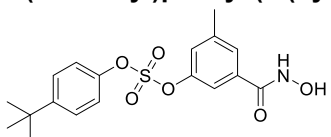
3-(Hydroxycarbamoyl)-5-methylphenyl (4-isopropylphenyl) sulfate (B9): Using general procedure 4 on a 20 μ mol scale using resin B and 4-isopropylphenol, compound **B9** (2.9 mg, 40 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR of major rotamer (600 MHz, DMSO) δ 11.37 (s, 1H), 9.16 (s, 1H), 7.66 (t, J = 1.3 Hz, 1H), 7.60–7.56 (m, 1H), 7.46–7.31 (m, 5H), 2.95 (tq, J = 14.1, 6.9 Hz, 1H), 2.41–2.36 (m, 3H), 1.20 (dd, J = 12.8, 6.9 Hz, 6H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 158.9, 149.8, 148.5, 147.9, 140.9, 130.9, 128.2, 124.8, 120.9, 114.0, 32.9, 23.7, 20.7. Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_R 9.584 min (>98%, UV_{215.4}). HRMS m/z 366.1019 ([M+H]⁺, C₁₇H₂₀NO₆S⁺, calcd. 366.1006).



3-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)-5-methylphenyl) sulfate (B11). Using general procedure 4 on a 20 μ mol scale using resin B and 3-(tert-butyl)phenol, compound **B11** (1.1 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.37 (s, 1H), 9.16 (s, 1H), 7.66 (q, J = 1.2 Hz, 1H), 7.60–7.56 (m, 1H), 7.52–7.42 (m, 2H), 7.39 (ddd, J = 2.4, 1.5, 0.8 Hz, 1H), 7.32–7.30 (m, 1H), 7.28 (dt, J = 6.6, 2.4 Hz, 1H), 2.39 (d, J = 0.8 Hz, 3H), 1.27 (s, 9H). ¹³C NMR (151 MHz, DMSO) δ 162.4, 153.9, 149.9, 149.7, 141.0, 134.7, 130.1, 127.0, 125.0, 123.9, 118.0, 117.8, 116.5, 34.7, 30.8, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.678 min (>98%, UV_{215.4}). HRMS m/z 380.1164 ([M+H]⁺, C₁₈H₂₂NO₆S⁺, calcd. 380.1162).

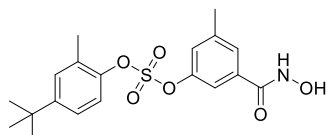


4-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)-5-methylphenyl) sulfate (B12). Using general procedure 4 on a 20 μ mol scale using resin B and 4-(tert-butyl)phenol, compound **B12** (1.1 mg, 15 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.37 (s, 1H), 9.16 (s, 1H), 7.66 (q, J = 1.3 Hz, 1H), 7.58 (dd, J = 2.5, 1.4 Hz, 1H), 7.57–7.54 (m, 2H), 7.40 (ddd, J = 2.4, 1.5, 0.8 Hz, 1H), 7.38–

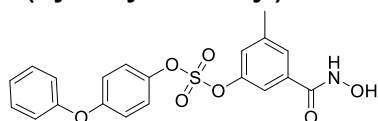


7.34 (m, 2H), 2.40 (s, 3H), 1.30 (s, 9H). ¹³C NMR (151 MHz, DMSO) δ 162.4, 150.8, 149.7, 147.6, 141.0, 134.7, 127.3, 127.1, 124.0, 120.5, 116.5, 34.4, 31.0, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), *t*_R 7.793 min (>98%, UV_{215.4}). HRMS *m/z* 380.1163 ([M+H]⁺, C₁₈H₂₂NO₆S⁺, calcd. 380.1162).

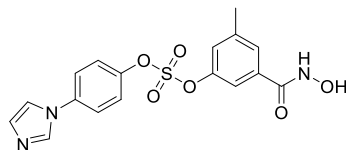
4-(tert-Butyl)-2-methylphenyl (3-(hydroxycarbamoyl)-5-methylphenyl) sulfate (B19). Using general procedure 4 on a 20 μmol scale using resin B and 4-(*tert*-butyl)-2-methylphenol, compound **B19** (0.8 mg, 10 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.37 (s, 1H), 9.16 (s, 1H), 7.67 (q, *J* = 1.1 Hz, 1H), 7.60 (t, *J* = 2.1 Hz, 1H), 7.43 (dd, *J* = 2.5, 0.8 Hz, 1H), 7.42–7.39 (m, 1H), 7.36 (ddd, *J* = 8.7, 2.6, 0.6 Hz, 1H), 7.29 (d, *J* = 8.6 Hz, 1H), 2.40 (d, *J* = 0.8 Hz, 3H), 2.25 (s, 3H), 1.28 (s, 9H). ¹³C NMR (151 MHz, DMSO) δ 162.4, 150.6, 149.6, 146.4, 140.9, 134.7, 129.6, 129.0, 127.1, 124.6, 124.1, 120.2, 116.6, 34.3, 31.0, 20.7, 15.9. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), *t*_R 10.631 min (>98%, UV_{215.4}). HRMS *m/z* 394.1318 ([M+H]⁺, C₁₉H₂₄NO₆S⁺, calcd. 394.1319).



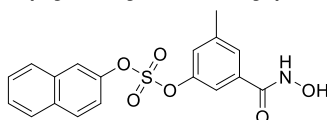
3-(Hydroxycarbamoyl)-5-methylphenyl (4-phenoxyphenyl) sulfate (B21). Using general procedure 4 on a 20 μmol scale using resin B and 4-phenoxyphenol, compound **B21** (1.1 mg, 13 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.39–11.36 (m, 1H), 9.17 (d, *J* = 1.7 Hz, 1H), 7.66 (td, *J* = 1.5, 0.7 Hz, 1H), 7.57 (ddd, *J* = 2.3, 1.4, 0.6 Hz, 1H), 7.49–7.41 (m, 5H), 7.20 (tt, *J* = 7.5, 1.1 Hz, 1H), 7.15–7.11 (m, 2H), 7.10–7.07 (m, 2H), 2.41 (s, 3H). ¹³C NMR (151 MHz, DMSO) δ 162.4, 156.2, 155.9, 149.6, 144.9, 141.0, 134.7, 130.3, 127.1, 124.2, 124.0, 122.9, 119.7, 119.2, 116.4, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), *t*_R 7.621 min (>98%, UV_{215.4}). HRMS *m/z* 416.0799 ([M+H]⁺, C₂₀H₁₈NO₇S⁺, calcd. 416.0798).



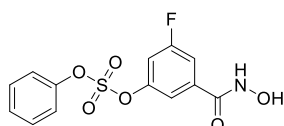
4-(1H-imidazol-1-yl)phenyl (3-(hydroxycarbamoyl)-5-methylphenyl) sulfate (B23). Using general procedure 4 on a 20 μmol scale using resin B and 4-(1H-imidazol-1-yl)phenol, compound **B23** (1.6 mg, 21 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.40 (s, 1H), 9.39 (t, *J* = 1.4 Hz, 1H), 8.20 (t, *J* = 1.7 Hz, 1H), 7.99–7.93 (m, 2H), 7.79–7.72 (m, 3H), 7.69 (td, *J* = 1.6, 0.8 Hz, 1H), 7.60 (ddd, *J* = 2.3, 1.5, 0.7 Hz, 1H), 7.48 (ddd, *J* = 2.4, 1.5, 0.8 Hz, 1H), 2.42 (d, *J* = 0.8 Hz, 3H). ¹³C NMR (151 MHz, DMSO) δ 149.6, 149.4, 141.1, 135.3, 134.8, 134.8, 127.2, 124.1, 124.0, 123.2, 122.9, 120.5, 116.5, 20.7. Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), *t*_R 4.391 min (>98%, UV_{215.4}). HRMS *m/z* 390.0754 ([M+H]⁺, C₁₇H₁₆N₃O₆S⁺, calcd. 390.0754).



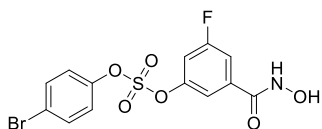
3-(Hydroxycarbamoyl)-5-methylphenyl naphthalen-2-yl sulfate (B24): Using general procedure 4 on a 40 μmol scale using resin B and naphthalen-2-ol, compound **B24** (3.1 mg, 21 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.38 (s, 1H), 9.18 (s, 1H), 8.13 (d, *J* = 9.0 Hz, 1H), 8.09–7.99 (m, 3H), 7.69–7.51 (m, 5H), 7.49–7.45 (m, 1H), 2.42–2.36 (m, 3H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 162.4, 149.7, 147.4, 141.0, 134.7, 133.1, 131.8, 130.7, 128.0, 127.9, 127.5, 127.1, 124.1, 119.7, 118.6, 116.6, 114.1, 20.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), *t*_R 7.069 min (> 98%, UV_{215.4}). HRMS *m/z* 374.0709 ([M+H]⁺, C₁₈H₁₆NO₆S⁺, calcd. 374.0693).



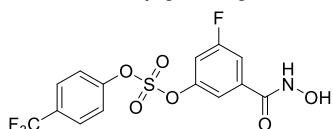
3-Fluoro-5-(hydroxycarbamoyl)phenyl phenyl sulfate (C1): Using general procedure 4 on a 40 μmol scale using resin C and penol, compound **C1** (4.5 mg, 34 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.53 (s, 1H), 7.69 (dd, *J* = 8.0, 2.3 Hz, 2H), 7.65 (dt, *J* = 8.7, 2.3 Hz, 1H), 7.59–7.51 (m, 2H), 7.50–7.43 (m, 3H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 161.94 (d, *J* = 248.7 Hz), 150.03 (d, *J* = 11.5 Hz), 149.8, 136.35 (d, *J* = 8.2 Hz), 130.6, 128.4, 121.1, 115.8, 113.69 (d, *J* = 22.9 Hz), 112.13 (d, *J* = 25.9 Hz). Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. ¹⁹F NMR (376 MHz, DMSO) δ -74.1 (CF₃, TFA), -108.1. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), *t*_R 6.017 min (96%, UV_{215.4}). HRMS *m/z* 328.0300 ([M+H]⁺, C₁₃H₁₁NO₆FS⁺, calcd. 353.0801).



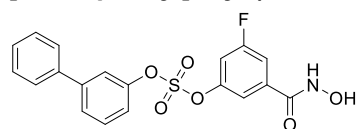
4-Bromophenyl (3-fluoro-5-(hydroxycarbamoyl)phenyl) sulfate (C2). Using general procedure 4 on a 20 μ mol scale using resin C and 4-bromophenol, compound **C2** (1.0 mg, 12 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.53 (s, 1H), 9.33 (br. s, 1H), 7.79–7.72 (m, 2H), 7.69 (tt, J = 5.1, 2.1 Hz, 3H), 7.50–7.44 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 161.9 (d, J = 248.9 Hz), 161.0, 149.9 (d, J = 11.4 Hz), 148.9, 136.4 (d, J = 8.0 Hz), 133.5, 123.4, 120.9, 115.8 (d, J = 3.2 Hz), 113.8 (d, J = 23.1 Hz), 112.2 (d, J = 26.0 Hz). ^{19}F NMR (376 MHz, DMSO) δ -74.08 ($\text{CF}_{3,\text{TFA}}$), -108.06. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.786 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 427.9211 ($[\text{M}+\text{Na}]^+$, $\text{C}_{13}\text{H}_9\text{NO}_6\text{FSBrNa}^+$, calcd. 427.9210).



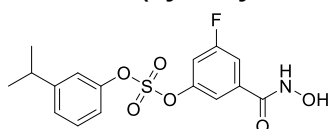
3-Fluoro-5-(hydroxycarbamoyl)phenyl (4-(trifluoromethyl)phenyl) sulfate (C3). Using general procedure 4 on a 20 μ mol scale using resin C and 4-(trifluoromethyl)phenol, compound **C3** (0.9 mg, 11 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.53 (s, 1H), 9.33 (s, 1H), 8.00–7.90 (m, 2H), 7.78–7.68 (m, 5H). ^{13}C NMR (151 MHz, DMSO) δ 161.95 (d, J = 248.6 Hz), 160.95, 152.14, 149.87 (d, J = 11.6 Hz), 136.40 (d, J = 8.1 Hz), 128.61 (d, J = 32.2 Hz), 128.11 (q, J = 3.8 Hz), 122.24, 115.85 (d, J = 3.1 Hz), 113.95 (d, J = 22.9 Hz), 112.27 (d, J = 26.0 Hz). The peak for CF_3 was not visible in ^{13}C NMR due to high coupling constant and overlap with neighbouring peaks. ^{19}F NMR (376 MHz, DMSO) δ -60.99, -73.43 ($\text{CF}_{3,\text{TFA}}$), -108.00. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 8.932 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 396.0157 ($[\text{M}+\text{H}]^+$, $\text{C}_{14}\text{H}_{10}\text{NO}_6\text{F}_4\text{S}^+$, calcd. 396.0159).



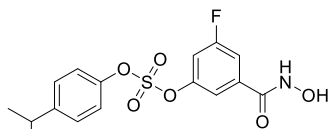
[1,1'-Biphenyl]-3-yl (3-fluoro-5-(hydroxycarbamoyl)phenyl) sulfate (C4): Using general procedure 4 on a 40 μ mol scale using resin C and [1,1'-biphenyl]-3-ol, compound **C4** (4.5 mg, 28 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.48 (s, 1H), 7.68–7.61 (m, 2H), 7.60–7.51 (m, 4H), 7.45–7.34 (m, 6H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 161.8 (d, J = 248.5 Hz), 161.2, 149.8 (d, J = 11.5 Hz), 146.7, 136.2 (d, J = 7.9 Hz), 135.5, 134.5, 132.0, 129.6, 129.0, 128.7, 128.4, 128.0, 121.6, 115.7, 113.5 (d, J = 22.7 Hz), 111.8 (d, J = 26.0 Hz). ^{19}F NMR of major rotamer (376 MHz, DMSO) δ -74.6 ($\text{CF}_{3,\text{TFA}}$), -108.3. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.195 min (95%, $\text{UV}_{215.4}$). HRMS m/z 426.0418 ($[\text{M}+\text{Na}]^+$, $\text{C}_{19}\text{H}_{14}\text{FNO}_6\text{SNa}^+$, calcd. 426.0433).



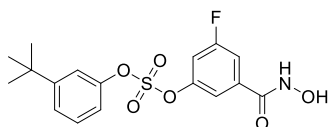
3-Fluoro-5-(hydroxycarbamoyl)phenyl (3-isopropylphenyl) sulfate (C8). Using general procedure 4 on a 20 μ mol scale using resin C and 3-isopropylphenol, compound **C8** (2.1 mg, 28 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.51 (s, 1H), 9.32 (s, 1H), 7.69 (tt, J = 3.7, 1.4 Hz, 2H), 7.62 (dt, J = 8.7, 2.3 Hz, 1H), 7.47 (t, J = 7.9 Hz, 1H), 7.38–7.33 (m, 1H), 7.31–7.25 (m, 2H), 2.96 (hept, J = 6.9 Hz, 1H), 1.20 (d, J = 6.9 Hz, 6H). ^{13}C NMR (151 MHz, DMSO) δ 161.9 (d, J = 248.6 Hz), 151.6, 150.1 (d, J = 11.5 Hz), 149.9, 136.3 (d, J = 8.0 Hz), 130.4, 126.3, 118.8, 118.3, 115.8, 113.6 (d, J = 23.3 Hz), 112.1 (d, J = 25.8 Hz), 33.1, 23.5. Peak for $\text{C}(=\text{O})\text{NH}$ was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation via the nearby ^{14}N -nuclei. ^{19}F NMR (376 MHz, DMSO) δ -73.42 ($\text{CF}_{3,\text{TFA}}$), -108.25. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.487 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 370.0754 ($[\text{M}+\text{H}]^+$, $\text{C}_{16}\text{H}_{17}\text{NO}_6\text{FS}^+$, calcd. 370.0755).



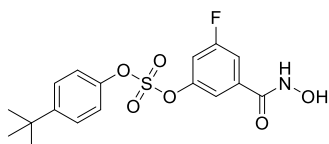
3-Fluoro-5-(hydroxycarbamoyl)phenyl (4-isopropylphenyl) sulfate (C9): Using general procedure 4 on a 40 μ mol scale using resin C and 4-isopropylphenol, compound **C9** (5.0 mg, 30 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.53 (s, 1H), 7.69 (dd, J = 8.6, 2.4 Hz, 2H), 7.62 (dt, J = 8.8, 2.3 Hz, 1H), 7.43–7.34 (m, 4H), 2.95 (tp, J = 13.6, 7.0 Hz, 1H), 1.23–1.16 (m, 6H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 161.93 (d, J = 248.6 Hz), 150.06 (d, J = 11.5 Hz), 148.7, 147.8, 136.33 (d, J = 7.9 Hz), 128.3, 120.9, 115.8, 113.64 (d, J = 23.0 Hz), 112.09 (d, J = 26.0 Hz), 32.9, 23.7. ^{19}F NMR (376 MHz, DMSO) δ -74.3 ($\text{CF}_{3,\text{TFA}}$), -108.2. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.419 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 370.0771 ($[\text{M}+\text{H}]^+$, $\text{C}_{16}\text{H}_{17}\text{NO}_6\text{FS}^+$, calcd. 370.0755).



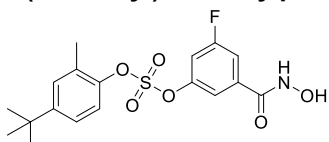
3-(*tert*-Butyl)phenyl (3-fluoro-5-(hydroxycarbamoyl)phenyl) sulfate (C11). Using general procedure 4 on a 20 μ mol scale using resin C and 3-*tert*-butylphenol, compound **C11** (1.2 mg, 15 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.52 (s, 1H), 9.32 (br. s, 1H), 7.69 (q, J = 4.1 Hz, 2H), 7.61 (dt, J = 8.9, 2.4 Hz, 1H), 7.52–7.45 (m, 2H), 7.33 (ddd, J = 2.3, 1.6, 0.6 Hz, 1H), 7.30 (ddd, J = 7.1, 2.5, 1.8 Hz, 1H), 1.27 (s, 9H). ¹³C NMR (151 MHz, DMSO) δ 161.9 (d, J = 248.8 Hz), 161.0, 153.9, 150.1 (d, J = 11.6 Hz), 149.8, 136.3 (d, J = 7.6 Hz), 130.2, 125.2, 118.0, 117.8, 115.8 (d, J = 3.3 Hz), 113.6 (d, J = 21.2 Hz), 112.1 (d, J = 25.1 Hz), 34.7, 30.8. ¹⁹F NMR (376 MHz, DMSO) δ -73.92 (CF_{3,TFA}), -108.24. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_r 7.652 min (>98%, UV_{215.4}). HRMS m/z 384.0909 ([M+H]⁺, C₁₇H₁₉NO₆FS⁺, calcd. 384.0911).



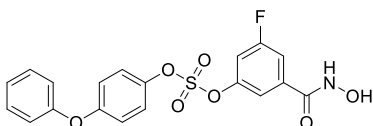
4-(*tert*-Butyl)phenyl (3-fluoro-5-(hydroxycarbamoyl)phenyl) sulfate (C12). Using general procedure 4 on a 20 μ mol scale using resin C and 4-*tert*-butylphenol, compound **C12** (2.5 mg, 33 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.53 (s, 1H), 9.33 (s, 1H), 7.70 (dd, J = 8.0, 2.3 Hz, 2H), 7.63 (dt, J = 8.7, 2.3 Hz, 1H), 7.59–7.53 (m, 2H), 7.42–7.36 (m, 2H), 1.30 (s, 9H). ¹³C NMR (151 MHz, DMSO) δ 161.9 (d, J = 248.7 Hz), 161.0, 150.9, 150.0 (d, J = 11.6 Hz), 147.6, 136.3 (d, J = 7.8 Hz), 127.3, 120.5, 115.8, 113.7 (d, J = 23.2 Hz), 112.1 (d, J = 25.7 Hz), 34.4, 31.0. ¹⁹F NMR (376 MHz, DMSO) δ -73.81 (CF_{3,TFA}), -108.19. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_r 10.087 min (>98%, UV_{215.4}). HRMS m/z 384.0910 ([M+H]⁺, C₁₇H₁₉NO₆FS⁺, calcd. 384.0911).



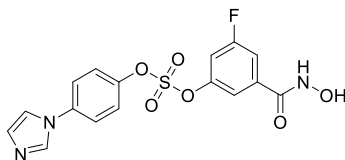
4-(*tert*-Butyl)-2-methylphenyl (3-fluoro-5-(hydroxycarbamoyl)phenyl) sulfate (C19). Using general procedure 4 on a 20 μ mol scale using resin C and 4-(*tert*-butyl)-2-methylphenol, compound **C19** (1.1 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.52 (s, 1H), 9.32 (s, 1H), 7.73–7.67 (m, 2H), 7.63 (dt, J = 8.7, 2.3 Hz, 1H), 7.43 (dd, J = 2.5, 0.9 Hz, 1H), 7.36 (ddd, J = 8.7, 2.6, 0.6 Hz, 1H), 7.31 (d, J = 8.7 Hz, 1H), 2.26 (s, 3H), 1.28 (s, 9H). ¹³C NMR (151 MHz, DMSO) δ 161.9 (d, J = 248.7 Hz), 150.8, 150.0 (d, J = 11.6 Hz), 146.4, 136.3 (d, J = 7.9 Hz), 129.6, 129.1, 124.7, 120.3, 116.0, 113.7 (d, J = 22.9 Hz), 112.3 (d, J = 26.1 Hz), 34.3, 31.0, 15.9. ¹⁹F NMR (376 MHz, DMSO) δ -74.26 (CF_{3,TFA}), -108.26. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_r 10.575 min (>98%, UV_{215.4}). HRMS m/z 398.1068 ([M+H]⁺, C₁₈H₂₁NO₆FS⁺, calcd. 398.1068).



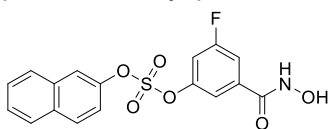
3-Fluoro-5-(hydroxycarbamoyl)phenyl (4-phenoxyphenyl) sulfate (C21). Using general procedure 4 on a 20 μ mol scale using resin C and 4-phenoxyphenol, compound **C21** (2.6 mg, 31 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.53 (s, 1H), 9.32 (br. s, 1H), 7.72–7.67 (m, 2H), 7.65 (dt, J = 8.7, 2.3 Hz, 1H), 7.52–7.46 (m, 2H), 7.46–7.41 (m, 2H), 7.20 (tt, J = 7.3, 1.1 Hz, 1H), 7.15–7.11 (m, 2H), 7.11–7.06 (m, 2H). ¹³C NMR (151 MHz, DMSO) δ 161.93 (d, J = 248.7 Hz), 156.35, 155.89, 150.03 (d, J = 11.5 Hz), 144.85, 136.34 (d, J = 8.1 Hz), 130.26, 124.23, 122.95, 119.67, 119.22, 113.68 (d, J = 23.0 Hz), 112.12 (d, J = 25.7 Hz). Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation via the nearby ¹⁴N-nuclei. ¹⁹F NMR (376 MHz, DMSO) δ -74.33 (CF_{3,TFA}), -108.16. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_r 7.589 min (>98%, UV_{215.4}). Impurity seen around 7.85 min originates from the column. HRMS m/z 420.0546 ([M+H]⁺, C₁₉H₁₅NO₇FS⁺, calcd. 420.0547).



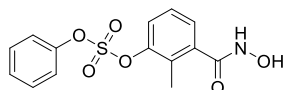
4-(1*H*-imidazol-1-yl)phenyl (3-fluoro-5-(hydroxycarbamoyl)phenyl) sulfate (C23). Using general procedure 4 on a 20 μ mol scale using resin C and 4-(1*H*-imidazol-1-yl)phenol, compound **C23** (2.3 mg, 29 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 11.55 (s, 1H), 9.45 (s, 1H), 8.22 (t, J = 1.7 Hz, 1H), 7.99–7.95 (m, 2H), 7.82–7.76 (m, 3H), 7.74–7.70 (m, 3H). ¹³C NMR (151 MHz, DMSO) δ 162.0 (d, J = 248.7 Hz), 158.1 (q, J = 33.5 Hz, CO_{TFA}), 150.0 (d, J = 11.5 Hz), 149.4, 136.4 (d, J = 8.0 Hz), 135.3, 134.9, 124.3, 123.0, 122.8, 120.6, 115.9, 113.8 (d, J = 23.0 Hz), 112.2 (d, J = 26.2 Hz). ¹⁹F NMR (376 MHz, DMSO) δ -74.14 (CF_{3,TFA}), -108.04. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_r 4.248 min (>98%, UV_{215.4}). HRMS m/z 394.0501 ([M+H]⁺, C₁₆H₁₃N₃O₆FS⁺, calcd. 394.0503).



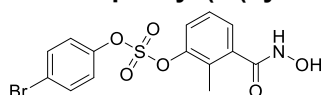
3-Fluoro-5-(hydroxycarbamoyl)phenyl naphthalen-2-yl sulfate (C24): Using general procedure 4 on a 40 μmol scale using resin C and naphthalen-2-ol, compound **C24** (4.3 mg, 29 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.54 (s, 1H), 8.15–8.02 (m, 4H), 7.79–7.48 (m, 6H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 161.9 (d, $J = 248.7$ Hz), 150.0 (d, $J = 11.6$ Hz), 147.3, 136.4 (d, $J = 8.2$ Hz), 133.1, 131.9, 130.8, 128.1, 127.9, 127.5, 127.1, 119.7, 118.7, 115.9, 113.7 (d, $J = 23.1$ Hz), 112.2 (d, $J = 26.2$ Hz). C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation via the nearby ^{14}N -nuclei. ^{19}F NMR of major rotamer (376 MHz, DMSO) δ -74.5 (CF_3 , TFA), -108.1. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.275 min (> 98%, $\text{UV}_{215.4}$). HRMS m/z 378.0458 ($[\text{M}+\text{H}]^+$, $\text{C}_{17}\text{H}_{13}\text{FNO}_6\text{S}^+$, calcd. 378.0442).



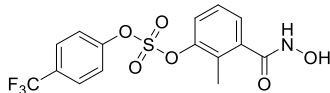
3-(Hydroxycarbamoyl)-2-methylphenyl phenyl sulfate (D1): Using general procedure 4 on a 40 μmol scale using resin D and phenol, compound **D1** (6.5 mg, 50 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.01 (s, 1H), 7.60–7.53 (m, 2H), 7.47 (dtd, $J = 7.1, 3.3, 1.8$ Hz, 4H), 7.41 (t, $J = 7.9$ Hz, 1H), 7.35 (dd, $J = 7.6, 1.3$ Hz, 1H), 2.44 (s, 1H, minor rotamer), 2.24 (s, 2H, major rotamer). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 164.5, 149.8, 148.7, 137.7, 130.6, 128.8, 128.3, 127.6, 127.1, 122.1, 121.2, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 7.209 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 324.0549 ($[\text{M}+\text{H}]^+$, $\text{C}_{14}\text{H}_{14}\text{NO}_6\text{S}^+$, calcd. 324.0536).



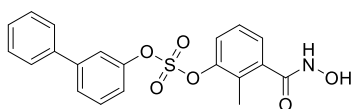
4-Bromophenyl (3-(hydroxycarbamoyl)-2-methylphenyl) sulfate (D2): Using general procedure 4 on a 20 μmol scale using resin D and 4-bromophenol, compound **D2** (1.7 mg, 21 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.00 (d, $J = 1.7$ Hz, 1H), 9.20 (d, $J = 1.7$ Hz, 1H), 7.79–7.73 (m, 2H), 7.50 (dd, $J = 8.3, 1.3$ Hz, 1H), 7.48–7.44 (m, 2H), 7.41 (ddd, $J = 8.2, 7.6, 0.7$ Hz, 1H), 7.35 (dd, $J = 7.6, 1.3$ Hz, 1H), 2.24 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 164.4, 148.9, 148.6, 137.7, 133.5, 128.8, 127.6, 127.1, 123.5, 122.1, 120.8, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 8.382 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 401.9638 ($[\text{M}+\text{H}]^+$, $\text{C}_{14}\text{H}_{13}\text{NO}_6\text{SBr}^+$, calcd. 401.9641).



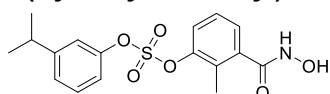
3-(Hydroxycarbamoyl)-2-methylphenyl (4-(trifluoromethyl)phenyl) sulfate (D3): Using general procedure 4 on a 20 μmol scale using resin D and 4-(trifluoromethyl)phenol, compound **D3** (1.8 mg, 23 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.01 (d, $J = 1.7$ Hz, 1H), 9.21 (d, $J = 1.7$ Hz, 1H), 8.00–7.93 (m, 2H), 7.76–7.71 (m, 2H), 7.54 (dd, $J = 8.3, 1.2$ Hz, 1H), 7.42 (ddd, $J = 8.2, 7.6, 0.7$ Hz, 1H), 7.36 (dd, $J = 7.6, 1.2$ Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 164.4, 152.2, 148.6, 137.7, 128.8, 128.6 (q, $J = 32.8$ Hz), 128.1 (q, $J = 3.7$ Hz), 127.7, 127.2, 123.6 (q, $J = 272.5$ Hz), 122.3, 122.1, 12.7. ^{19}F NMR (376 MHz, DMSO) δ -60.98. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 8.652 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 392.0407 ($[\text{M}+\text{H}]^+$, $\text{C}_{15}\text{H}_{13}\text{NO}_7\text{F}_3\text{S}^+$, calcd. 392.0410).



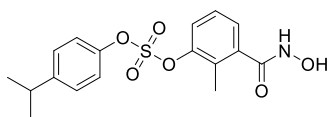
[1,1'-Biphenyl]-3-yl (3-(hydroxycarbamoyl)-2-methylphenyl) sulfate (D4): Using general procedure 4 on a 40 μmol scale using resin D and [1,1'-biphenyl]-3-ol, compound **D4** (3.2 mg, 20 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 10.96 (s, 1H), 7.65–7.60 (m, 1H), 7.59–7.54 (m, 3H), 7.50–7.44 (m, 4H), 7.43–7.39 (m, 1H), 7.31–7.24 (m, 2H), 7.19–7.12 (m, 1H), 2.11 (s, 3H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 164.4, 148.5, 146.8, 137.5, 135.8, 134.5, 131.9, 129.5, 129.1, 128.7, 128.5, 128.5, 128.0, 127.4, 126.9, 121.9, 121.6, 12.5. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.034 min (> 98%, $\text{UV}_{215.4}$). HRMS m/z 400.0867 ($[\text{M}+\text{H}]^+$, $\text{C}_{20}\text{H}_{18}\text{NO}_6\text{S}^+$, calcd. 400.0849).



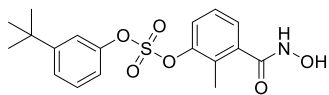
3-(Hydroxycarbamoyl)-2-methylphenyl (3-isopropylphenyl) sulfate (D8): Using general procedure 4 on a 20 μmol scale using resin D and 3-isopropylphenol, compound **D8** (1.5 mg, 20 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 10.99 (s, 1H), 9.19 (br. s, 1H), 7.50–7.43 (m, 2H), 7.42–7.38 (m, 1H), 7.37–7.32 (m, 2H), 7.31–7.25 (m, 2H), 2.97 (hept, $J = 6.9$ Hz, 1H), 2.22 (s, 3H), 1.20 (d, $J = 6.9$ Hz, 6H). ^{13}C NMR (151 MHz, DMSO) δ 164.5, 151.5, 149.9, 148.6, 137.7, 130.3, 128.8, 127.6, 127.0, 126.2, 122.0, 118.9, 118.5, 33.2, 23.5, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.198 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 366.1002 ($[\text{M}+\text{H}]^+$, $\text{C}_{17}\text{H}_{20}\text{NO}_6\text{S}^+$, calcd. 366.1006).



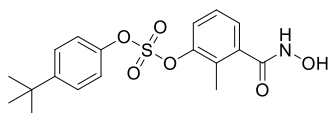
3-(Hydroxycarbamoyl)-2-methylphenyl (4-isopropylphenyl) sulfate (D9). Using general procedure 4 on a 20 μmol scale using resin D and 4-isopropylphenol, compound **D9** (1.7 mg, 23 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.00 (s, 1H), 9.19 (s, 1H), 7.47 (dd, $J = 8.3, 1.3$ Hz, 1H), 7.44–7.32 (m, 6H), 2.96 (hept, $J = 6.9$ Hz, 1H), 2.22 (s, 3H), 1.21 (d, $J = 6.9$ Hz, 6H). ^{13}C NMR (151 MHz, DMSO) δ 164.5, 148.7, 148.6, 147.8, 137.7, 128.8, 128.2, 127.6, 127.0, 122.0, 121.0, 32.9, 23.7, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.320 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 366.1004 ($[\text{M}+\text{H}]^+$, $\text{C}_{17}\text{H}_{20}\text{NO}_7\text{S}^+$, calcd. 366.1006).



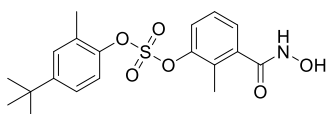
3-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)-2-methylphenyl) sulfate (D11): Using general procedure 4 on a 40 μmol scale using resin D and 3-(tert-butyl)phenol, compound **D11** (6.9 mg, 45 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.00 (s, 1H), 7.53–7.43 (m, 3H), 7.43–7.37 (m, 2H), 7.34 (dd, $J = 7.6, 1.4$ Hz, 1H), 7.28 (ddd, $J = 7.3, 2.5, 1.7$ Hz, 1H), 2.21 (s, 3H), 1.28 (s, 9H). ^{13}C NMR (151 MHz, DMSO) δ 164.5, 153.9, 149.9, 148.7, 137.7, 130.1, 128.8, 127.6, 127.0, 125.1, 122.0, 118.2, 118.0, 34.7, 30.8, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.642 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 380.1177 ($[\text{M}+\text{H}]^+$, $\text{C}_{18}\text{H}_{22}\text{NO}_6\text{S}^+$, calcd. 380.1162).



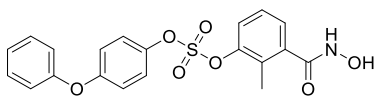
4-(tert-Butyl)phenyl (3-(hydroxycarbamoyl)-2-methylphenyl) sulfate (D12). Using general procedure 4 on a 20 μmol scale using resin D and 4-(tert-butyl)phenol, compound **D12** (2.0 mg, 26 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.00 (s, 1H), 9.19 (br. s, 1H), 7.59–7.53 (m, 2H), 7.47 (dd, $J = 8.3, 1.3$ Hz, 1H), 7.42–7.39 (m, 1H), 7.39–7.35 (m, 2H), 7.34 (dd, $J = 7.6, 1.3$ Hz, 1H), 2.22 (s, 3H), 1.30 (s, 9H). ^{13}C NMR (151 MHz, DMSO) δ 164.5, 150.8, 148.7, 147.6, 137.7, 128.8, 127.6, 127.3, 127.00, 122.0, 120.6, 34.4, 31.0, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.803 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 380.1160 ($[\text{M}+\text{H}]^+$, $\text{C}_{18}\text{H}_{22}\text{NO}_6\text{S}^+$, calcd. 380.1162).



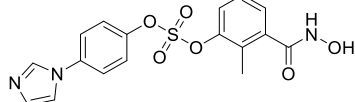
4-(tert-Butyl)-2-methylphenyl (3-(hydroxycarbamoyl)-2-methylphenyl) sulfate (D19): Using general procedure 4 on a 40 μmol scale using resin D and 4-(tert-butyl)-2-methylphenol, compound **D19** (7.1 mg, 45 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.01 (s, 1H), 7.47–7.37 (m, 3H), 7.37–7.32 (m, 2H), 7.27 (d, $J = 8.6$ Hz, 1H), 2.28 (s, 3H), 2.25 (s, 3H), 1.28 (d, $J = 2.2$ Hz, 9H). ^{13}C NMR (151 MHz, DMSO) δ 164.5, 150.7, 148.6, 146.4, 137.7, 129.7, 129.0, 128.9, 127.5, 127.1, 124.6, 122.3, 120.5, 34.3, 31.0, 15.9, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 10.253 min (95%, $\text{UV}_{215.4}$). HRMS m/z 394.1336 ($[\text{M}+\text{H}]^+$, $\text{C}_{19}\text{H}_{24}\text{NO}_6\text{S}^+$, calcd. 394.1319).



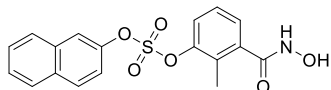
3-(Hydroxycarbamoyl)-2-methylphenyl (4-phenoxyphenyl) sulfate (D21). Using general procedure 4 on a 20 μmol scale using resin D and 4-phenoxyphenol, compound **D21** (1.5 mg, 18 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.00 (s, 1H), 9.20 (br. s, 1H), 7.50–7.46 (m, 3H), 7.45–7.39 (m, 3H), 7.34 (dd, $J = 7.6, 1.3$ Hz, 1H), 7.20 (tt, $J = 7.4, 1.1$ Hz, 1H), 7.16–7.12 (m, 2H), 7.10–7.05 (m, 2H), 2.24 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 164.5, 156.3, 156.0, 148.7, 144.9, 137.7, 130.3, 128.8, 127.6, 127.0, 124.2, 123.0, 122.0, 119.7, 119.1, 12.7. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 9.575 min (>98%, $\text{UV}_{215.4}$). HRMS m/z 416.0796 ($[\text{M}+\text{H}]^+$, $\text{C}_{20}\text{H}_{18}\text{NO}_7\text{S}^+$, calcd. 416.0798).



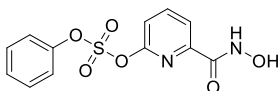
4-(1H-Imidazol-1-yl)phenyl (3-(hydroxycarbamoyl)-2-methylphenyl) sulfate (D23): Using general procedure 4 on a 20 μmol scale using resin D and 4-(1H-imidazol-1-yl)phenol, compound **D23** (4.9 mg, 63 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR, major rotamer (600 MHz, DMSO) δ 11.02 (s, 1H), 9.51 (dt, $J = 24.4, 1.4$ Hz, 1H), 8.25 (dt, $J = 9.4, 1.7$ Hz, 1H), 8.01–7.95 (m, 2H), 7.85–7.72 (m, 4H), 7.52 (ddd, $J = 29.5, 8.3, 1.3$ Hz, 1H), 7.45–7.34 (m, 2H), 2.27 (s, 3H). ^{13}C NMR, major rotamer (151 MHz, DMSO) δ 164.4, 159.9, 149.4, 148.7, 137.7, 135.3, 134.7, 128.8, 127.7, 127.2, 124.3, 123.0, 122.2, 120.7, 12.8. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 3.922 min (97%, $\text{UV}_{215.4}$). HRMS m/z 390.0768 ($[\text{M}+\text{H}]^+$, $\text{C}_{17}\text{H}_{16}\text{N}_3\text{O}_6\text{S}^+$, calcd. 390.0754).



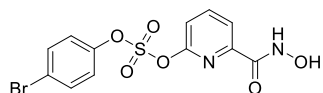
3-(Hydroxycarbamoyl)-2-methylphenyl naphthalen-2-yl sulfate (D24): Using general procedure 4 on a 40 μmol scale using resin D and naphthalen-2-ol, compound **D24** (3.4 mg, 23 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.01 (s, 1H), 8.14–8.11 (m, 1H), 8.09–8.03 (m, 3H), 7.66–7.61 (m, 2H), 7.61–7.52 (m, 2H), 7.42 (t, $J = 7.9$ Hz, 1H), 7.36 (dd, $J = 7.7, 1.2$ Hz, 1H), 2.25 (s, 3H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 164.5, 148.7, 147.3, 137.7, 133.1, 131.8, 130.7, 128.8, 128.0, 127.9, 127.8, 127.6, 127.5, 127.1, 122.2, 119.8, 118.7, 12.8. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.881 min (98%, UV_{215.4}). HRMS m/z 374.0710 ($[\text{M}+\text{H}]^+$, C₁₈H₁₆NO₆S⁺, calcd. 374.0693).



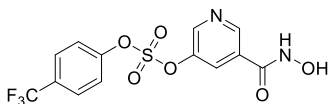
6-(Hydroxycarbamoyl)pyridin-2-yl phenyl sulfate (E1): Using general procedure 4 on a 100 μmol scale using resin F and phenol, compound **E1** (2.9 mg, 9 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.61 (s, 1H), 8.98 (d, $J = 1.7$ Hz, 1H), 8.86 (d, $J = 2.7$ Hz, 1H), 8.20 (t, $J = 2.2$ Hz, 1H), 7.57 (dtd, $J = 9.1, 7.1, 2.1$ Hz, 2H), 7.53–7.45 (m, 3H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 149.8, 147.1, 146.6, 144.9, 130.7, 130.0, 128.4, 127.4, 121.1. C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.140 min (>98%, UV_{215.4}). HRMS m/z 311.0331 ($[\text{M}+\text{H}]^+$, C₁₂H₁₁N₂O₆S⁺, calcd. 311.0332).



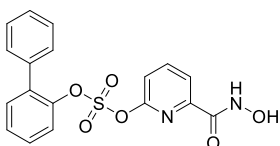
4-Bromophenyl (6-(hydroxycarbamoyl)pyridin-2-yl) sulfate (E2): Using general procedure 4 on a 85 μmol scale using resin F and 4-bromophenol, compound **E2** (9.0 mg, 27 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.61 (s, 1H), 8.99 (d, $J = 1.7$ Hz, 1H), 8.89 (d, $J = 2.7$ Hz, 1H), 8.22 (dd, $J = 2.7, 1.7$ Hz, 1H), 7.80–7.72 (m, 2H), 7.54–7.46 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 160.6, 148.9, 147.2, 146.6, 144.8, 133.5, 130.0, 127.4, 123.5, 121.0. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 5.929 min (>98%, UV_{215.4}). HRMS m/z 388.9439 ($[\text{M}+\text{H}]^+$, C₁₂H₁₀N₂O₆SBr⁺, calcd. 388.9437).



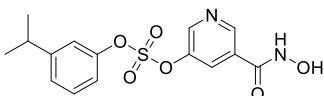
5-(Hydroxycarbamoyl)pyridin-3-yl (4-(trifluoromethyl)phenyl) sulfate (E3): Using general procedure 4 on a 40 μmol scale using resin E and 4-(trifluoromethyl)phenol, compound **E3** (1.5 mg, 10 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.62 (s, 1H), 9.40 (s, 1H), 9.00 (d, $J = 1.9$ Hz, 1H), 8.97–8.91 (m, 1H), 8.25 (t, $J = 2.4$ Hz, 1H), 7.98 (d, $J = 8.5$ Hz, 2H), 7.77 (dd, $J = 12.0, 8.6$ Hz, 2H). ^{13}C NMR (151 MHz, DMSO) δ 160.6, 152.1, 147.4, 146.6, 144.9, 130.1, 128.14 (q, $J = 3.8$ Hz), 127.5, 122.3, 122.2. ^{19}F NMR (376 MHz, DMSO) δ -61.0, -73.9 (CF₃, TFA). Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 7.765 min (96%, UV_{215.4}). HRMS m/z 379.0221 ($[\text{M}+\text{H}]^+$, C₁₃H₁₀N₂O₆F₃S⁺, calcd. 379.0206).



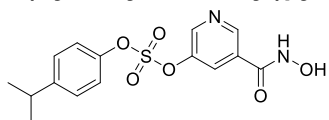
[1,1'-Biphenyl]-2-yl (6-(hydroxycarbamoyl)pyridin-2-yl) sulfate (E4): Using general procedure 4 on a 85 μmol scale using resin F and [1,1'-biphenyl]-2-ol, compound **E4** (13.8 mg, 42 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.57 (s, 1H), 8.92 (d, $J = 1.7$ Hz, 1H), 8.62 (d, $J = 2.7$ Hz, 1H), 8.01 (dd, $J = 2.7, 1.7$ Hz, 1H), 7.72–7.66 (m, 1H), 7.62–7.51 (m, 3H), 7.44–7.41 (m, 2H), 7.41–7.37 (m, 2H), 7.37–7.33 (m, 1H). ^{13}C NMR (151 MHz, DMSO) δ 146.9, 146.7, 146.5, 144.5, 135.5, 134.5, 132.0, 129.8, 129.6, 129.0, 128.8, 128.4, 128.0, 127.0, 121.7. C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.391 min (>98%, UV_{215.4}). HRMS m/z 387.0648 ($[\text{M}+\text{H}]^+$, C₁₈H₁₅N₂O₆S⁺, calcd. 387.0645).



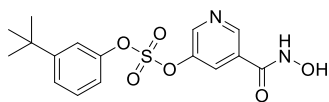
5-(Hydroxycarbamoyl)pyridin-3-yl (3-isopropylphenyl) sulfate (E8): Using general procedure 4 on a 40 μmol scale using resin E and 3-isopropylphenol, compound **E8** (2.1 mg, 15 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.62 (s, 1H), 8.95 (dd, $J = 1.7$ Hz, 1H, both rotamers), 8.78 (d, $J = 2.7$ Hz, 1H, both rotamers), 8.19 (dd, $J = 2.7, 1.7$ Hz, 1H, major rotamer), 7.99 (dd, $J = 2.7, 1.7$ Hz, 0H, minor rotamer), 7.50–7.43 (m, 1H), 7.38–7.26 (m, 3H), 3.02–2.89 (m, 1H), 1.19 (dd, $J = 16.6, 6.9$ Hz, 6H). ^{13}C NMR (151 MHz, DMSO) δ 157.2, 151.7, 149.9, 147.1, 146.7, 144.9, 130.4, 126.4, 124.4, 118.9, 118.3, 33.2, 23.5. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.524 min (>98%, UV_{215.4}). HRMS m/z 353.0814 ($[\text{M}+\text{H}]^+$, C₁₅H₁₇N₂O₆S⁺, calcd. 353.0801).



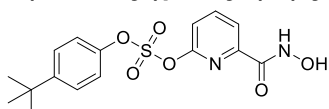
5-(Hydroxycarbamoyl)pyridin-3-yl (4-isopropylphenyl) sulfate (E9): Using general procedure 4 on a 40 μmol scale using resin E and 4-isopropylphenol, compound **E9** (2.6 mg, 18 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.61 (s, 1H), 9.38 (s, 1H), 8.98 (d, $J = 1.7$ Hz, 1H), 8.86 (d, $J = 2.7$ Hz, 1H), 8.20 (dd, $J = 2.7, 1.7$ Hz, 1H), 7.45–7.36 (m, 4H), 3.01–2.90 (m, 1H), 1.20 (dd, $J = 14.2, 6.9$ Hz, 6H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 158.2, 157.2, 148.8, 147.8, 147.1, 146.7, 128.3, 124.3, 120.9, 32.9, 23.7. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.609 min (97%, UV_{215.4}). HRMS m/z 353.0817 ($[\text{M}+\text{H}]^+$, C₁₅H₁₇N₂O₆S⁺, calcd. 353.0801).



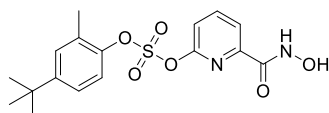
3-(tert-Butyl)phenyl (5-(hydroxycarbamoyl)pyridin-3-yl) sulfate (E11): Using general procedure 4 on a 40 μmol scale using resin E and 3-(tert-butyl)phenol, compound **E11** (2.2 mg, 15 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.61 (s, 1H), 9.39 (s, 1H), 8.98 (d, $J = 1.8$ Hz, 1H), 8.84 (d, $J = 2.7$ Hz, 1H), 8.18 (t, $J = 2.2$ Hz, 1H), 7.53–7.44 (m, 2H), 7.38–7.28 (m, 2H), 1.28 (s, 9H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 154.0, 149.8, 147.0, 146.7, 144.8, 130.2, 130.0, 127.3, 125.3, 118.0, 117.9, 34.7, 30.8. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_{R} 8.771 min (>98%, UV_{215.4}). HRMS m/z 367.0972 ($[\text{M}+\text{H}]^+$, C₁₆H₁₉N₂O₆S⁺, calcd. 367.0958).



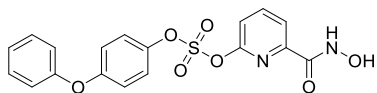
4-(tert-Butyl)phenyl (6-(hydroxycarbamoyl)pyridin-2-yl) sulfate (E12): Using general procedure 4 on a 100 μmol scale using resin F and 4-(tert-butyl) phenol, compound **E12** (4.8 mg, 13 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.61 (s, 1H), 8.98 (d, $J = 1.7$ Hz, 1H), 8.86 (d, $J = 2.7$ Hz, 1H), 8.20 (t, $J = 2.2$ Hz, 1H), 7.59–7.52 (m, 2H), 7.43–7.36 (m, 2H), 1.30 (s, 9H). ^{13}C NMR (151 MHz, DMSO) δ 151.0, 147.6, 147.1, 146.7, 144.9, 130.0, 127.4 (2C), 120.5, 34.5, 31.0. C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.040 min (>98%, UV_{215.4}). HRMS m/z 367.0959 ($[\text{M}+\text{H}]^+$, C₁₆H₁₉N₂O₆S⁺, calcd. 367.0958).



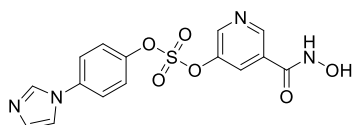
4-(tert-Butyl)-2-methylphenyl (6-(hydroxycarbamoyl)pyridin-2-yl) sulfate (E19): Using general procedure 4 on a 85 μmol scale using resin F and 4-(tert-butyl)-2-methylphenol, compound **E19** (1.0 mg, 3 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.61 (s, 1H), 8.99 (d, $J = 1.7$ Hz, 1H), 8.87 (d, $J = 2.7$ Hz, 1H), 8.21 (dd, $J = 2.7, 1.7$ Hz, 1H), 7.44 (dt, $J = 2.2, 0.8$ Hz, 1H), 7.39–7.31 (m, 2H), 2.25 (d, $J = 0.7$ Hz, 3H), 1.29 (s, 9H). ^{13}C NMR (151 MHz, DMSO) δ 160.6, 150.9, 147.2, 146.6, 146.4, 145.0, 129.9, 129.5, 129.1, 127.5, 124.7, 120.3, 34.3, 31.0, 15.9. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.307 min (96%, UV_{215.4}). HRMS m/z 381.1115 ($[\text{M}+\text{H}]^+$, C₁₇H₂₁N₂O₆S⁺, calcd. 381.1114).



6-(Hydroxycarbamoyl)pyridin-2-yl (4-phenoxyphenyl) sulfate (E21): Using general procedure 4 on a 85 μmol scale using resin F and 4-phenoxyphenol, compound **E21** (1.2 mg, 4 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 11.62 (s, 1H), 8.98 (d, $J = 1.7$ Hz, 1H), 8.87 (d, $J = 2.7$ Hz, 1H), 8.21–8.17 (m, 1H), 7.55–7.48 (m, 2H), 7.46–7.40 (m, 2H), 7.24–7.16 (m, 1H), 7.15–7.11 (m, 2H), 7.11–7.06 (m, 2H). ^{13}C NMR (151 MHz, DMSO) δ 156.4, 155.9, 147.1, 146.7, 144.8, 130.3, 130.0, 127.4, 124.3, 123.0, 122.9, 119.7, 119.2. C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 6.854 min (97%, UV_{215.4}). HRMS m/z 403.0597 ($[\text{M}+\text{H}]^+$, C₁₈H₁₅N₂O₇S⁺, calcd. 403.0594).



4-(1H-imidazol-1-yl)phenyl (5-(hydroxycarbamoyl)pyridin-3-yl) sulfate (E23): Using general procedure 4 on a 40 μmol scale using resin E and 4-(1H-imidazol-1-yl)phenol, compound **E23** (1.7 mg, 11 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 11.65 (s, 0H), 9.48 (d, $J = 11.5$ Hz, 1H), 8.97 (dd, $J = 35.4, 1.7$ Hz, 1H), 8.86 (dd, $J = 75.5, 2.7$ Hz, 1H), 8.27–8.19 (m, 1H), 8.04 (dd, $J = 2.7, 1.7$ Hz, 1H), 8.02–7.94 (m,



2H), 7.86–7.78 (m, 3H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 157.3, 149.5, 147.3, 146.8, 145.4, 142.9, 134.9, 130.1, 127.5, 126.3, 124.5, 122.9. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 3.525 min (>98%, UV_{215.4}). HRMS *m/z* 377.0565 ([M+H]⁺, C₁₅H₁₃N₄O₆S⁺, calcd. 377.0550).

5-(Hydroxycarbamoyl)pyridin-3-yl naphthalen-2-yl sulfate (E24): Using general procedure 4 on a 40 μmol scale using resin E and naphthalene-2-ol, compound **E24** (2.7 mg, 19 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR of major rotamer (600 MHz, DMSO) δ 11.63 (s, 1H), 9.00 (d, *J* = 1.7 Hz, 1H), 8.93 (d, *J* = 2.7 Hz, 1H), 8.28 (dd, *J* = 2.7, 1.8 Hz, 1H), 8.16–8.11 (m, 2H), 8.09–8.03 (m, 2H), 7.68–7.57 (m, 3H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 147.3, 147.2, 146.7, 144.9, 133.1, 131.9, 130.8, 130.0, 128.1, 127.9, 127.5, 127.5, 127.2, 119.7, 118.7. Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 15 min (C18), t_R 7.831 min (>98%, UV_{215.4}). HRMS *m/z* 383.0326 ([M+Na]⁺, C₁₆H₁₂N₂O₆SNa⁺, calcd. 383.0308).

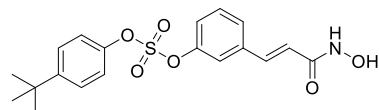
(E)-3-(3-(Hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl phenyl sulfate (F1): Using general procedure 4 on a 100 μmol scale using resin F and phenol, compound **F1** (4.8 mg, 14 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR of major rotamer (600 MHz, DMSO) δ 10.81 (s, 1H), 7.79–7.72 (m, 2H), 7.71–7.64 (m, 2H), 7.58 (td, *J* = 8.2, 1.3 Hz, 1H), 7.54–7.43 (m, 5H), 6.57–6.52 (m, 1H). ¹³C NMR (151 MHz, DMSO) δ 150.2, 149.0, 137.6, 136.5, 133.4, 131.1, 127.1, 123.4, 121.6, 121.4, 120.8, 119.7. Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.774 min (>98%, UV_{215.4}). HRMS *m/z* 336.0539 ([M+H]⁺, C₁₅H₁₄NO₆S⁺, calcd. 336.0536).

(E)-4-Bromophenyl (3-(3-(hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl) sulfate (F2): Using general procedure 4 on a 100 μmol scale using resin F and 4-bromophenol, compound **F2** (6.4 mg, 16 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR of major rotamer (600 MHz, DMSO) δ 10.80 (s, 1H), 7.69–7.61 (m, 2H), 7.60–7.53 (m, 3H), 7.53–7.47 (m, 1H), 7.47–7.42 (m, 1H), 7.39–7.32 (m, 2H), 6.54 (d, *J* = 15.8 Hz, 1H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 150.7, 150.3, 147.7, 138.9, 137.5, 131.0, 127.3, 127.0, 121.6, 121.3, 120.5, 119.6. Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 6.900 min (>98%, UV_{215.4}). HRMS *m/z* 413.9645 ([M+H]⁺, C₁₅H₁₃NO₆SBr⁺, calcd. 413.9641).

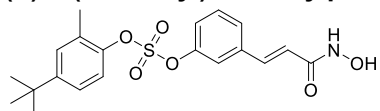
(E)-3-(3-(Hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl (4-(trifluoromethyl)phenyl) sulfate (F3): Using general procedure 4 on a 40 μmol scale using resin F and 4-(trifluoromethyl)phenol, compound **F3** (3.5 mg, 22 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR of major rotamer (600 MHz, DMSO) δ 10.81 (s, 1H), 7.99–7.92 (m, 2H), 7.73 (dt, *J* = 9.5, 2.0 Hz, 2H), 7.70–7.64 (m, 2H), 7.59 (t, *J* = 8.0 Hz, 1H), 7.54–7.46 (m, 2H), 6.55 (d, *J* = 15.8 Hz, 1H). ¹³C NMR of major rotamer (151 MHz, DMSO) δ 162.2, 152.2, 150.2, 137.6, 136.5, 131.1, 128.57 (q, *J* = 32.5 Hz), 128.06 (q, *J* = 3.5 Hz), 127.1, 123.58 (q, *J* = 272.3 Hz), 122.2, 121.7, 121.4, 119.8. ¹⁹F NMR (376 MHz, DMSO) δ -61.0, -73.4 (CF₃, TFA). Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.058 min (>98%, UV_{215.4}). HRMS *m/z* 404.0416 ([M+H]⁺, C₁₆H₁₃NO₆F₃S⁺, calcd. 404.0410).

(E)-[1,1'-Biphenyl]-2-yl (3-(3-(hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl) sulfate (F4): Using general procedure 4 on a 100 μmol scale using resin F and [1,1'-biphenyl]-2-ol, compound **F4** (5.3 mg, 12 %) was isolated as a colorless fluffy powder after lyophilization. ¹H NMR (600 MHz, DMSO) δ 10.81 (s, 1H), 7.64 (dt, *J* = 8.1, 1.4 Hz, 1H), 7.60–7.52 (m, 4H), 7.50–7.37 (m, 8H), 7.22 (dd, *J* = 8.2, 2.4 Hz, 1H), 6.49 (d, *J* = 15.8 Hz, 1H). ¹³C NMR (151 MHz, DMSO) δ 150.1, 146.8, 137.4, 135.7, 134.4, 131.9, 130.9, 129.6, 129.0, 128.5, 128.4, 128.0, 126.9, 121.5, 121.4, 121.2, 119.4. Peak for C(=O)NH was not visible in ¹³C NMR, probably due to fast quadrupolar relaxation vi the nearby ¹⁴N-nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_R 7.245 min (>98%, UV_{215.4}). HRMS *m/z* 412.0850 ([M+H]⁺, C₂₁H₁₈NO₆S⁺, calcd. 412.0849).

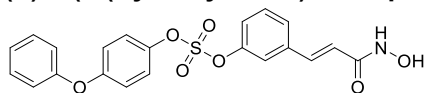
(E)-3-(tert-Butyl)phenyl (3-(3-(hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl) sulfate (F11): Using general procedure 4 on a 40 μmol scale using resin F and 3-(tert-butyl) phenol, compound **F11** (4.2 mg, 27 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 10.80 (s, 1H), 7.65 (dt, J = 7.8, 1.3 Hz, 1H), 7.62–7.56 (m, 2H), 7.50 (d, J = 8.4 Hz, 1H), 7.49–7.46 (m, 2H), 7.43 (ddd, J = 8.2, 2.6, 0.9 Hz, 1H), 7.28 (td, J = 3.1, 1.8 Hz, 2H), 6.53 (d, J = 15.8 Hz, 1H), 1.26 (s, 9H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 153.8, 150.3, 149.9, 137.5, 136.5, 131.0, 130.1, 127.0, 125.0, 121.6, 121.3, 119.5, 118.0, 117.8, 34.7, 30.8. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.673 min (>98%, UV_{215.4}). HRMS m/z 392.1162 ($[\text{M}+\text{H}]^+$, C₁₉H₂₂NO₆S⁺, calcd. 392.1162).



(E)-4-(tert-Butyl)phenyl (3-(3-(hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl) sulfate (F12): Using general procedure 4 on a 40 μmol scale using resin F and 4-(tert-butyl) phenol, compound **F12** (4.2 mg, 27 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR of major rotamer (600 MHz, DMSO) δ 7.69–7.61 (m, 2H), 7.60–7.53 (m, 3H), 7.53–7.48 (m, 1H), 7.44 (ddd, J = 8.2, 2.6, 1.0 Hz, 1H), 7.39–7.32 (m, 2H), 6.54 (d, J = 15.9 Hz, 1H), 1.30 (s, 9H). ^{13}C NMR of major rotamer (151 MHz, DMSO) δ 162.2, 150.7, 150.3, 147.6, 137.5, 136.5, 131.0, 127.3, 127.0, 121.6, 121.3, 120.5, 119.6, 34.4, 31.0. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.794 min (>98%, UV_{215.4}). HRMS m/z 414.0984 ($[\text{M}+\text{Na}]^+$, C₁₉H₂₁NO₆SNa⁺, calcd. 414.0981).



(E)-4-(tert-Butyl)-2-methylphenyl (3-(3-(hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl) sulfate (F19): Using general procedure 4 on a 40 μmol scale using resin F and 4-(tert-butyl)-2-methylphenol, compound **F19** (1.3 mg, 8 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 10.79 (s, 1H), 7.68–7.62 (m, 2H), 7.58 (t, J = 7.9 Hz, 1H), 7.53–7.47 (m, 1H), 7.45 (ddd, J = 8.1, 2.6, 1.0 Hz, 1H), 7.43–7.39 (m, 1H), 7.37–7.33 (m, 1H), 7.29 (dd, J = 8.6, 2.0 Hz, 1H), 6.54 (d, J = 15.8 Hz, 1H), 2.24 (s, 3H), 1.28 (s, 9H). ^{13}C NMR (151 MHz, DMSO) δ 150.6, 150.2, 146.4, 137.4, 136.5, 131.0, 129.6, 129.0, 127.1, 124.7, 121.8, 121.3, 120.3, 119.7, 34.3, 31.0, 15.9. C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 8.116 min (95%, UV_{215.4}). HRMS m/z 406.1322 ($[\text{M}+\text{H}]^+$, C₂₀H₂₄NO₆S⁺, calcd. 406.1319).



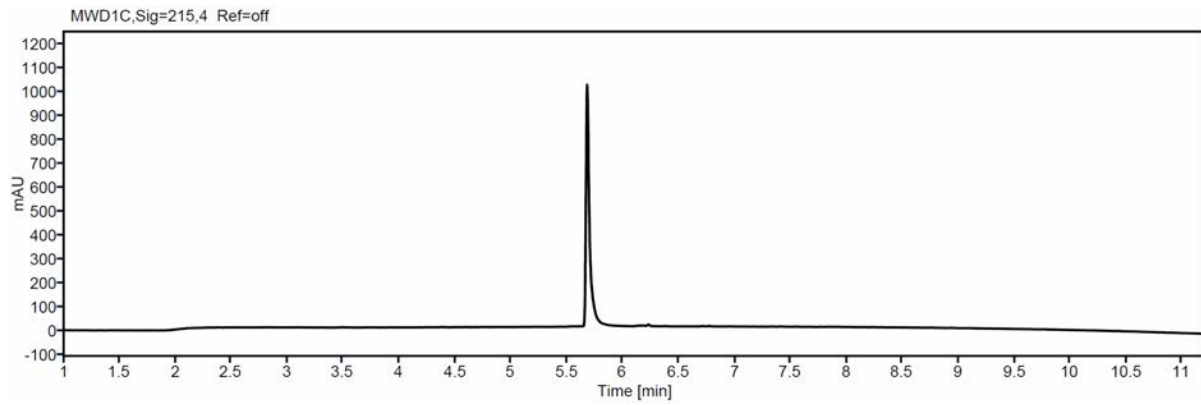
(E)-3-(3-(Hydroxyamino)-3-oxoprop-1-en-1-yl)phenyl (4-phenoxyphenyl) sulfate (F21): Using general procedure 4 on a 100 μmol scale using resin F and 4-phenoxyphenol, compound **F21** (7.6 mg, 17 %) was isolated as a colorless fluffy powder after lyophilization. ^1H NMR (600 MHz, DMSO) δ 10.80 (s, 1H), 7.65 (d, J = 8.2 Hz, 2H), 7.58 (t, J = 7.8 Hz, 1H), 7.54–7.40 (m, 6H), 7.20 (t, J = 7.4 Hz, 1H), 7.15–7.10 (m, 2H), 7.10–7.05 (m, 2H), 6.54 (d, J = 15.7 Hz, 1H). ^{13}C NMR (151 MHz, DMSO) δ 156.2, 155.9, 150.3, 144.9, 137.5, 131.0, 130.3, 127.0, 124.2, 122.9, 121.6, 121.4, 119.6, 119.6, 119.2. Peak for C(=O)NH was not visible in ^{13}C NMR, probably due to fast quadrupolar relaxation vi the nearby ^{14}N -nuclei. Analytical UPLC gradient 0–95% eluent II in eluent I over 10 min (C18), t_{R} 7.613 min (>98%, UV_{215.4}). HRMS m/z 428.0808 ($[\text{M}+\text{H}]^+$, C₂₁H₁₈NO₇S⁺, calcd. 428.0798).

Supplementary references

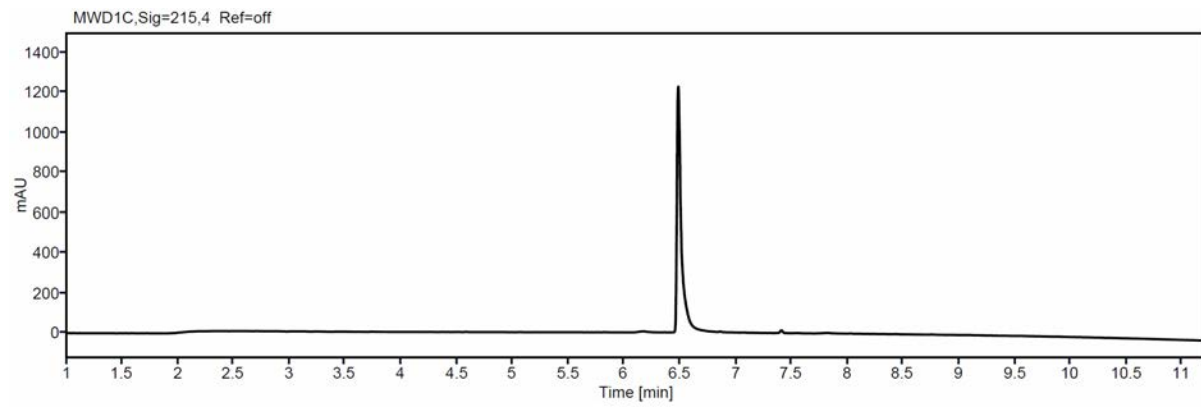
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2. Bradner, J. E.; West, N.; Grachan, M. L.; Greenberg, E. F.; Haggarty, S. J.; Warnow, T.; Mazitschek, R., Chemical Phylogenetics of Histone Deacetylases. *Nat. Chem. Biol.* **2010**, *6* (3), 238-243.
3. Moreno-Yruela, C.; Galleano, I.; Madsen, A. S.; Olsen, C. A., Histone Deacetylase 11 Is an ϵ -N-Myristoyllysine Hydrolase. *Cell Chem. Biol.* **2018**, *25* (7), 849-856.e8.
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Analytical UPLC Chromatograms of Final Compounds

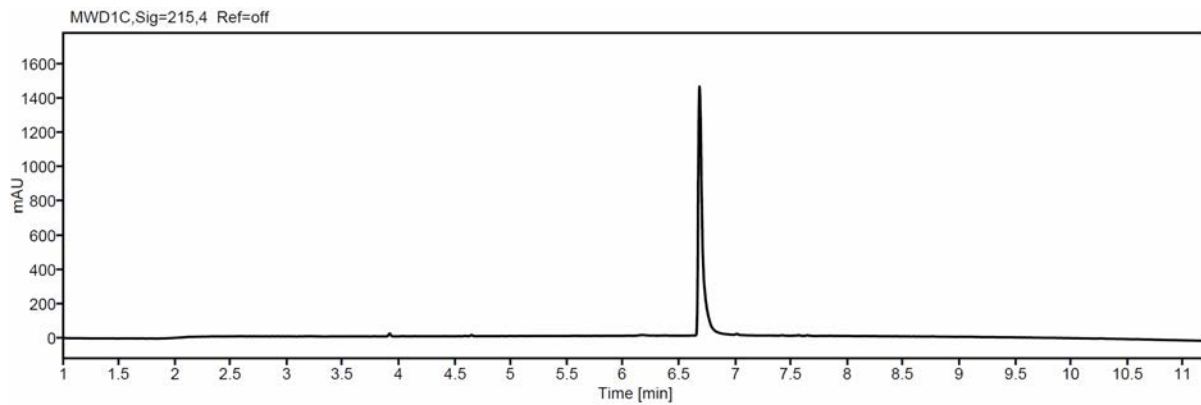
Analytical UPLC chromatogram of **A1**



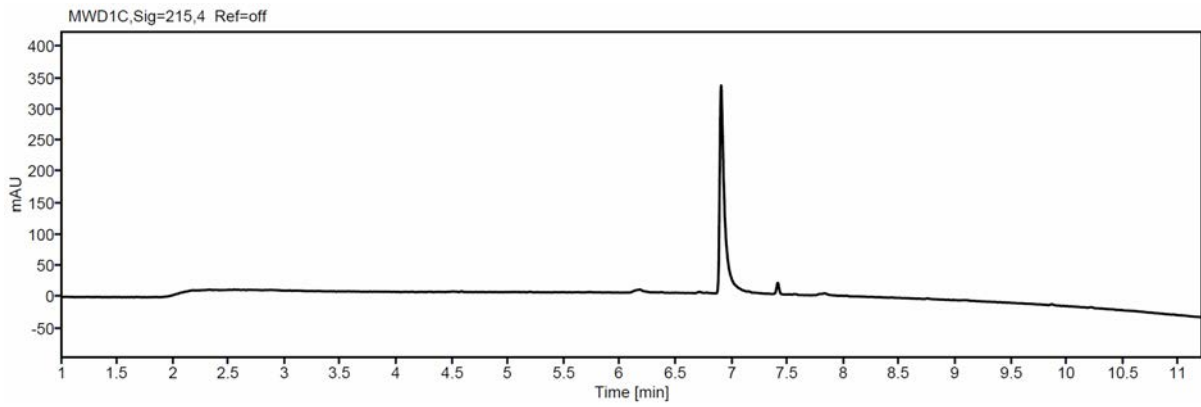
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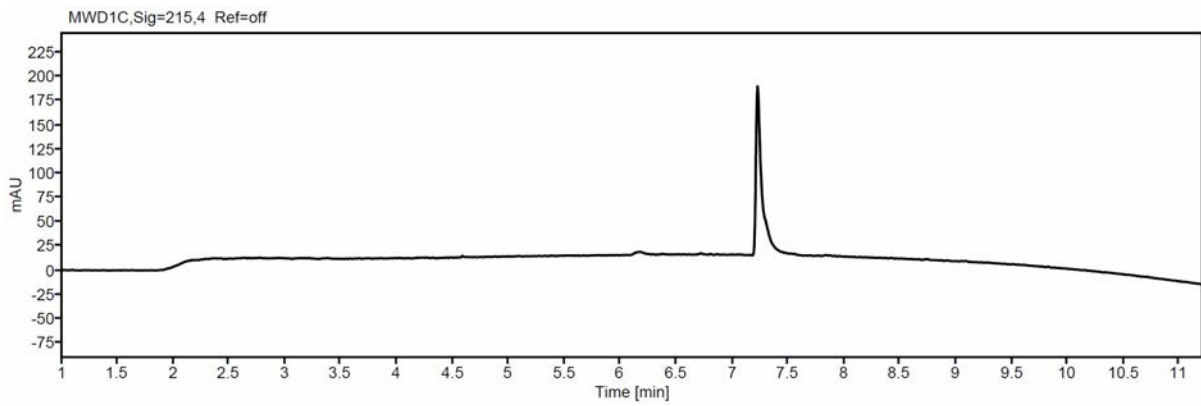
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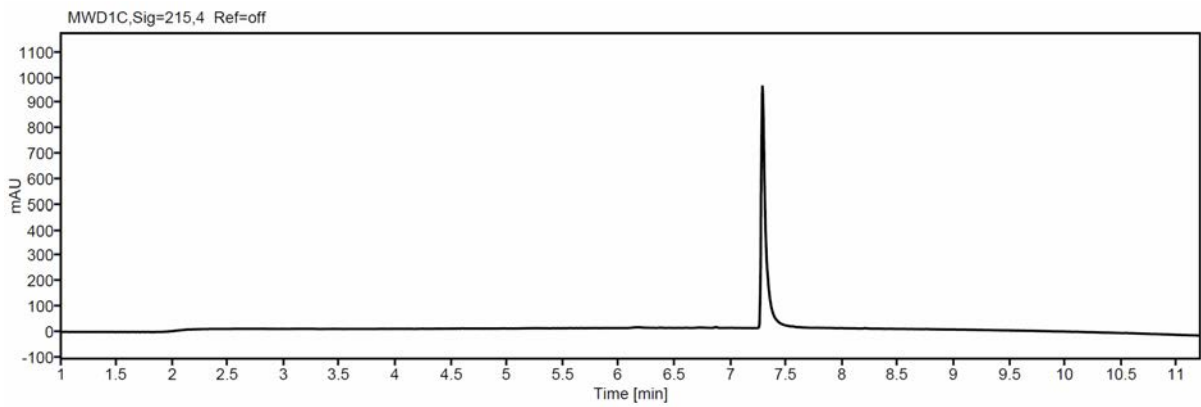
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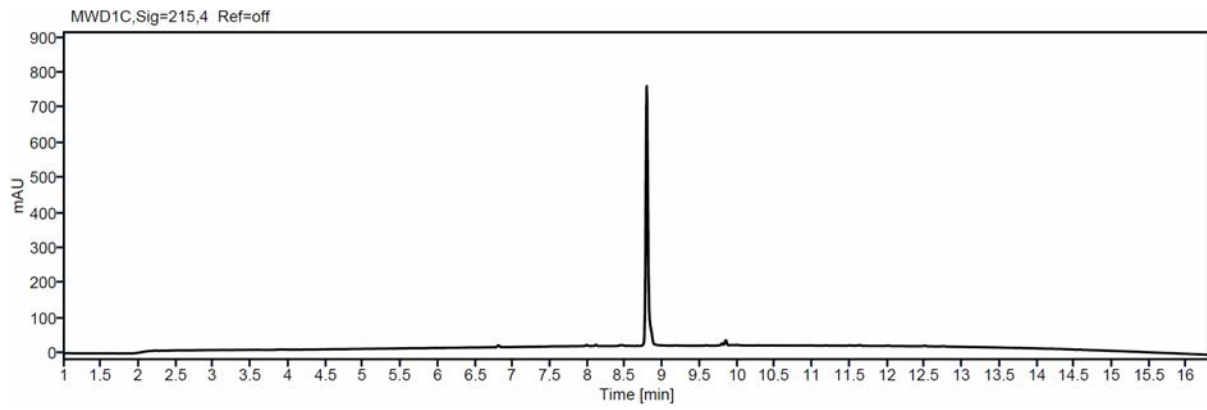
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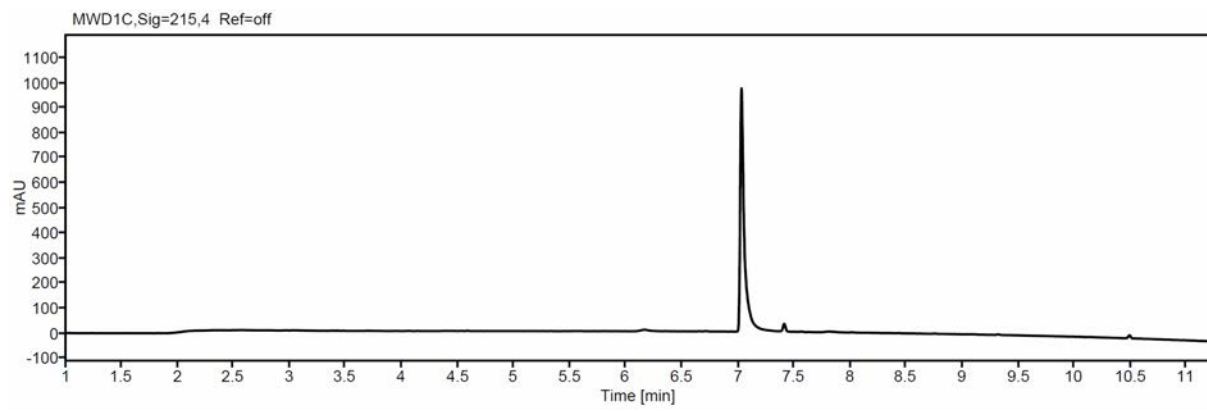
Analytical UPLC chromatogram of **A6**



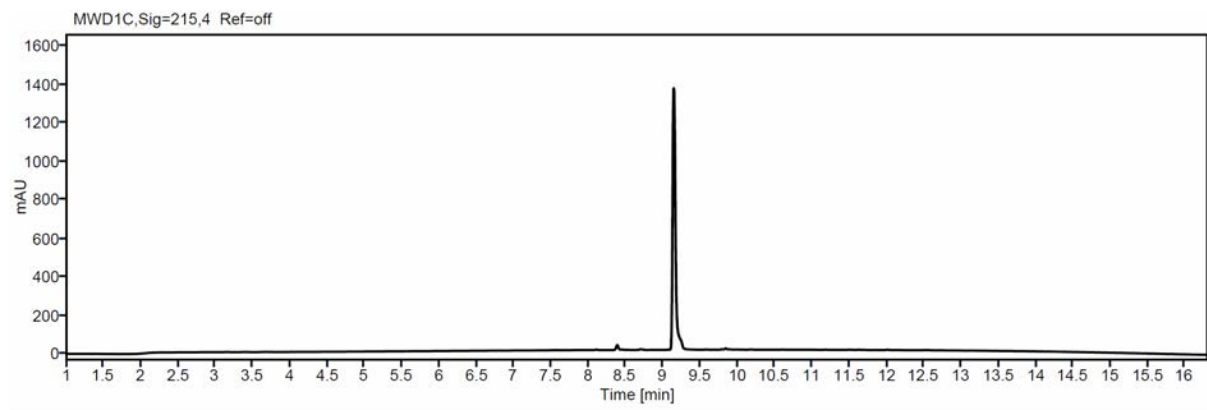
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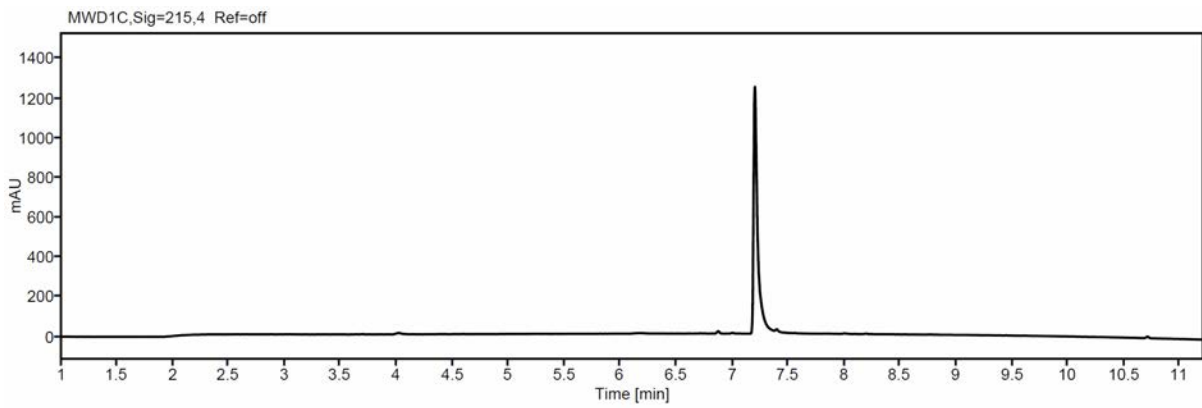
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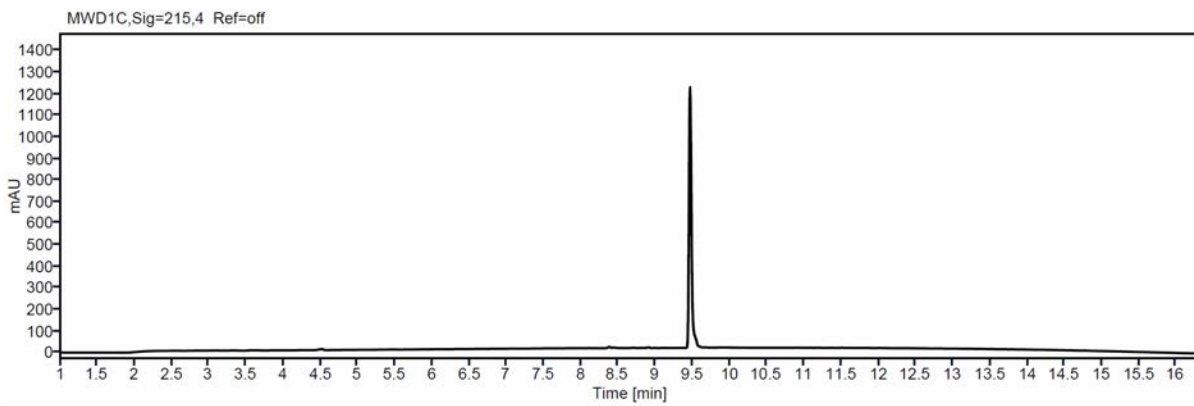
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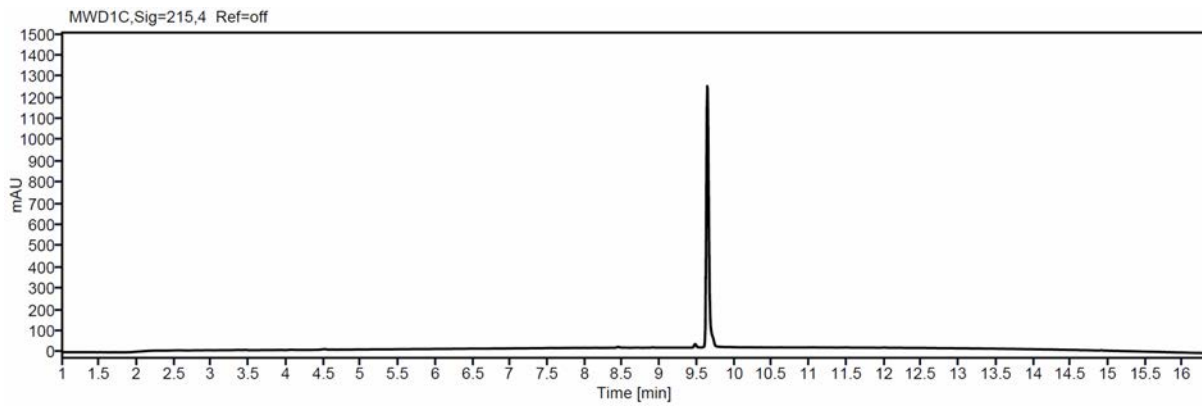
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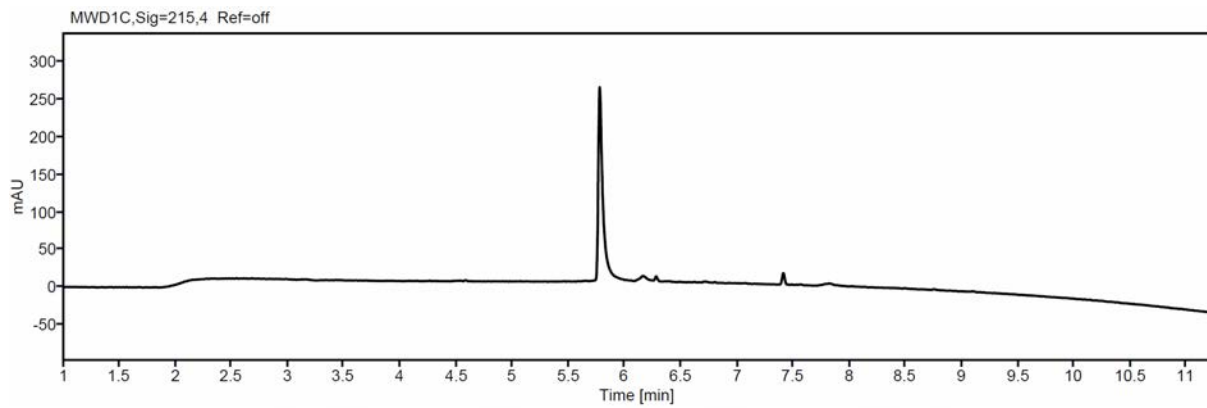
Analytical UPLC chromatogram of **A11**



Analytical UPLC chromatogram of **A12**

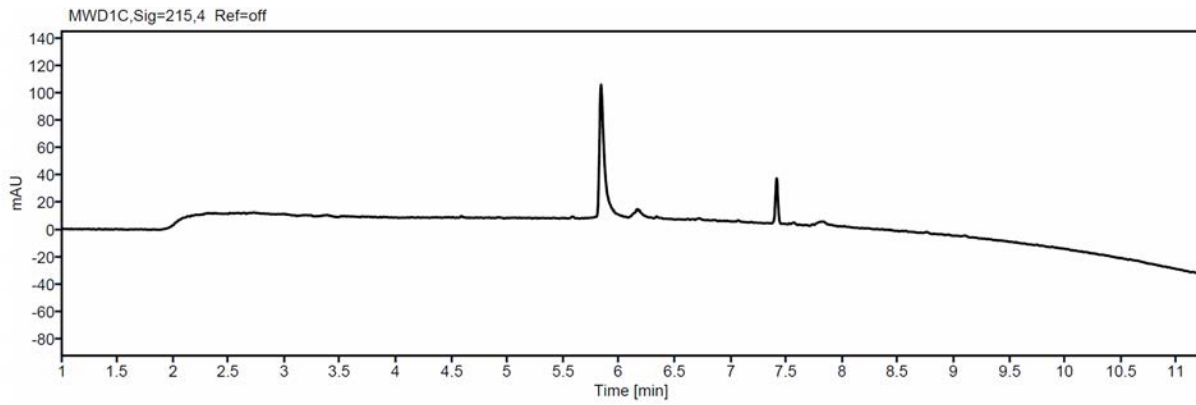


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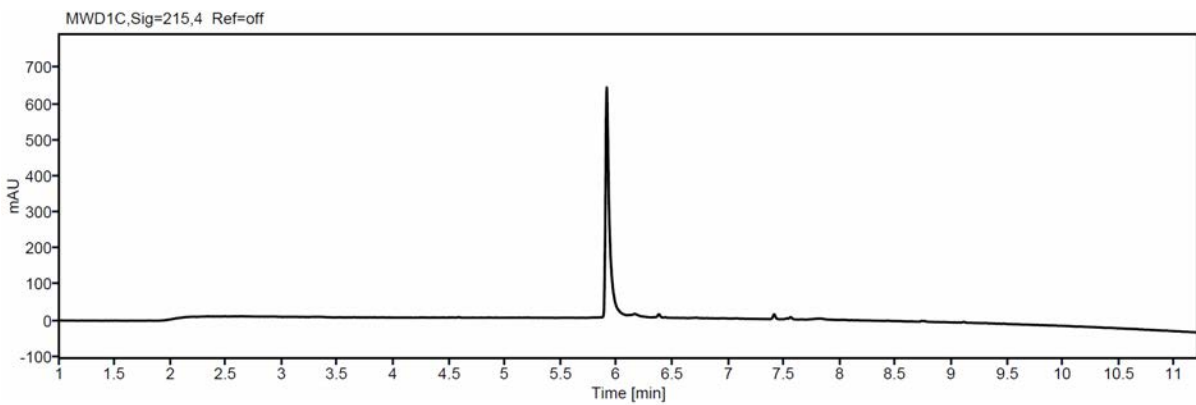
*Peak at 6.2 and ~7.45 min arises from an impurity on the column.

Analytical UPLC chromatogram of **A14**

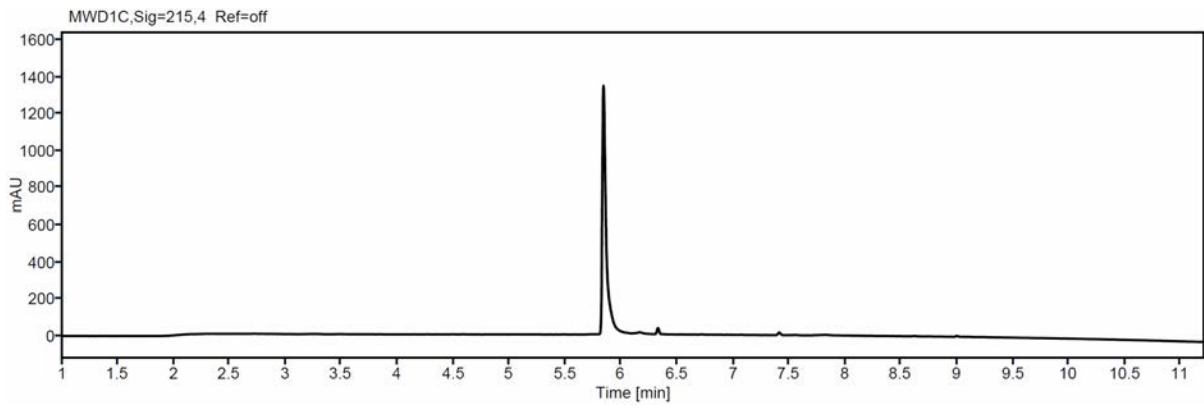


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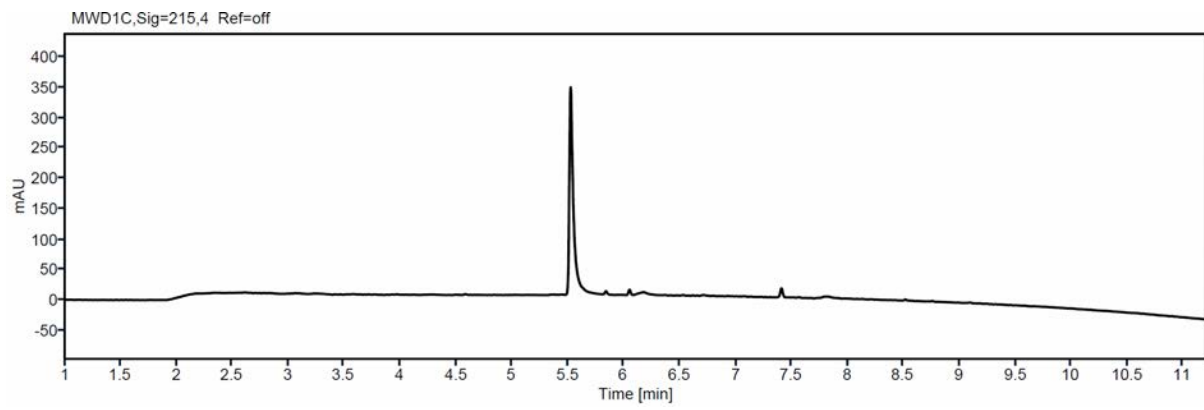
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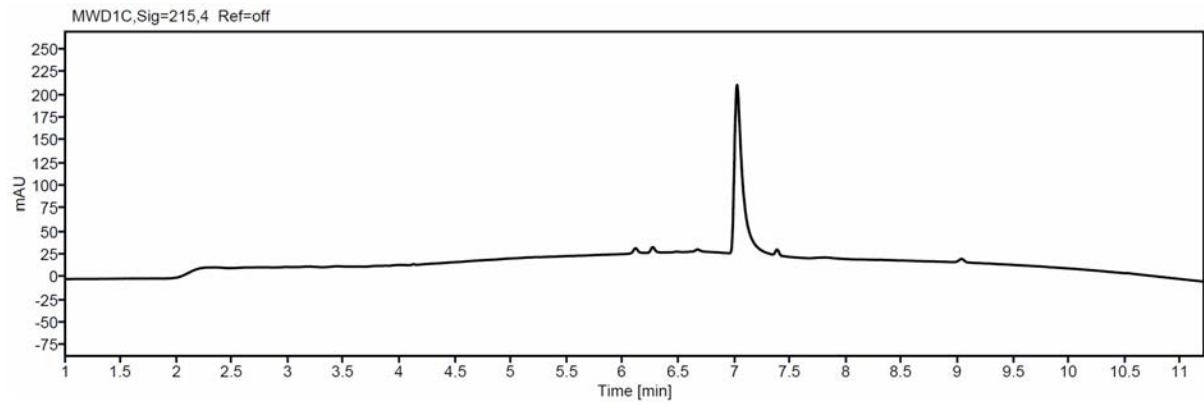
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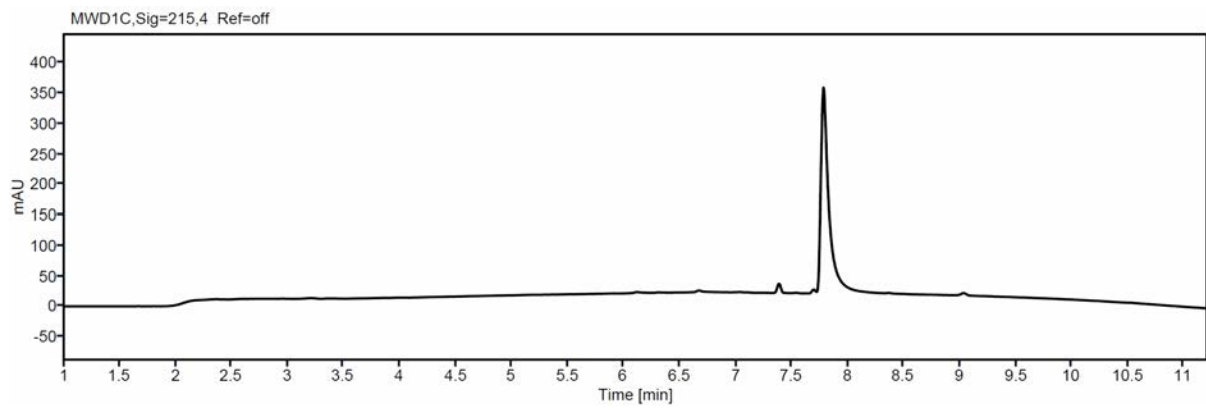
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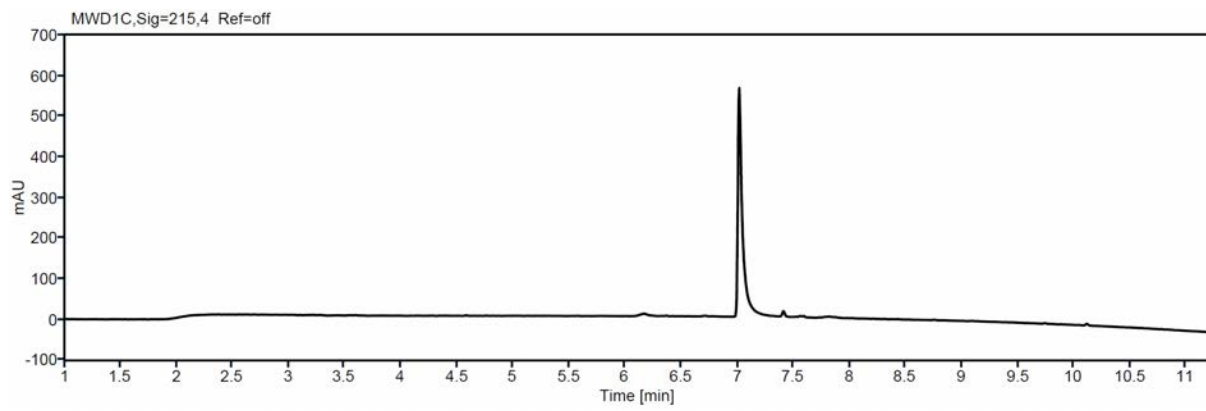
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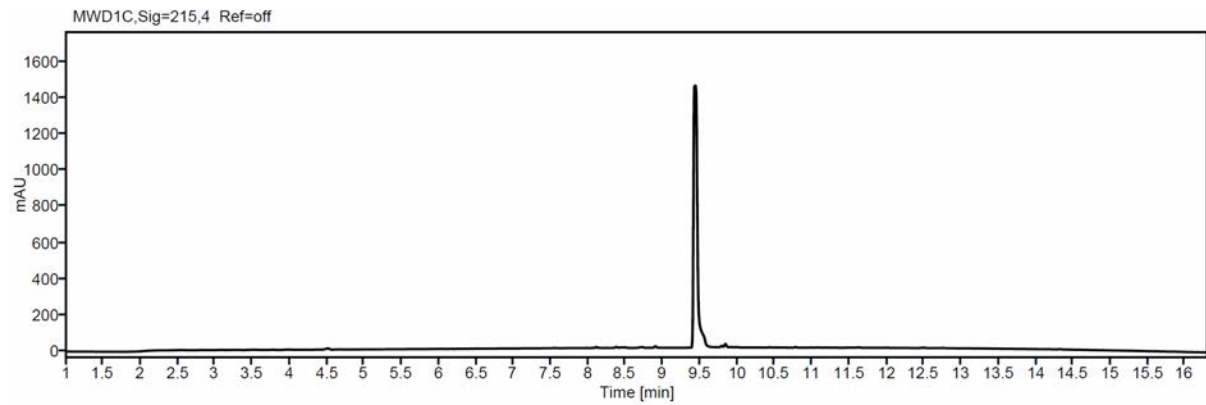
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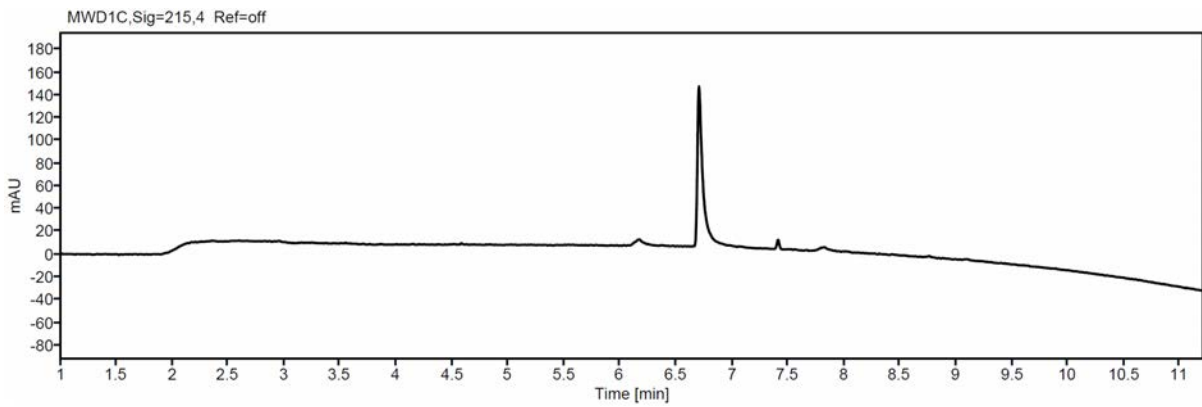
Analytical UPLC chromatogram of **A20**



Analytical UPLC chromatogram of **A21**

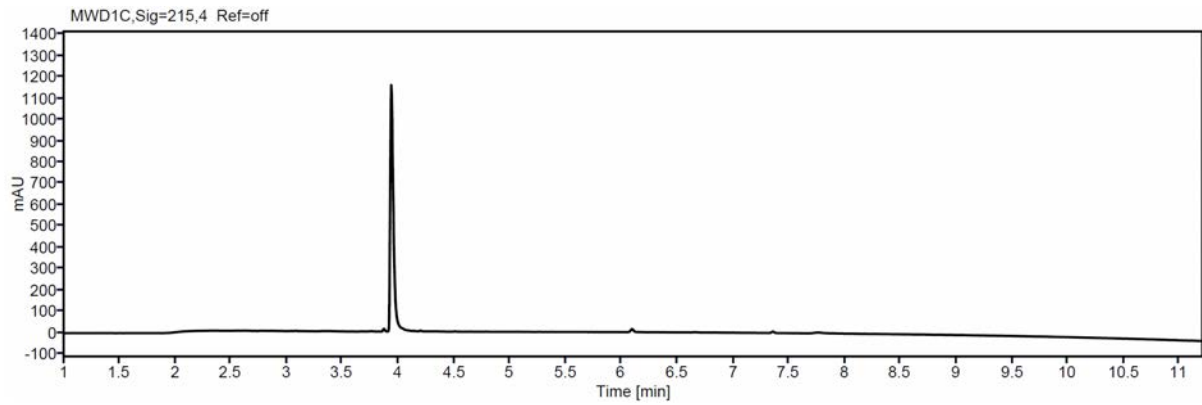


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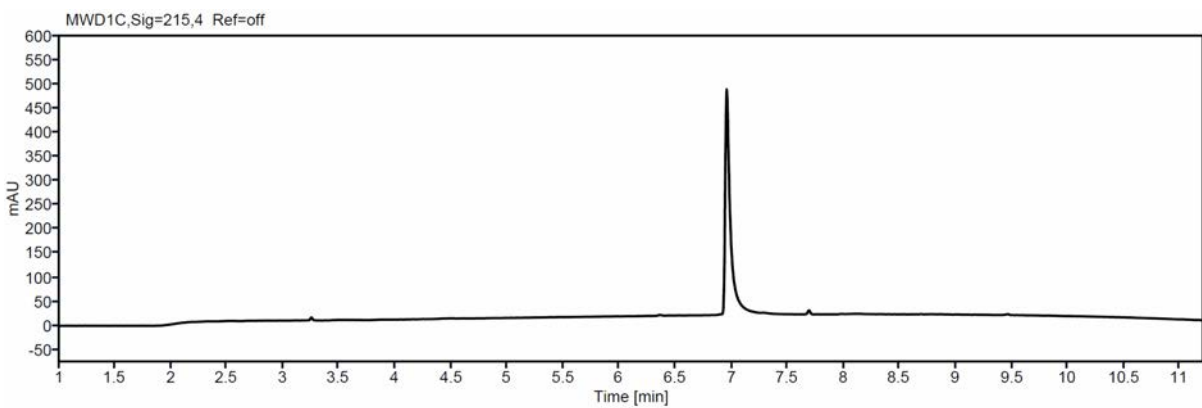


*Peak at 6.2 and ~7.45 min arises from an impurity on the column.

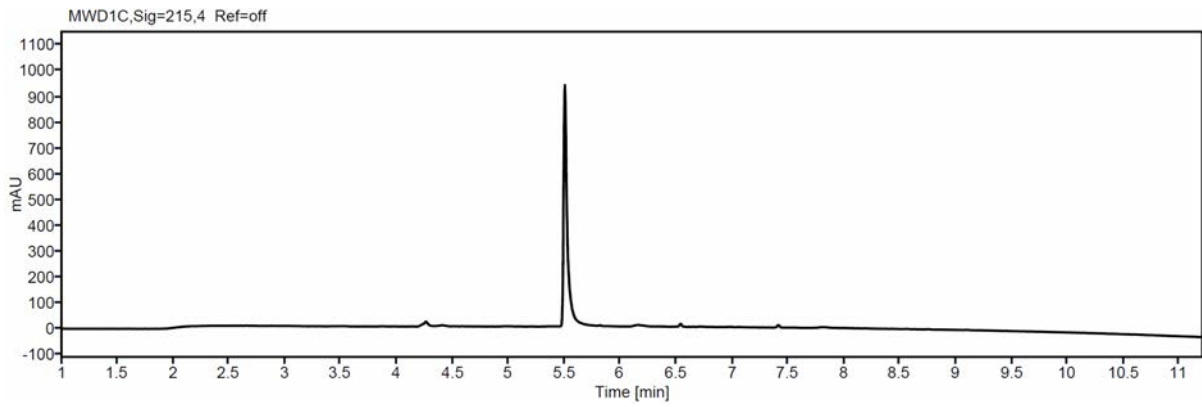
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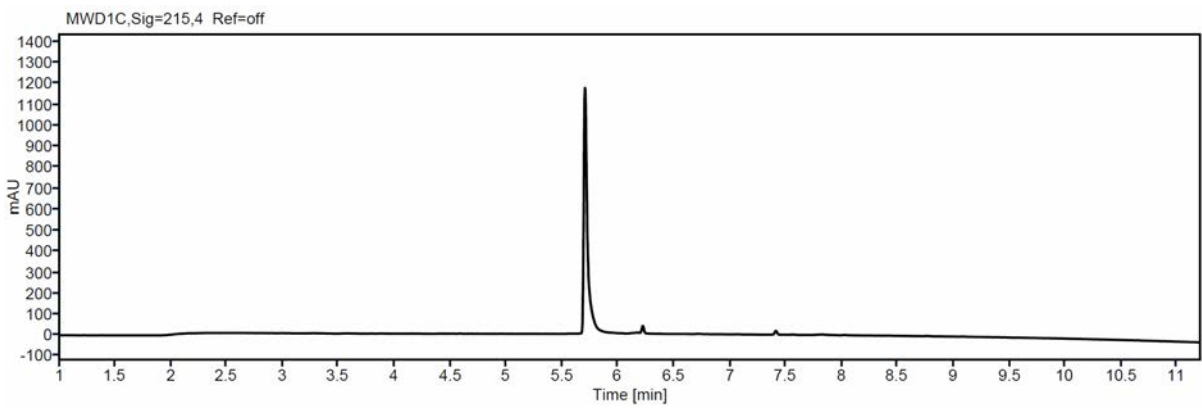
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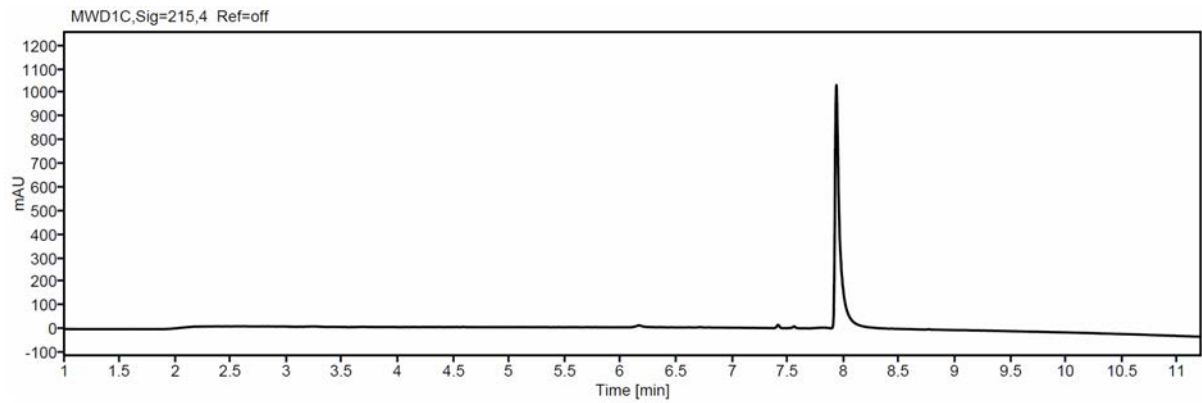
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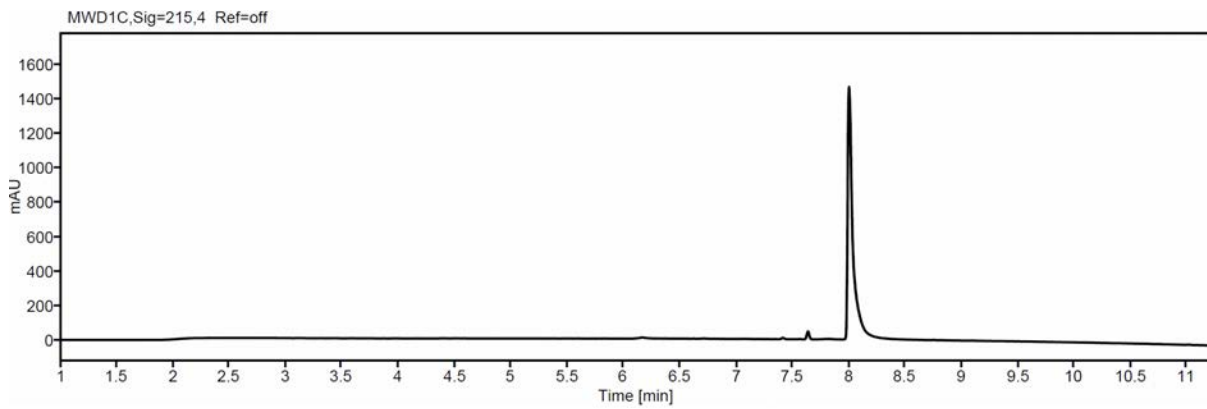
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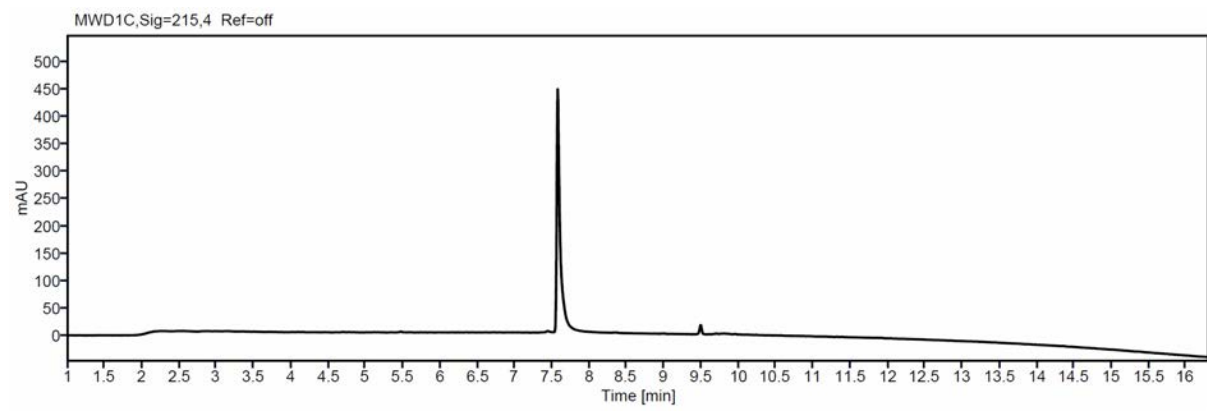
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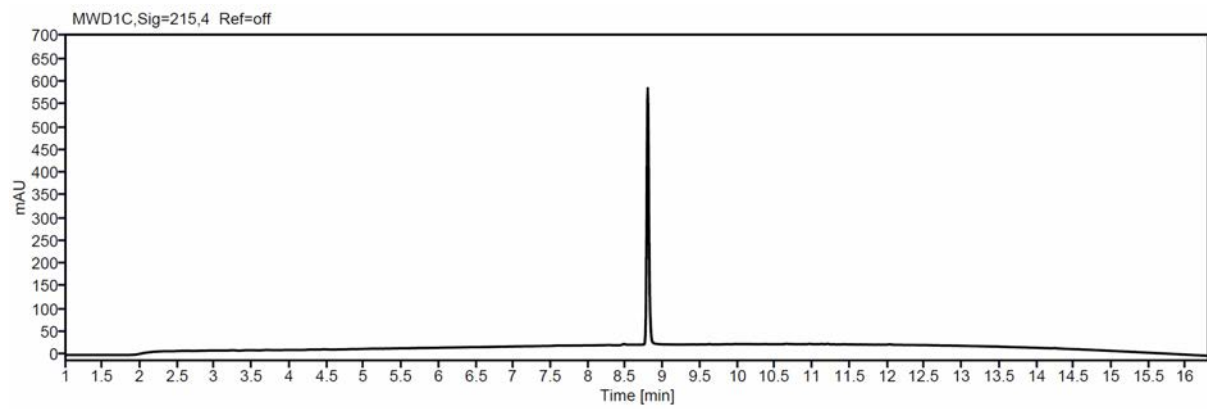
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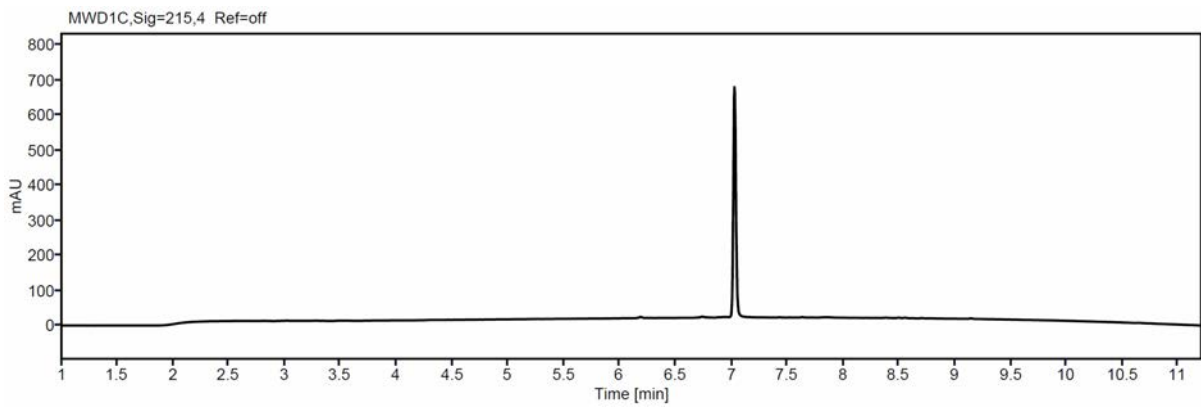
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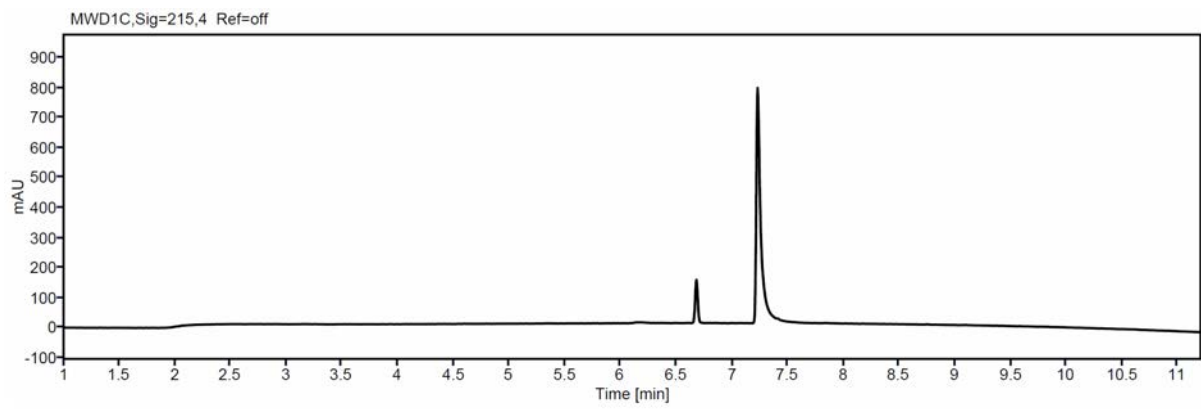
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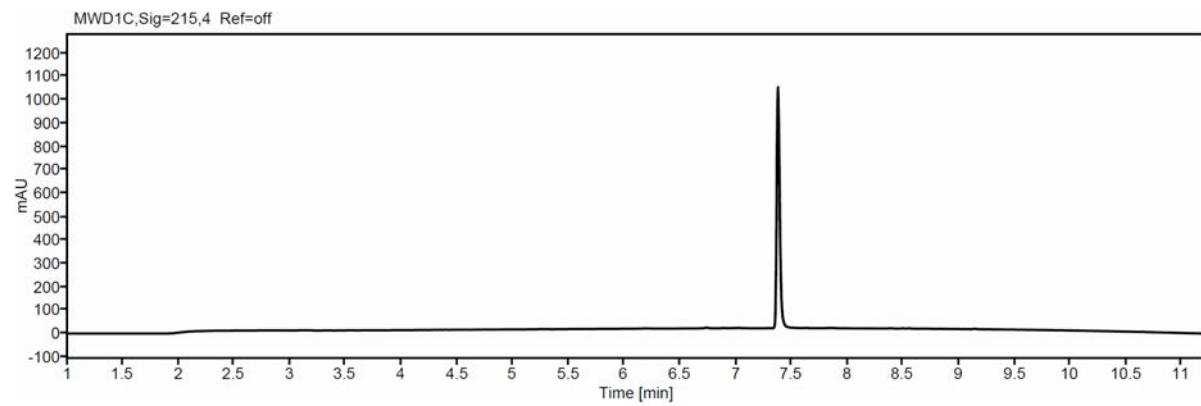
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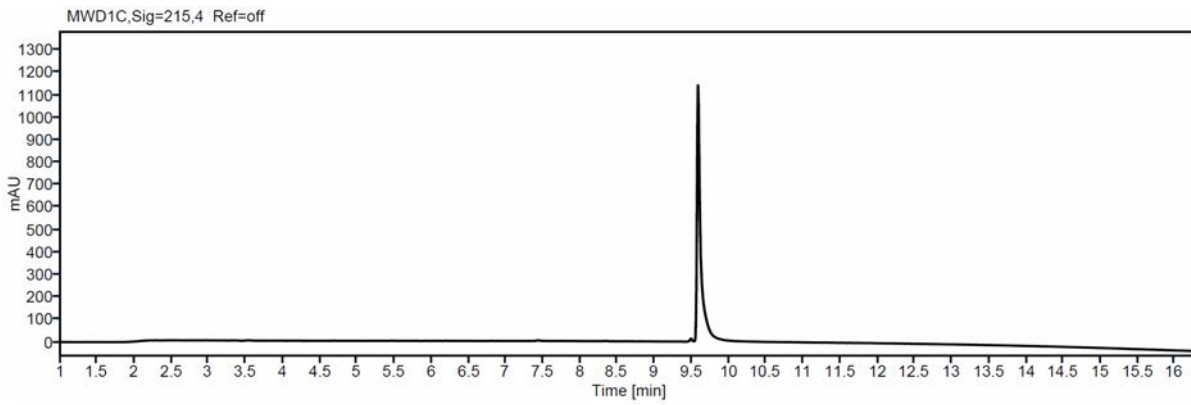
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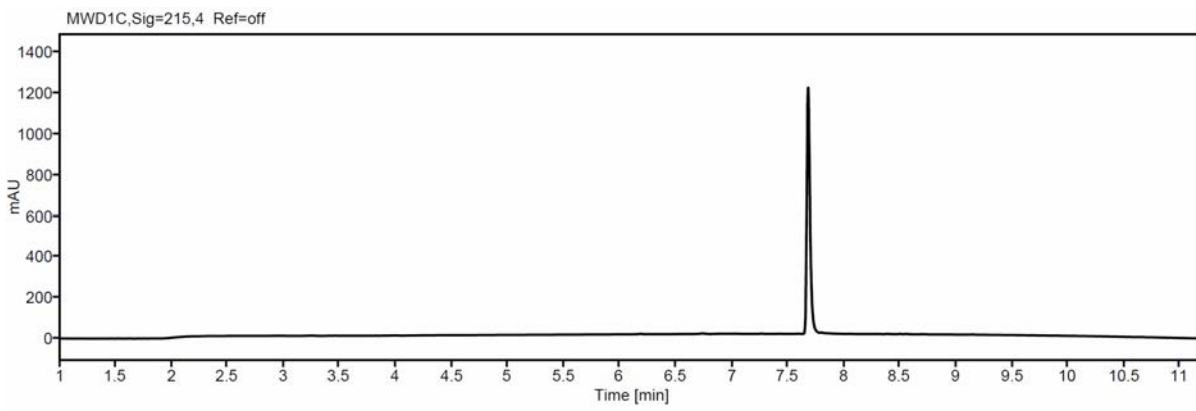
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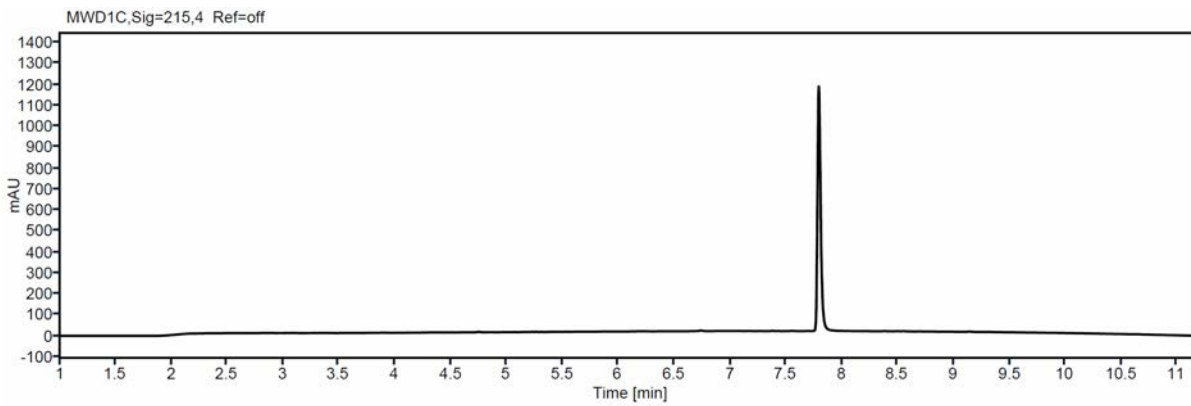
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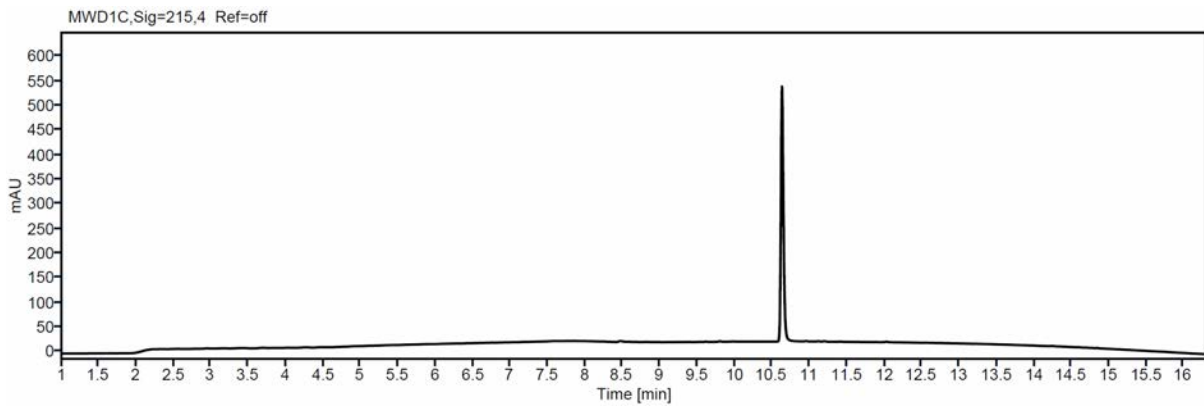
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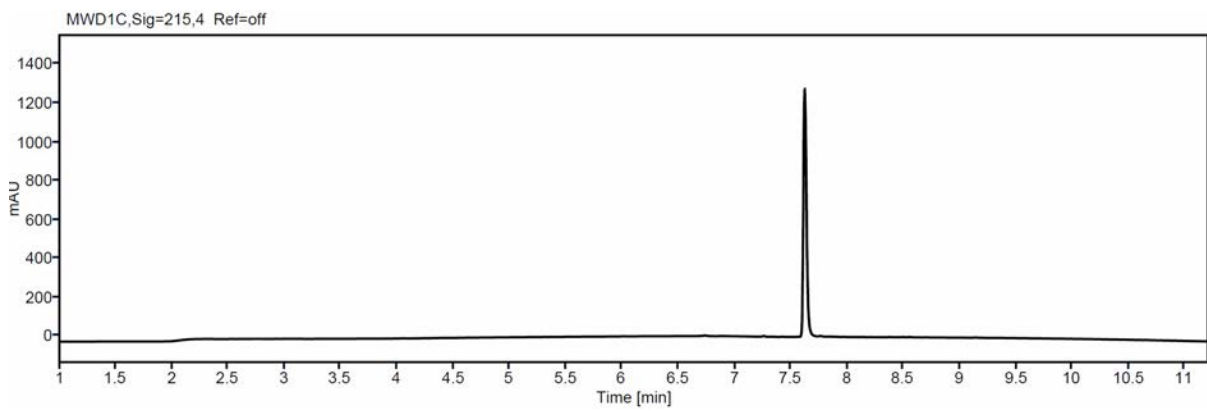
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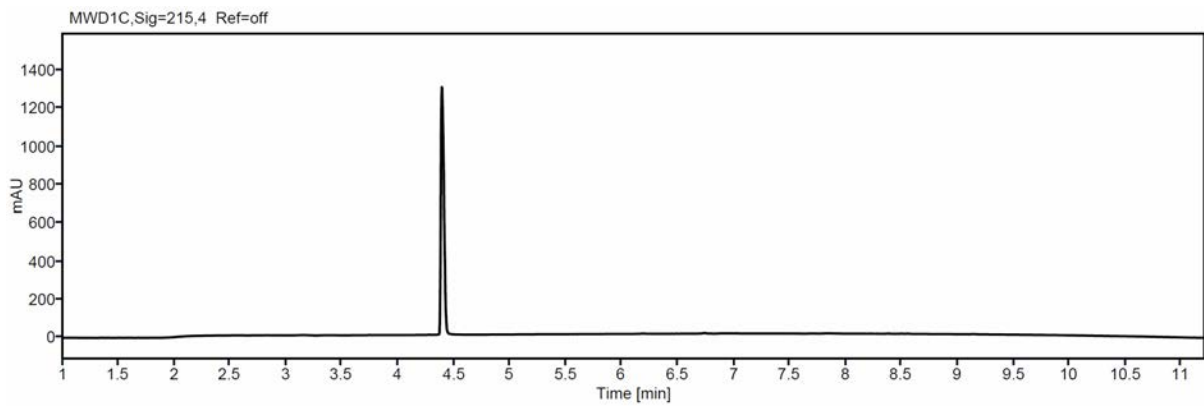
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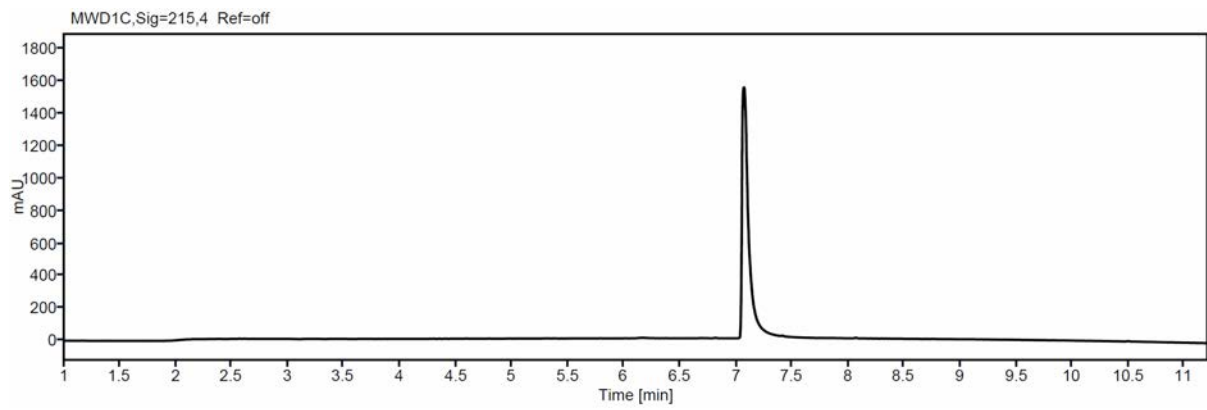
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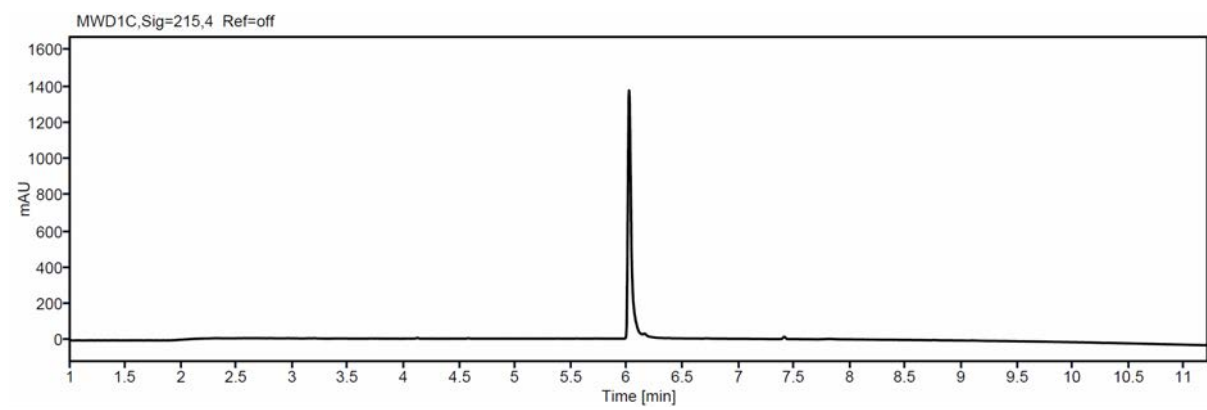
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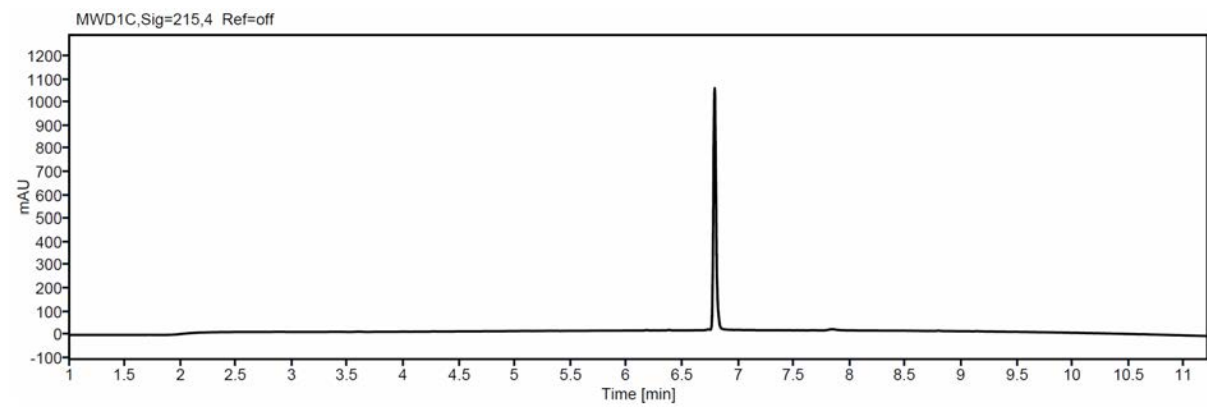
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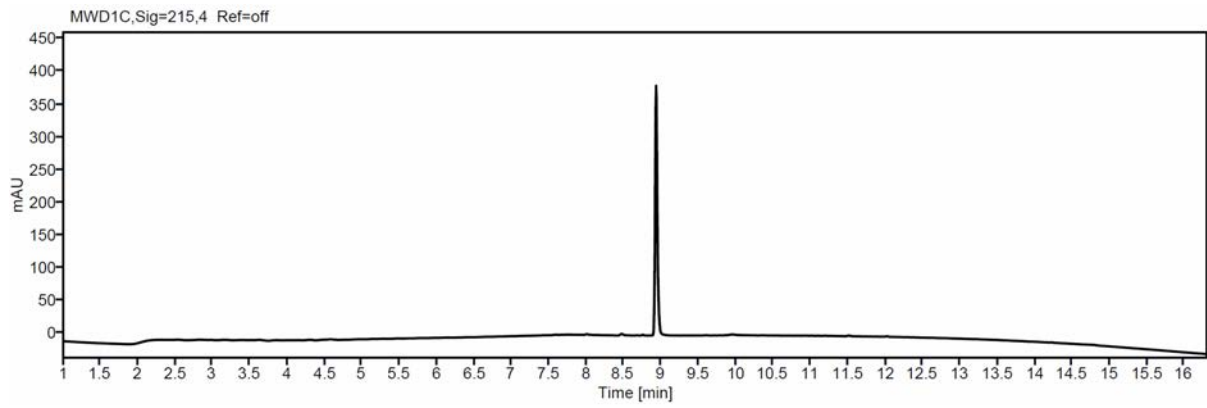
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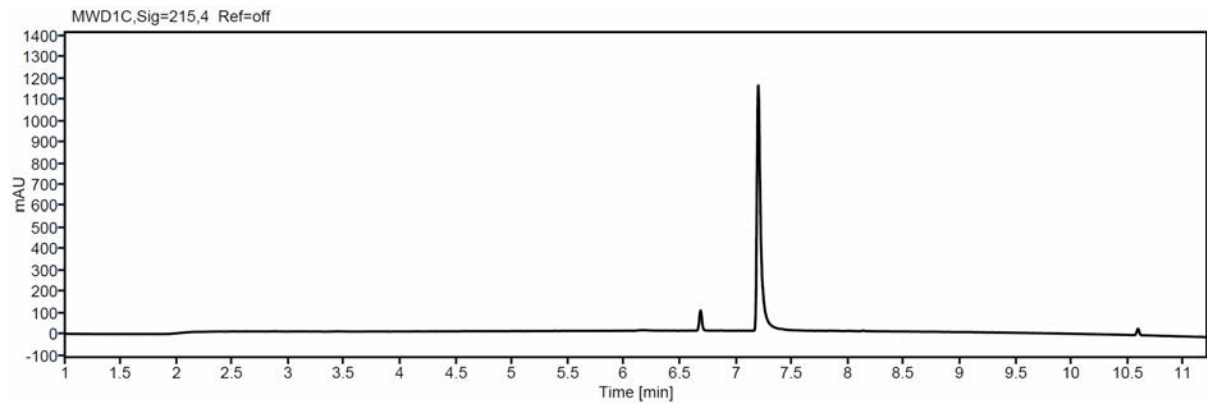
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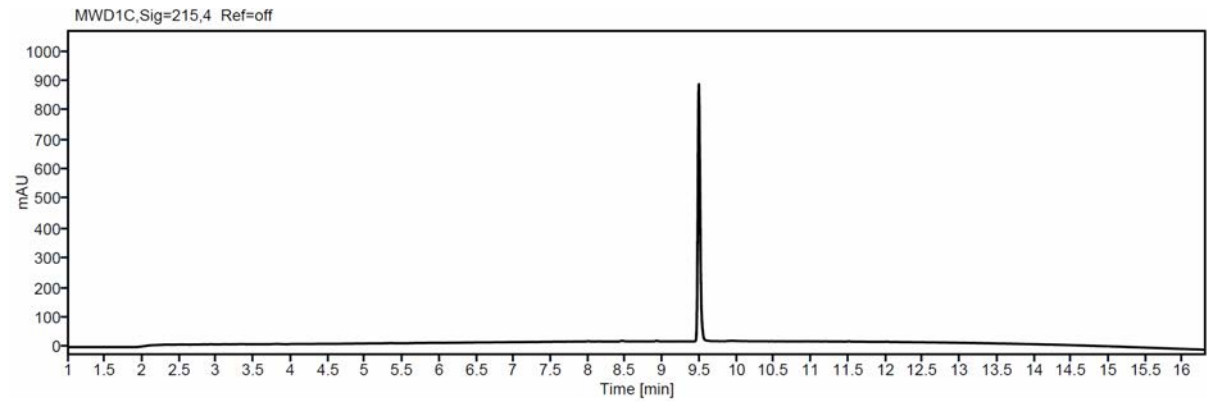
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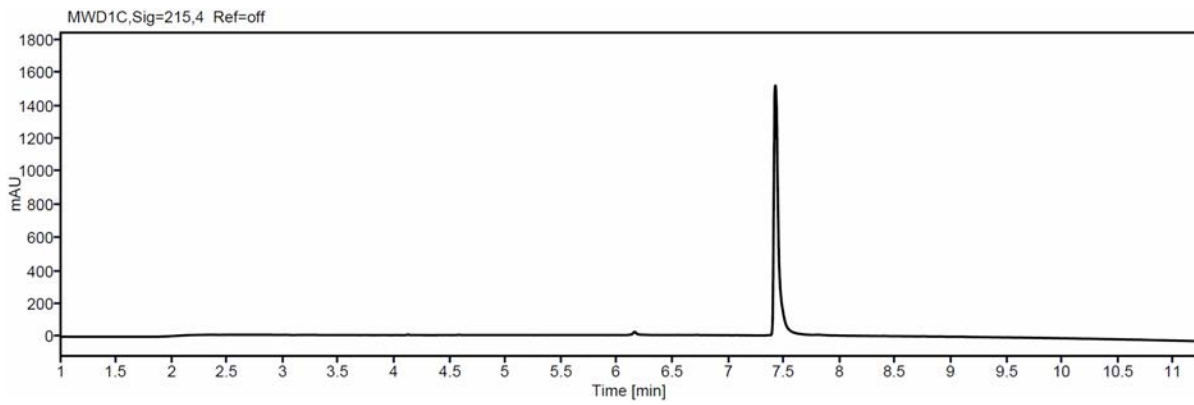
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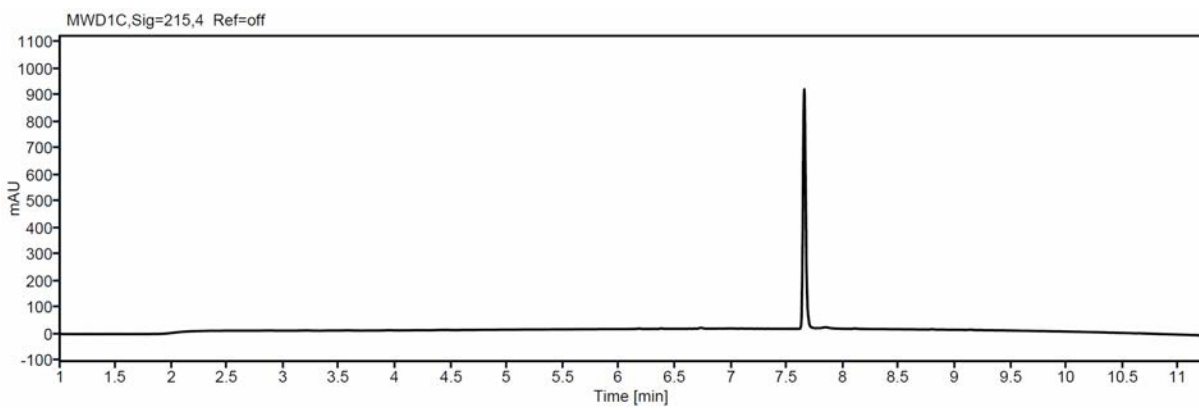
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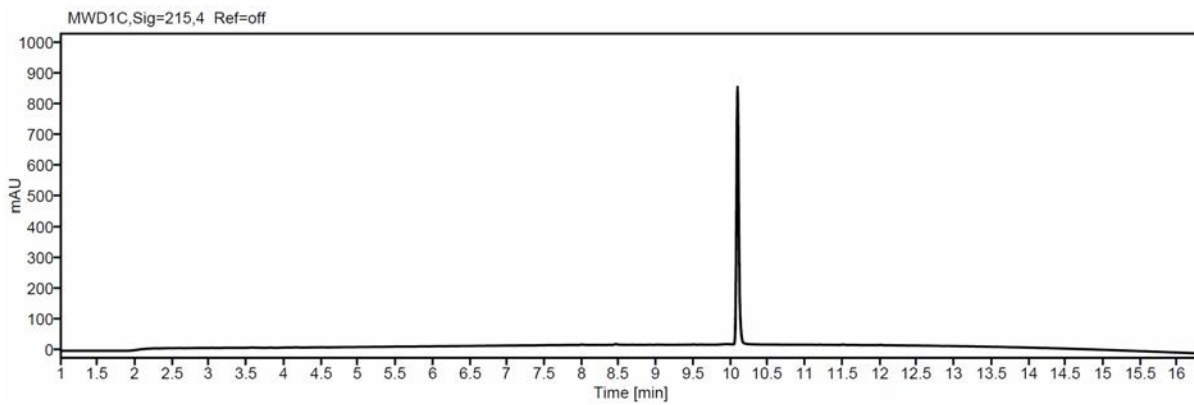
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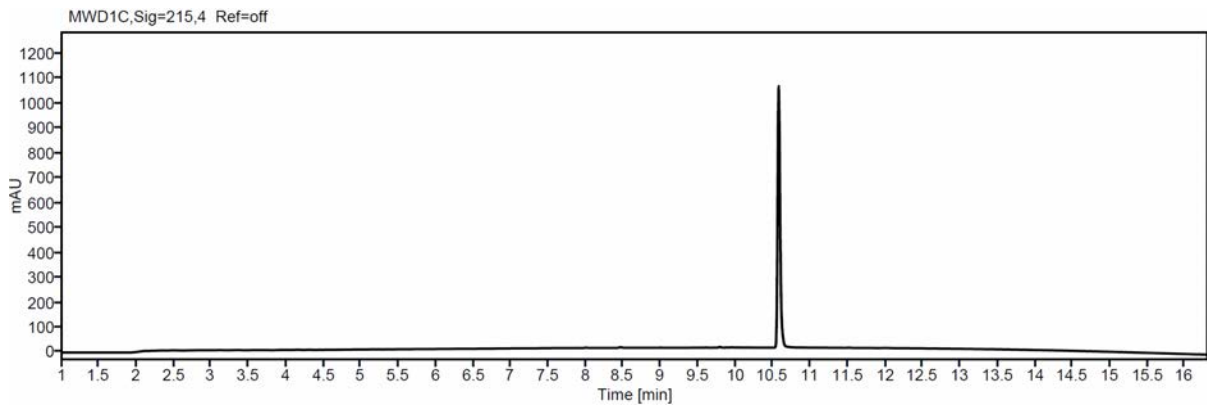
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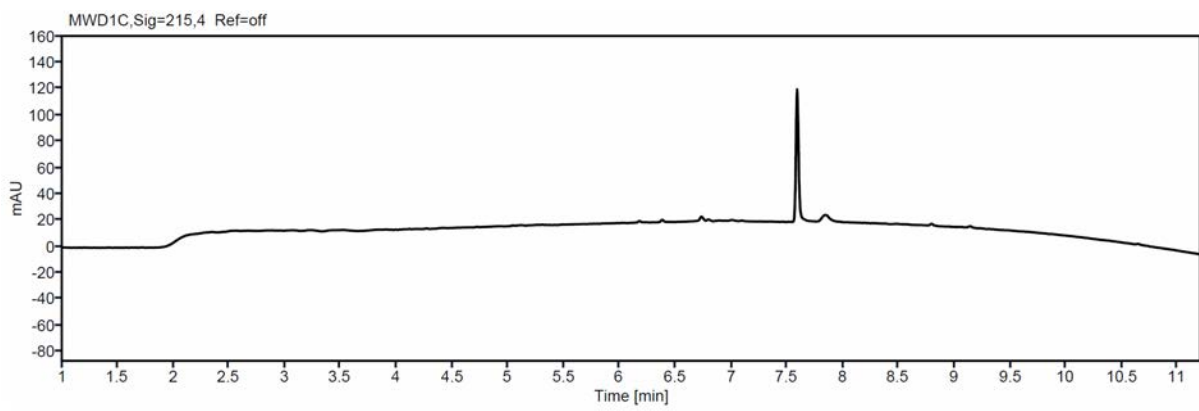
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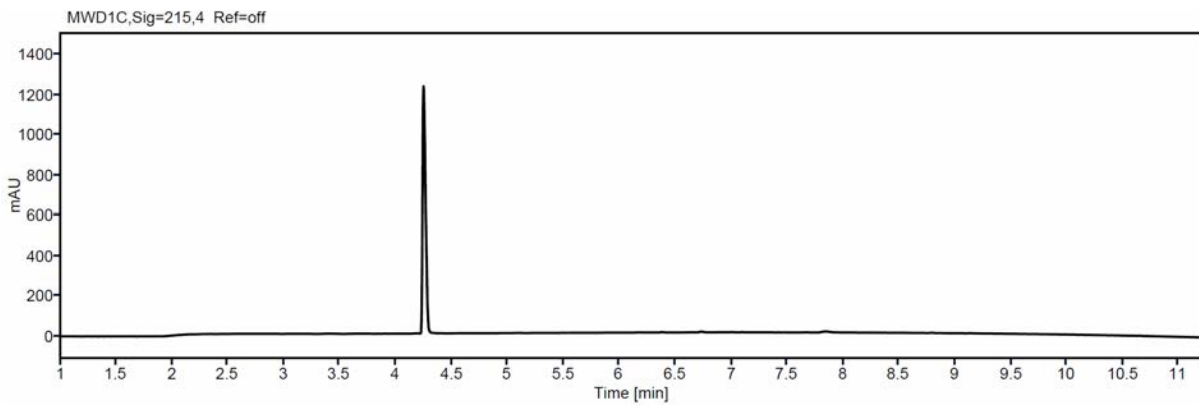
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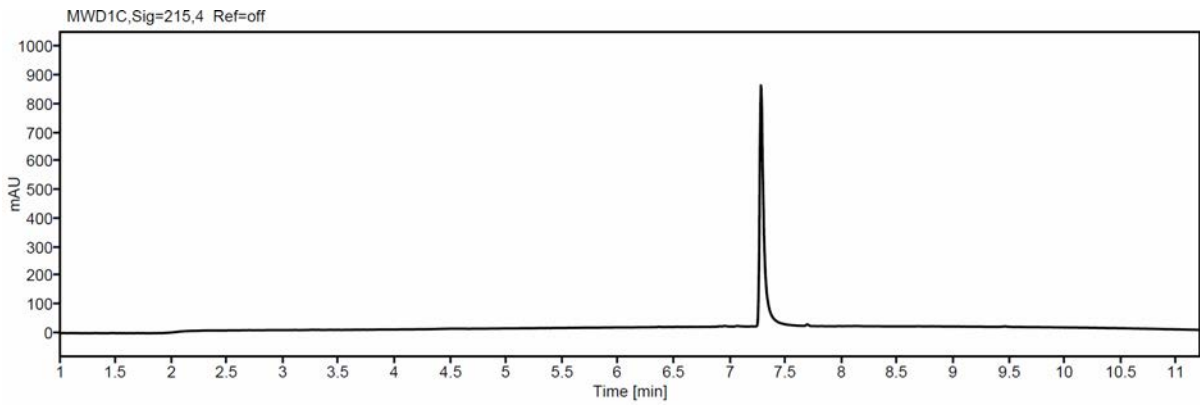
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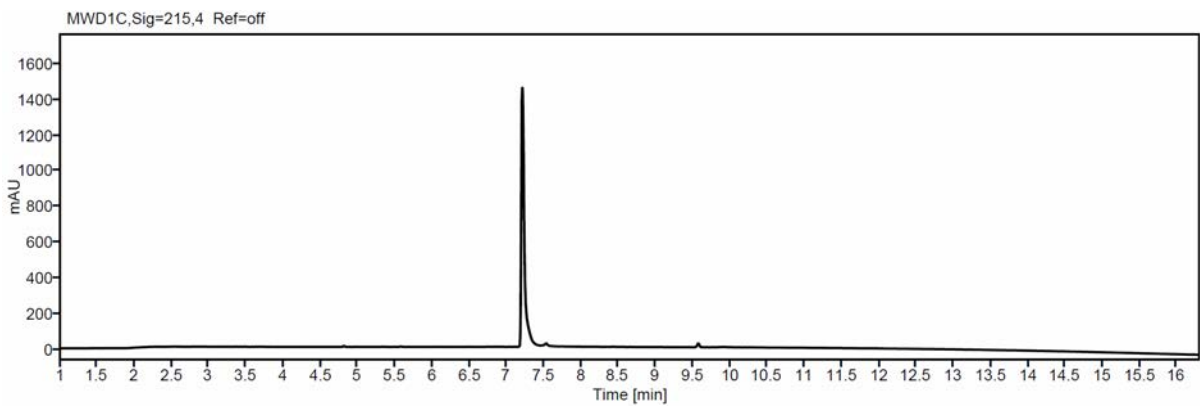
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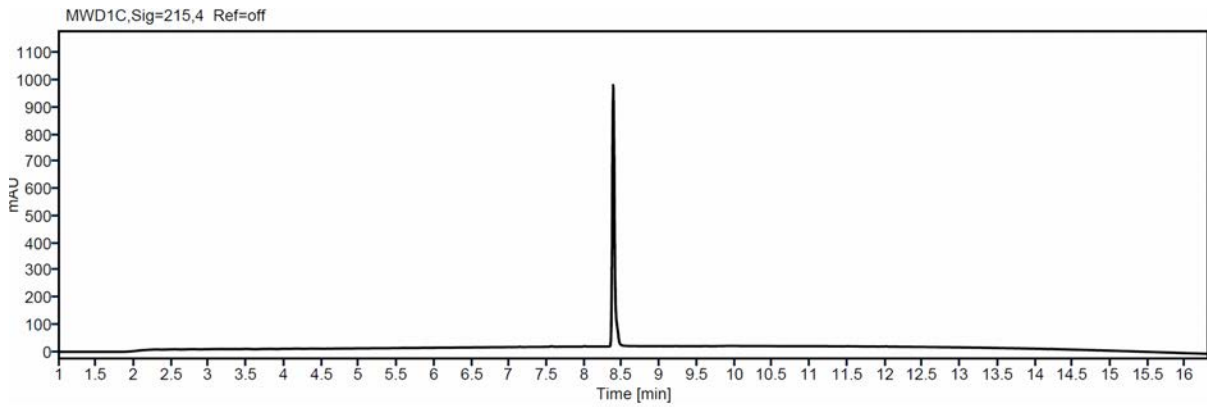
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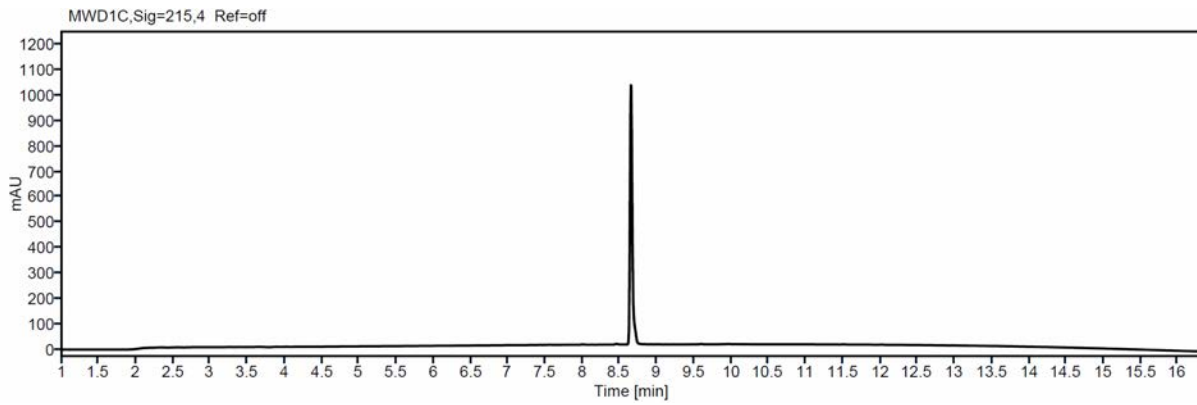
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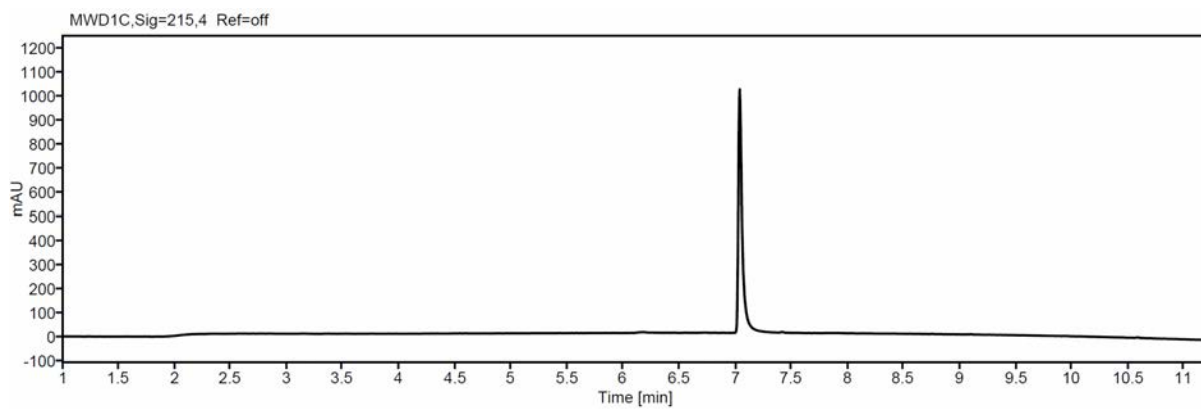
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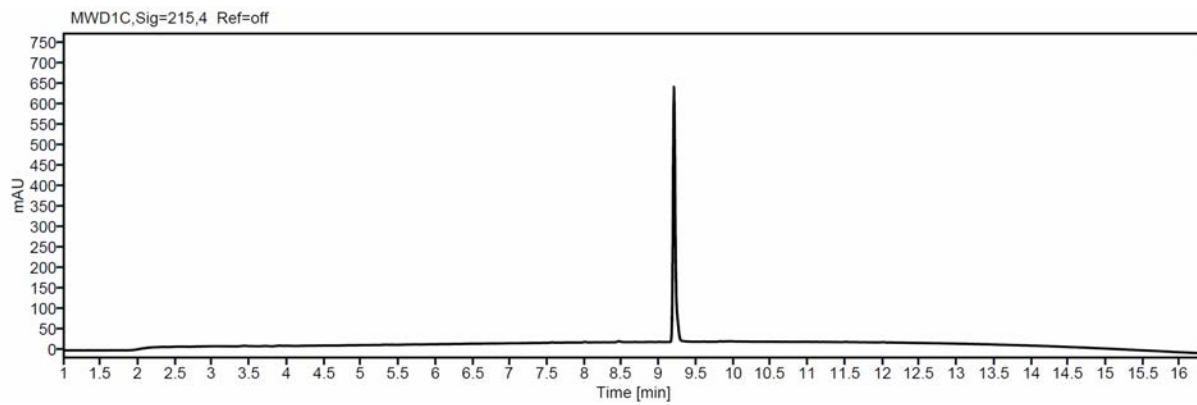
Analytical UPLC chromatogram of **D3**



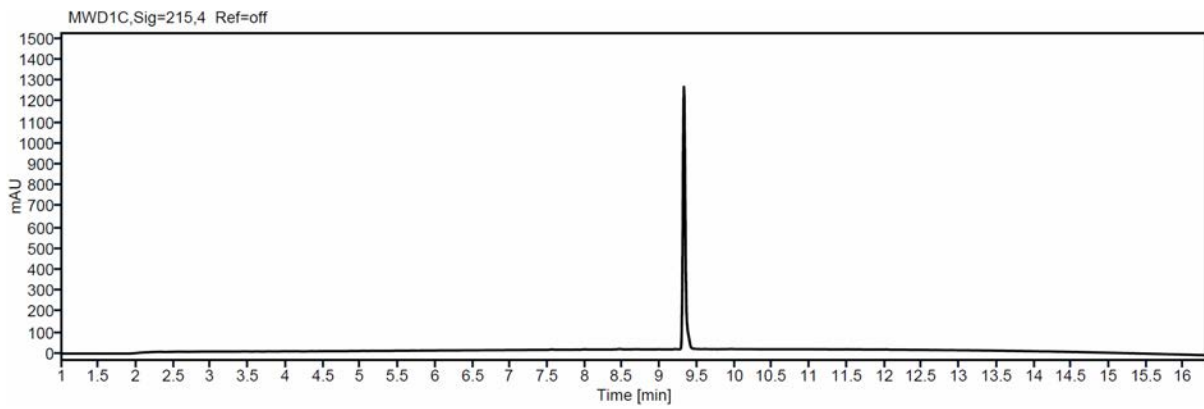
Analytical UPLC chromatogram of **D4**



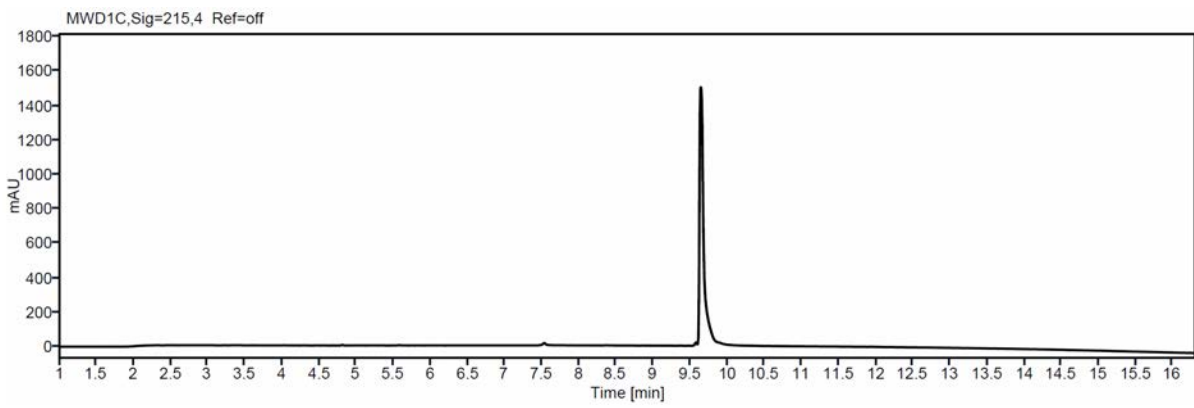
Analytical UPLC chromatogram of **D8**



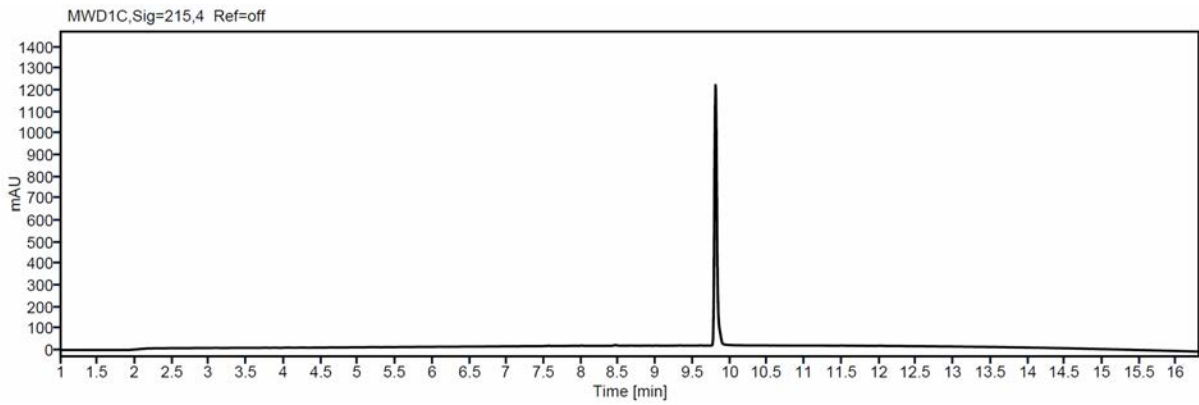
Analytical UPLC chromatogram of **D9**



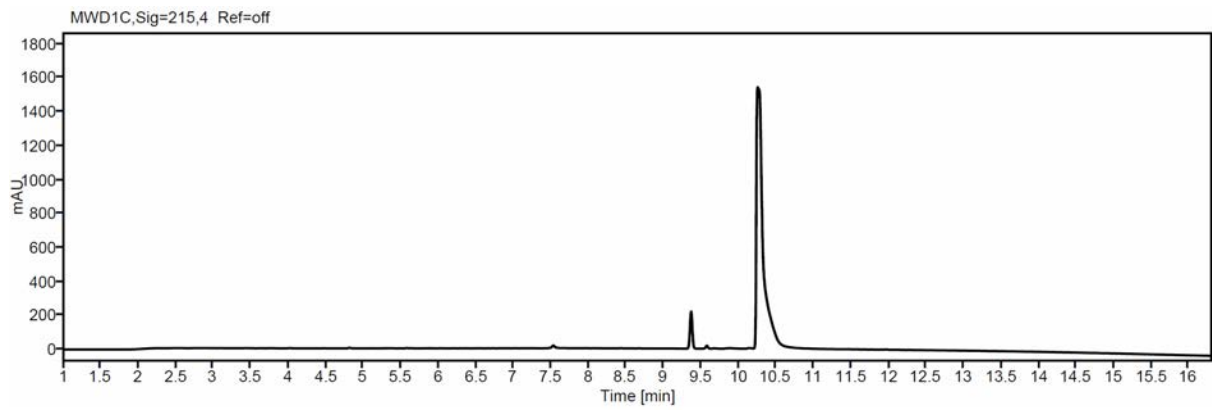
Analytical UPLC chromatogram of **D11**



Analytical UPLC chromatogram of **D12**

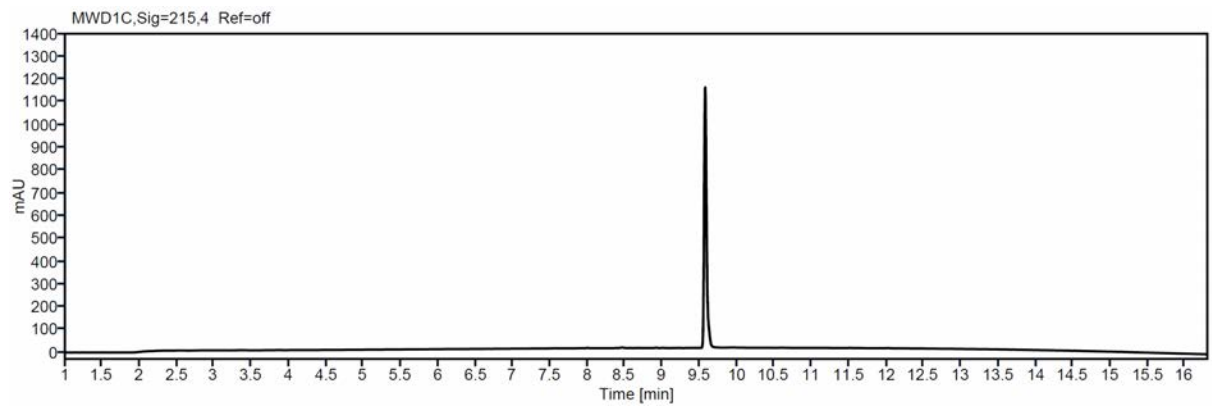


Analytical UPLC chromatogram of **D19**

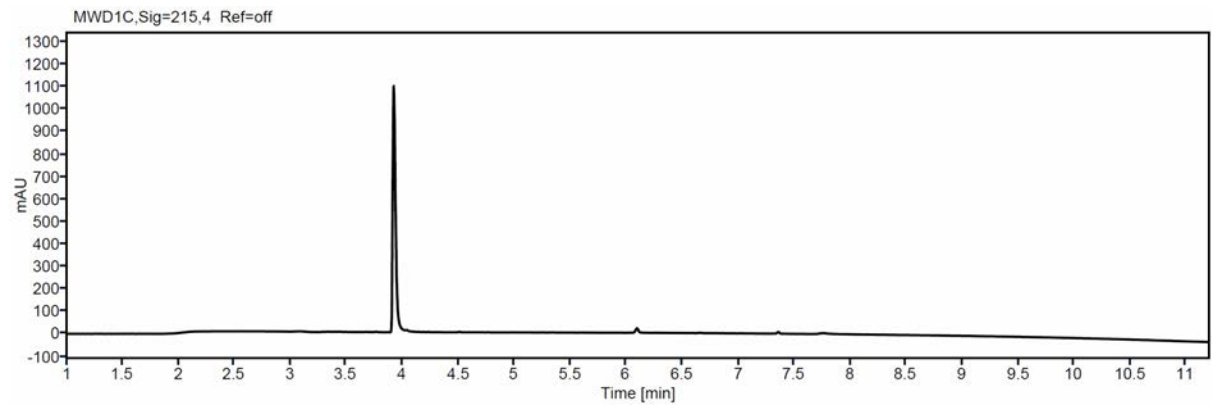


*Peak at ~9.4 min arises from an impurity on the column.

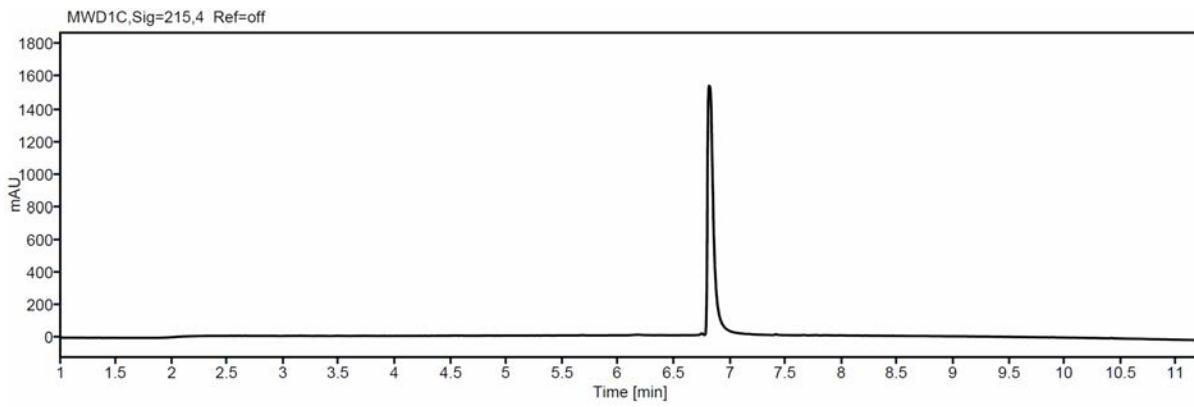
Analytical UPLC chromatogram of **D21**



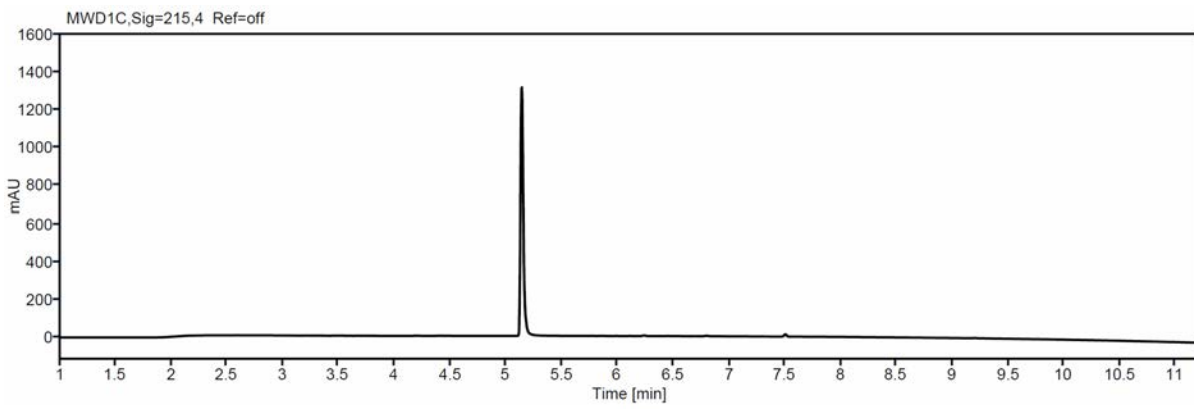
Analytical UPLC chromatogram of **D23**



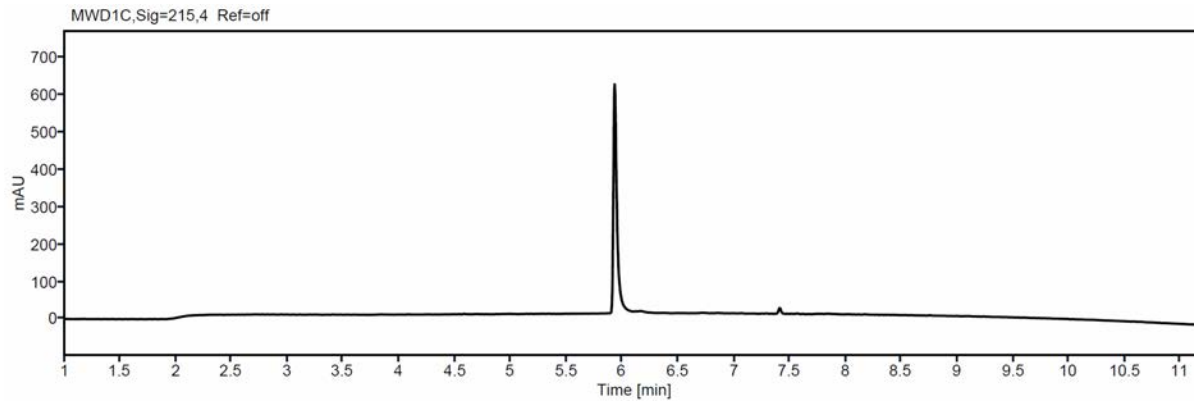
Analytical UPLC chromatogram of **D24**



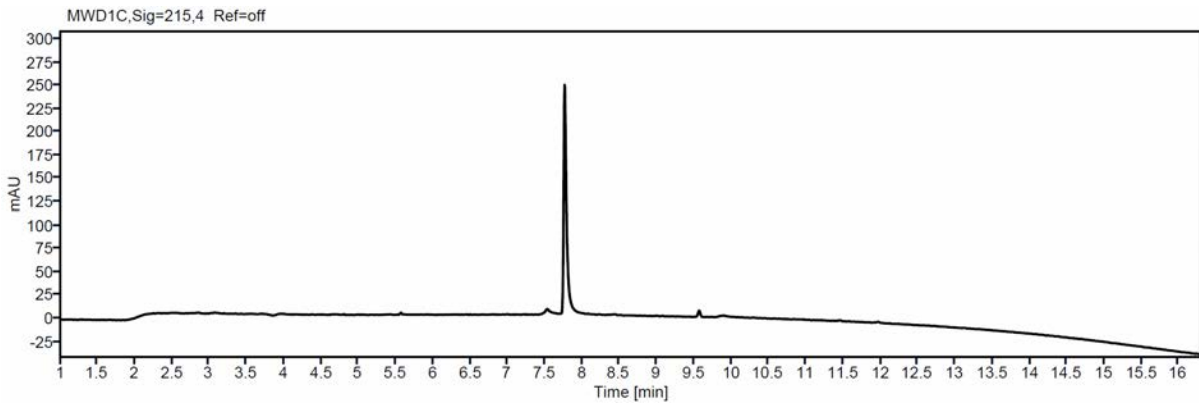
Analytical UPLC chromatogram of **E1**



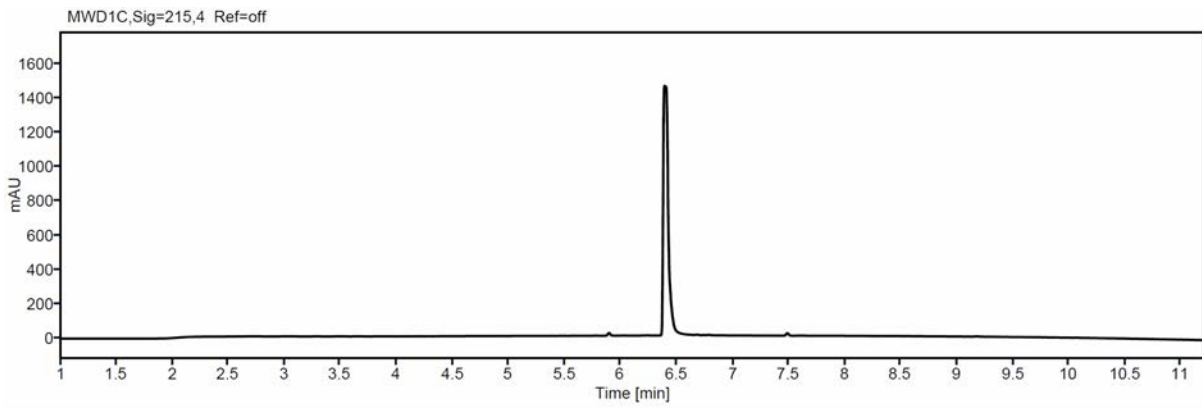
Analytical UPLC chromatogram of **E2**



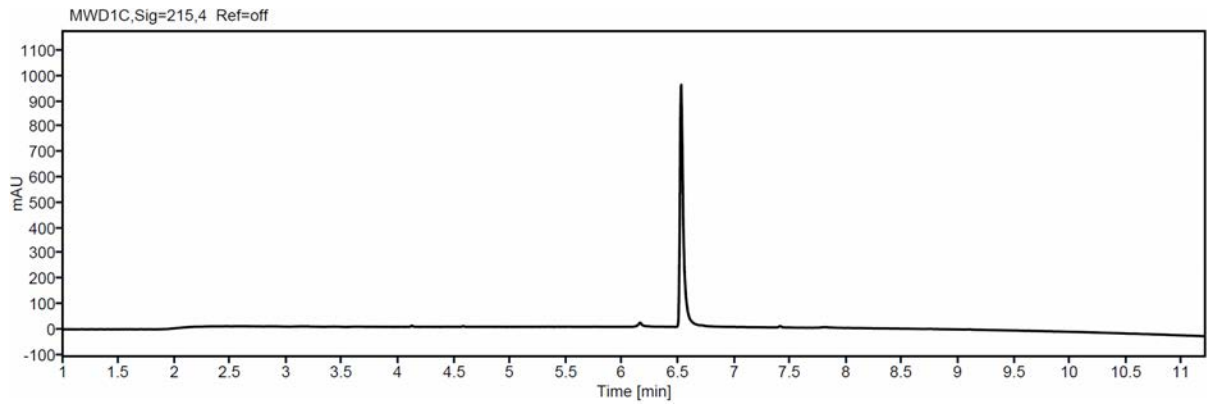
Analytical UPLC chromatogram of **E3**



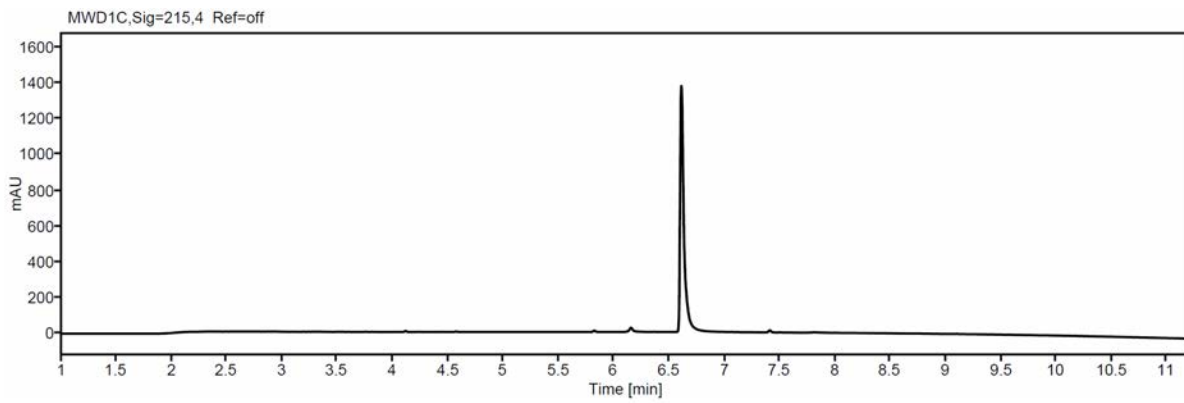
Analytical UPLC chromatogram of **E4**



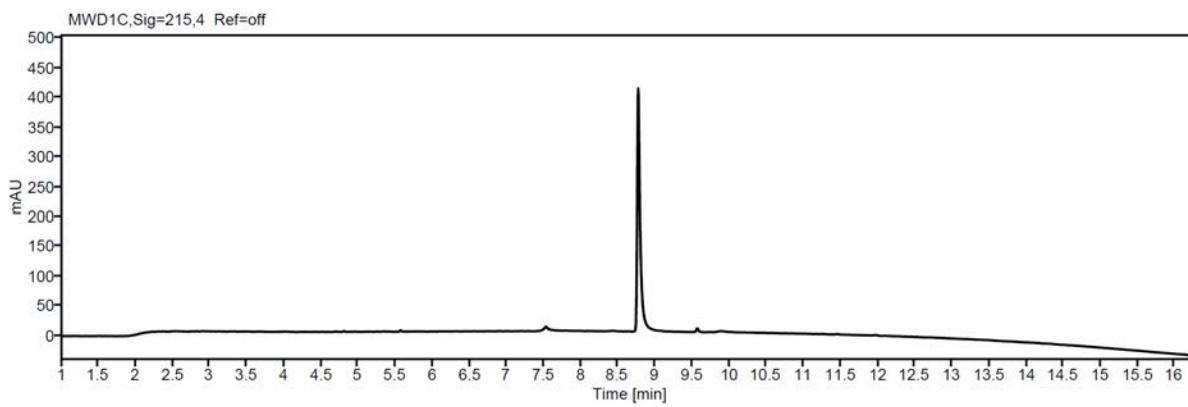
Analytical UPLC chromatogram of **E8**



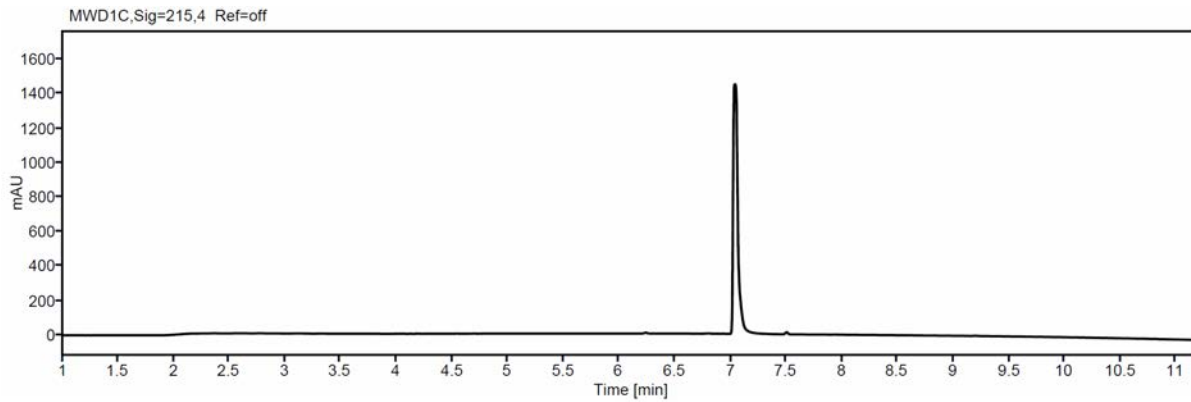
Analytical UPLC chromatogram of **E9**



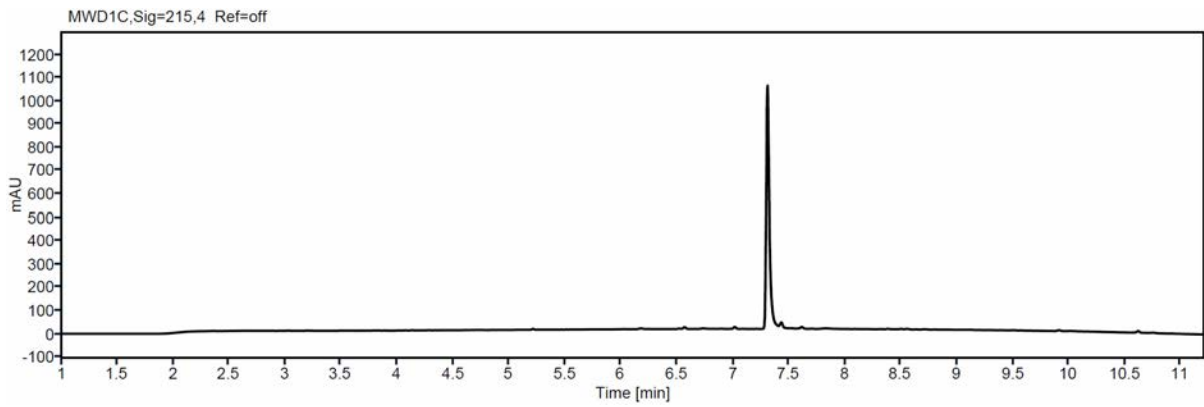
Analytical UPLC chromatogram of **E11**



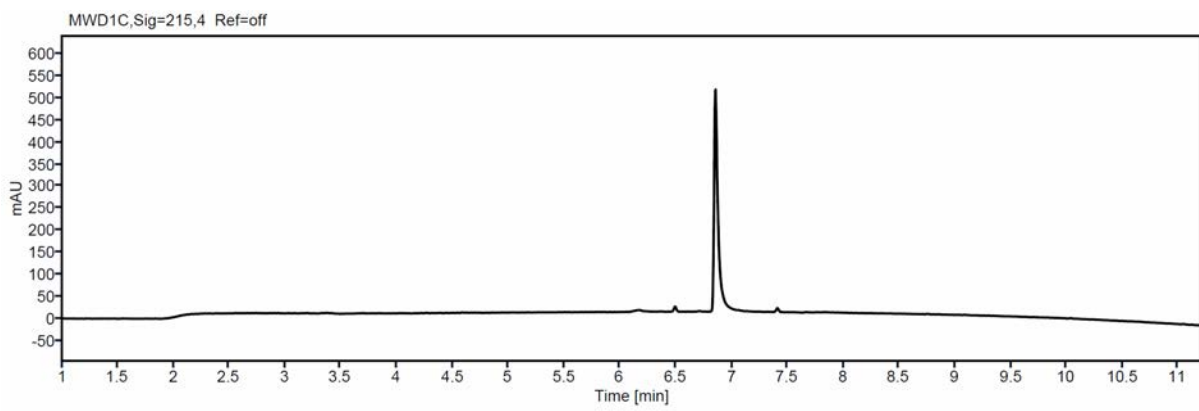
Analytical UPLC chromatogram of **E12**



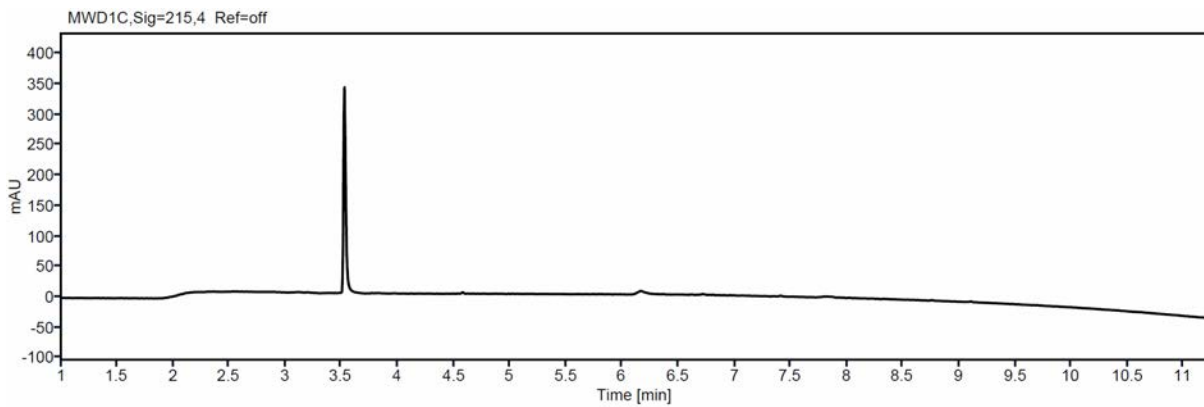
Analytical UPLC chromatogram of **E19**



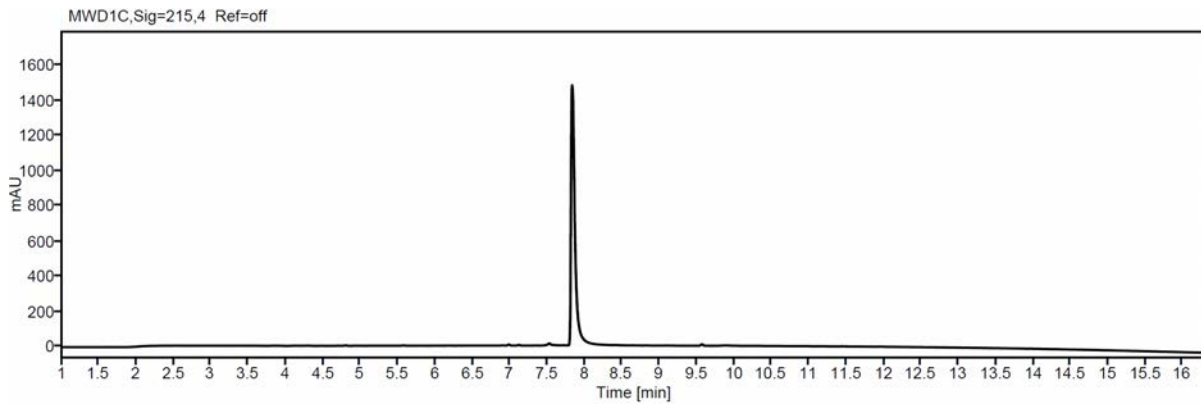
Analytical UPLC chromatogram of **E21**



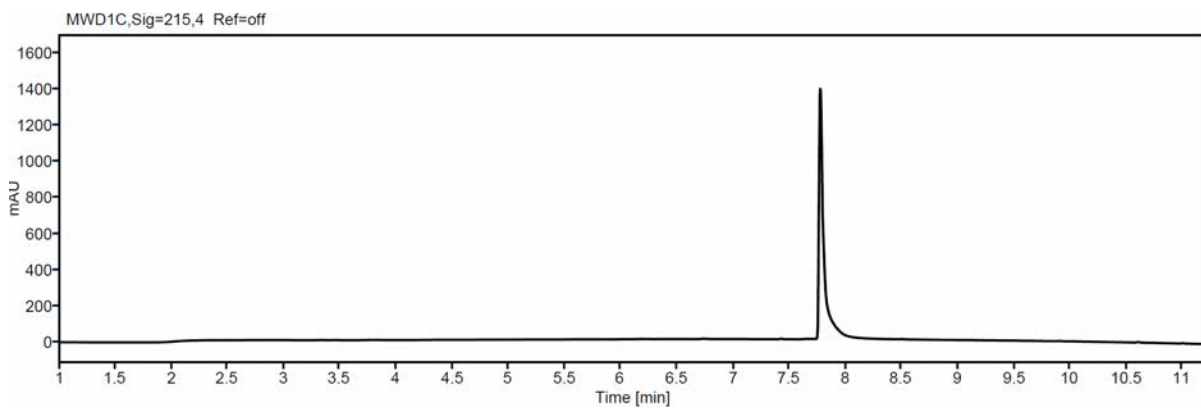
Analytical UPLC chromatogram of **E23**



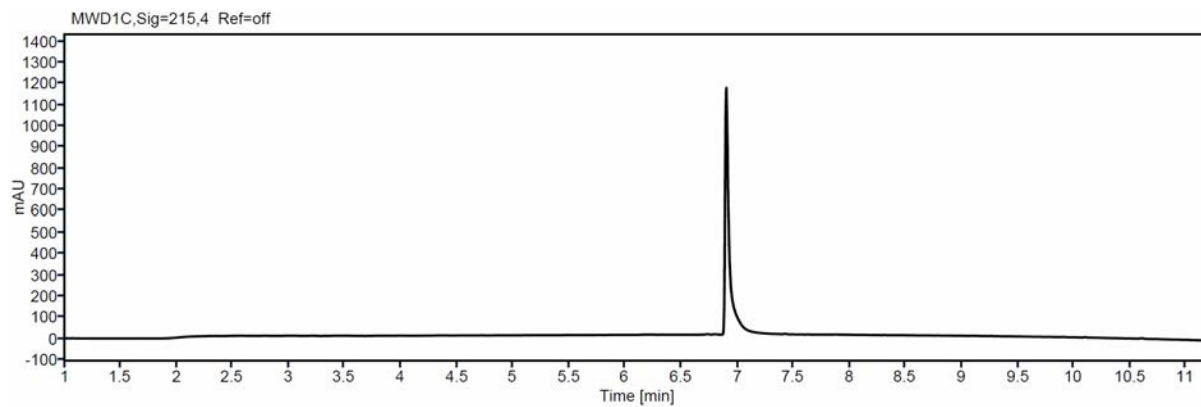
Analytical UPLC chromatogram of **E24**



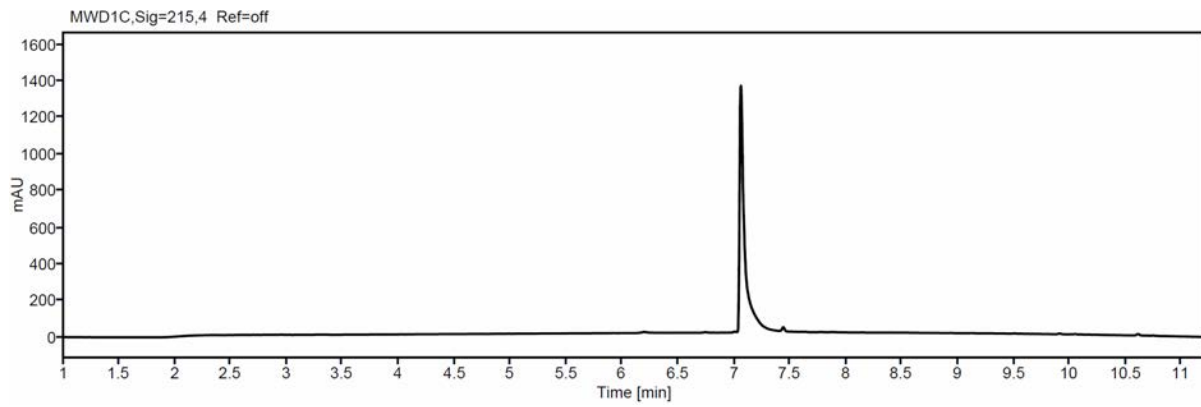
Analytical UPLC chromatogram of **F1**



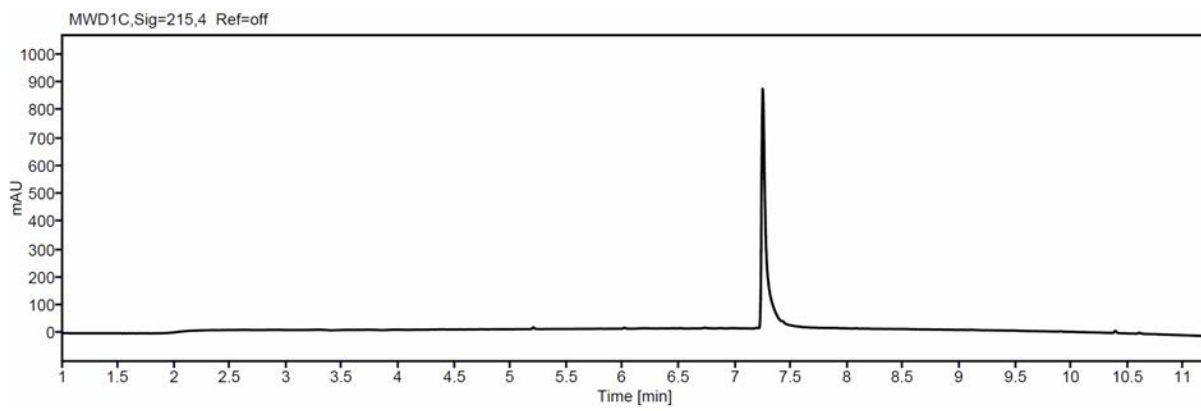
Analytical UPLC chromatogram of **F2**



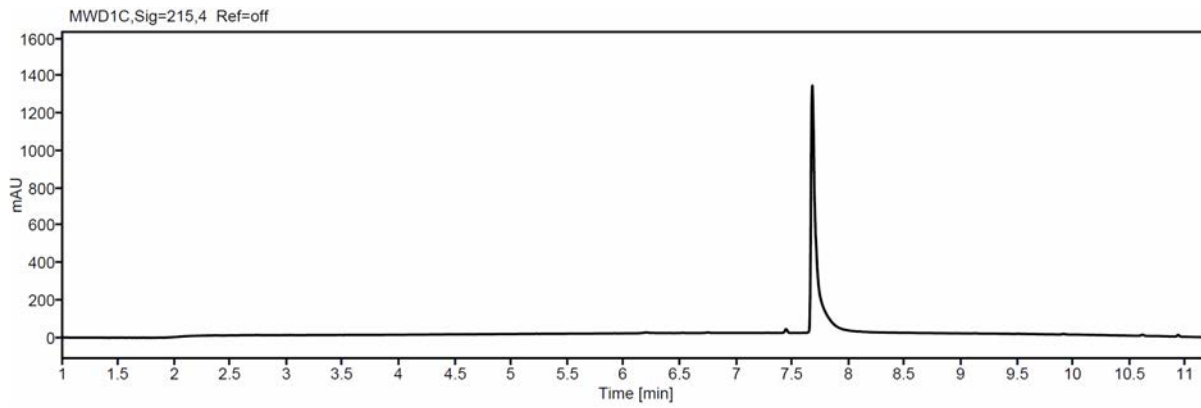
Analytical UPLC chromatogram of **F3**



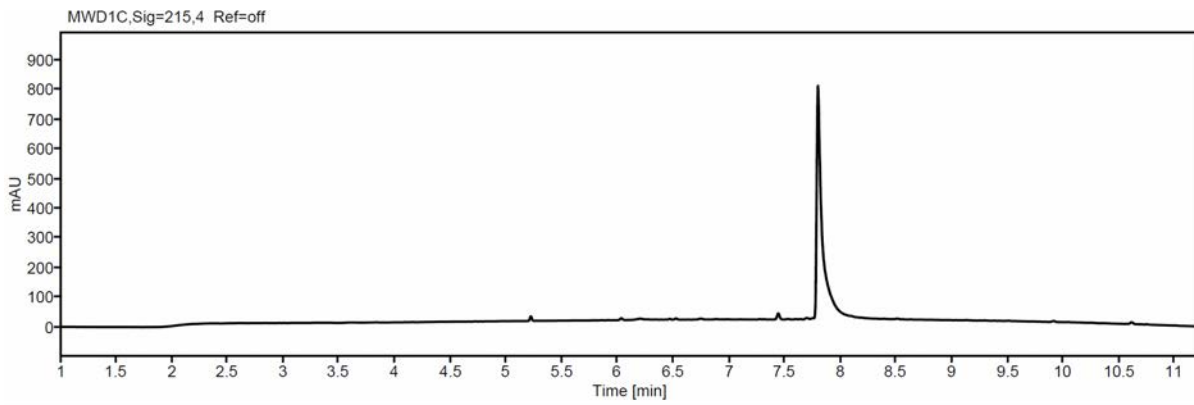
Analytical UPLC chromatogram of **F4**



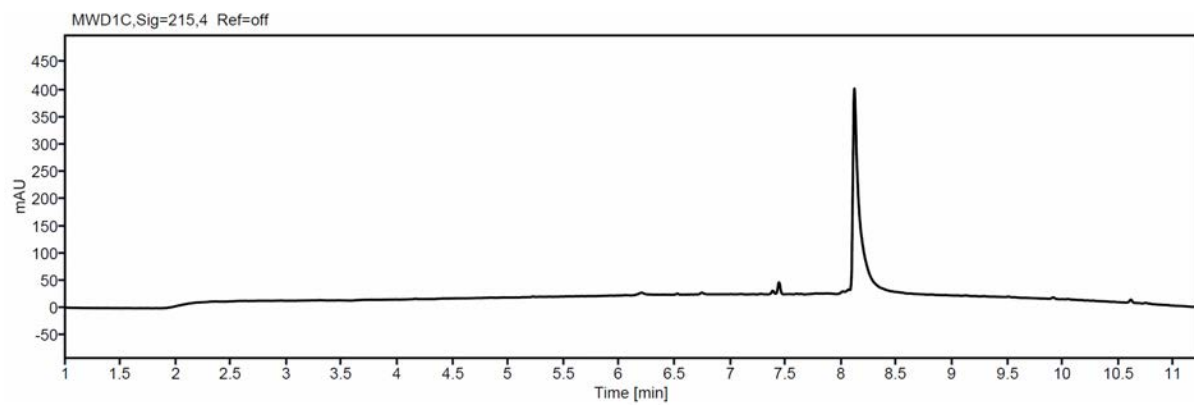
Analytical UPLC chromatogram of **F11**



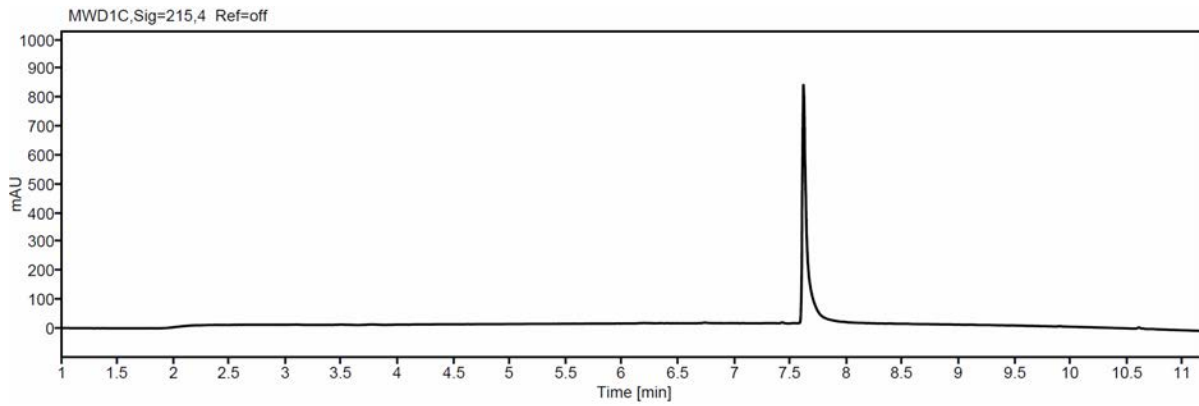
Analytical UPLC chromatogram of **F12**



Analytical UPLC chromatogram of **F19**

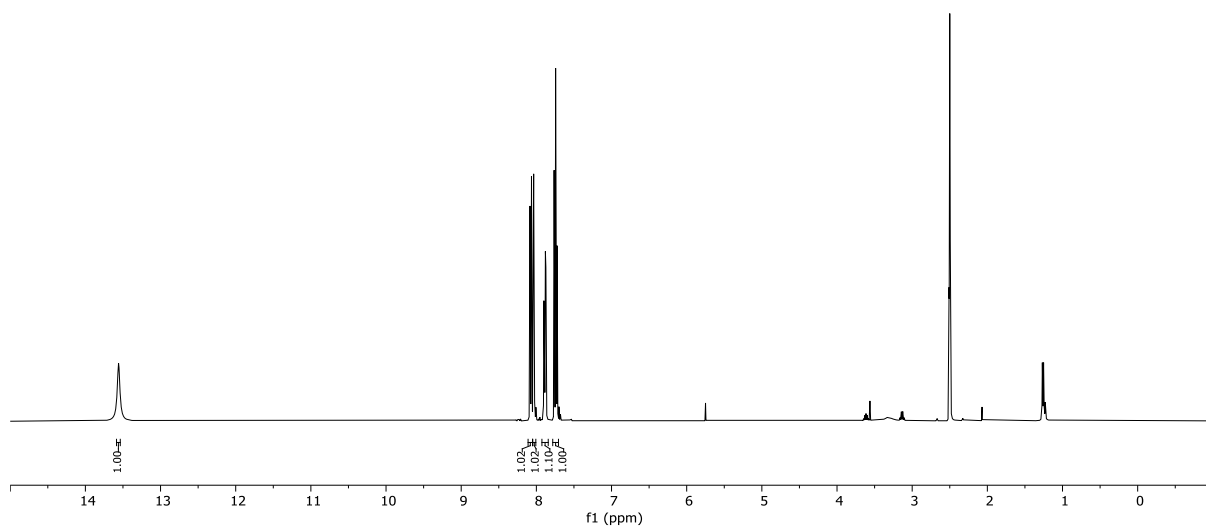
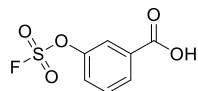


Analytical UPLC chromatogram of **F21**

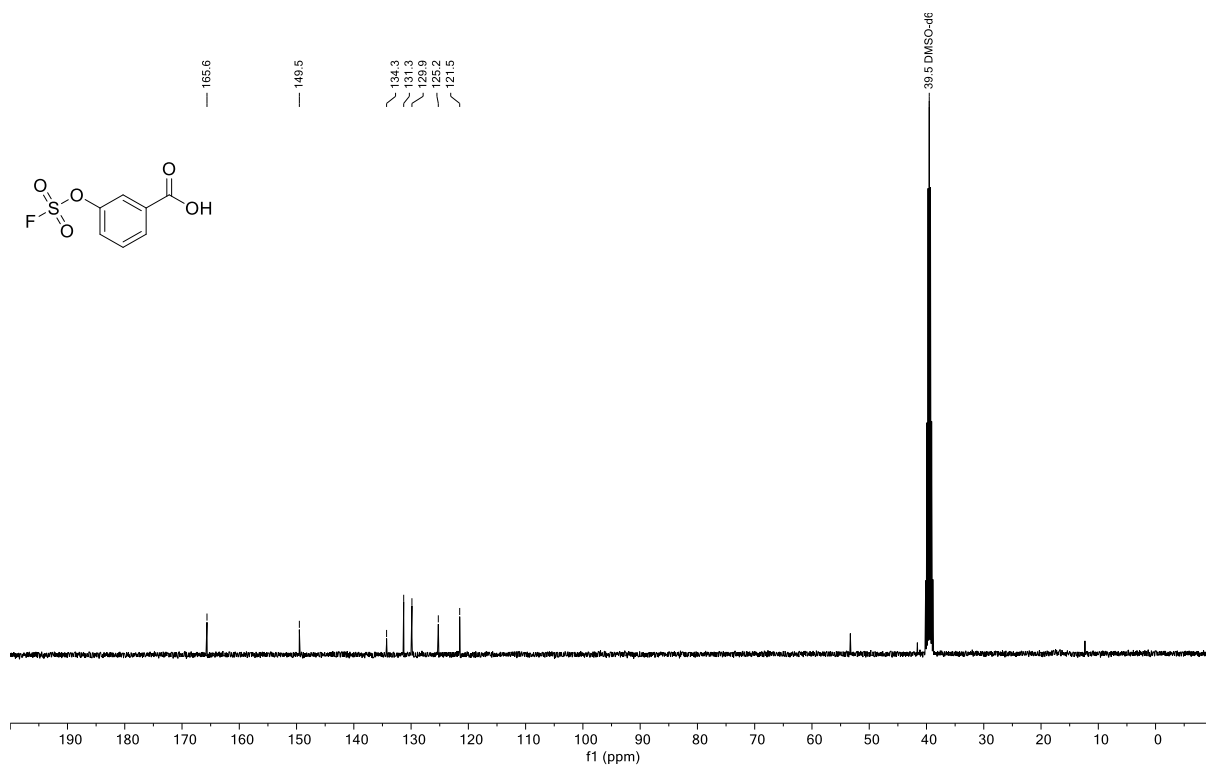
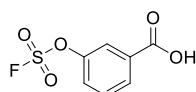


NMR spectra

^1H NMR spectra (400 MHz, $\text{DMSO-}d_6$) of compound **S1**

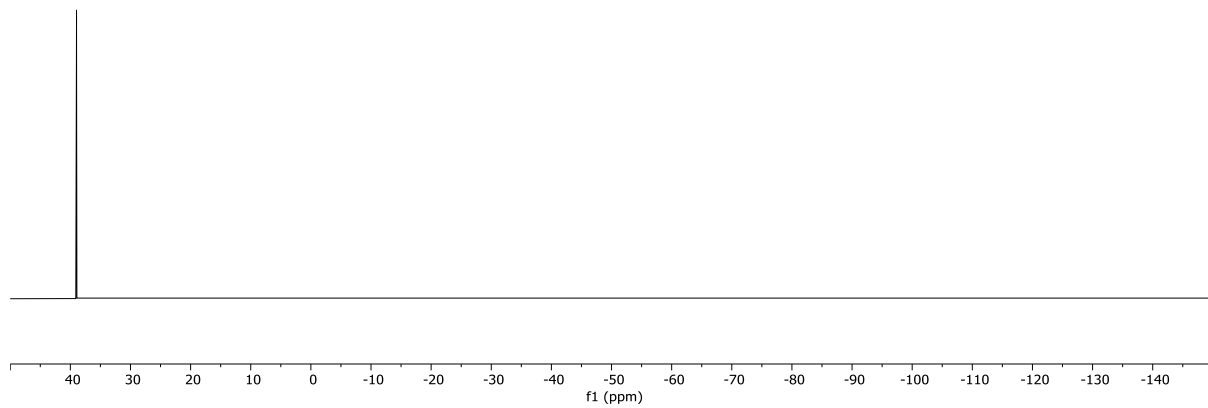
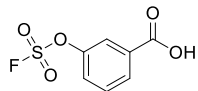


^{13}C NMR spectra (101 MHz, $\text{DMSO-}d_6$) of compound **S1**

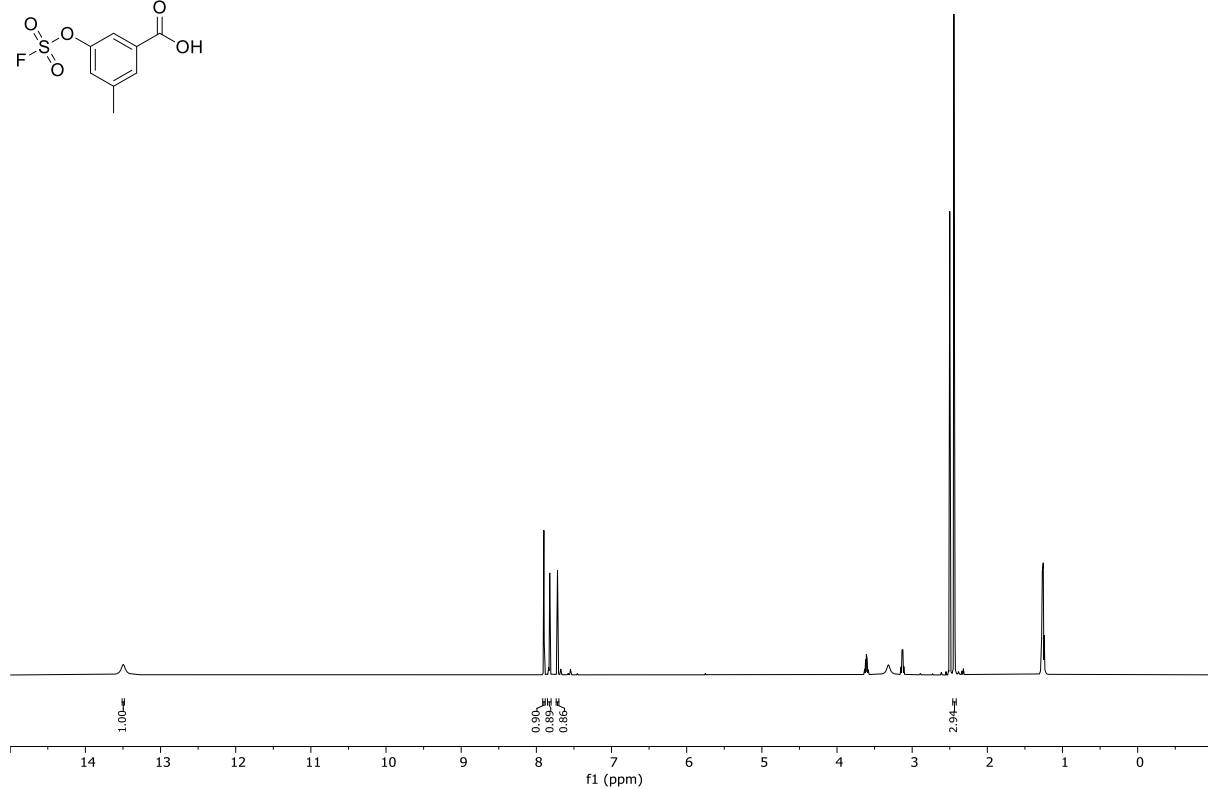
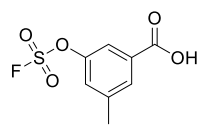


^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **S1**

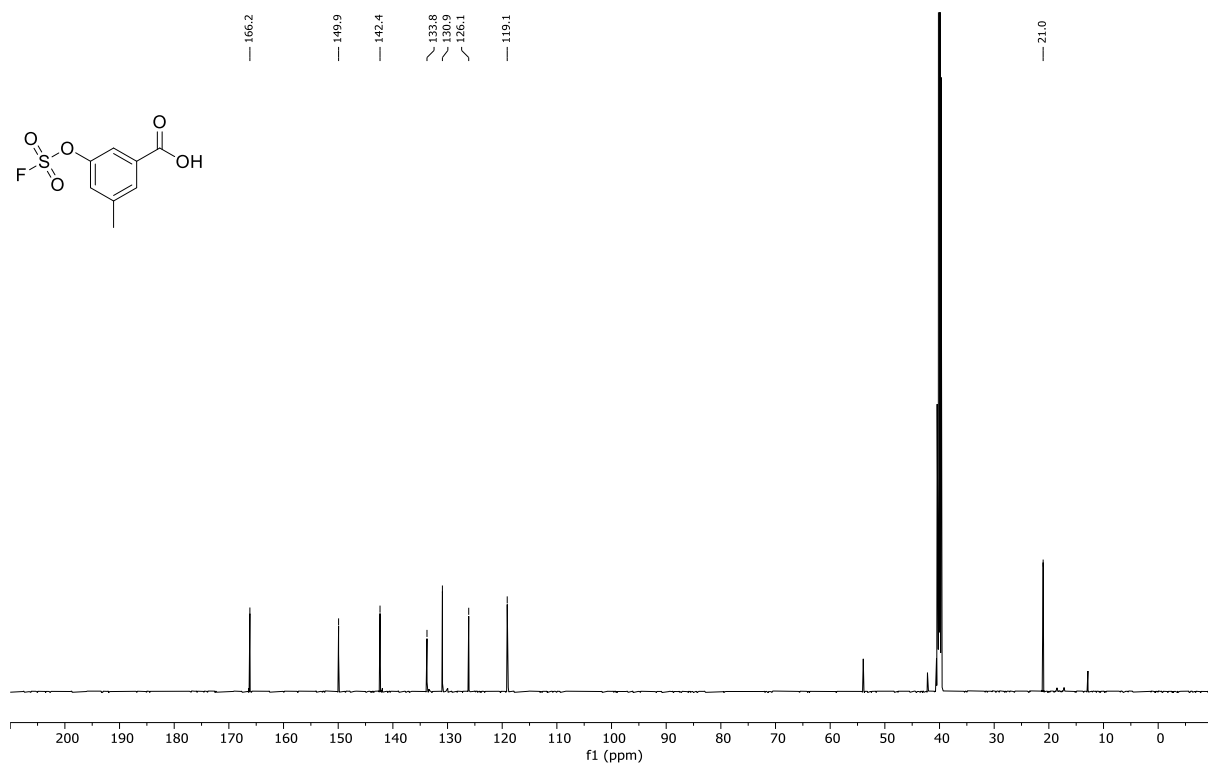
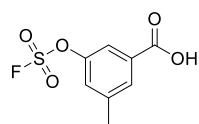
— 39.0



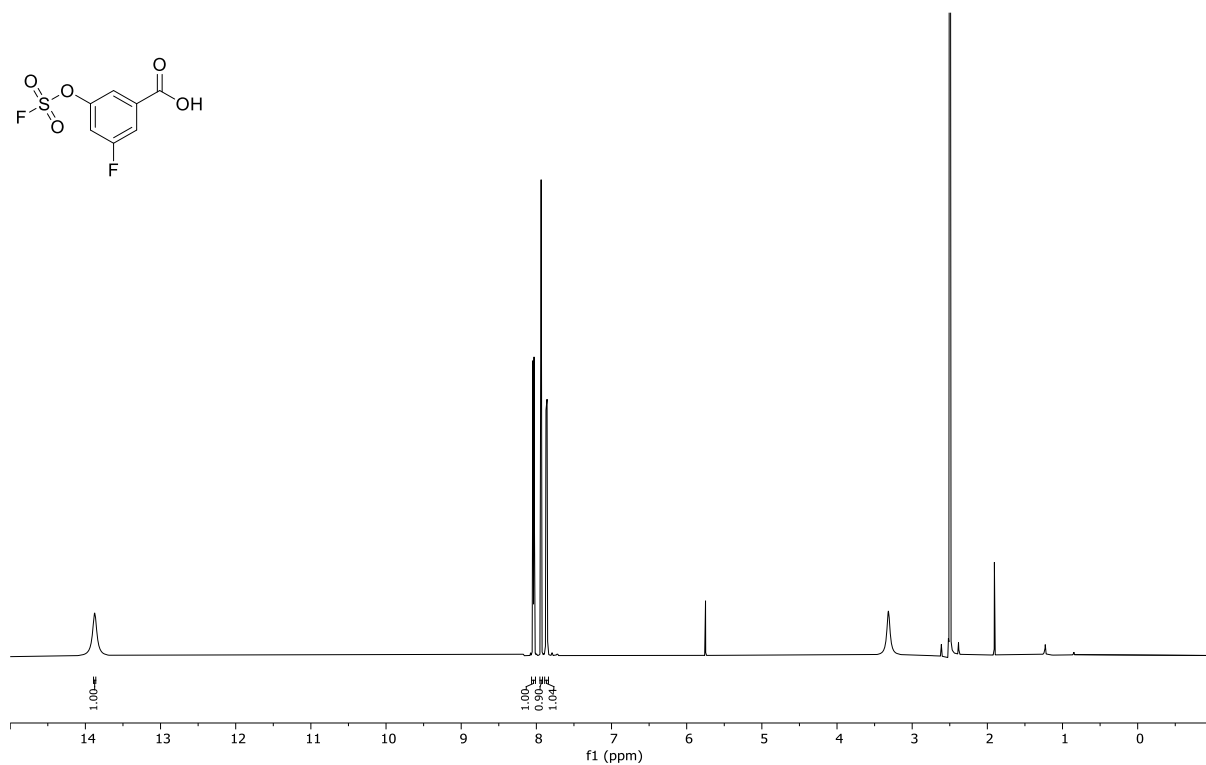
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **S2**



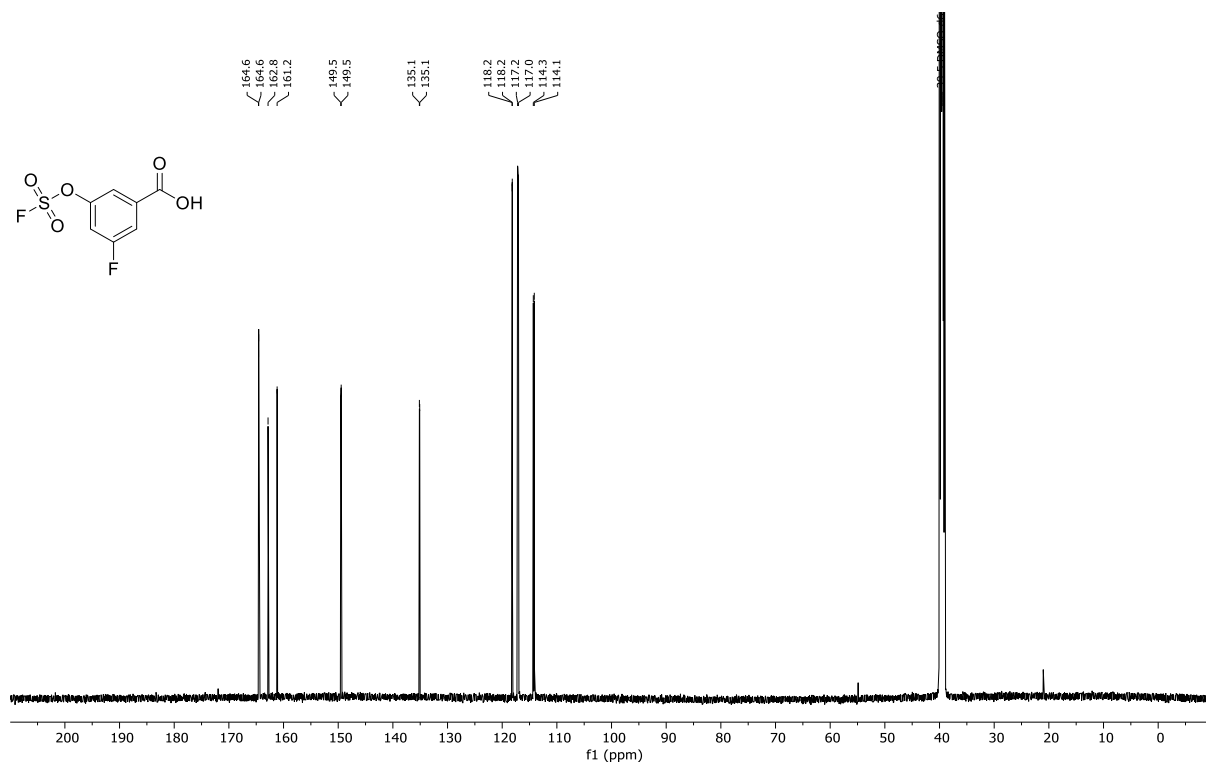
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **S2**



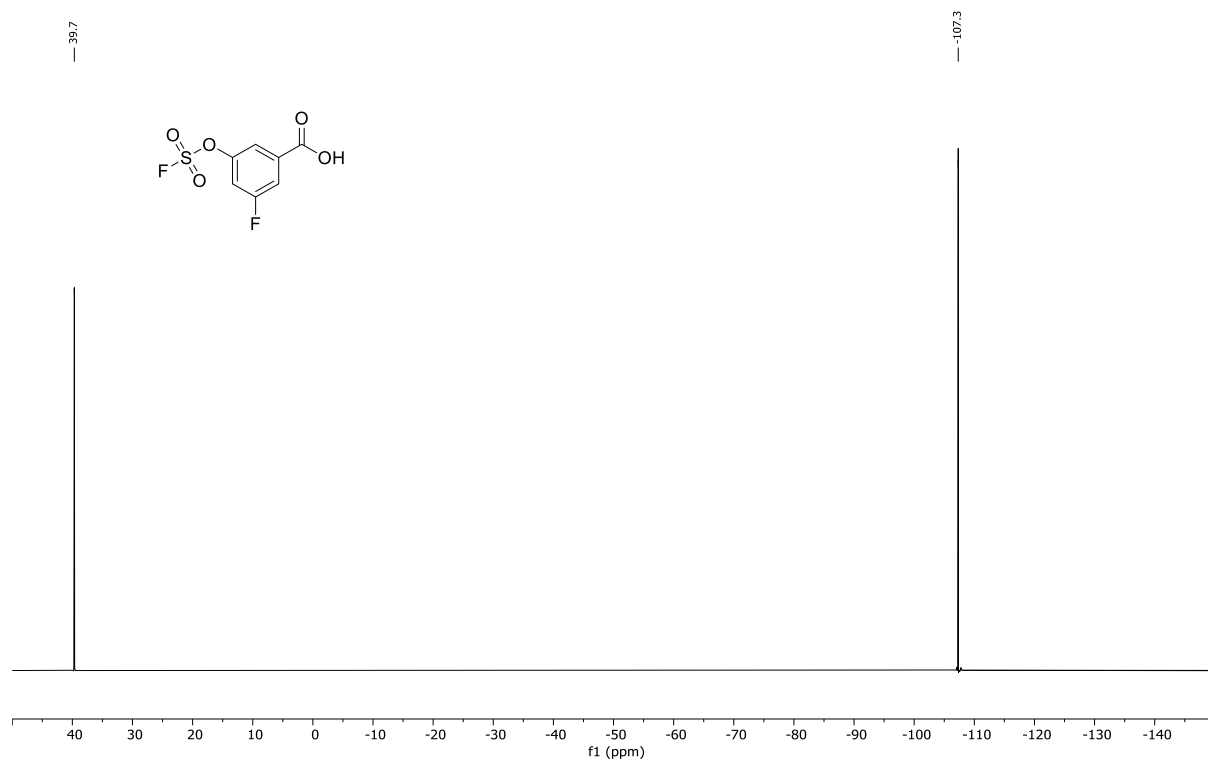
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **S3**



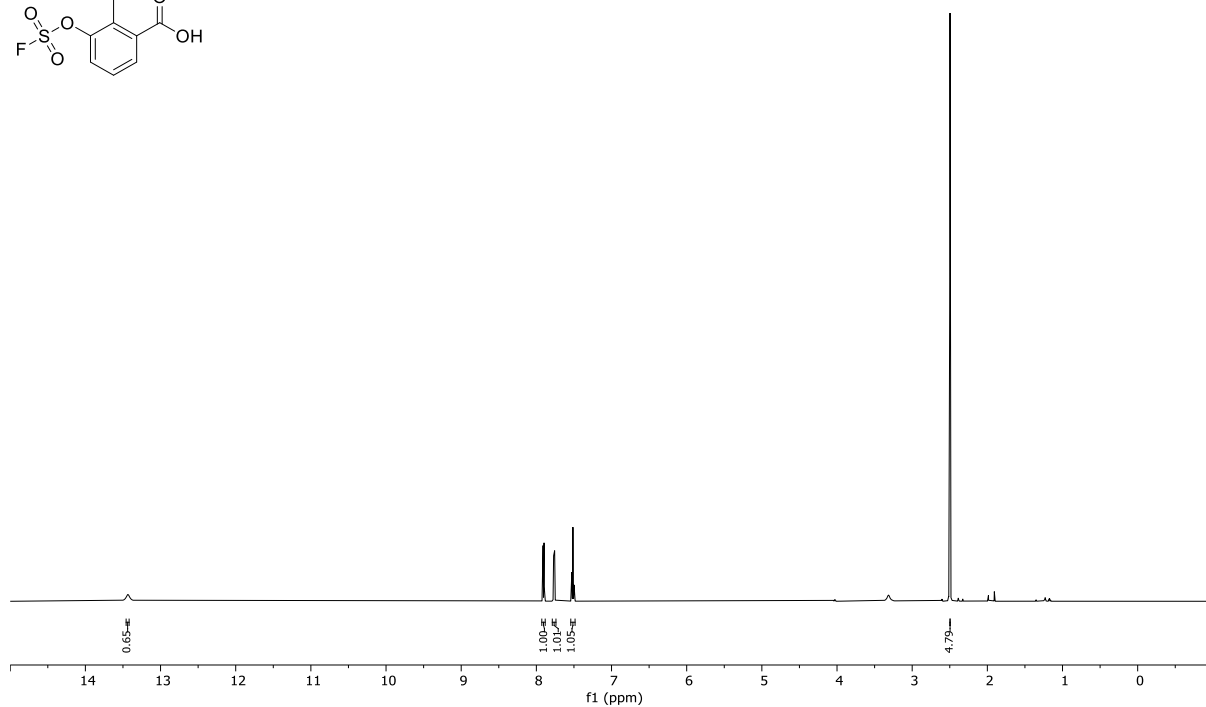
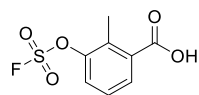
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **S3**



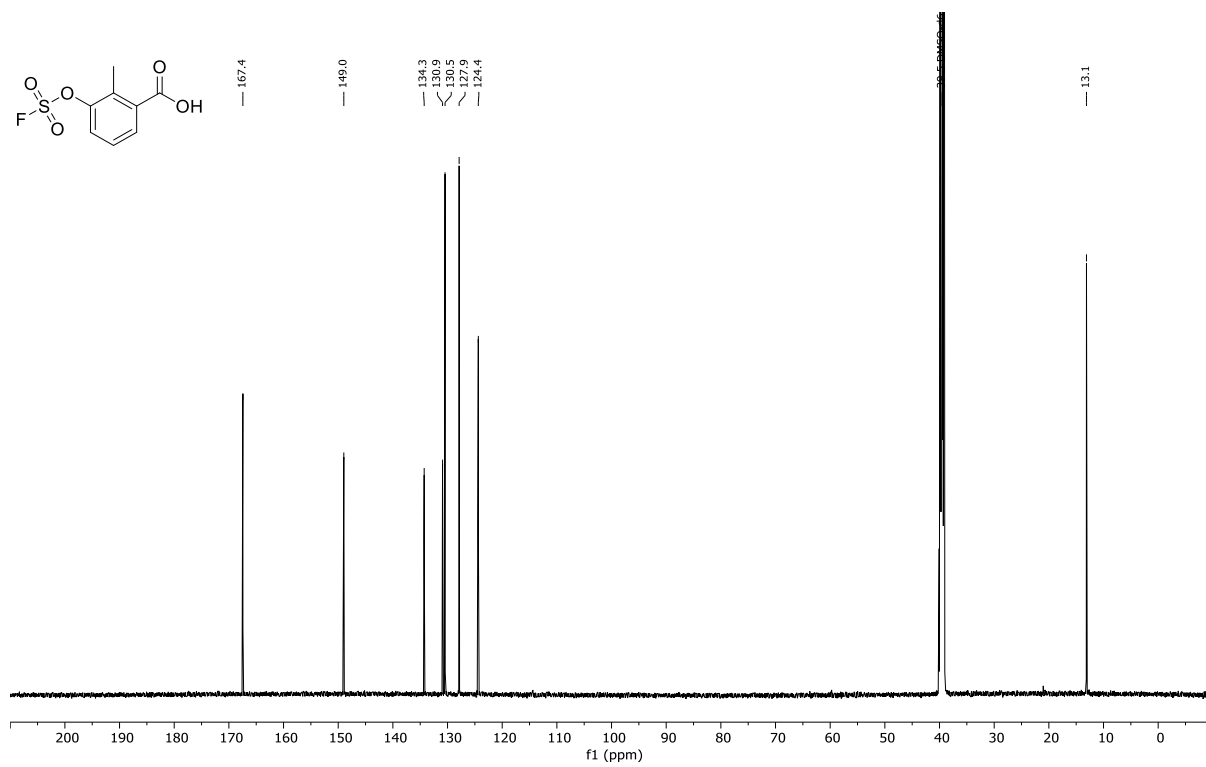
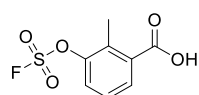
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **S3**



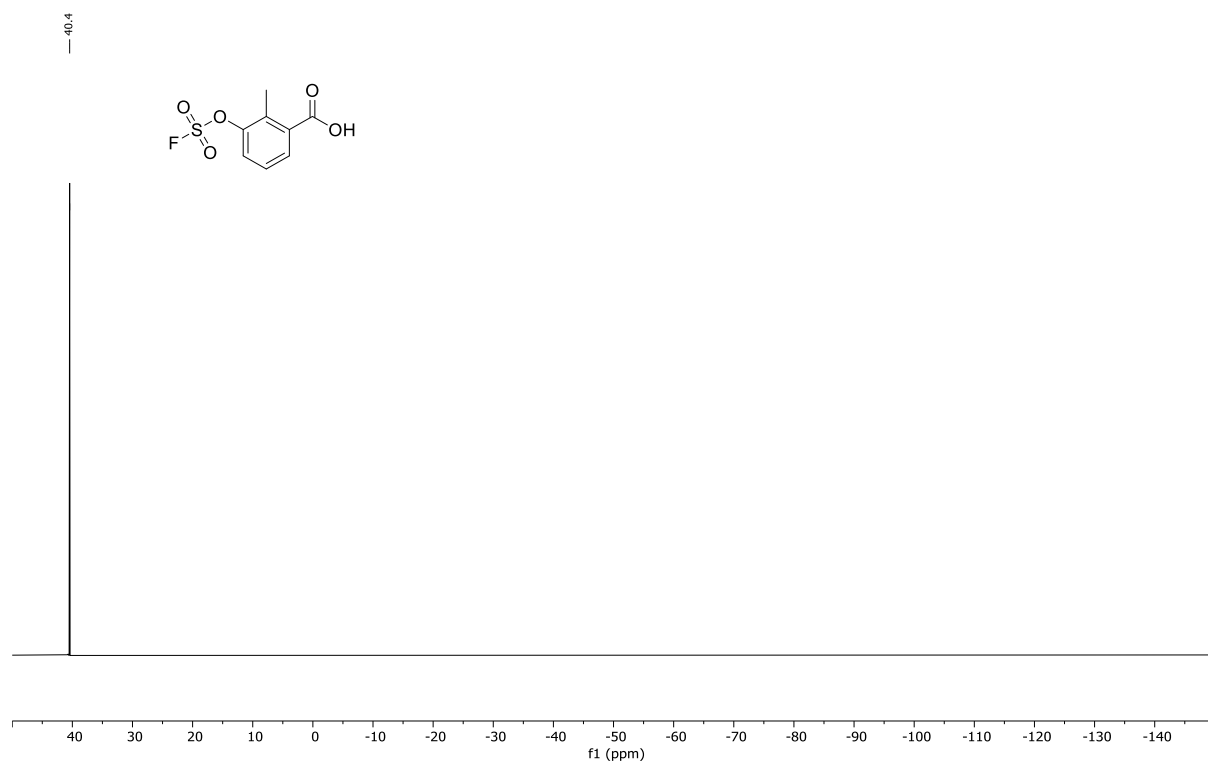
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **S4**



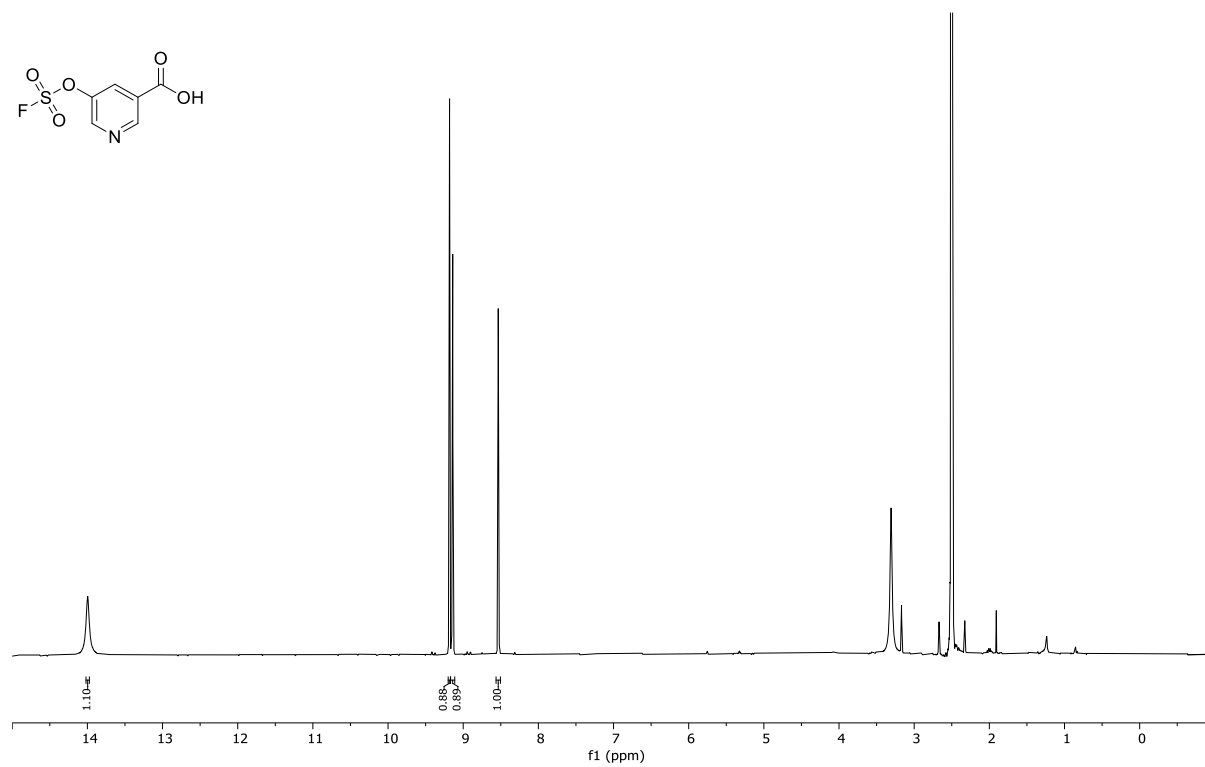
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **S4**



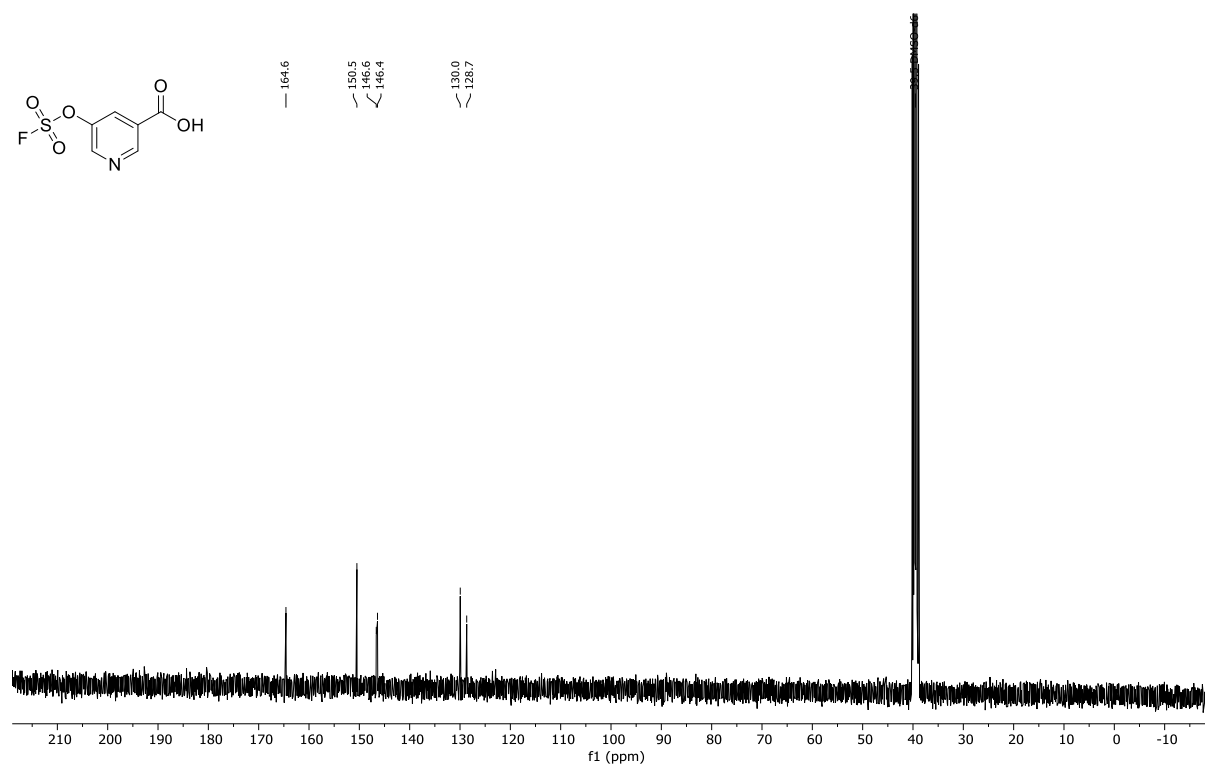
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **S4**



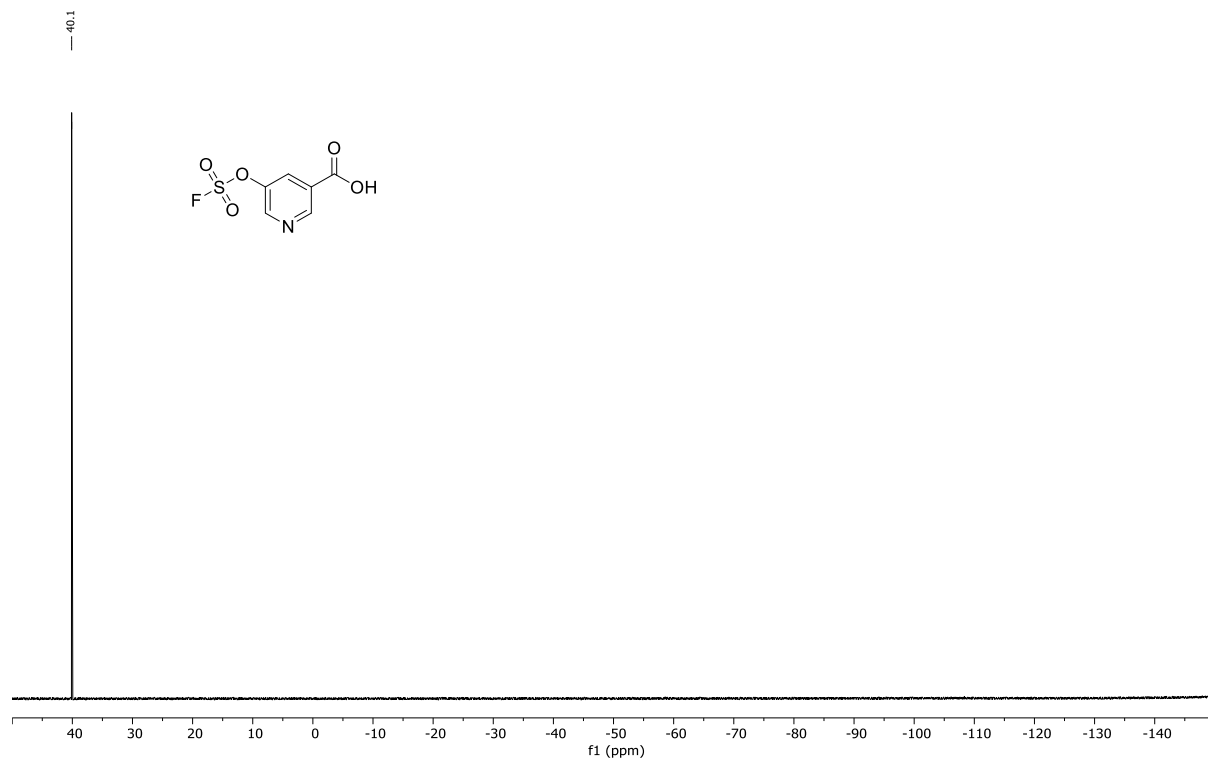
¹H NMR spectra (400 MHz, DMSO-*d*₆) of compound **S5**



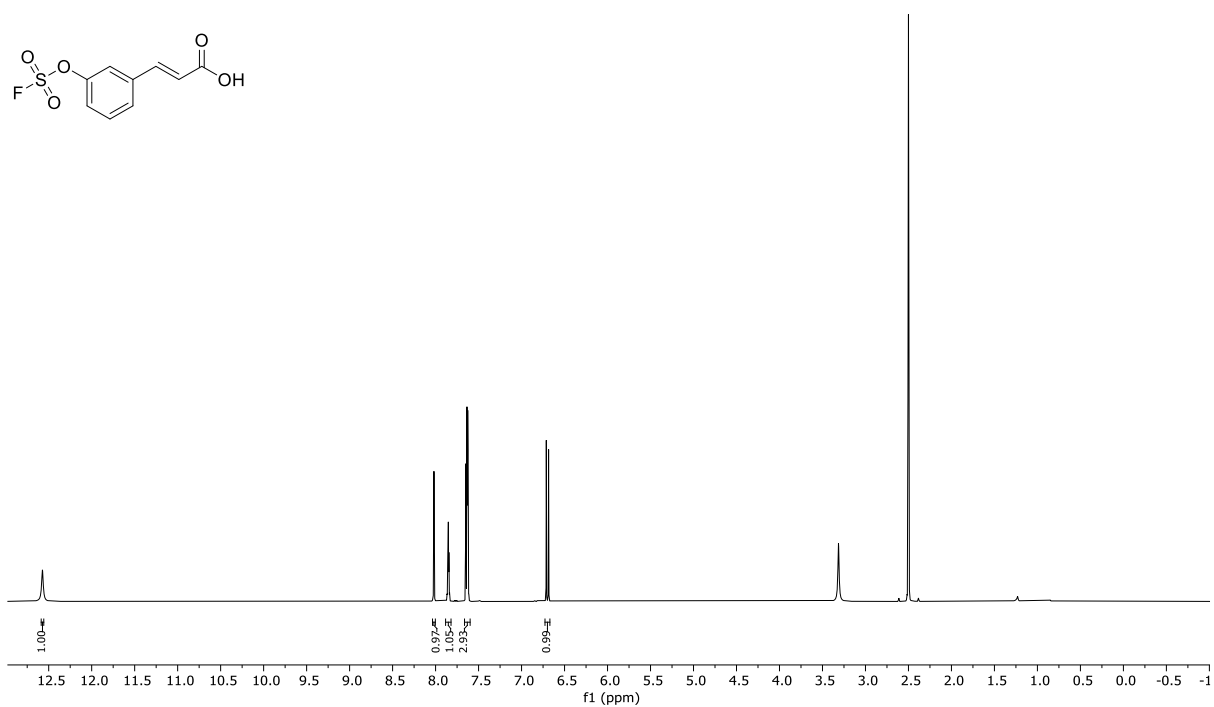
¹³C NMR spectra (101 MHz, DMSO-*d*₆) of compound **S5**



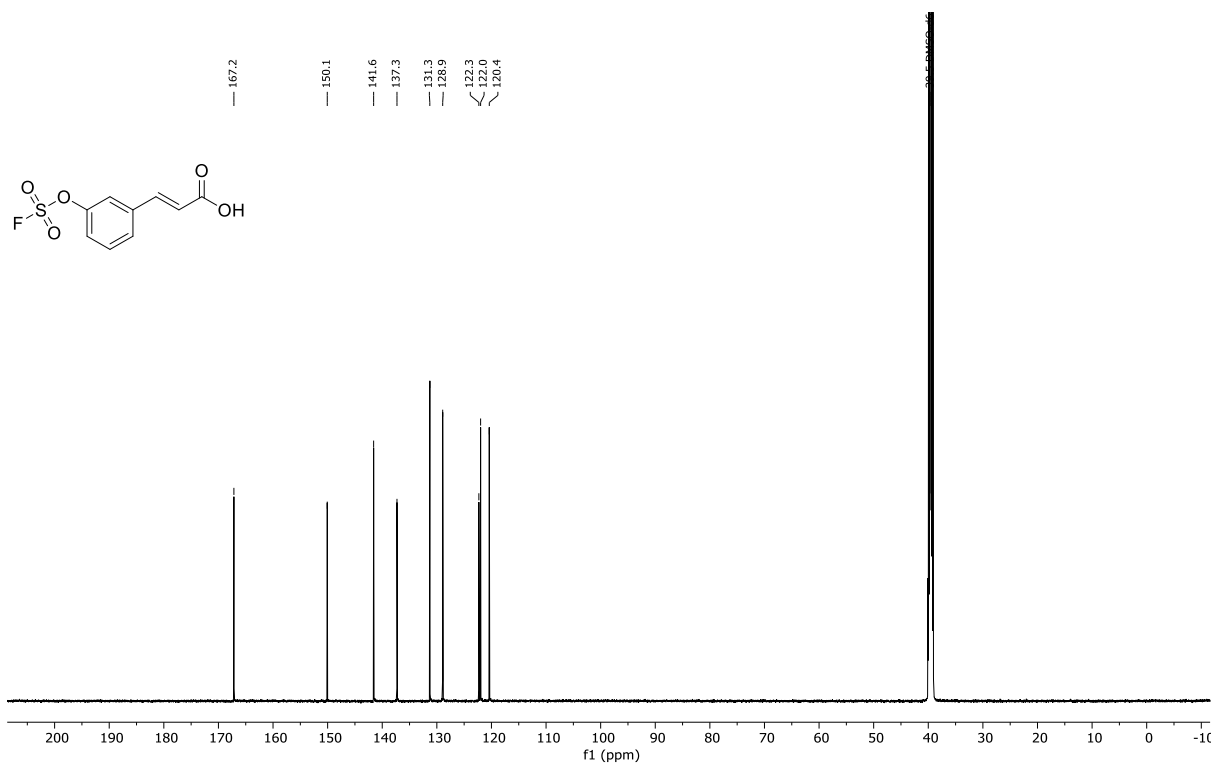
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **S5**



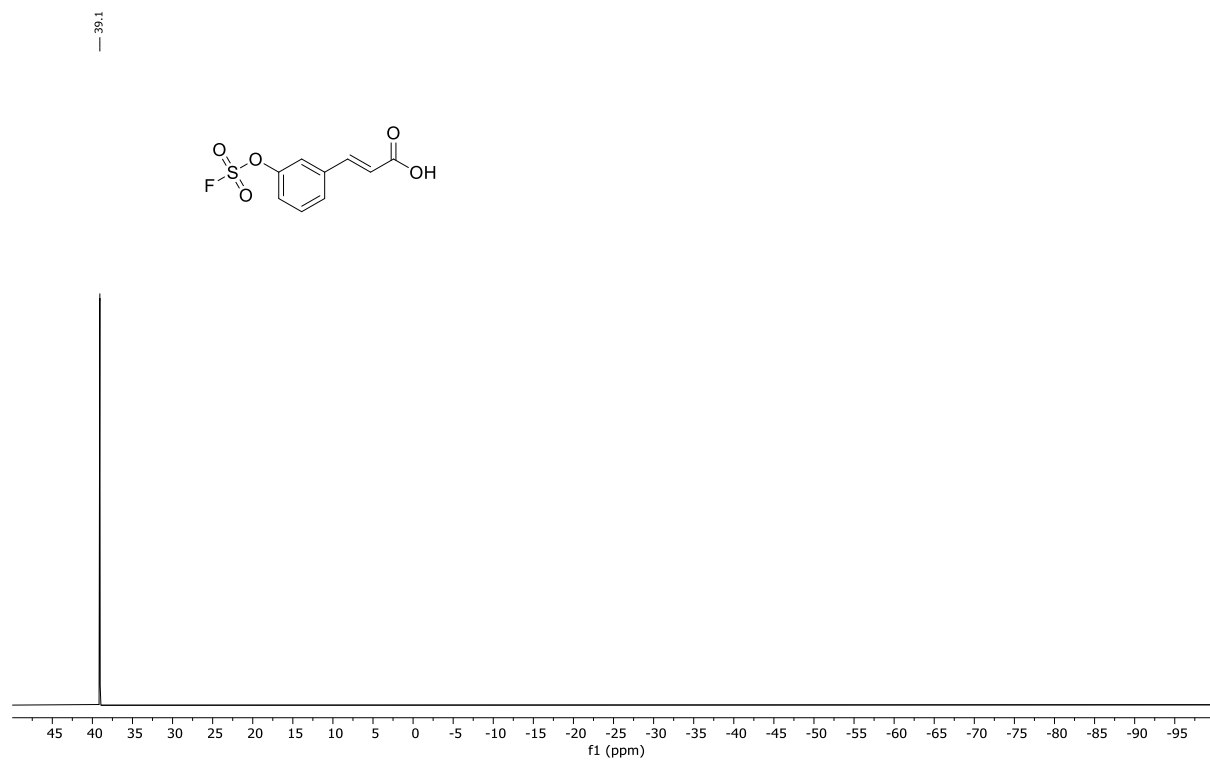
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **S6**



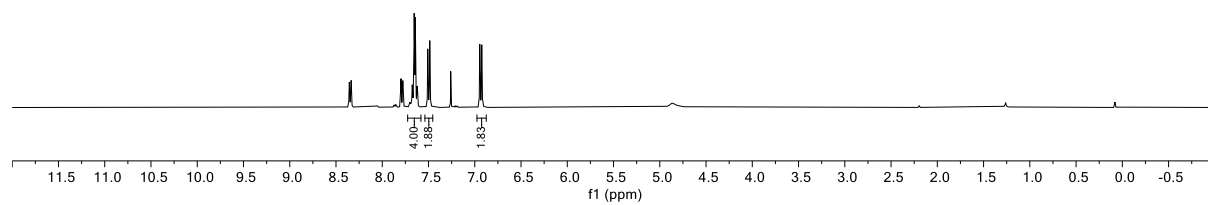
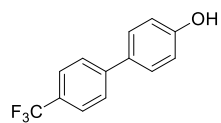
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **S6**



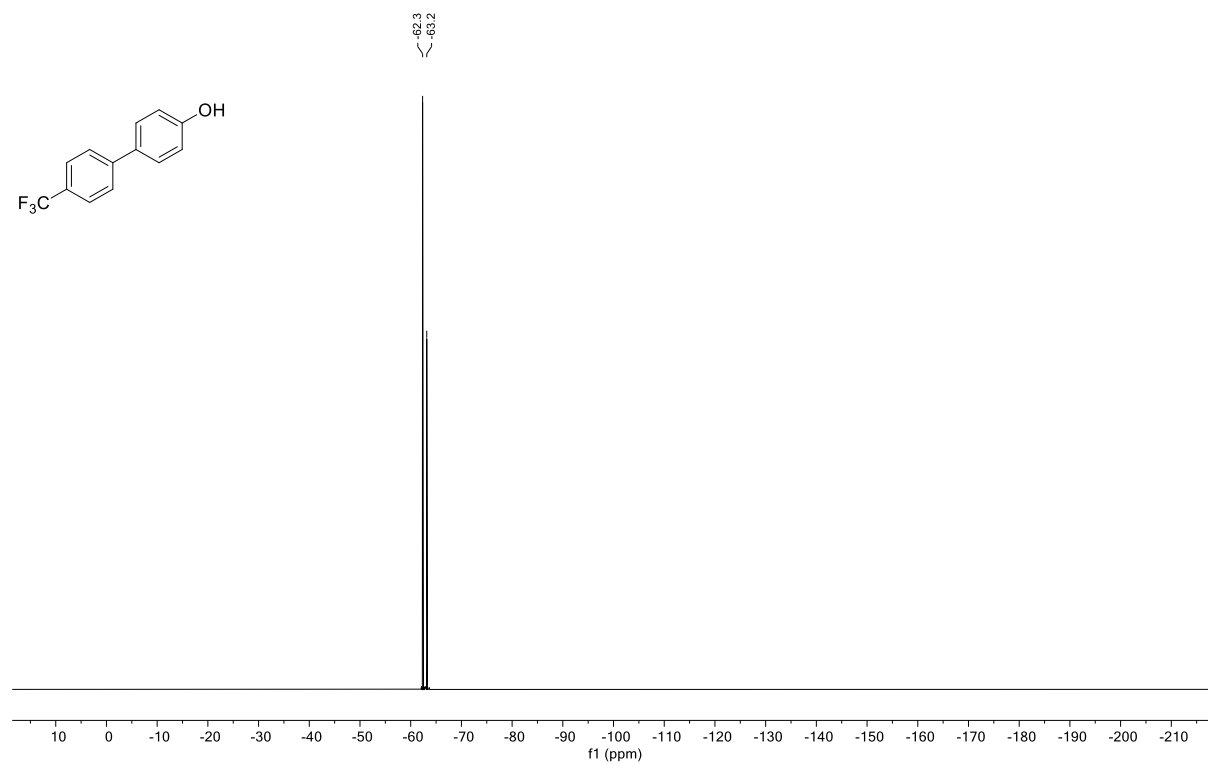
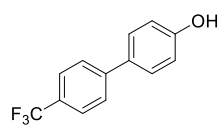
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **S6**



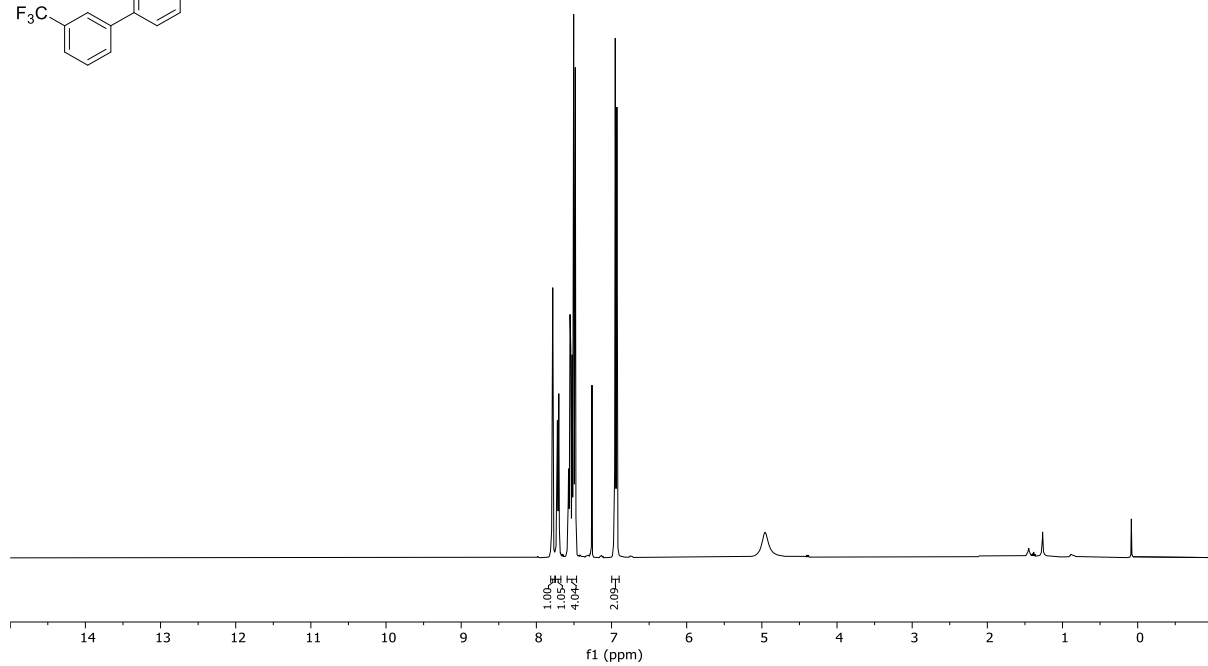
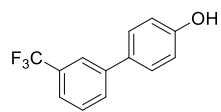
¹H NMR spectra (400 MHz, CDCl₃) of compound **S7**



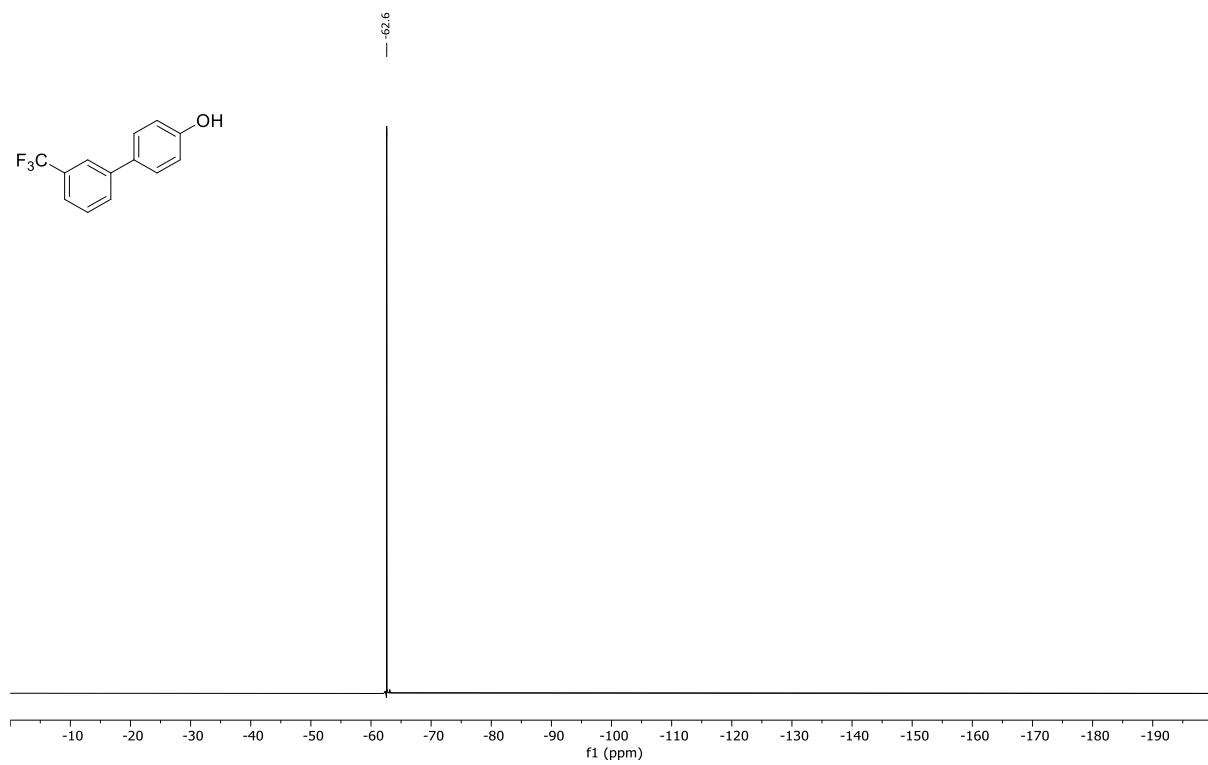
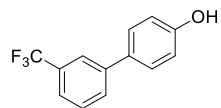
¹⁹F NMR spectra (376 MHz, CDCl₃) of compound **S7**



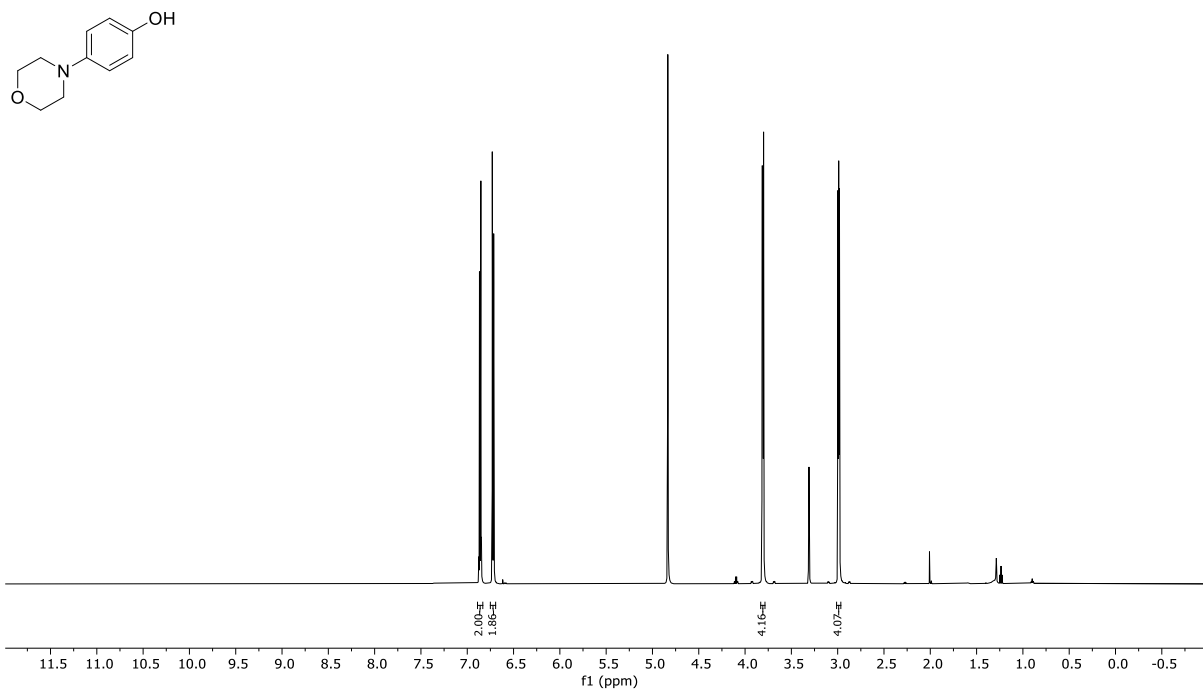
¹H NMR spectra (400 MHz, CDCl₃) of compound **S8**



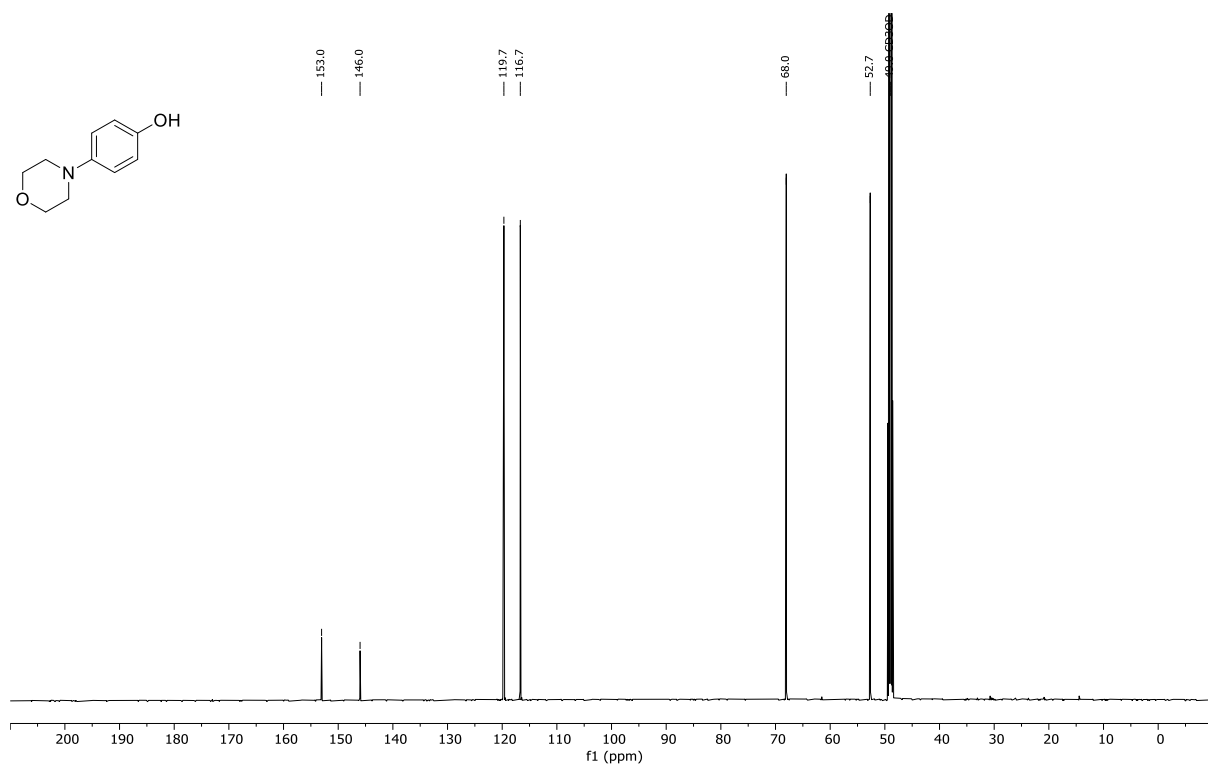
¹⁹F NMR spectra (376 MHz, CDCl₃) of compound **S8**



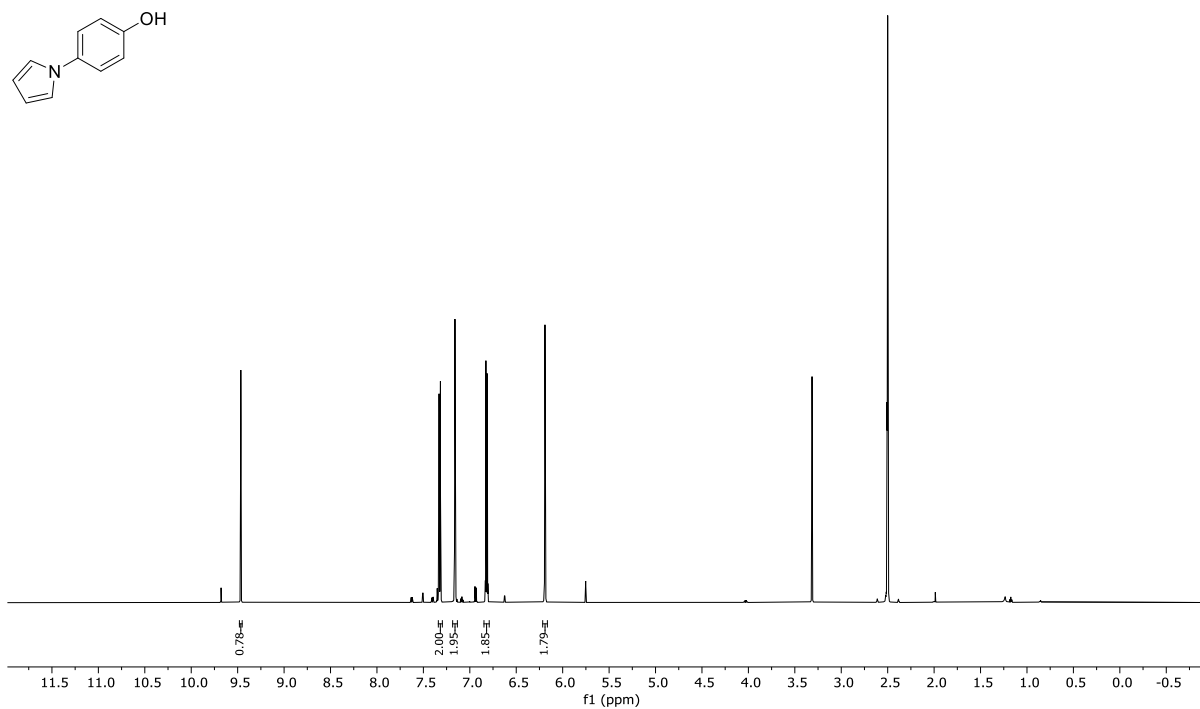
¹H NMR spectra (600 MHz, MeOD-d₄) of compound **S9**



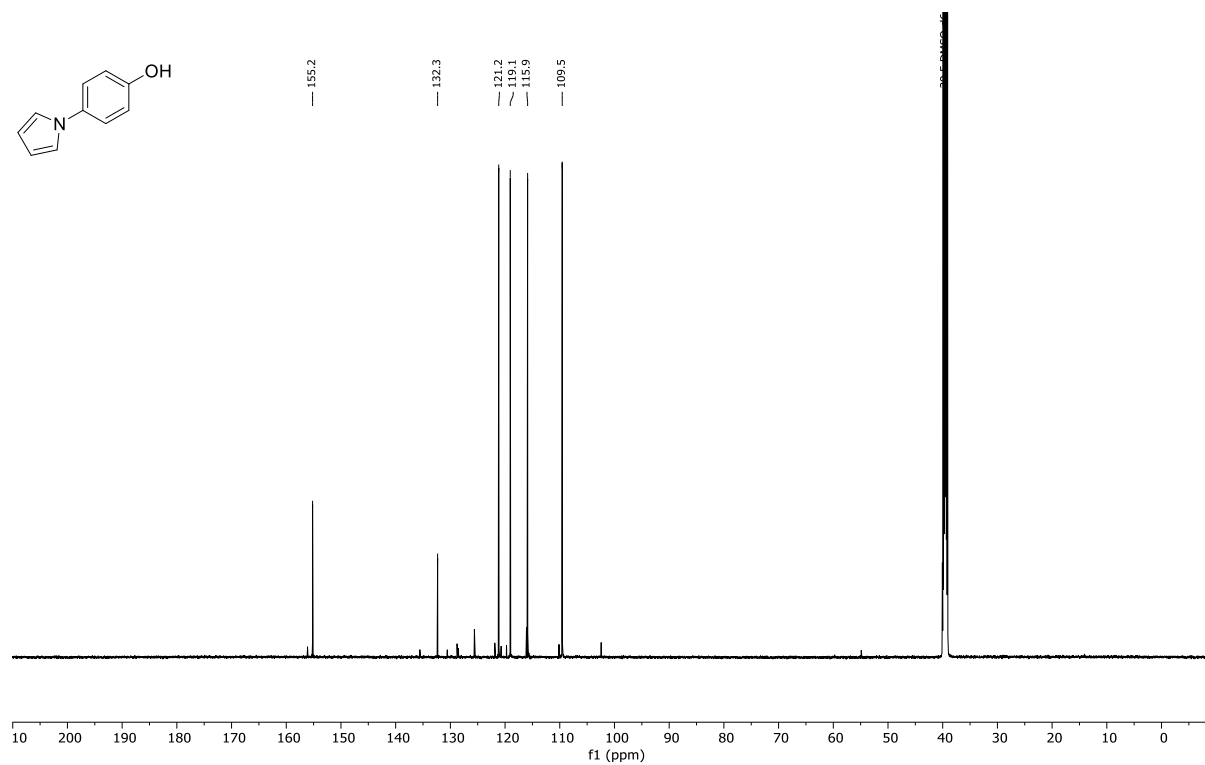
¹³C NMR spectra (151 MHz, MeOD-d₄) of compound **S9**



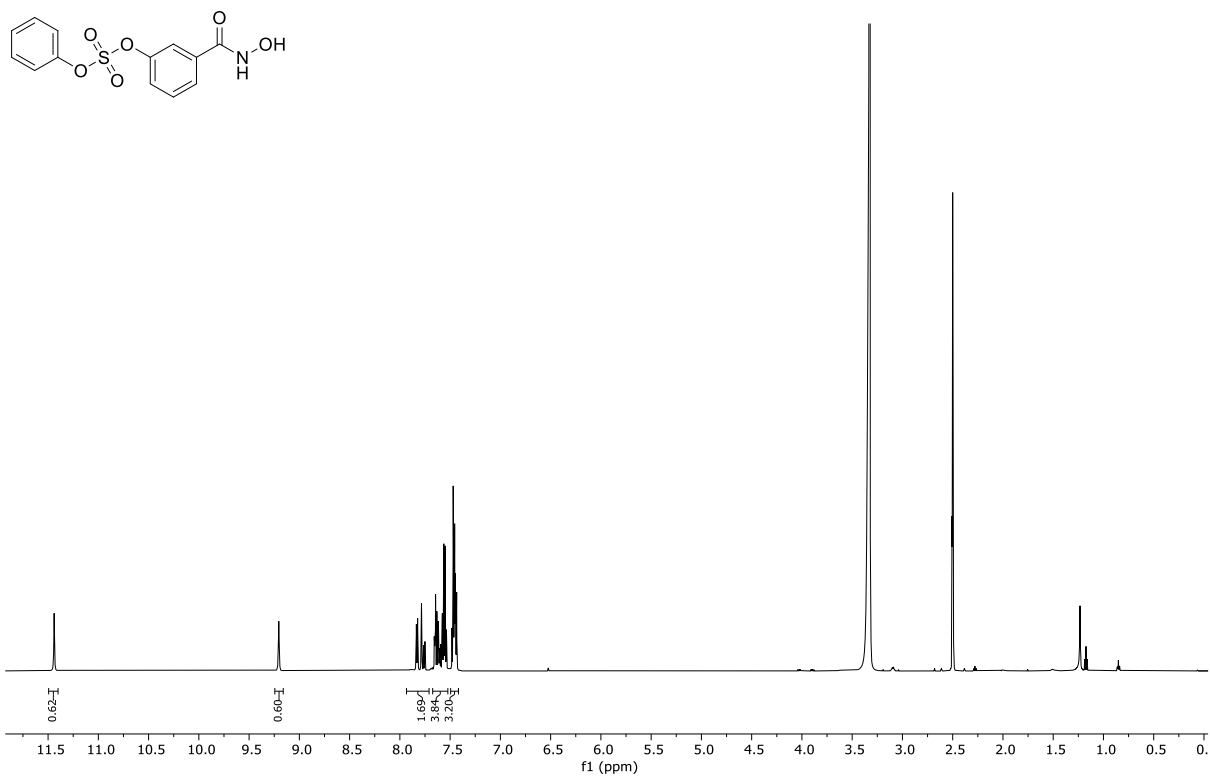
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **S10**



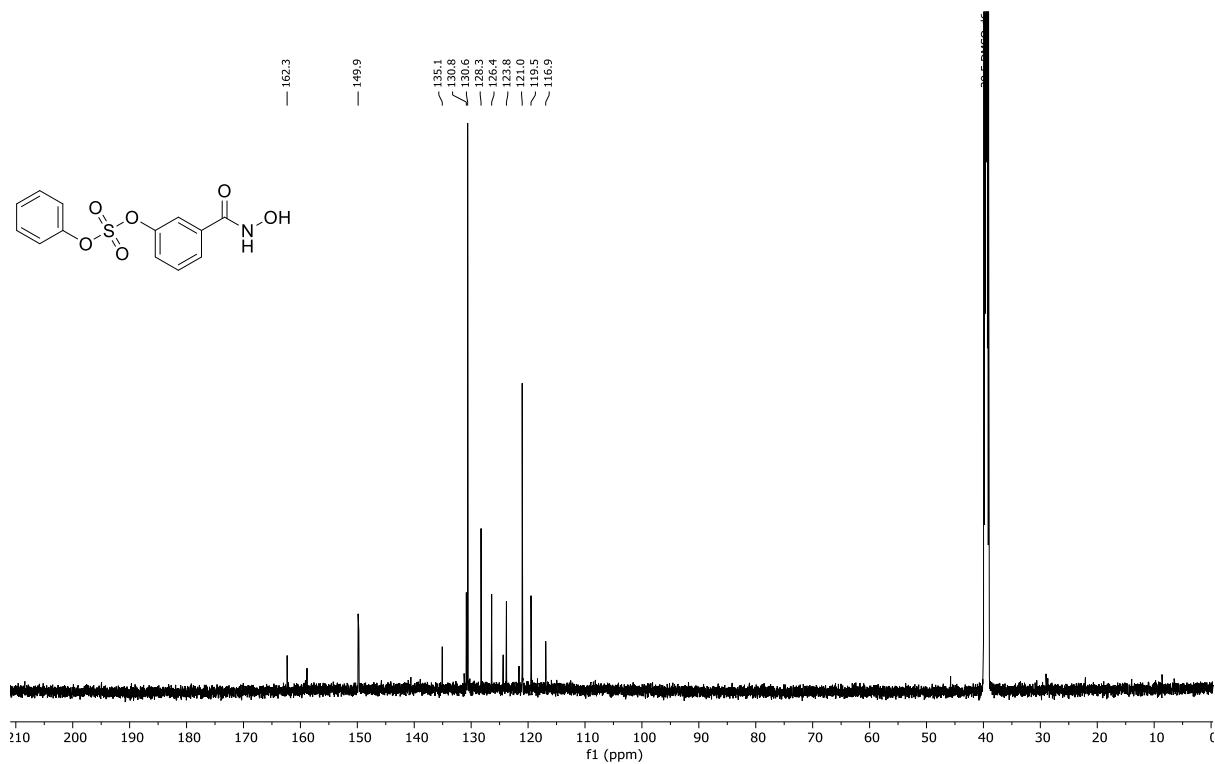
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **S10**



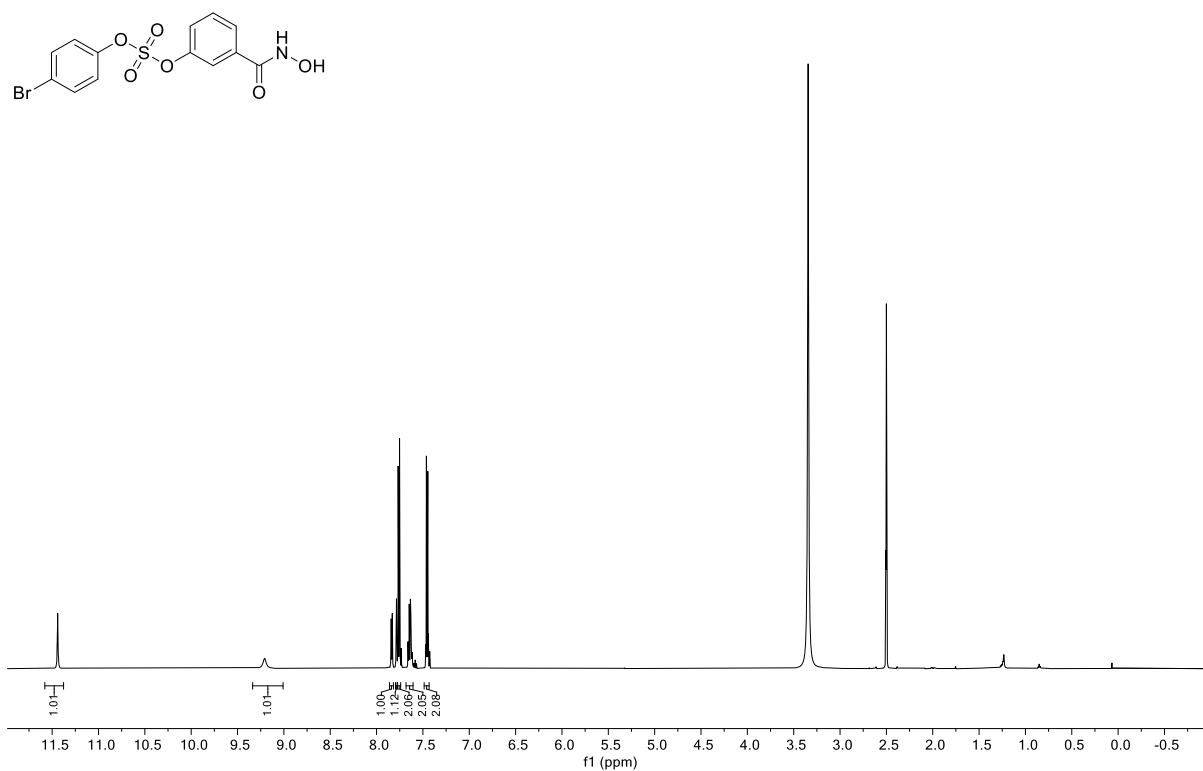
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A1**



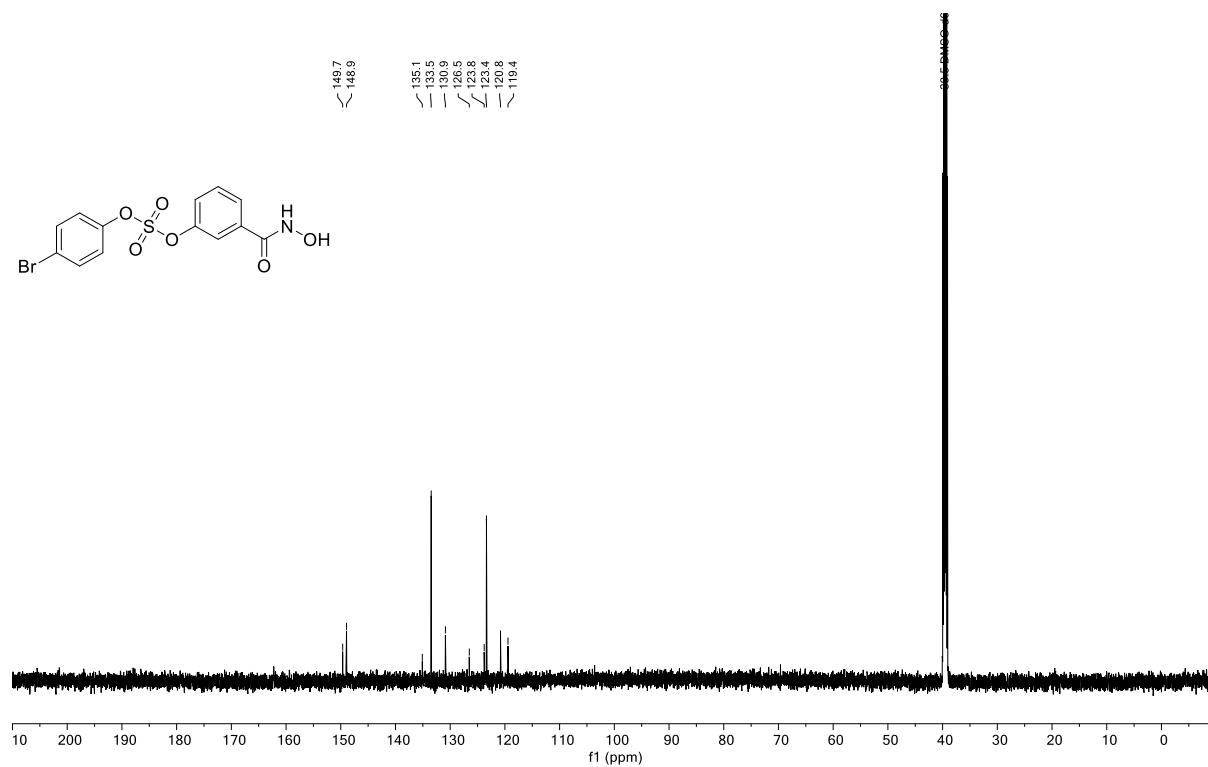
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A1** (minor contamination with TFA is observed at 158.8 ppm)



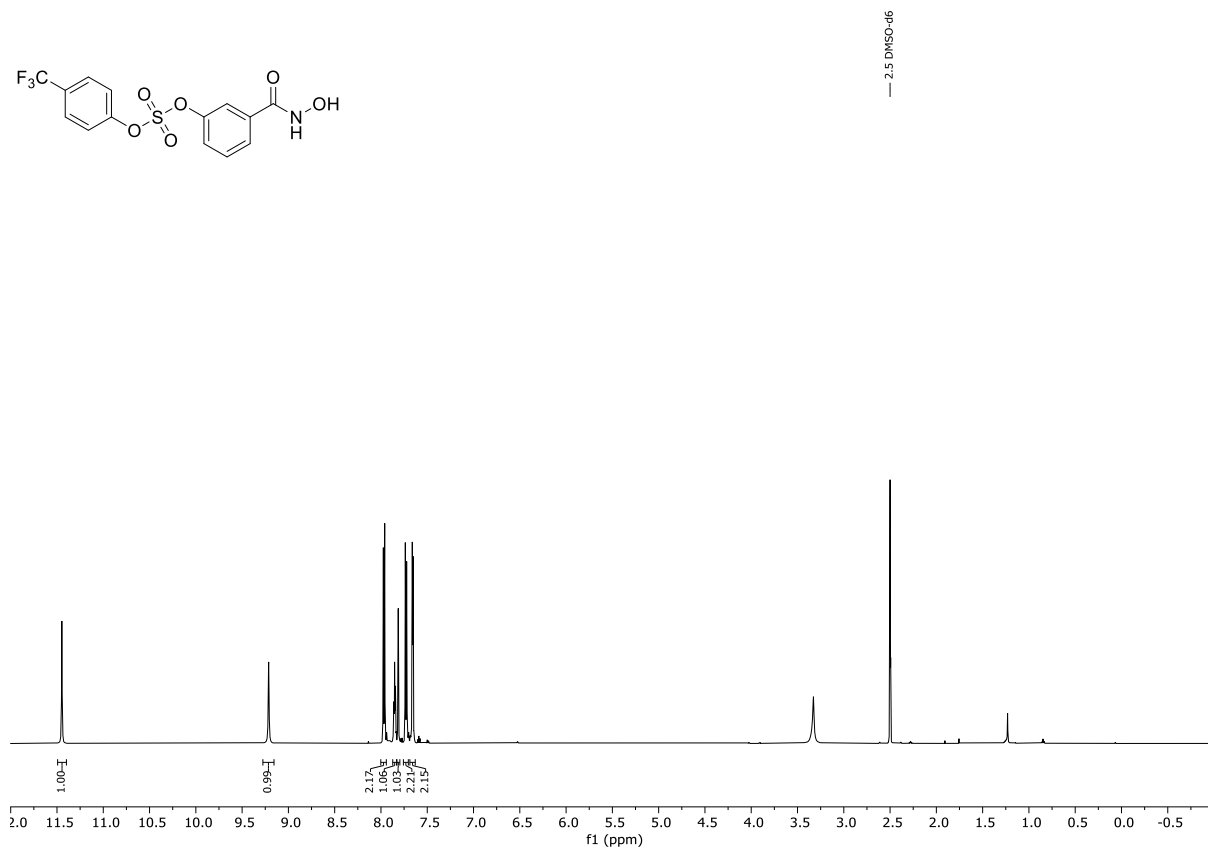
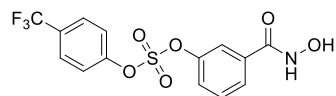
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **A2**



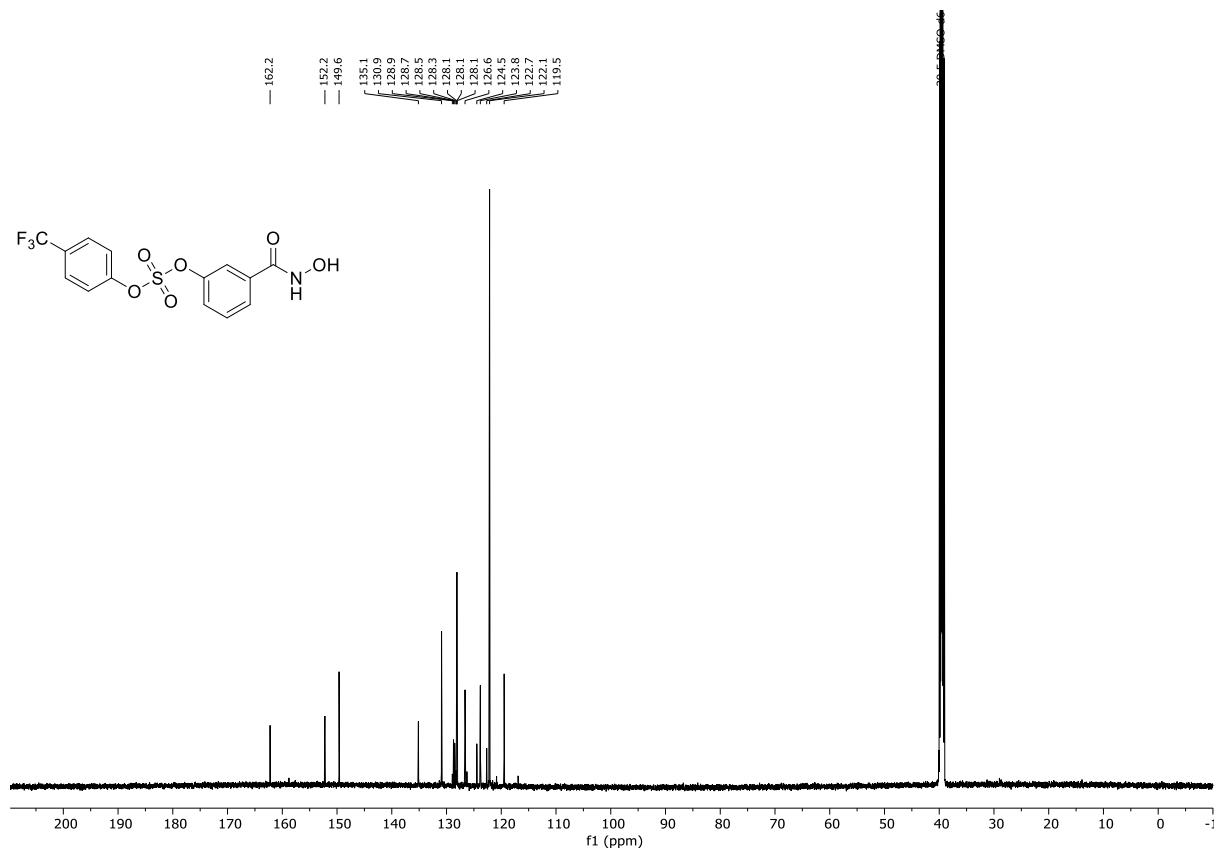
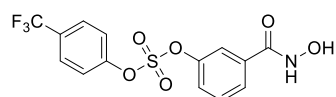
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **A2**



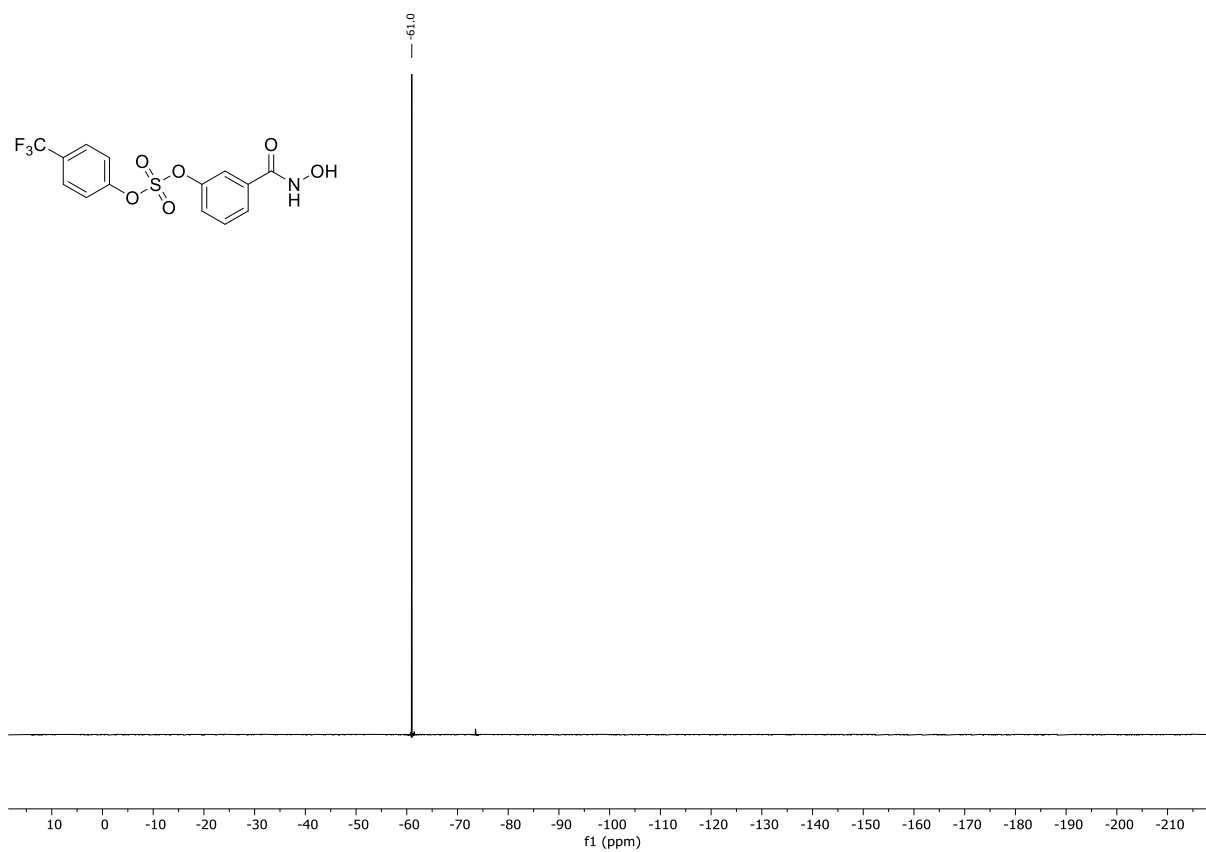
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A3**



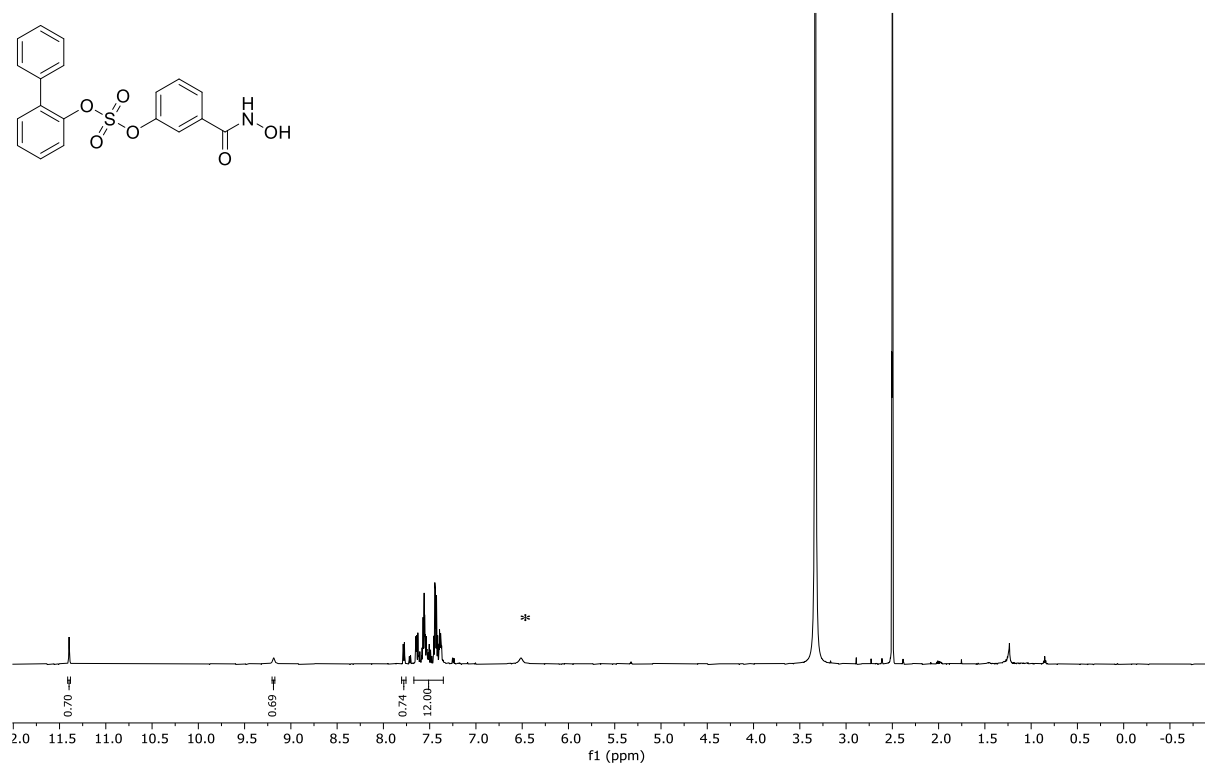
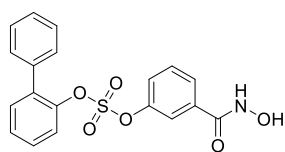
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A3**



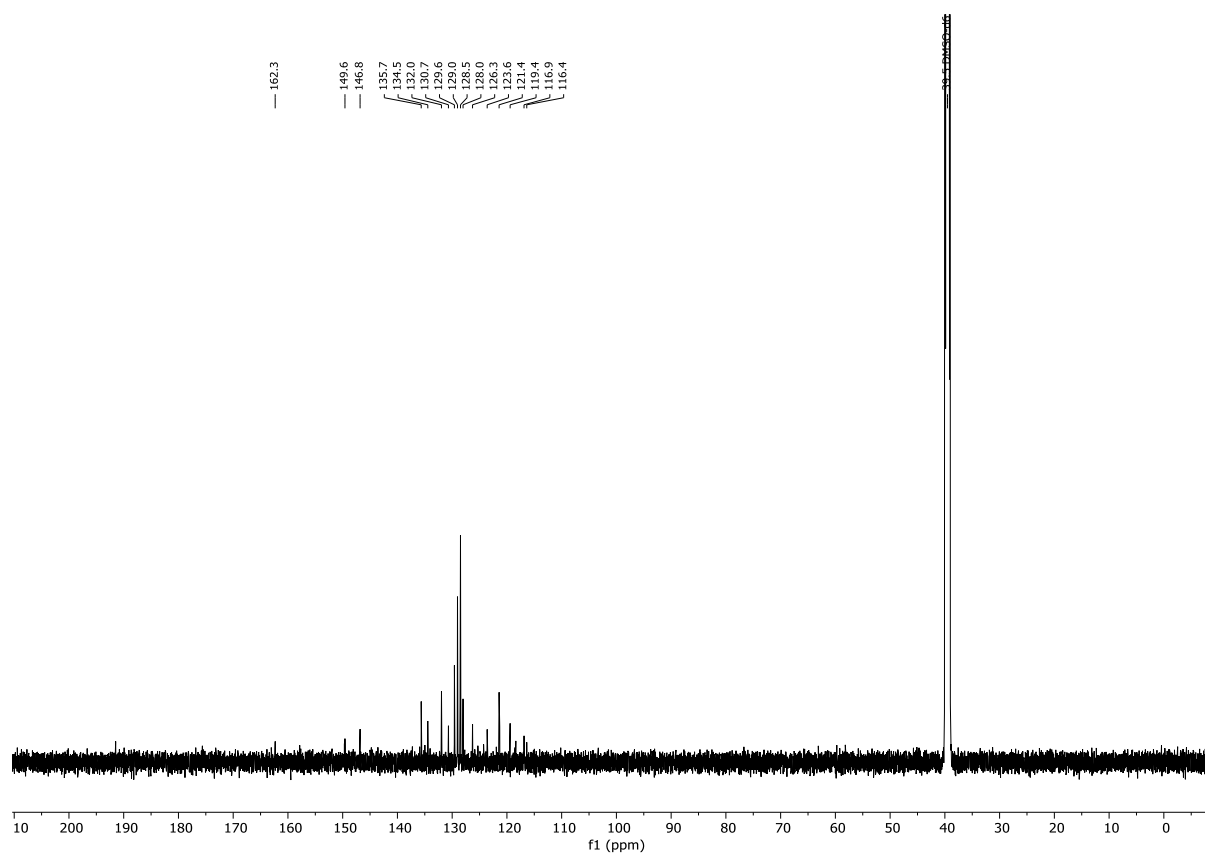
^{19}F NMR spectra (376 MHz, CDCl_3) of compound **A3**



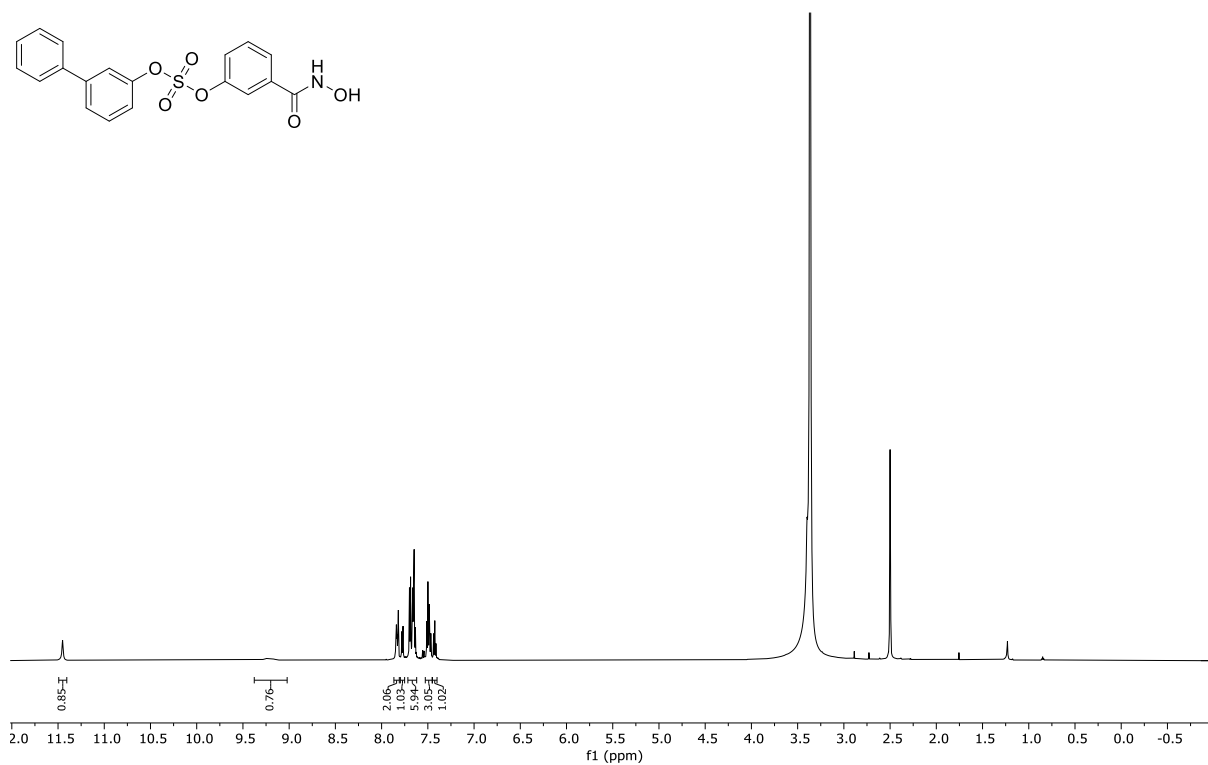
^1H NMR spectra (600 MHz, $\text{DMSO-}d_6$) of compound **A4** (* unidentified impurity)



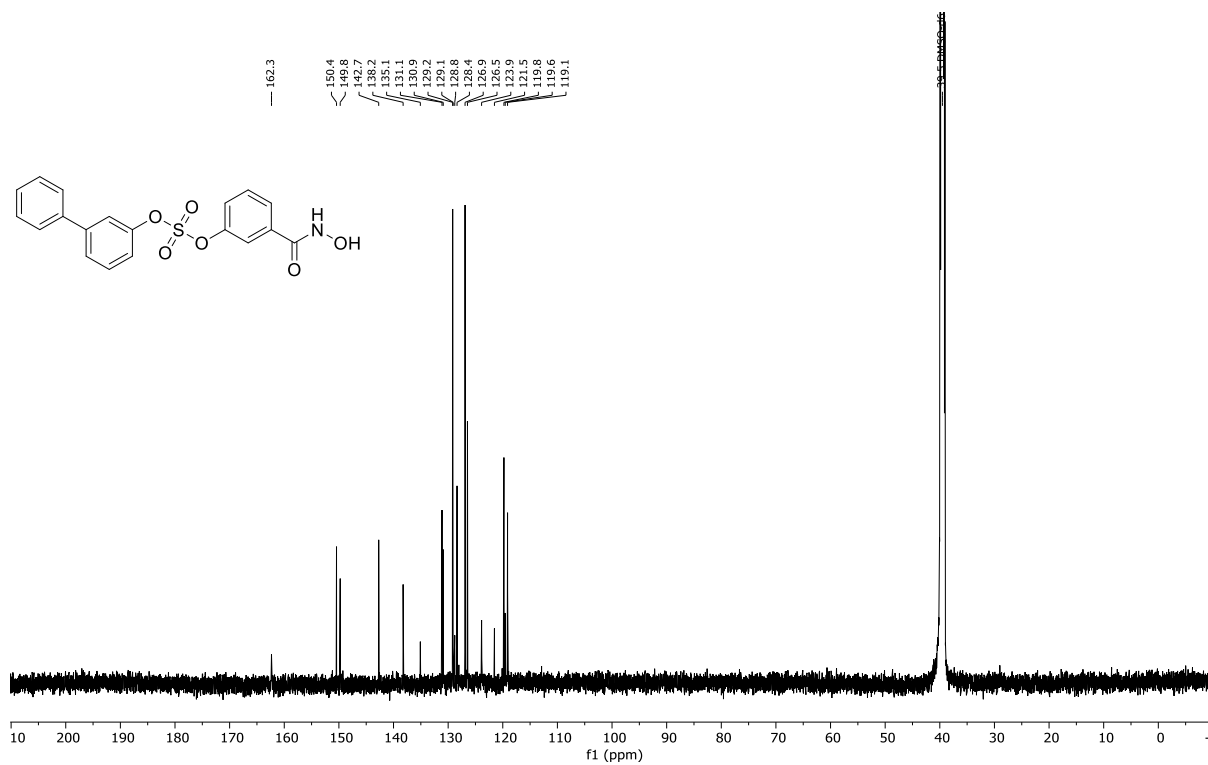
^{13}C NMR spectra (151 MHz, $\text{DMSO-}d_6$) of compound **A4** (minor contamination with TFA is observed at 158.8 ppm)



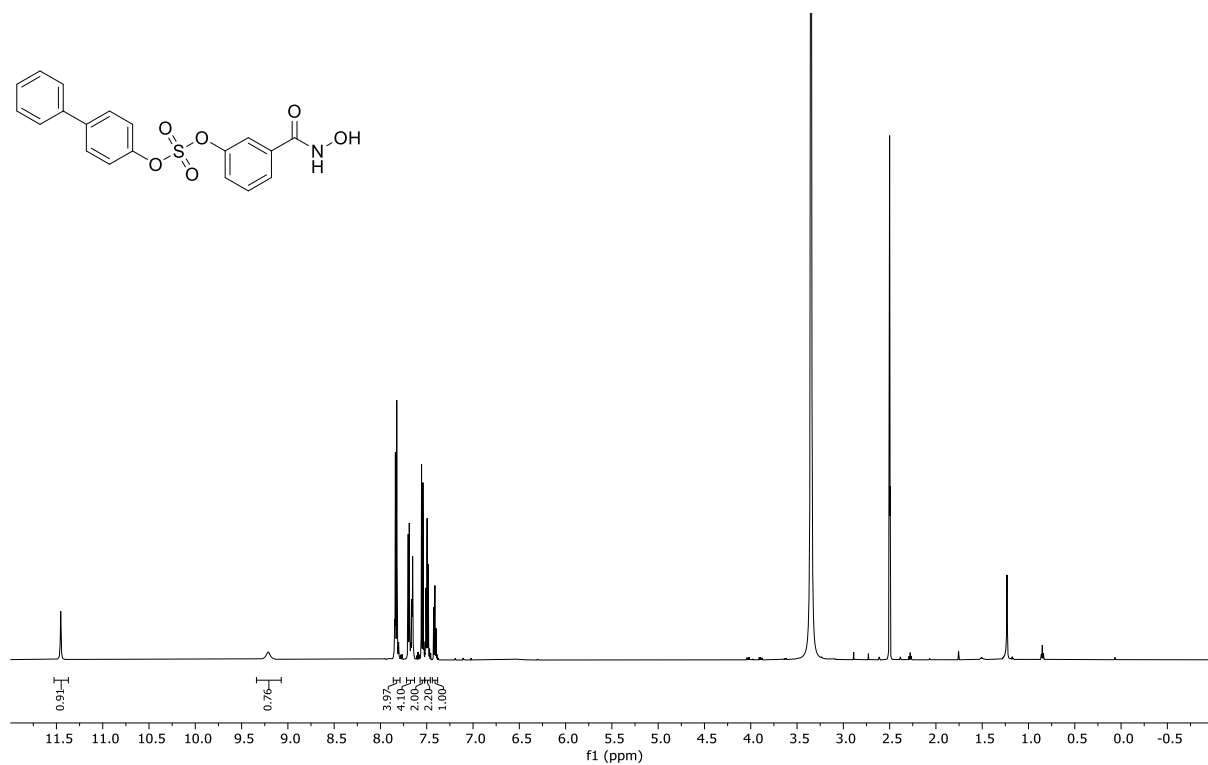
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A5**



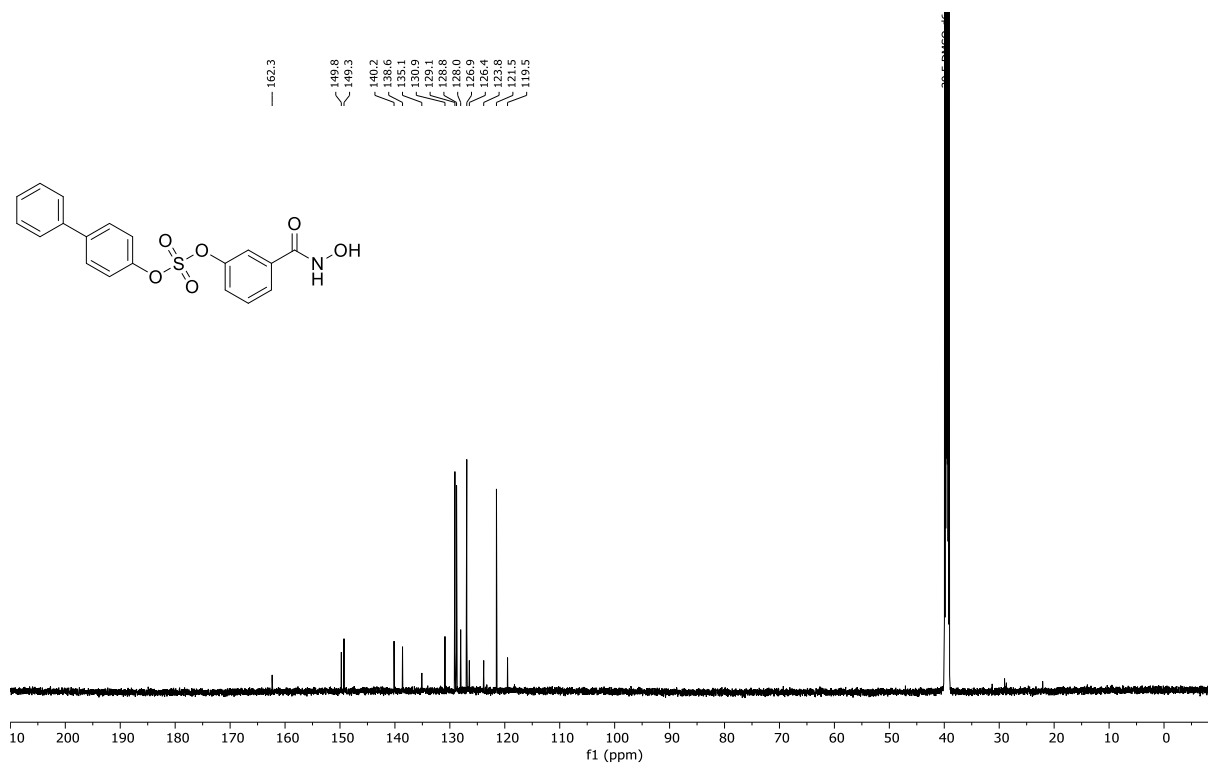
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A5**



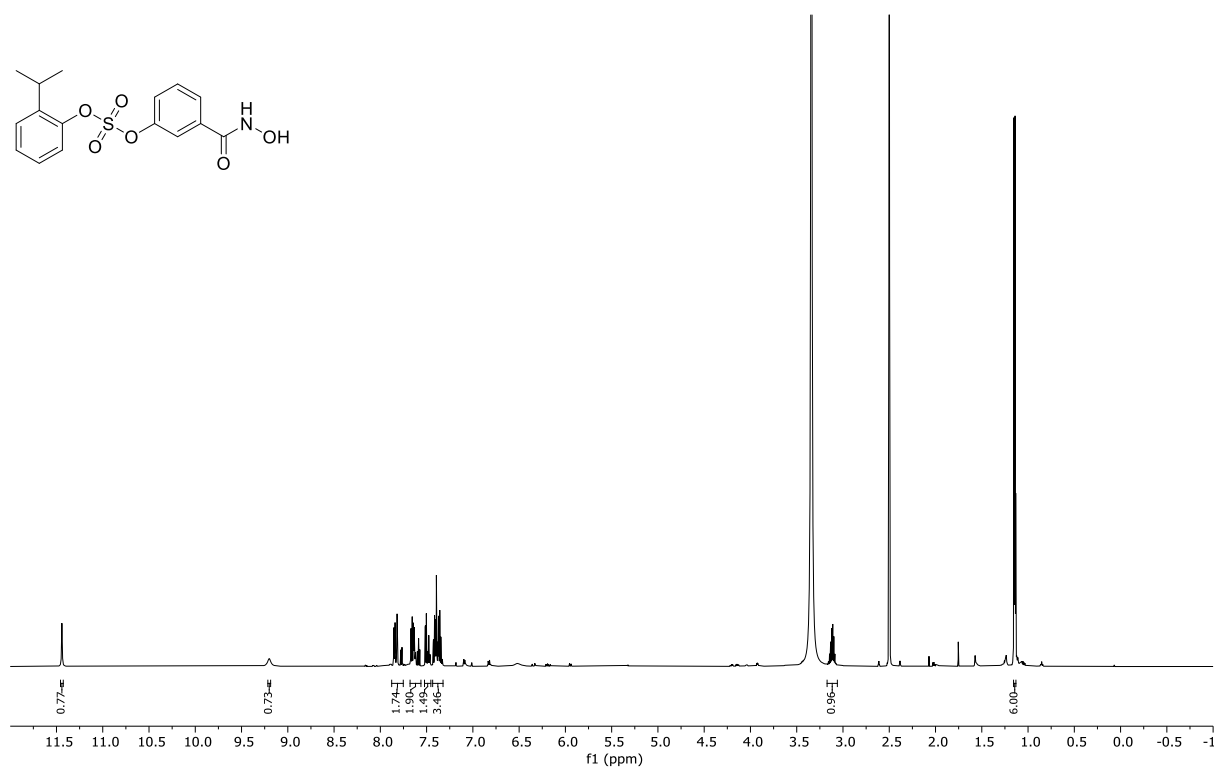
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A6**



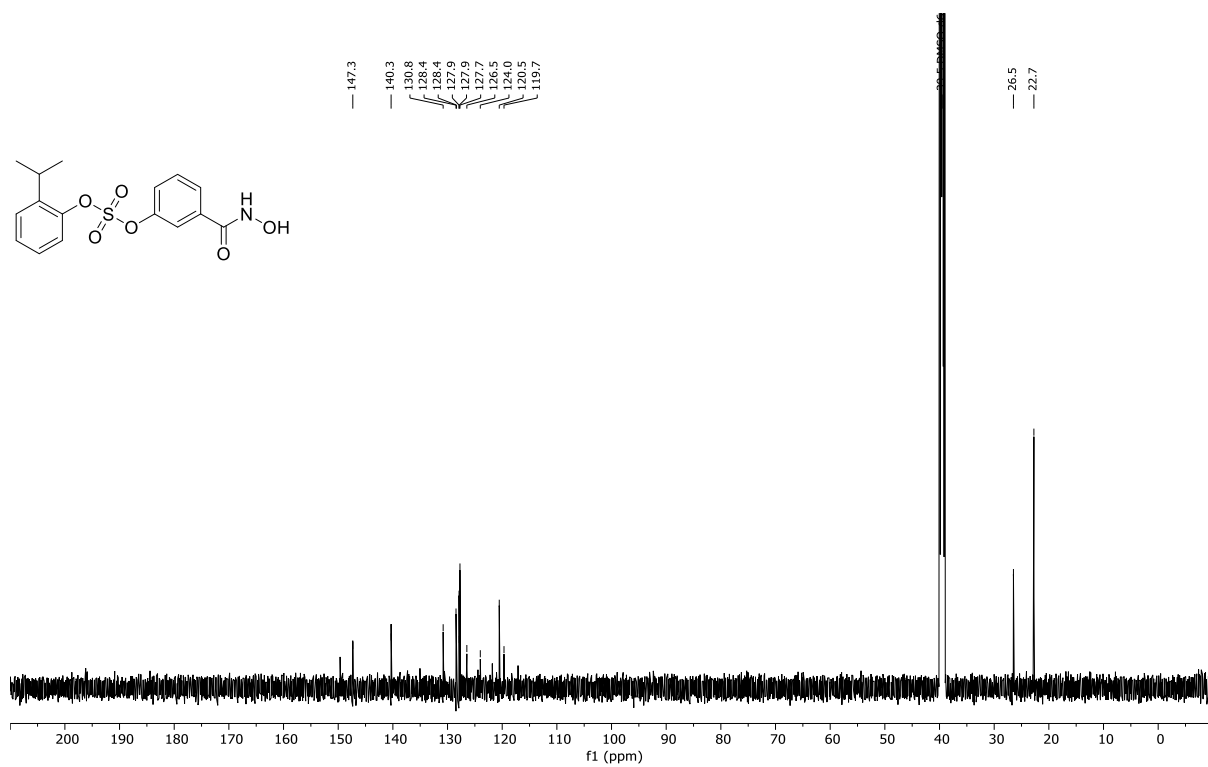
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A6**



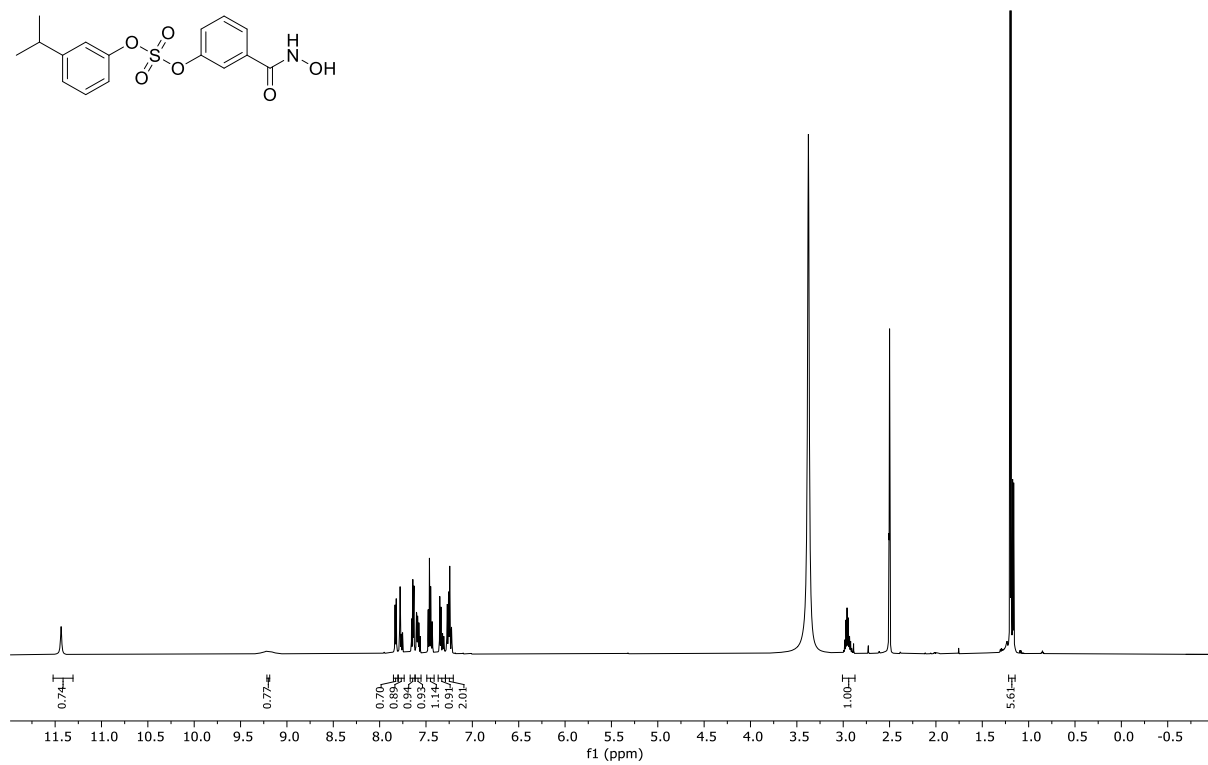
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A7**



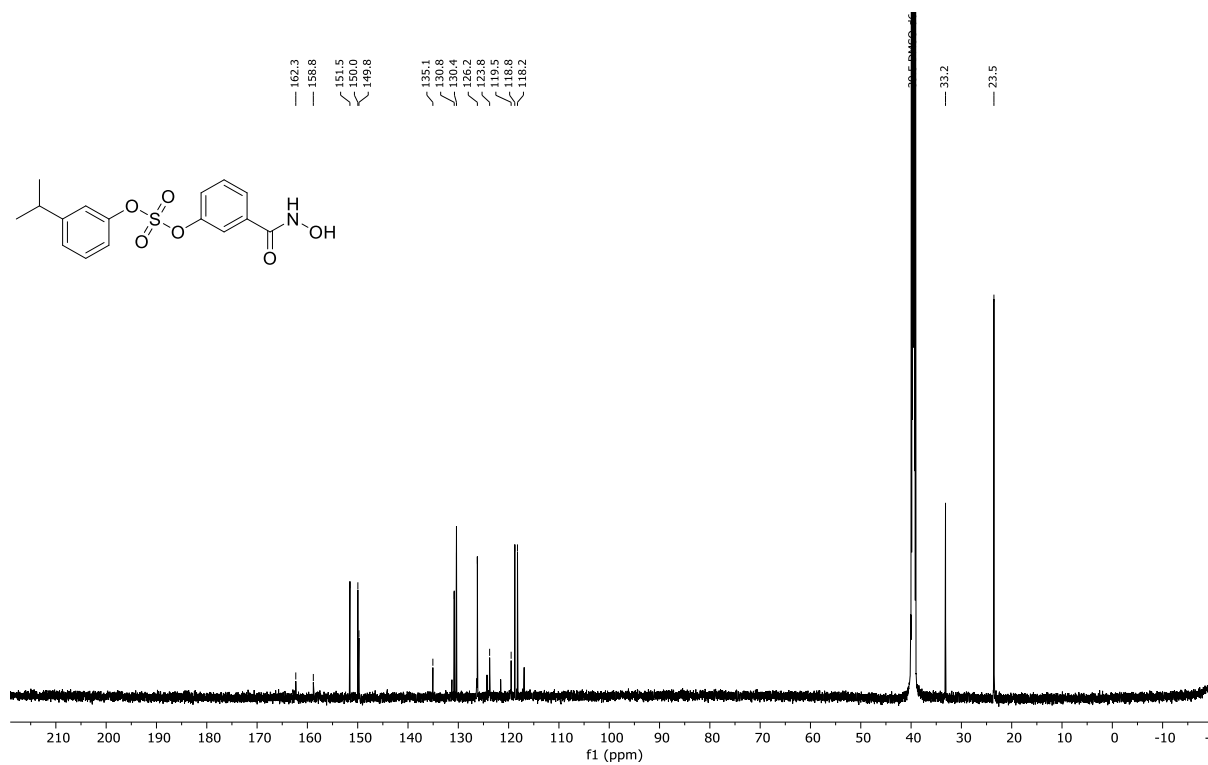
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A7**



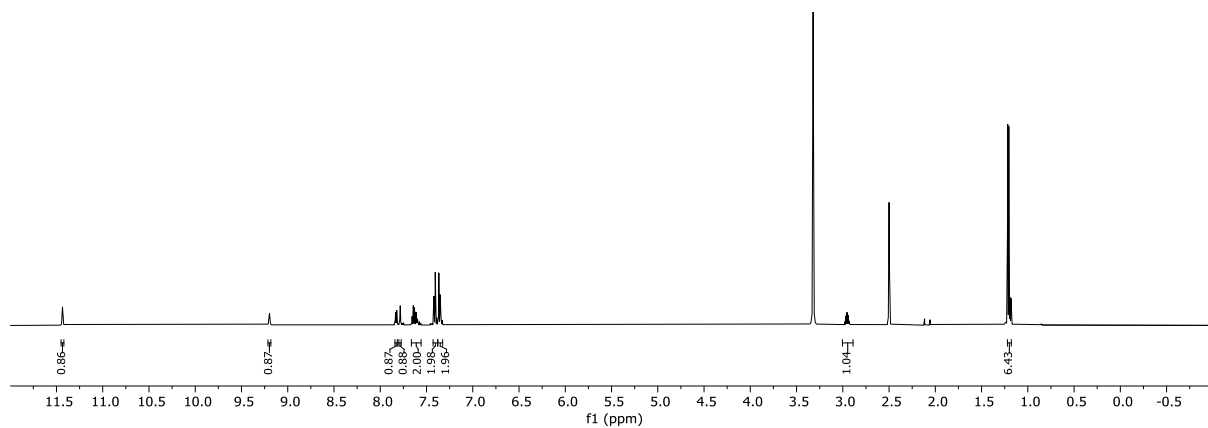
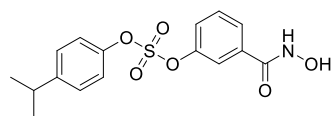
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A8**



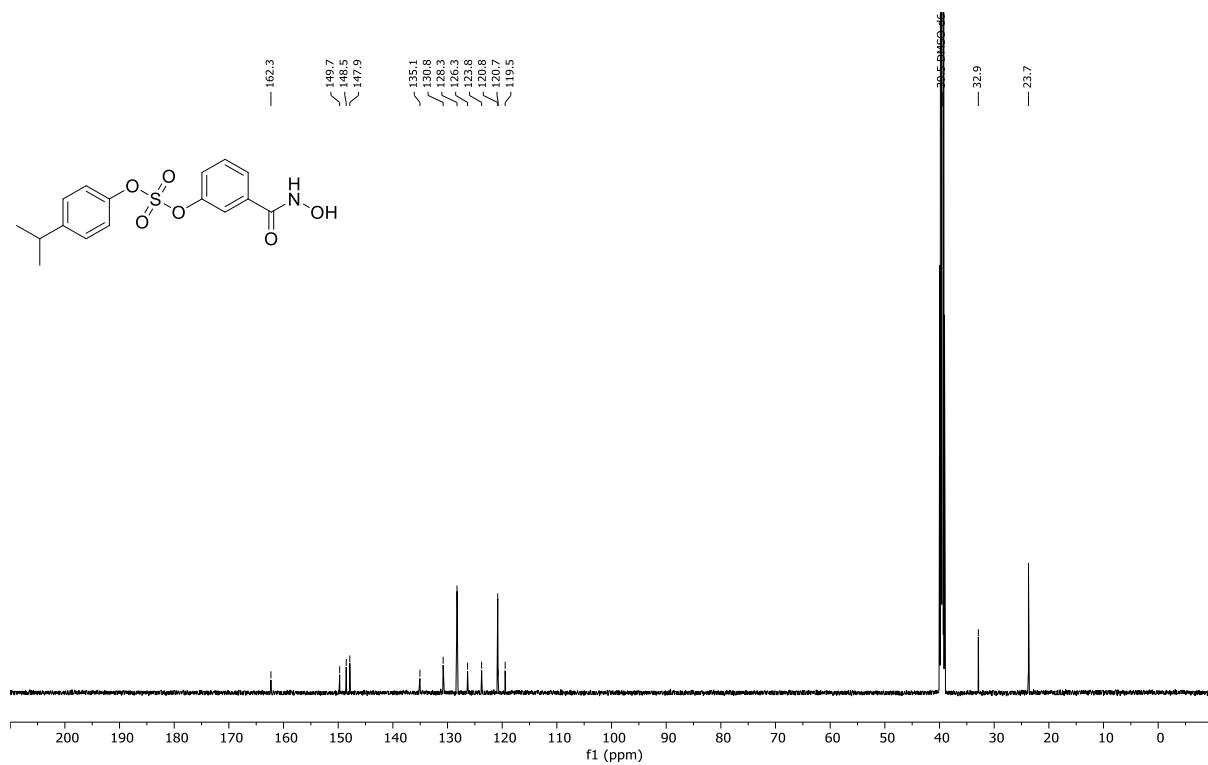
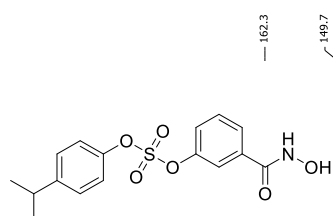
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A8**



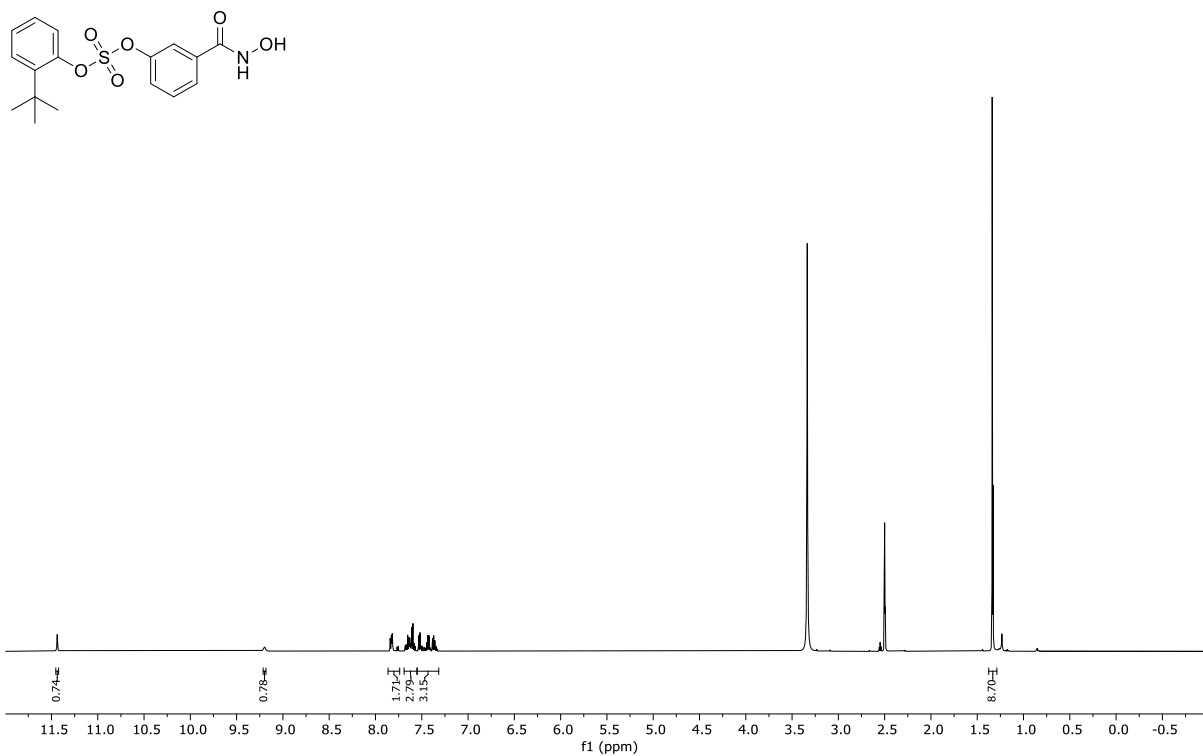
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A9**



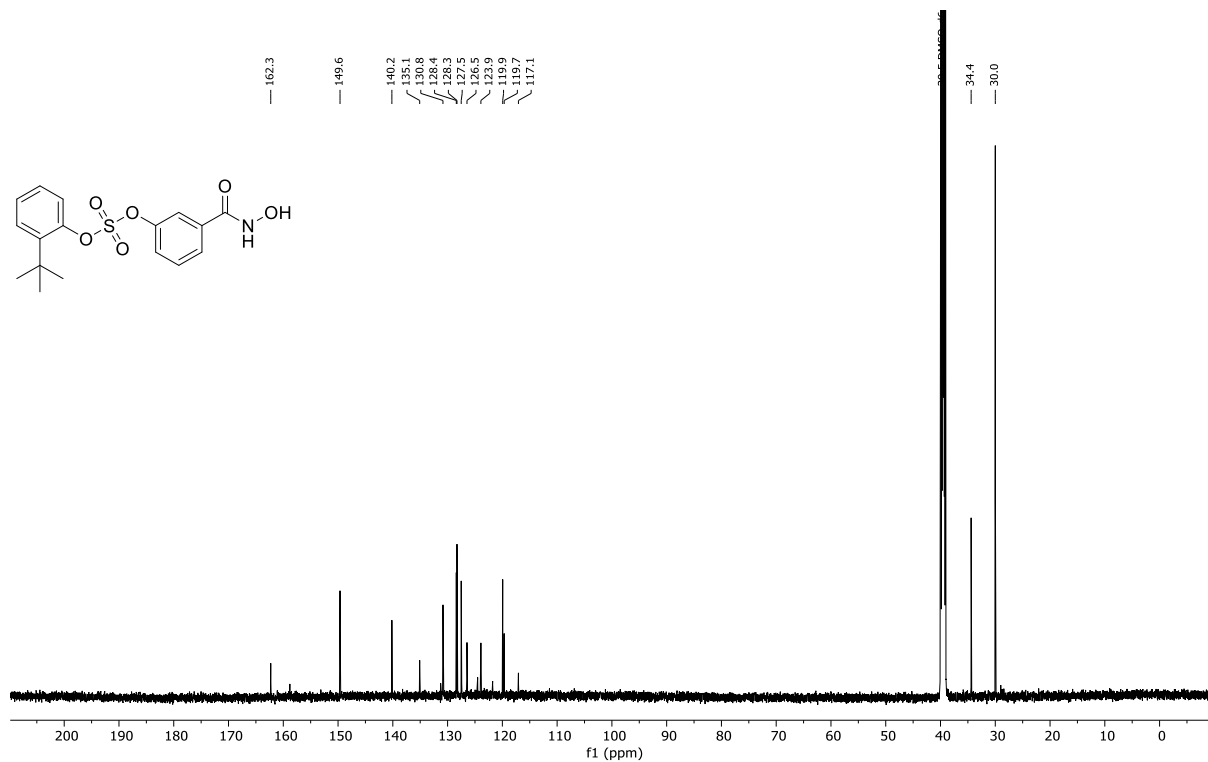
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A9**



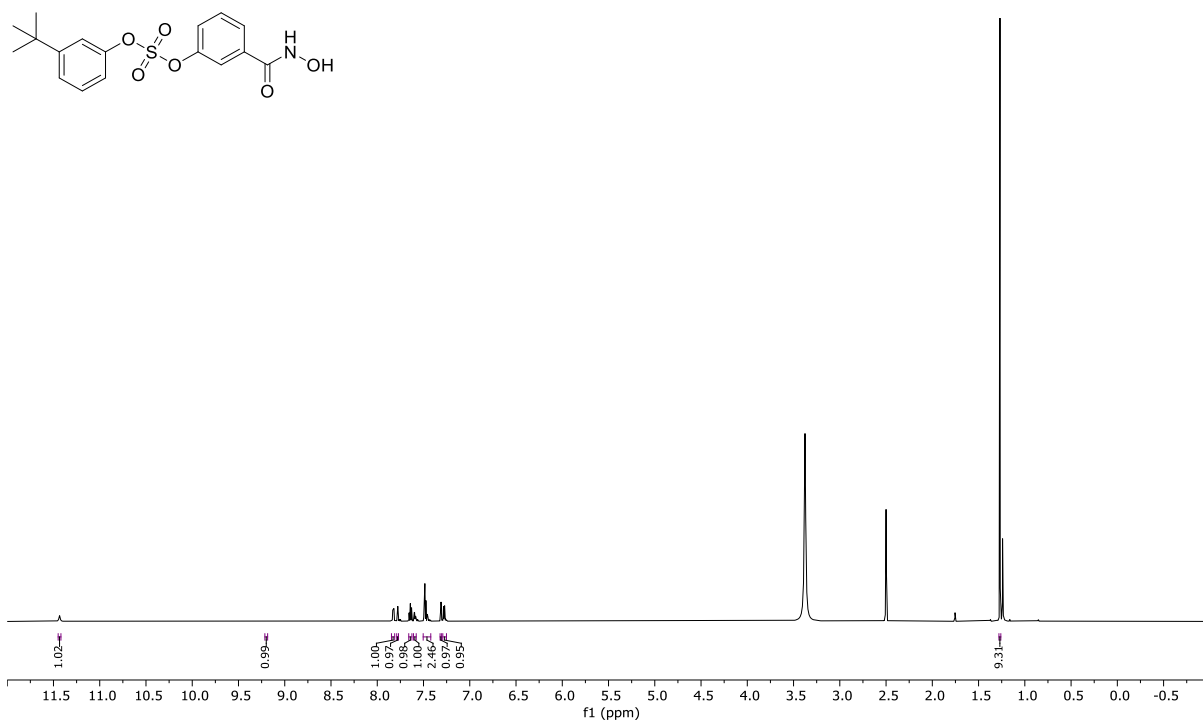
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **A10**



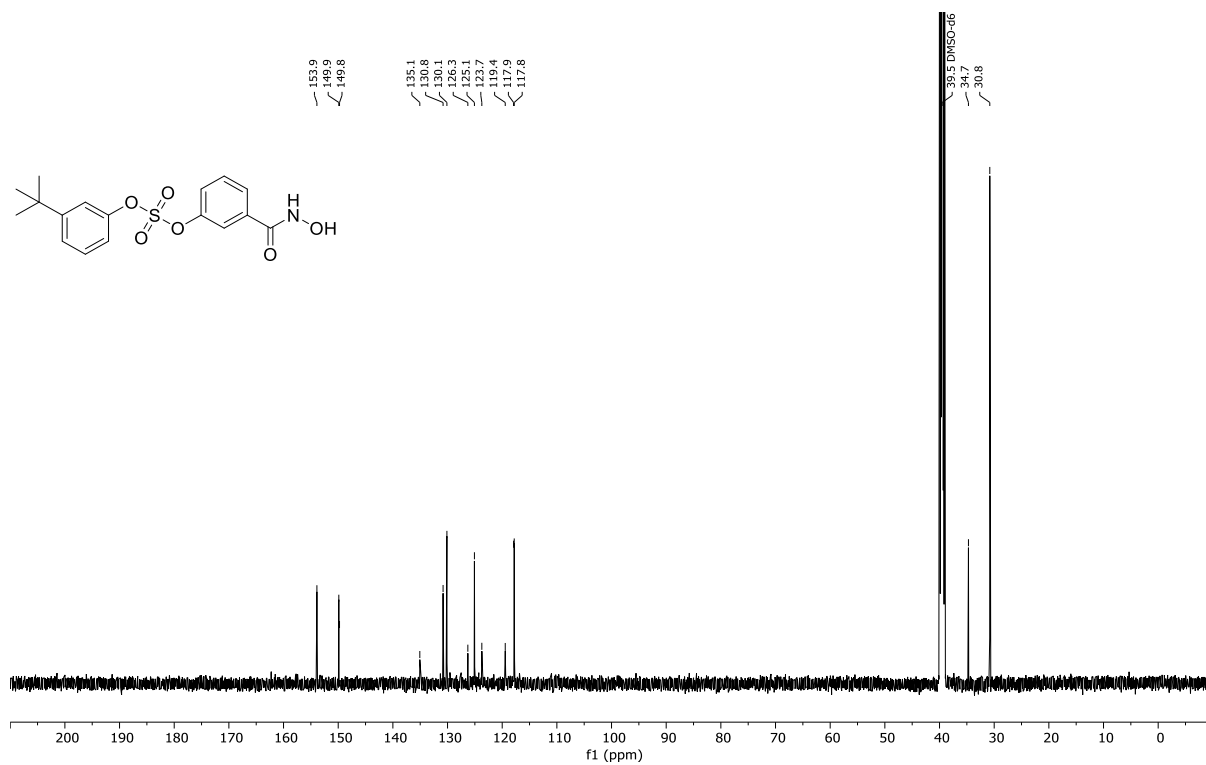
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **A10** (minor contamination with TFA is observed at 158.8 ppm)



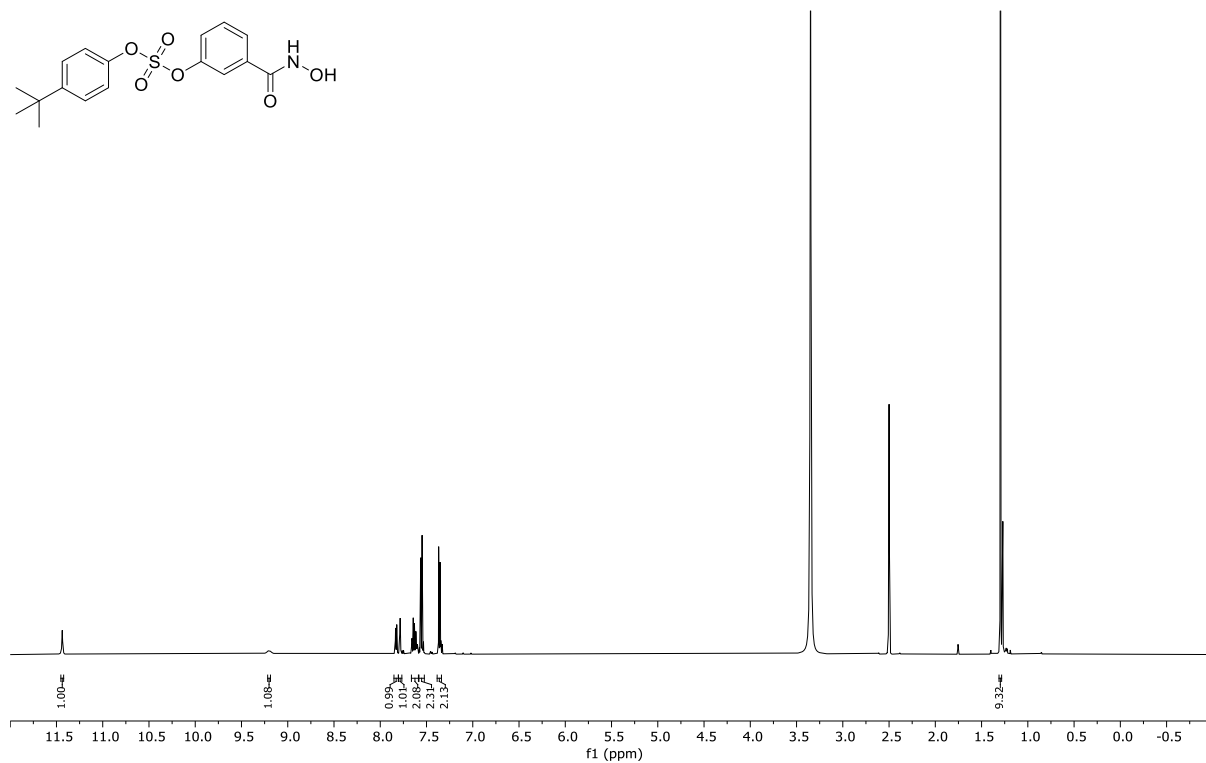
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A11**



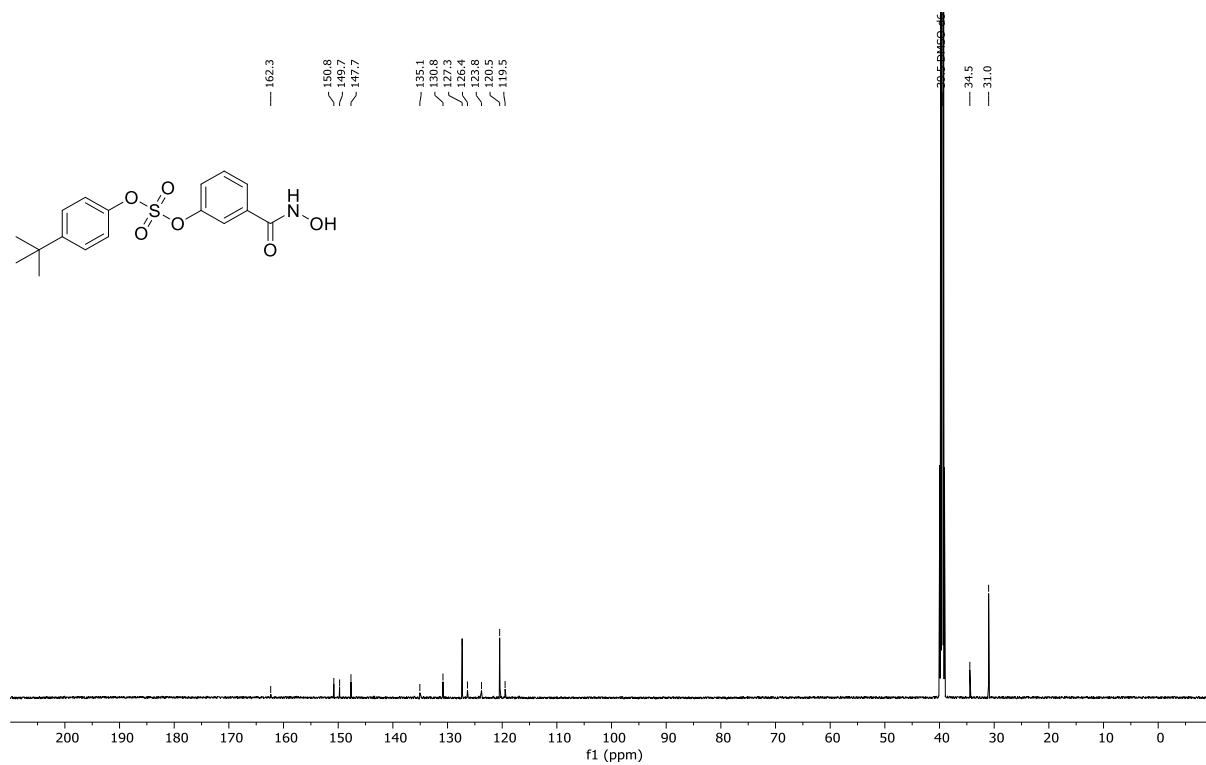
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A11**



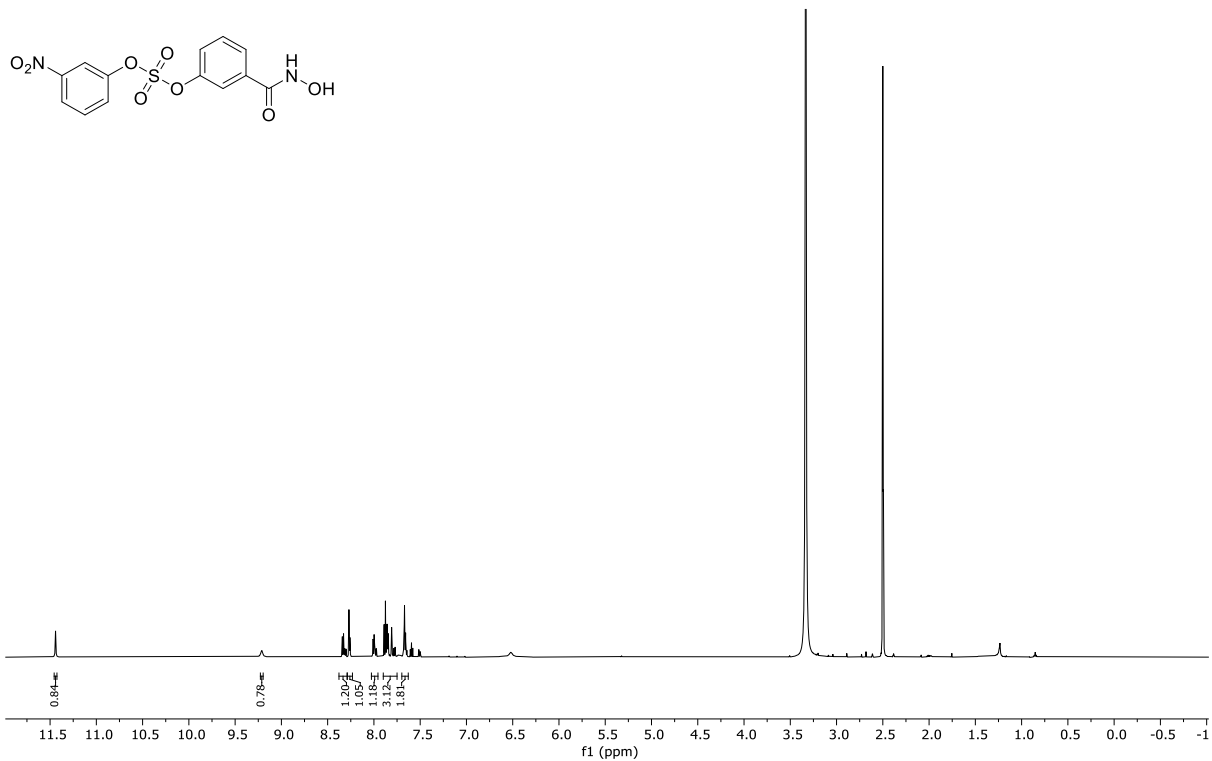
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A12**



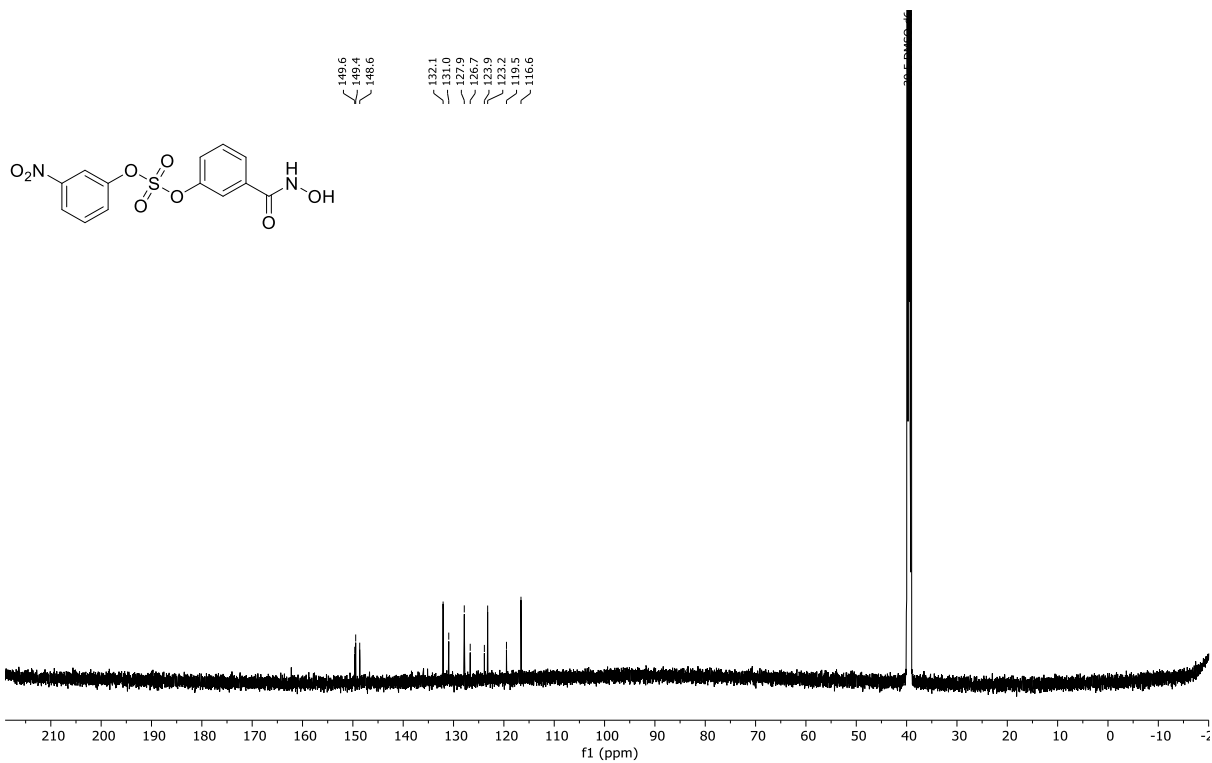
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A12**



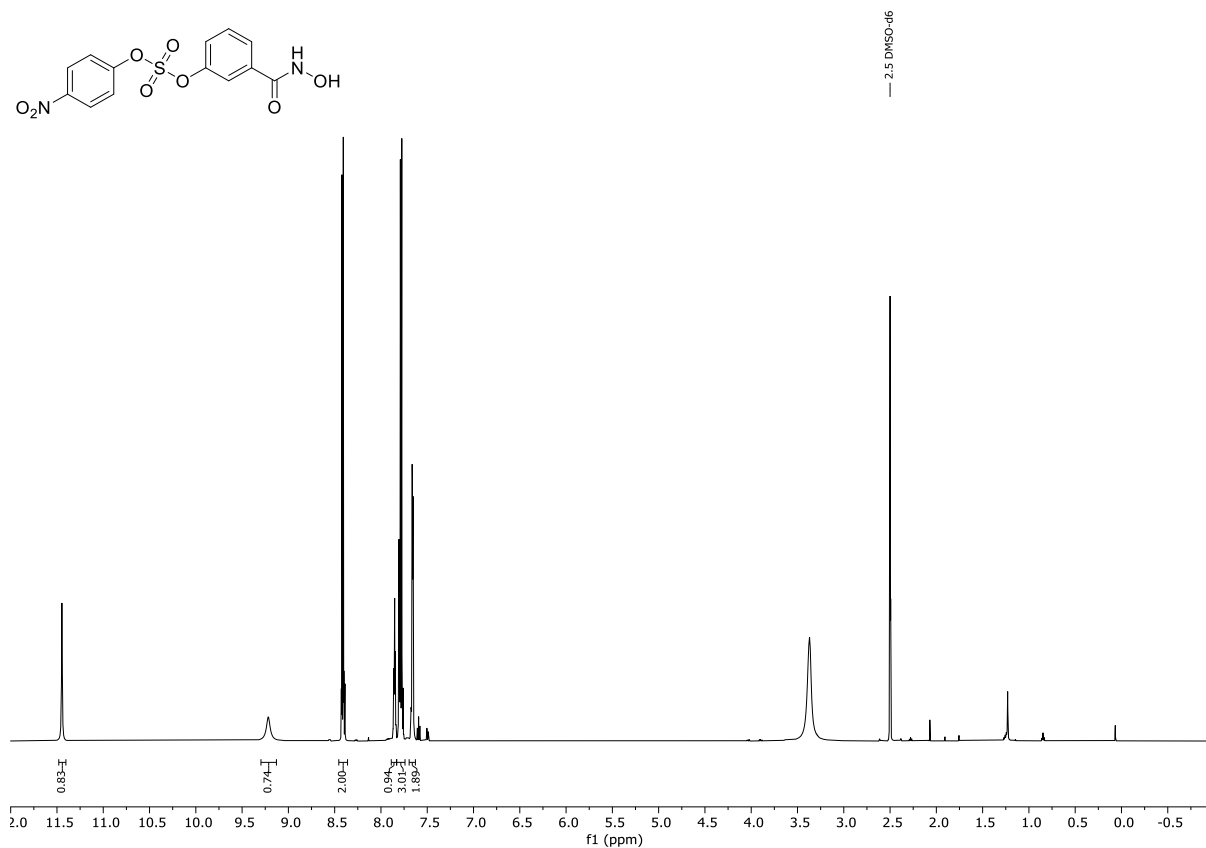
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A13**



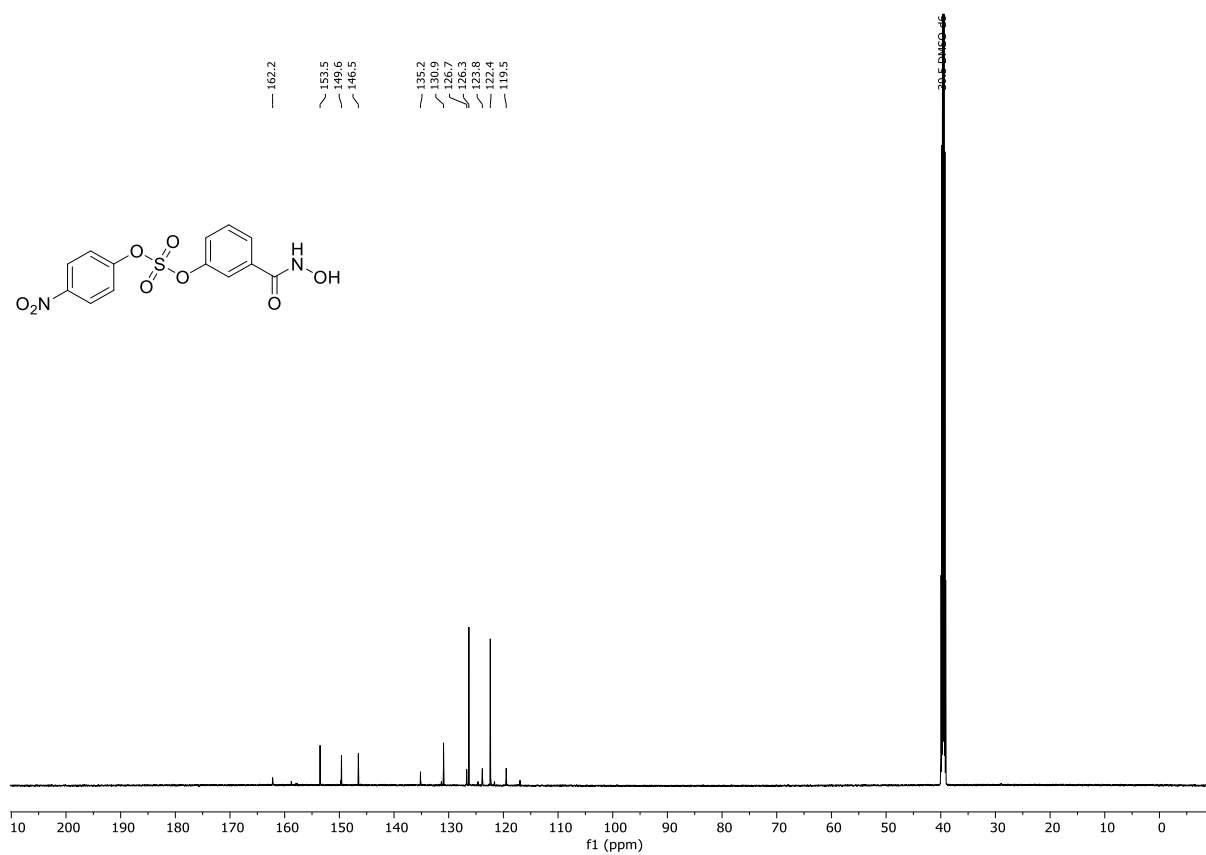
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A13**



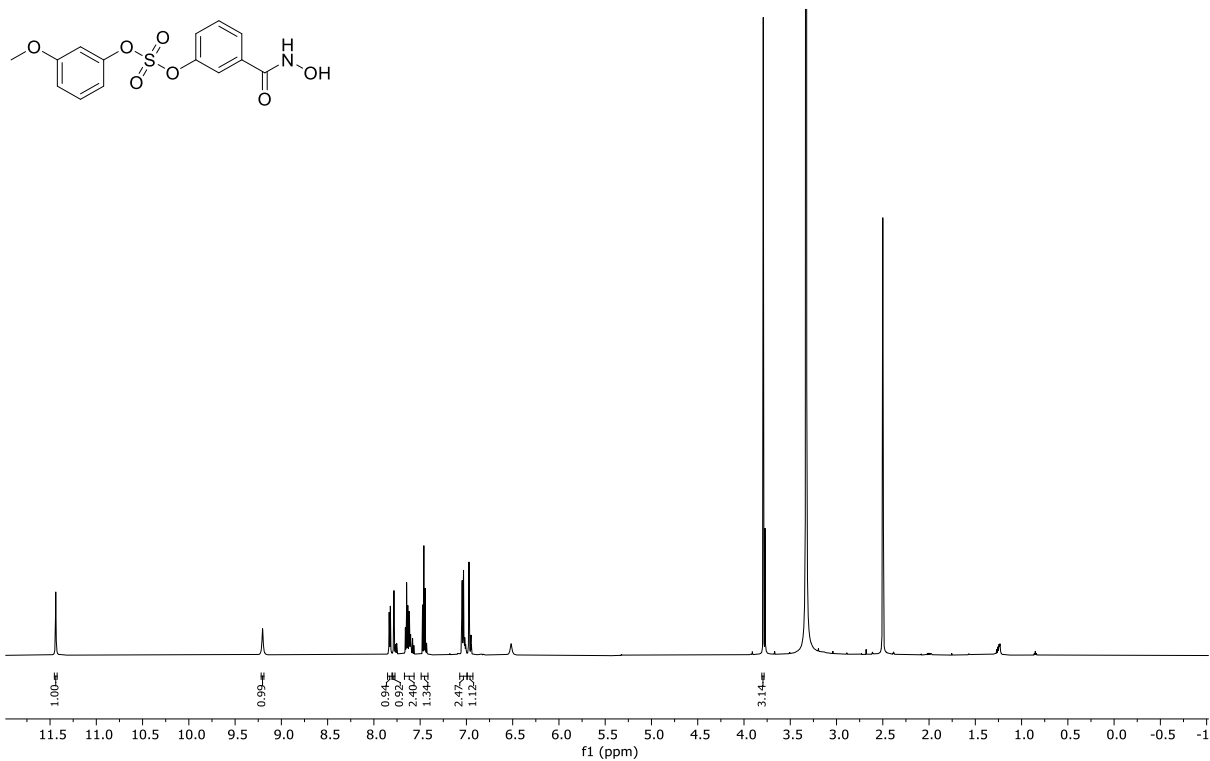
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A14**



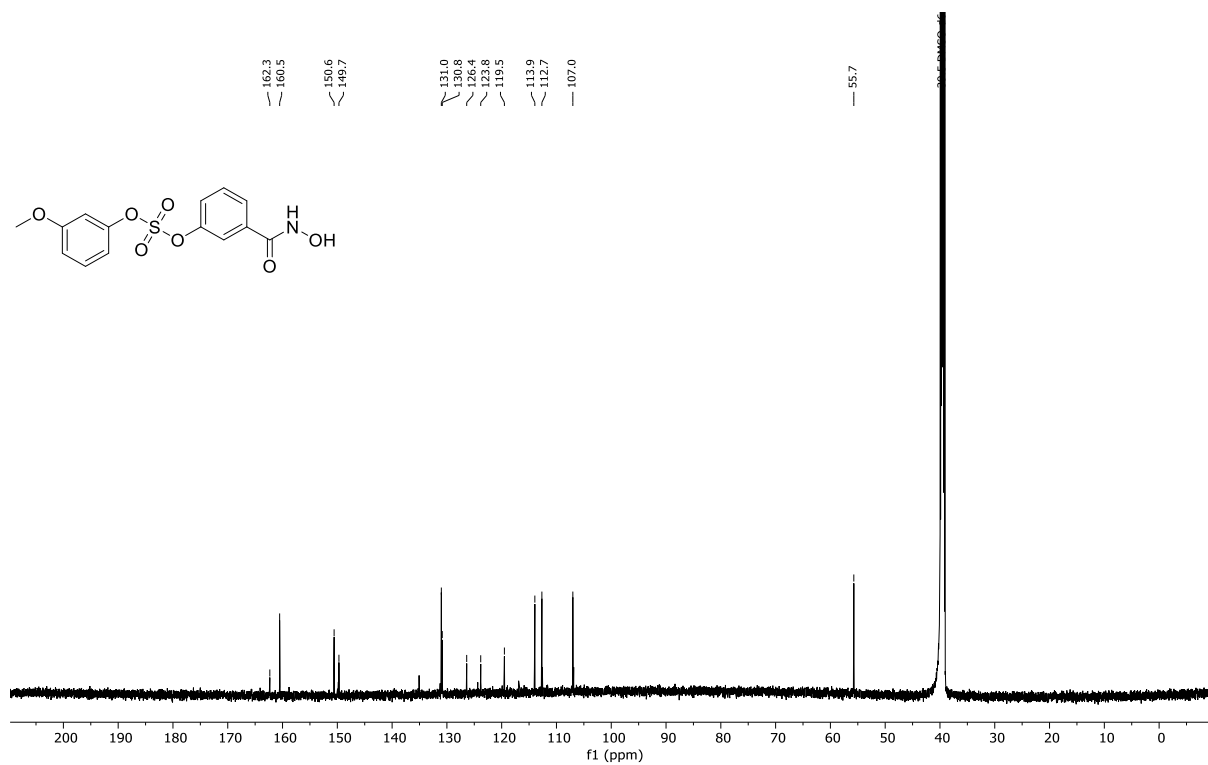
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A14** (minor contamination with TFA is observed at 158.8 ppm)



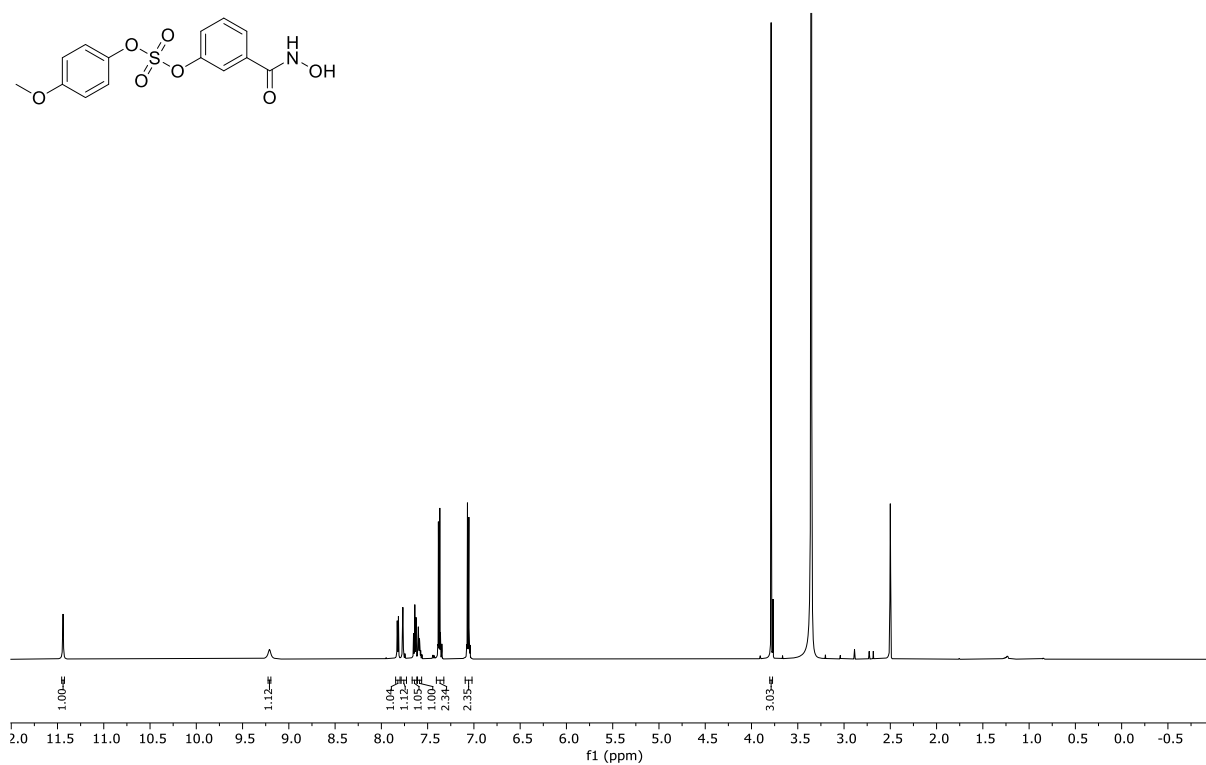
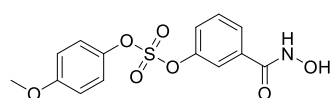
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A15**



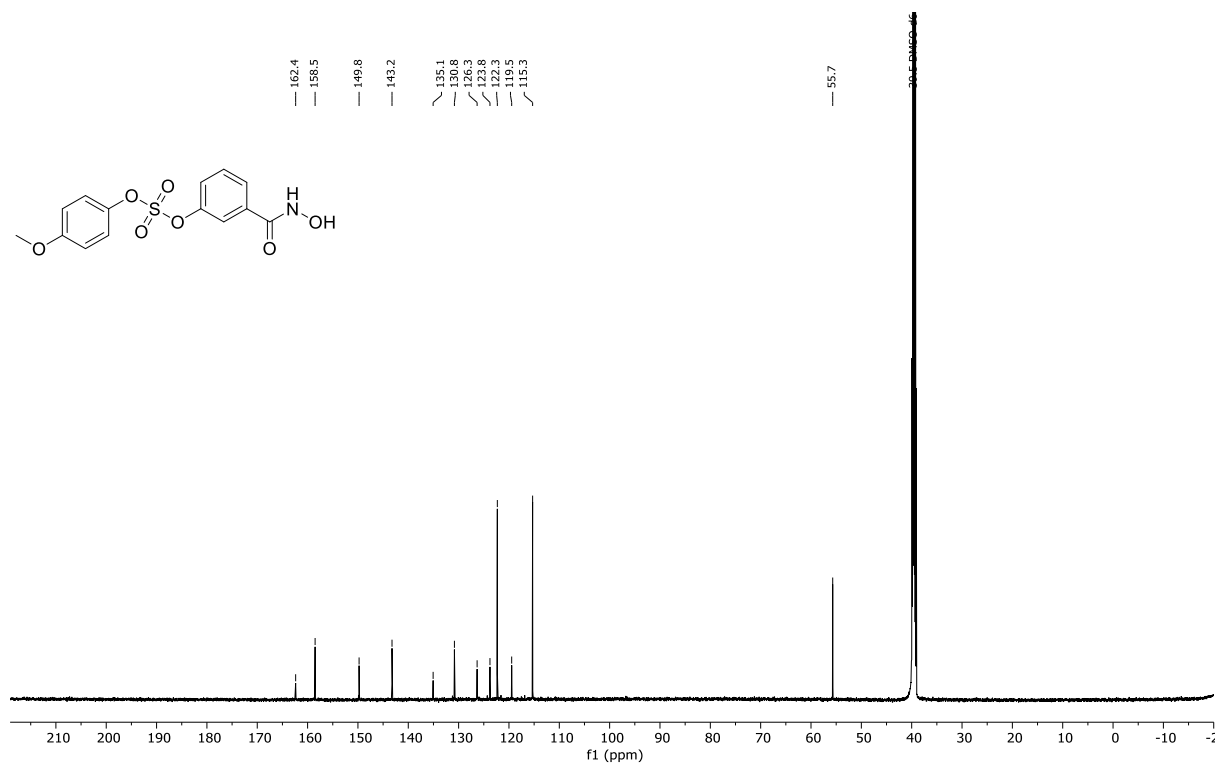
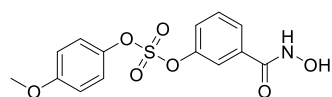
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A15**



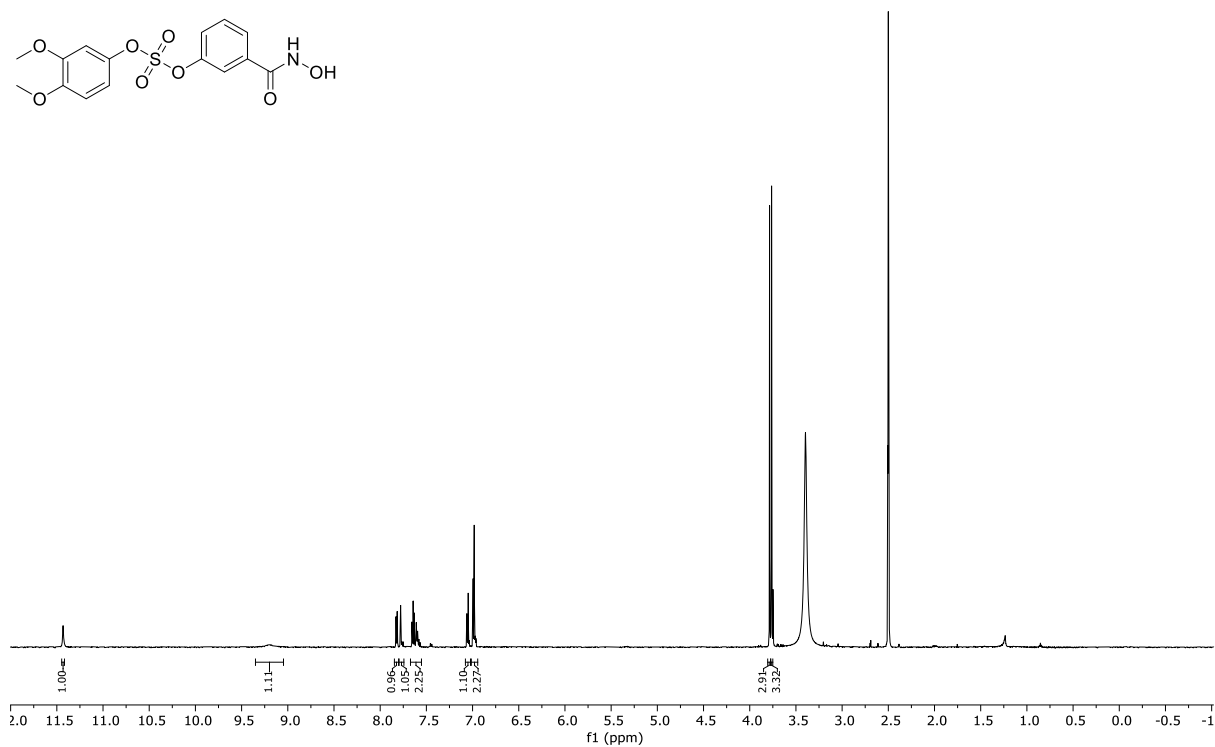
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A16**



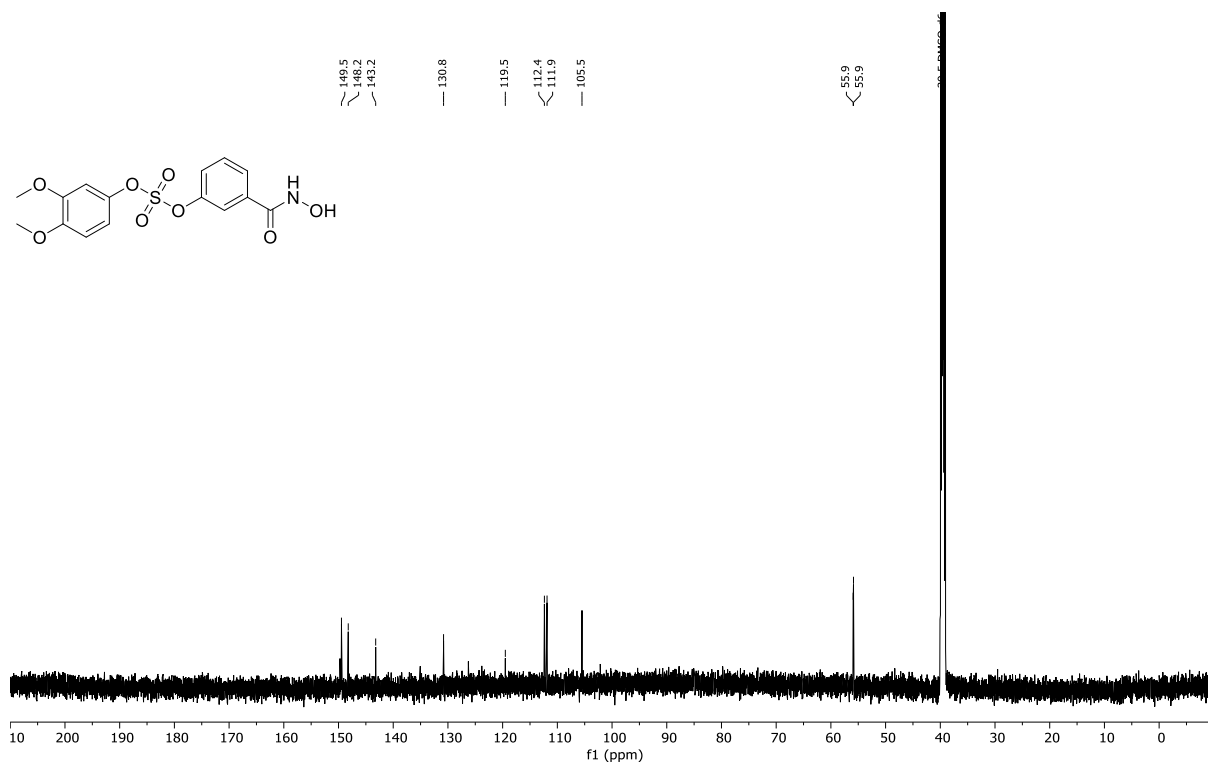
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A16**



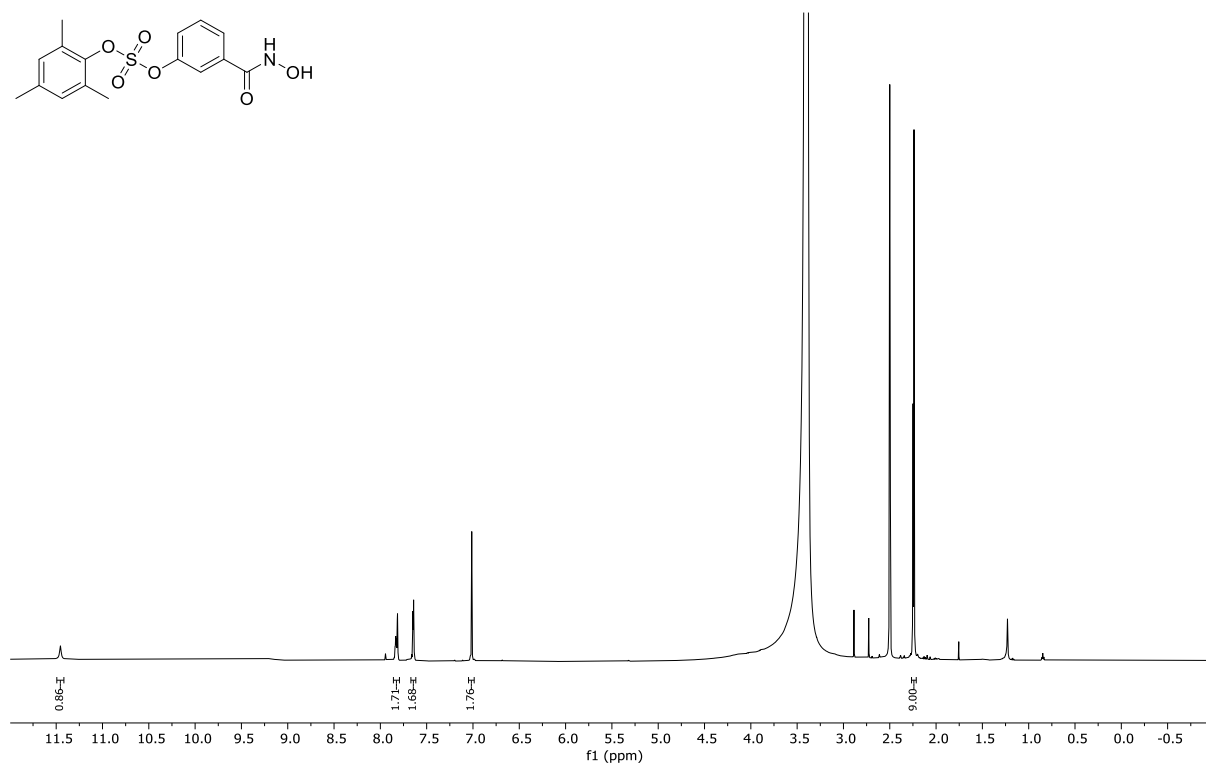
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A17**



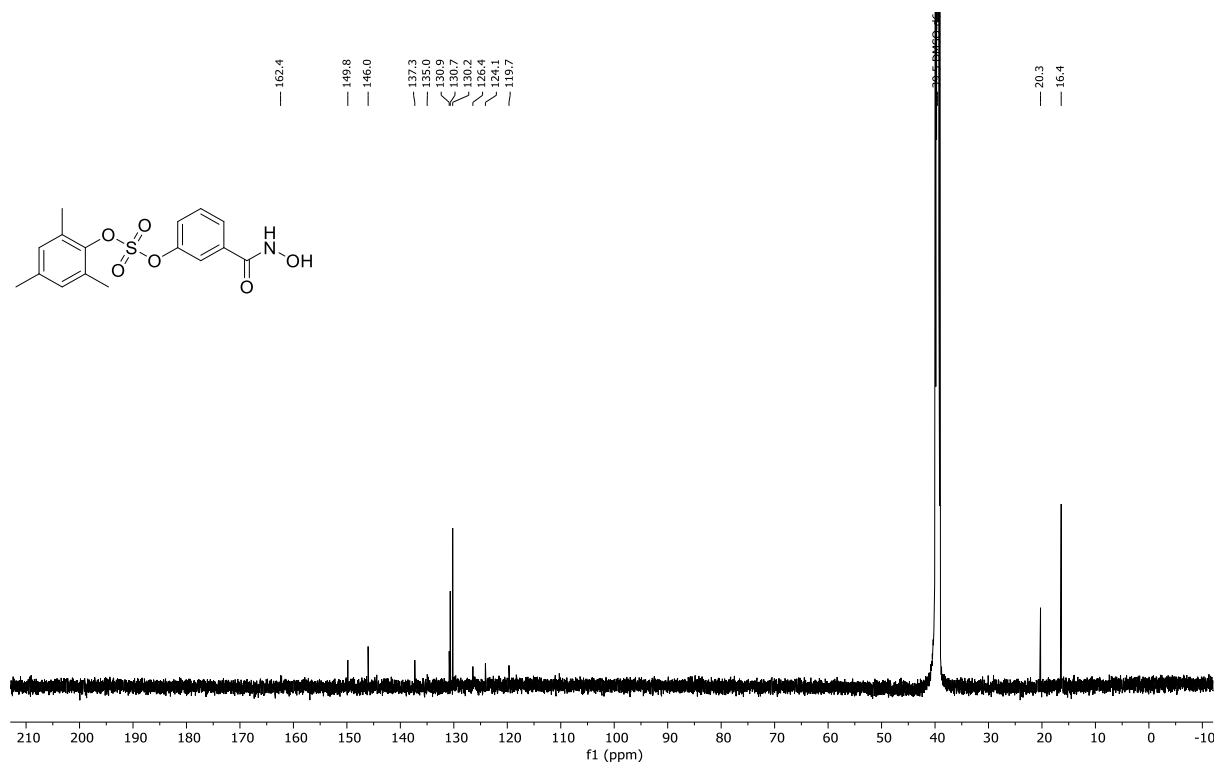
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A17**



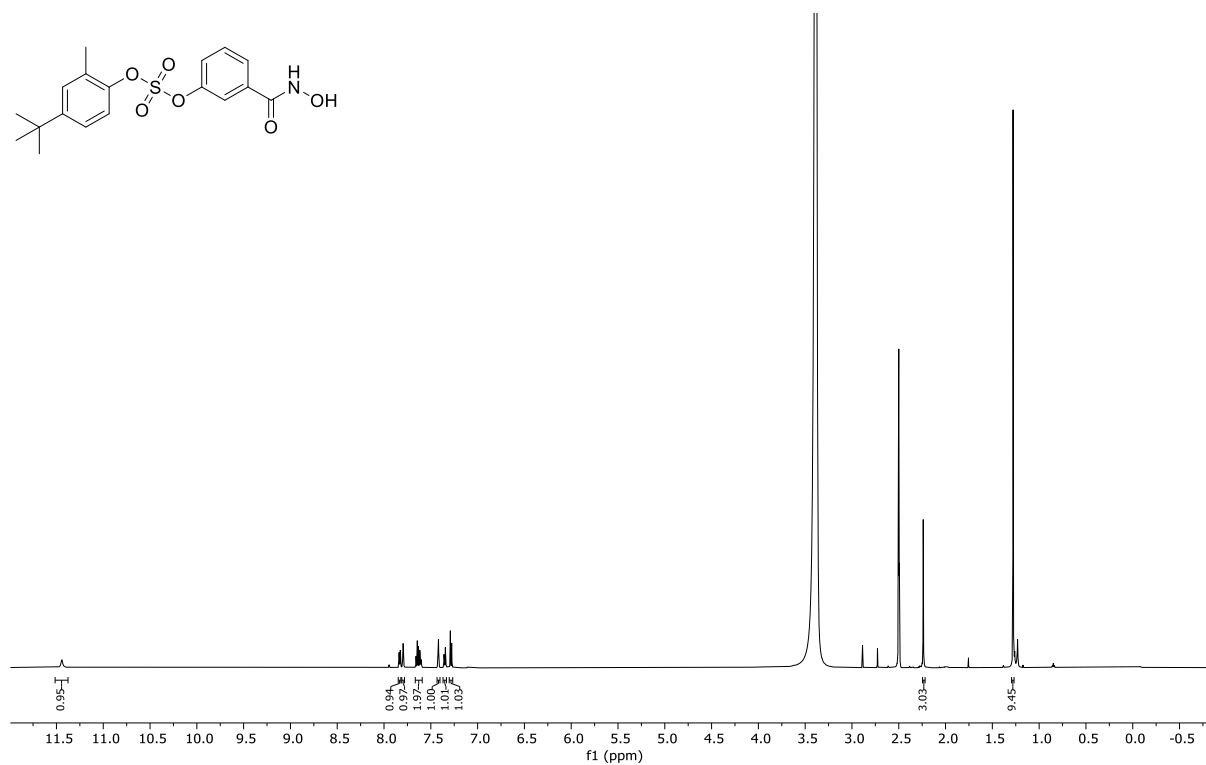
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A18**



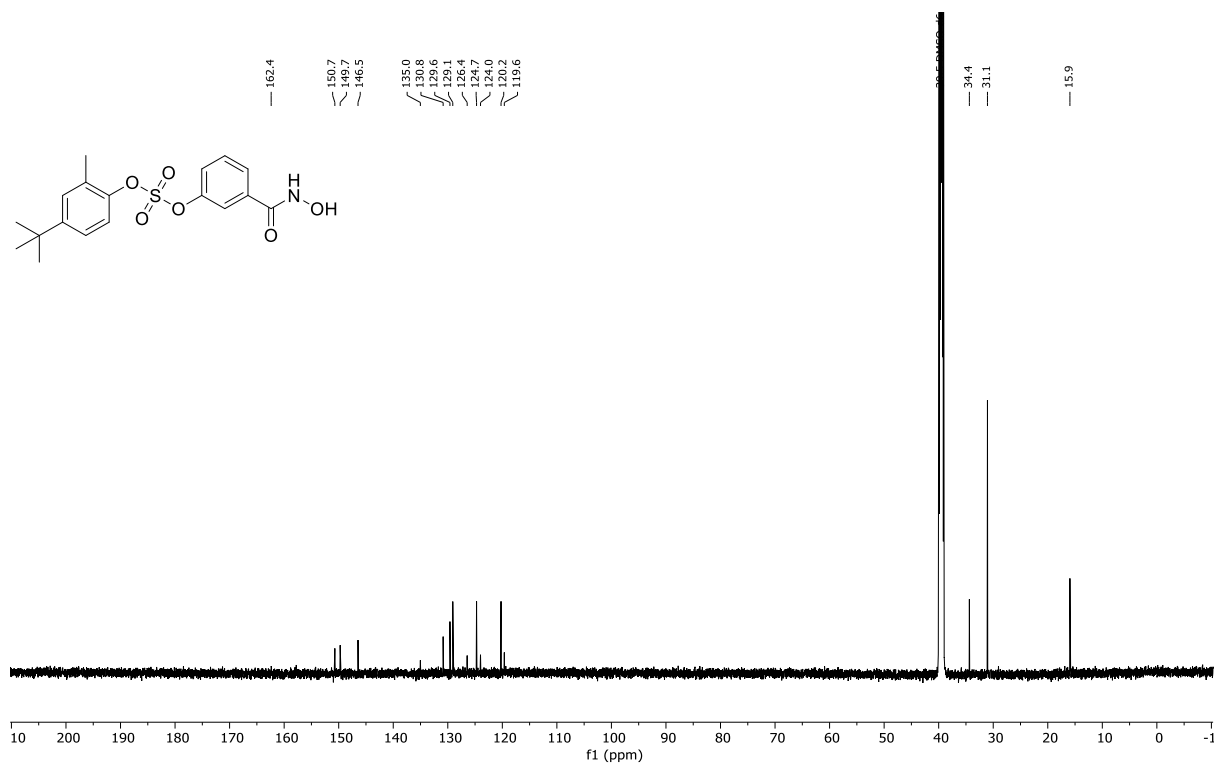
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A18**



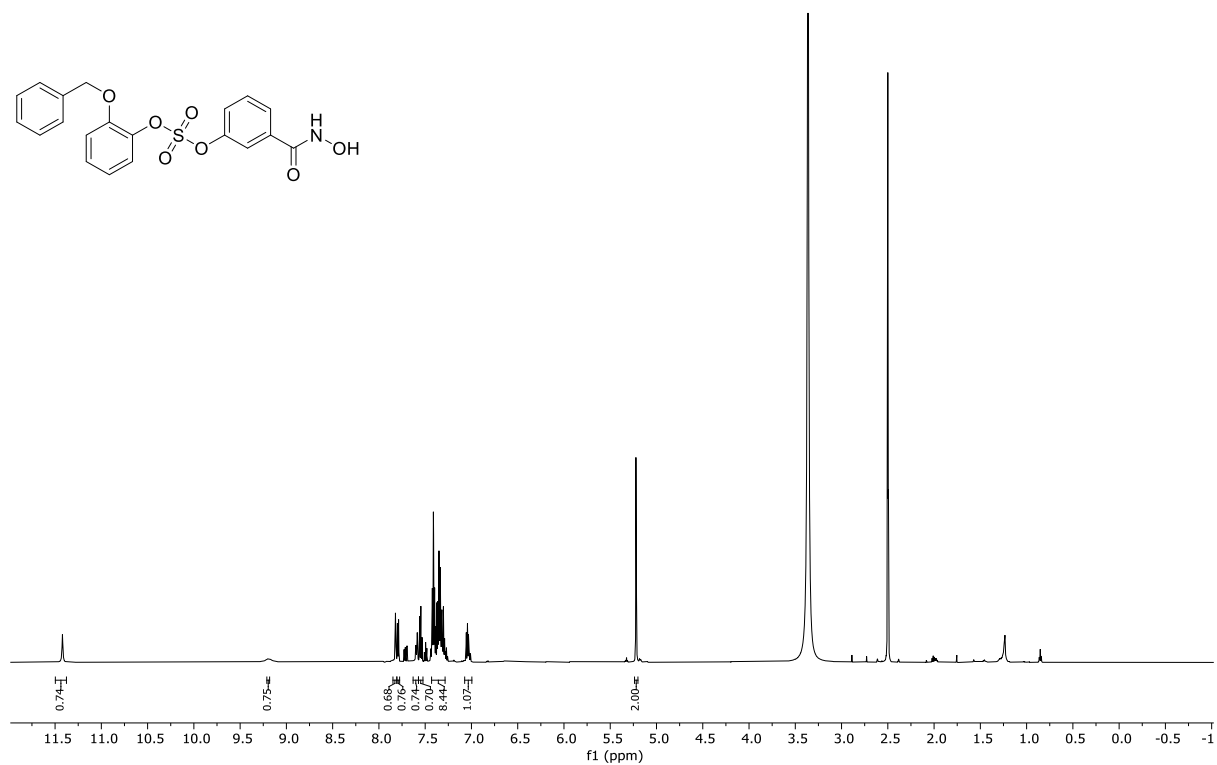
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A19**



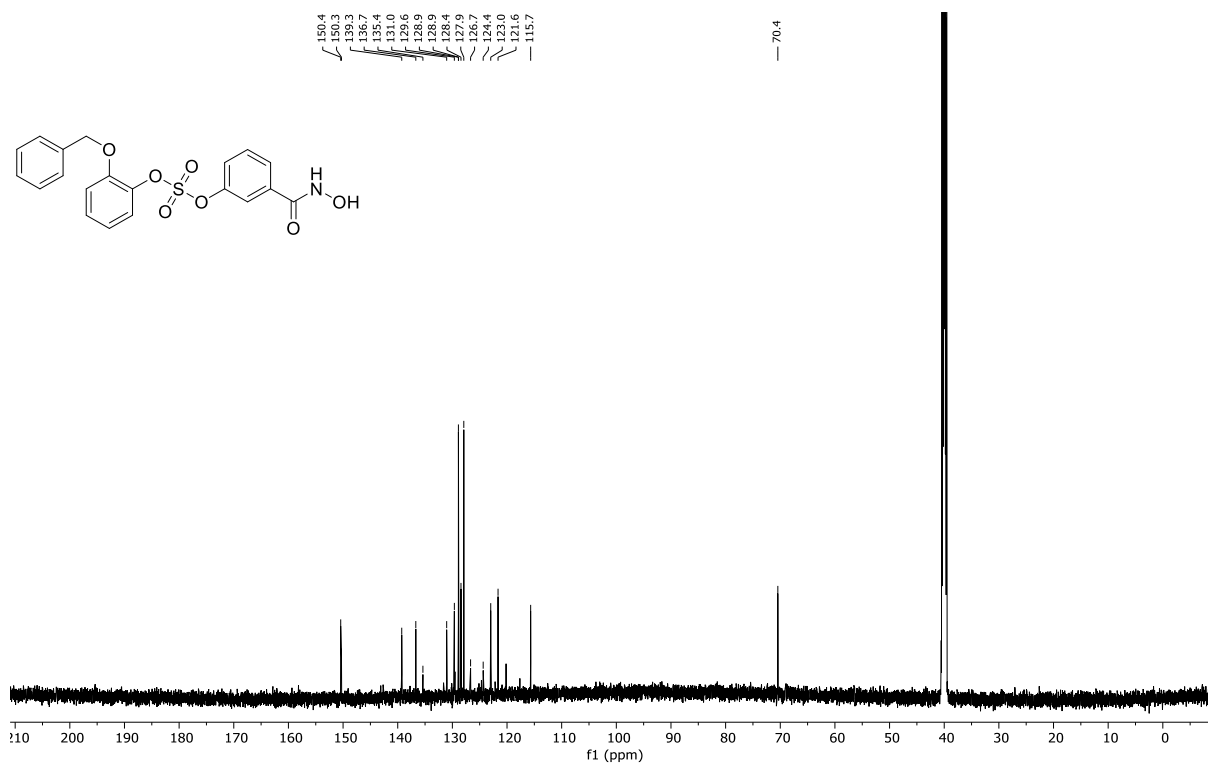
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A19**



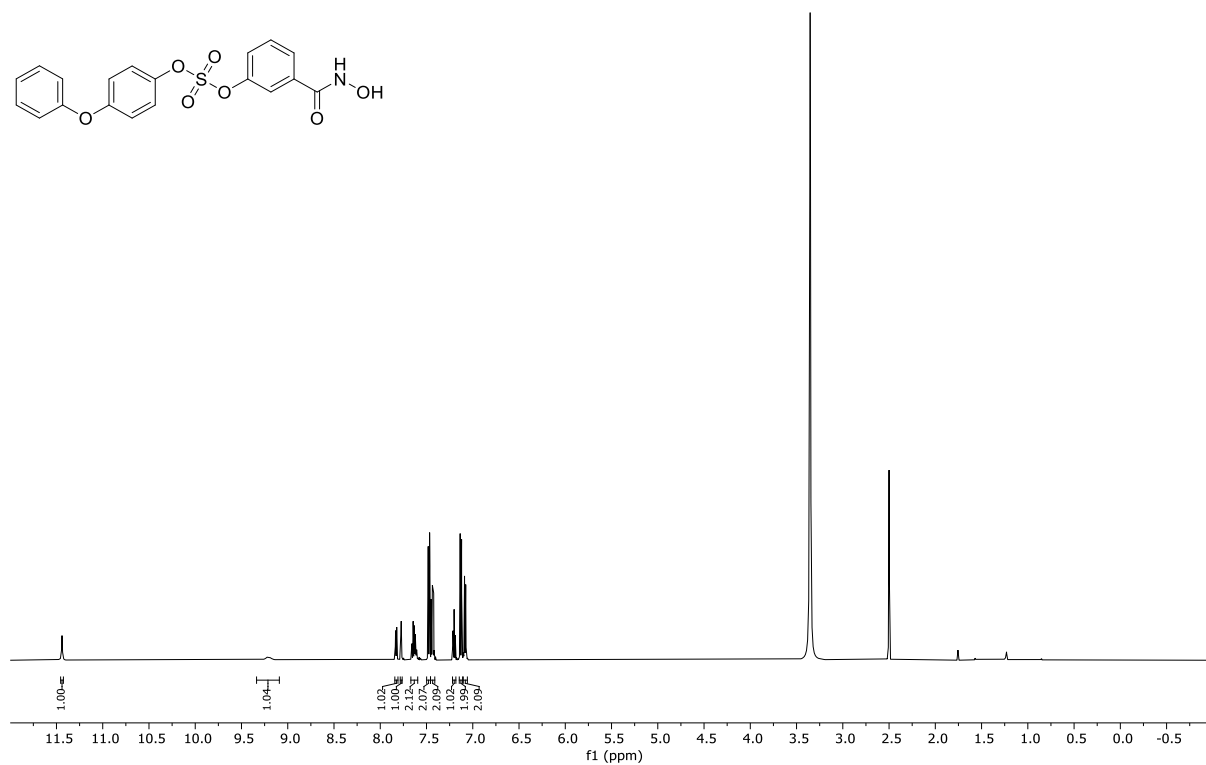
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A20**



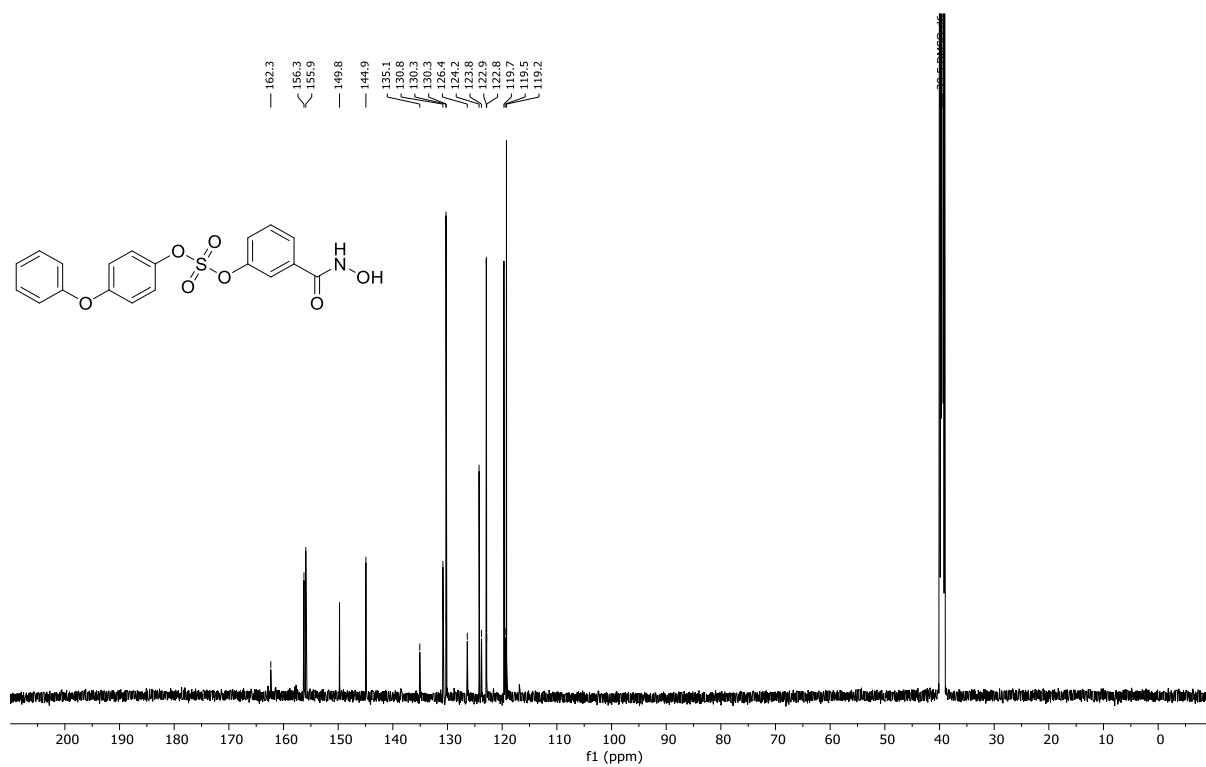
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A20**



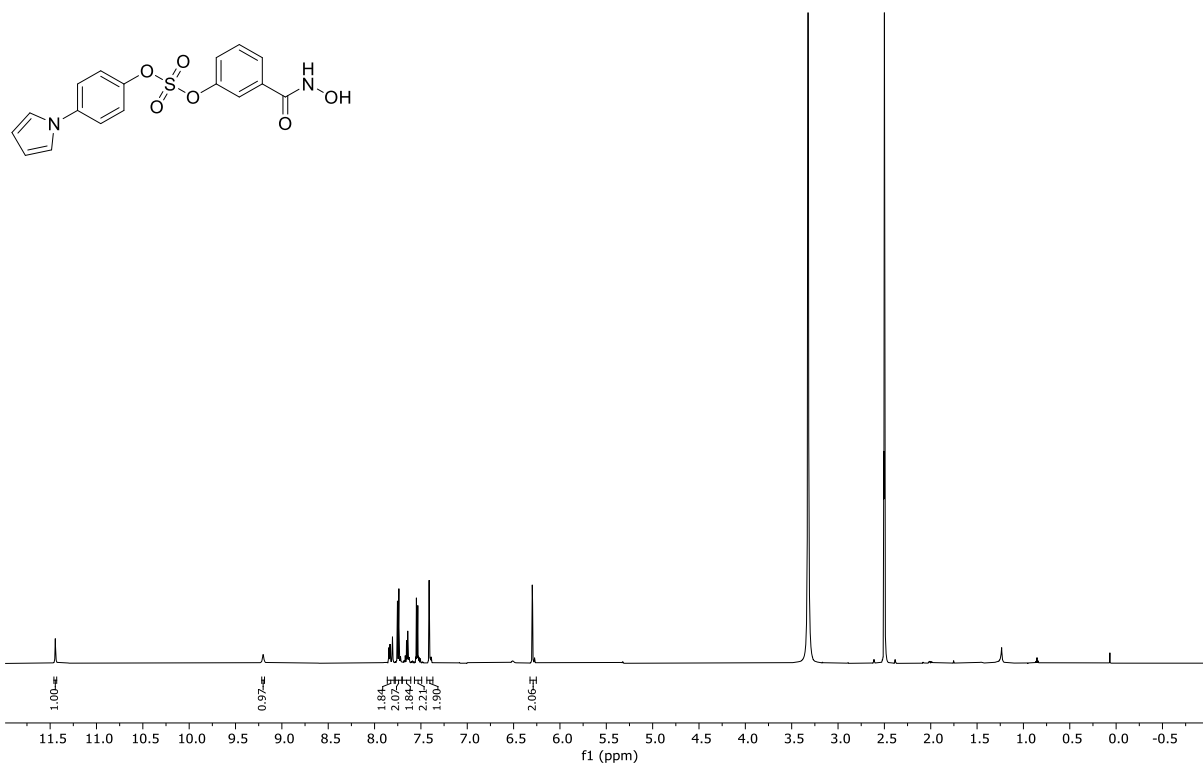
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A21**



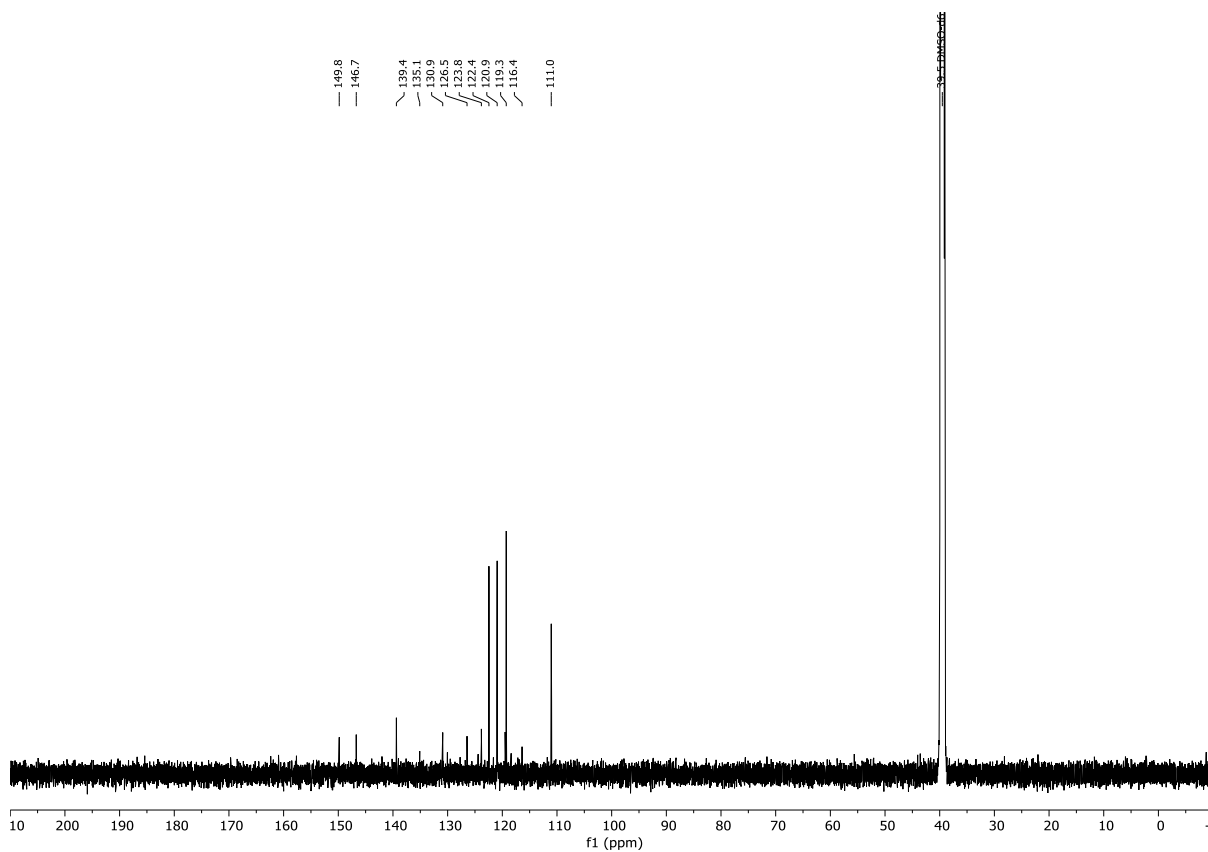
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A21**



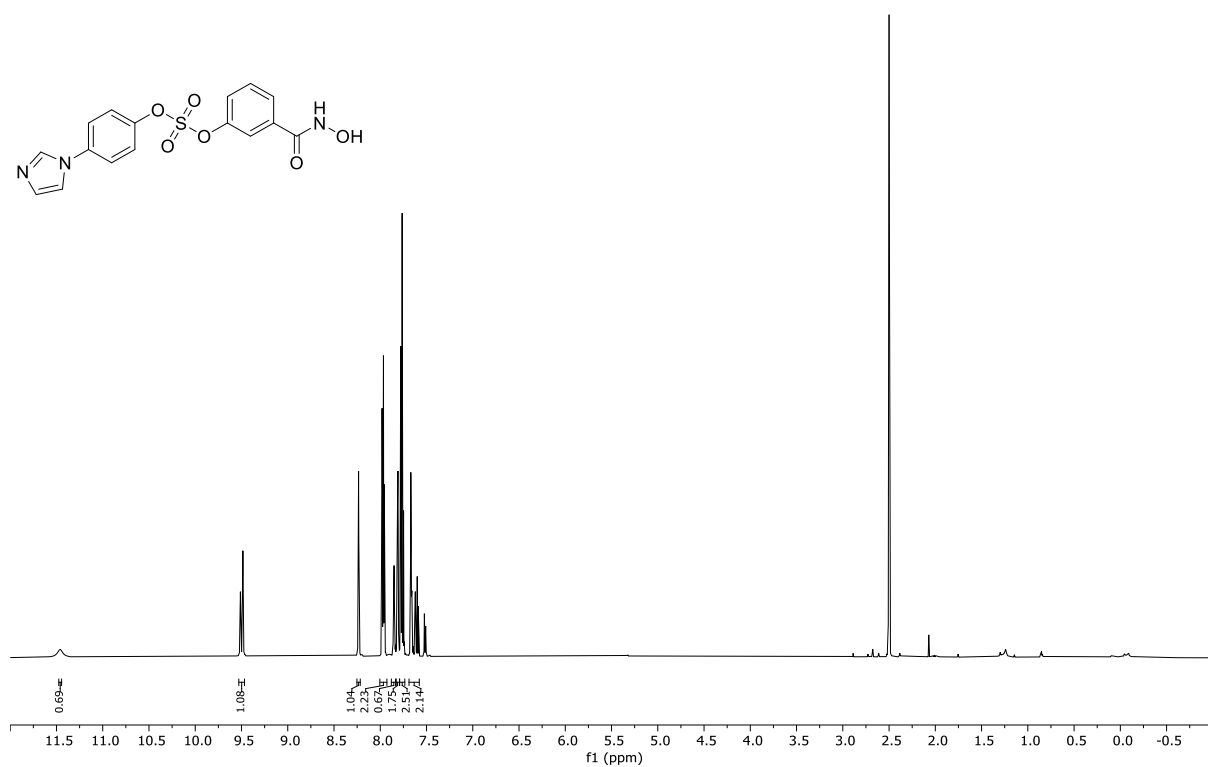
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A22**



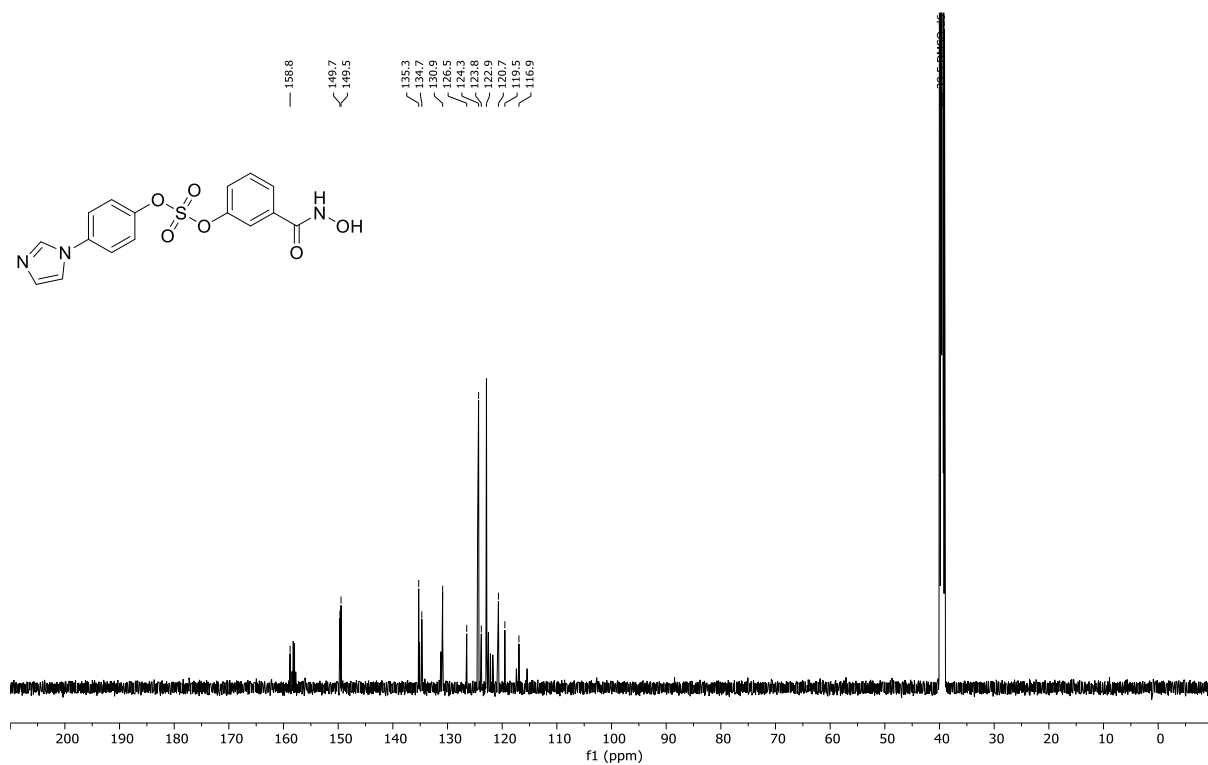
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A22**



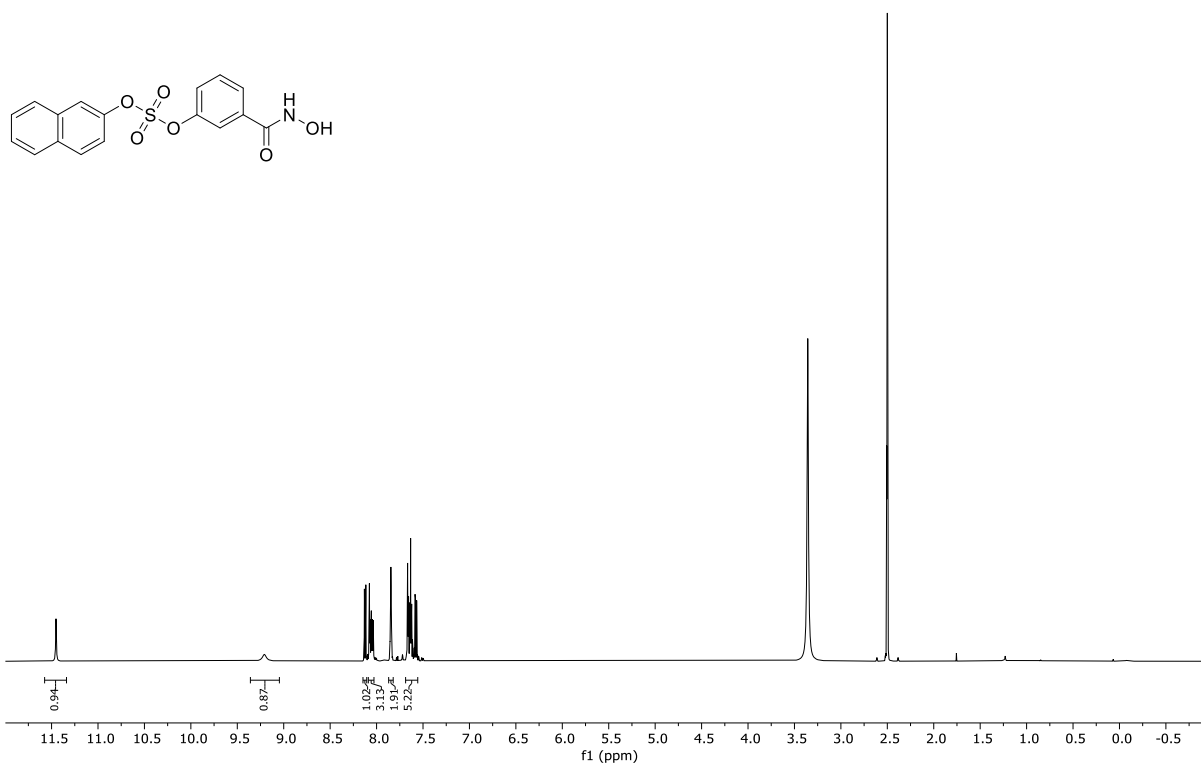
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **A23**



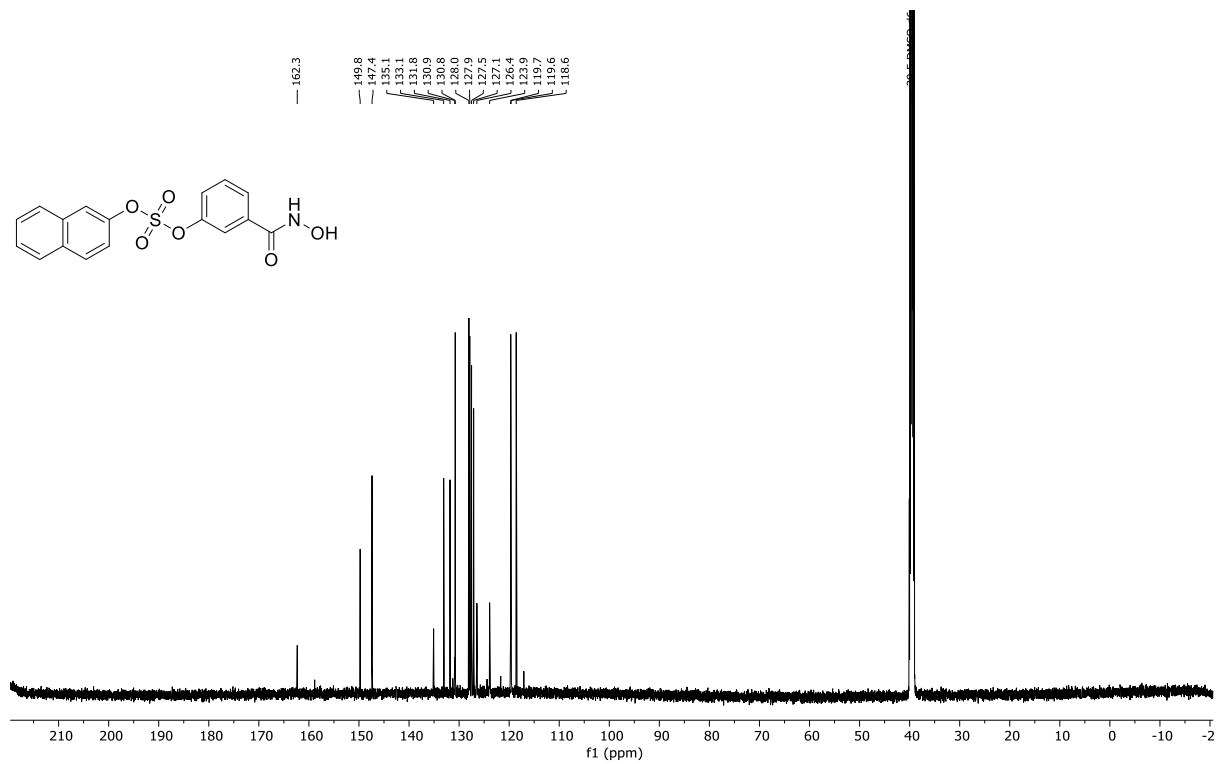
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **A23** (minor contamination with TFA is observed at 158.8 ppm)



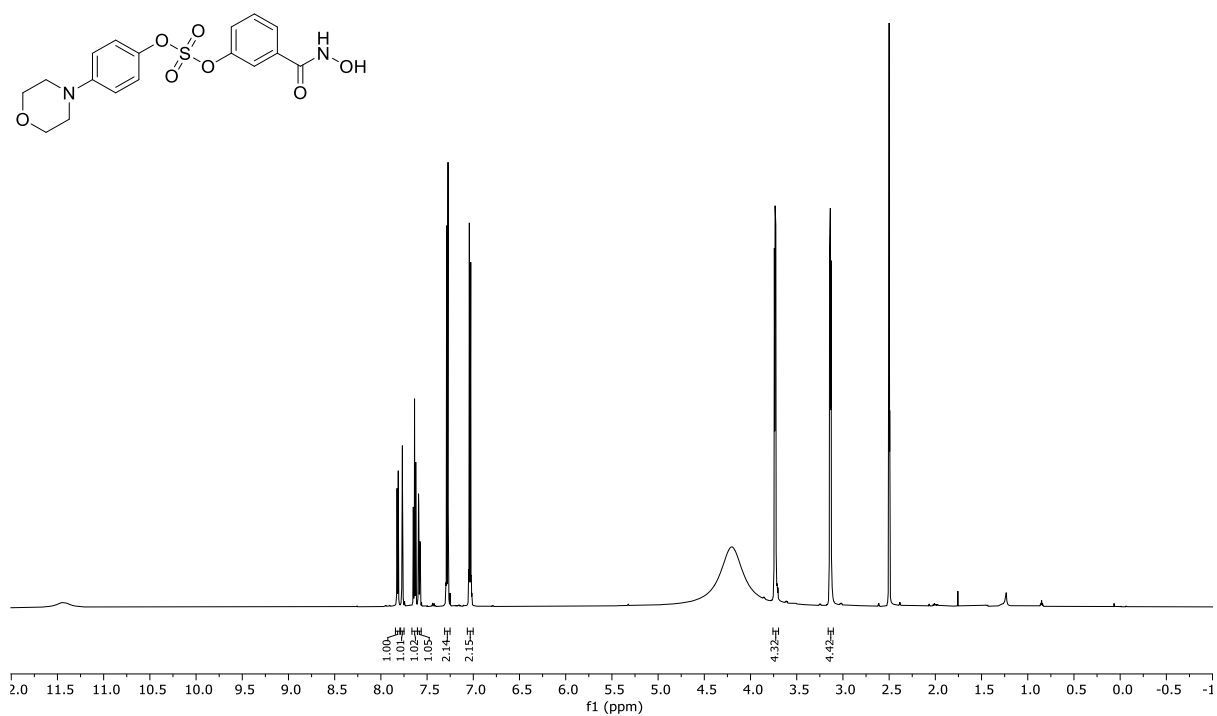
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A24**



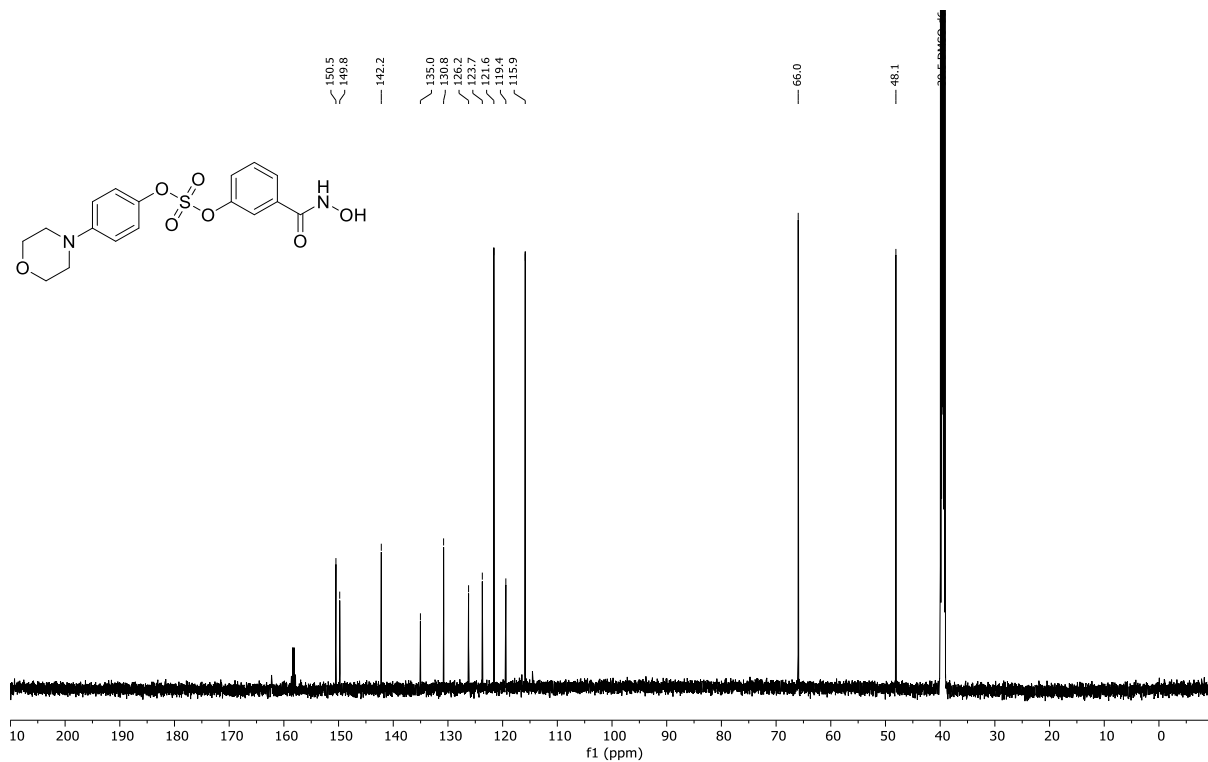
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A24** (minor contamination with TFA is observed at 158.8 ppm)



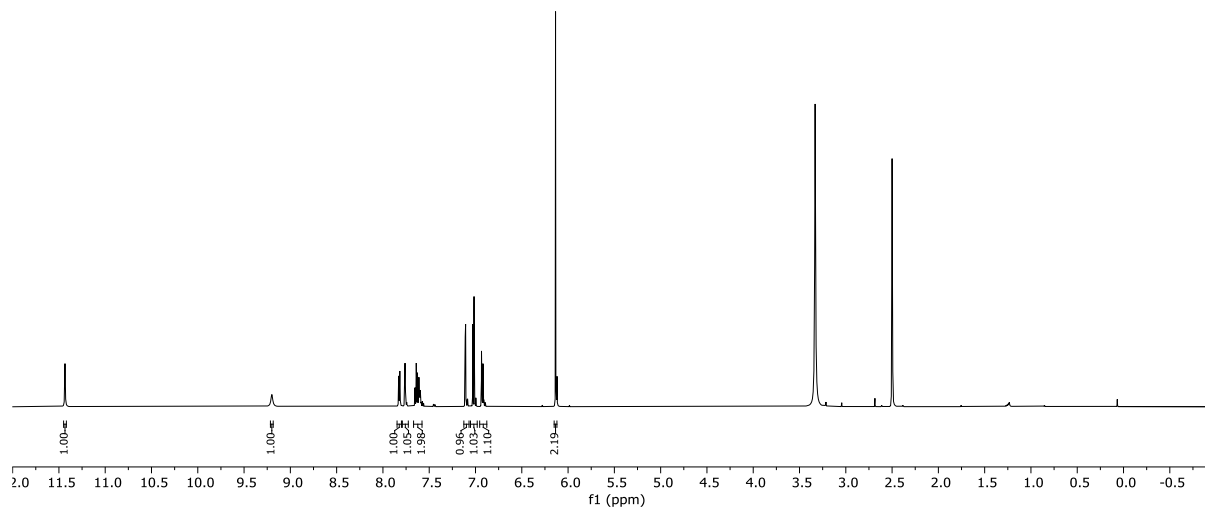
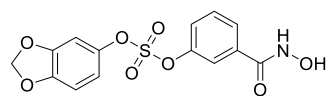
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **A25**



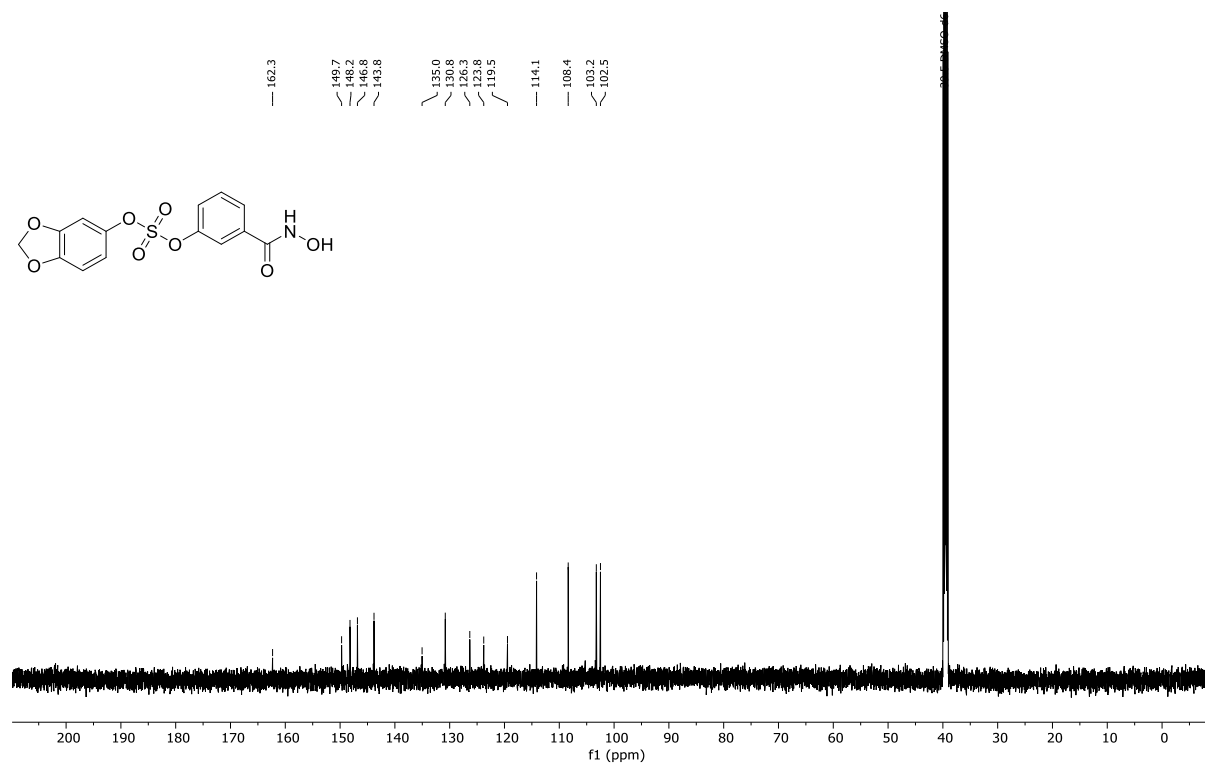
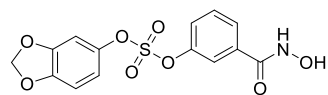
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **A25** (minor contamination with TFA is observed at 158.8 ppm)



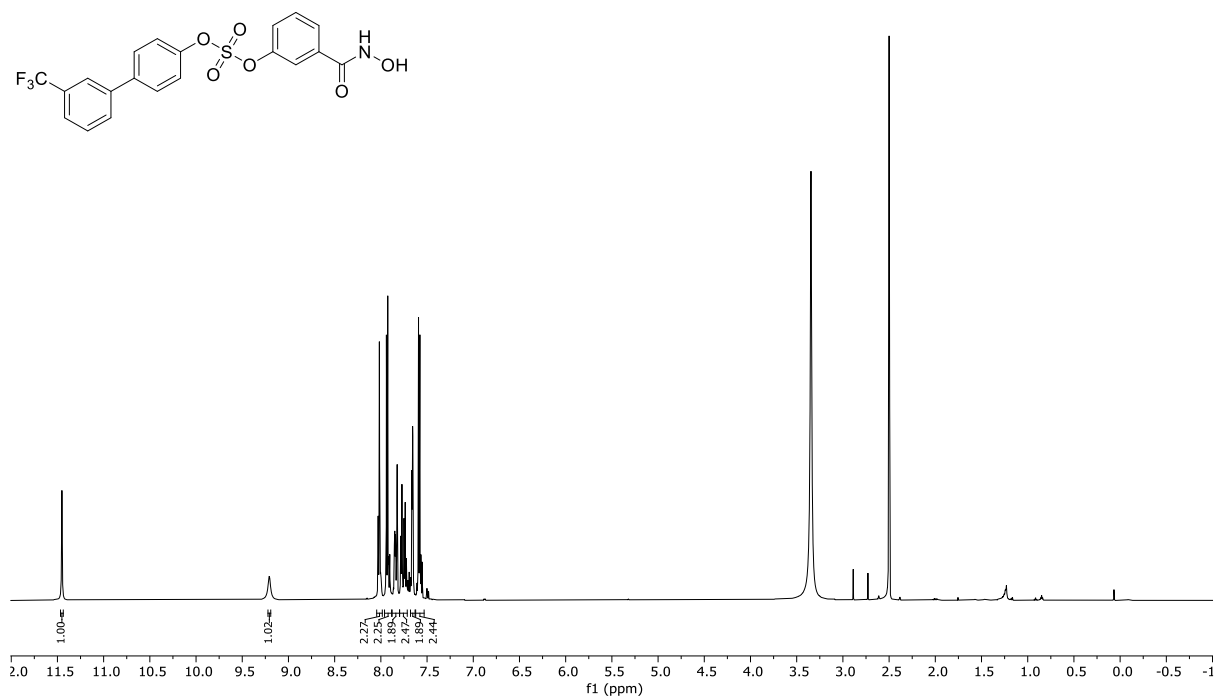
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A26**



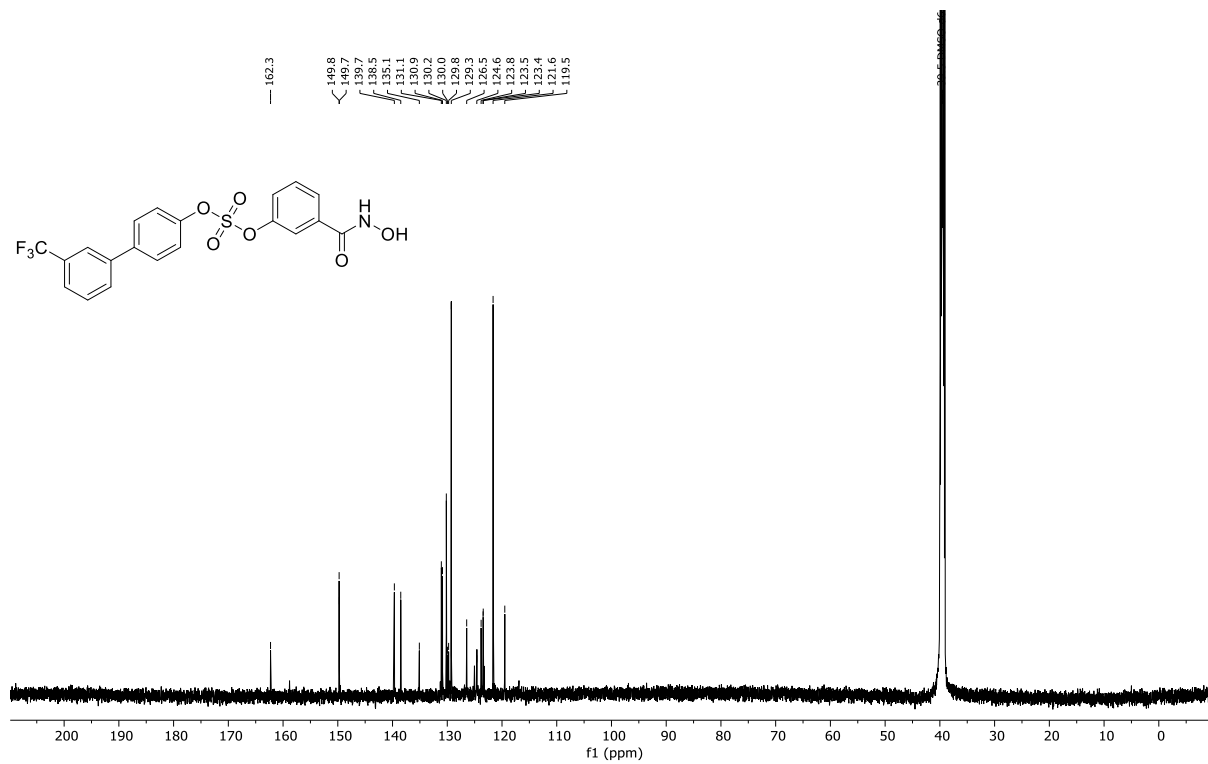
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A26**



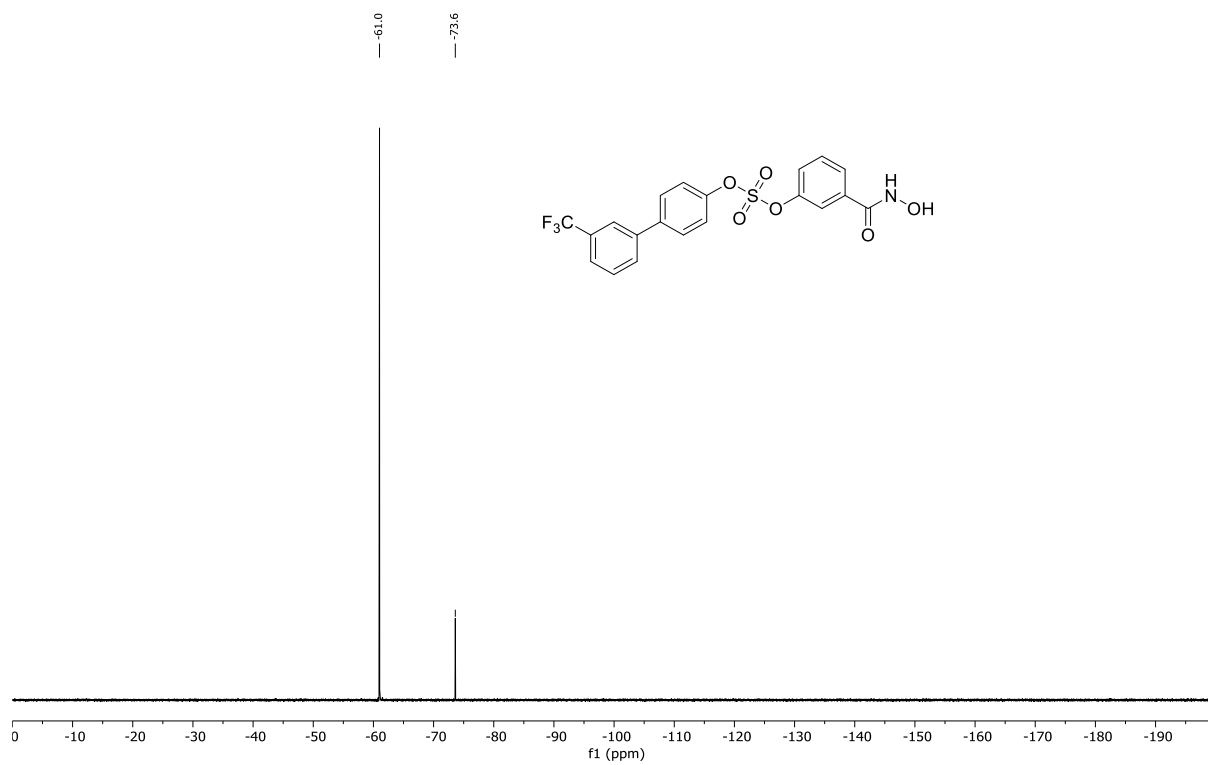
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **A27**



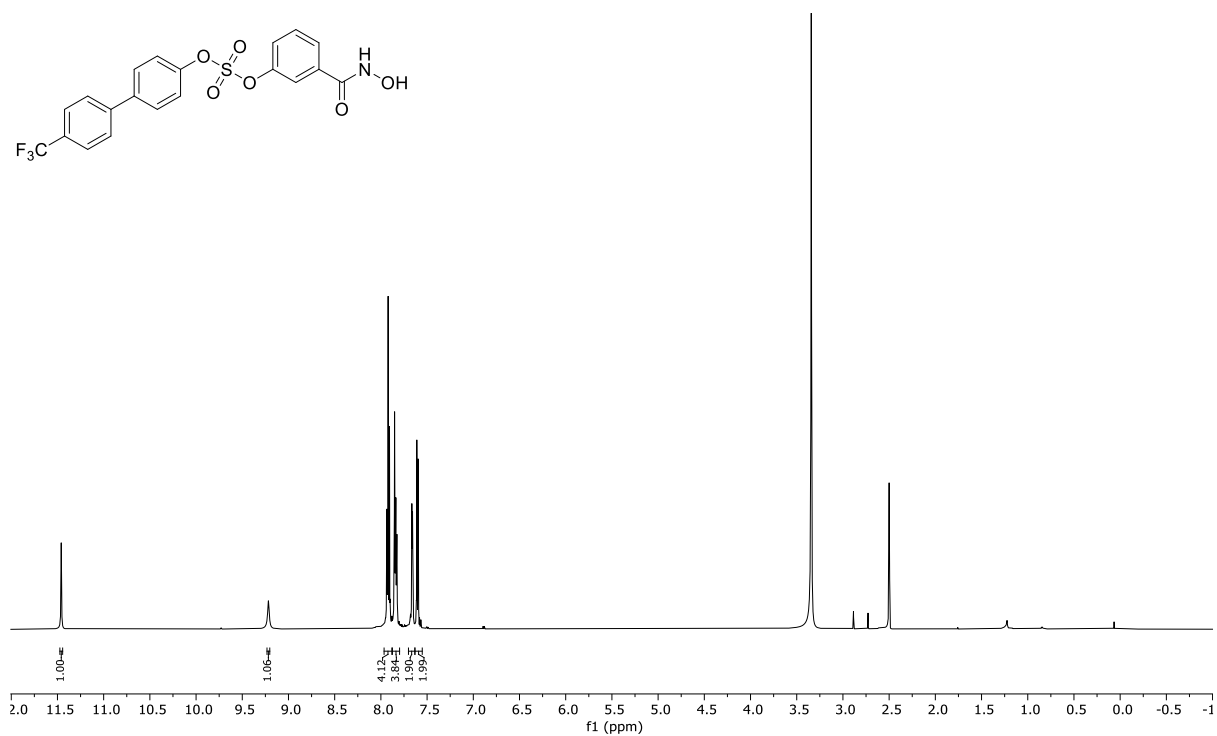
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **A27** (minor contamination with TFA is observed at 158.8 ppm)



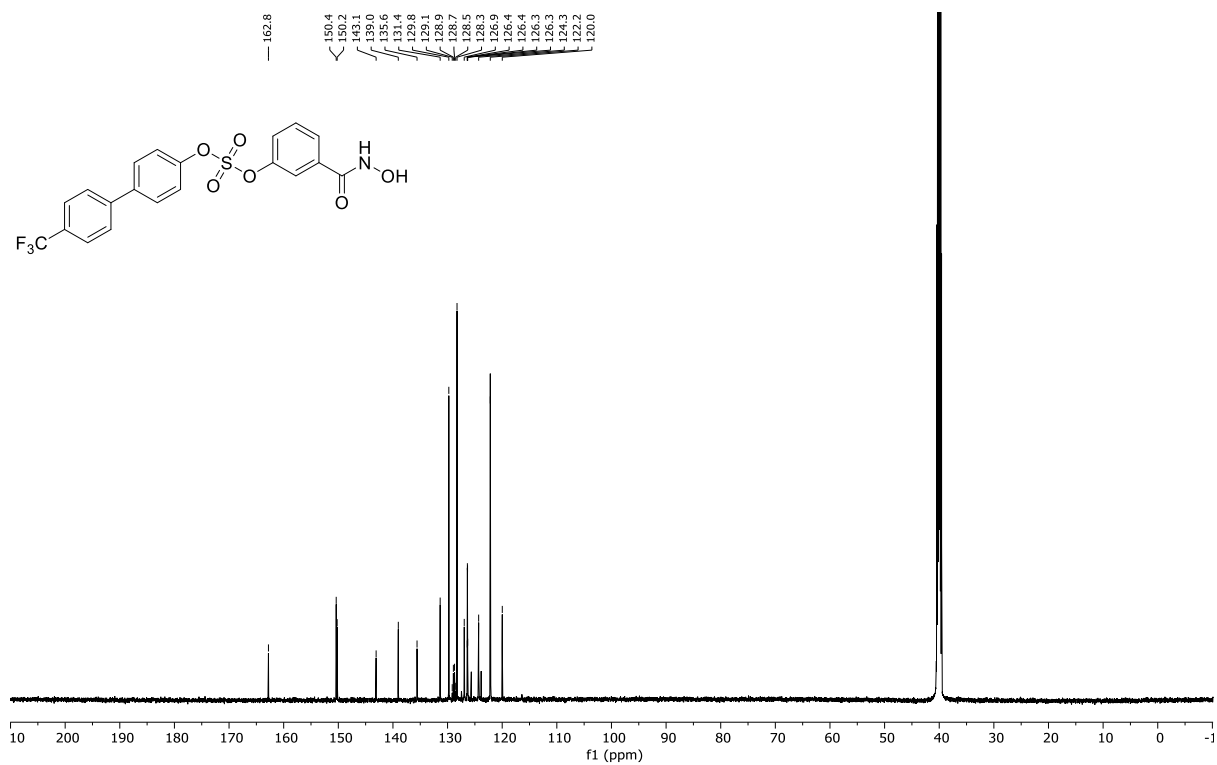
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **A27**



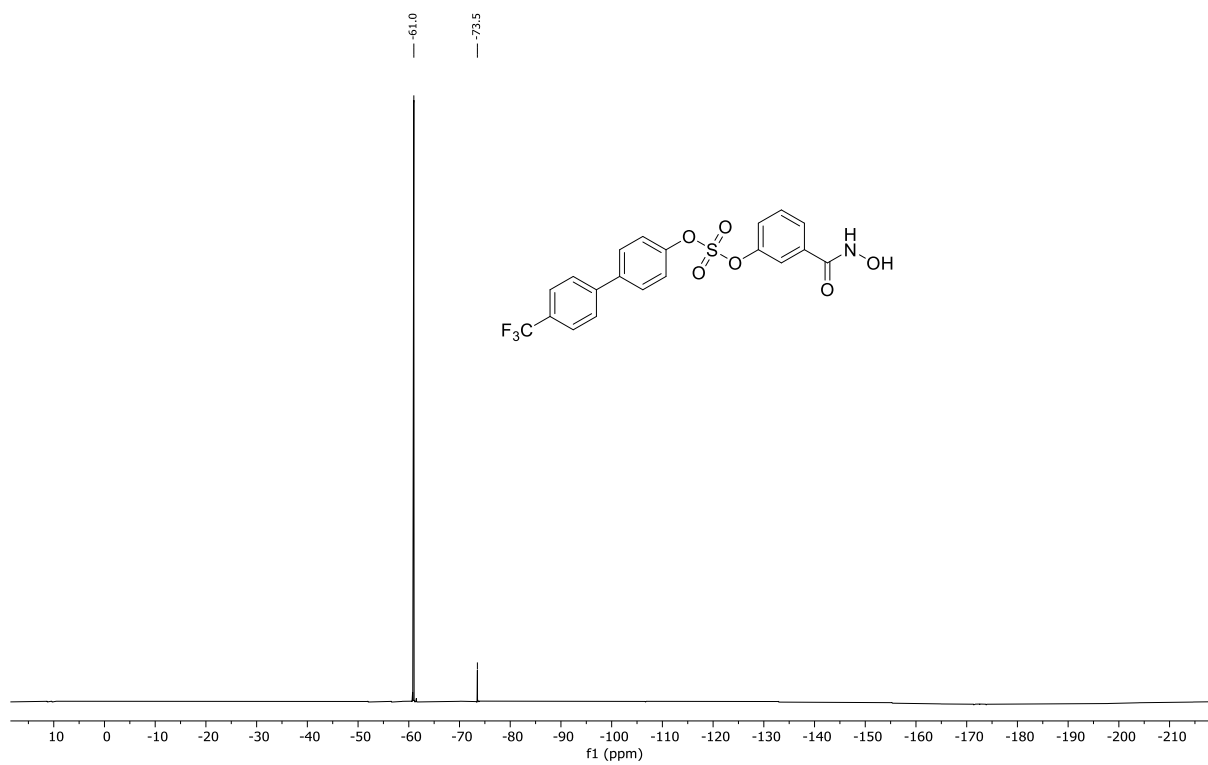
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **A28**



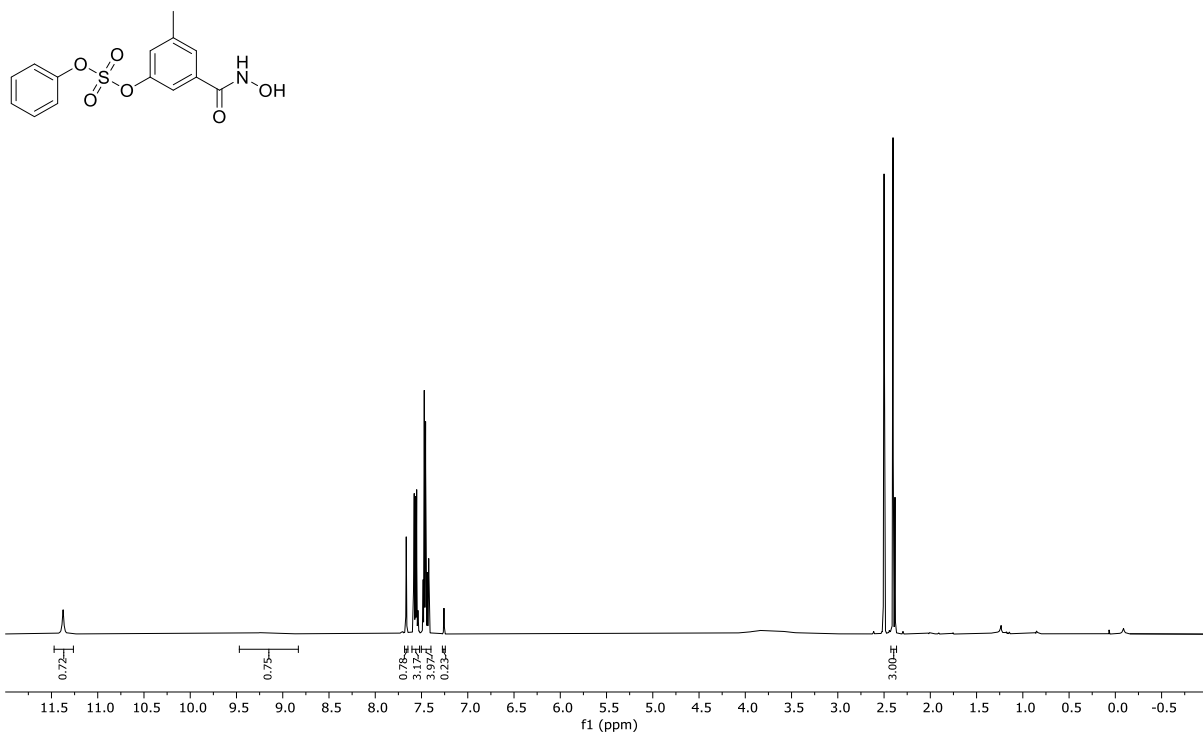
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **A28**



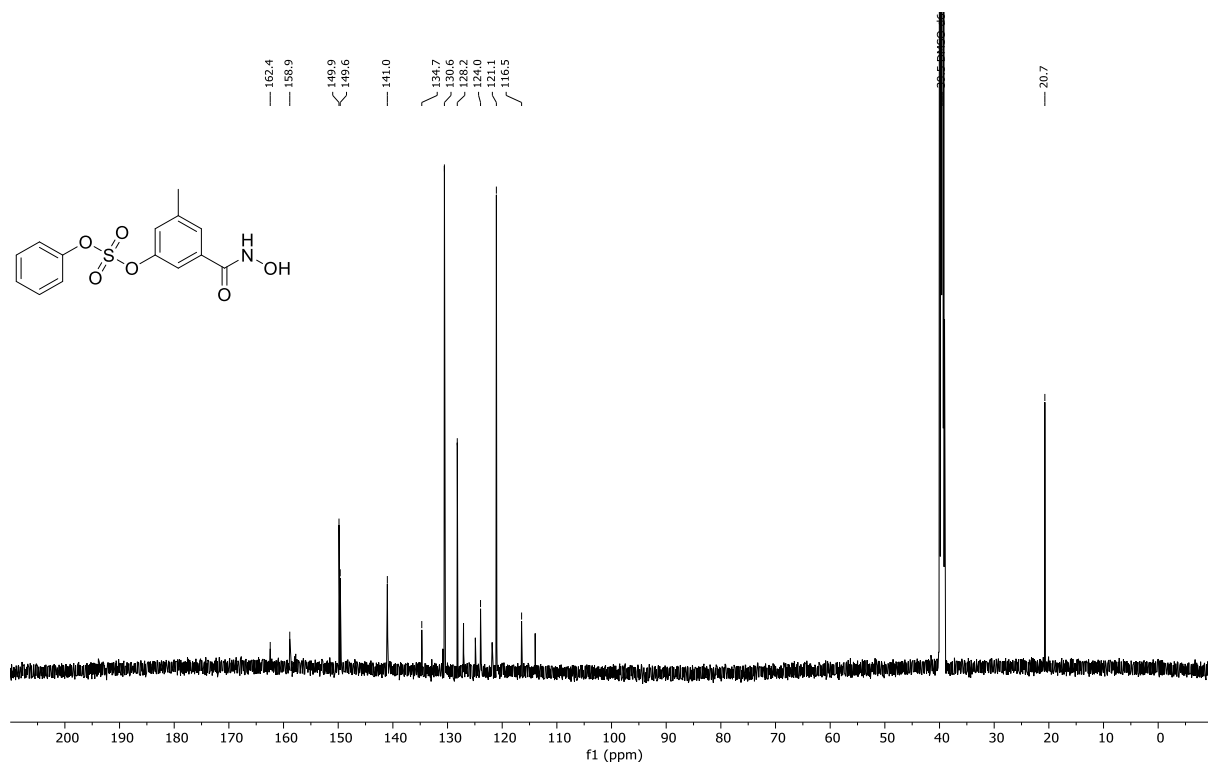
¹⁹F NMR spectra (376 MHz, DMSO-d₆) of compound **A28**



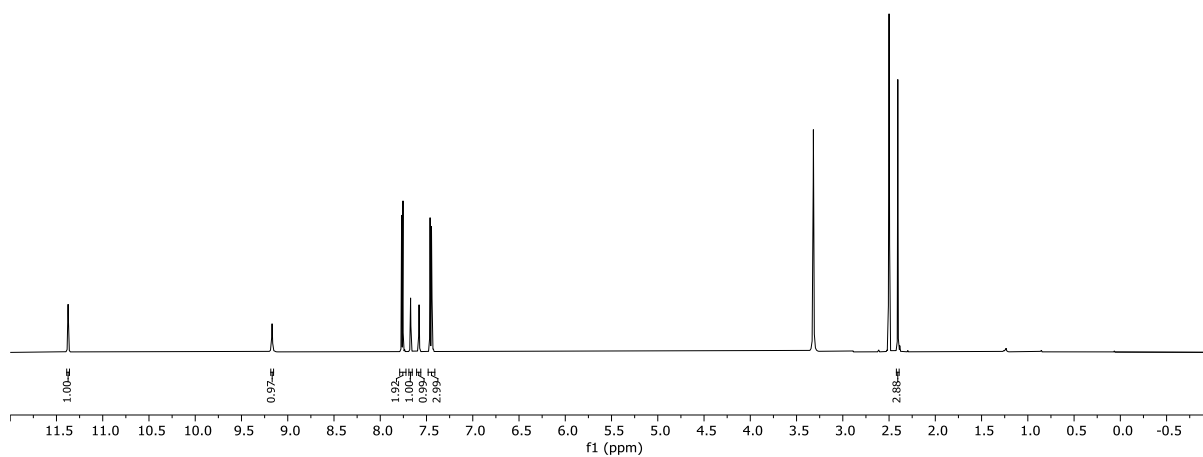
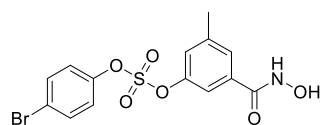
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **B1**



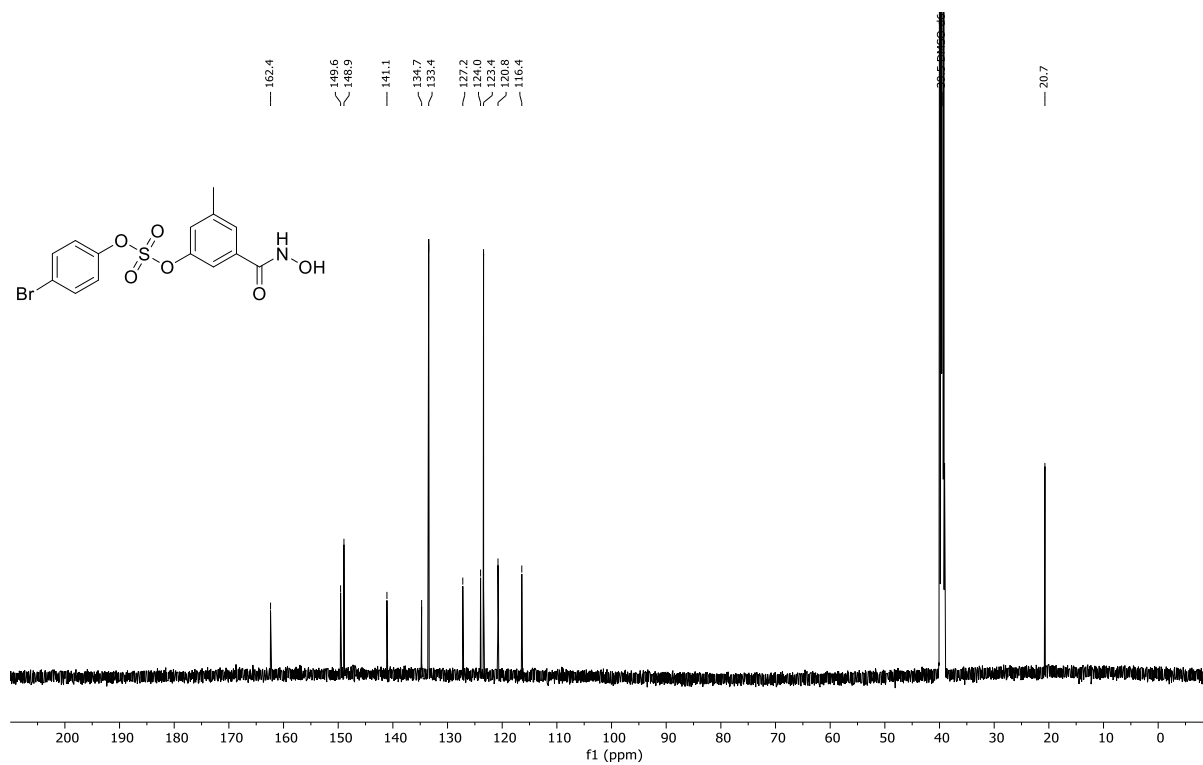
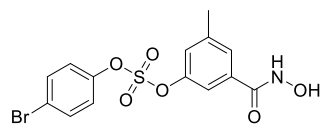
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **B1**



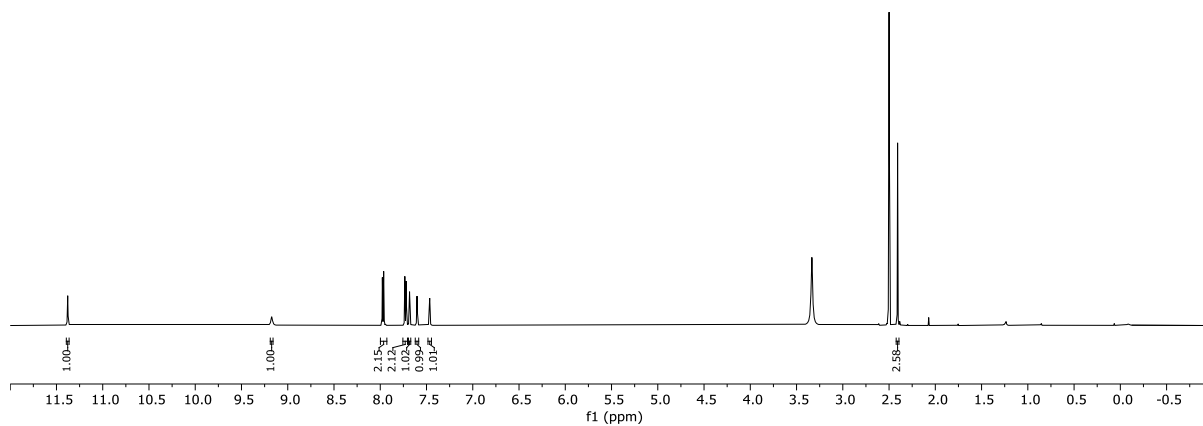
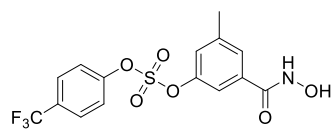
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **B2**



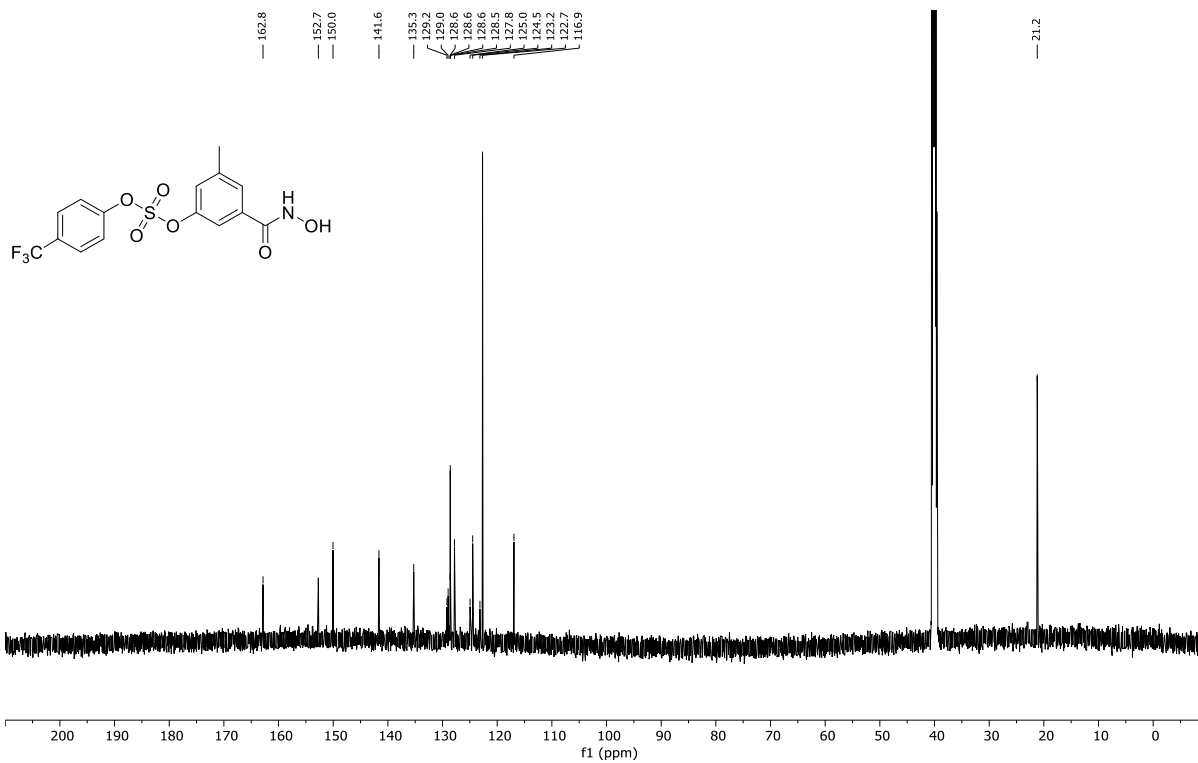
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **B2**



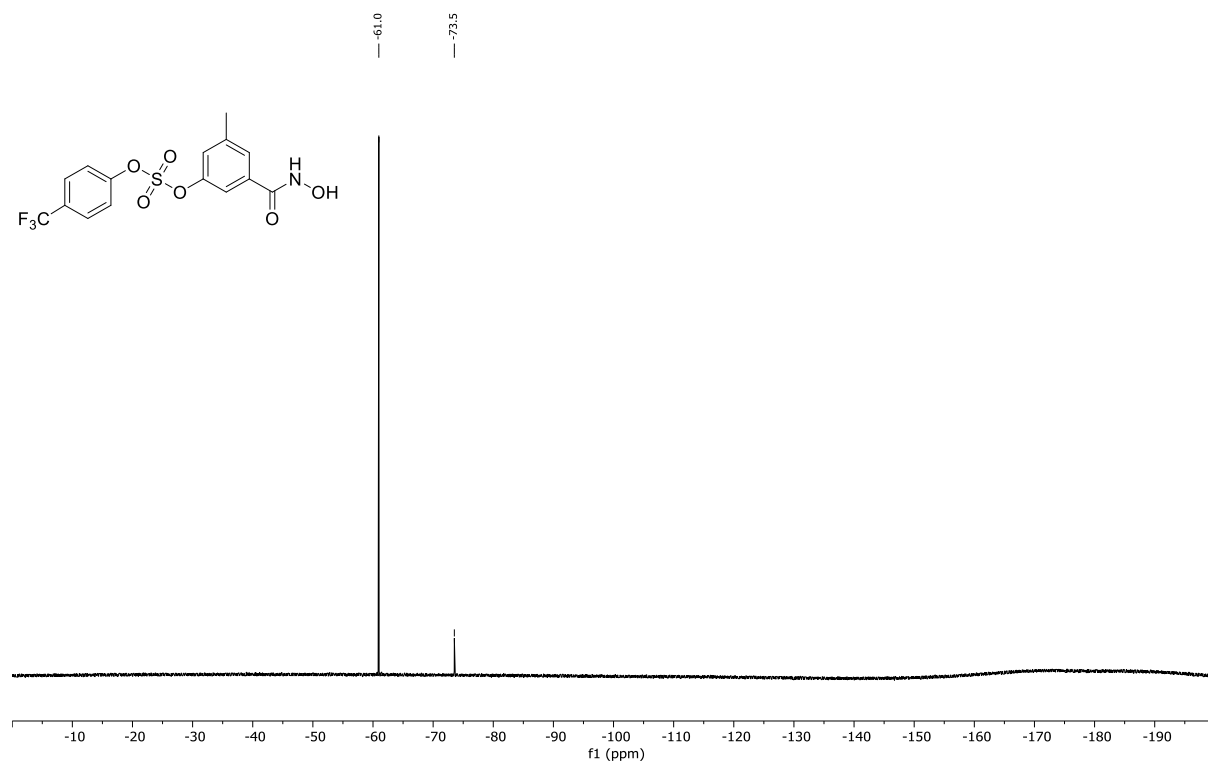
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **B3**



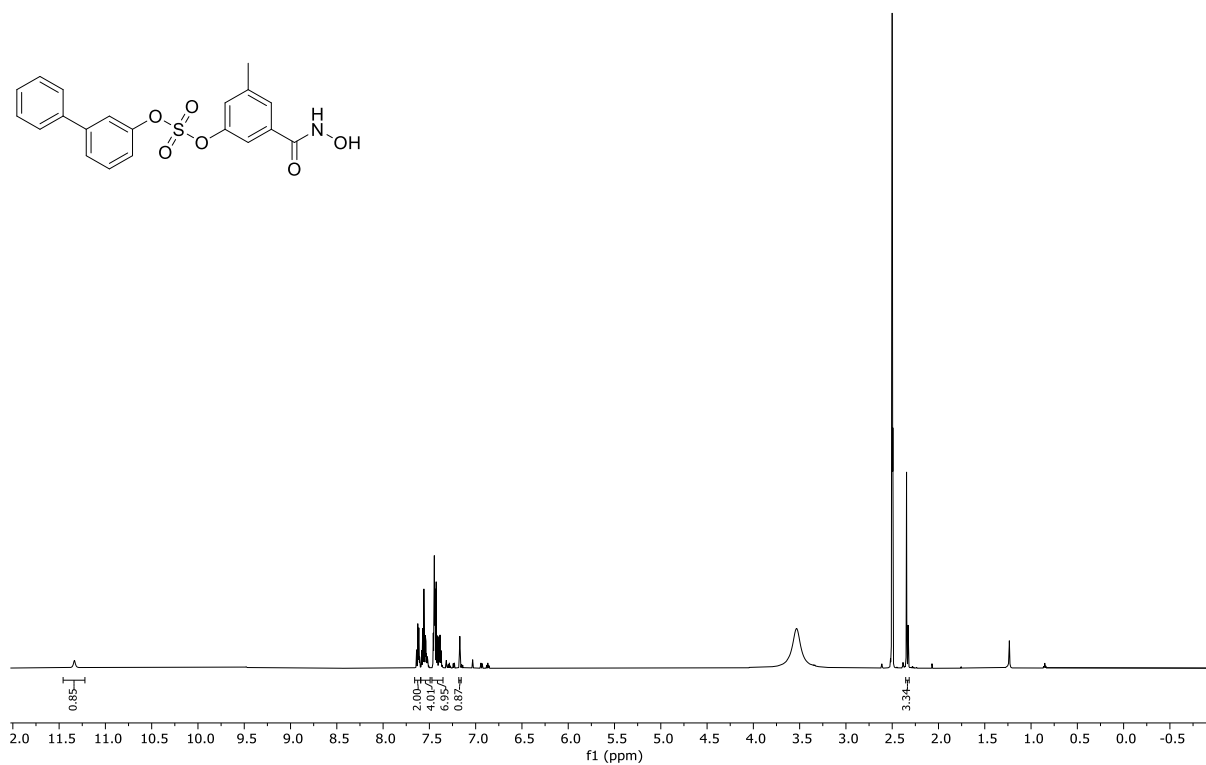
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **B3**



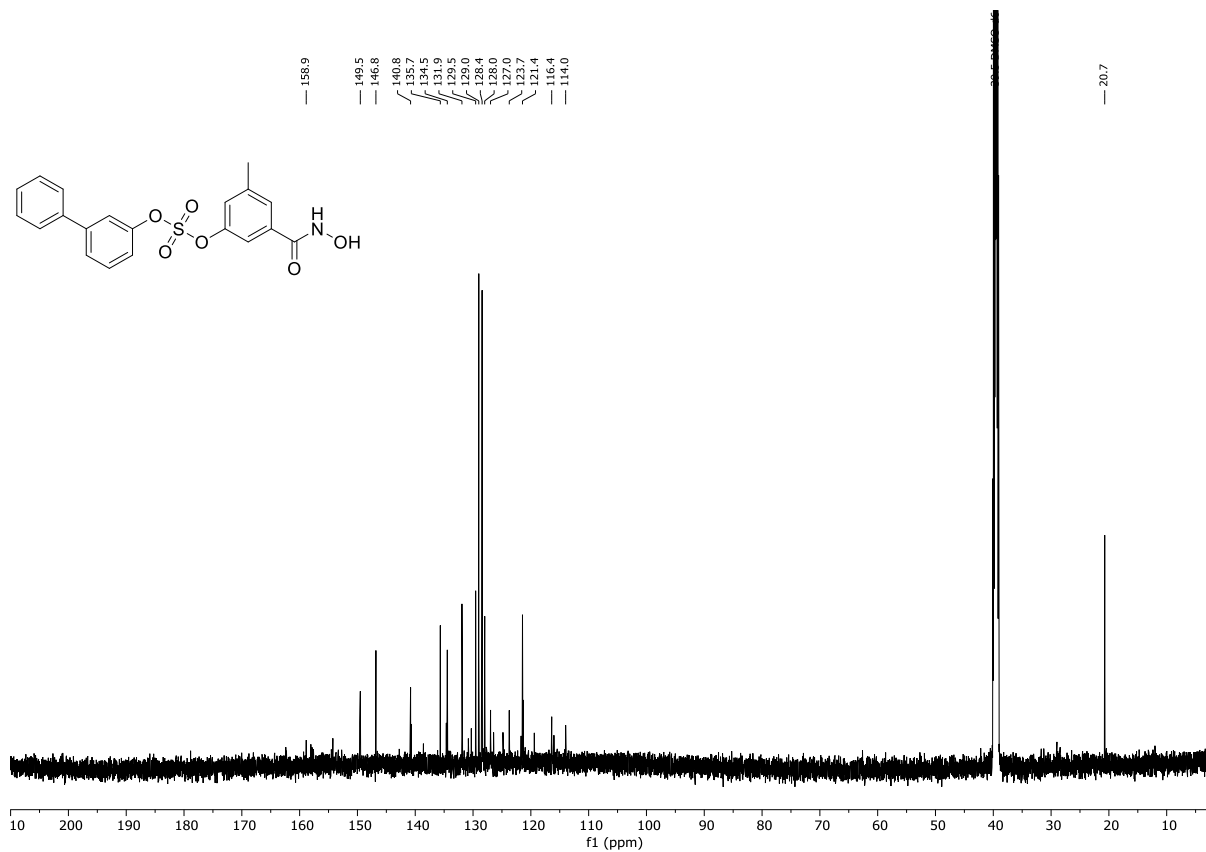
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **B3**



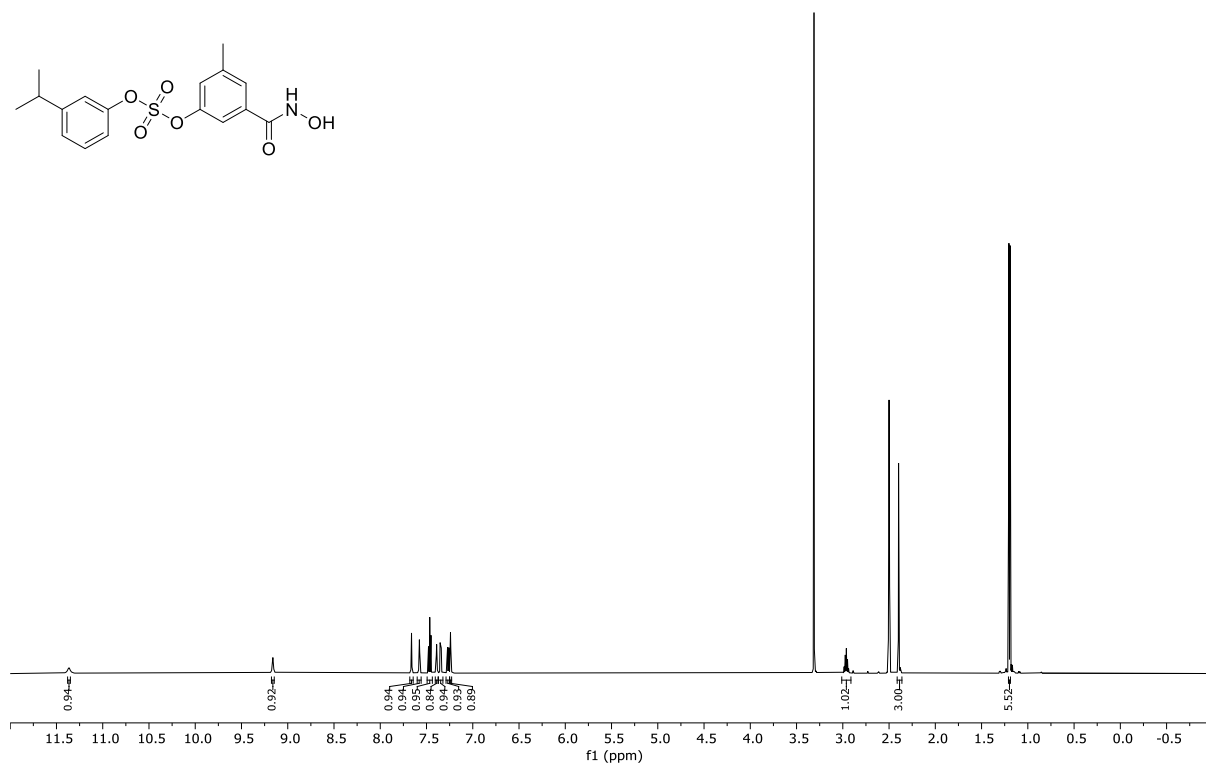
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B4**



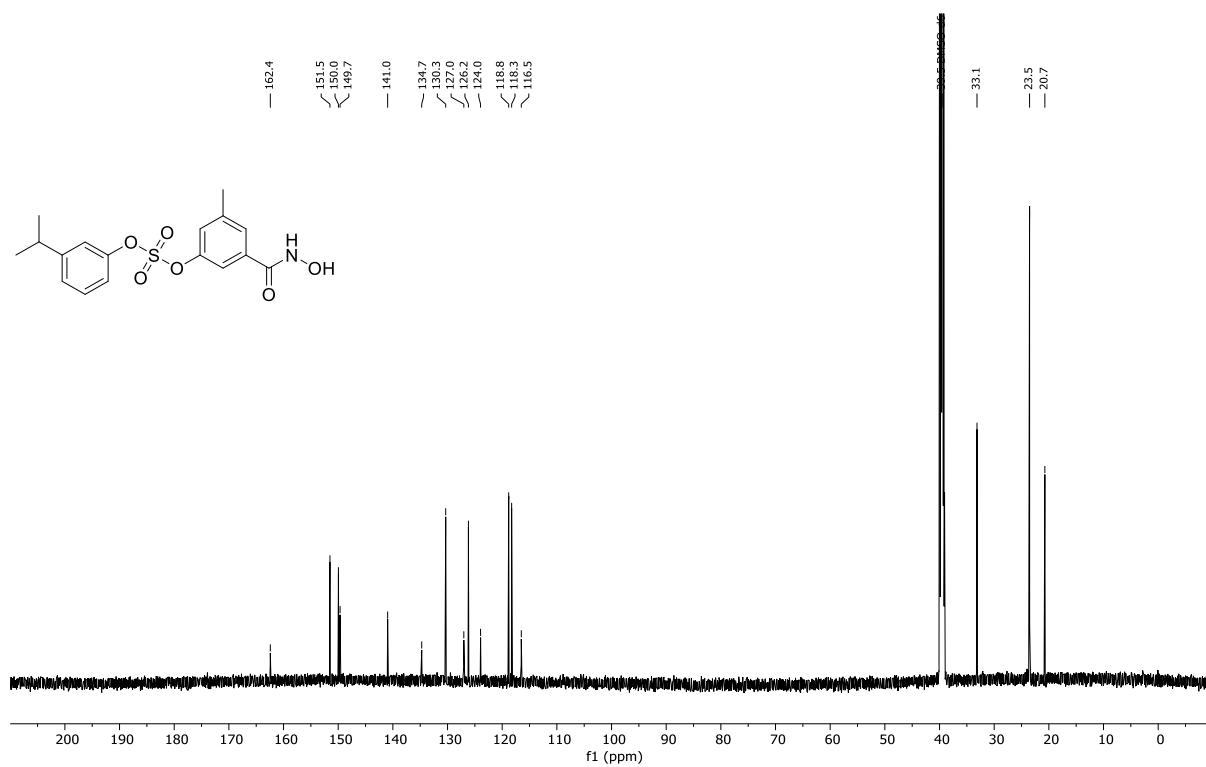
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B4** (minor contamination with TFA is observed at 158.8 ppm)



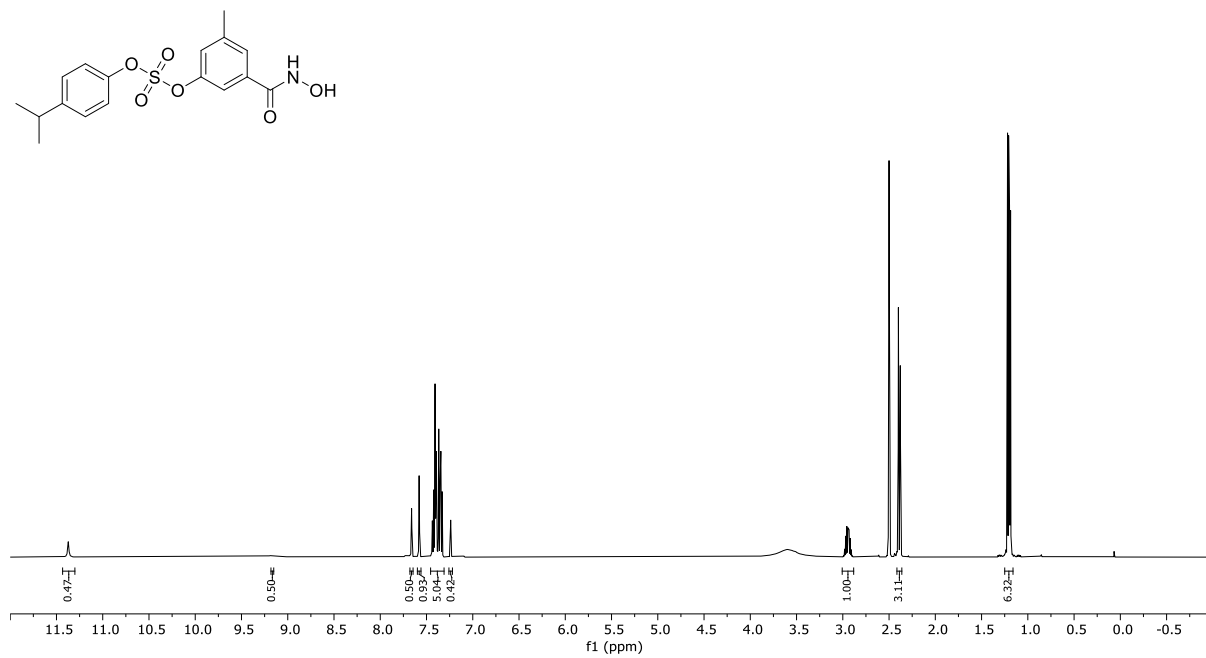
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B8**



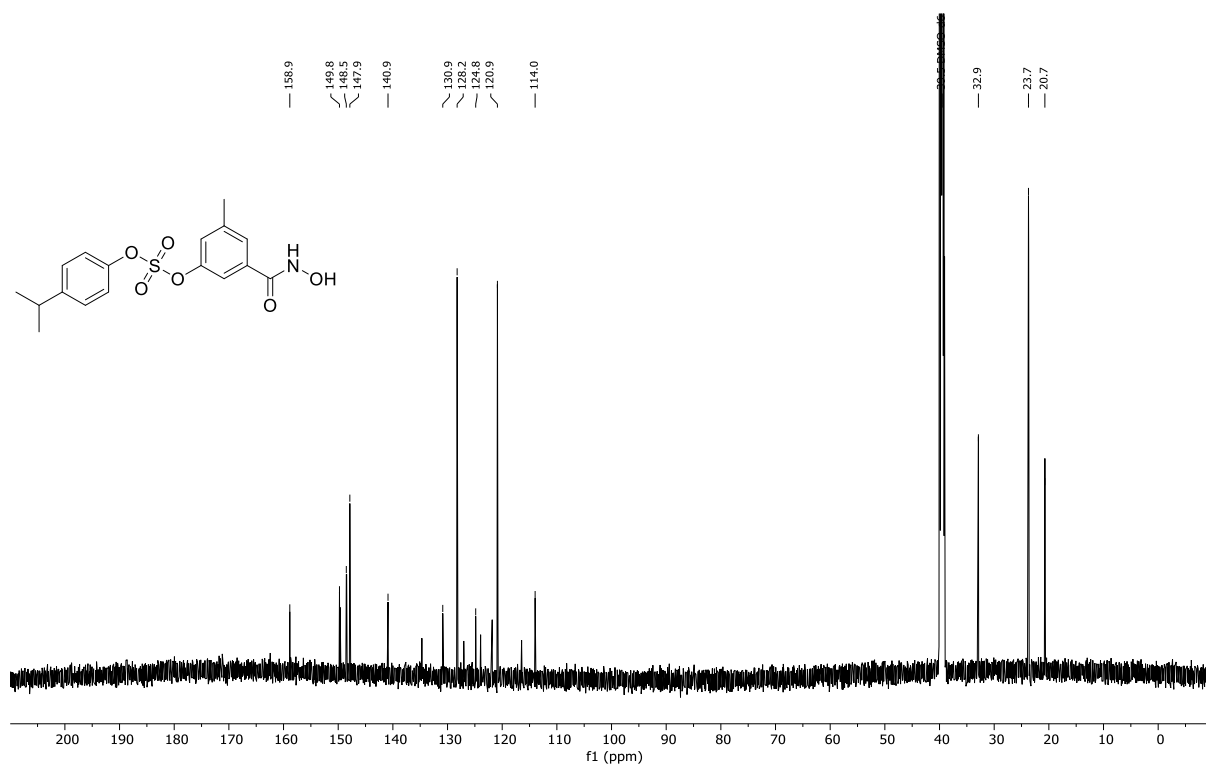
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B8**



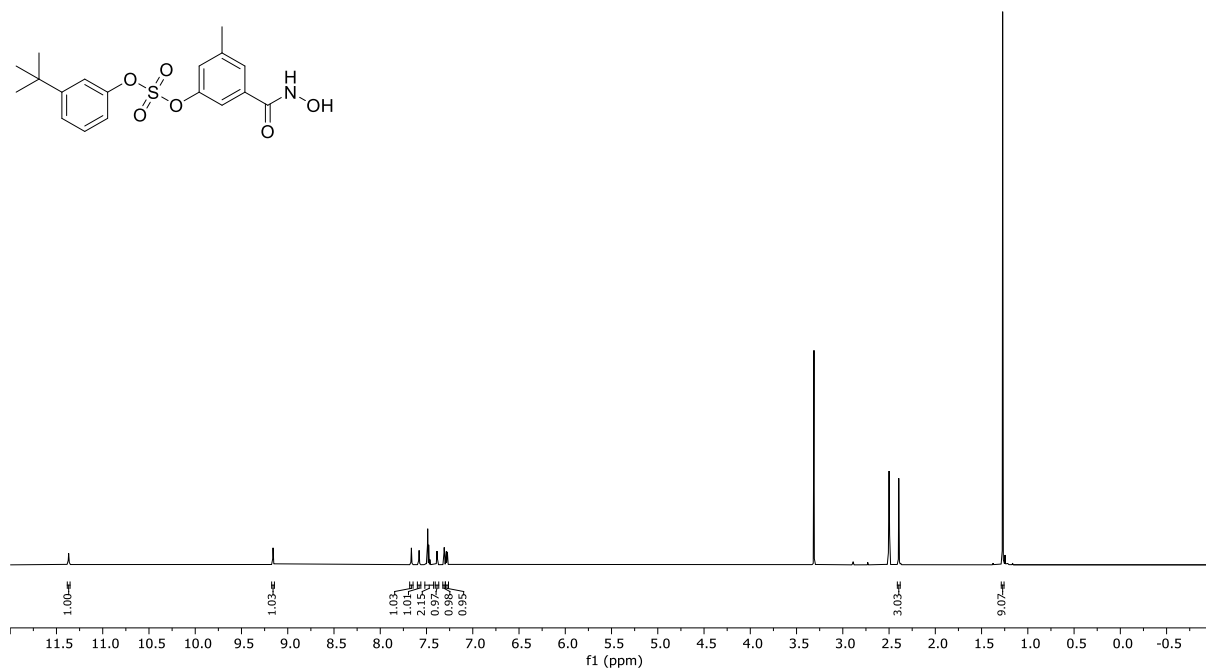
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B9**



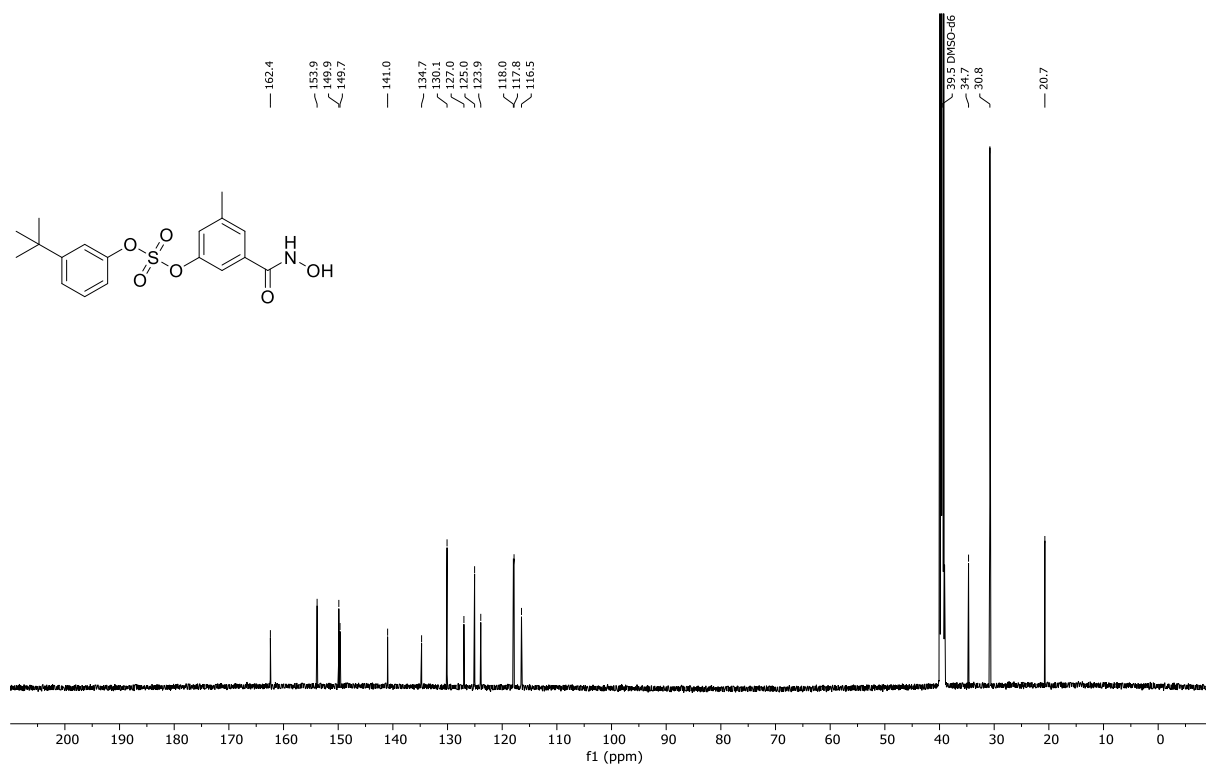
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B9**



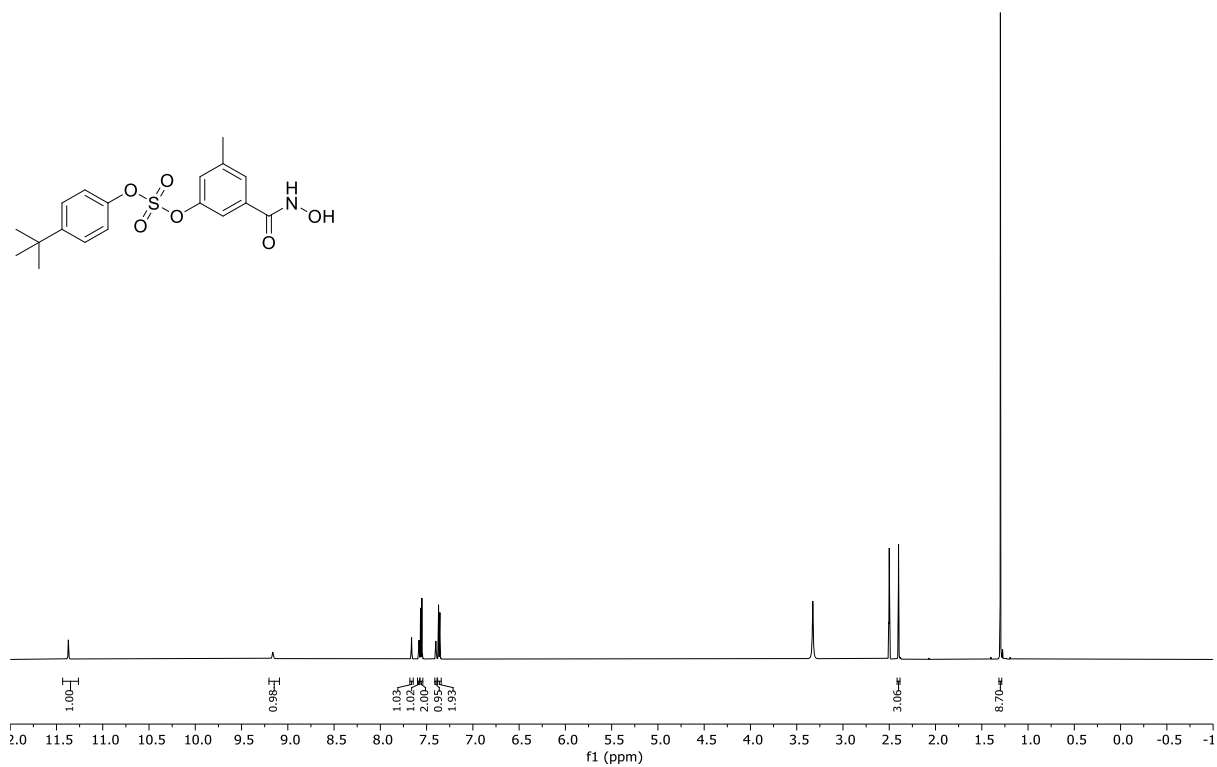
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **B11**



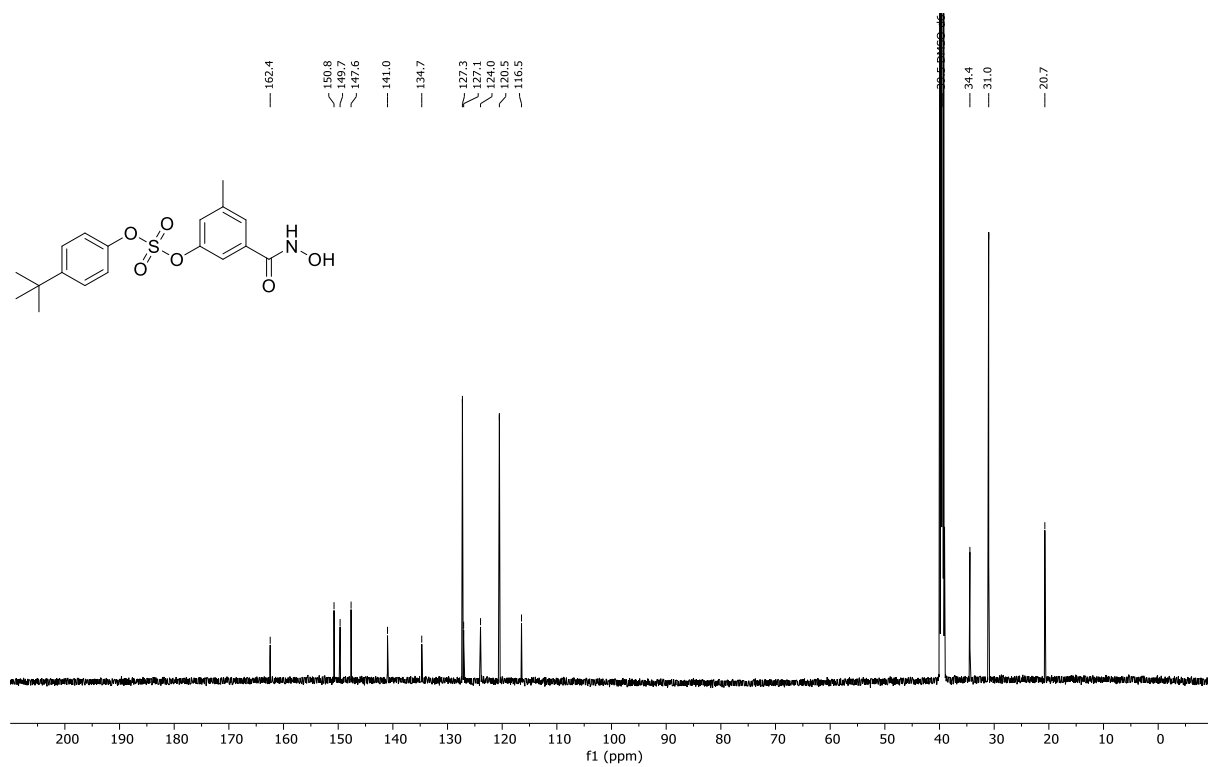
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **B11**



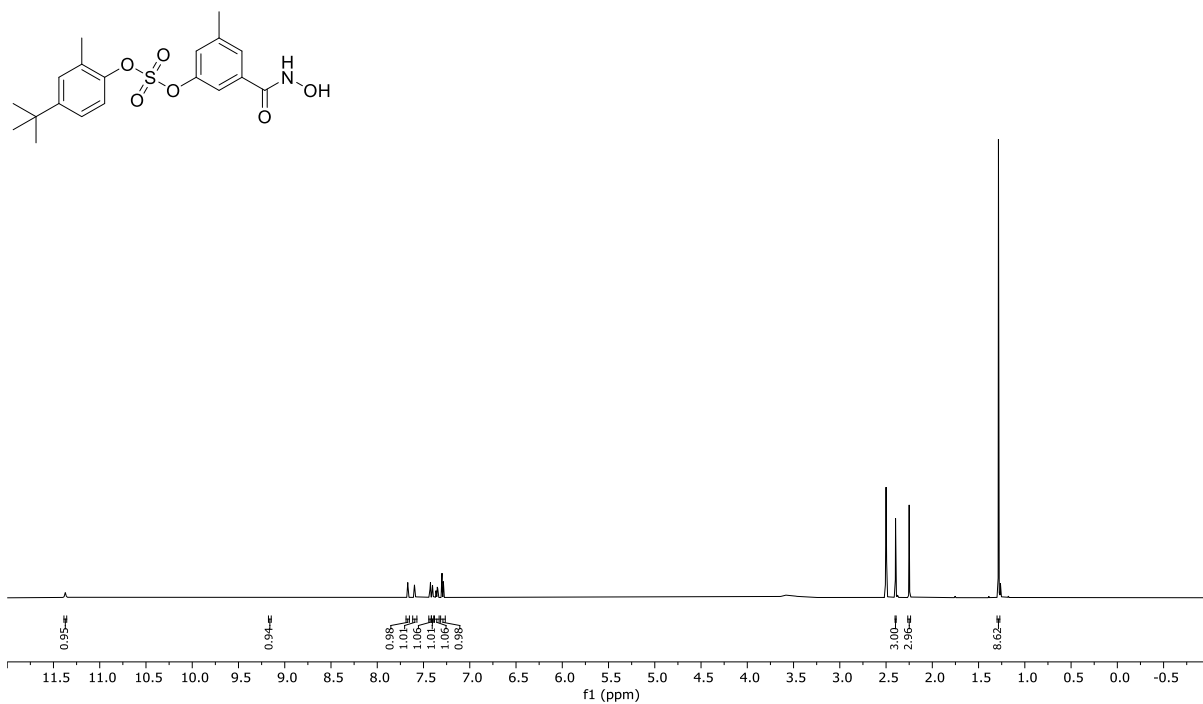
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B12**



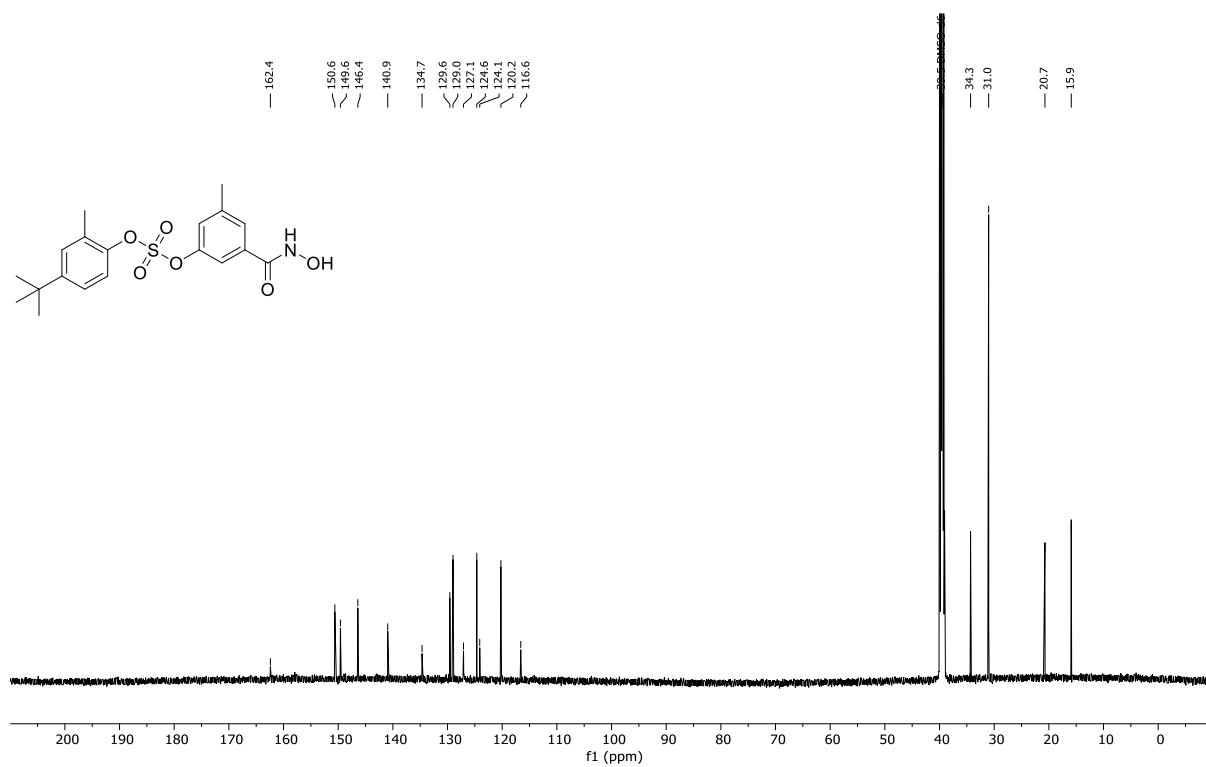
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B12**



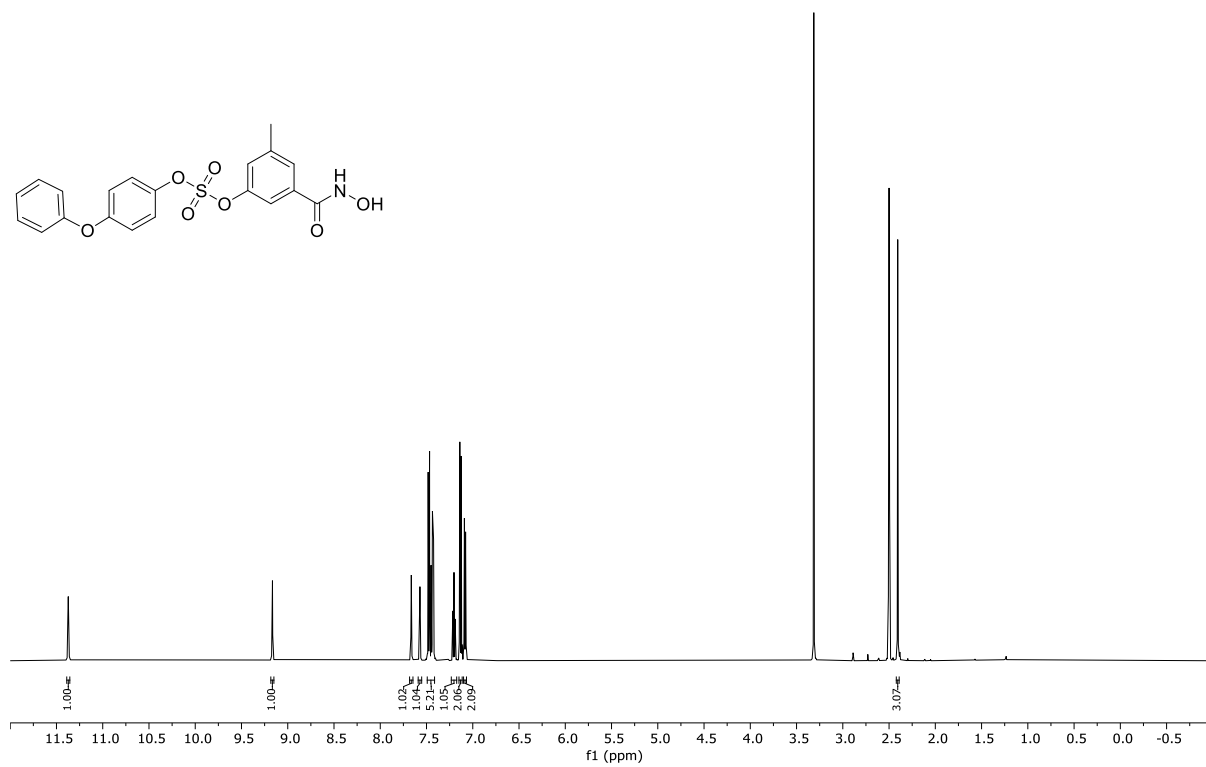
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B19**



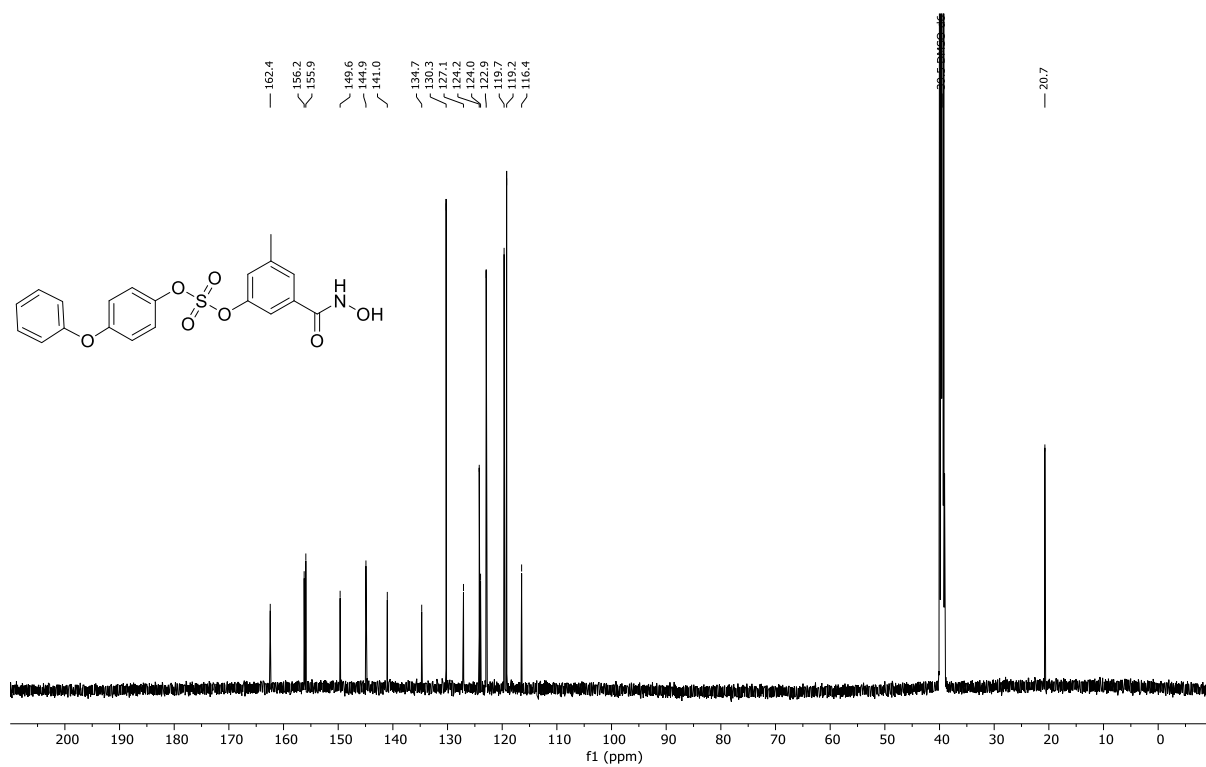
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B19**



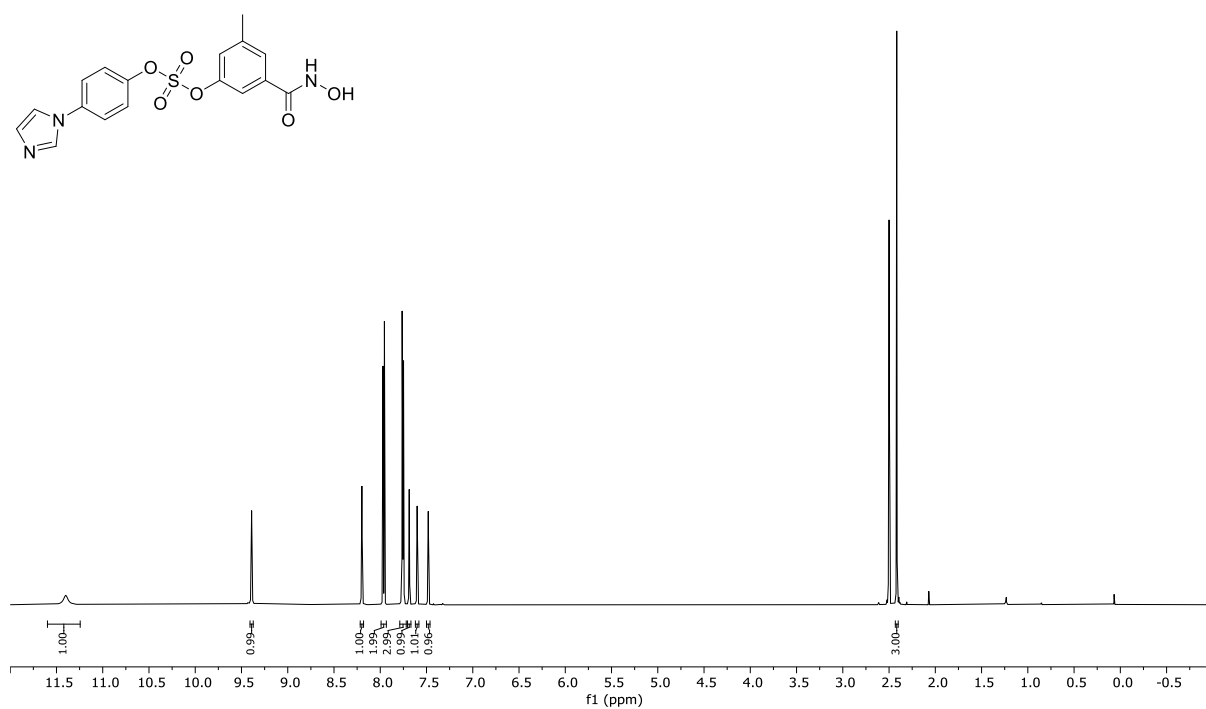
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B21**



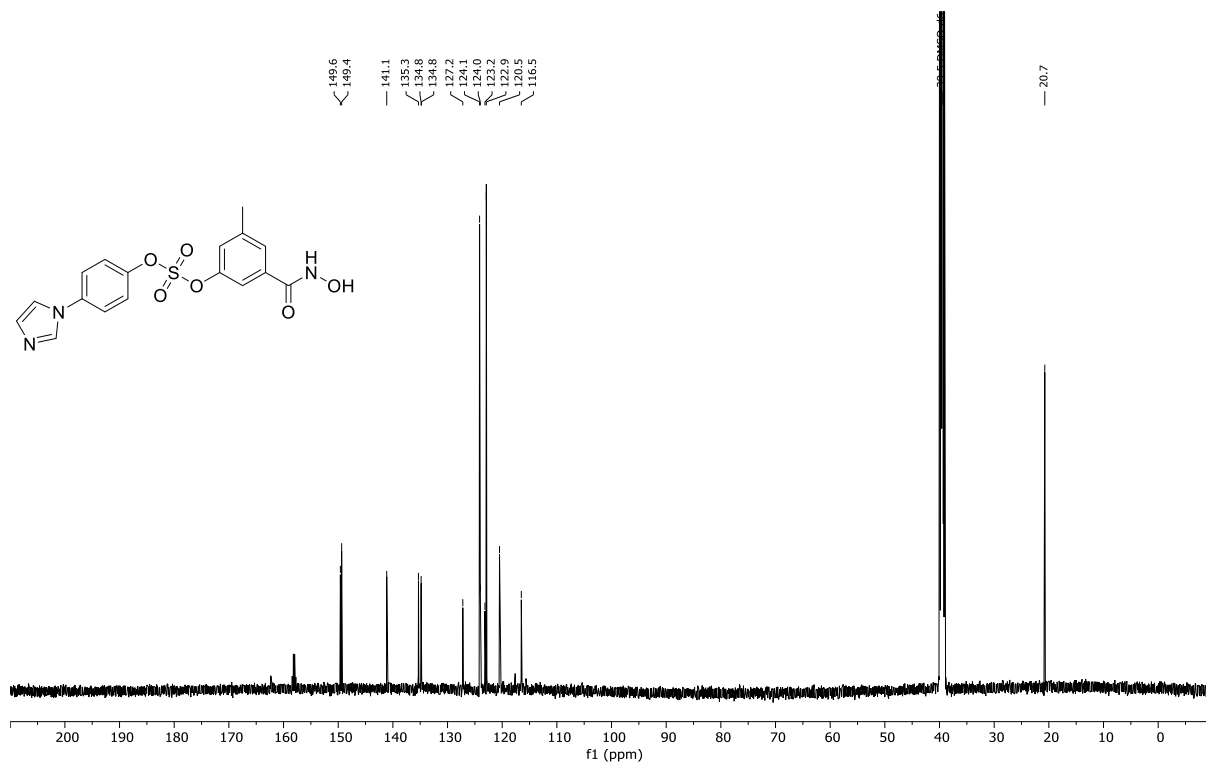
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B21**



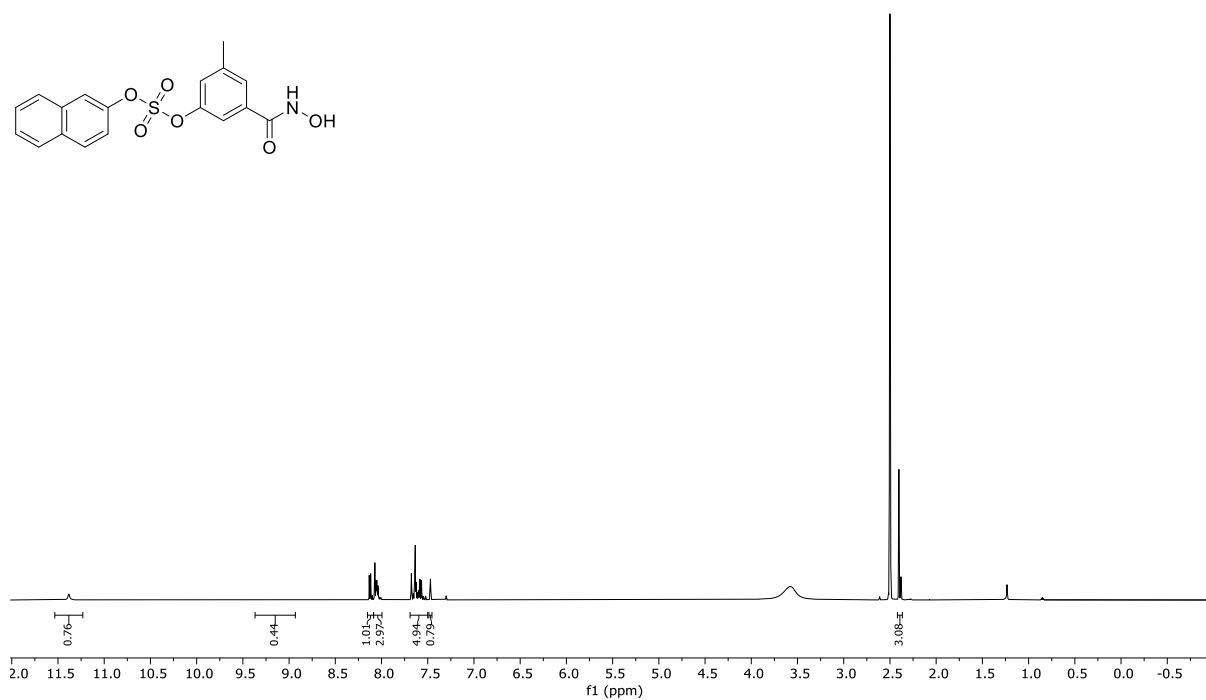
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B23**



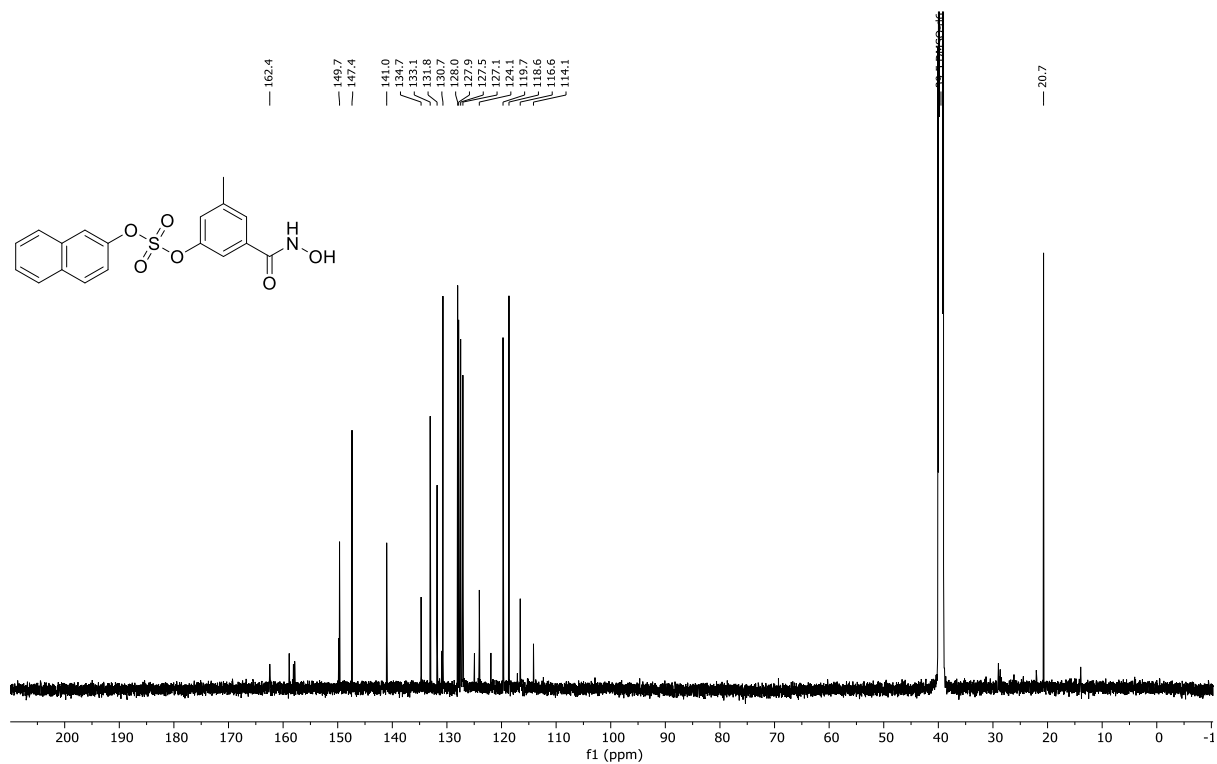
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B23** (minor contamination with TFA is observed at 158.8 ppm)



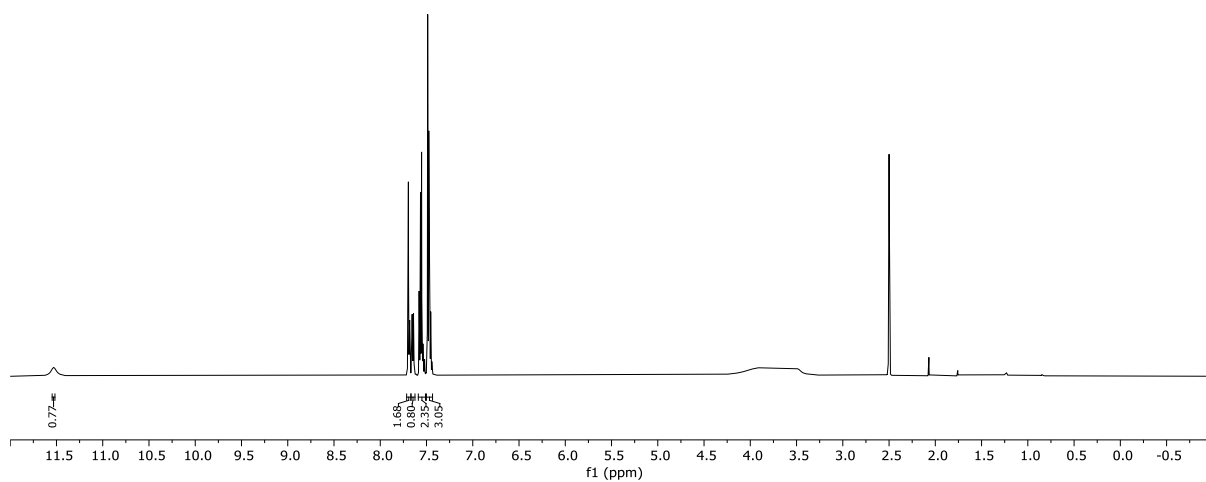
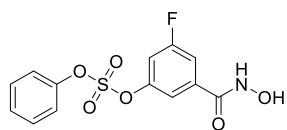
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **B24**



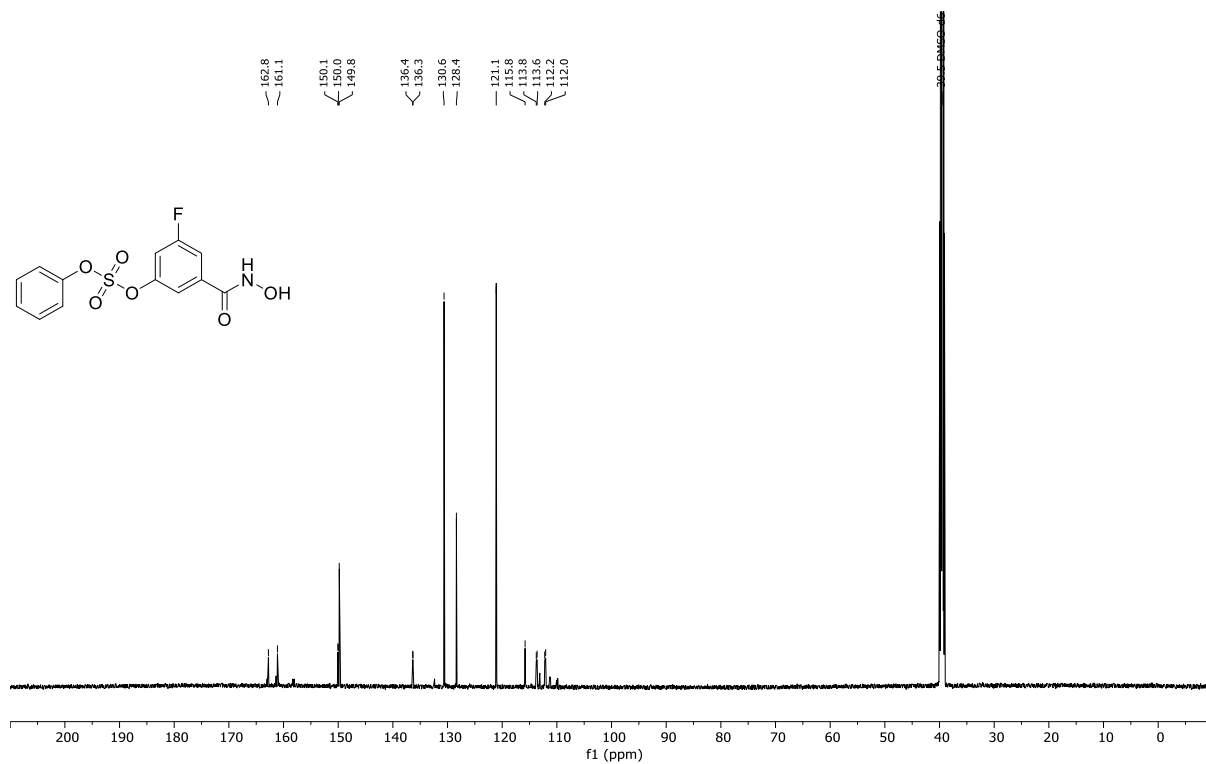
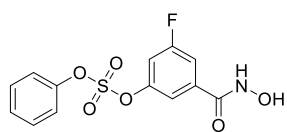
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **B24** (minor contamination with TFA is observed at 158.8 ppm)



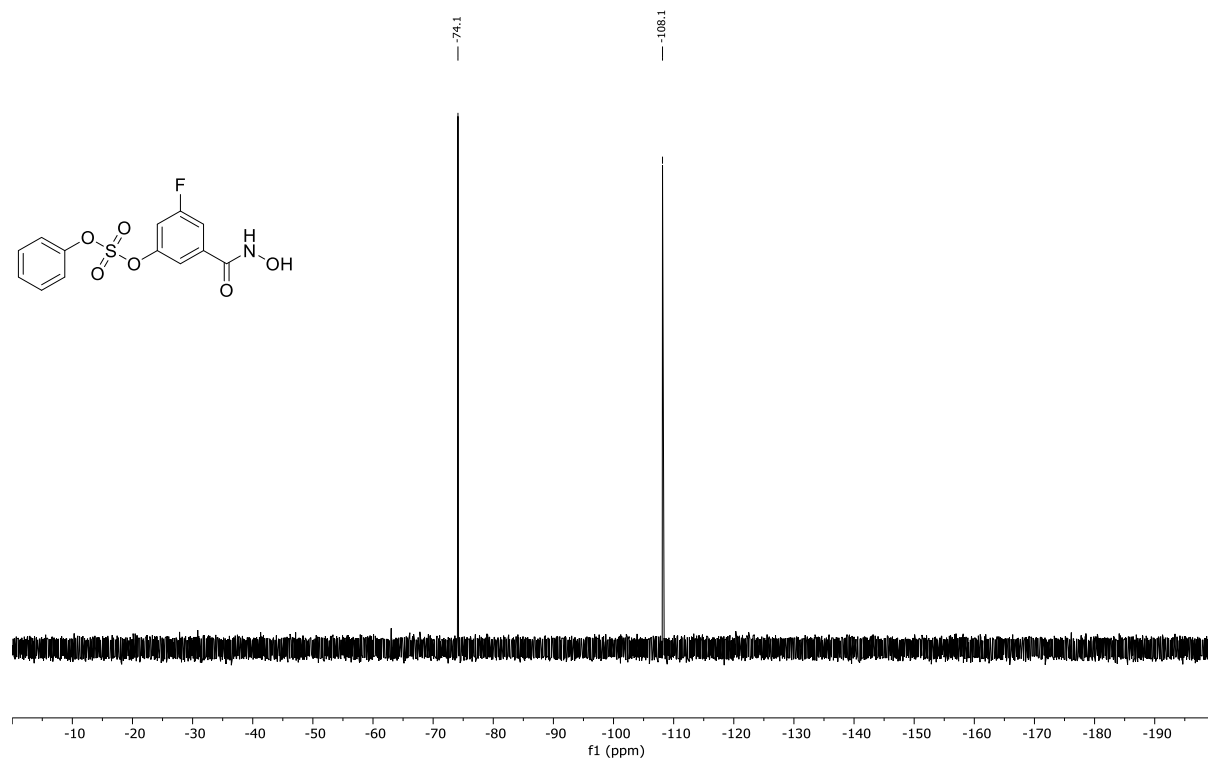
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **C1**



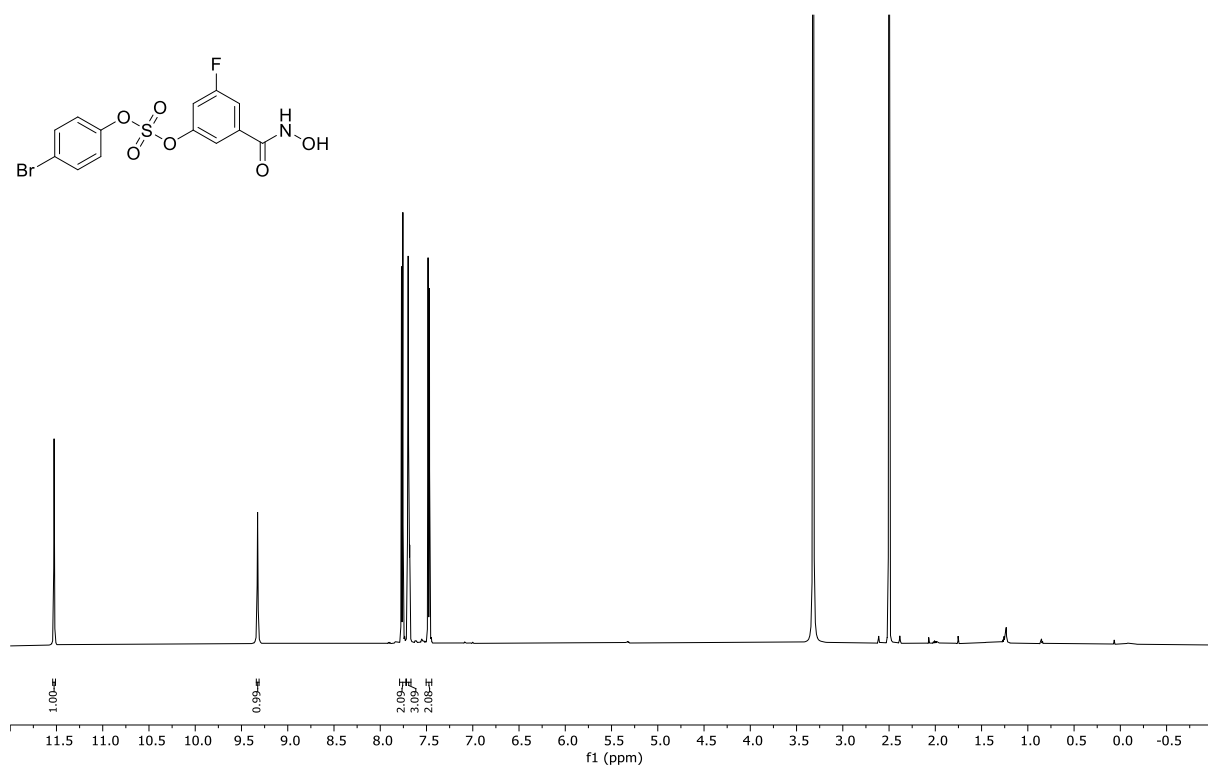
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **C1** (minor contamination with TFA is observed at 158.8 ppm)



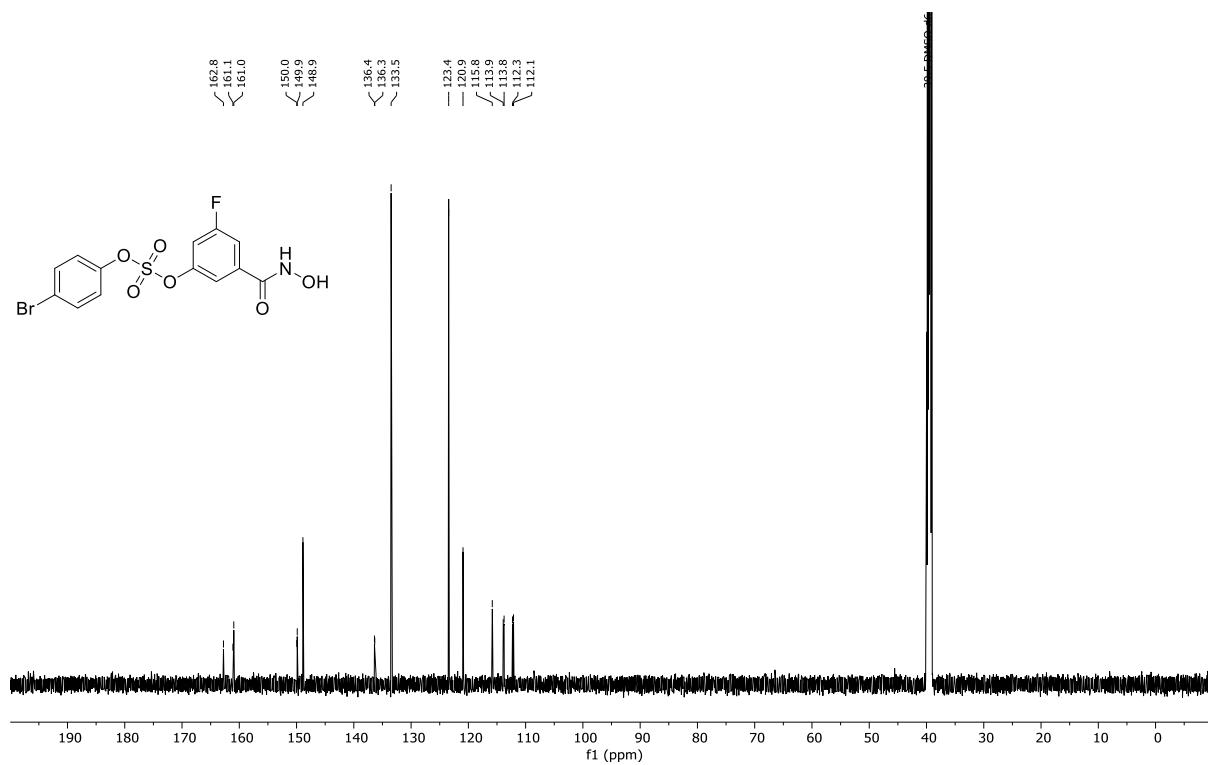
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C1**



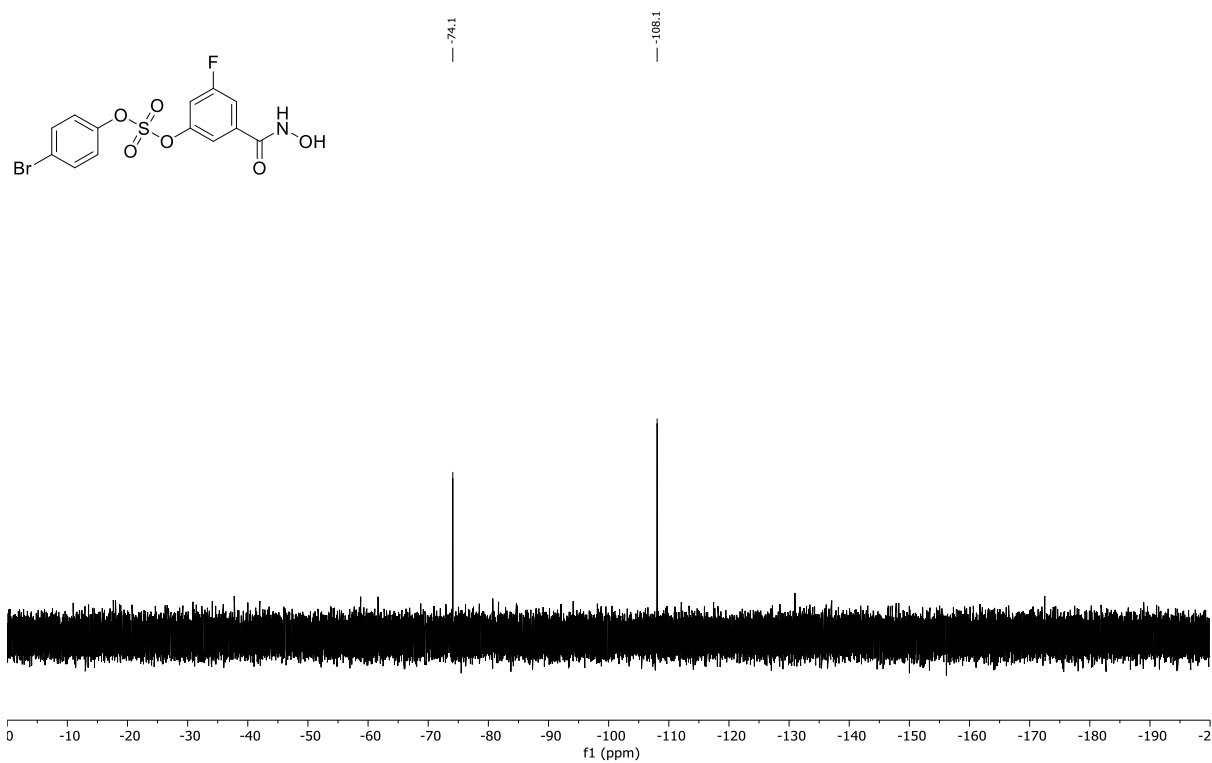
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C2**



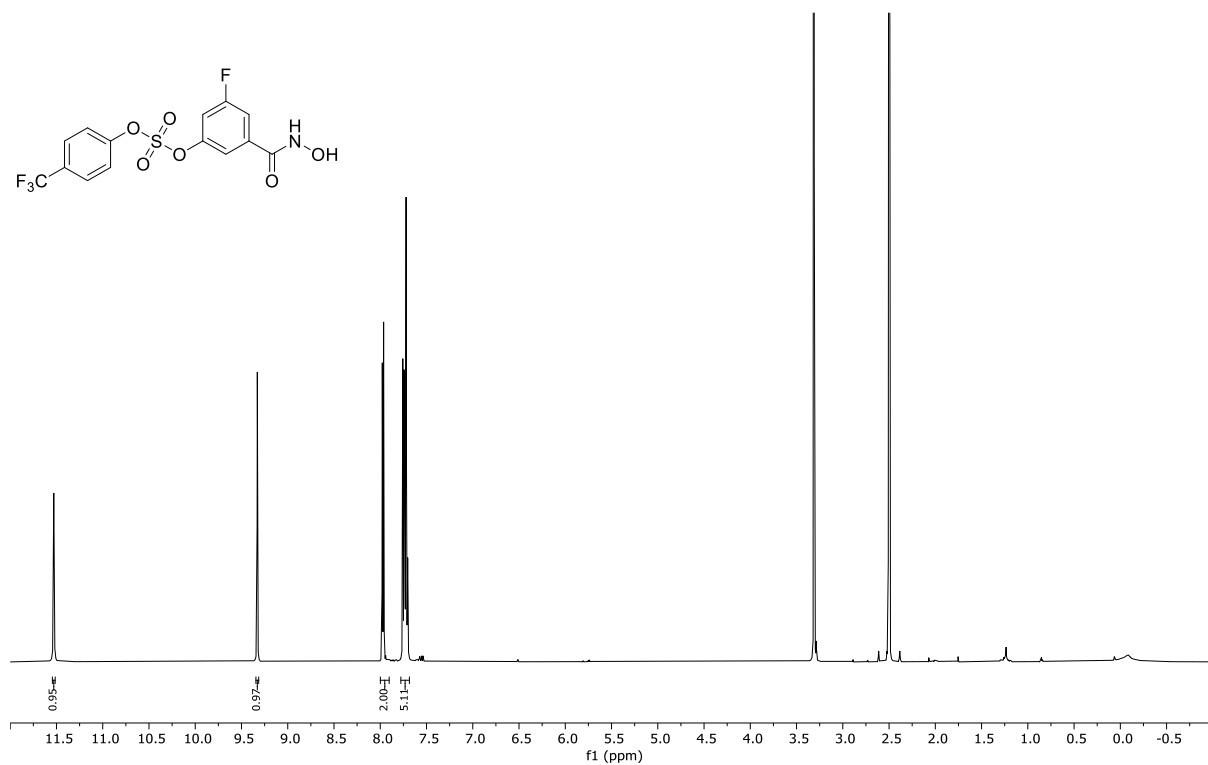
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C2**



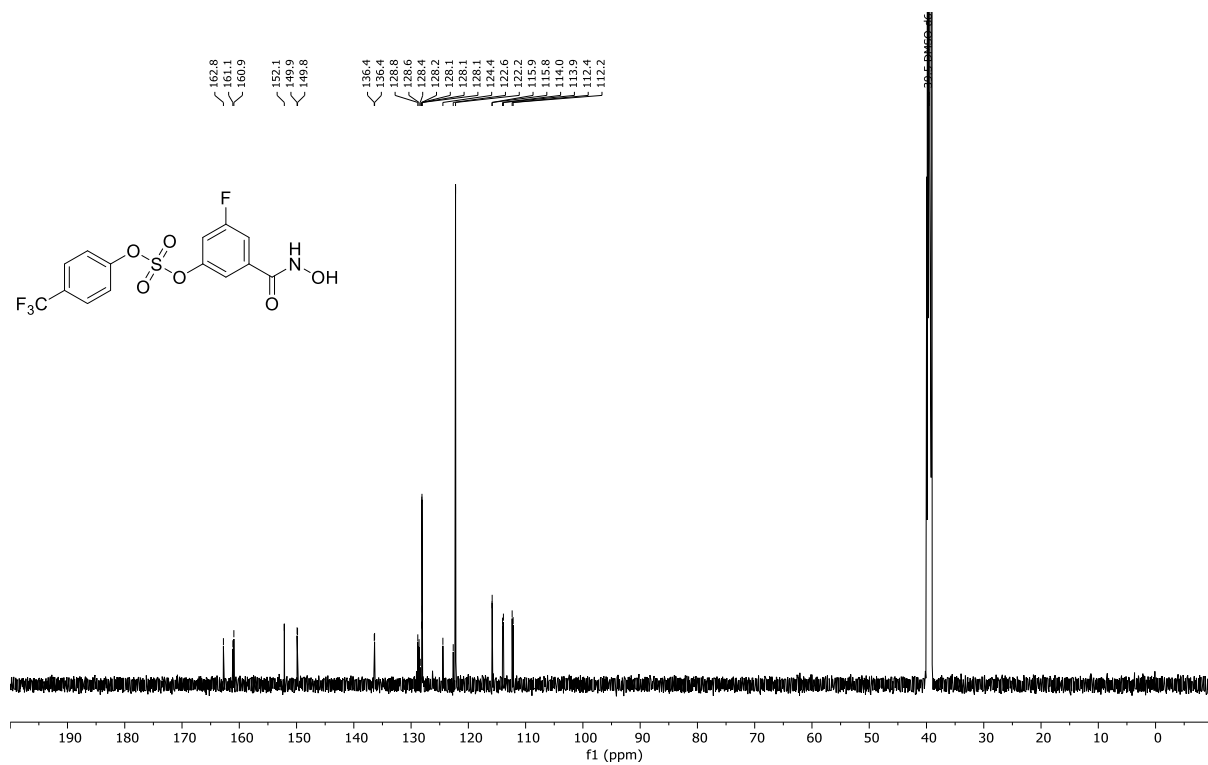
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C2**



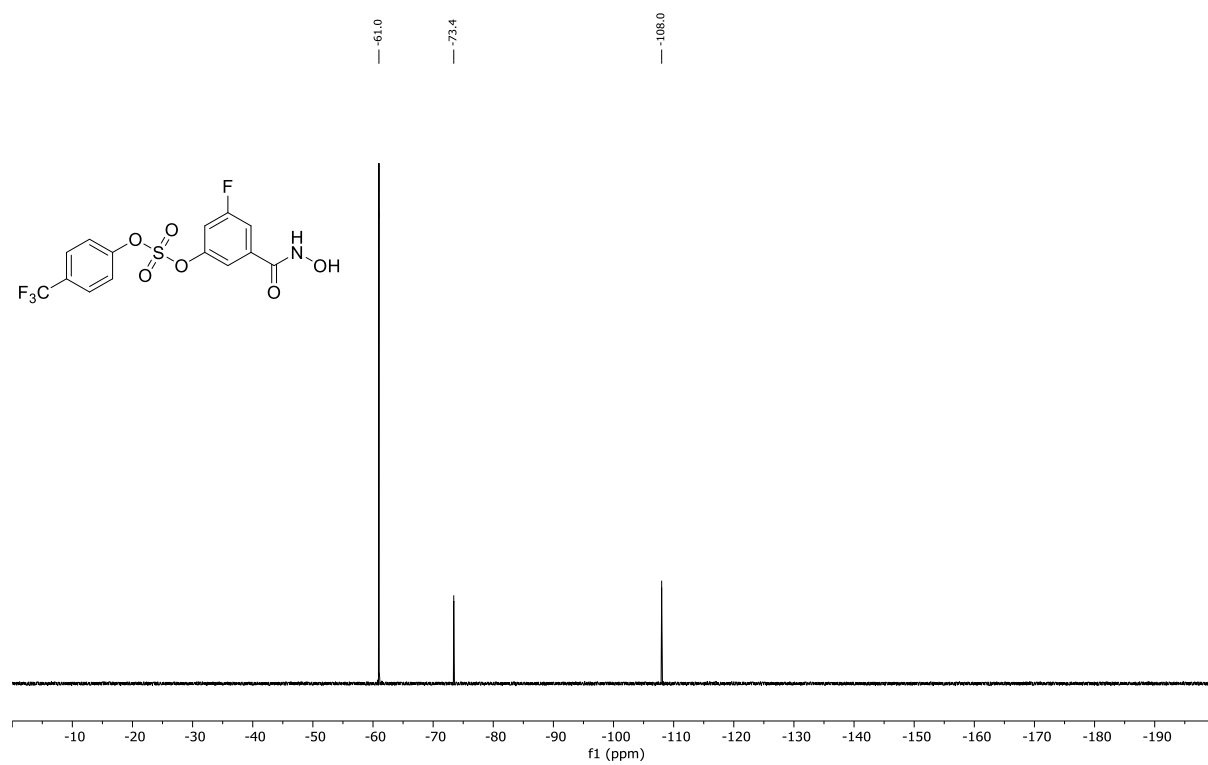
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C3**



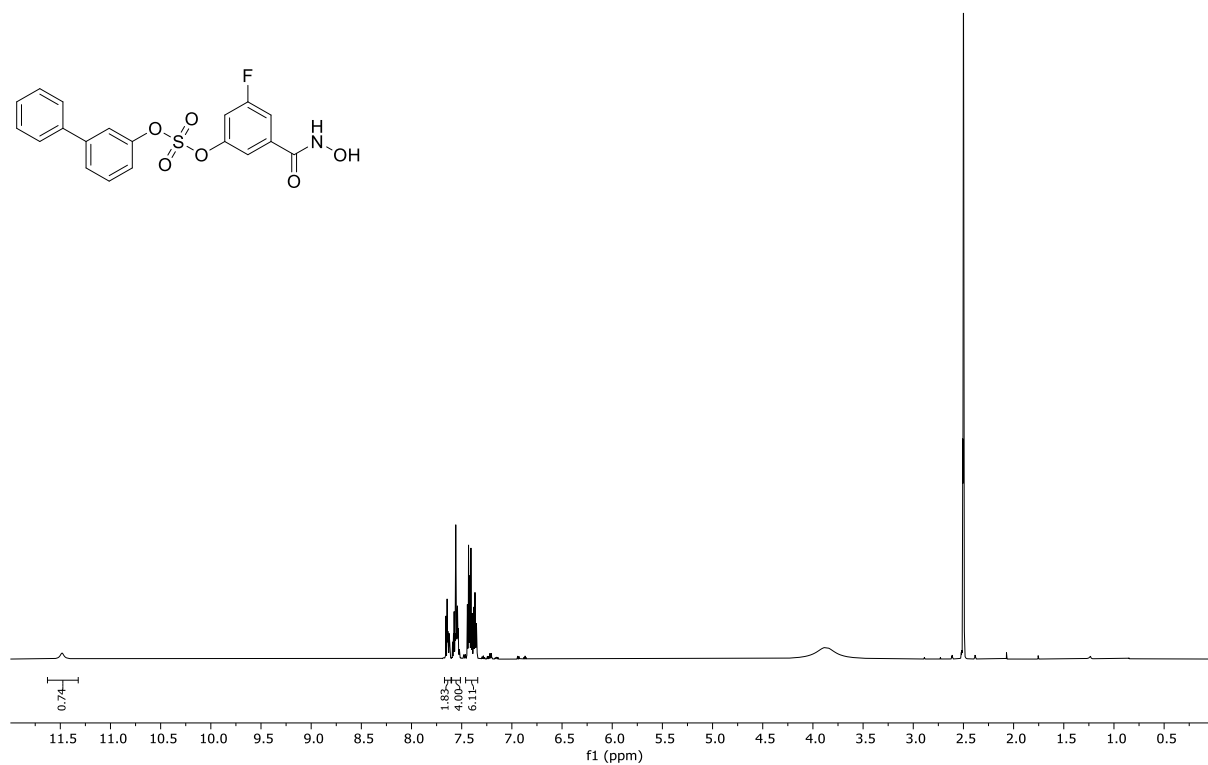
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C3**



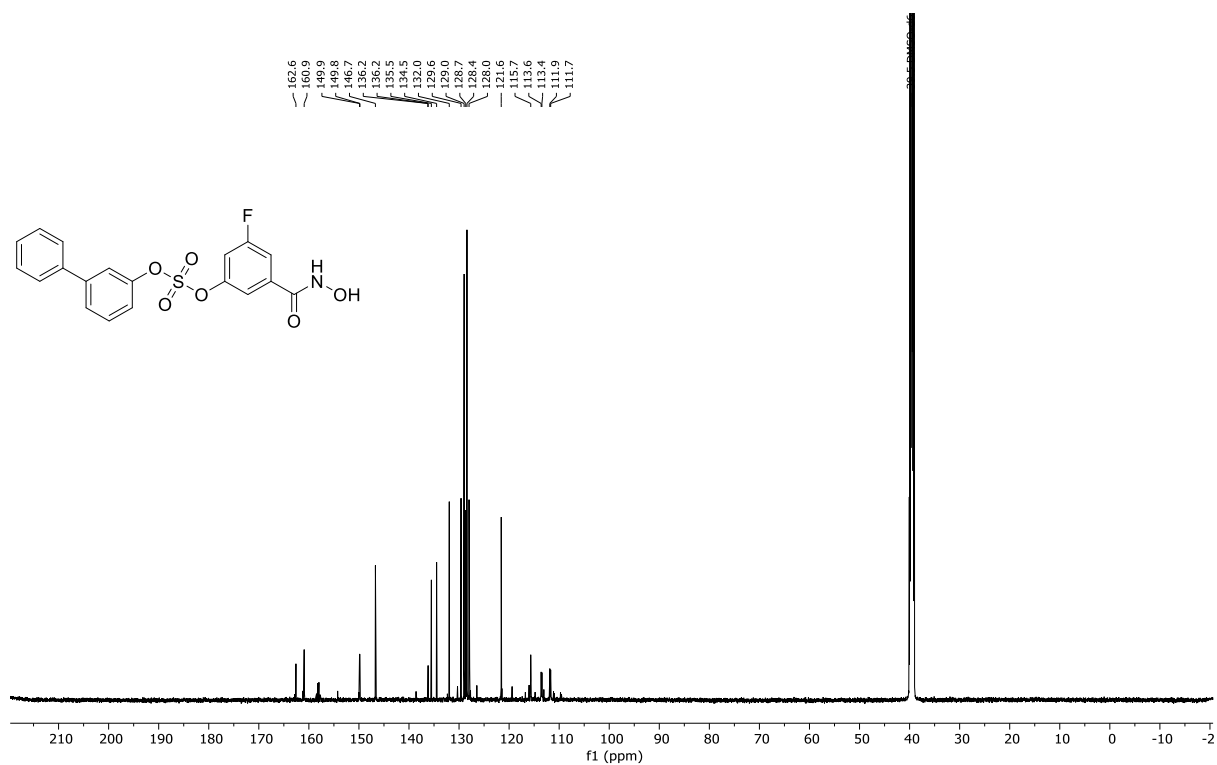
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C3**



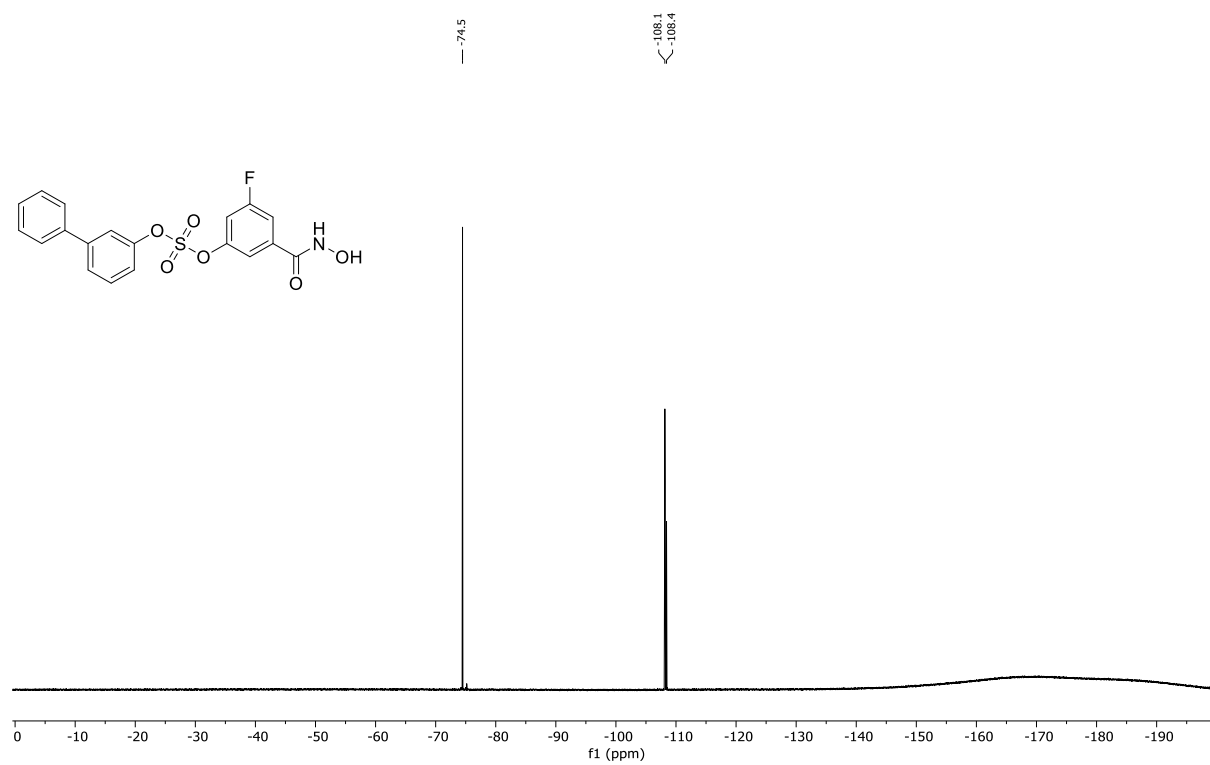
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C4**



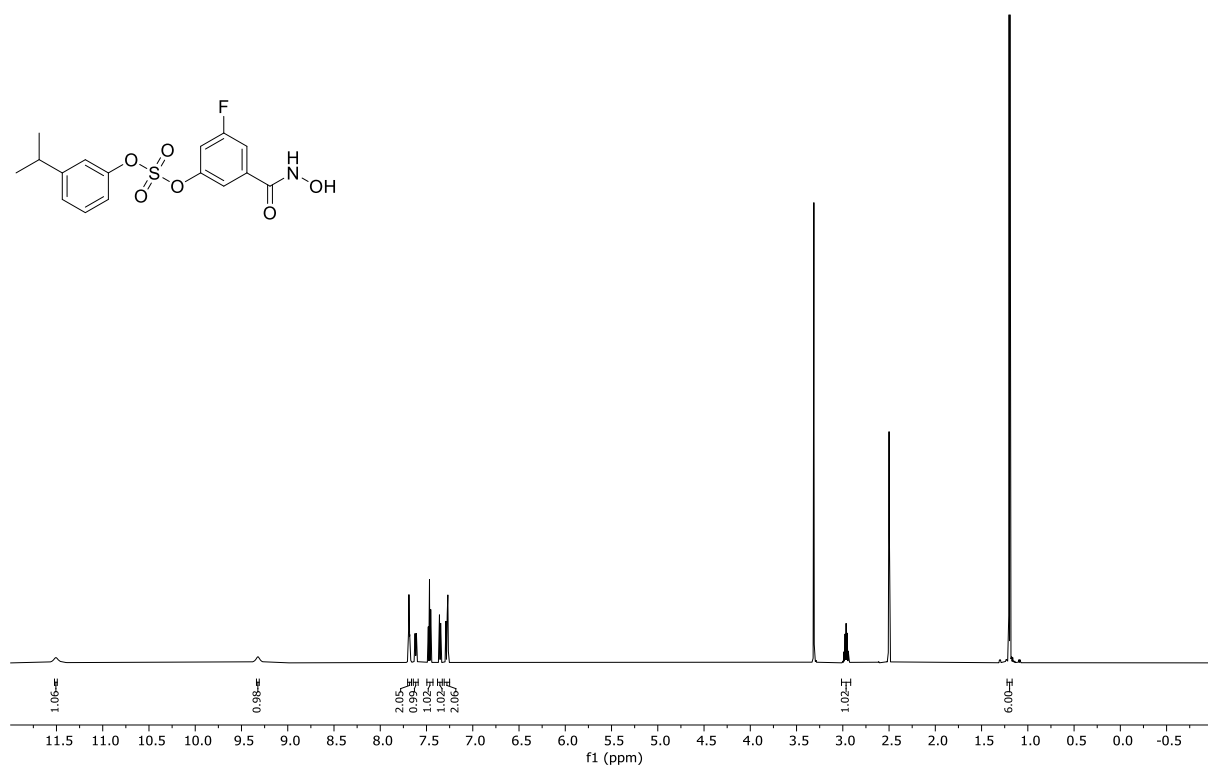
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C4** (minor contamination with TFA is observed at 158.8 ppm)



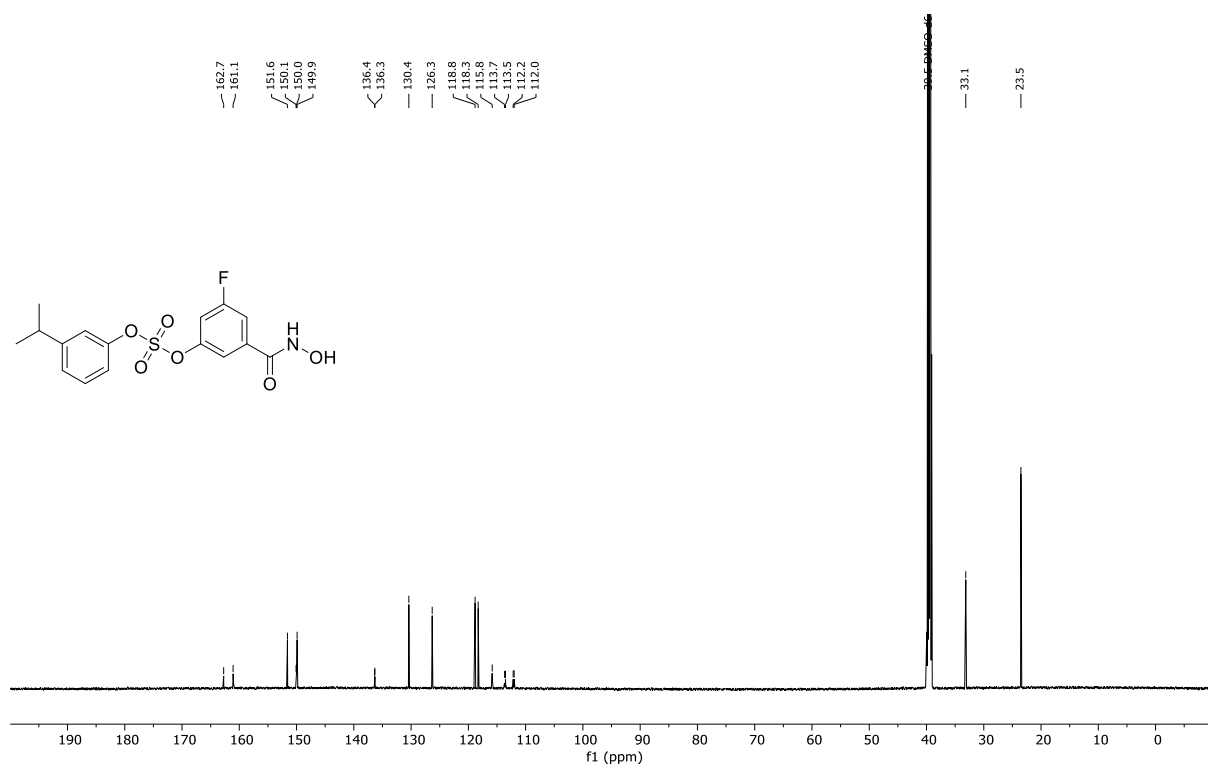
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C4**



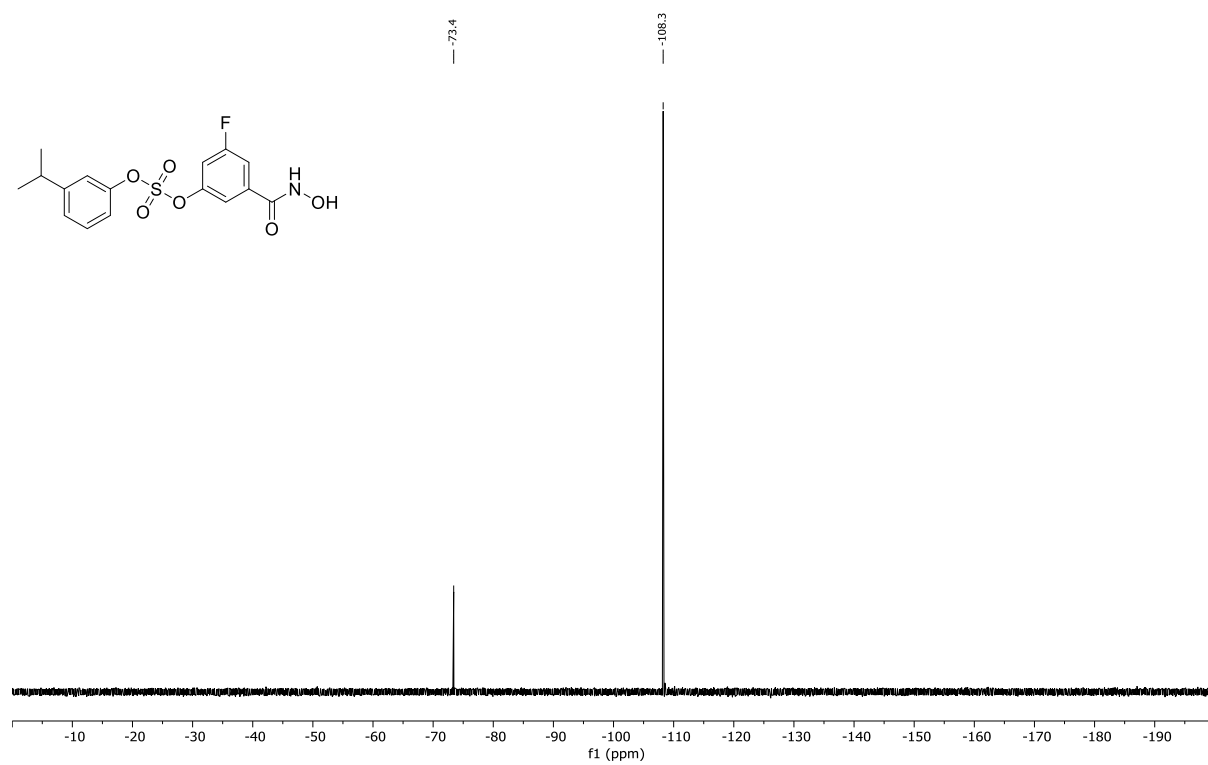
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C8**



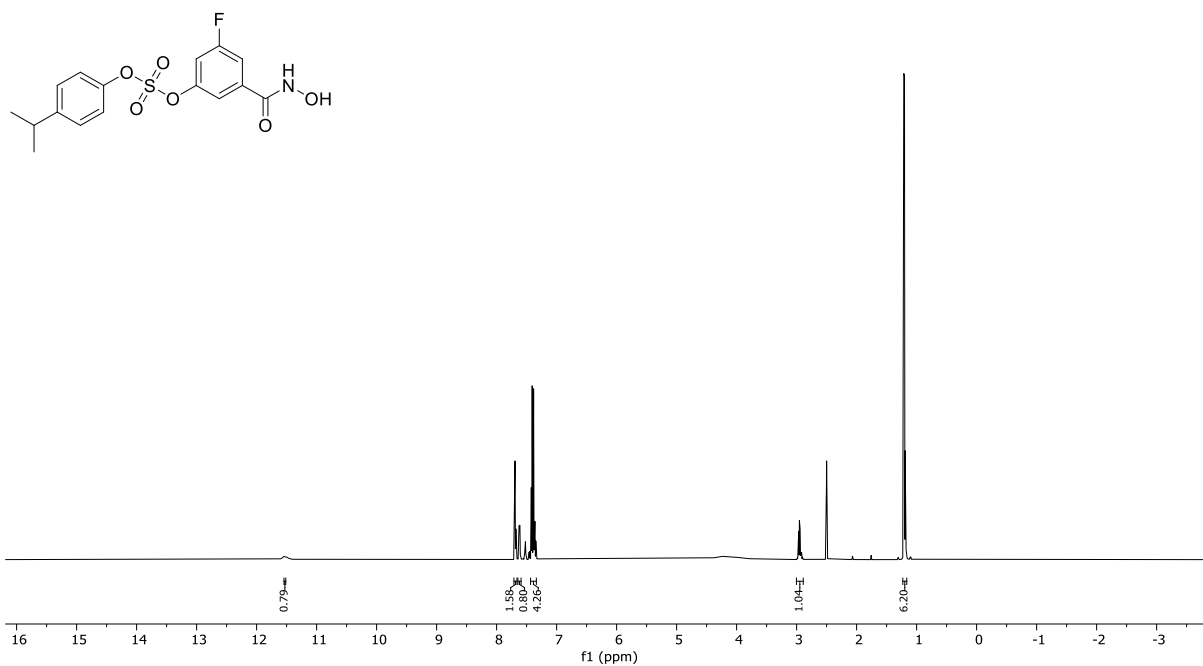
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C8**



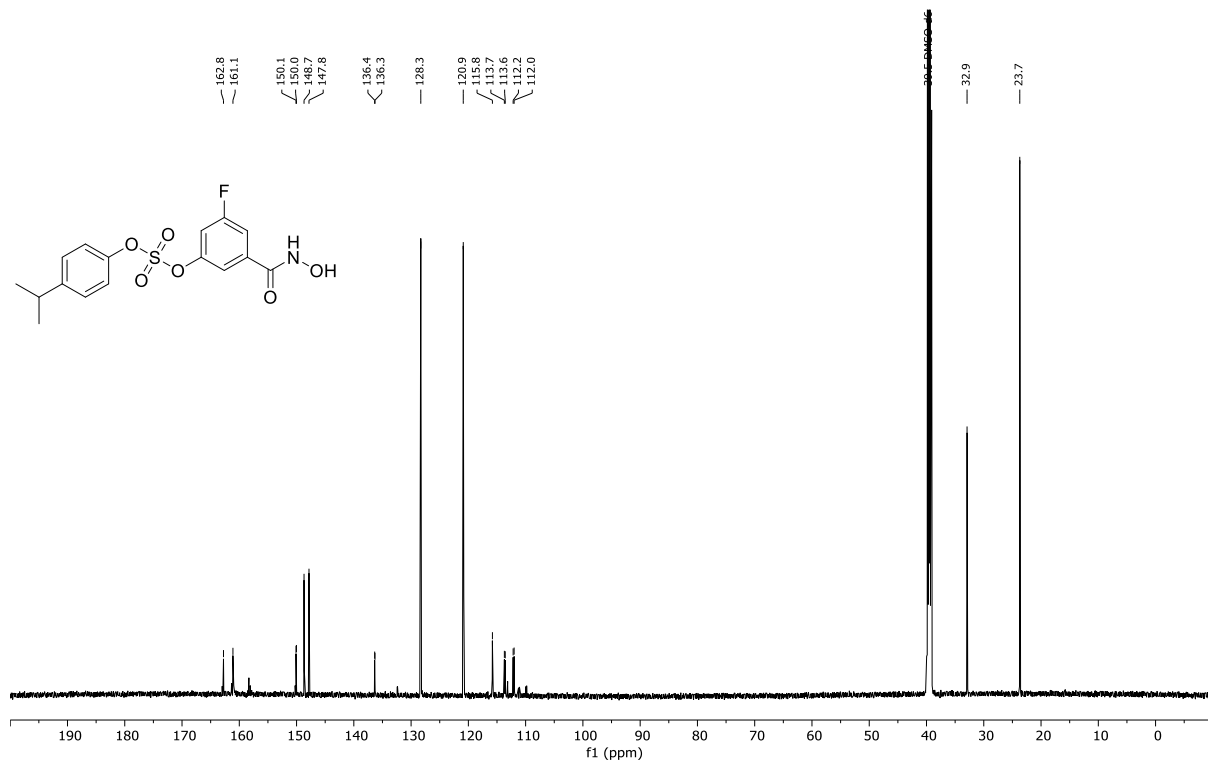
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C8**



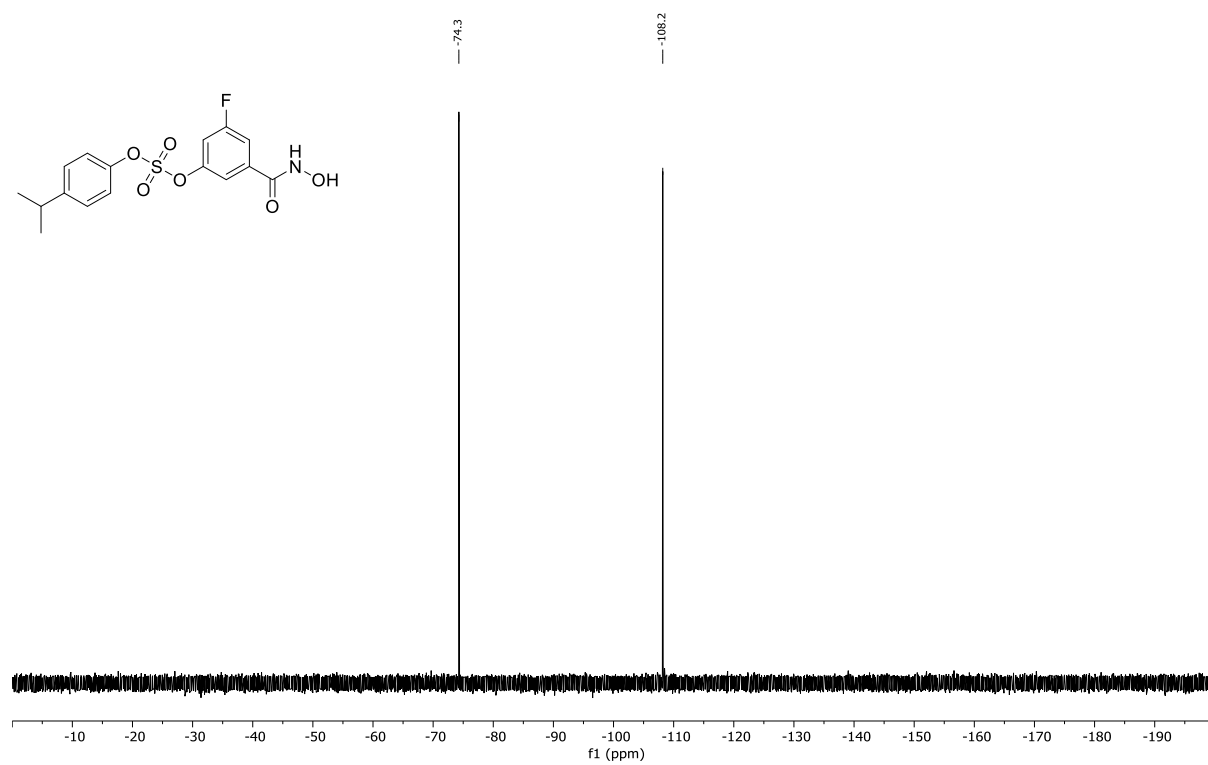
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C9**



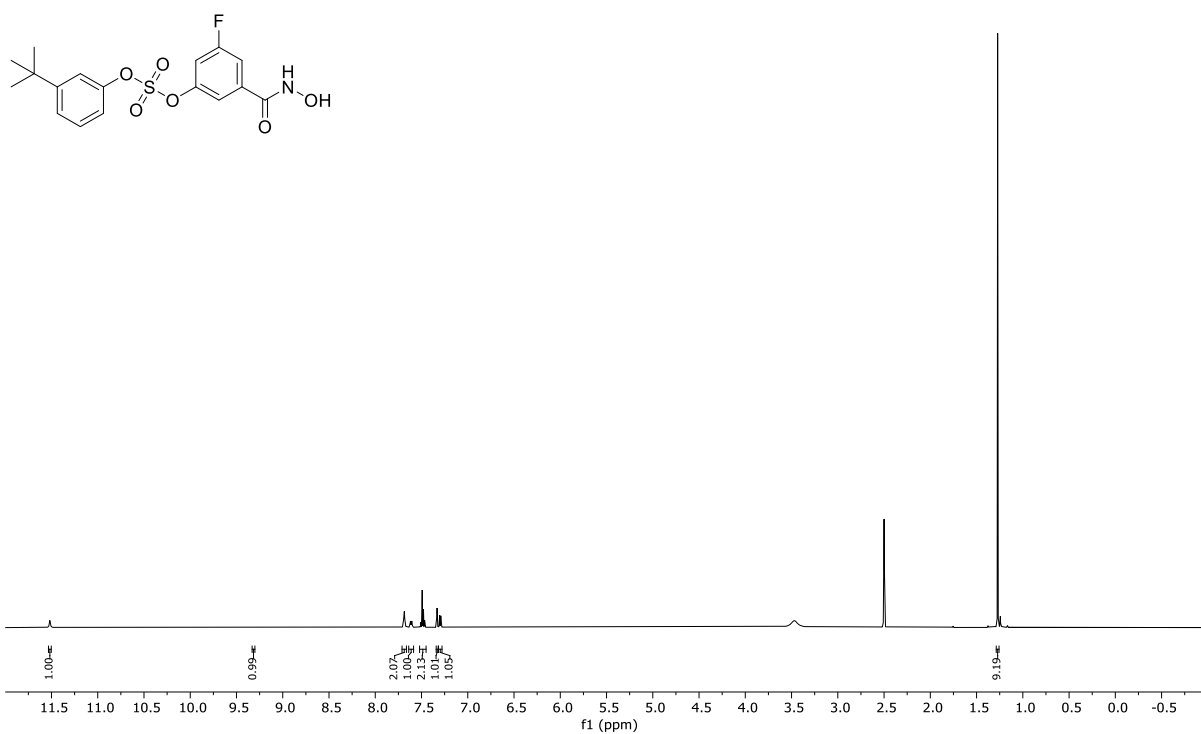
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C9** (minor contamination with TFA is observed at 158.8 ppm)



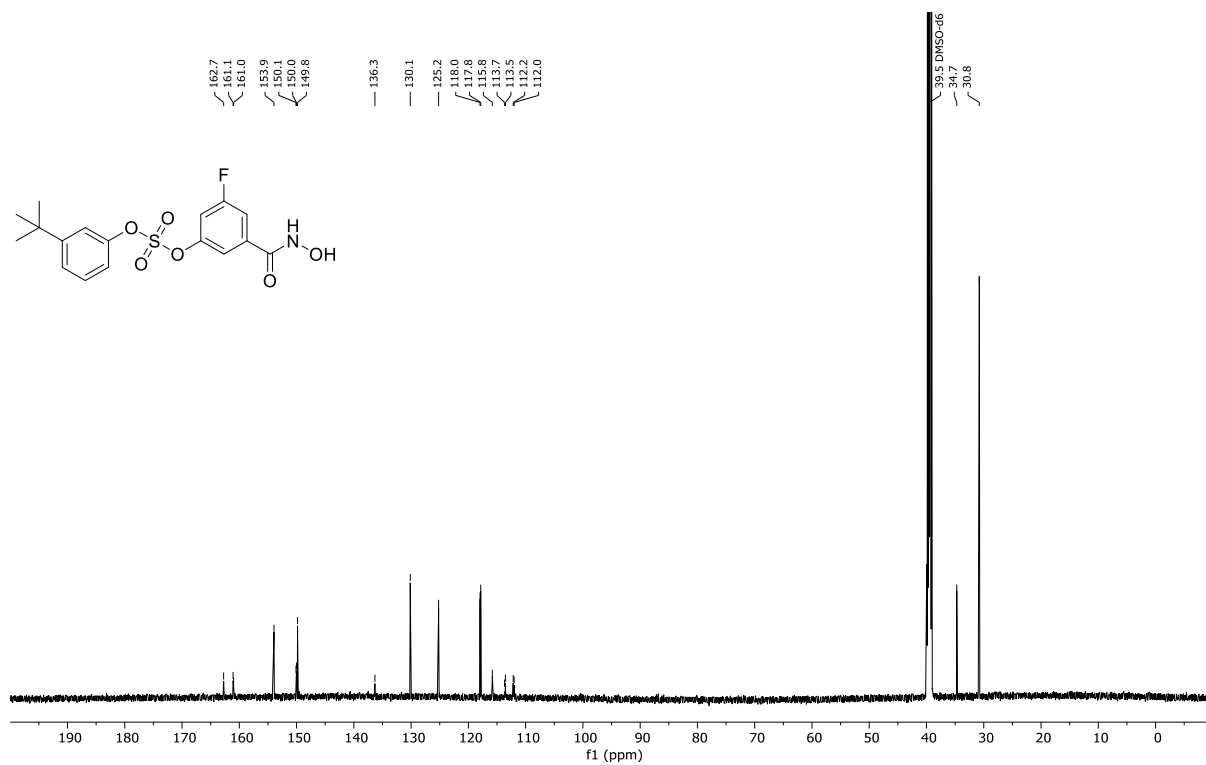
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C9**



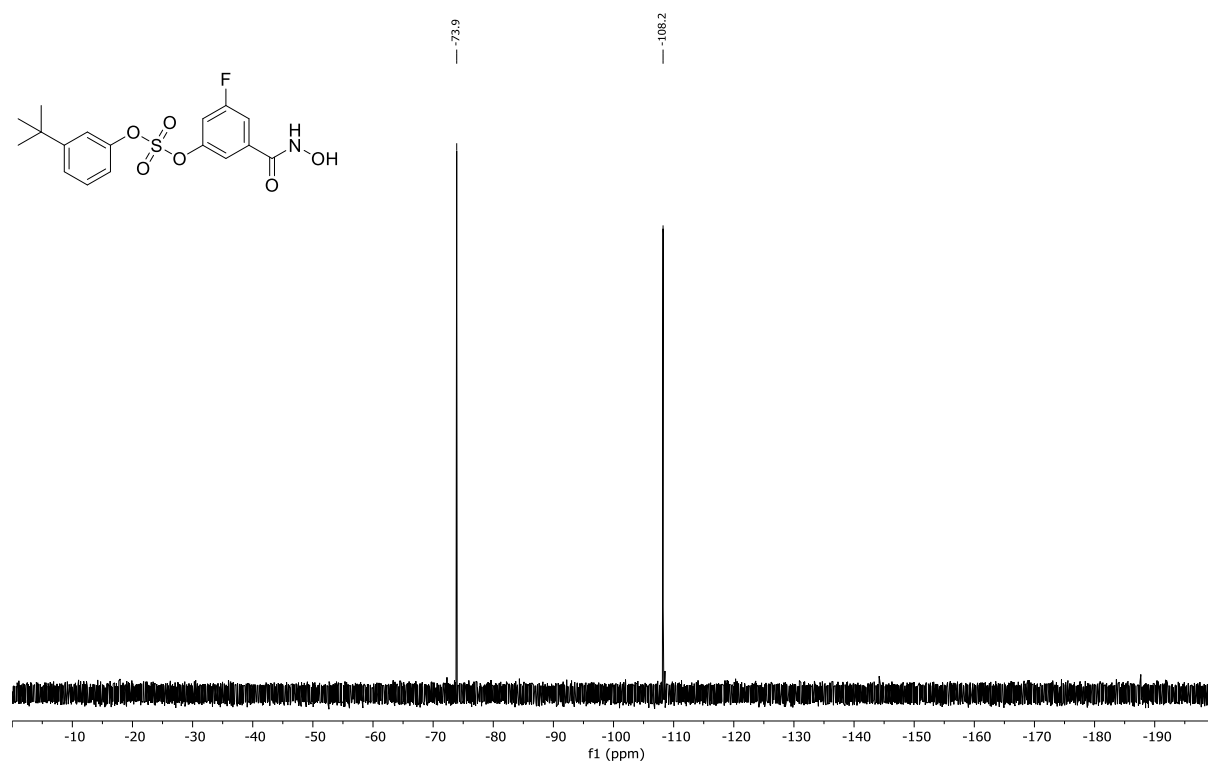
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C11**



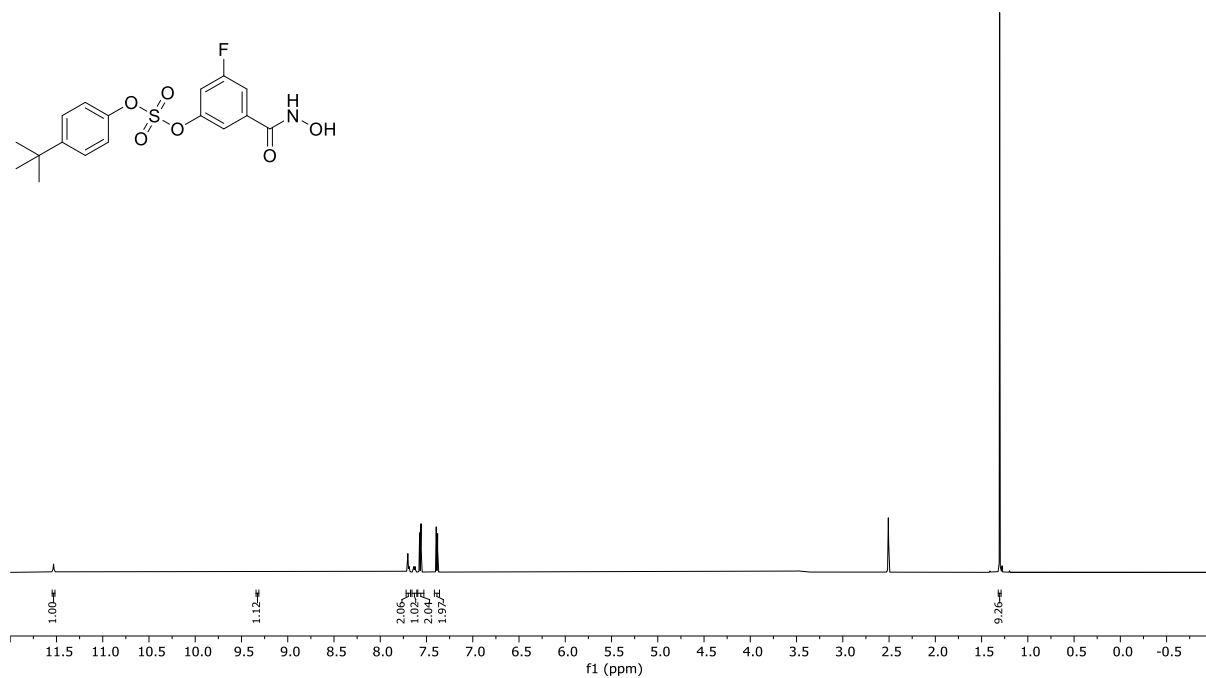
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C11**



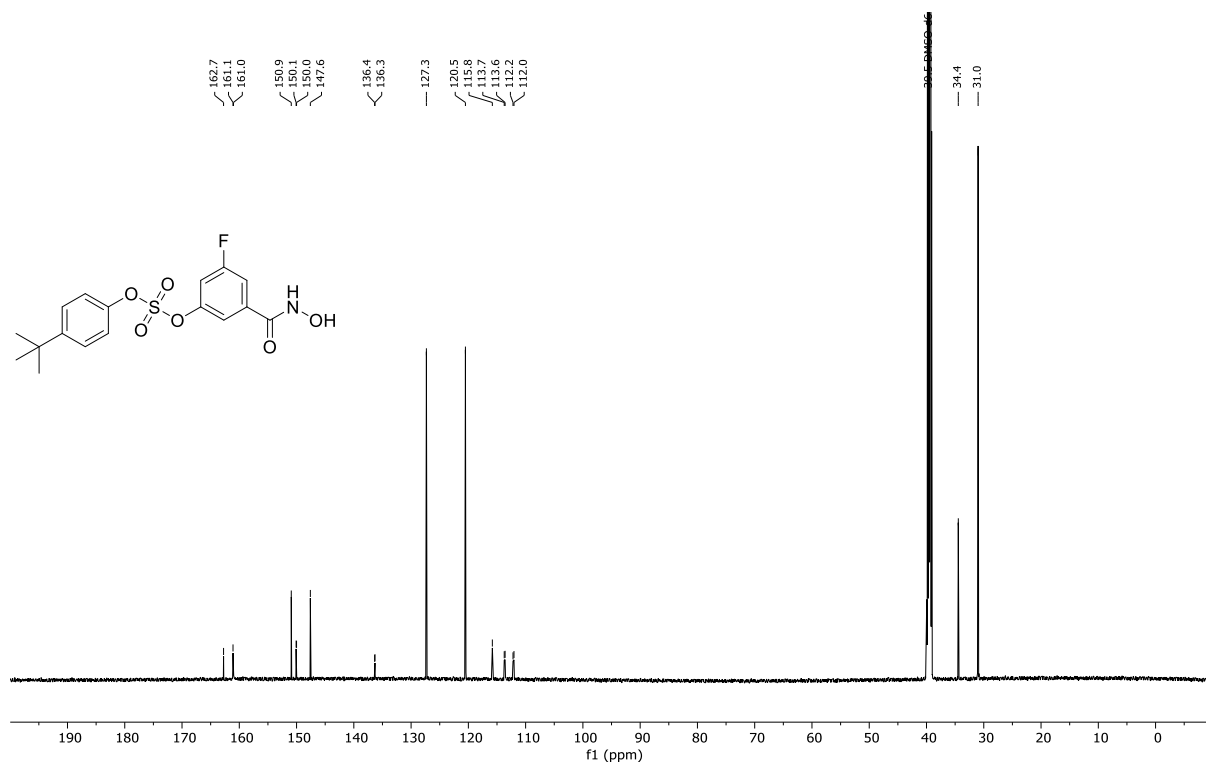
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C11**



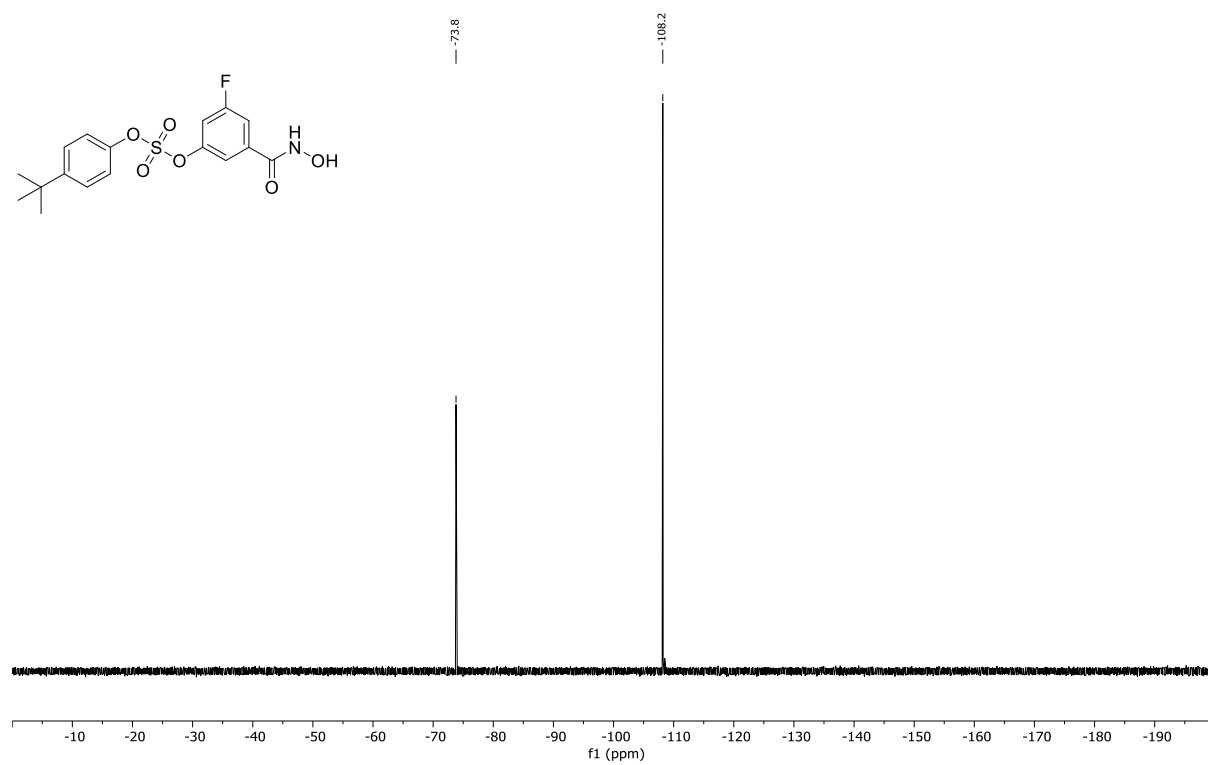
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C12**



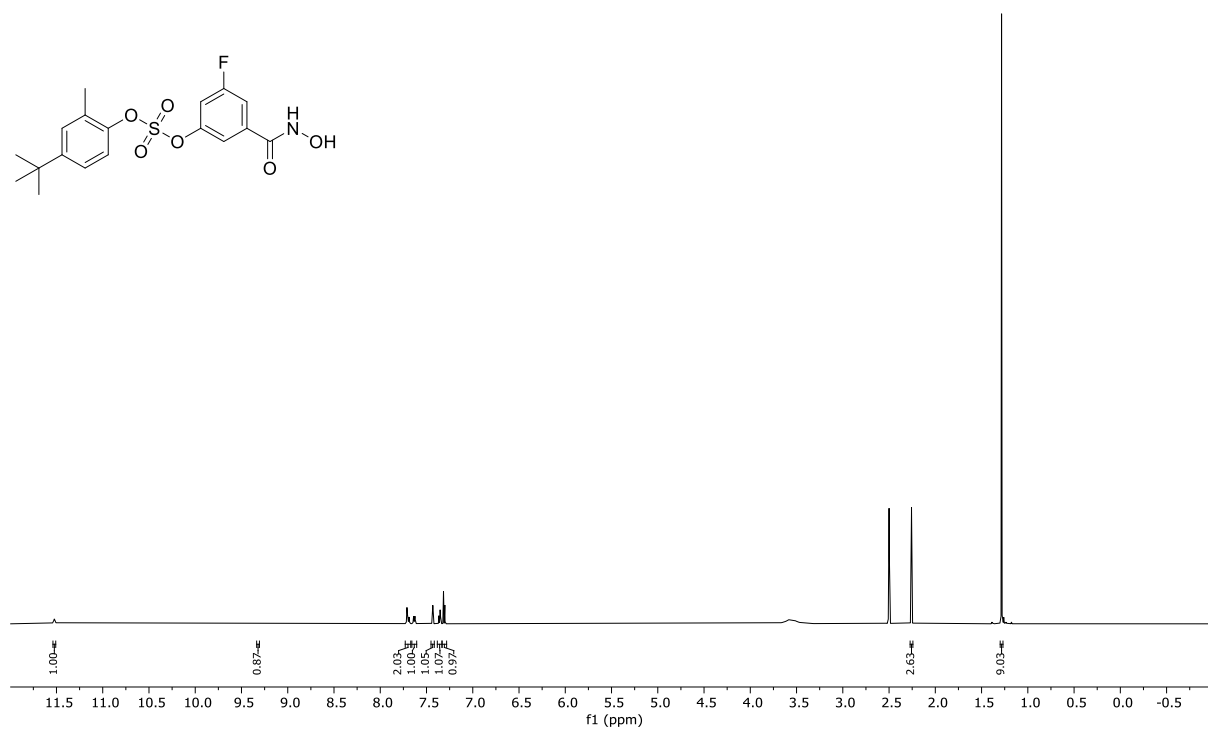
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C12**



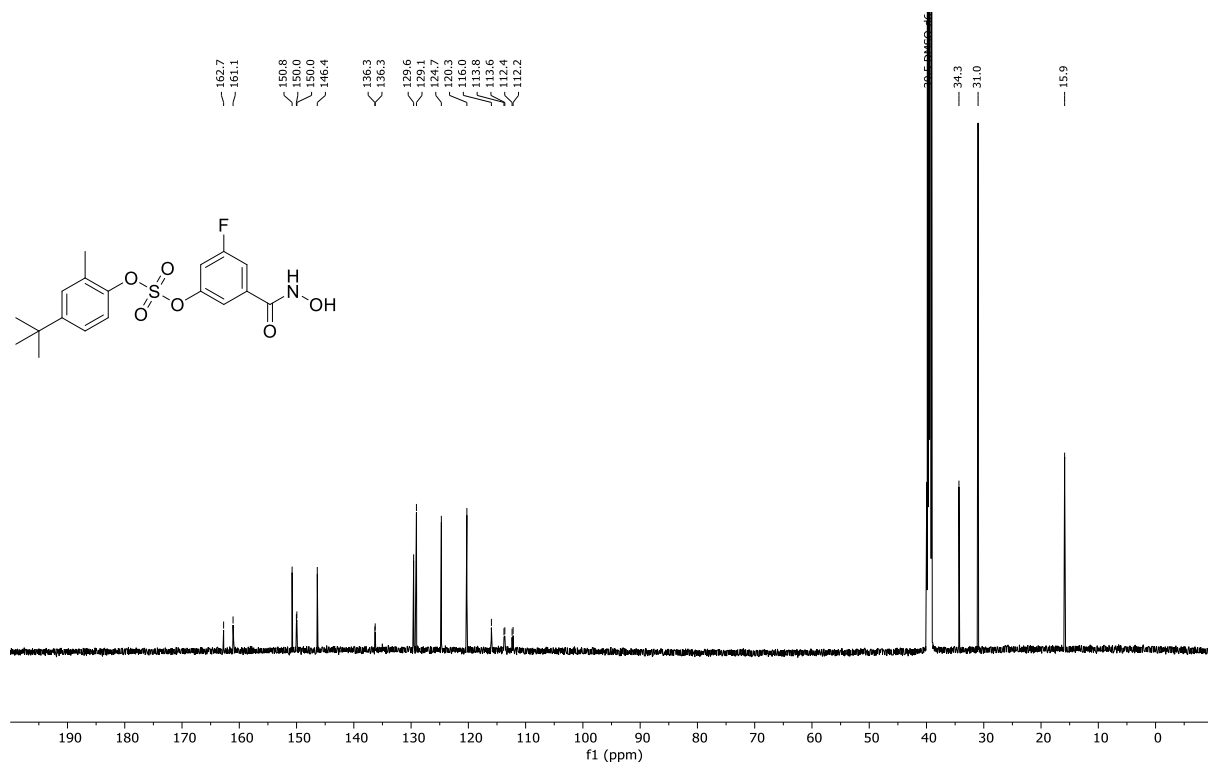
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C12**



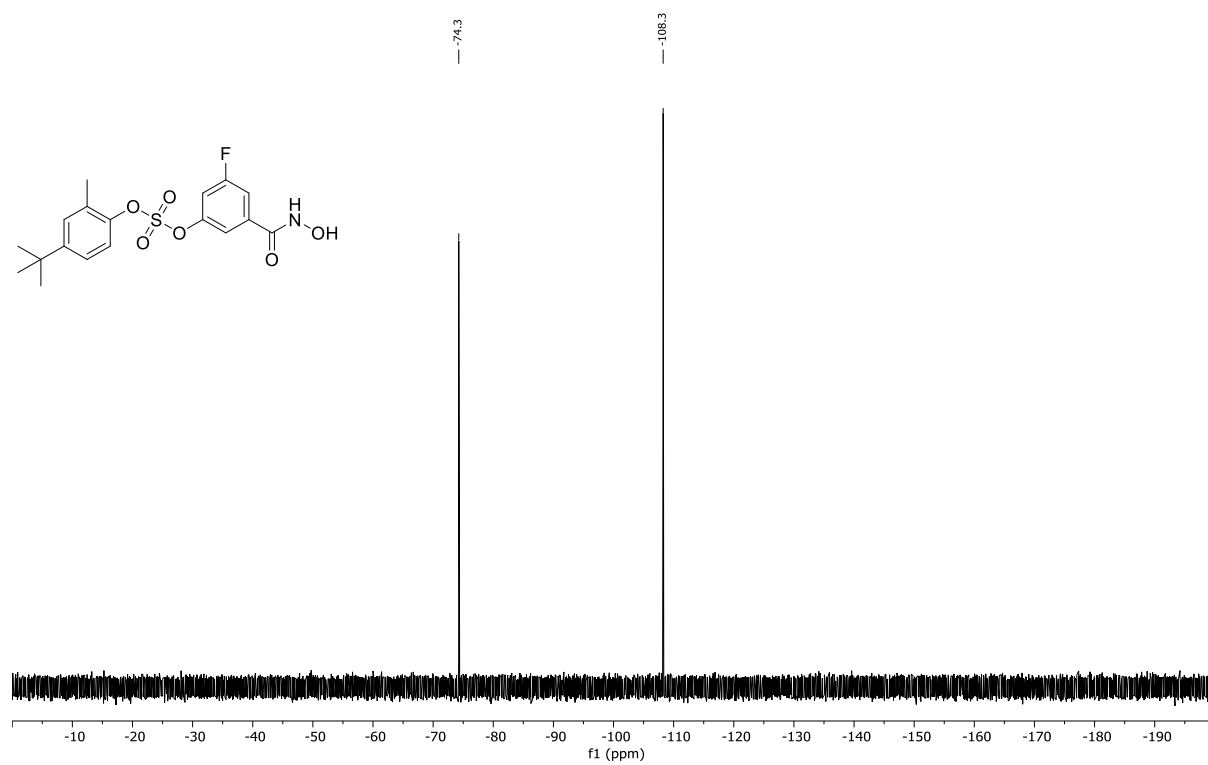
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C19**



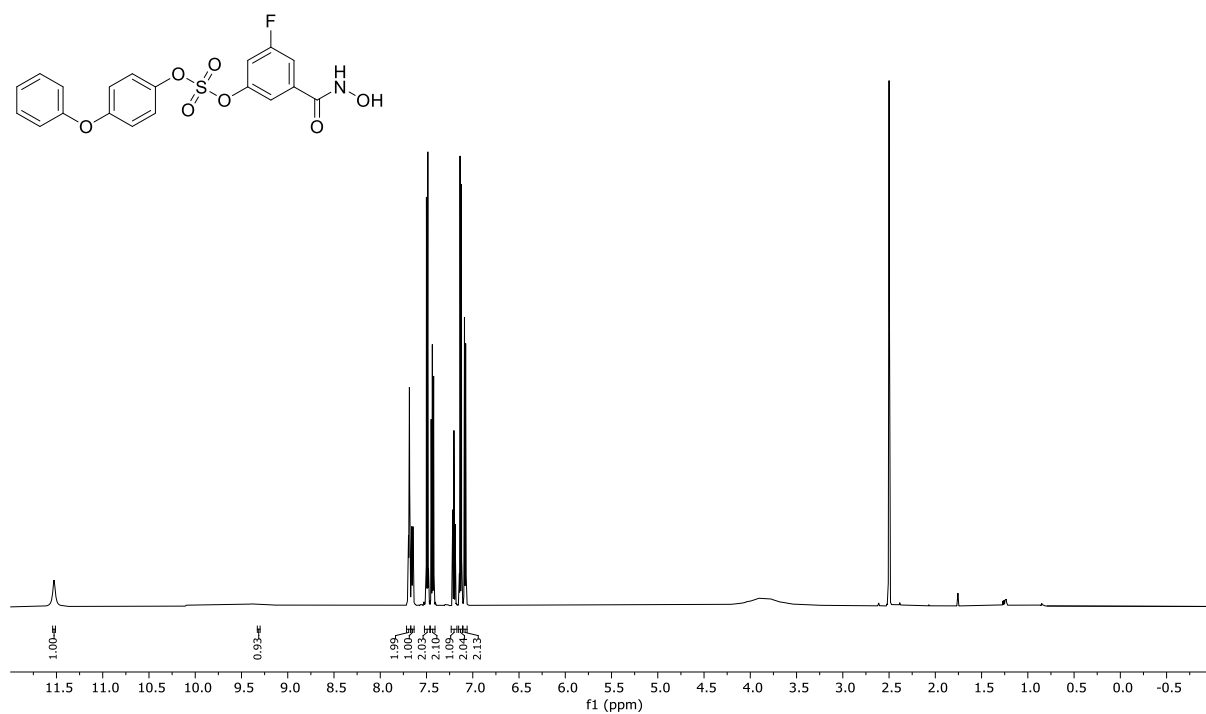
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C19**



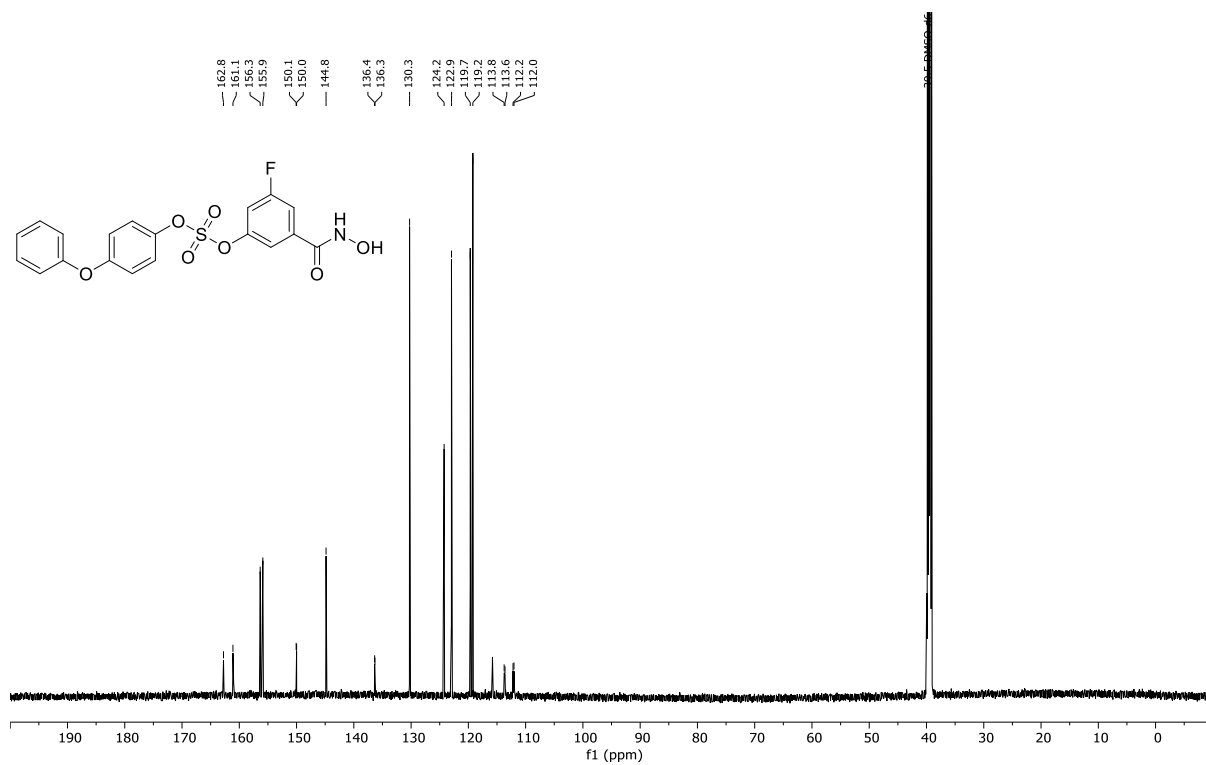
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C19**



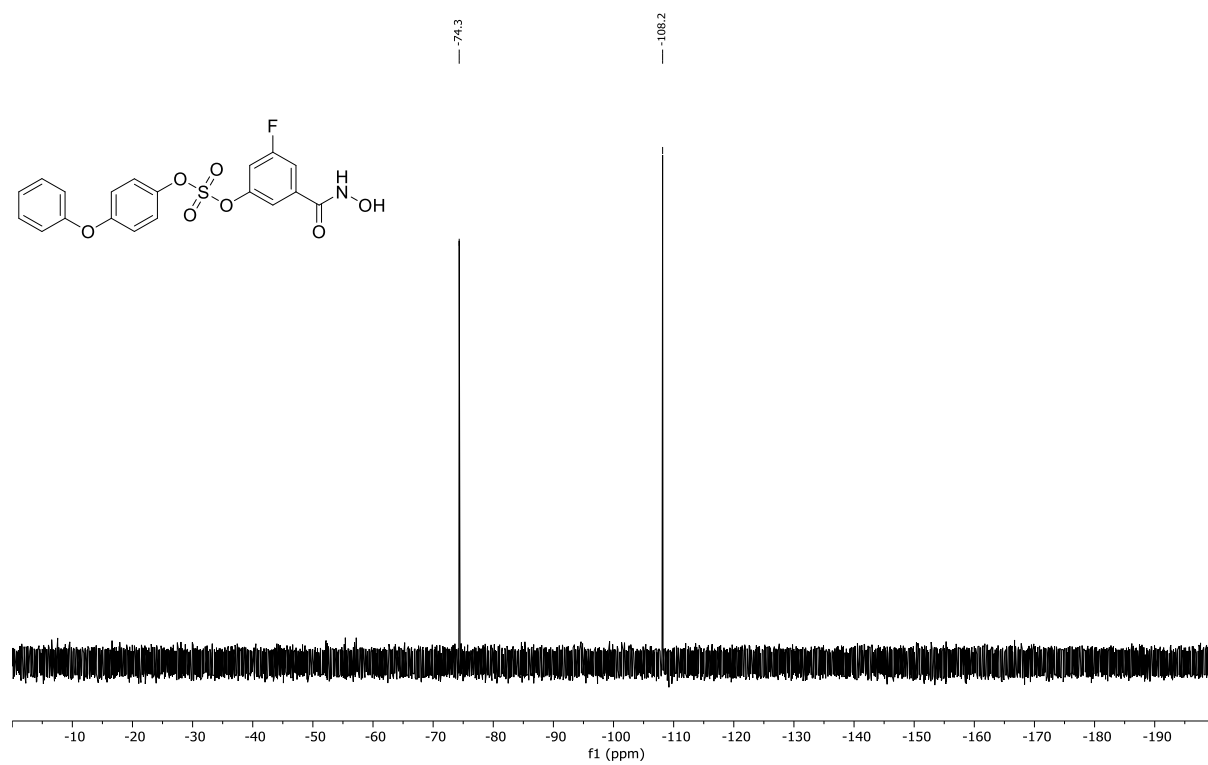
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C21**



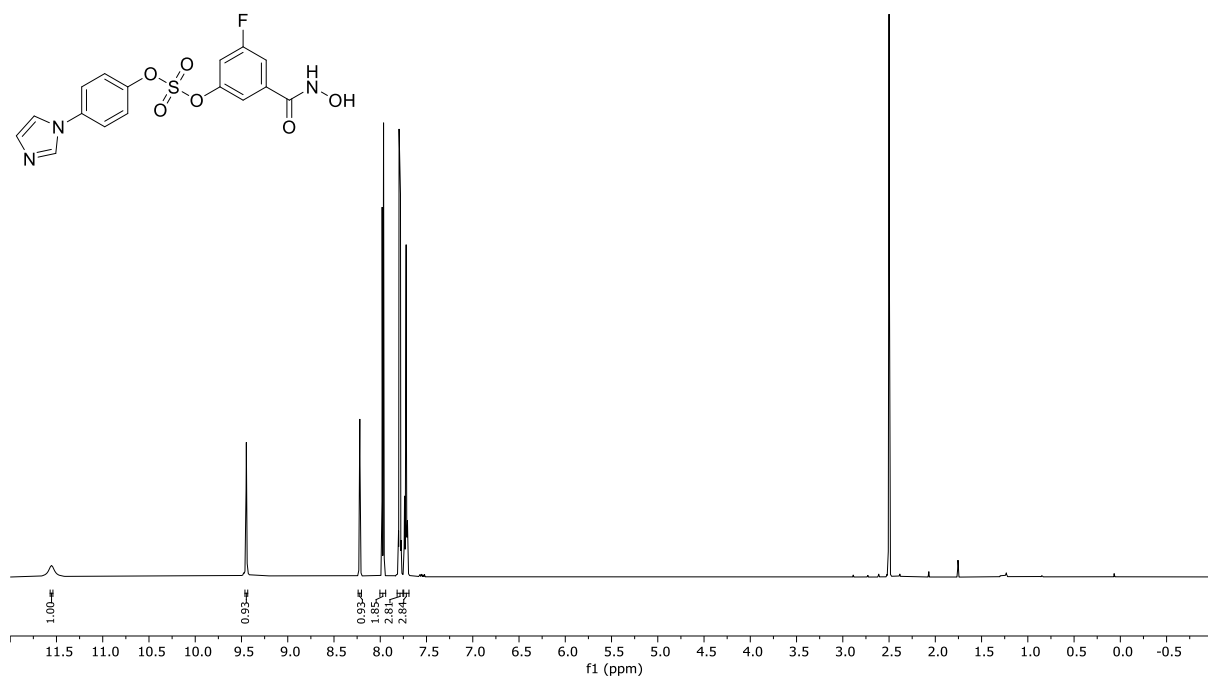
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C21**



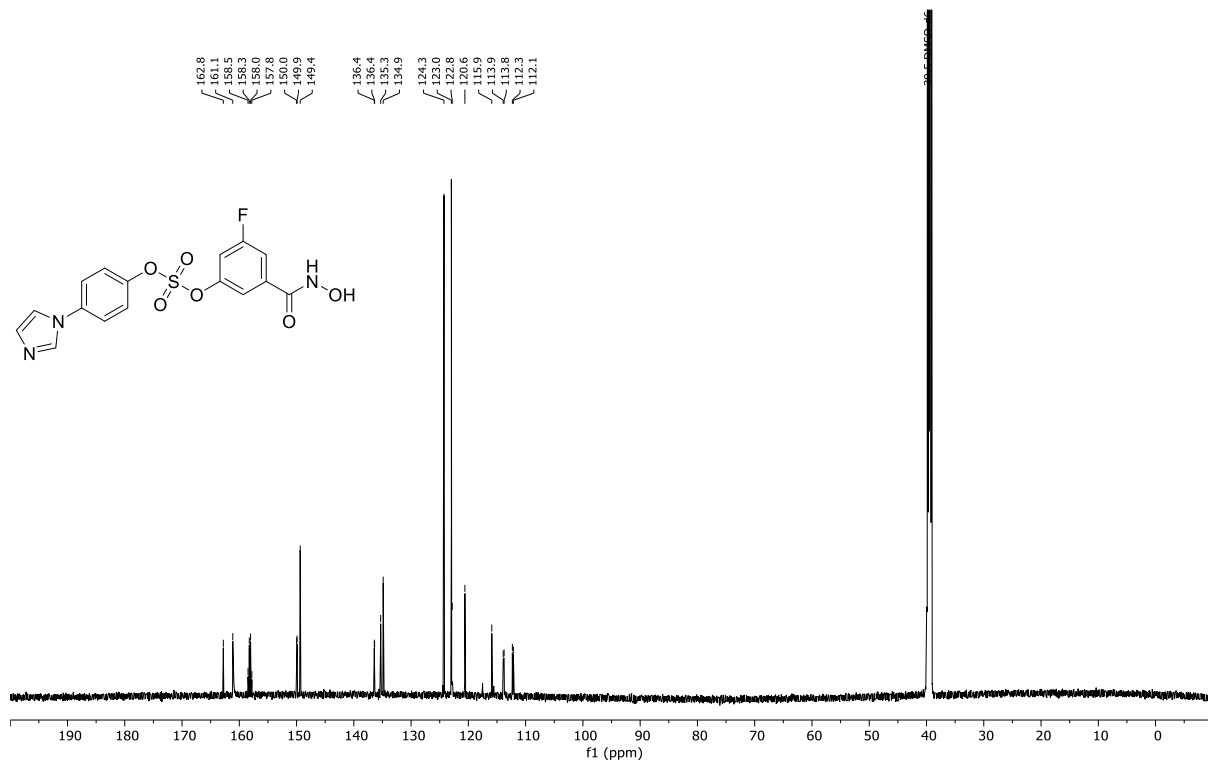
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C21**



¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C23**



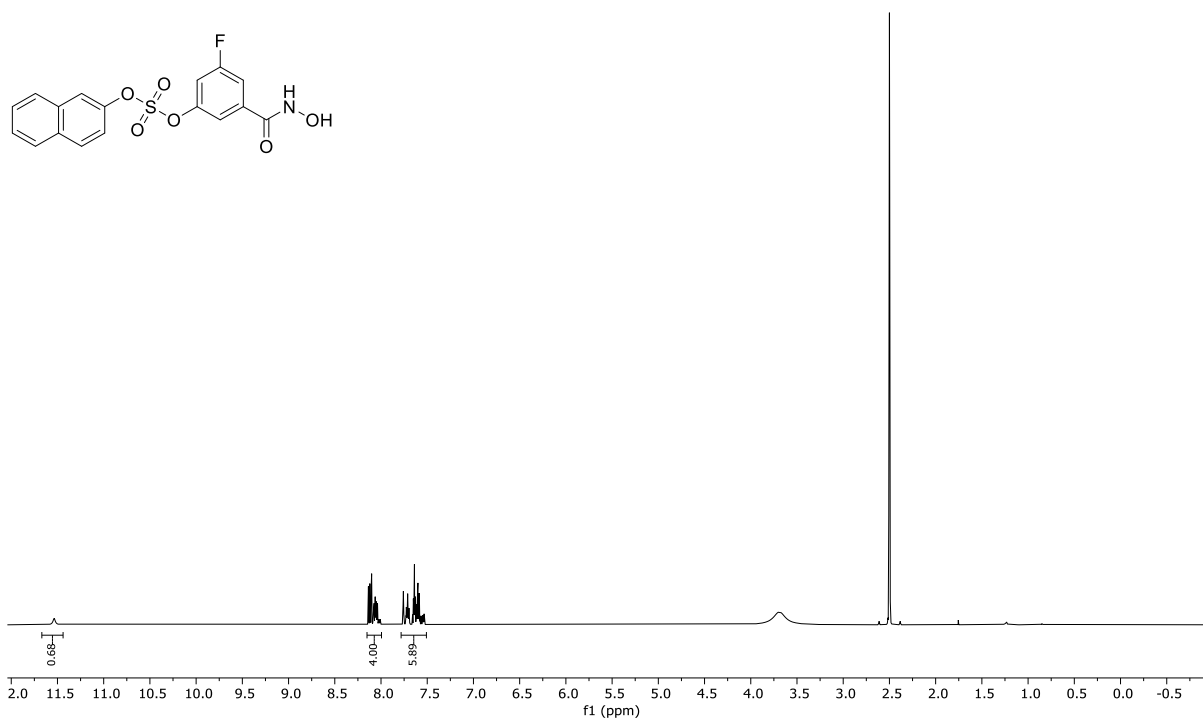
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C23** (minor contamination with TFA is observed at 158.8 ppm)



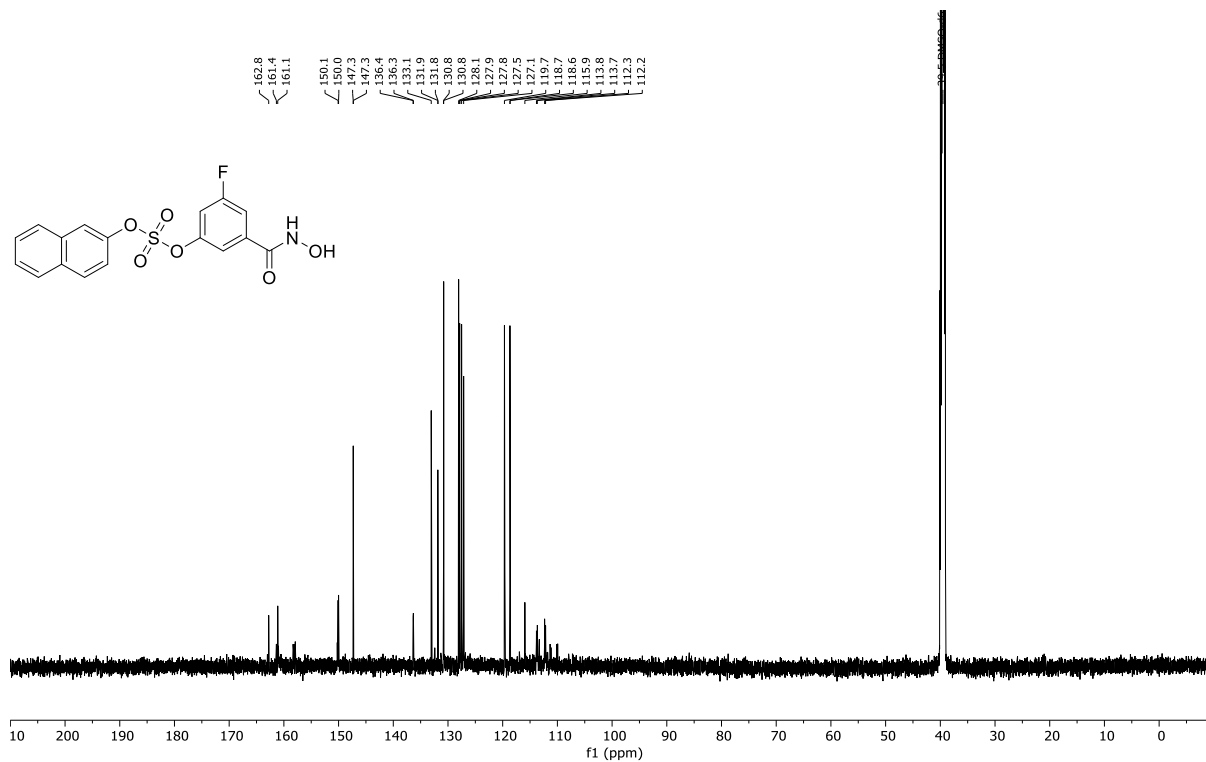
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C23**



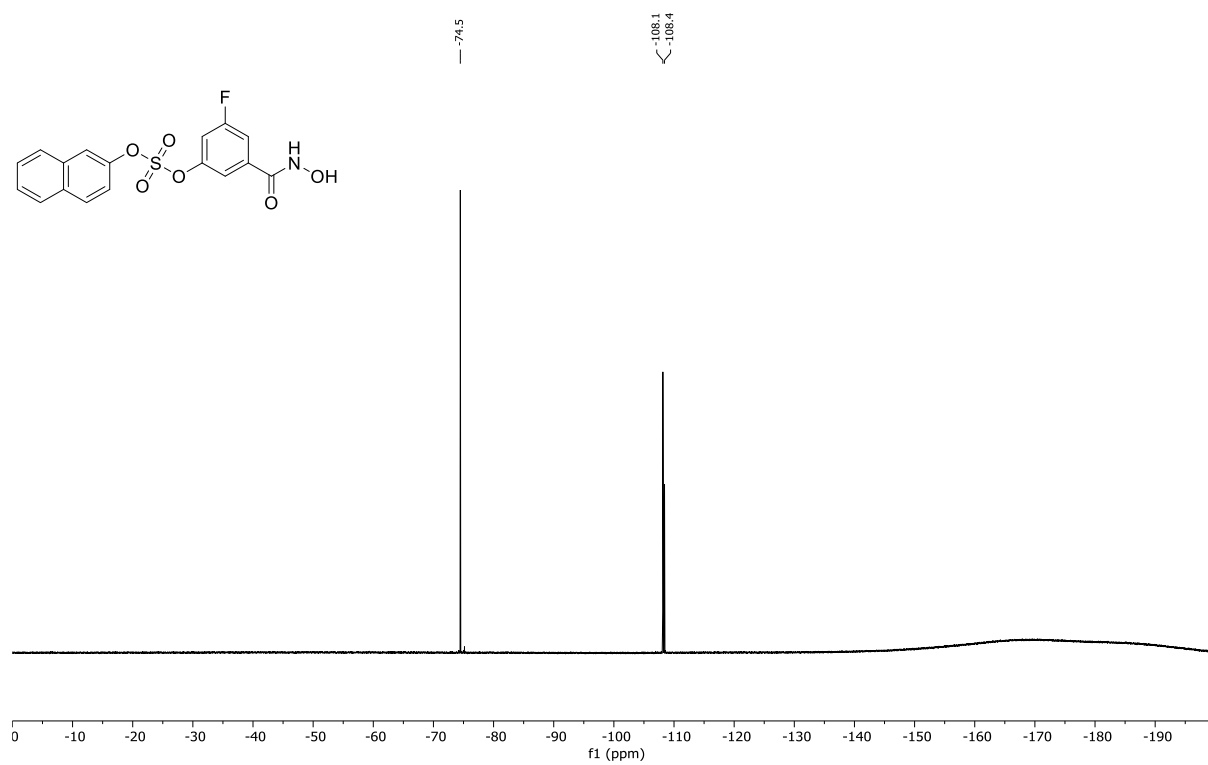
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **C24**



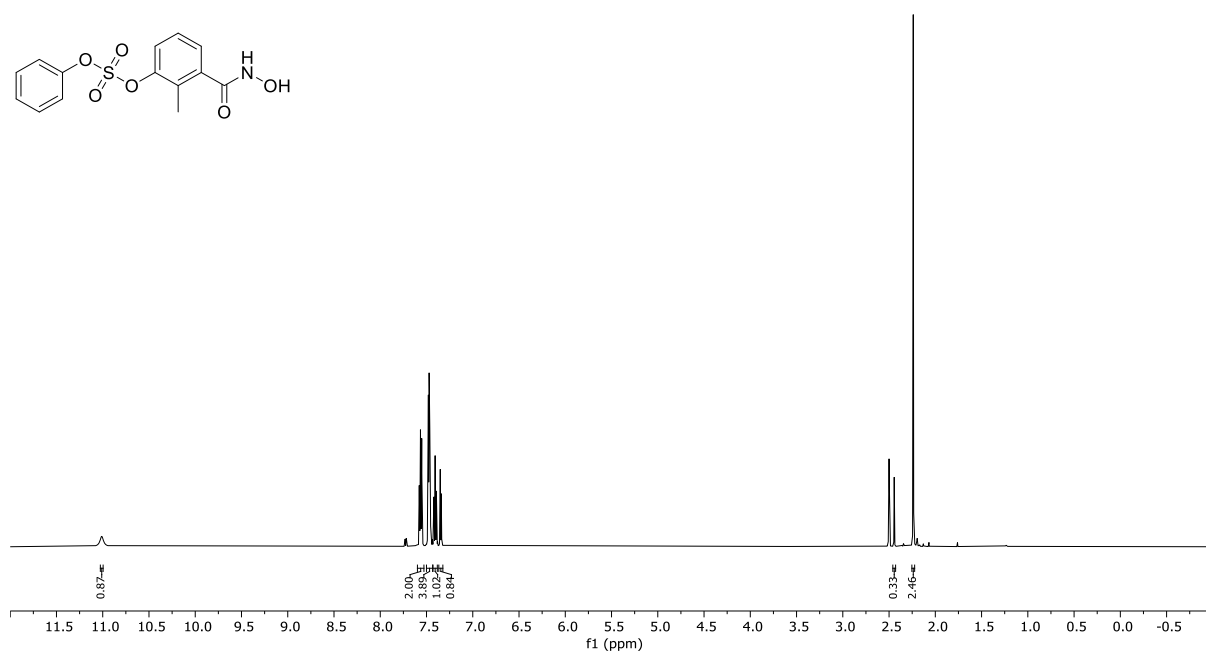
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **C24** (minor contamination with TFA is observed at 158.8 ppm)



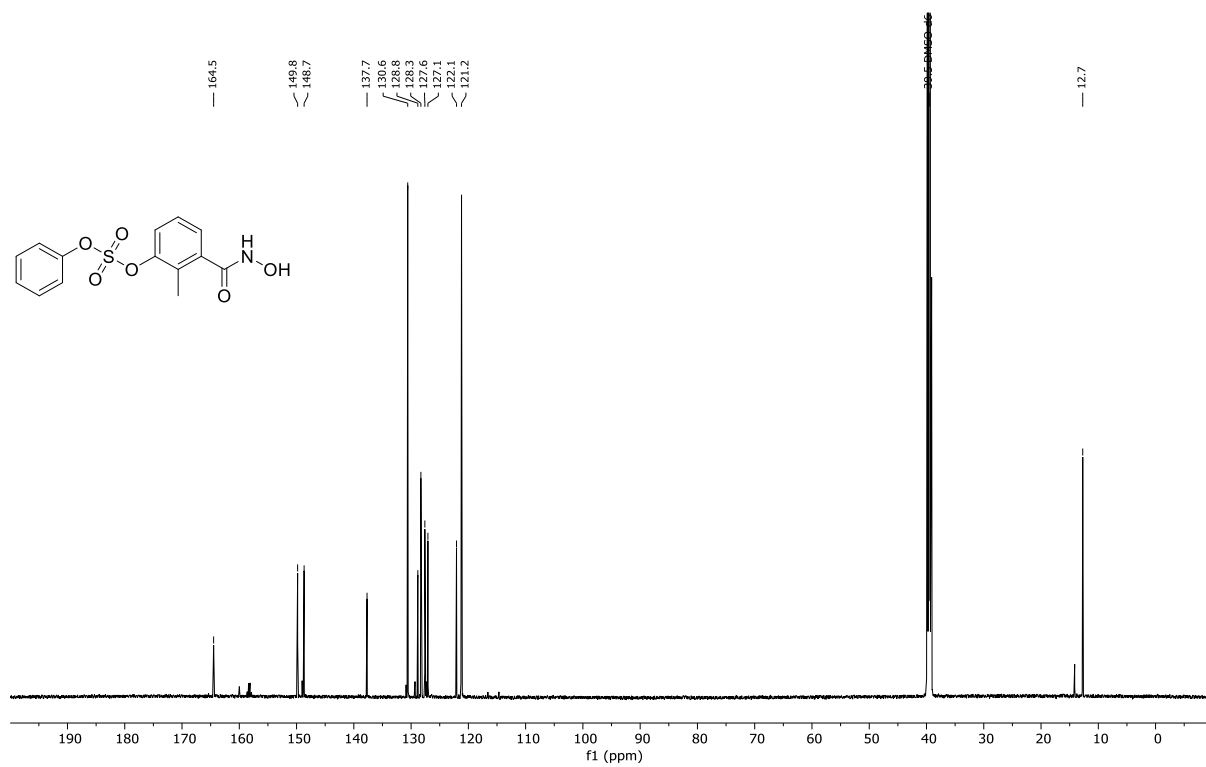
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **C24**



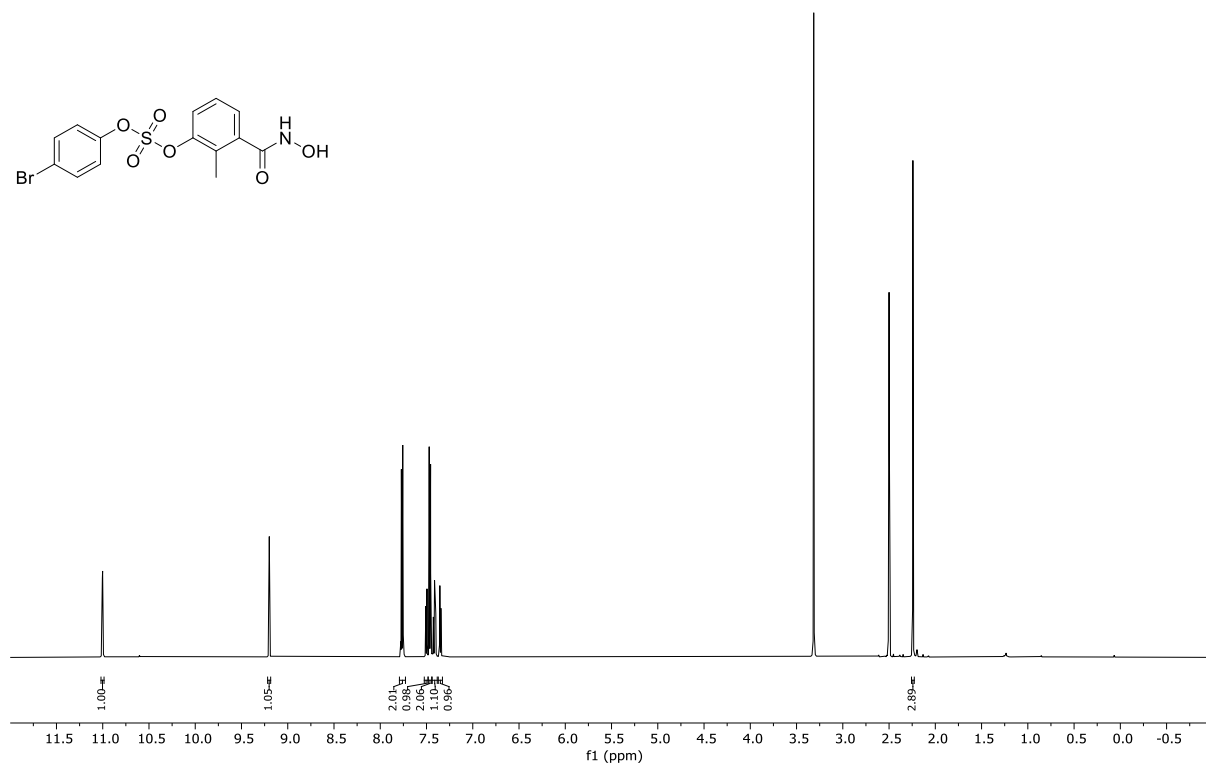
^1H NMR spectra (600 MHz, $\text{DMSO-}d_6$) of compound **D1**



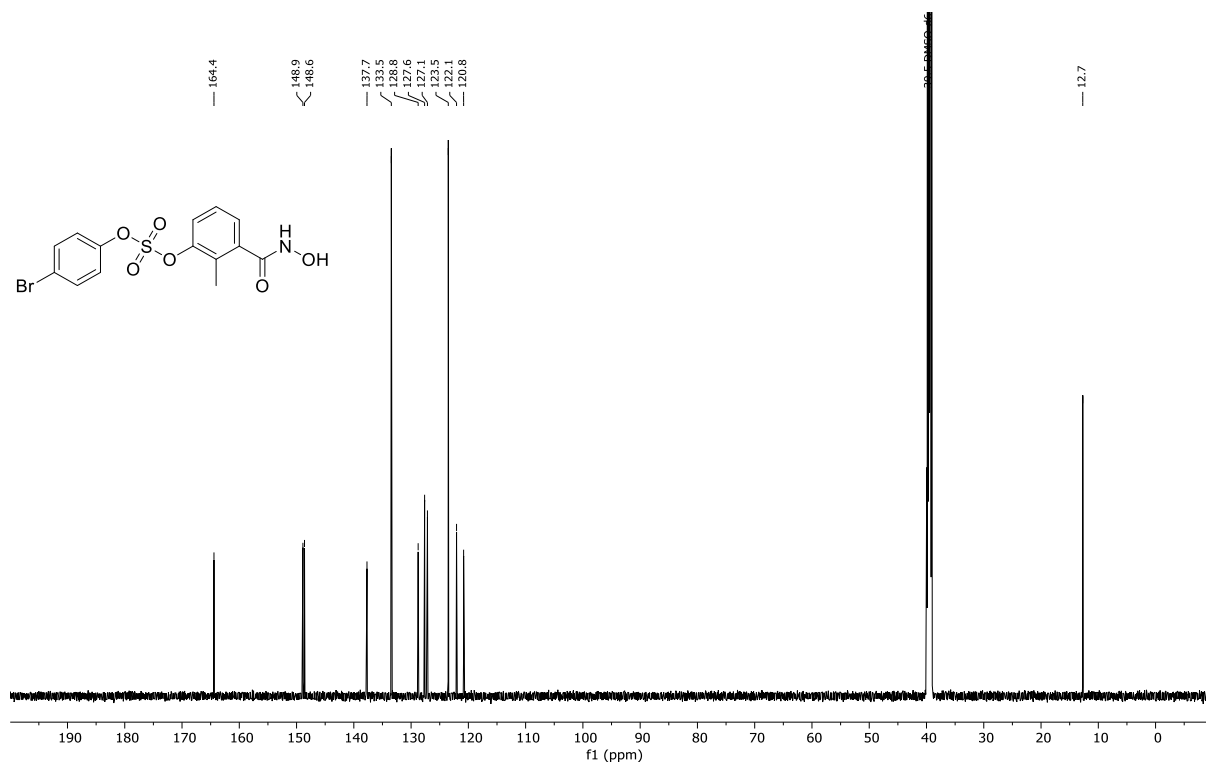
^{13}C NMR spectra (151 MHz, $\text{DMSO-}d_6$) of compound **D1** (minor contamination with TFA is observed at 158.8 ppm)



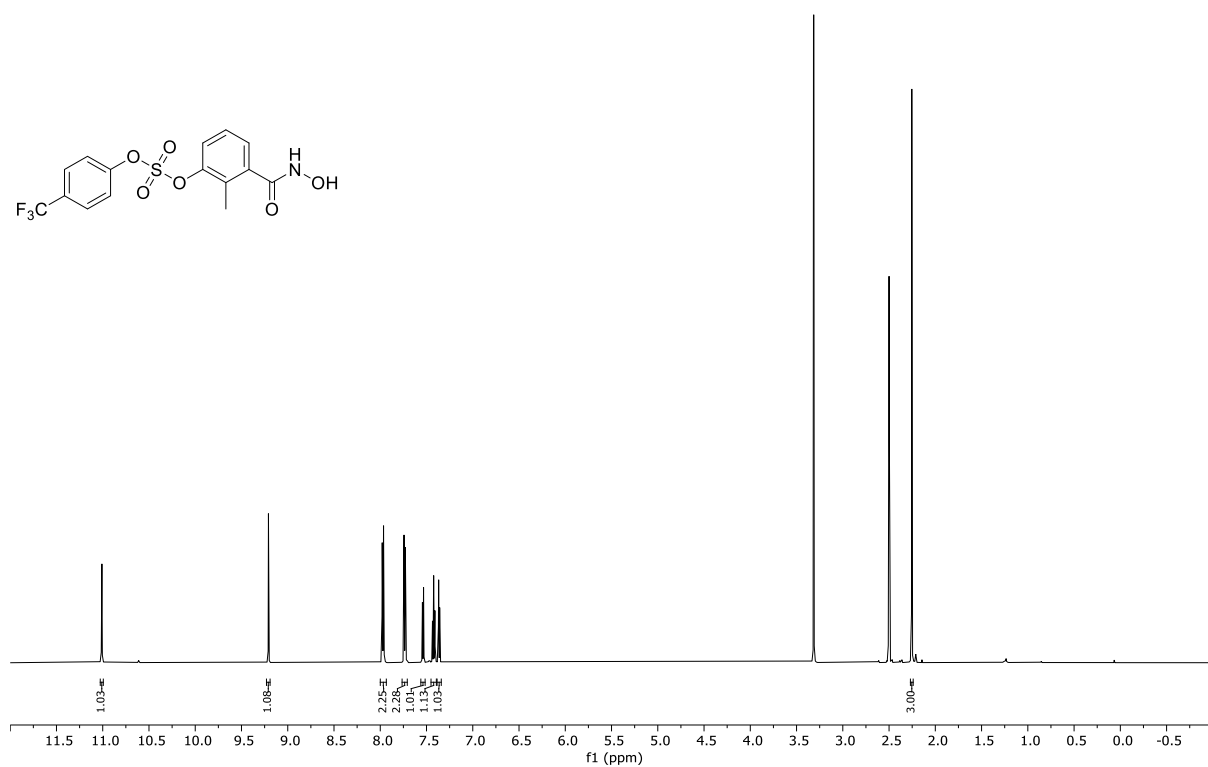
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D2**



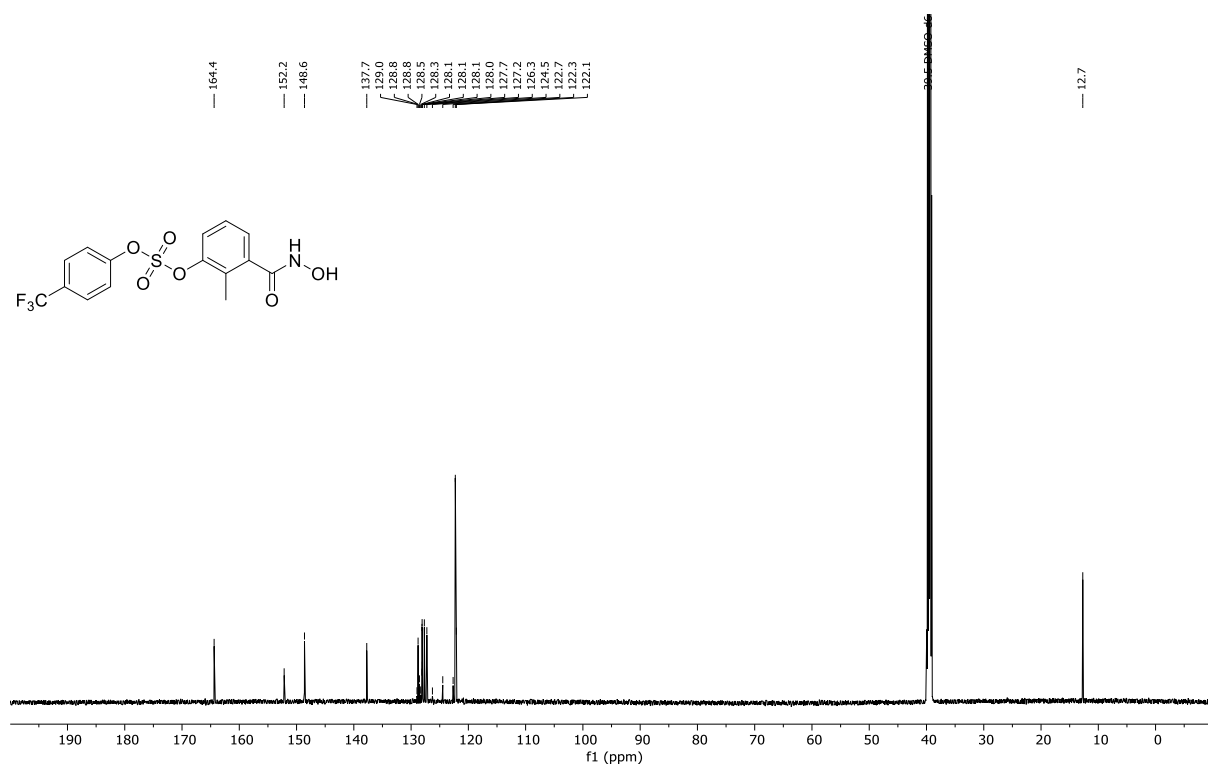
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D2**



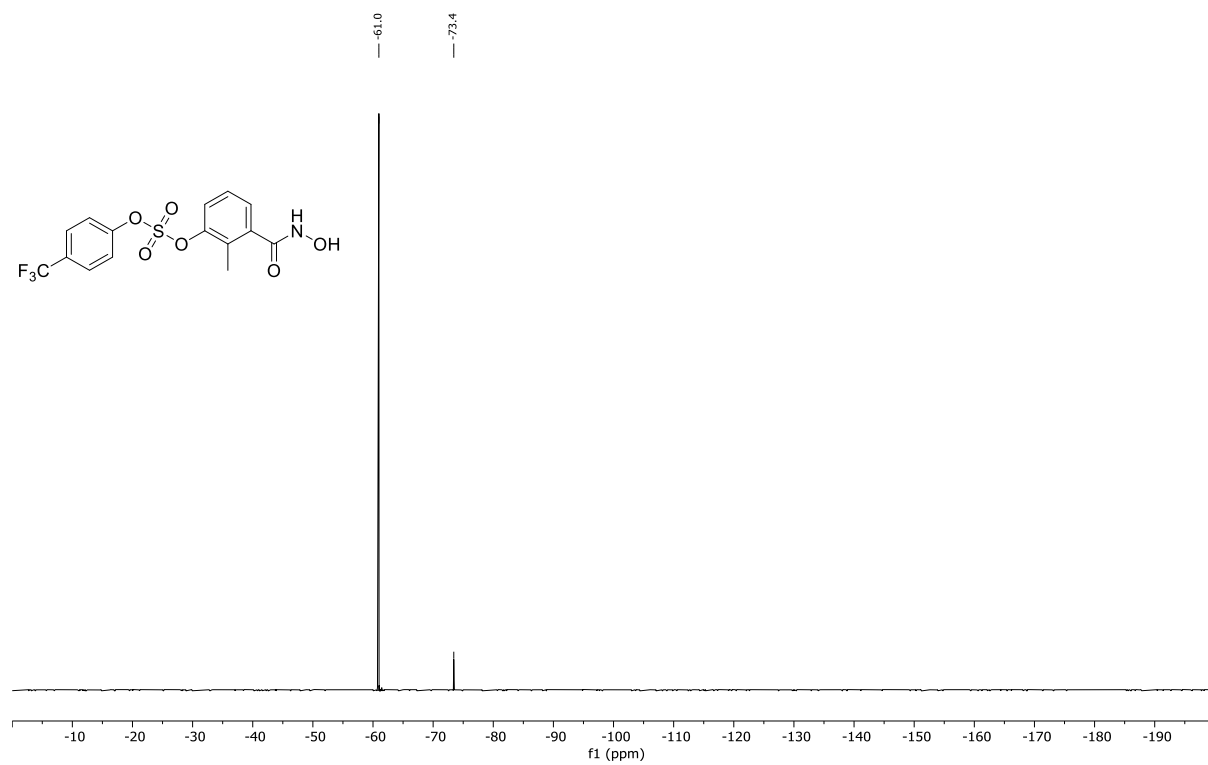
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D3**



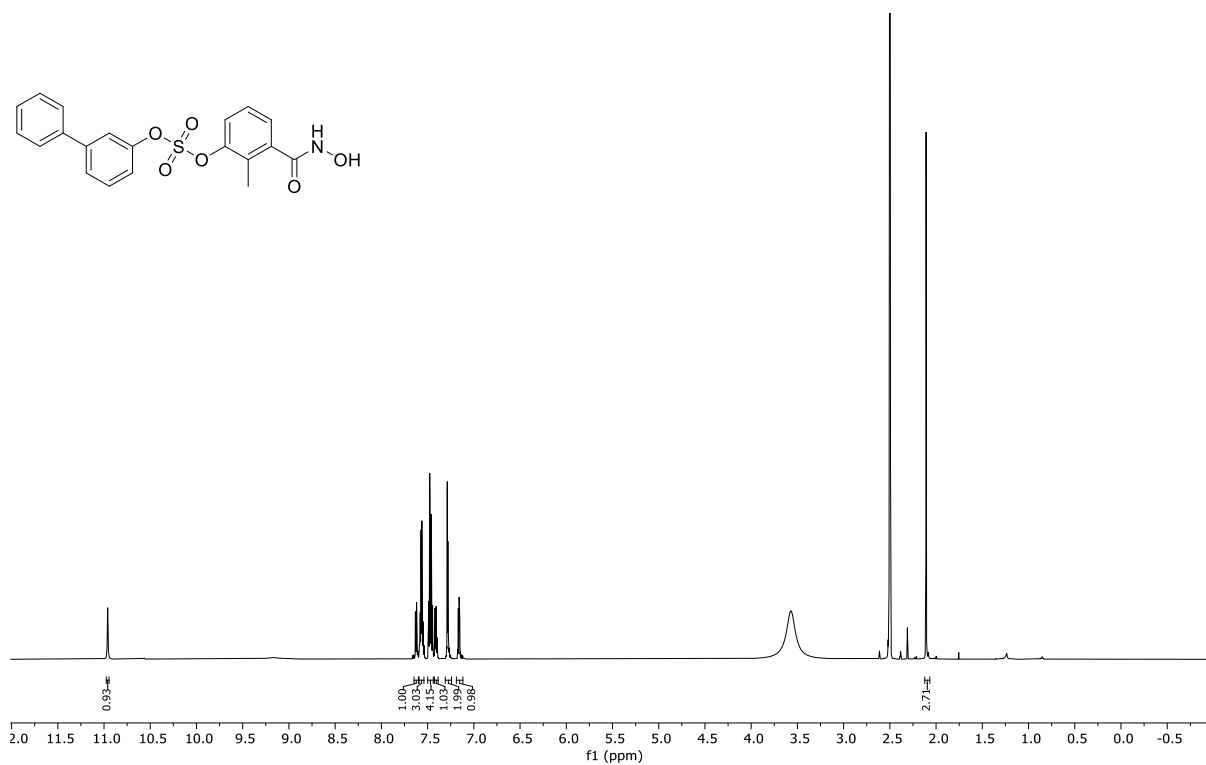
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D3**



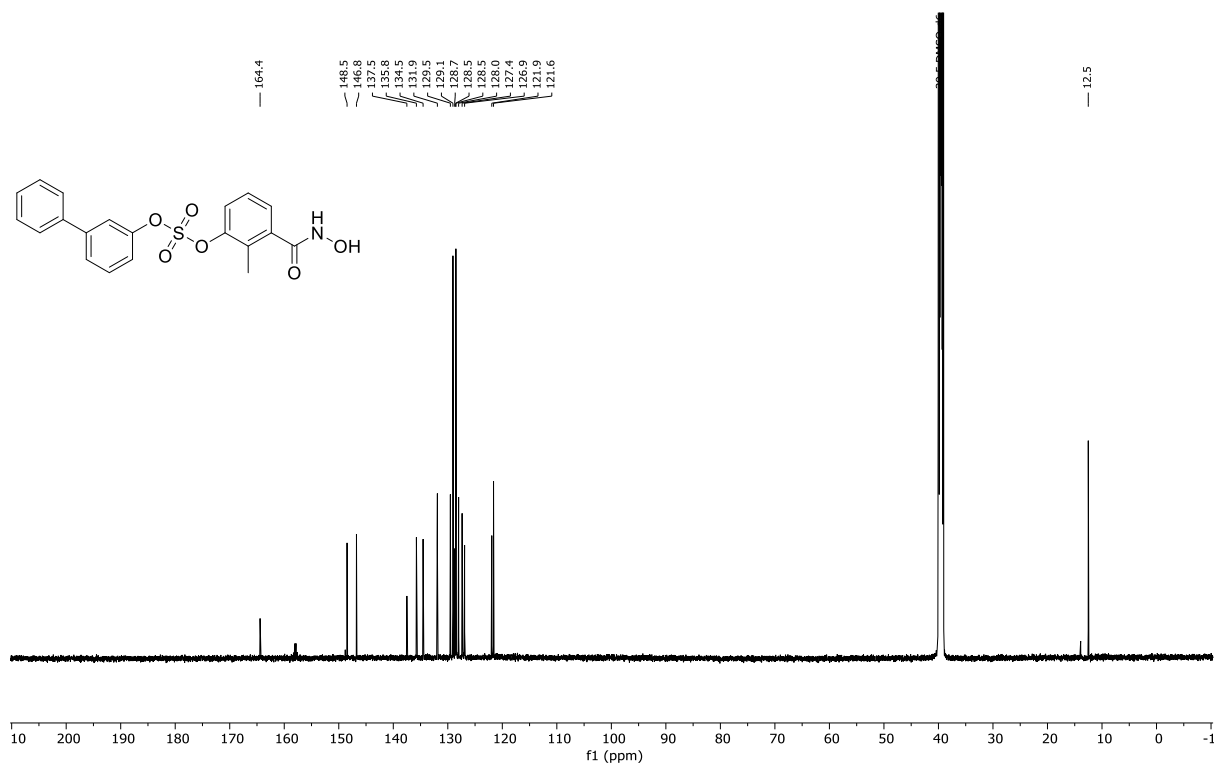
¹⁹F NMR spectra (376 MHz, DMSO-d₆) of compound **D3**



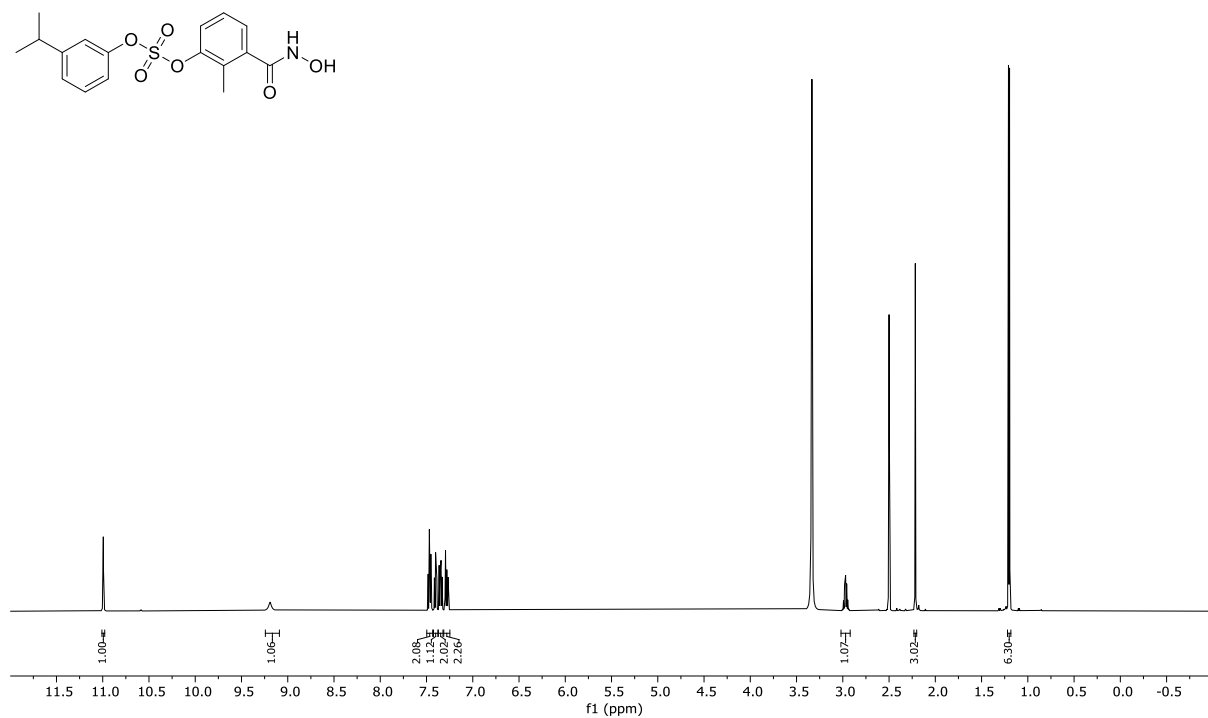
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D4**



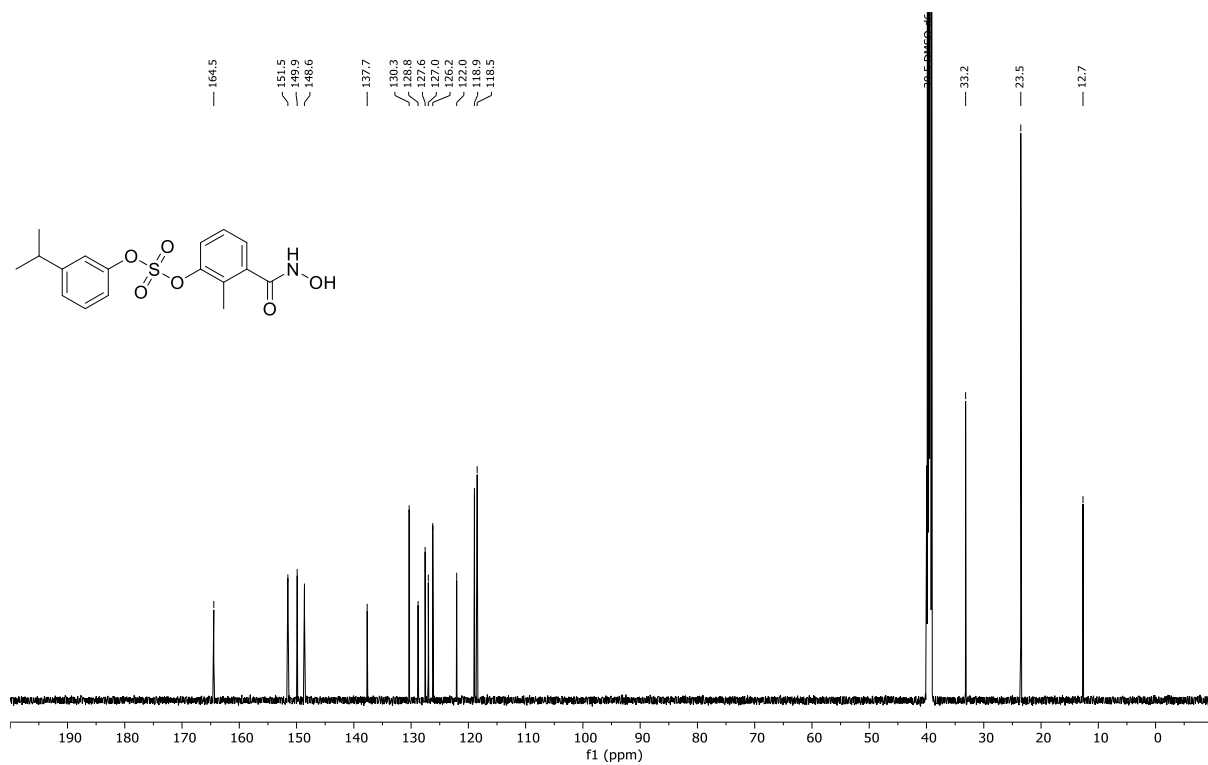
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D4** (minor contamination with TFA is observed at 158.8 ppm)



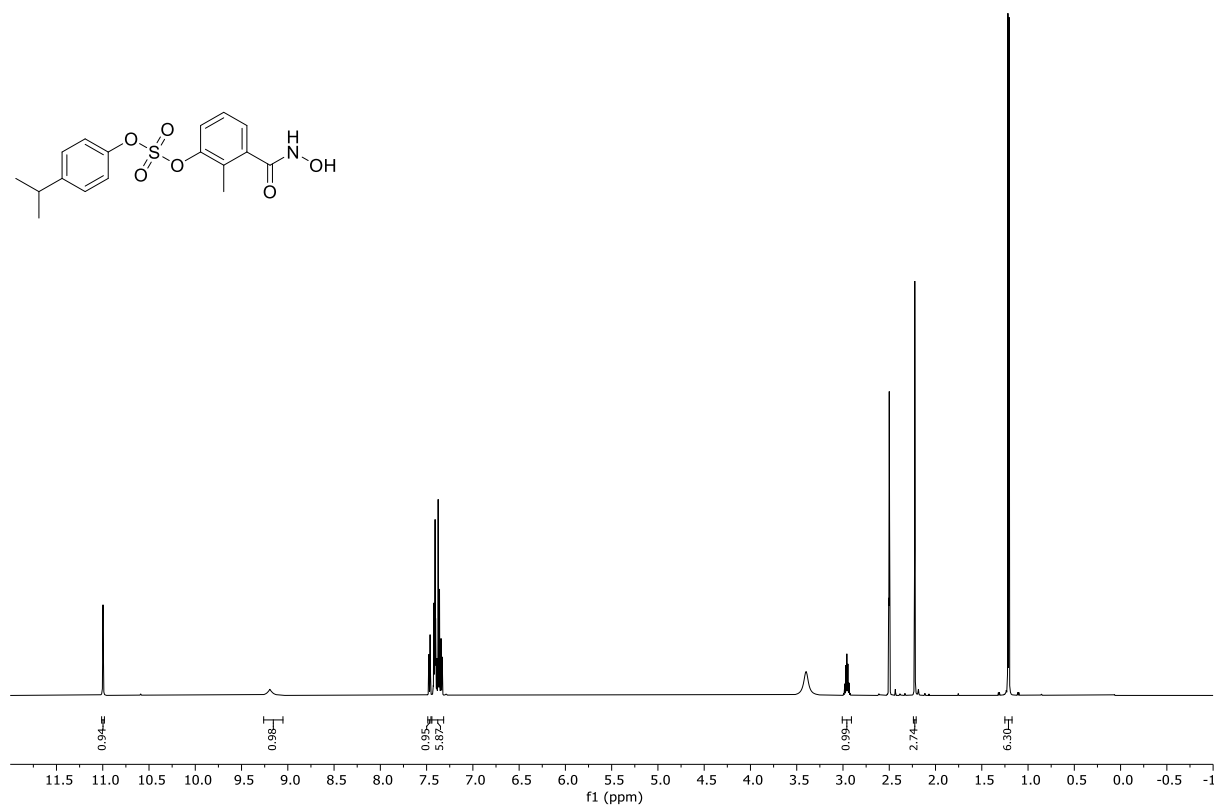
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D8**



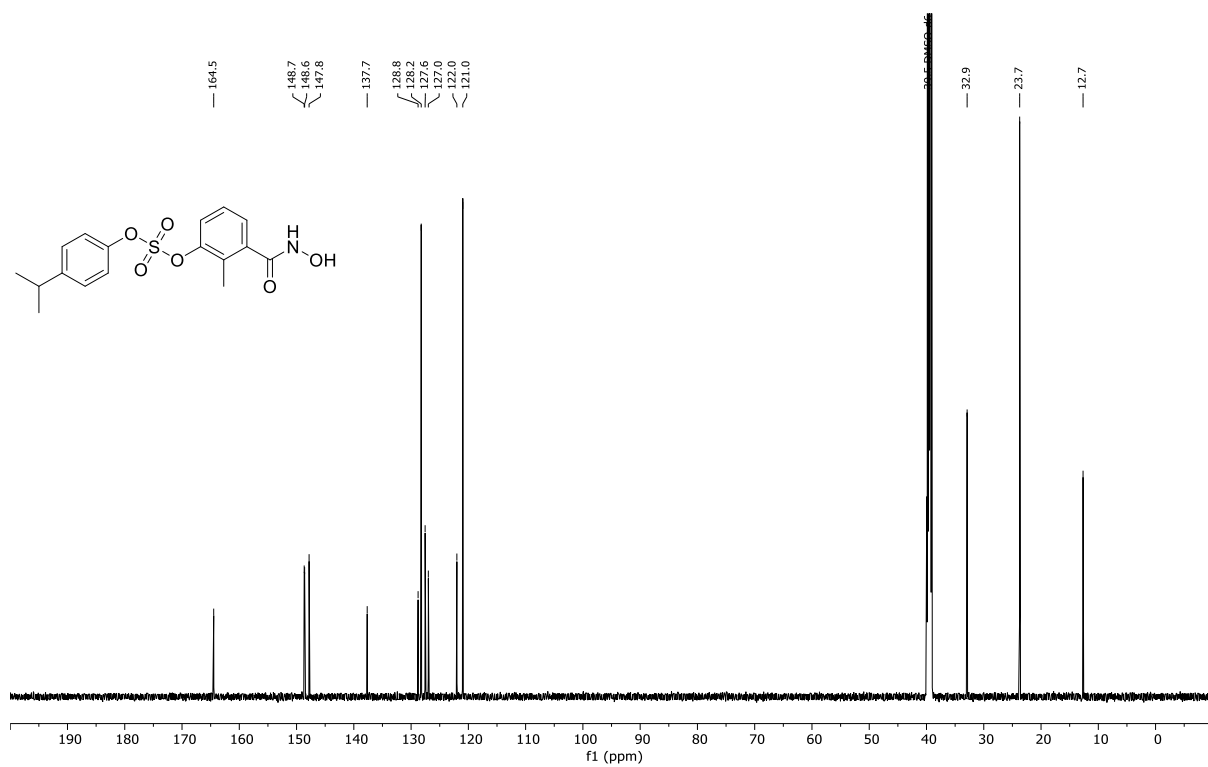
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D8**



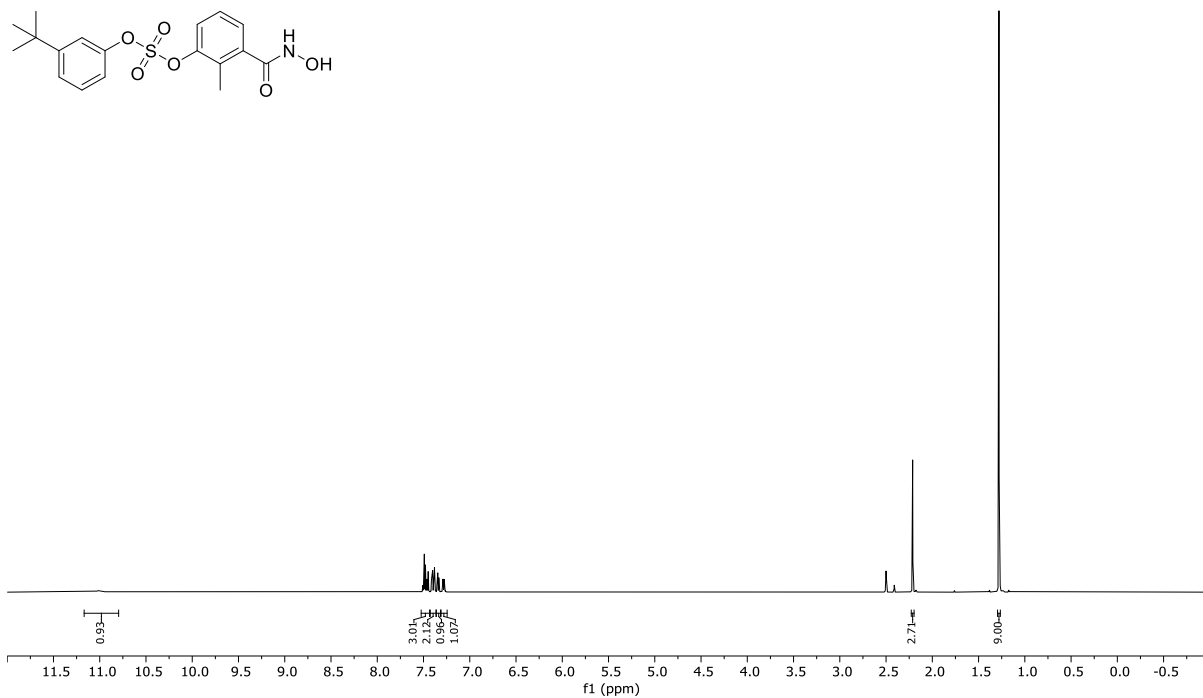
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D9**



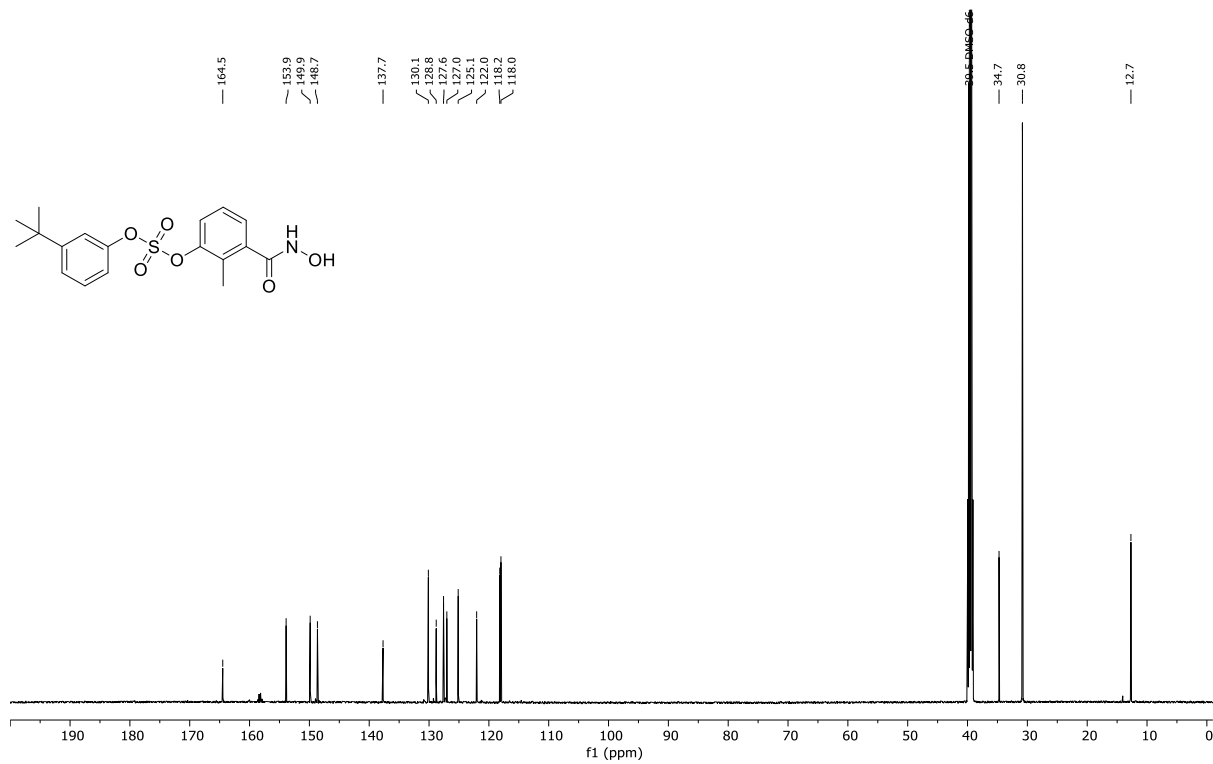
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D9**



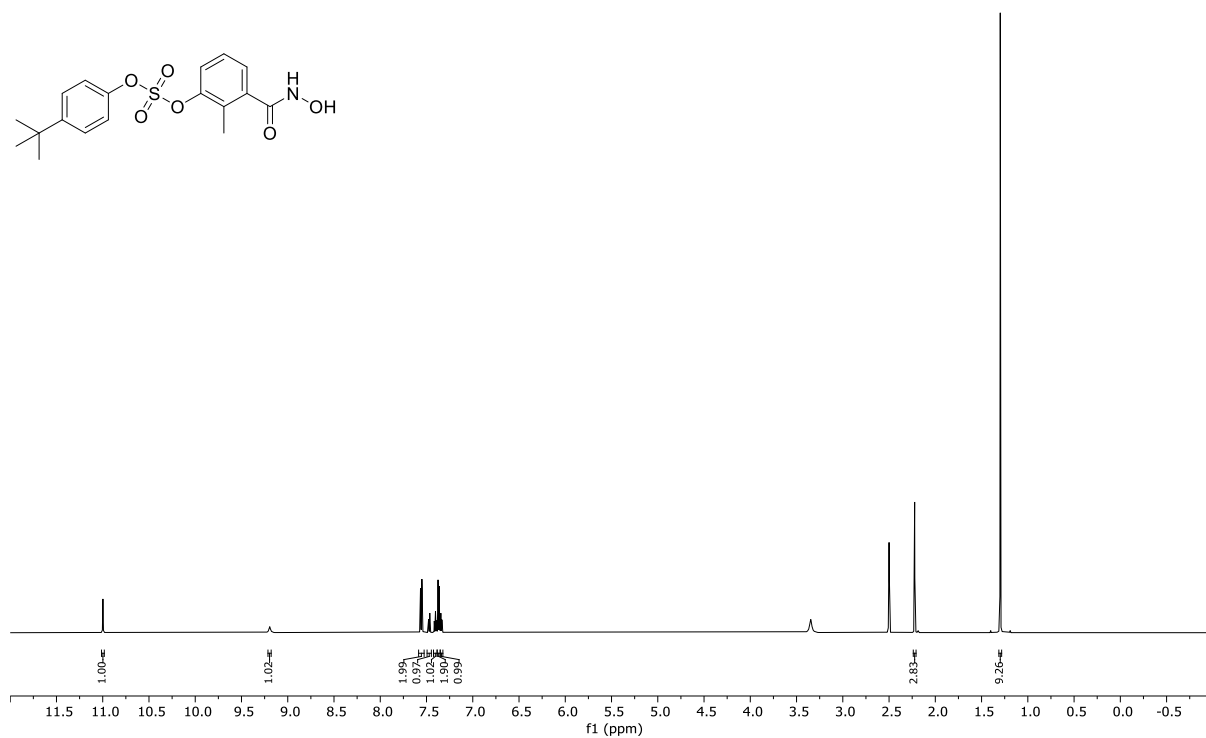
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D11**



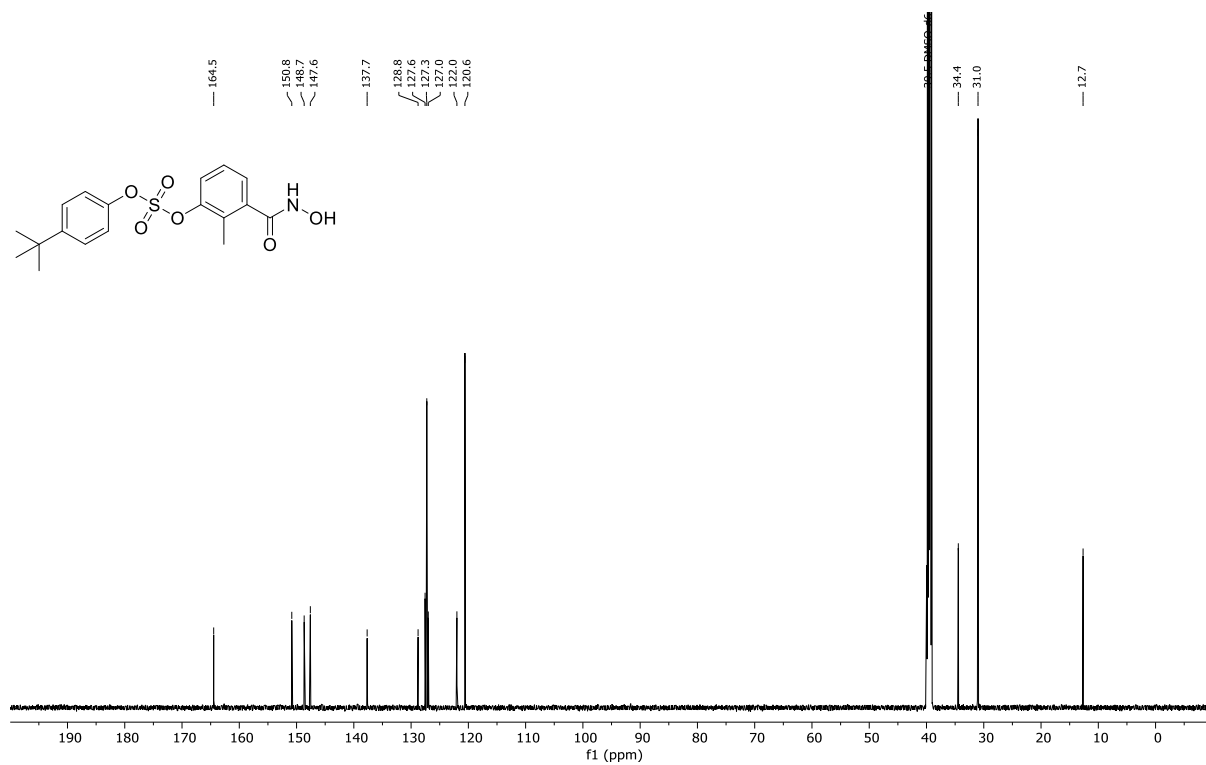
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D11** (minor contamination with TFA is observed at 158.8 ppm)



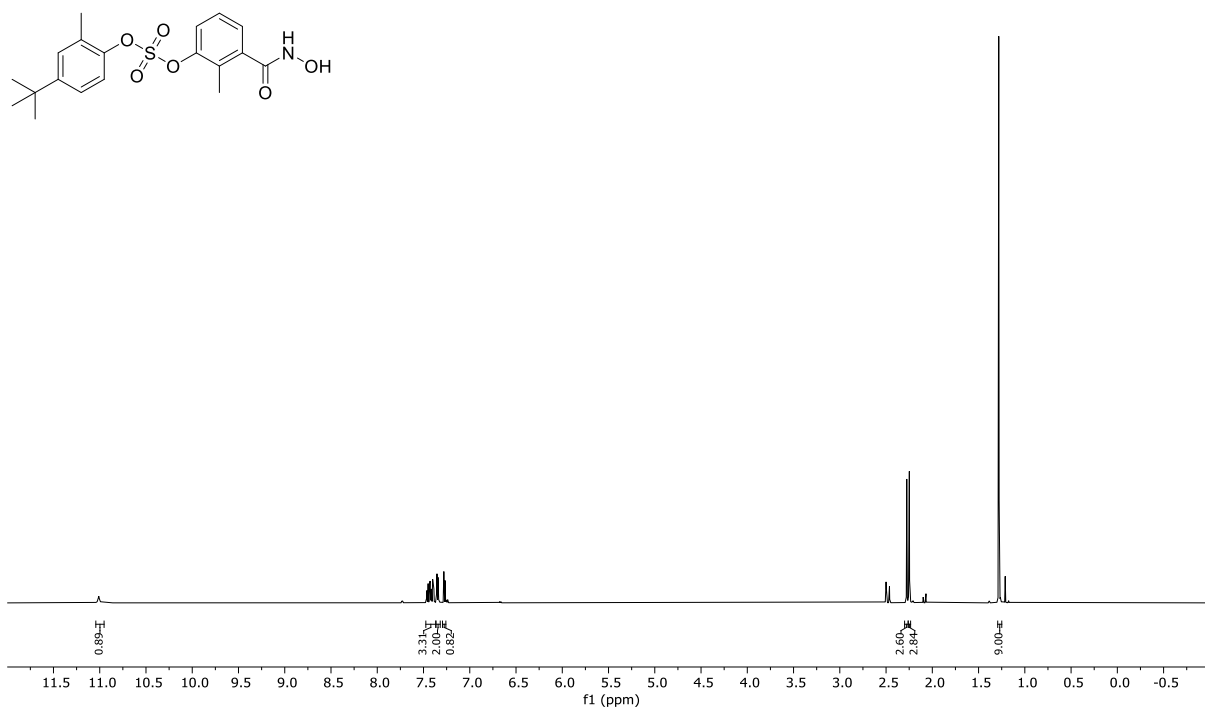
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D12**



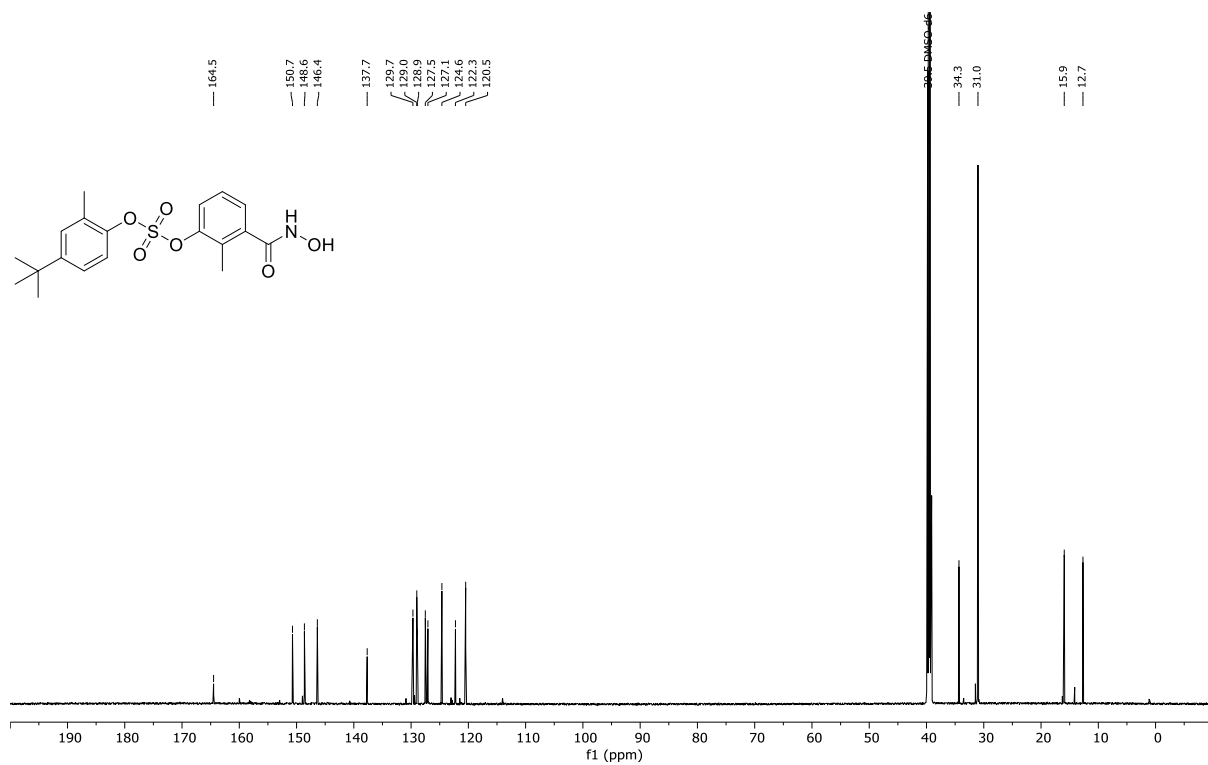
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D12**



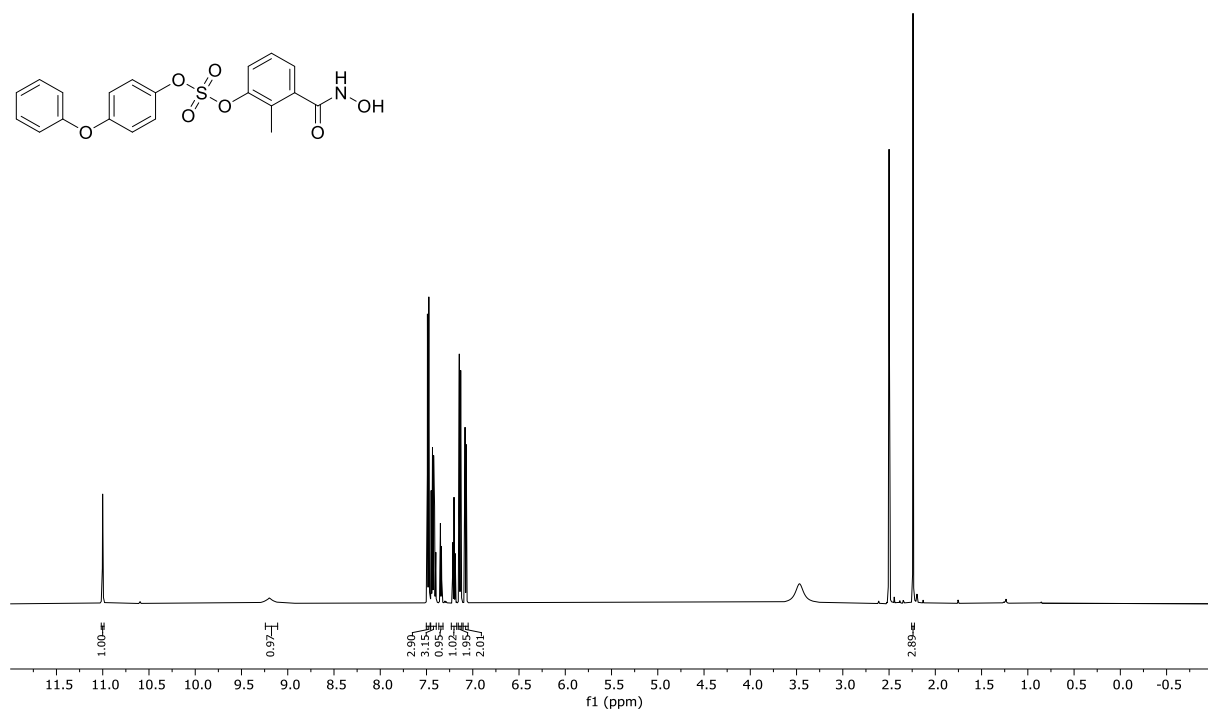
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D19**



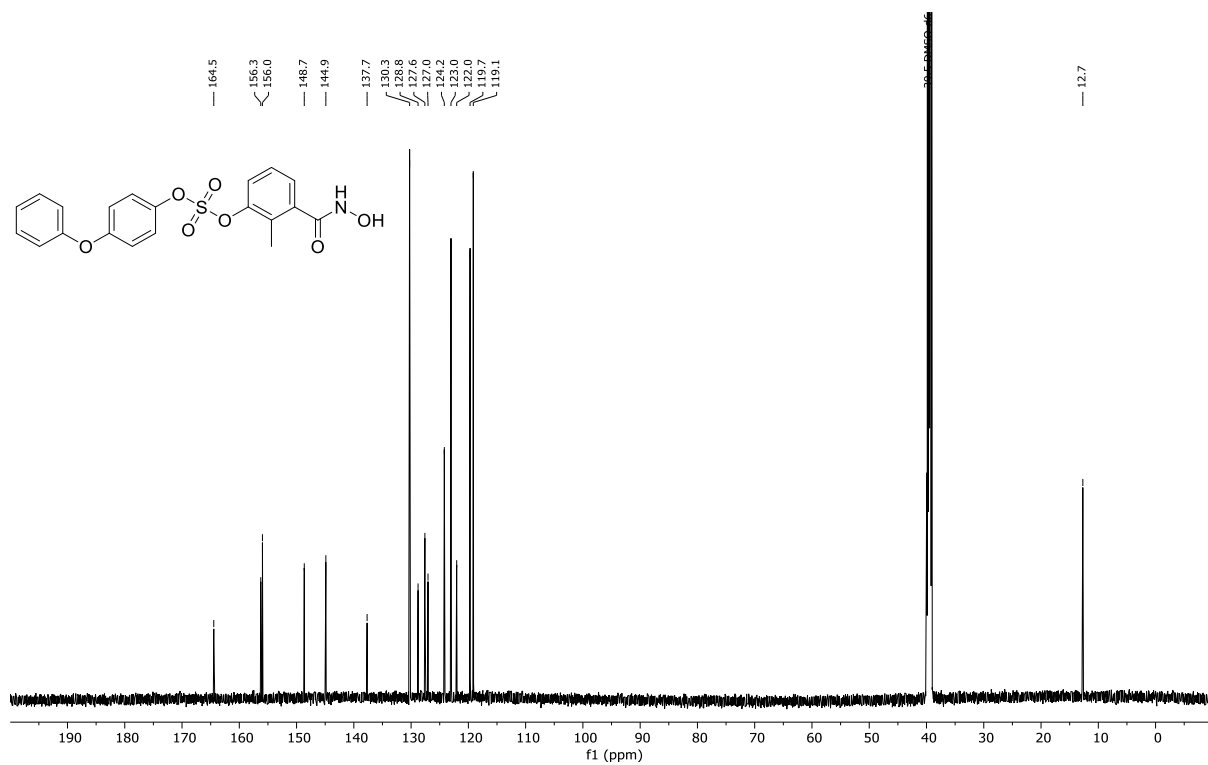
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D19**



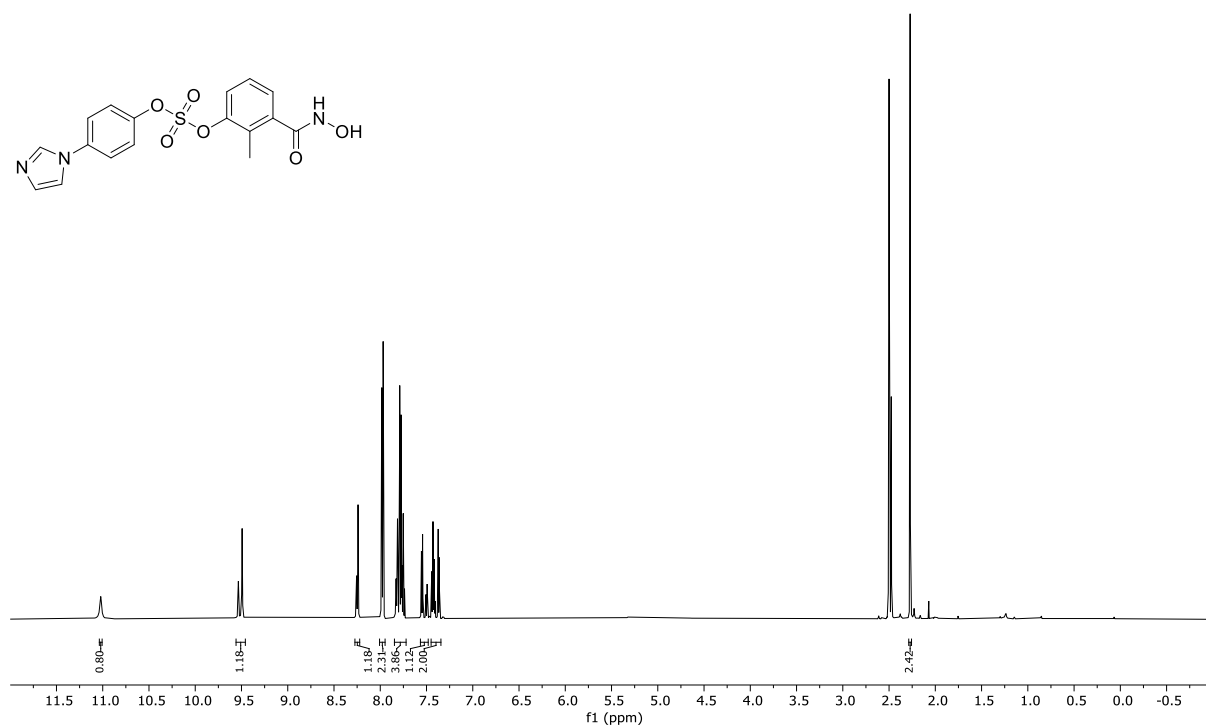
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D21**



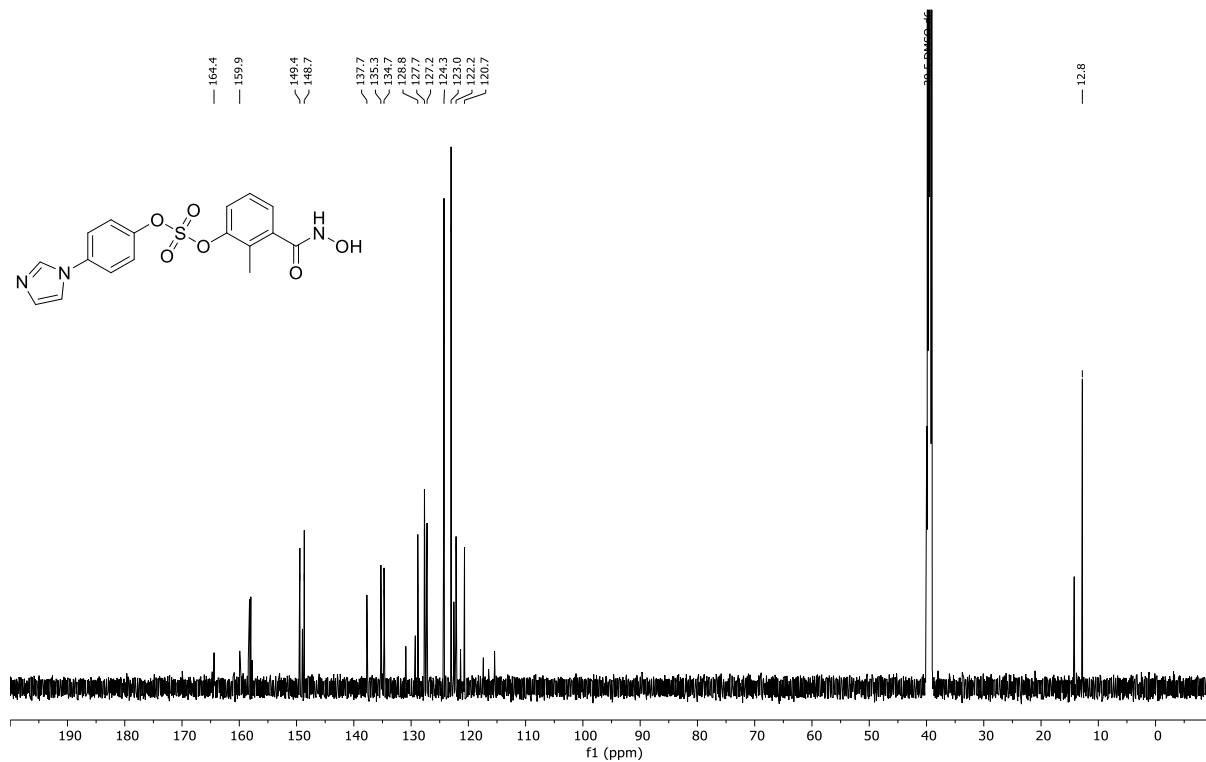
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D21**



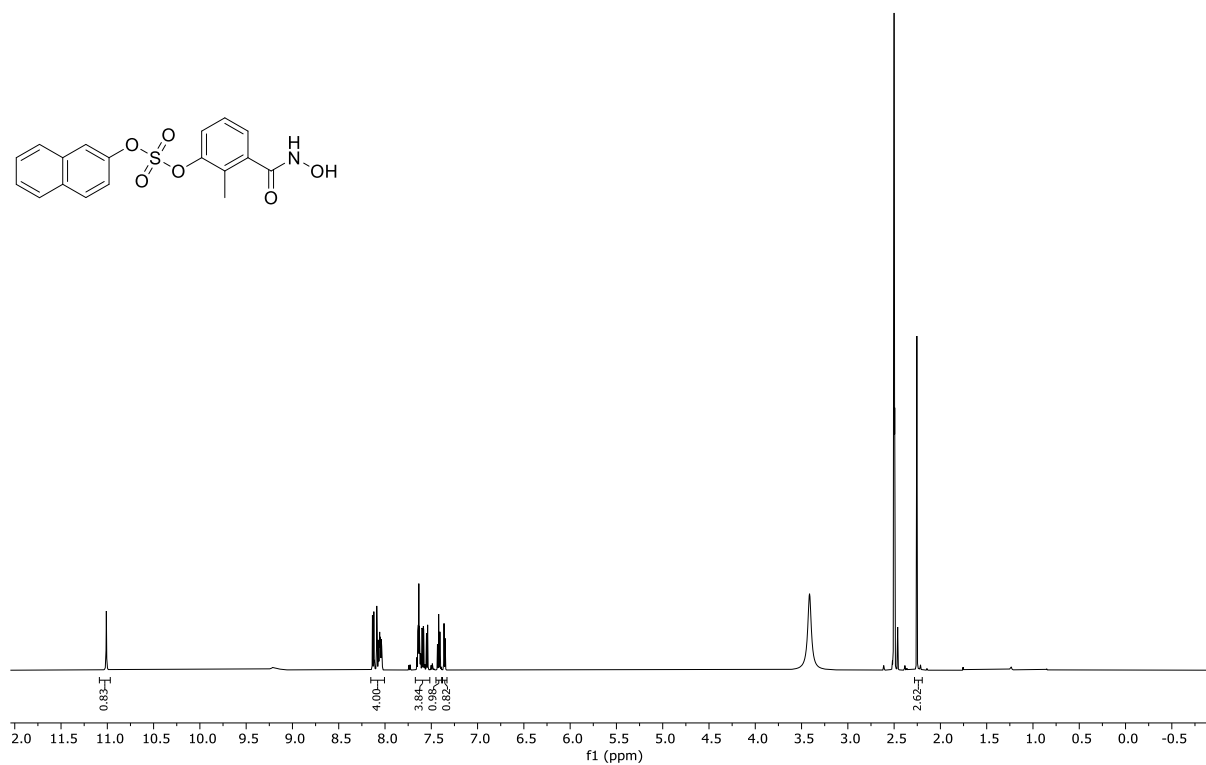
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D23**



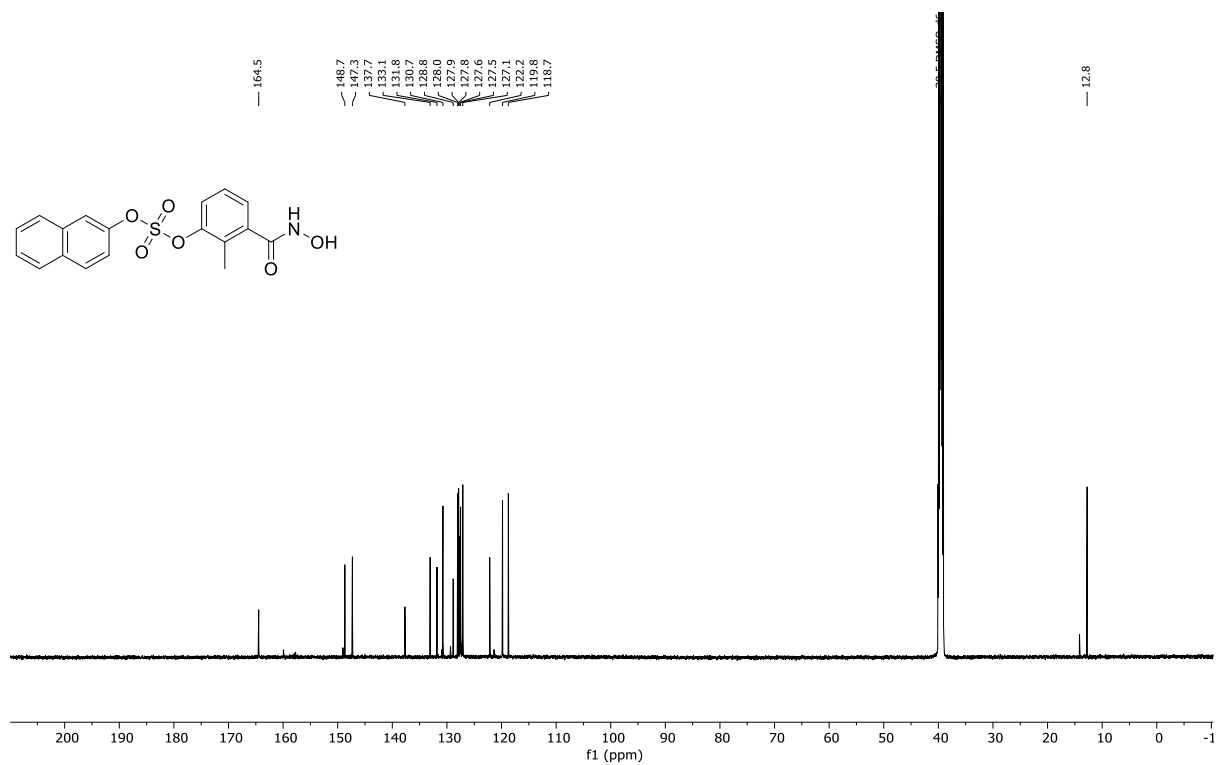
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D23** (minor contamination with TFA is observed at 158.8 ppm)



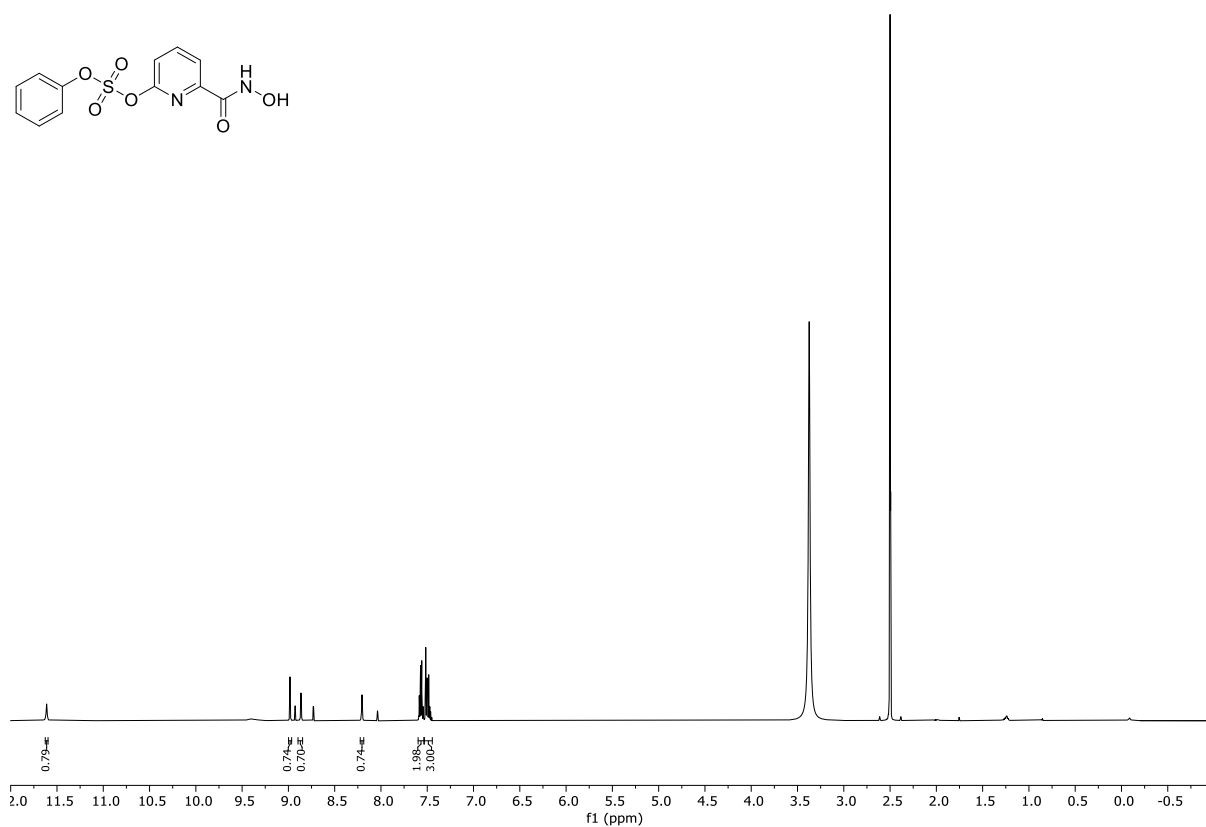
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **D24**



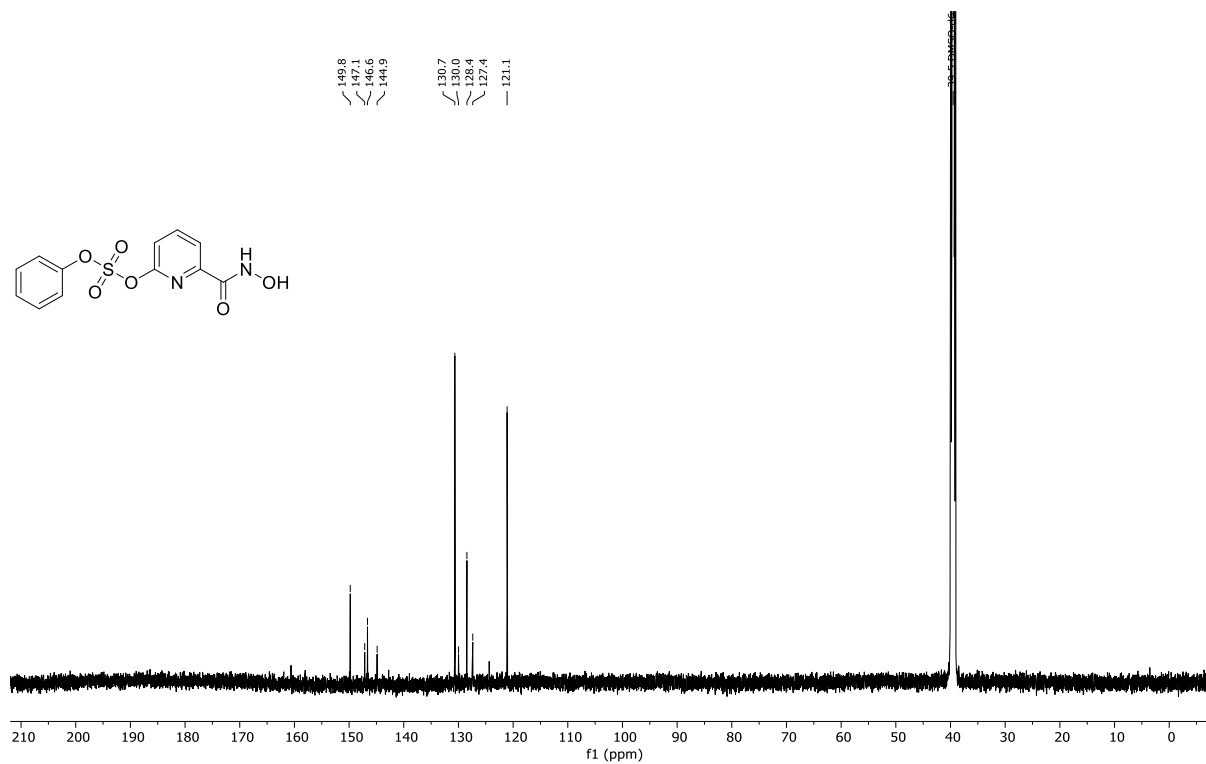
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **D24**



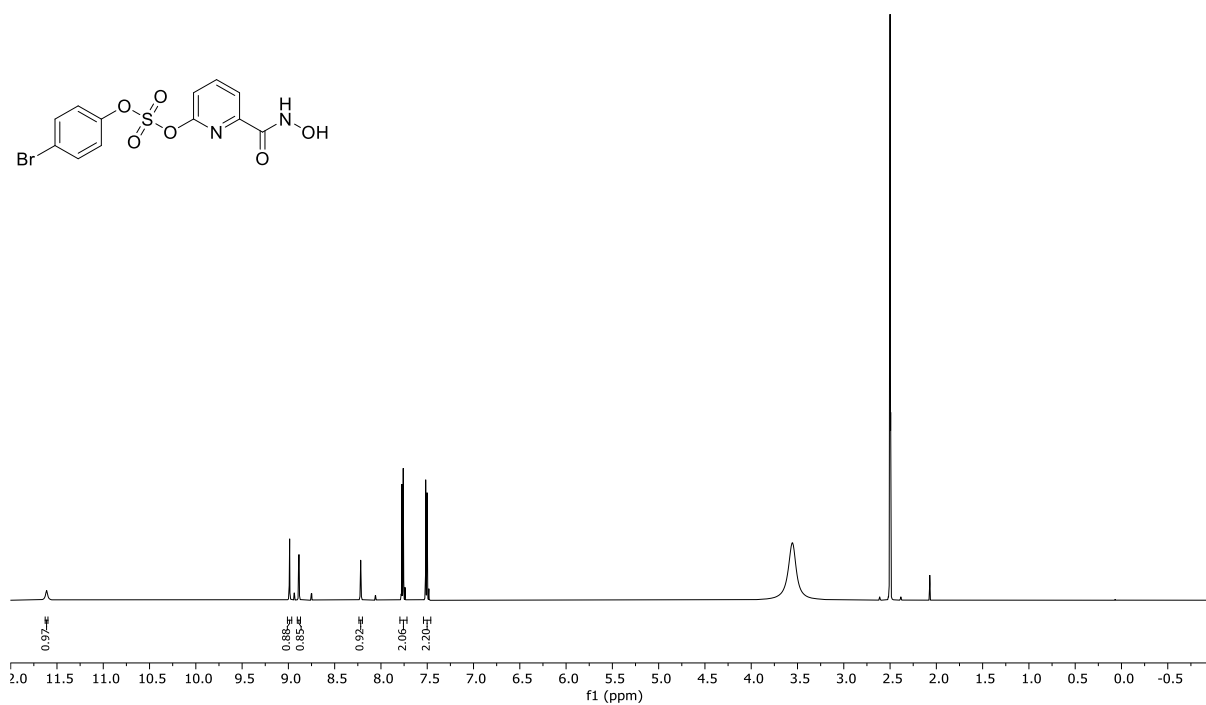
¹H NMR spectra (600 MHz, DMSO-d₆) of compound E1



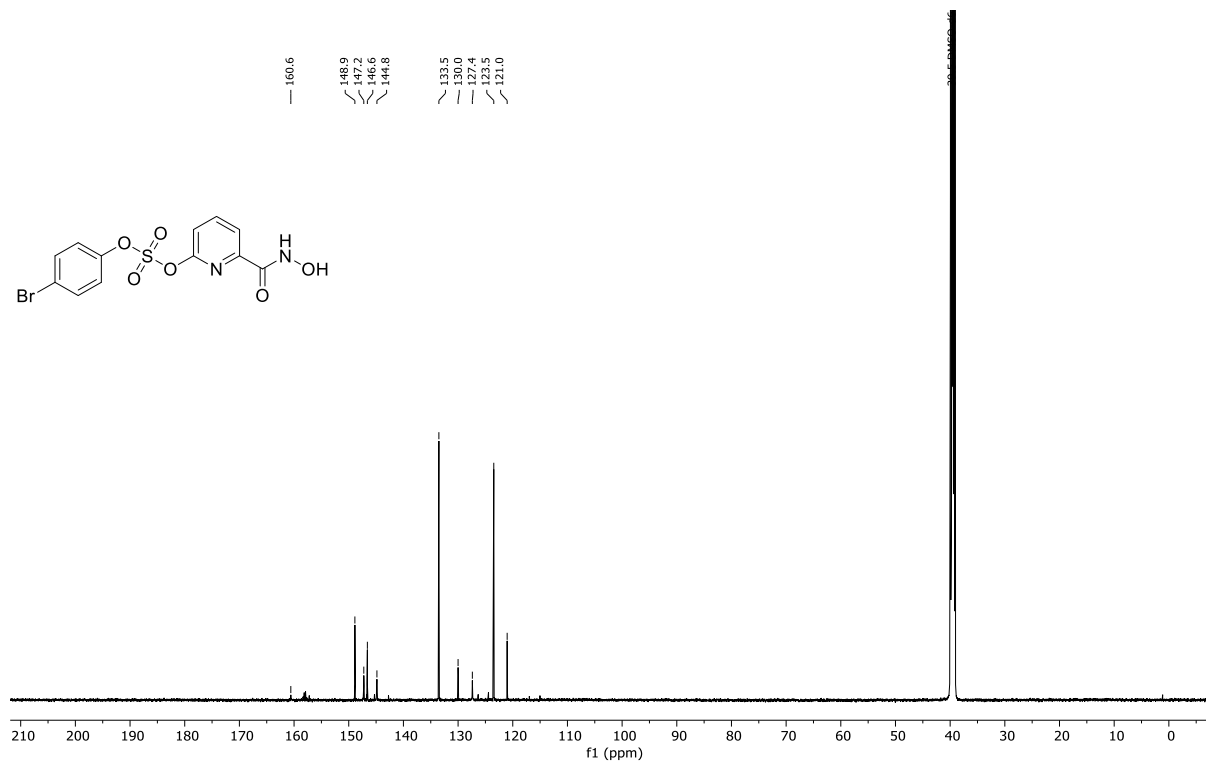
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound E1



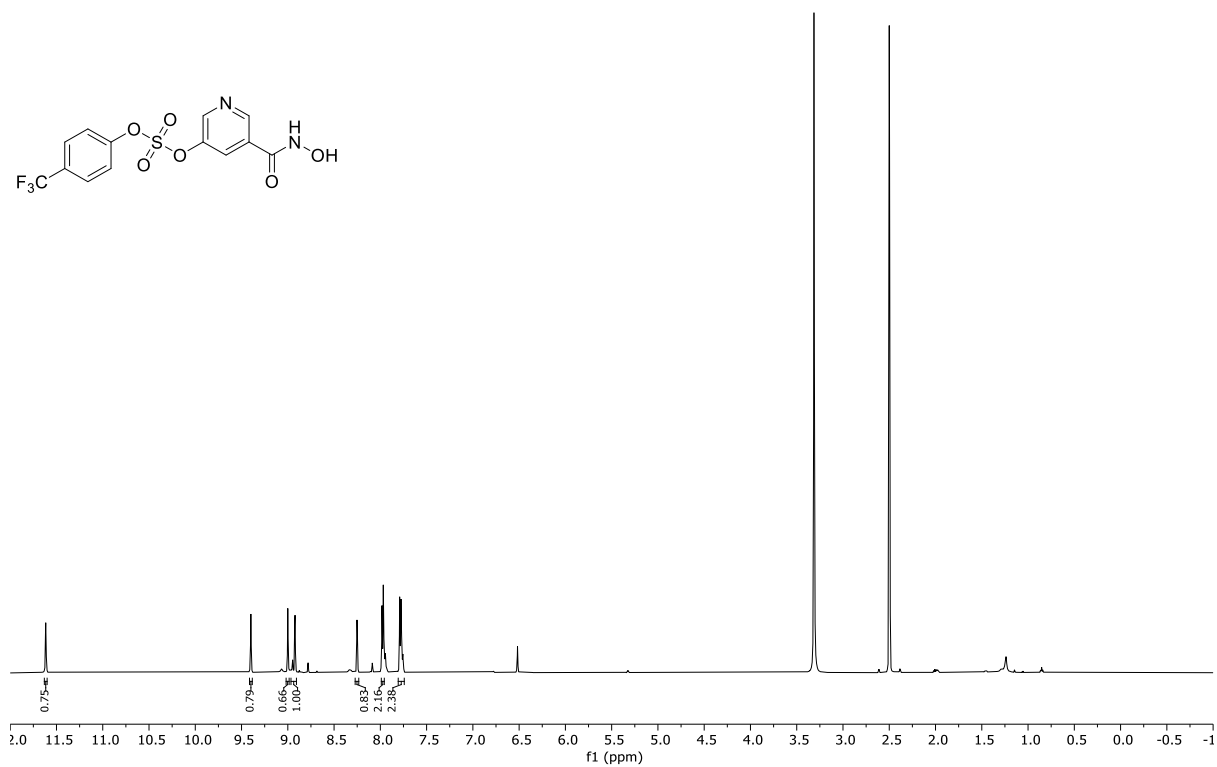
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E2**



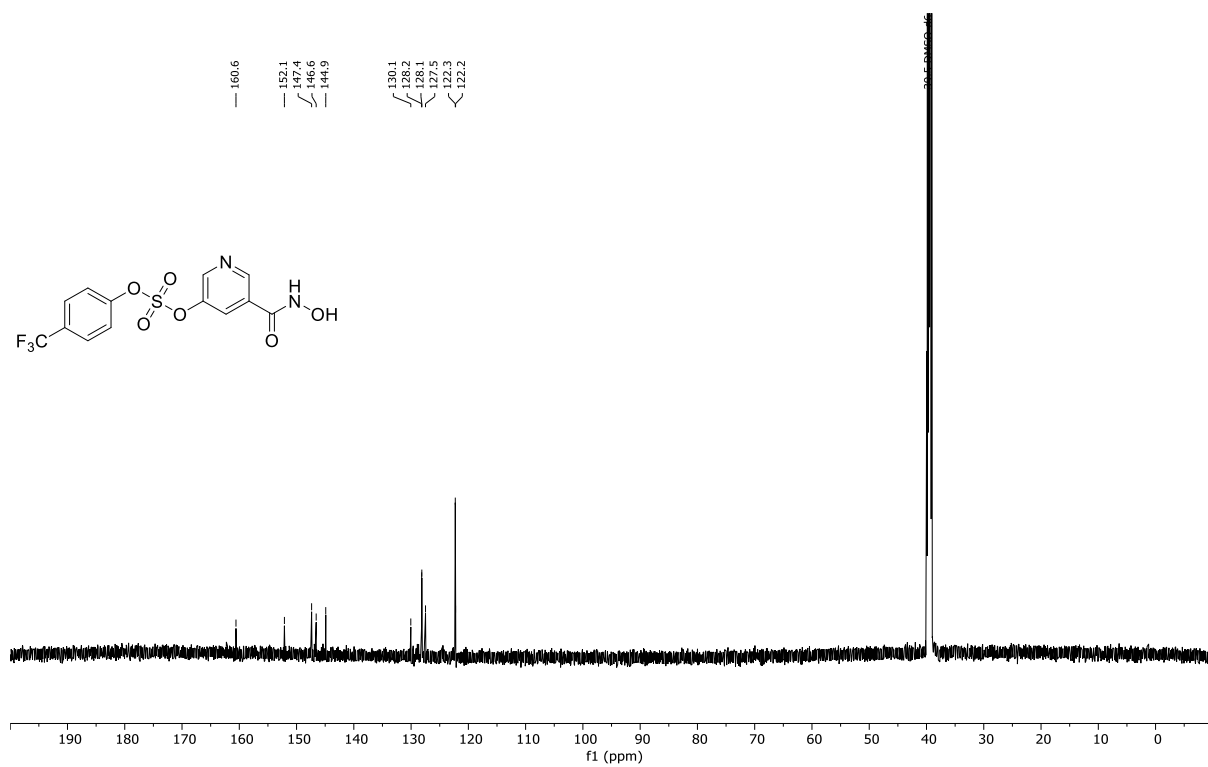
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E2** (minor contamination with TFA is observed at 158.8 ppm)



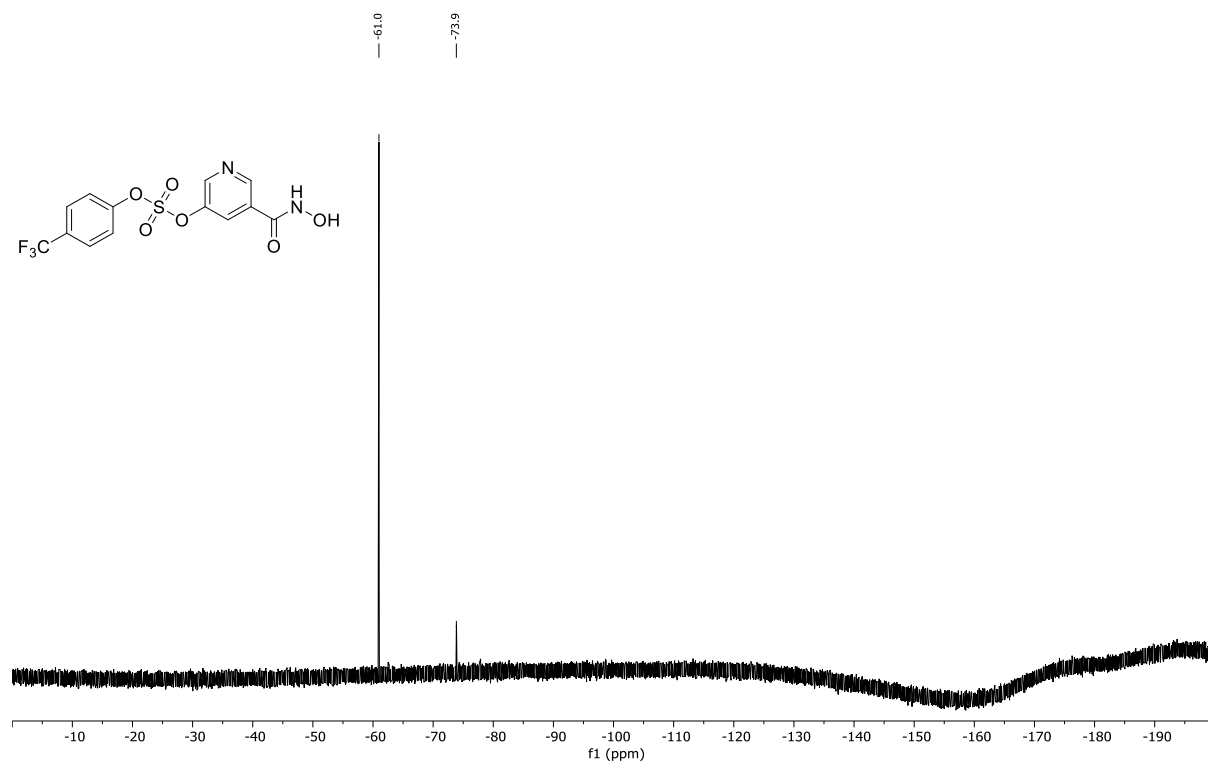
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E3**



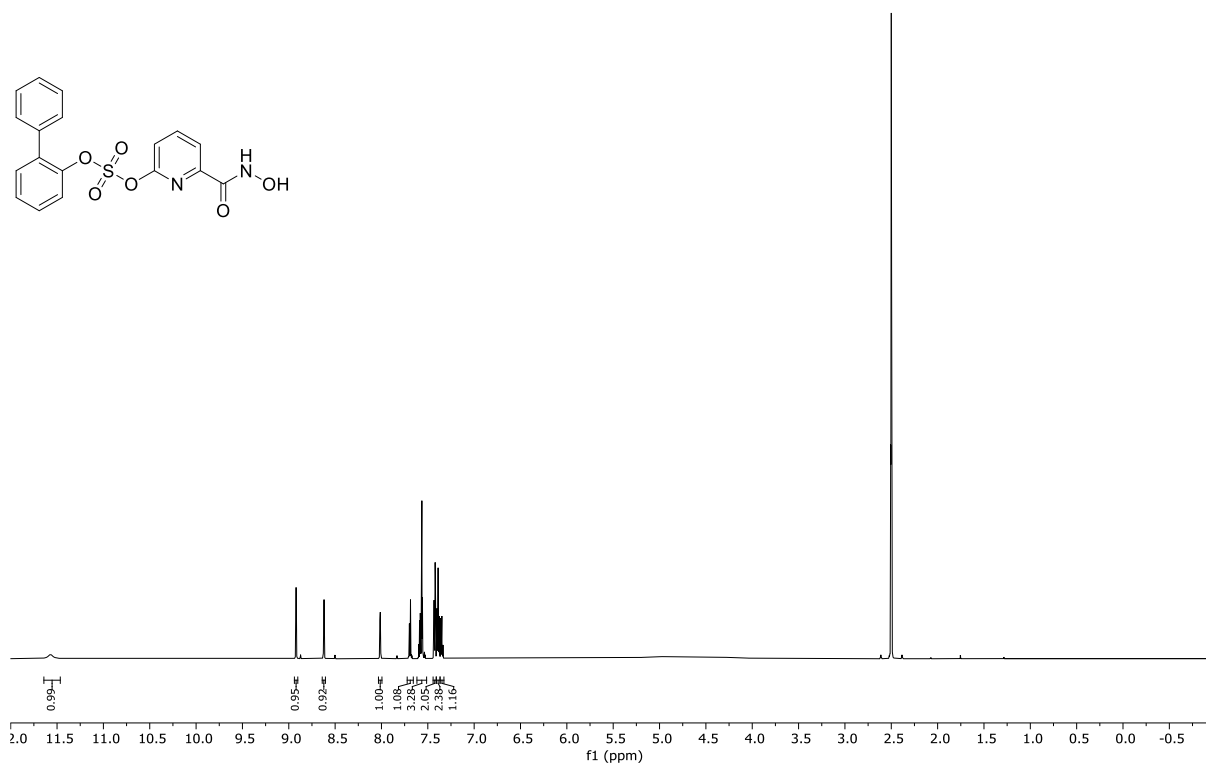
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E3**



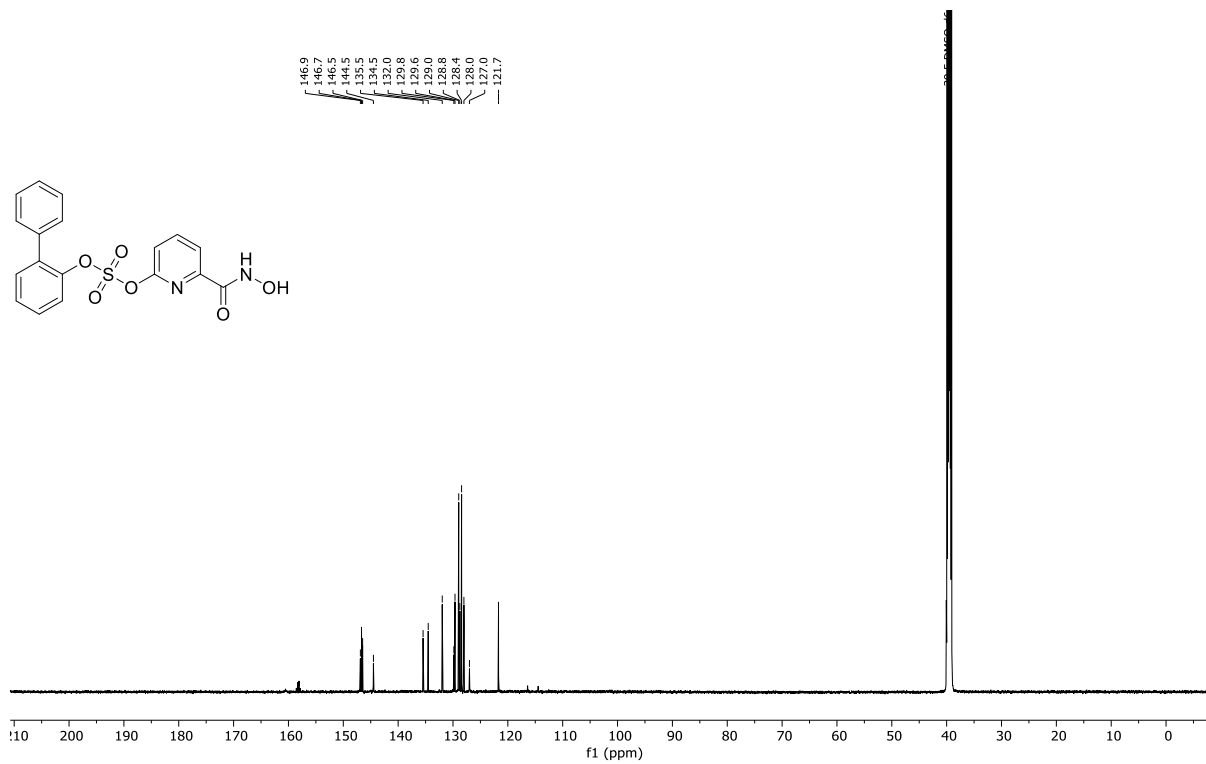
^{19}F NMR spectra (376 MHz, $\text{DMSO-}d_6$) of compound **E3**



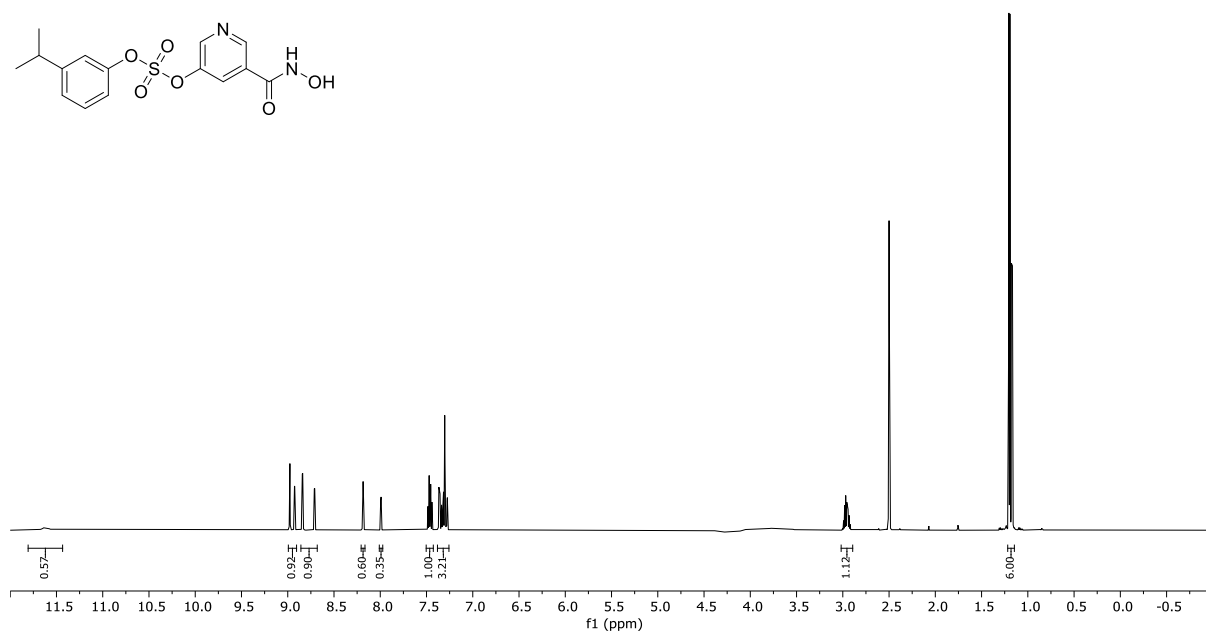
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E4**



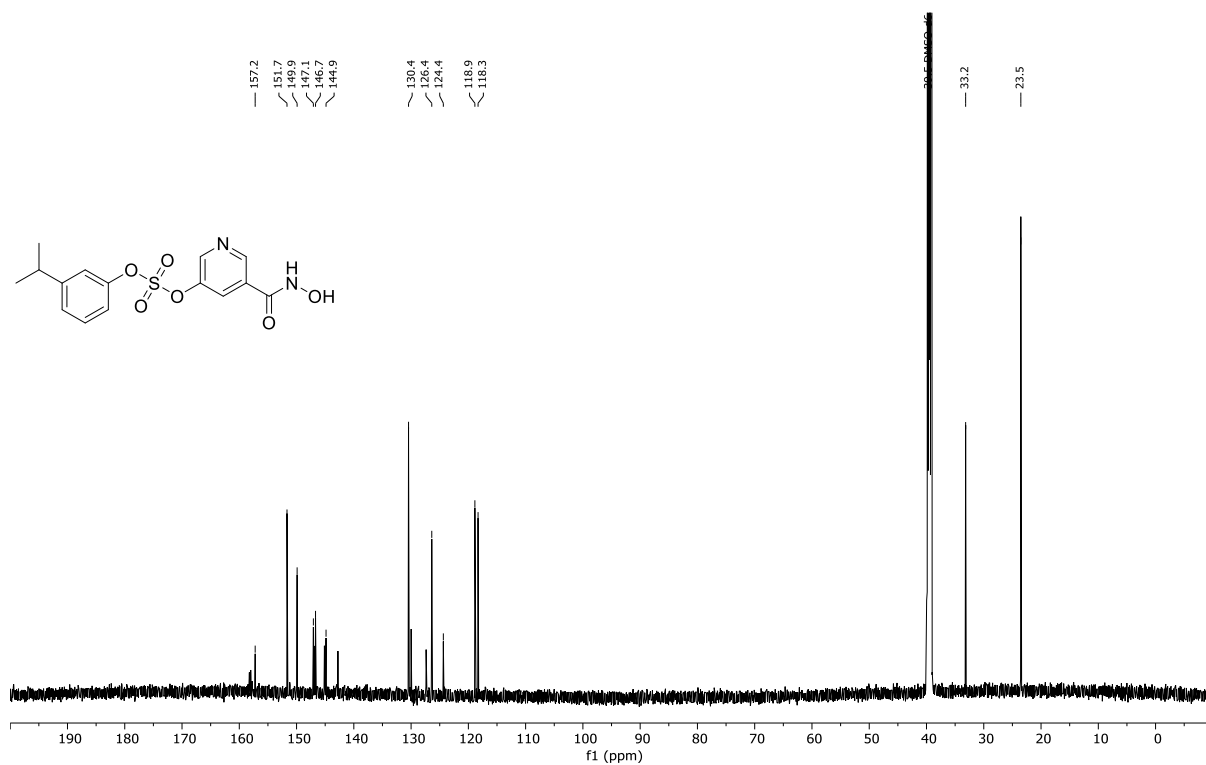
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E4** (minor contamination with TFA is observed at 158.8 ppm)



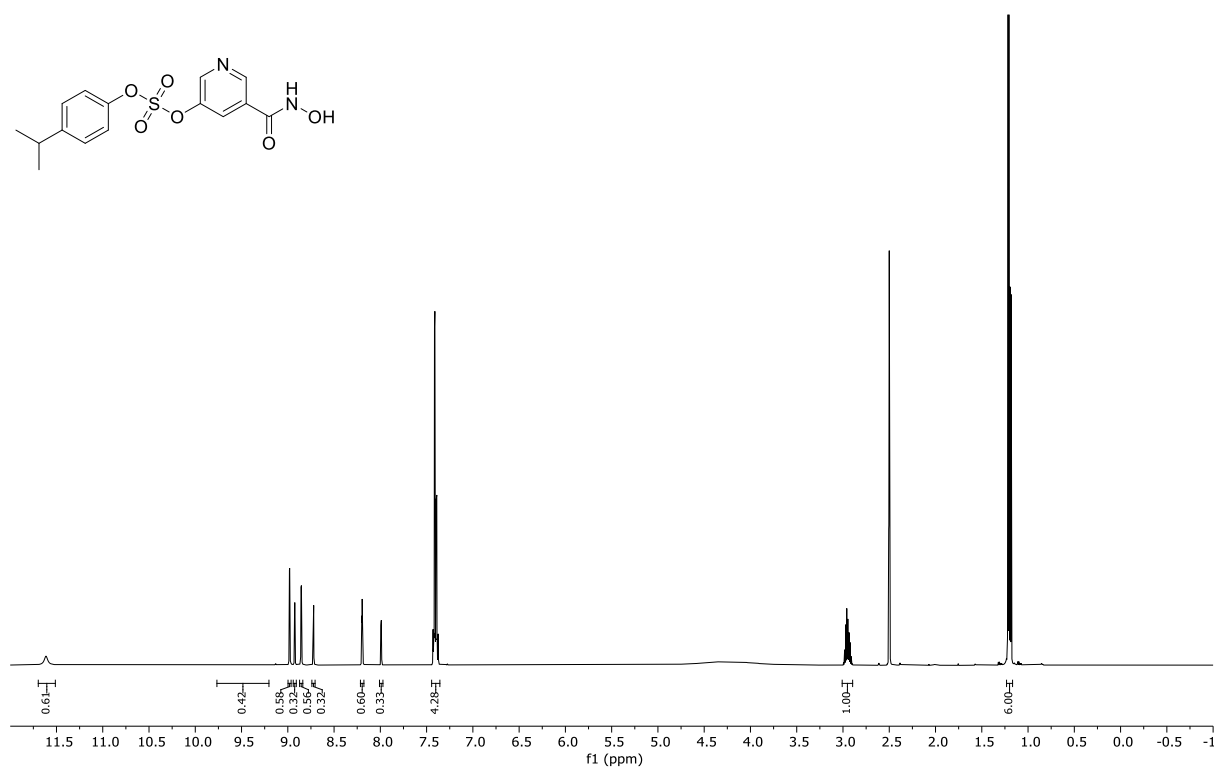
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E8**



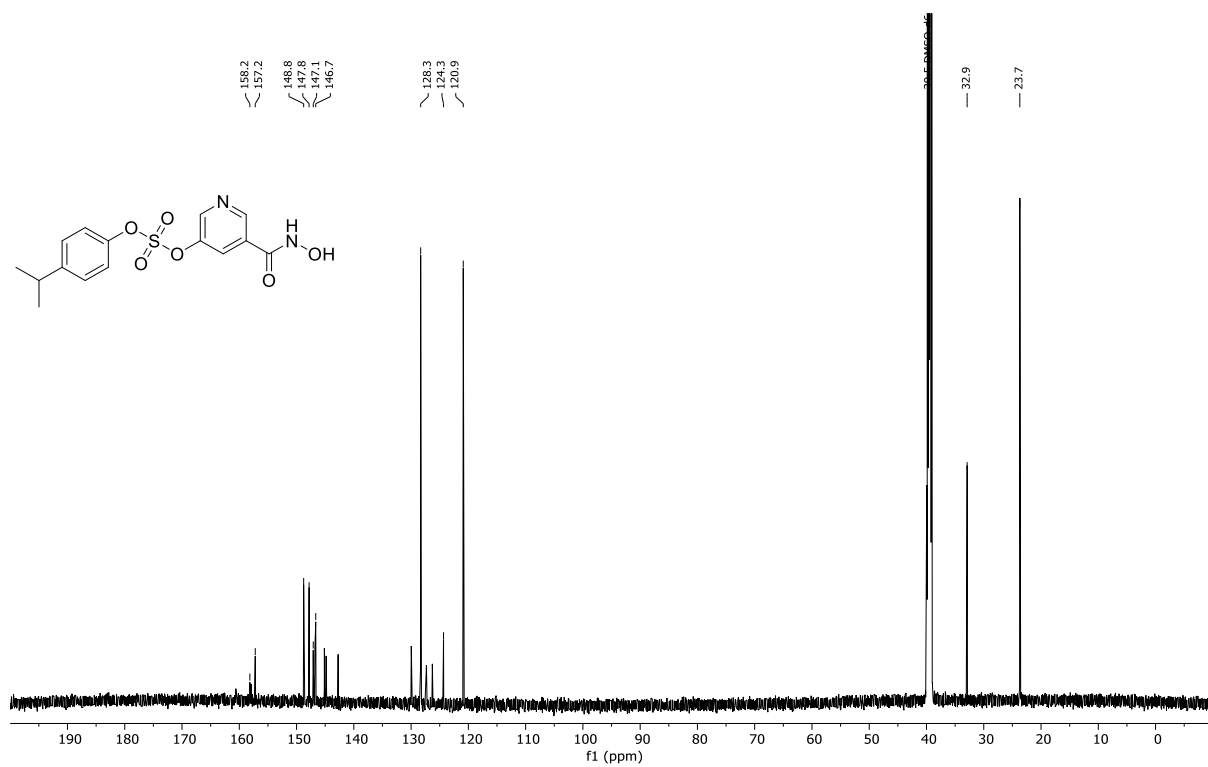
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E8** (minor contamination with TFA is observed at 158.8 ppm)



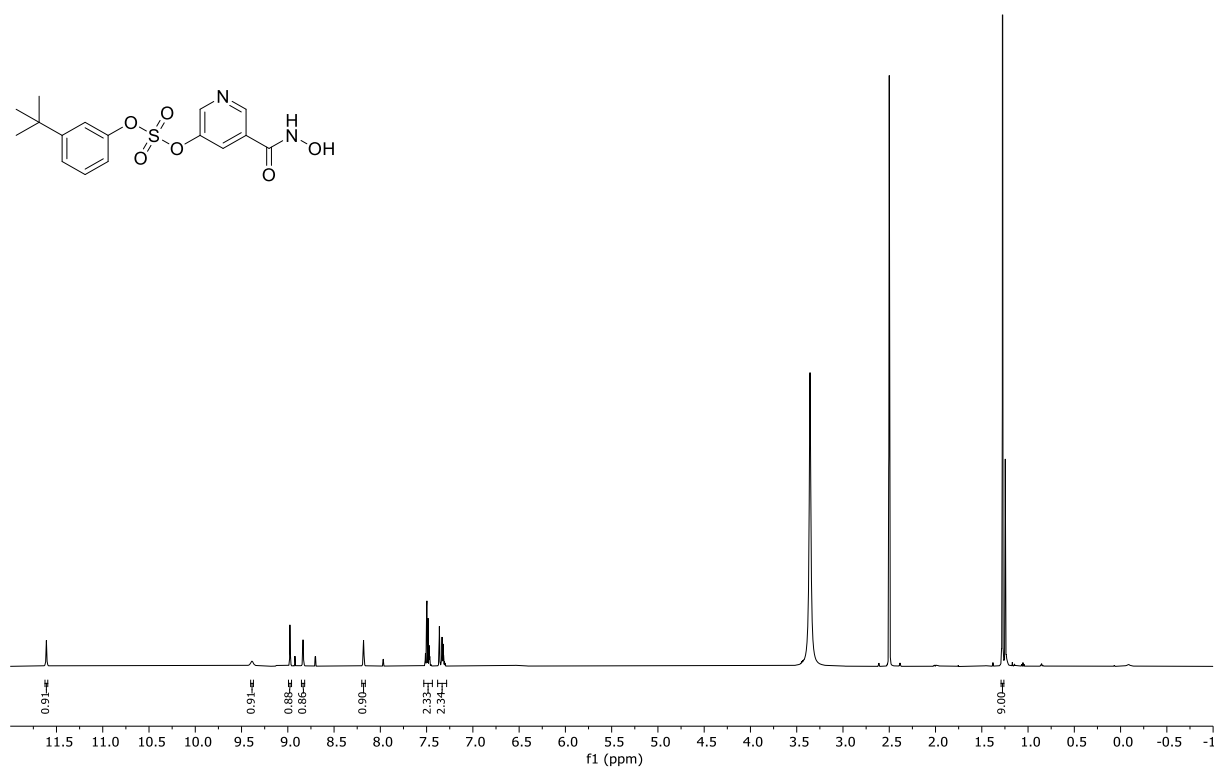
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E9**



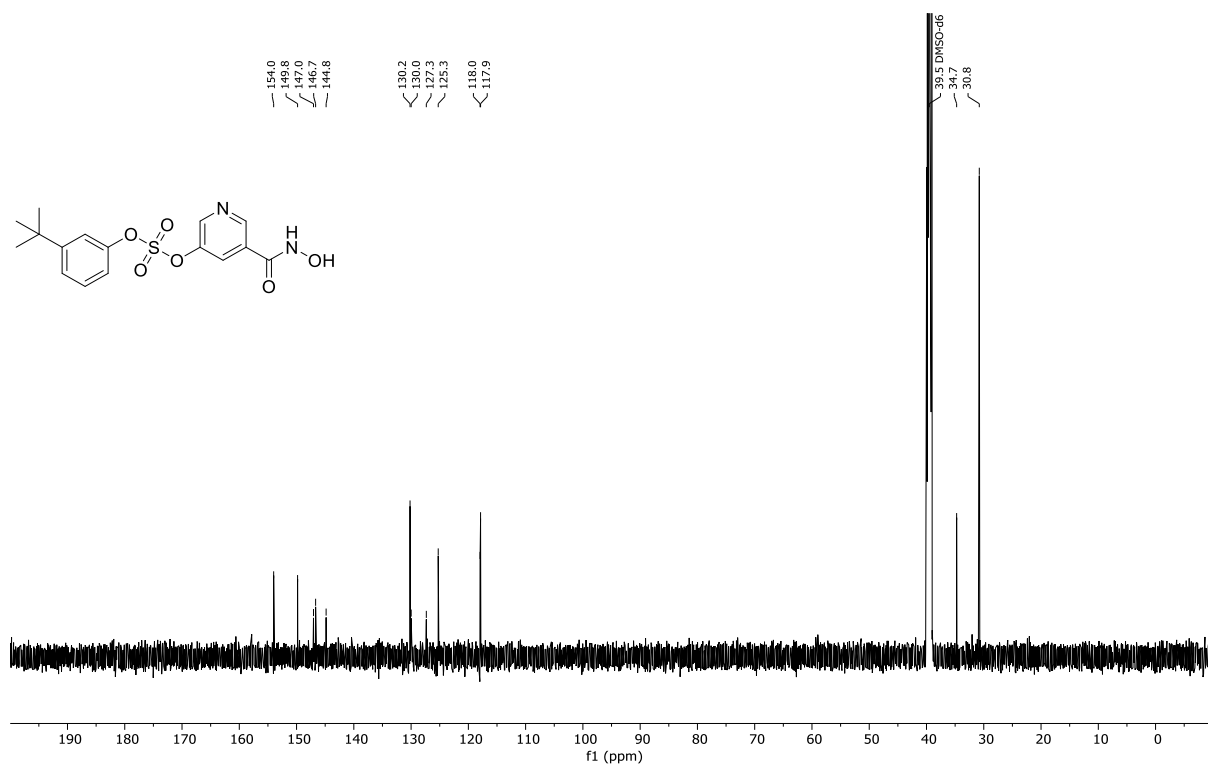
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E9**



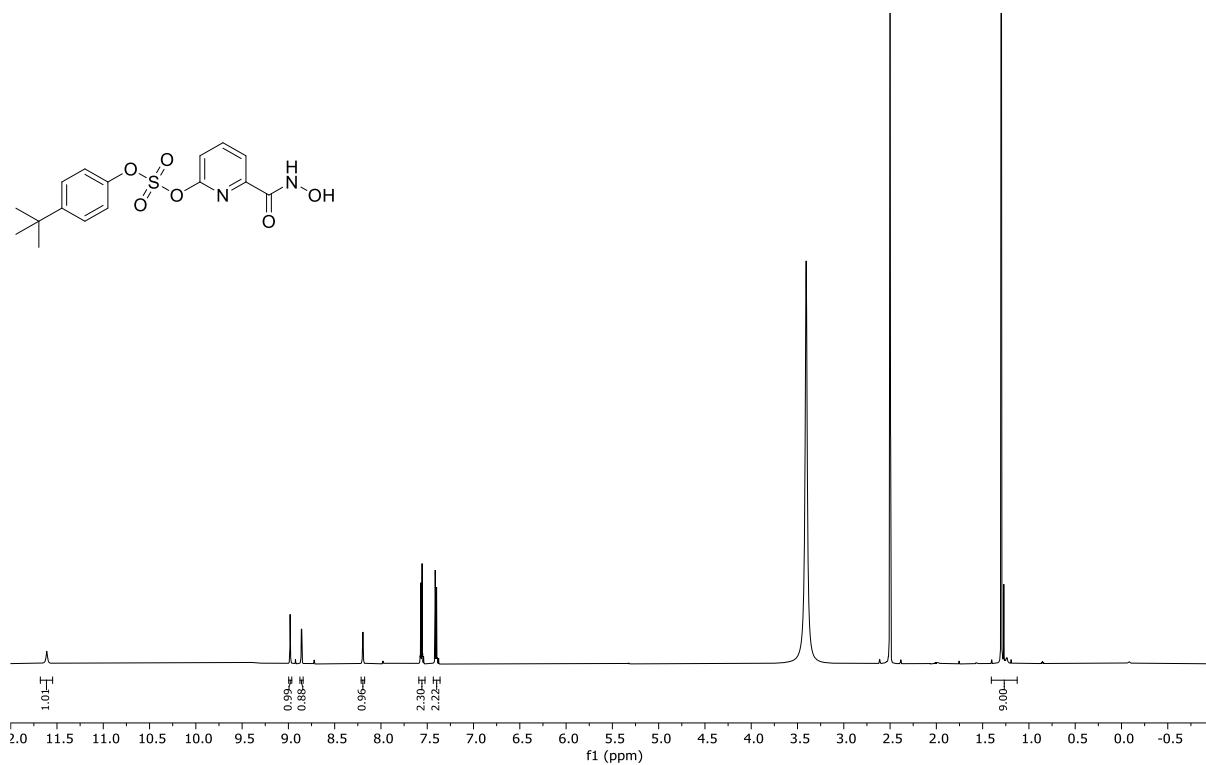
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E11**



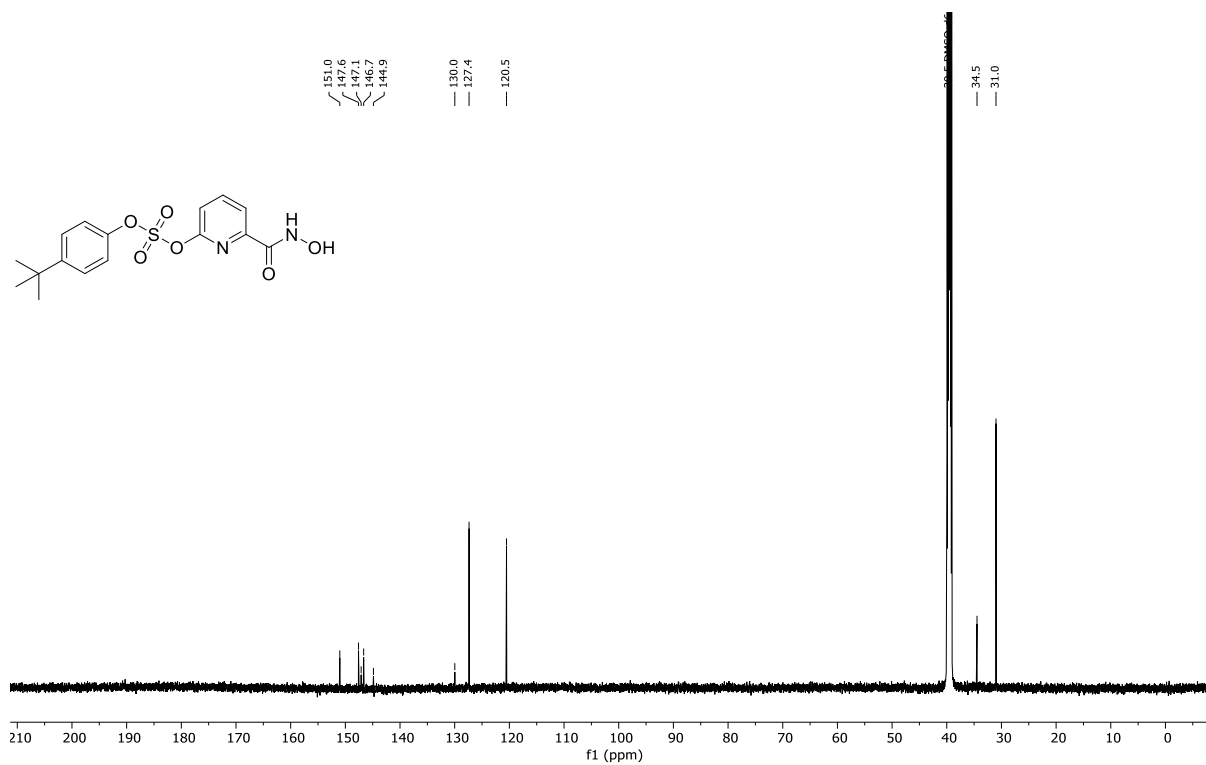
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E11**



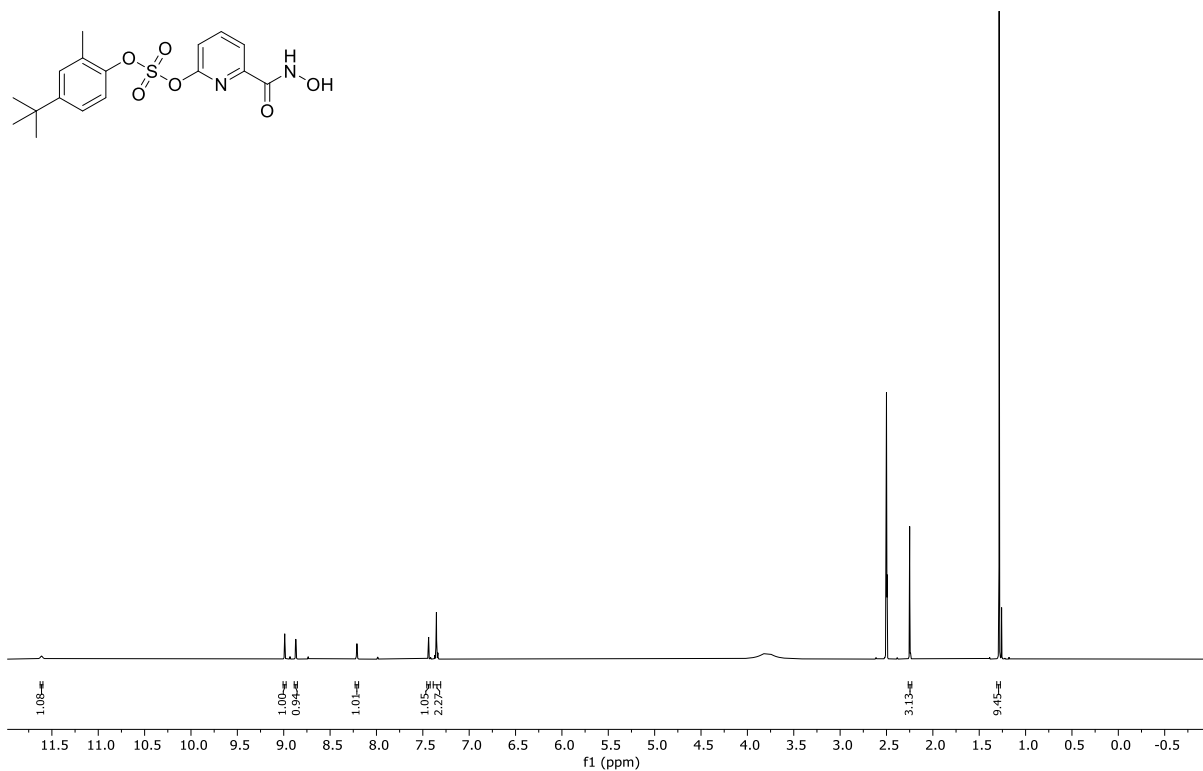
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E12**



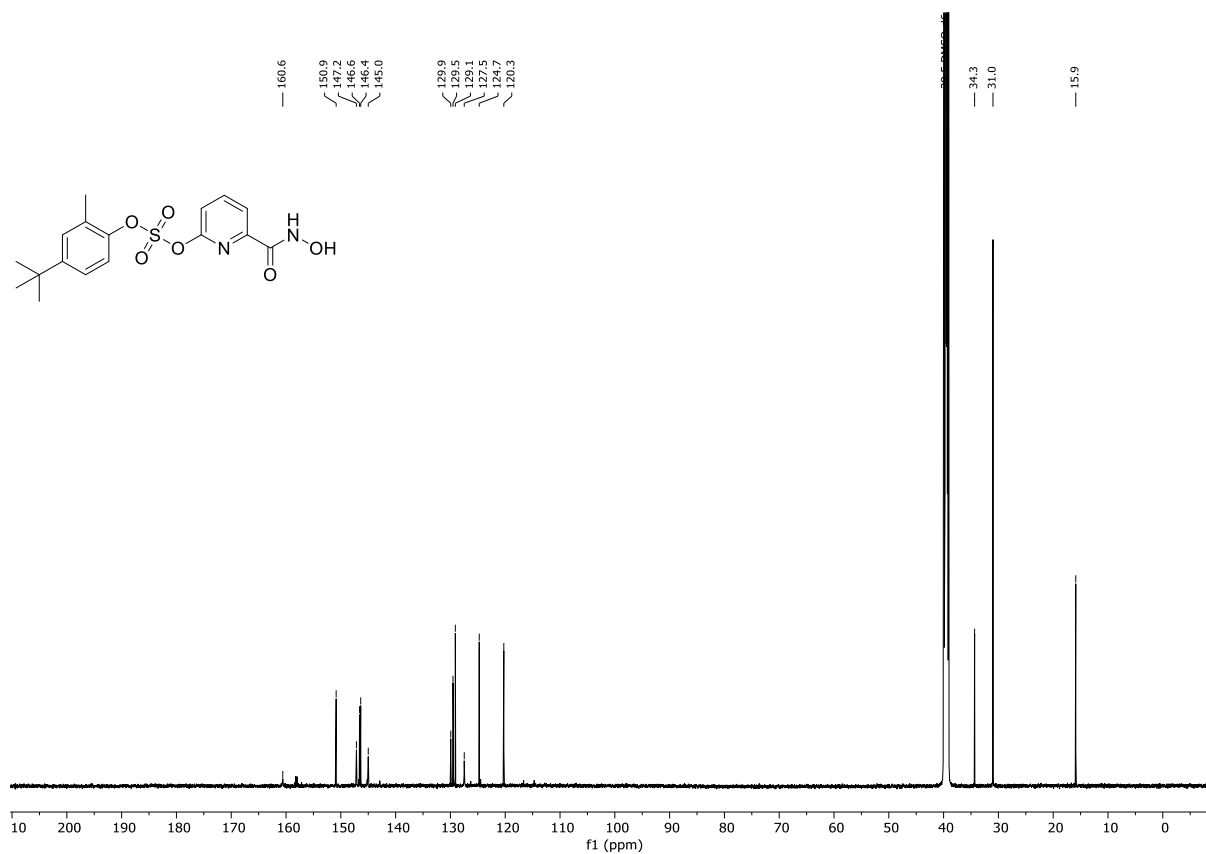
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E12**



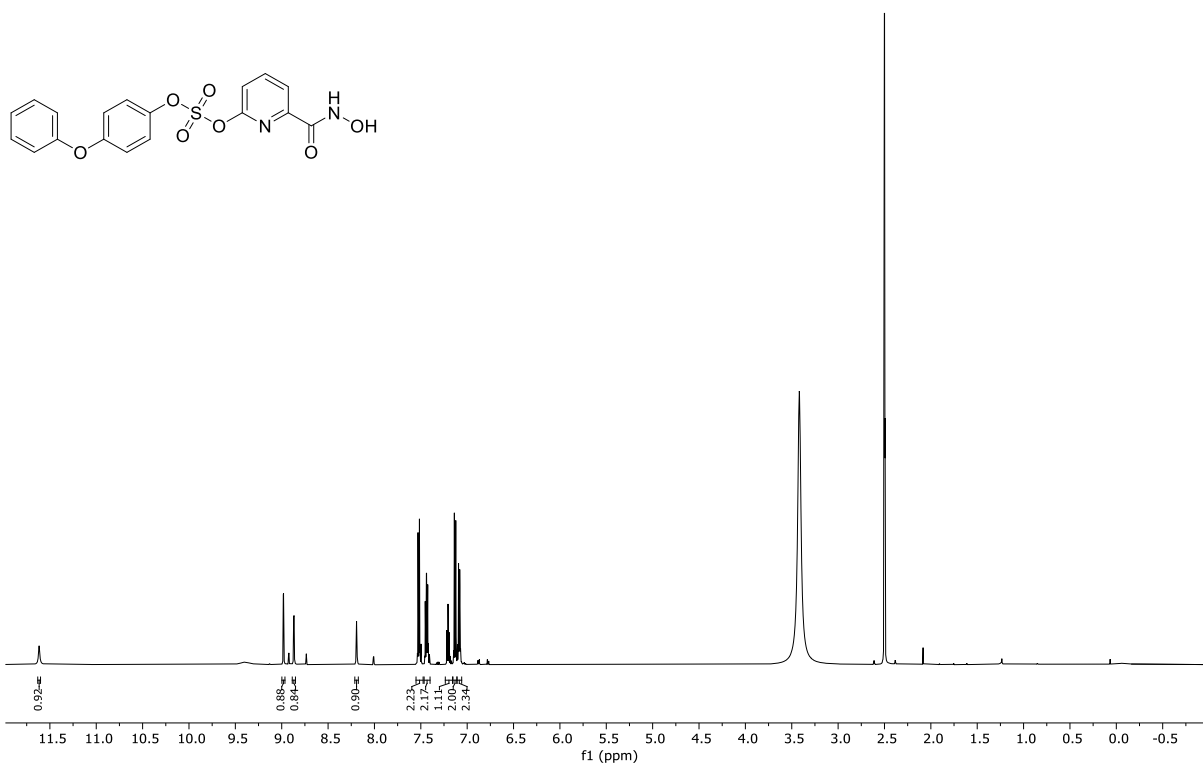
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **E19**



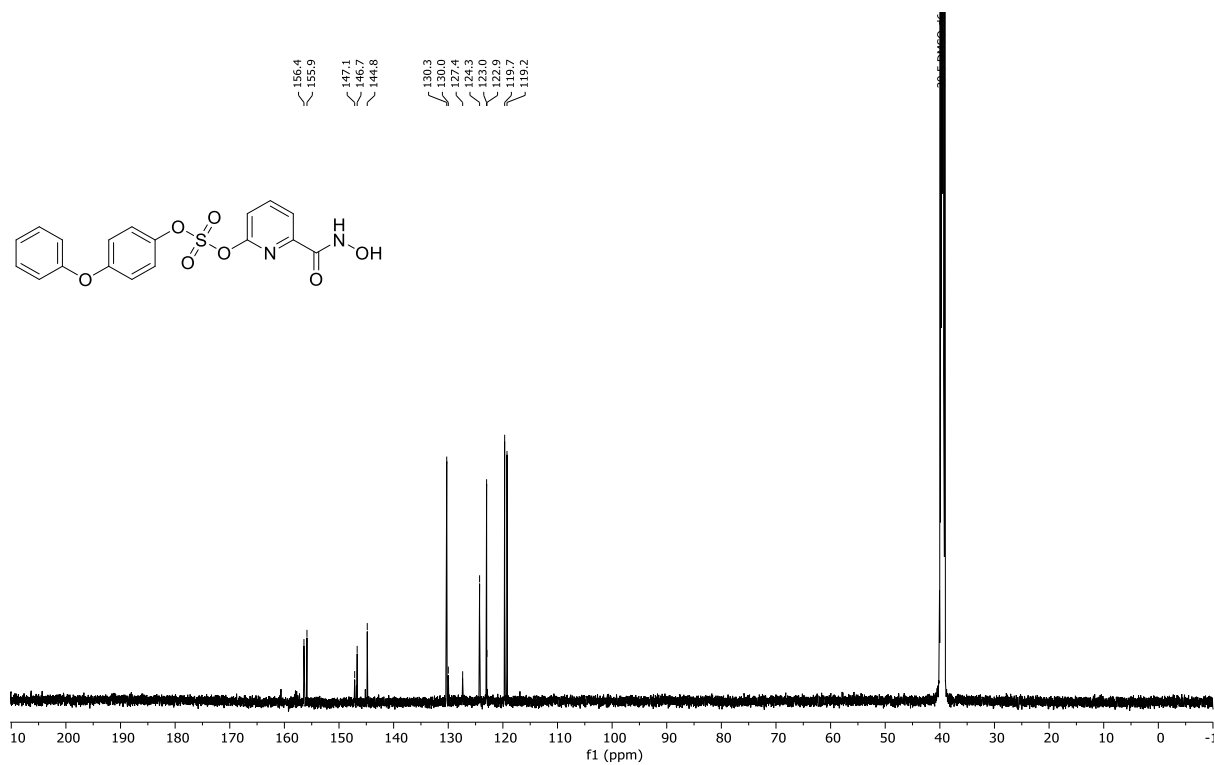
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **E19** (minor contamination with TFA is observed at 158.8 ppm)



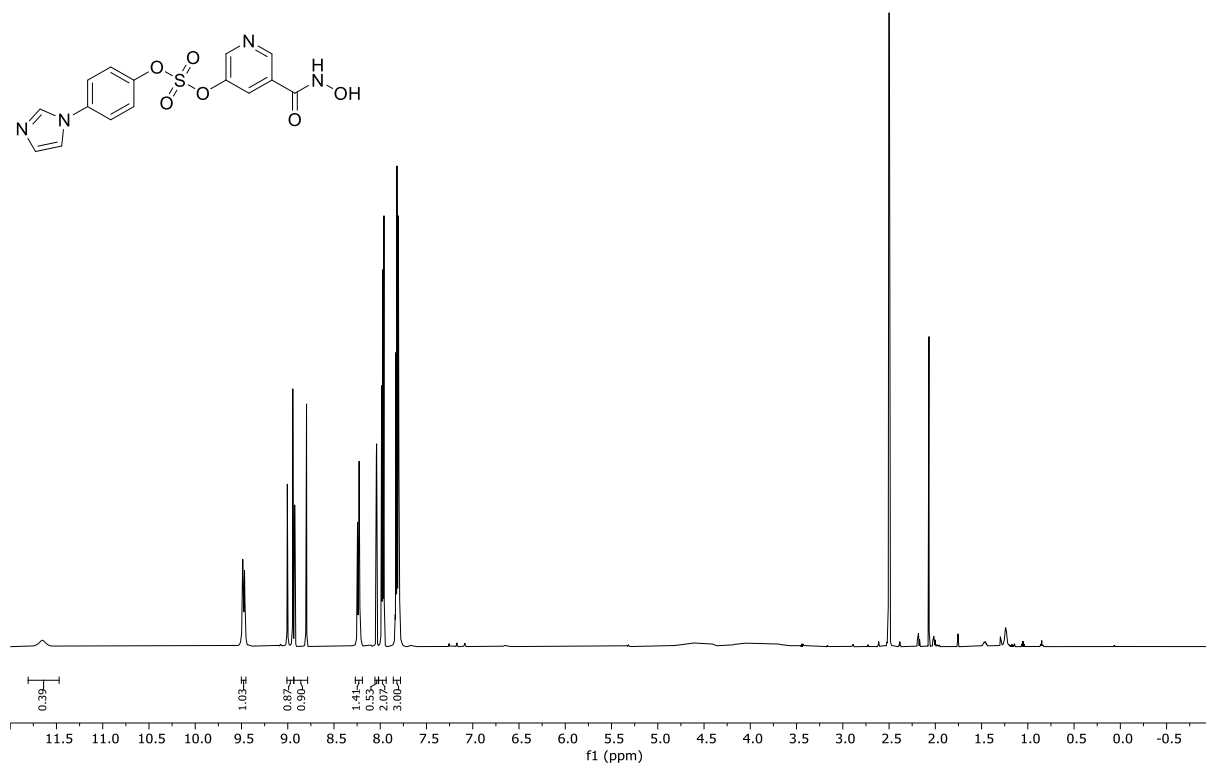
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E21**



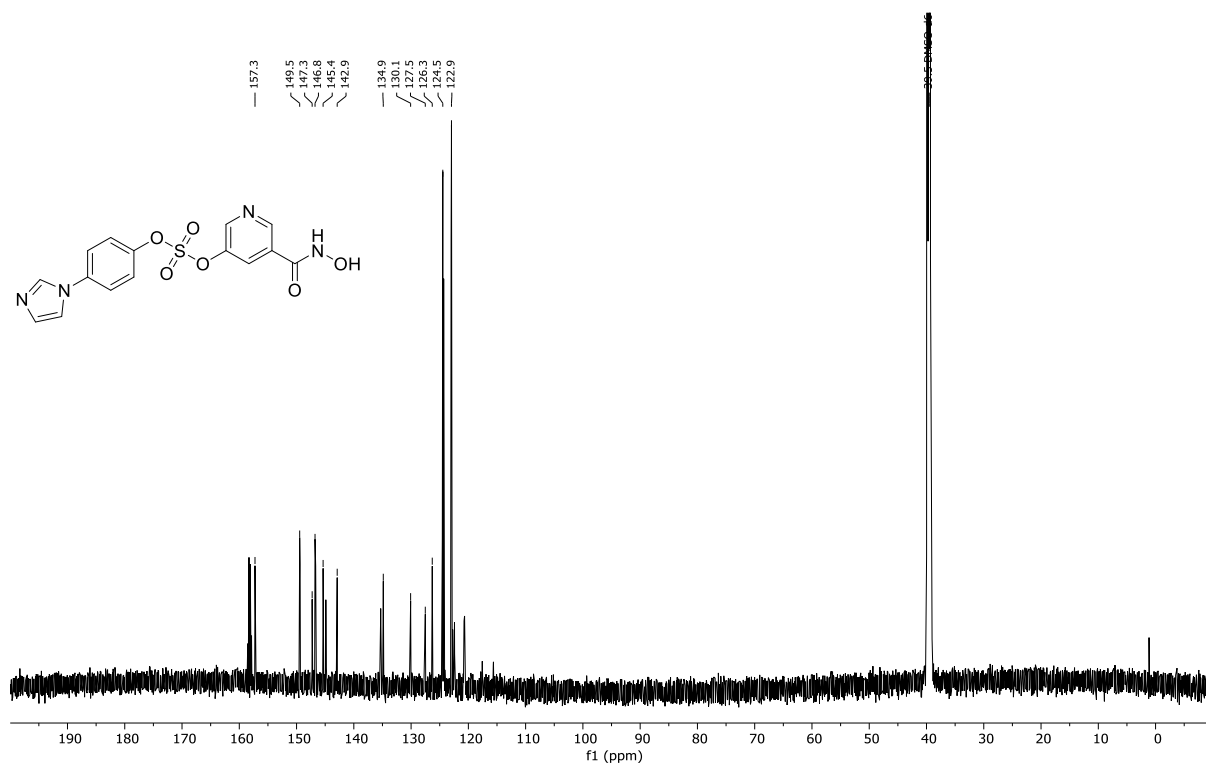
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E21**



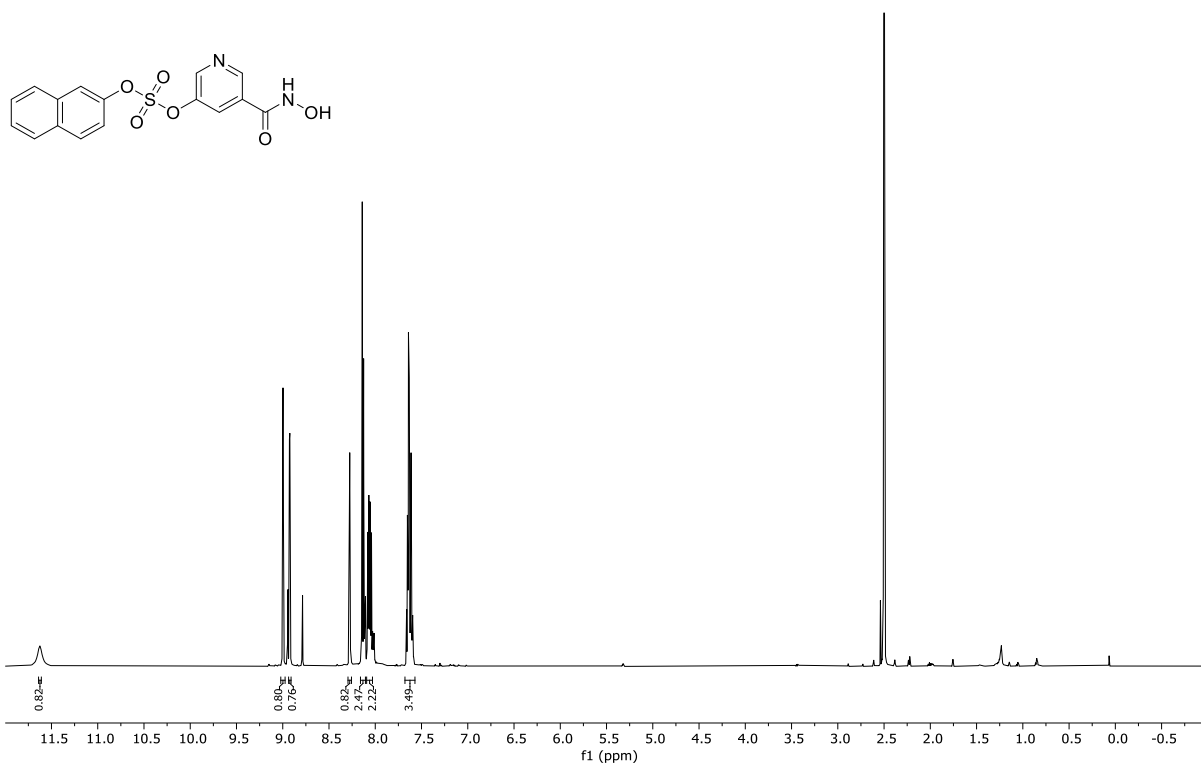
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **E23**



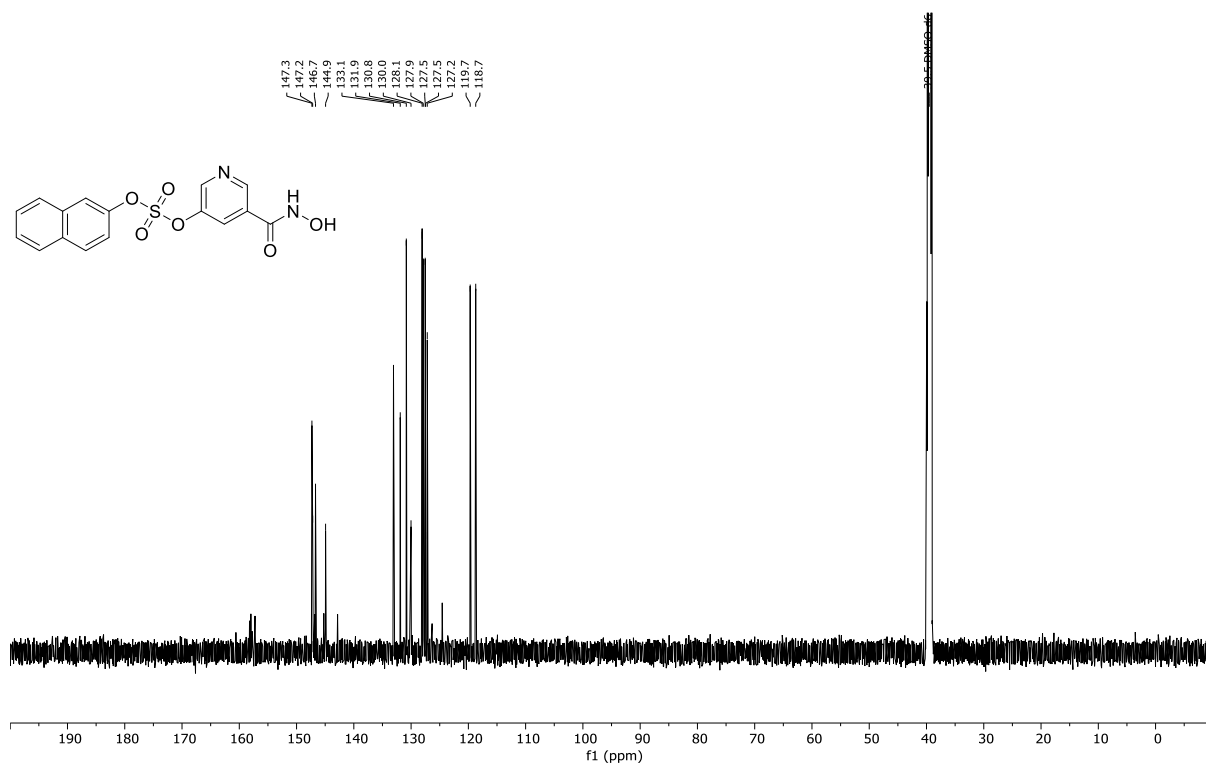
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **E23** (minor contamination with TFA is observed at 158.8 ppm)



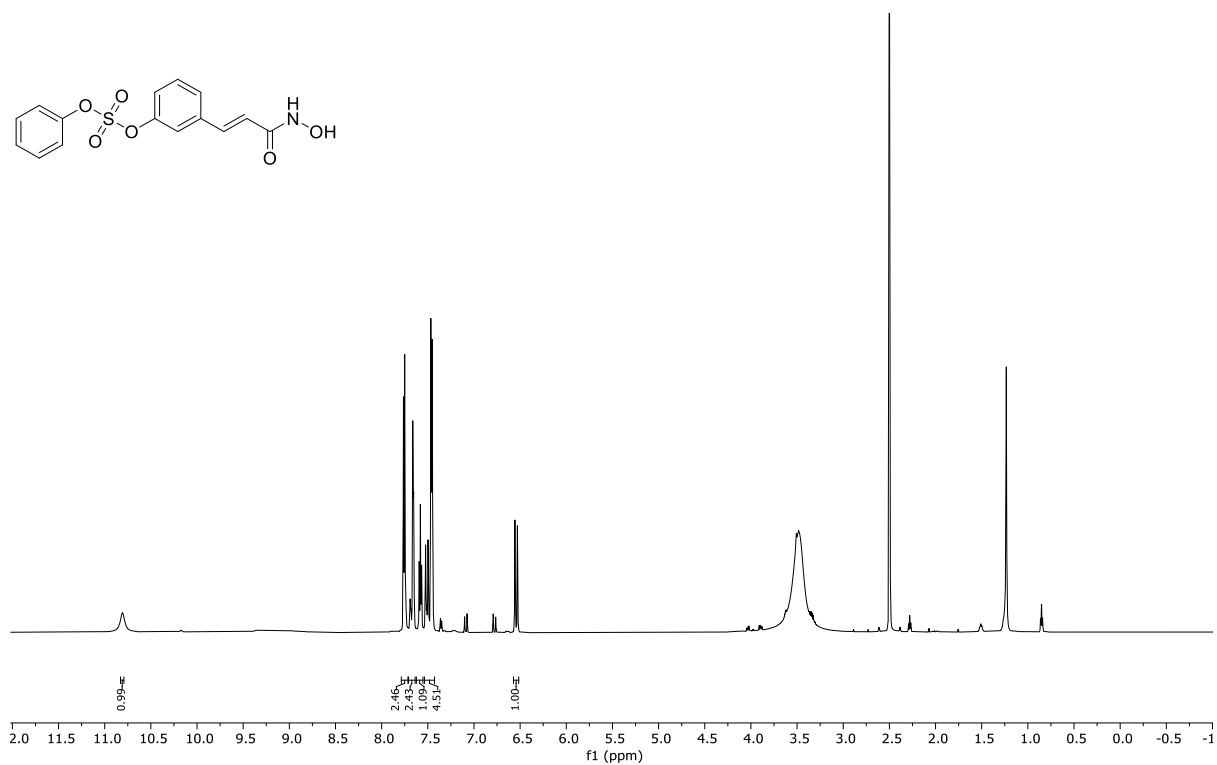
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **E24**



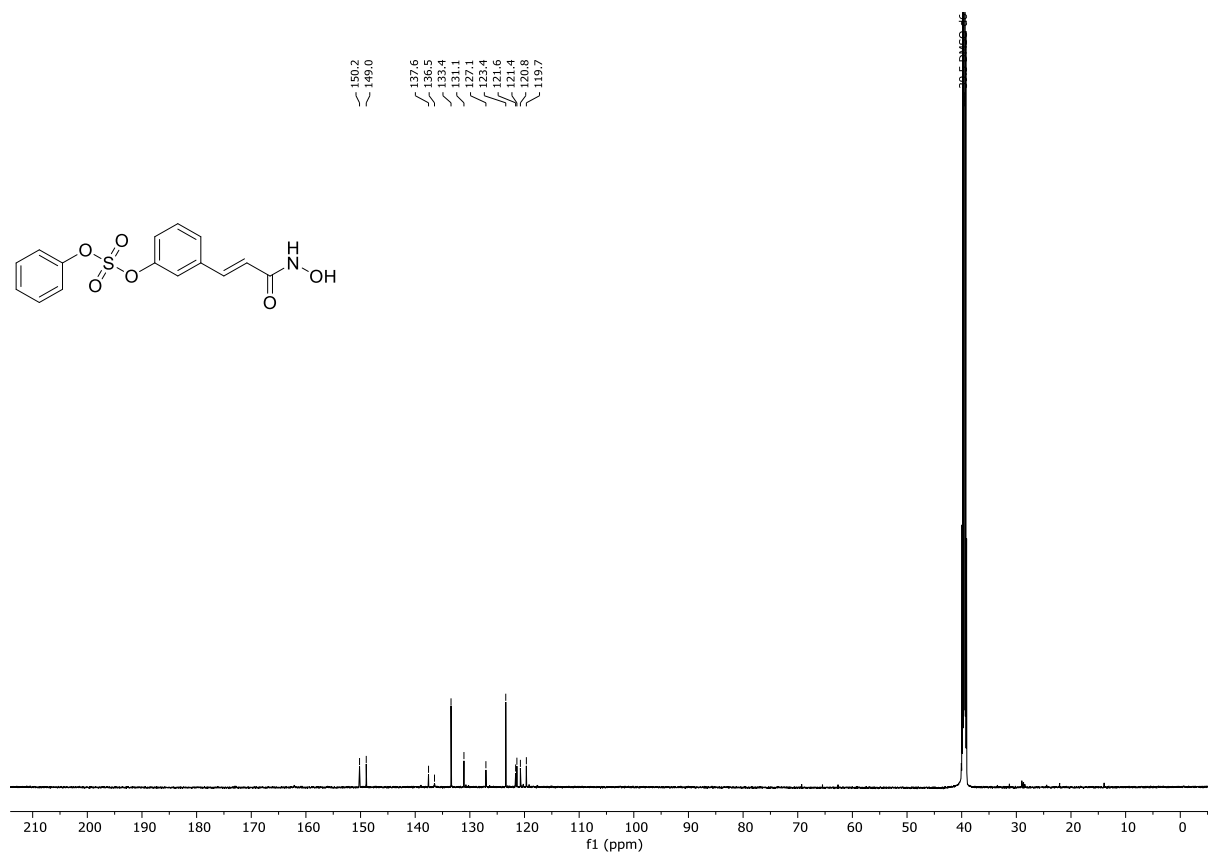
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **E24** (minor contamination with TFA is observed at 158.8 ppm)



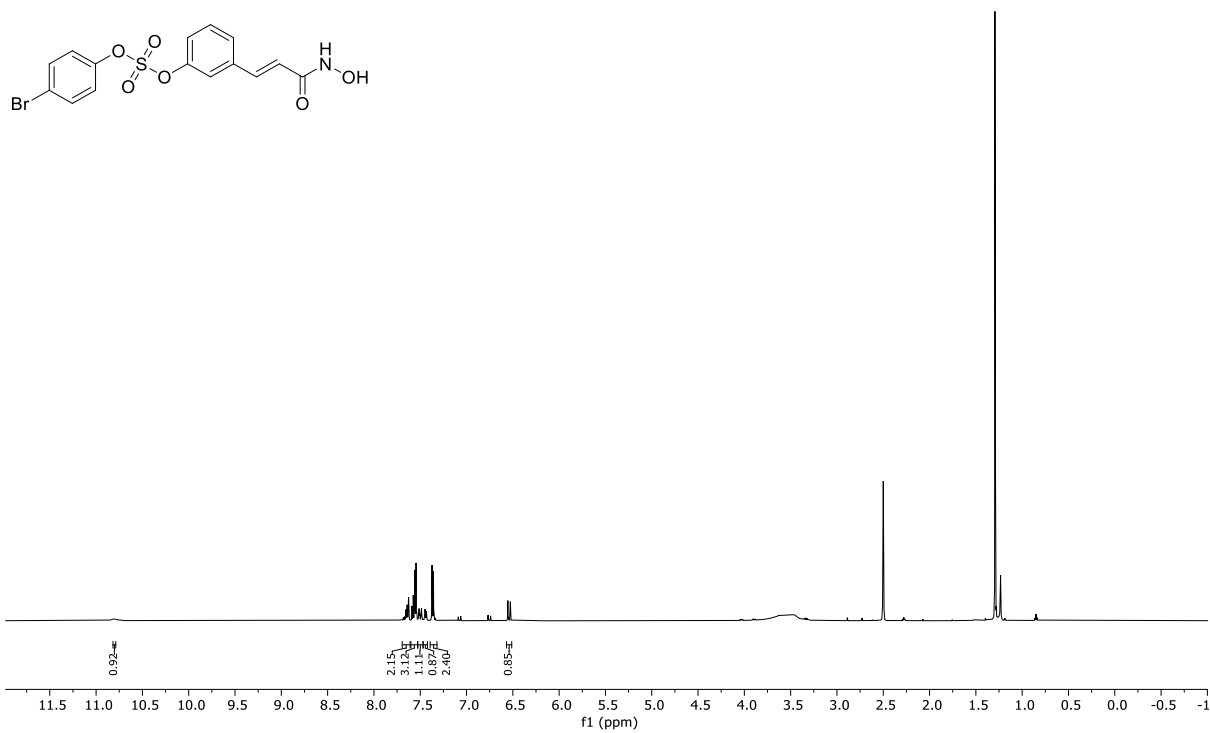
¹H NMR spectra (600 MHz, DMSO-d₆) of compound F1



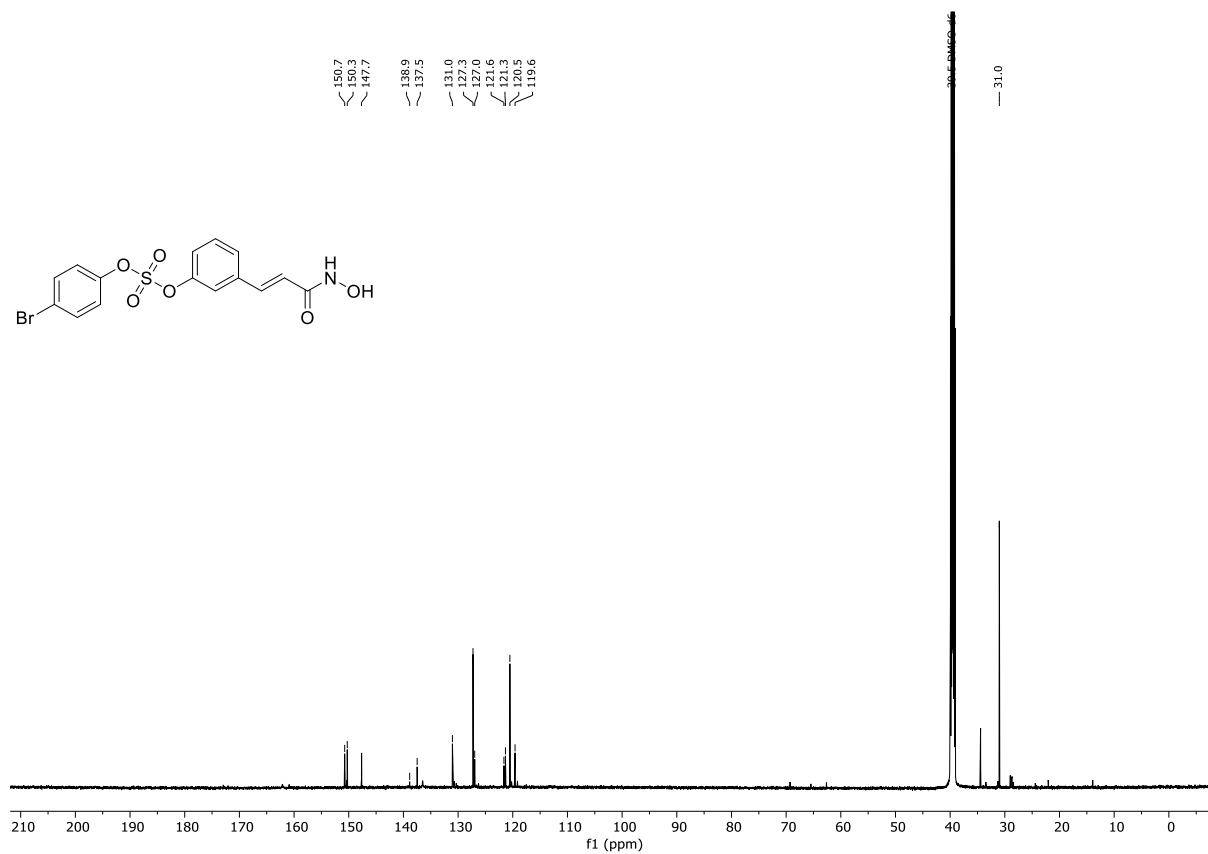
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound F1



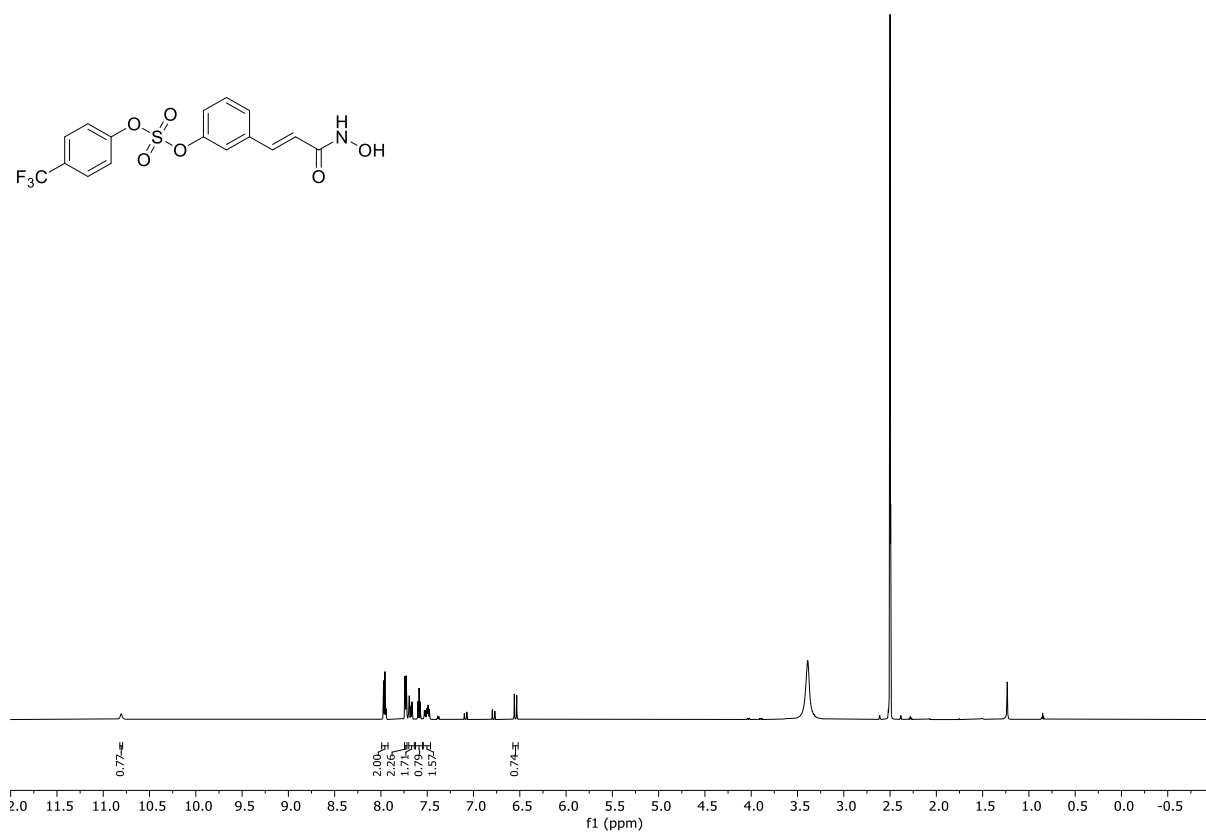
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **F2**



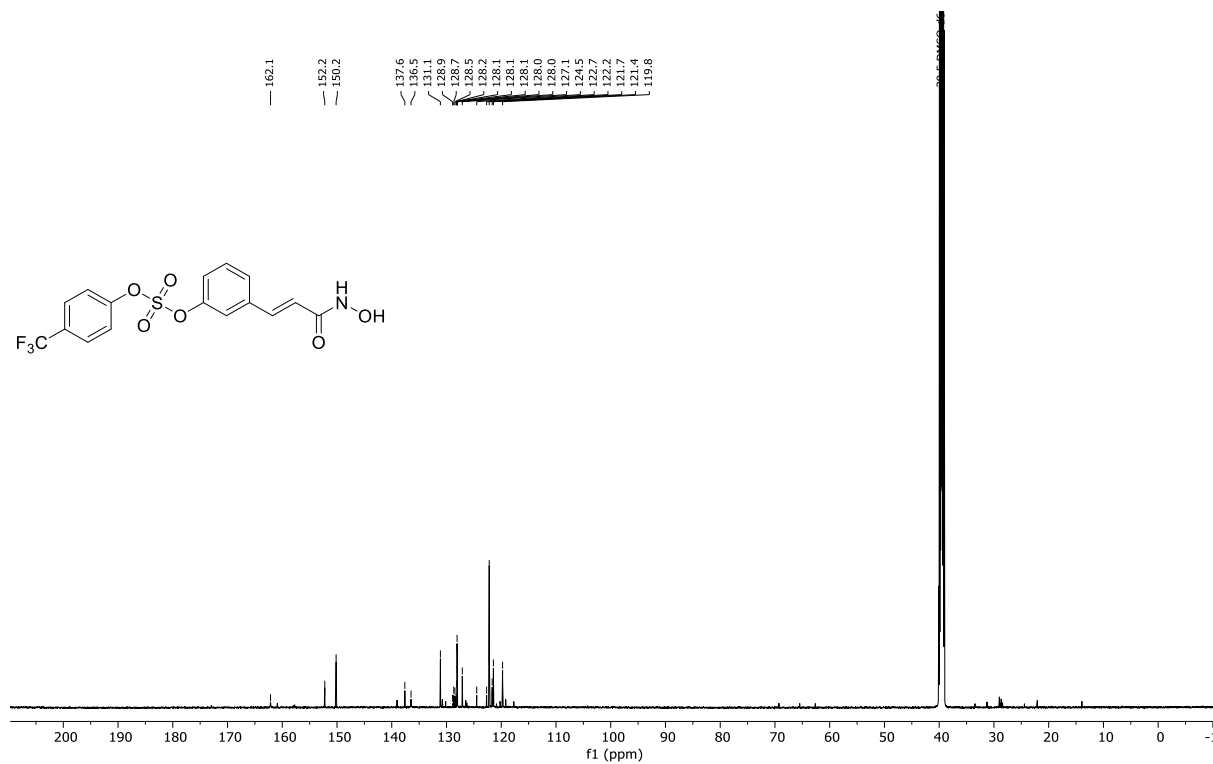
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **F2**



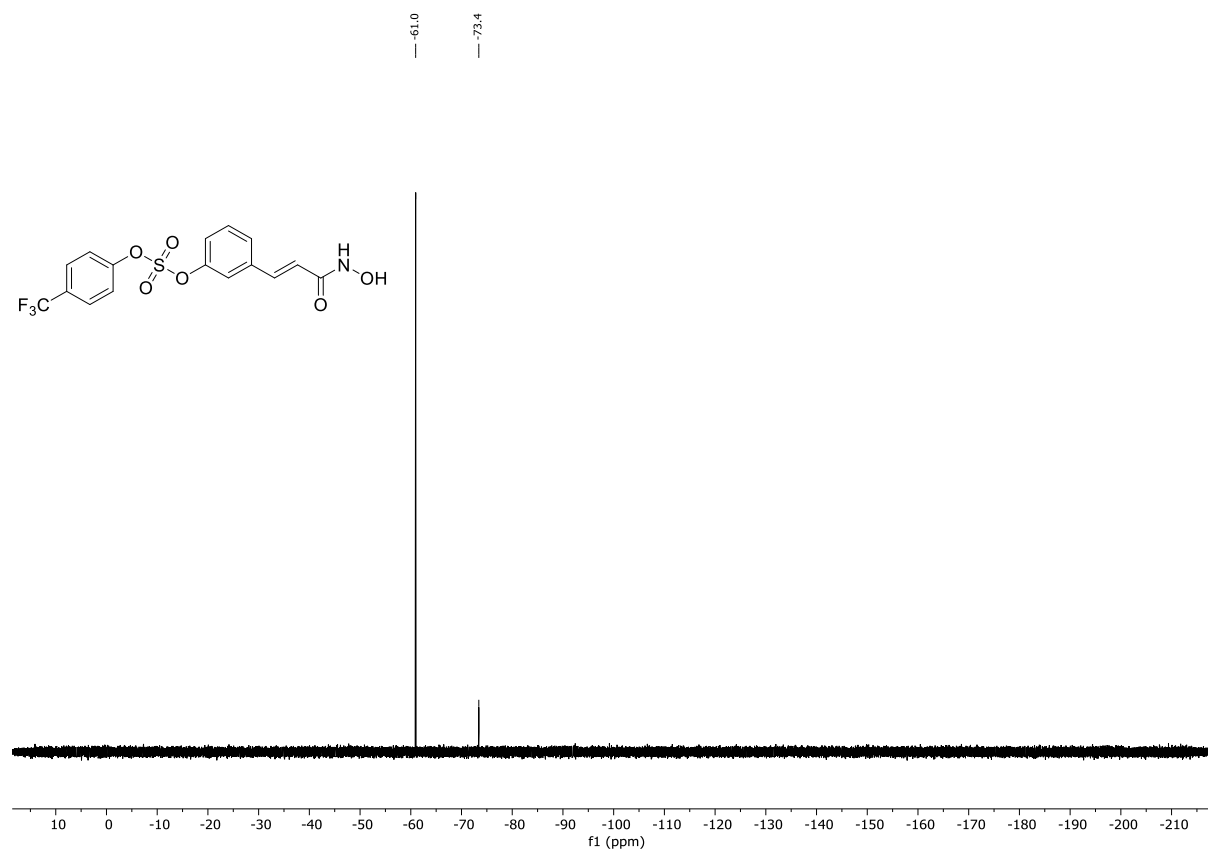
¹H NMR spectra (600 MHz, DMSO-*d*₆) of compound **F3**



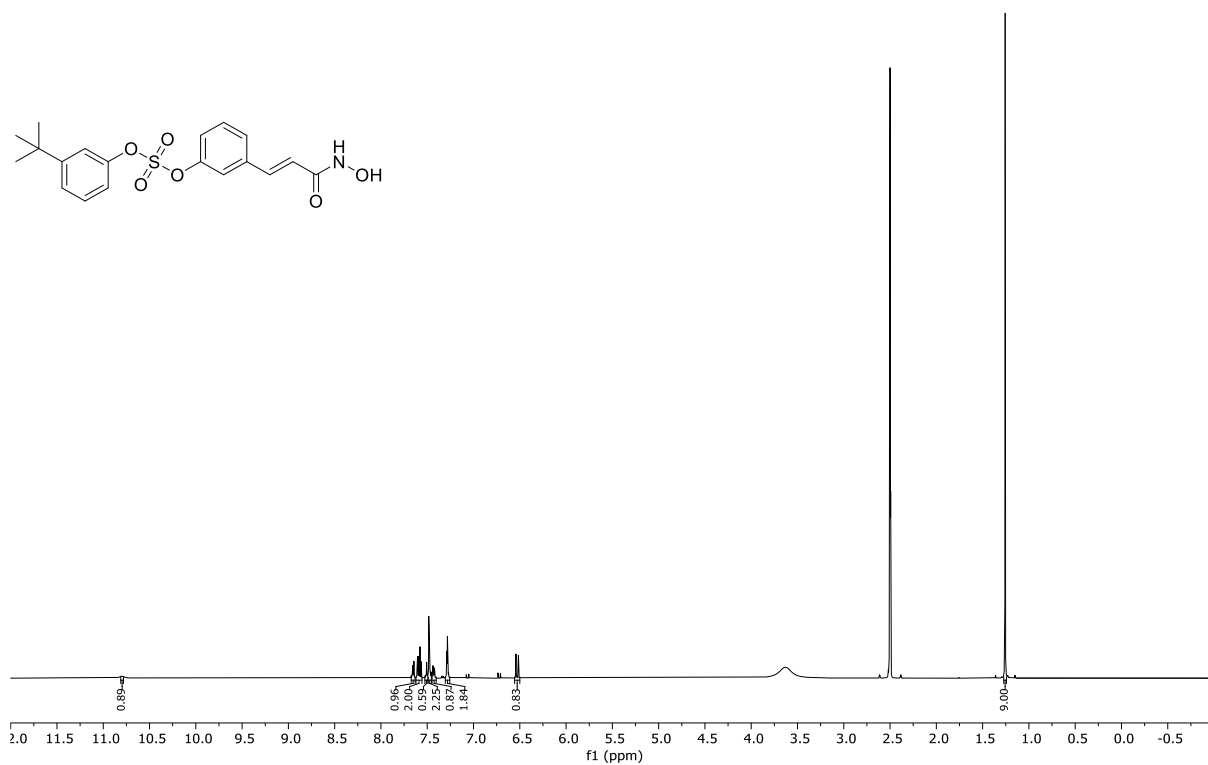
¹³C NMR spectra (151 MHz, DMSO-*d*₆) of compound **F3**



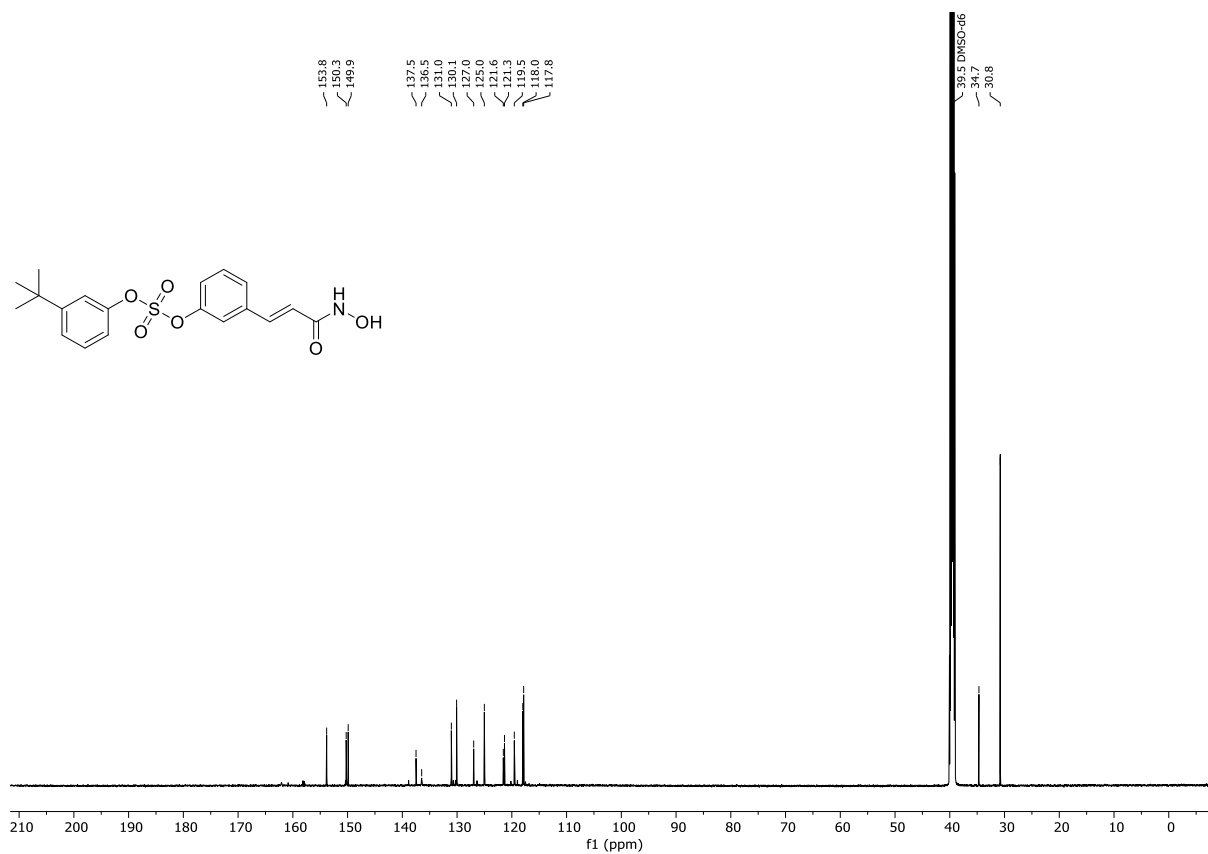
^{19}F NMR spectra (376 MHz, CDCl_3) of compound **F3**



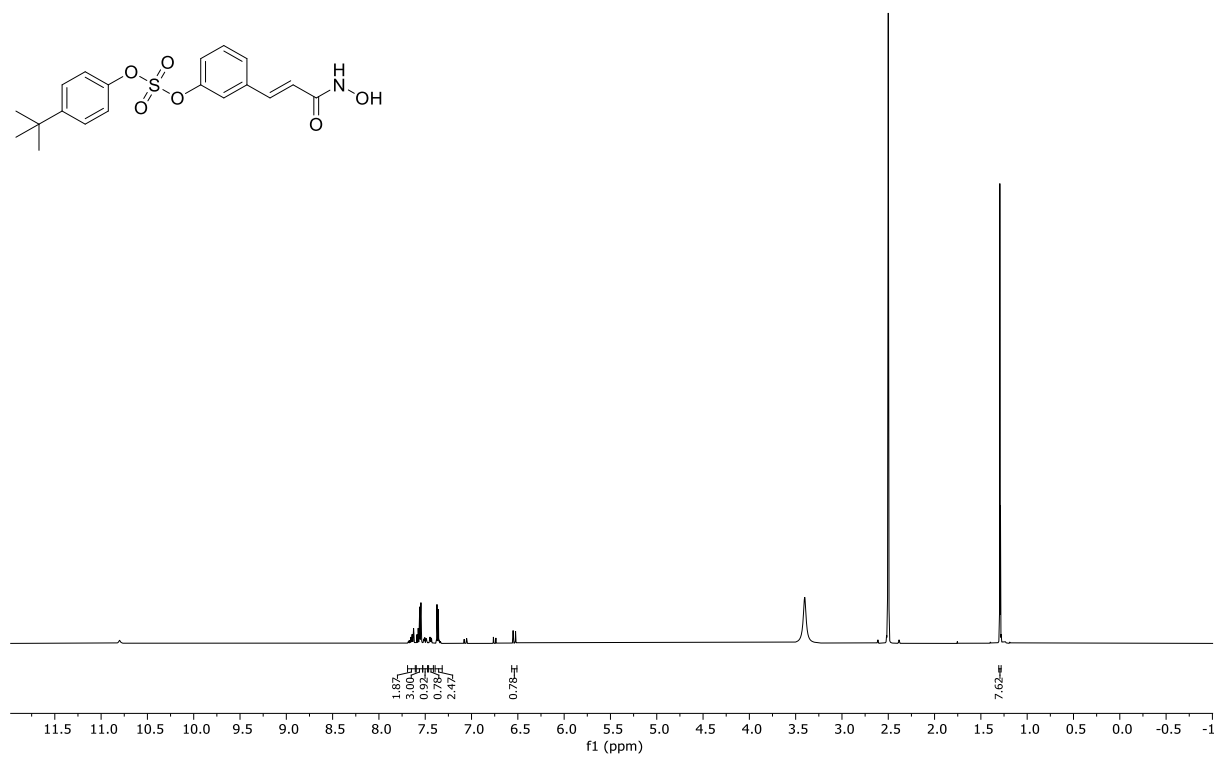
¹H NMR spectra (600 MHz, DMSO-d₆) of compound F11



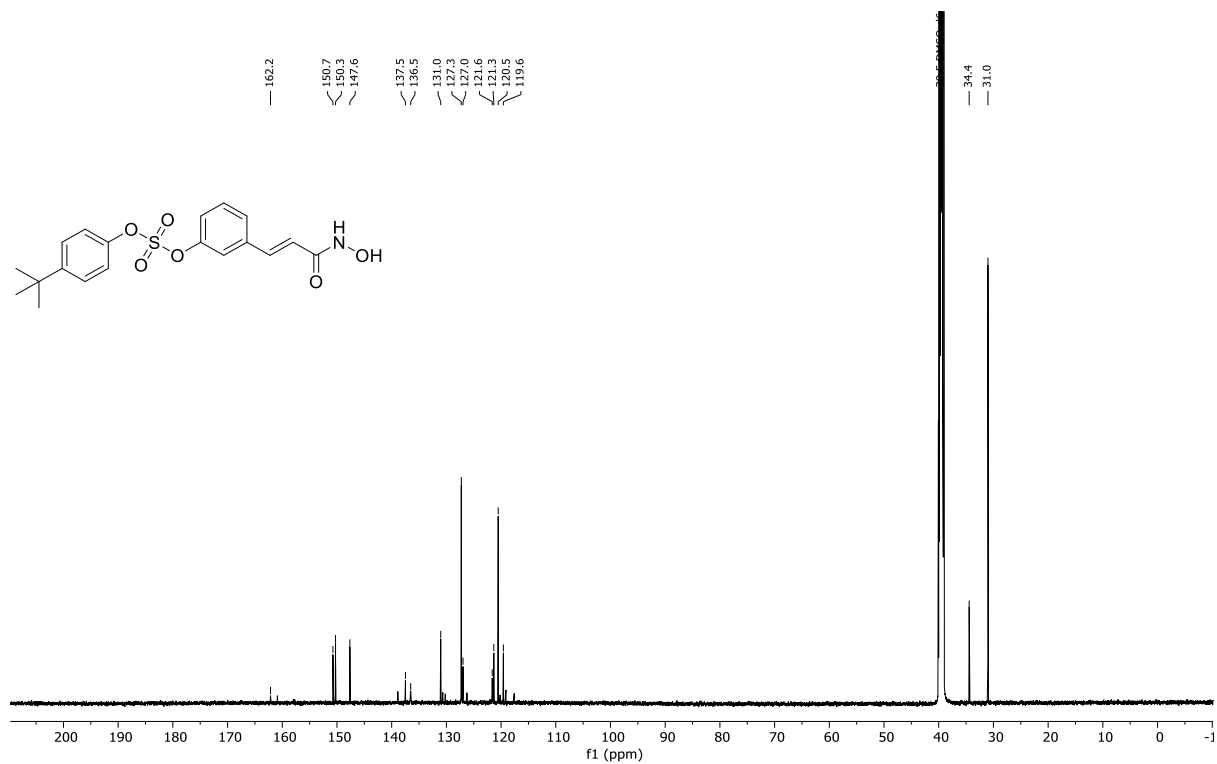
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound F11



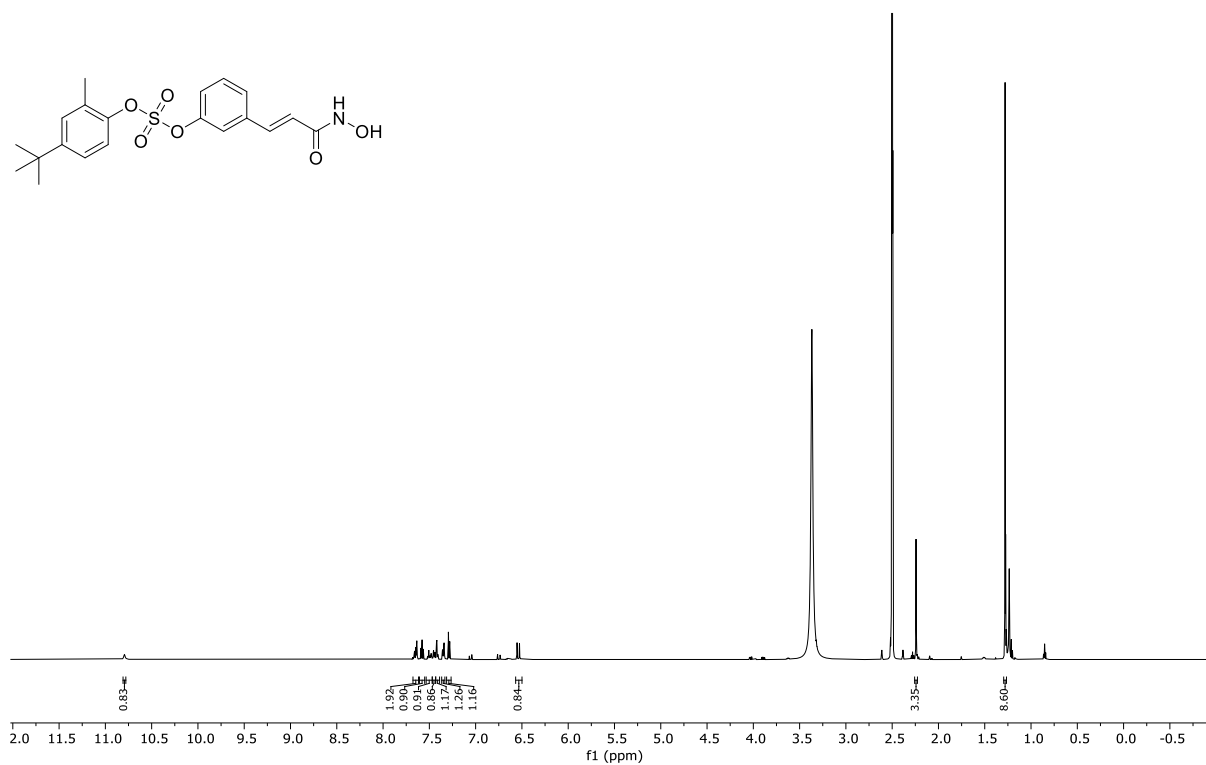
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **F12**



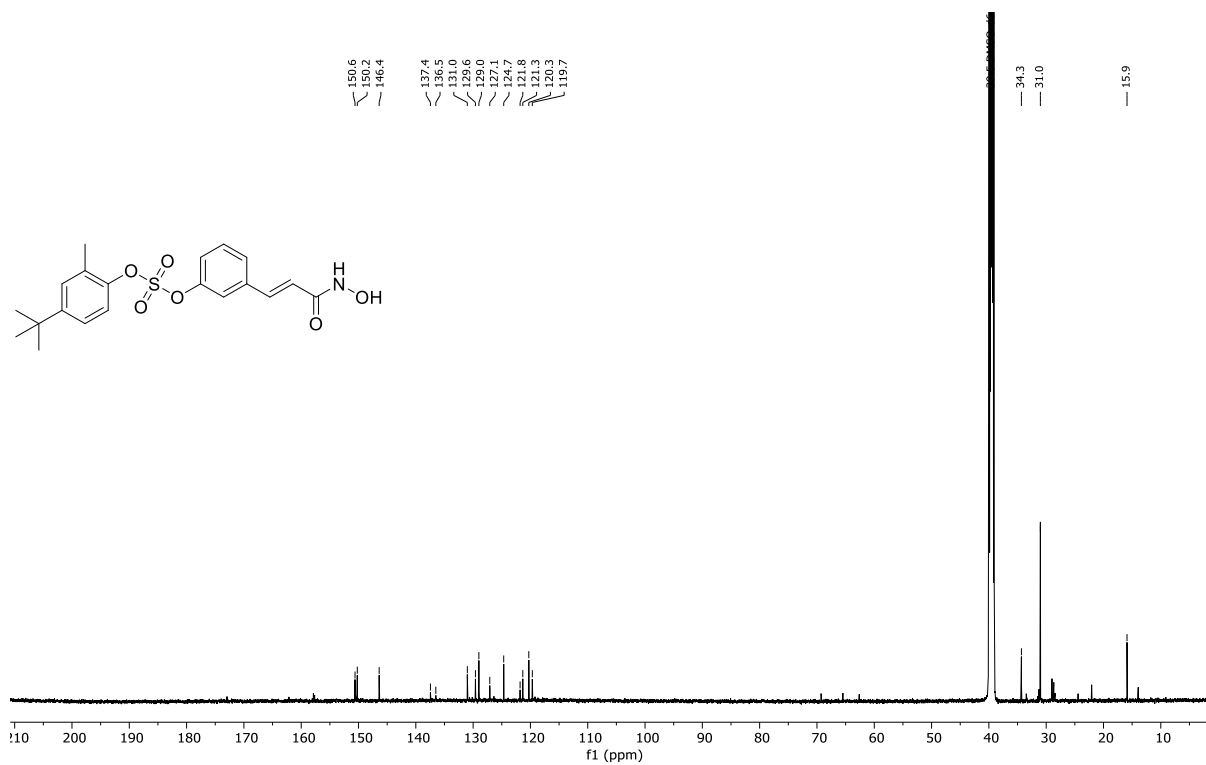
¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **F12**



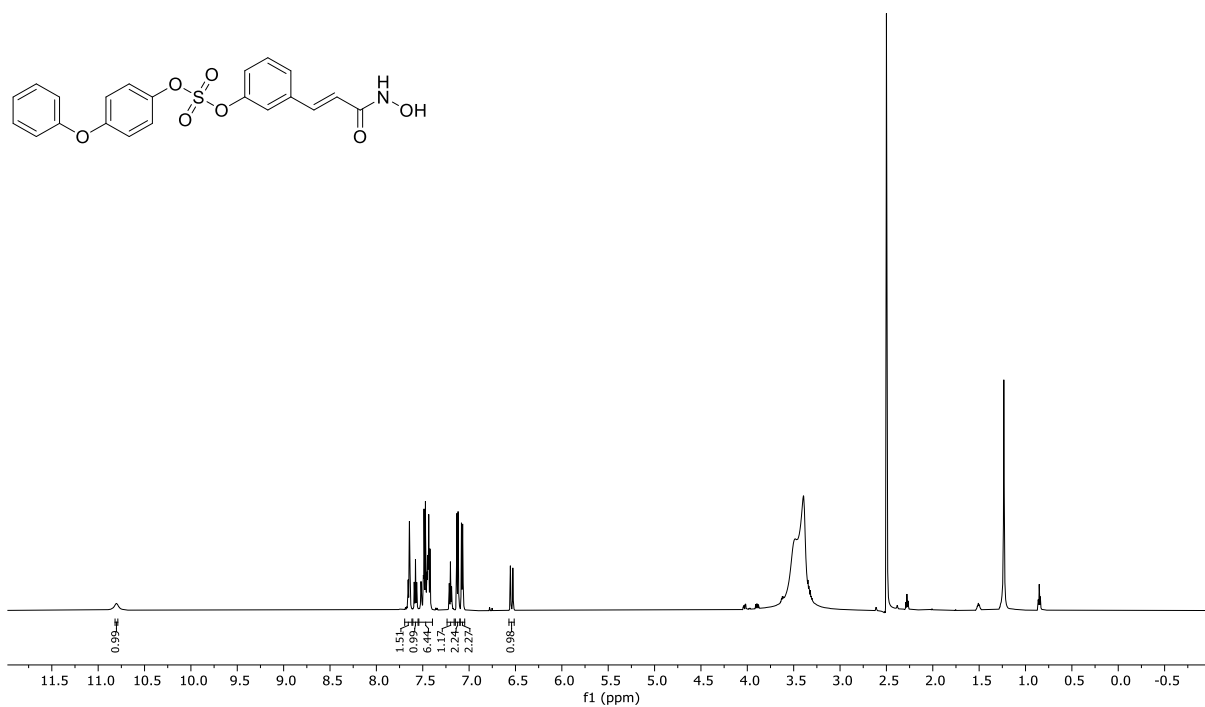
¹H NMR spectra (600 MHz, DMSO-d₆) of compound **F19**



¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **F19**



¹H NMR spectra (600 MHz, DMSO-d₆) of compound **F21**



¹³C NMR spectra (151 MHz, DMSO-d₆) of compound **F21**

