SUPPORTING INFORMATION

Tetrasubstitued Cyclopentadienones as Suitable Enantiopure Ligands with Axial Chirality

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Content:

Table S1 Computed energy in kcal/mol at B3LYP/6-31G(d) level for compounds 1a-e.

Compounds			*E*	1 aryl rotation		3 aryl rotation	
				TS-(0°)	**TS-(180°)**	**TS-(0°)**	**TS-(180°)**
1a	**(1*P*,3*M*)**	*in-in*	1.252	(*in*) 17.9_3 (*out*) 17.9_4	(*in*) 24.8 (*out*) 26.3	-	-
		in-out	0.216				
		out-out	0.439				
	(1*P*,3*P*)	*in-in*	0.432				
		in-out	0.795				
		out-out	0.000				
1b	**(1*P*,3*M*)**	*in-in*	0.973	(*in*) 19.6 (*out*) 19.9	(*in*) 26.2 (*out*) 27.2	-	-
		in-out	0.714				
		out-out	-[a]				
	(1*P*,3*P*)	*in-in*	0.000				
		in-out	-[a]				
		out-out	1.370				
1c	**(1*P*,3*M*)**	*in-in*	-[a]	(3*in*) 19.6_1 (3*out*) 19.6_4	(3*in*) 26.2 (3*out*) 27.9	(1*in*) 17.7 (1*out*) 18.3	(1*in*) 24.7 (1*out*) 25.5
		1*in*-3*out*	0.000				
		1*out*-3*in*	0.864				
		out-out	0.947				
	(1*P*,3*P*)	*in-in*	0.185				
		1*in*-3*out*	0.605				
		1*out*-3*in*	1.322				
		out-out	0.640				
1d	**(1*P*,3*M*)**	3*in*	0.343	(3*in*) 33.3 (3*out*) 34.7	(3*in*) 31.8 (3*out*) 33.4	18.3	26.6
		3*out*	0.296				
	(1*P*,3*P*)	3*in*	0.643				
		3*out*	0.000				
1e	**1P**	GS1	0.000	(1) 32.4 (2) 35.9	(1) 31.1 (2) 34.7	-	-
		GS2	0.487				

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[a] Conformation not found.

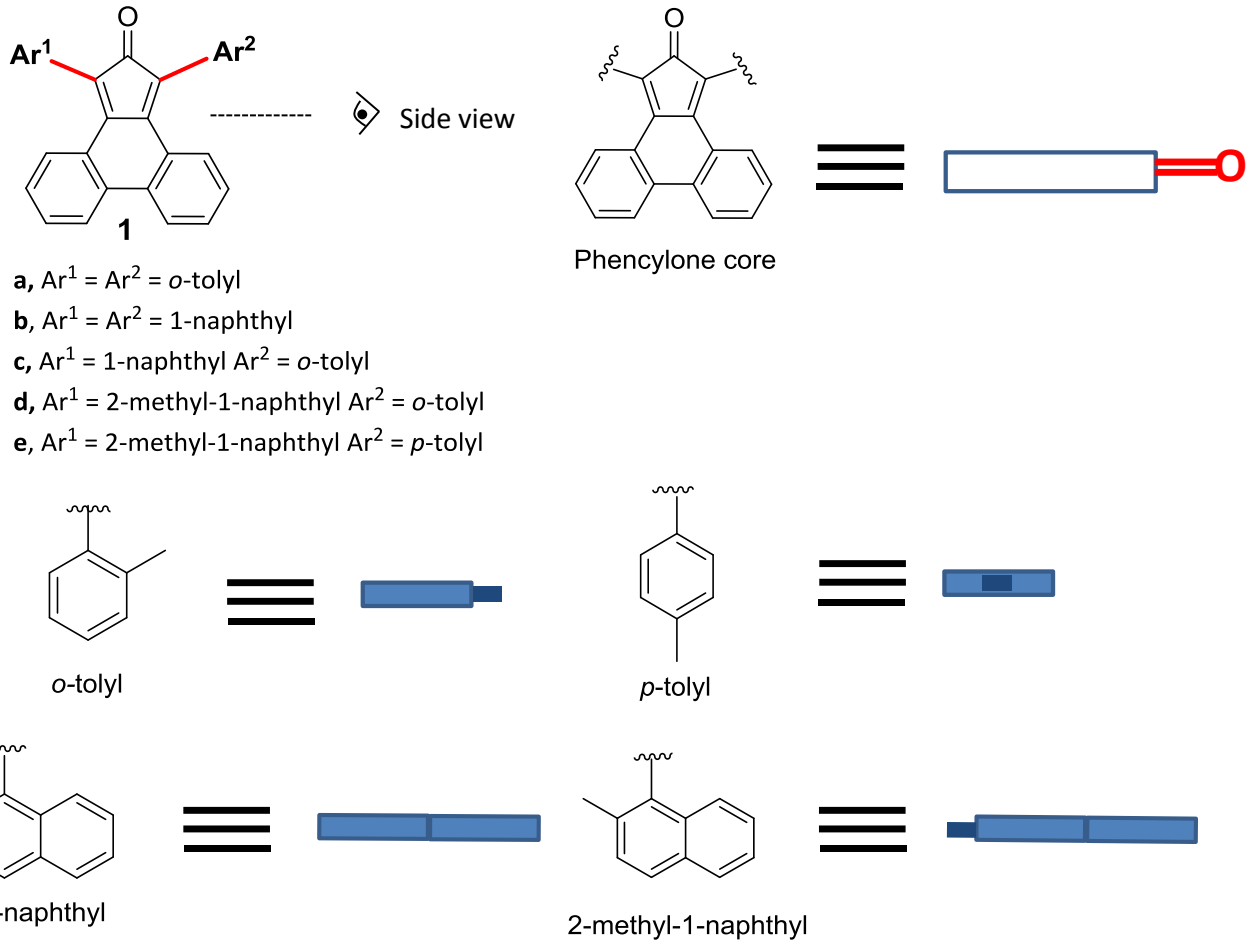


Figure S1 Legend for the conformational schemes of the phencyclones

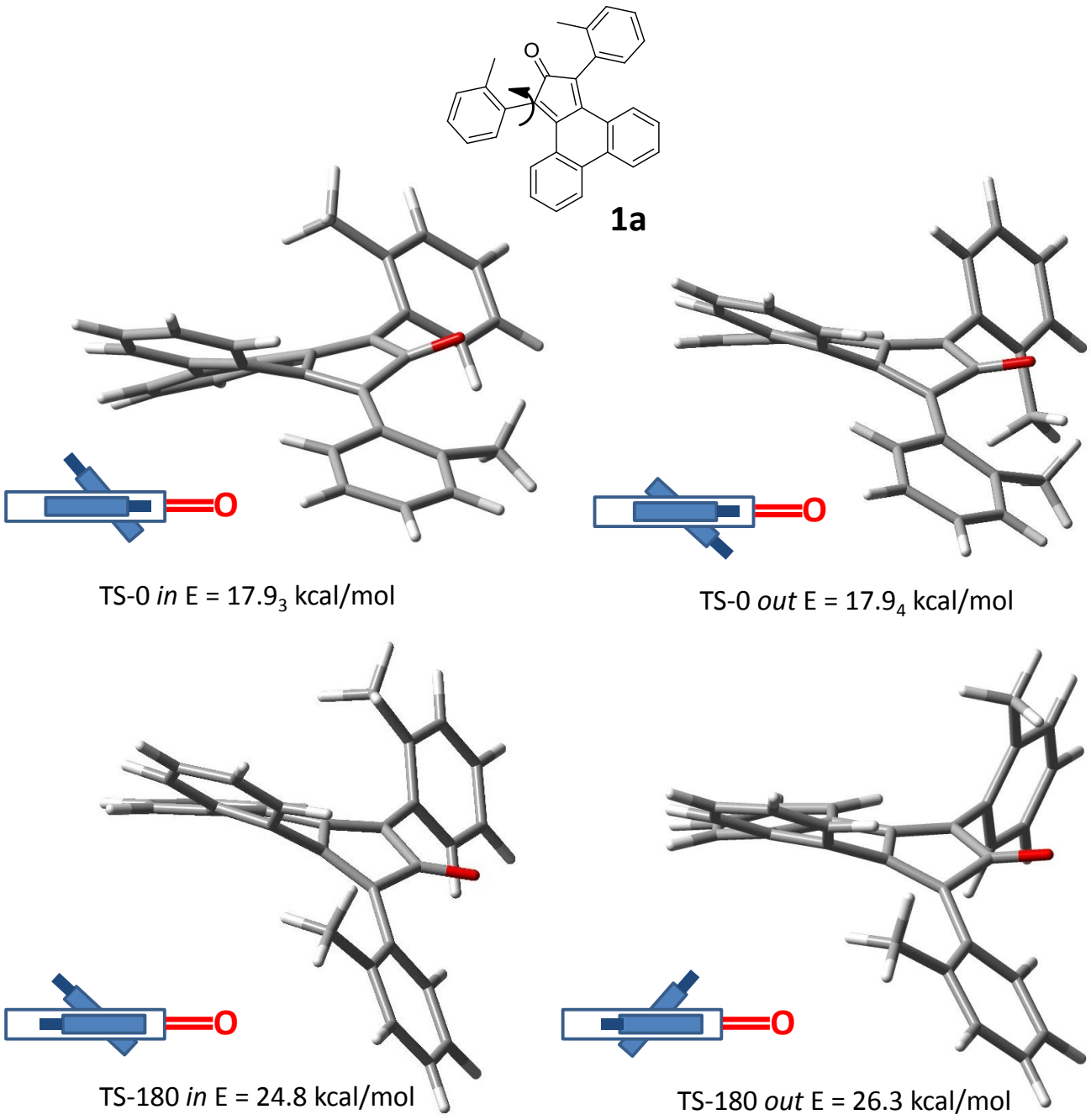


Figure S2 Transition states of 1a optimized at B3LYP/6-31G(d) level.

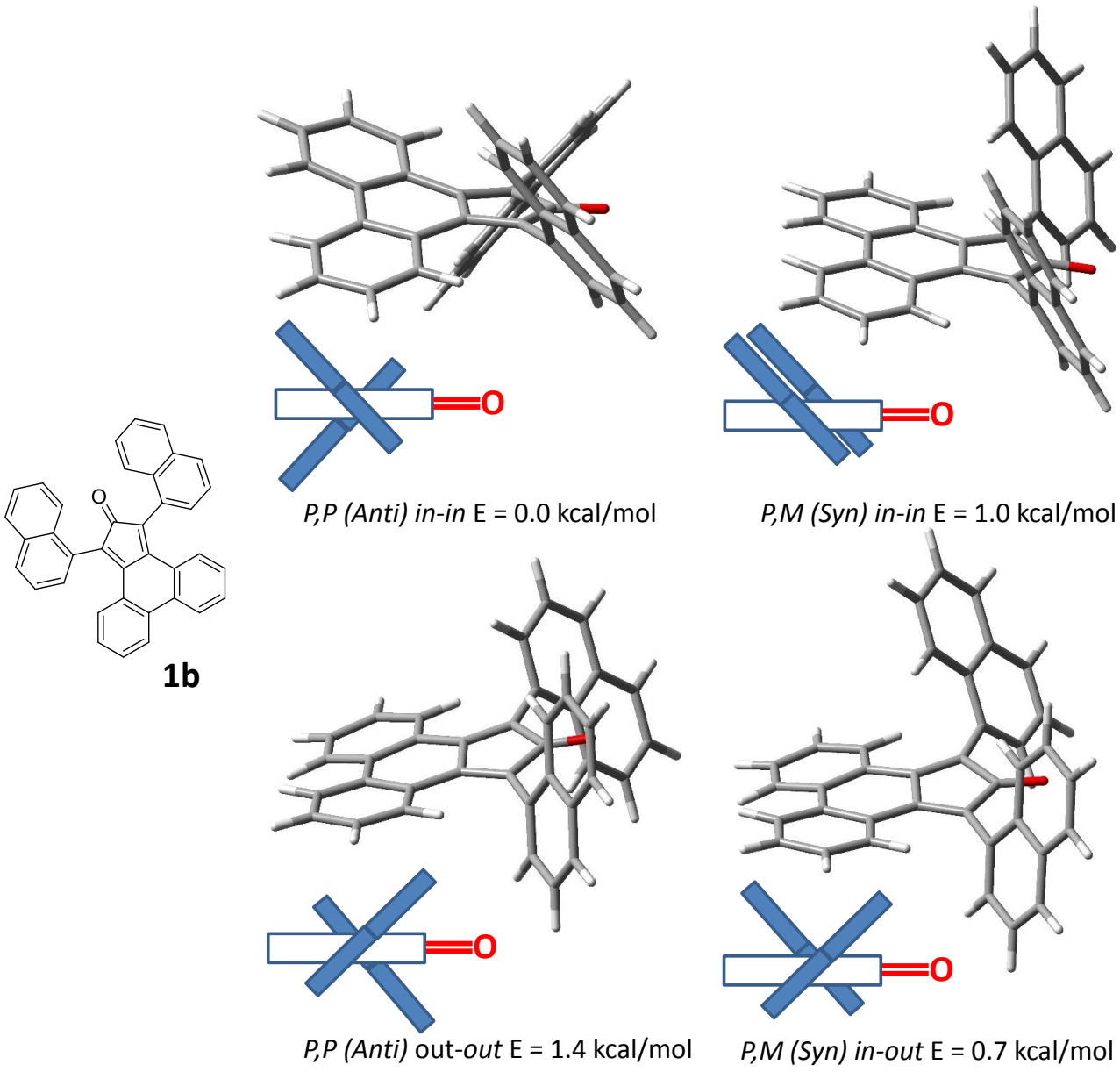


Figure S3 Most stable ground states of 1b optimized at B3LYP/6-31G(d) level.

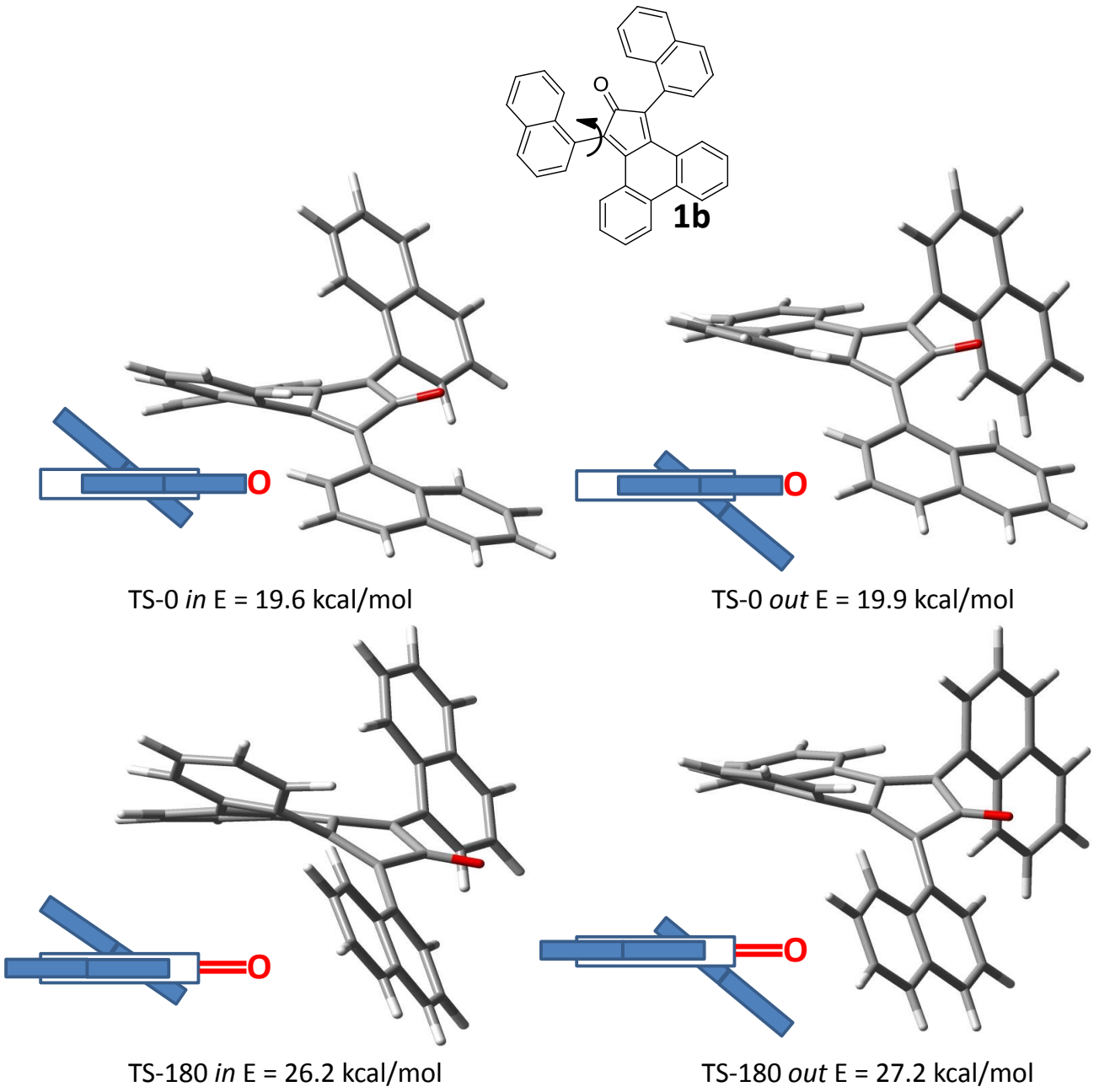


Figure S4 Transition states of 1b optimized at B3LYP/6-31G(d) level

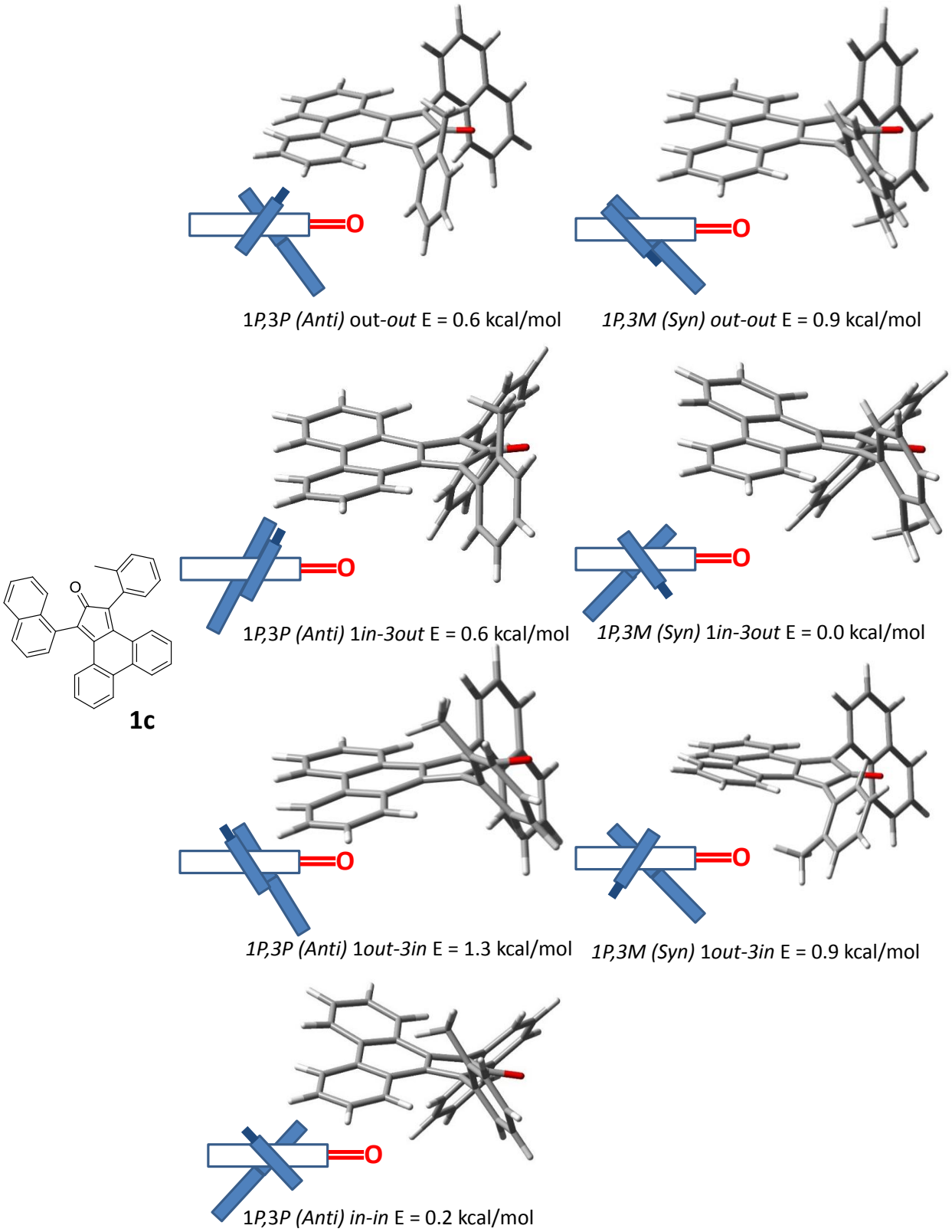


Figure S5 Most stable ground states of 1c (1-(naphthalen-1-yl)-3-(o-tolyl)-3b,7a-dihydro-2H-cyclopenta[l]phenanthren-2-one) optimized at B3LYP/6-31G(d) level

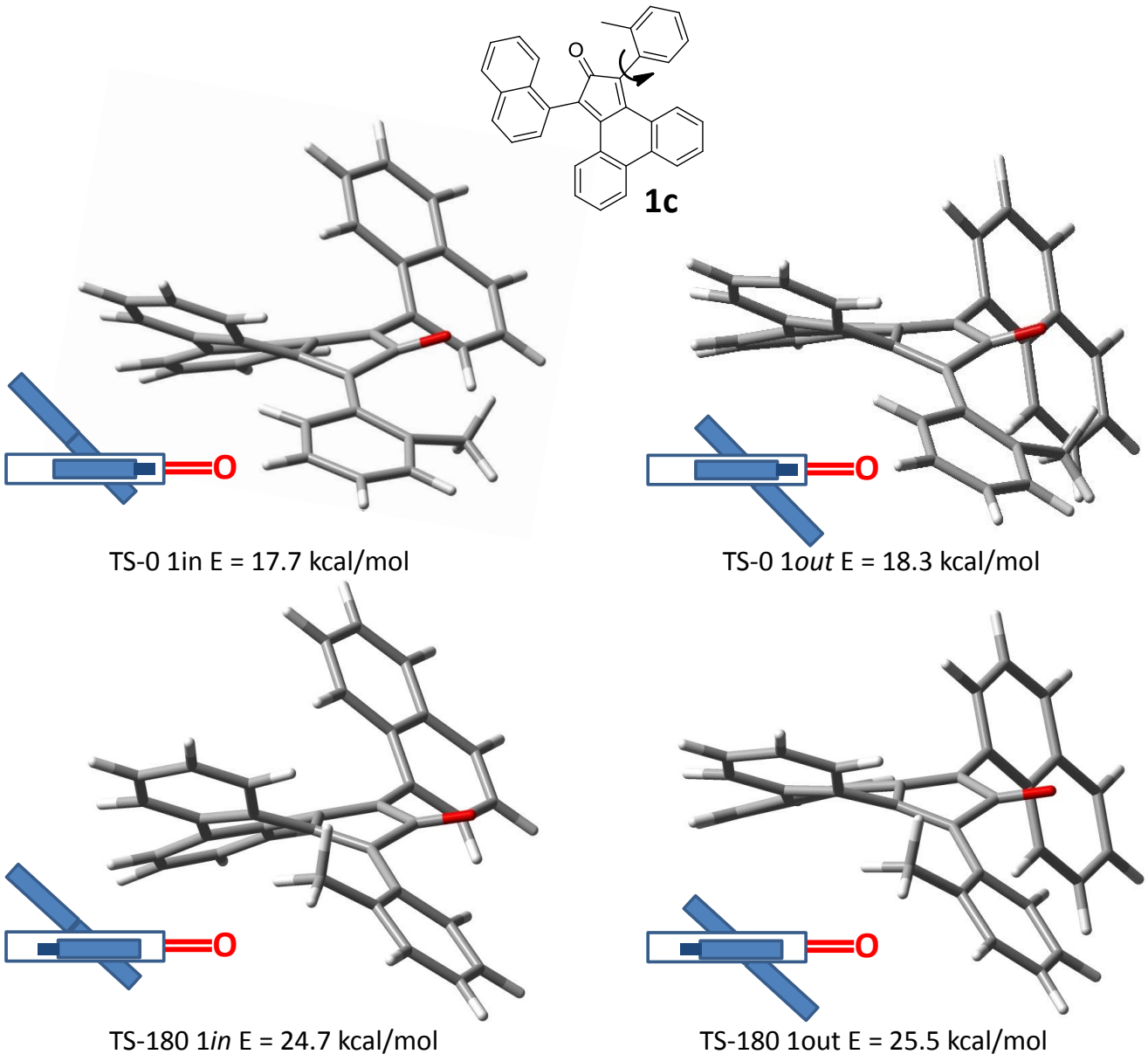


Figure S6 Transition states for the rotation of *o*-tolyl of 1c (1-(naphthalen-1-yl)-3-(*o*-tolyl)-3b,7a-dihydro-2H-cyclopenta[l]phenanthren-2-one) optimized at B3LYP/6-31G(d) level

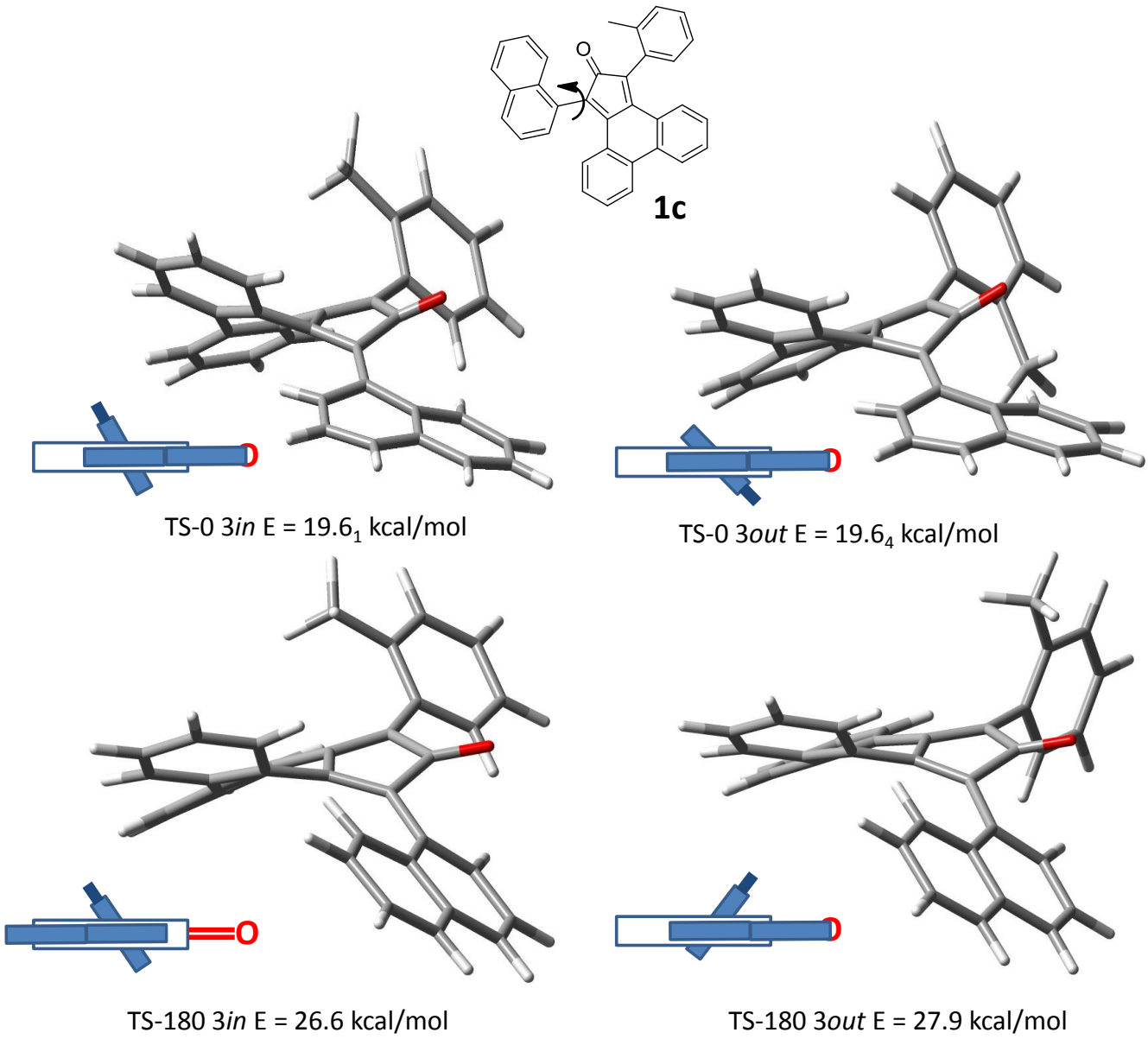
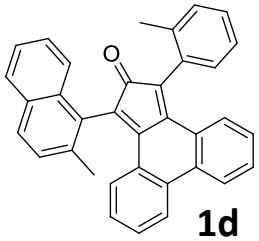
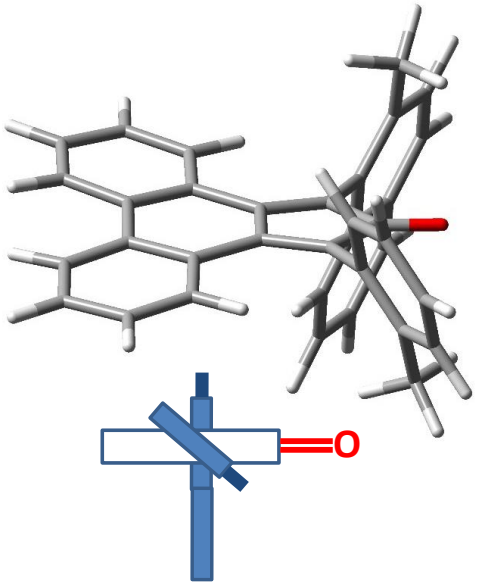


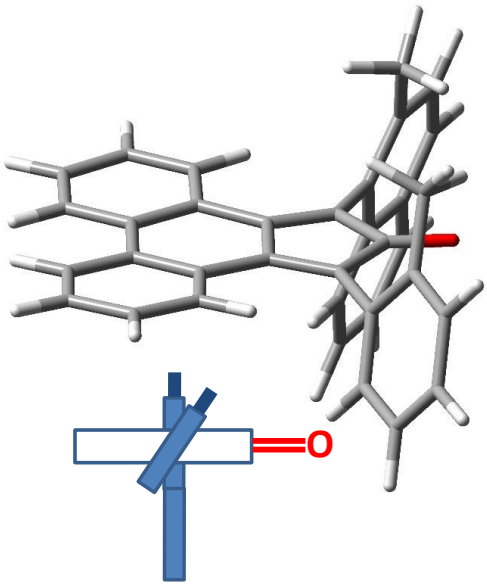
Figure S7 Transition states for the rotation of 1-naphthyl of 1c (1-(naphthalen-1-yl)-3-(*o*-tolyl)-3b,7a-dihydro-2H-cyclopenta[l]phenanthren-2-one) optimized at B3LYP/6-31G(d) level



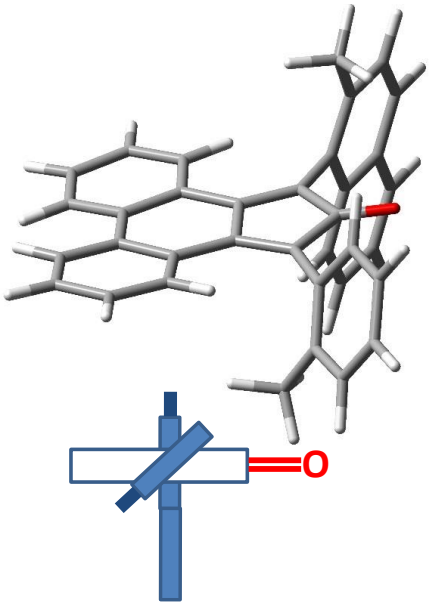
1d



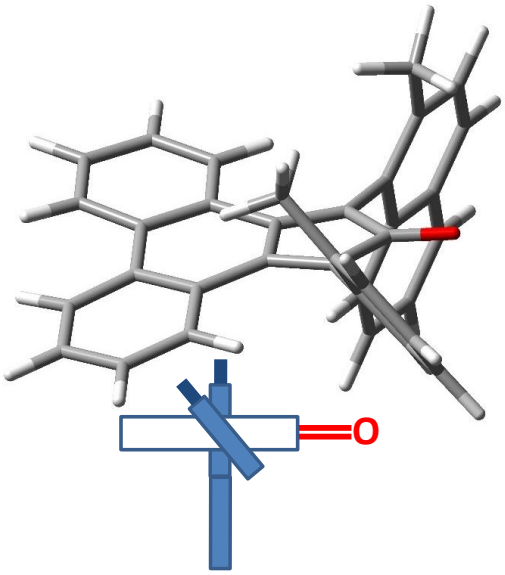
1*P*,3*M* *3out* E = 0.3 kcal/mol



1P,3P 3*out* E = 0.0 kcal/mol



1*P*,3*M* *3in* E = 0.3 kcal/mol



1P,3P 3*in* E = 0.6 kcal/mol

Figure S8 Stable ground states of 1d (1-(2-methylnaphthalen-1-yl)-3-(*o*-tolyl)-3b,7a-dihydro-2H-cyclopenta[l]phenanthren-2-one) optimized at B3LYP/6-31G(d) level

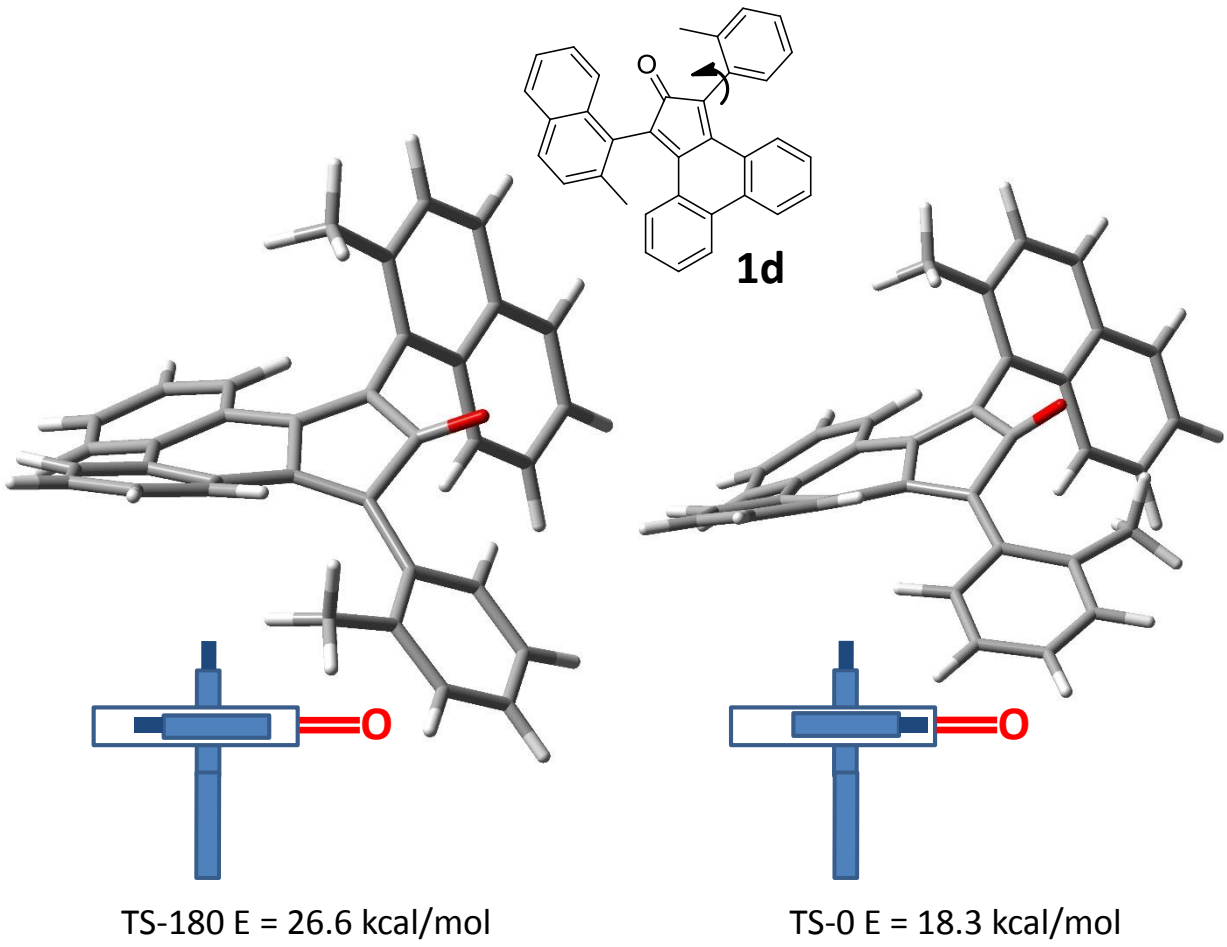


Figure S9 Transition states for the rotation of *o*-tolyl of 1d (1-(2-methylnaphthalen-1-yl)-3-(*o*-tolyl)-3b,7a-dihydro-2H-cyclopenta[l]phenanthren-2-one) optimized at B3LYP/6-31G(d) level

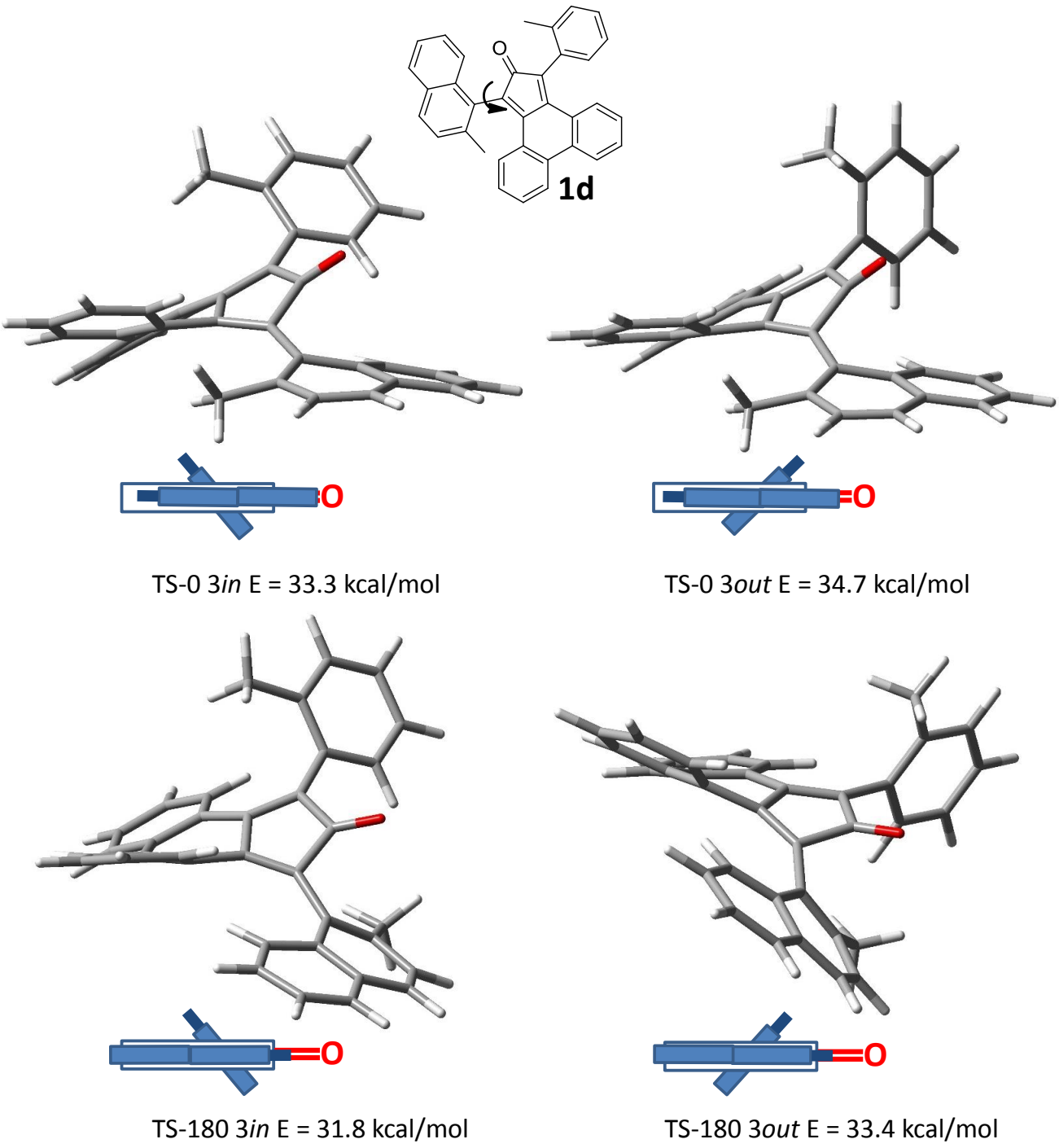


Figure S10 Transition states for the rotation of 2-methyl-1-naphthyl of 1d (1-(2-methylnaphthalen-1-yl)-3-(*o*-tolyl)-3b,7a-dihydro-2H-cyclopenta[l]phenanthren-2-one) optimized at B3LYP/6-31G(d) level.

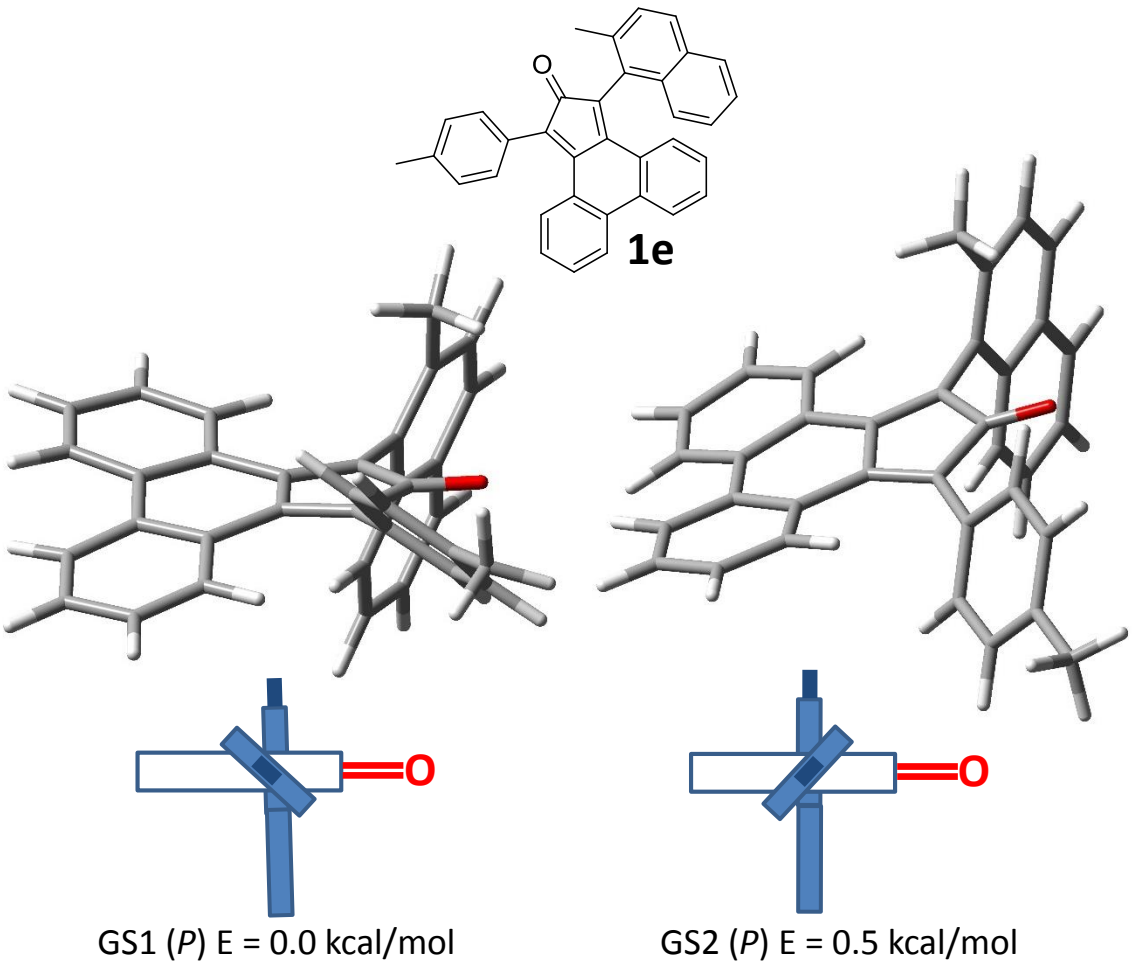


Figure S11 Stable ground states of 1e optimized at B3LYP/6-31G(d) level

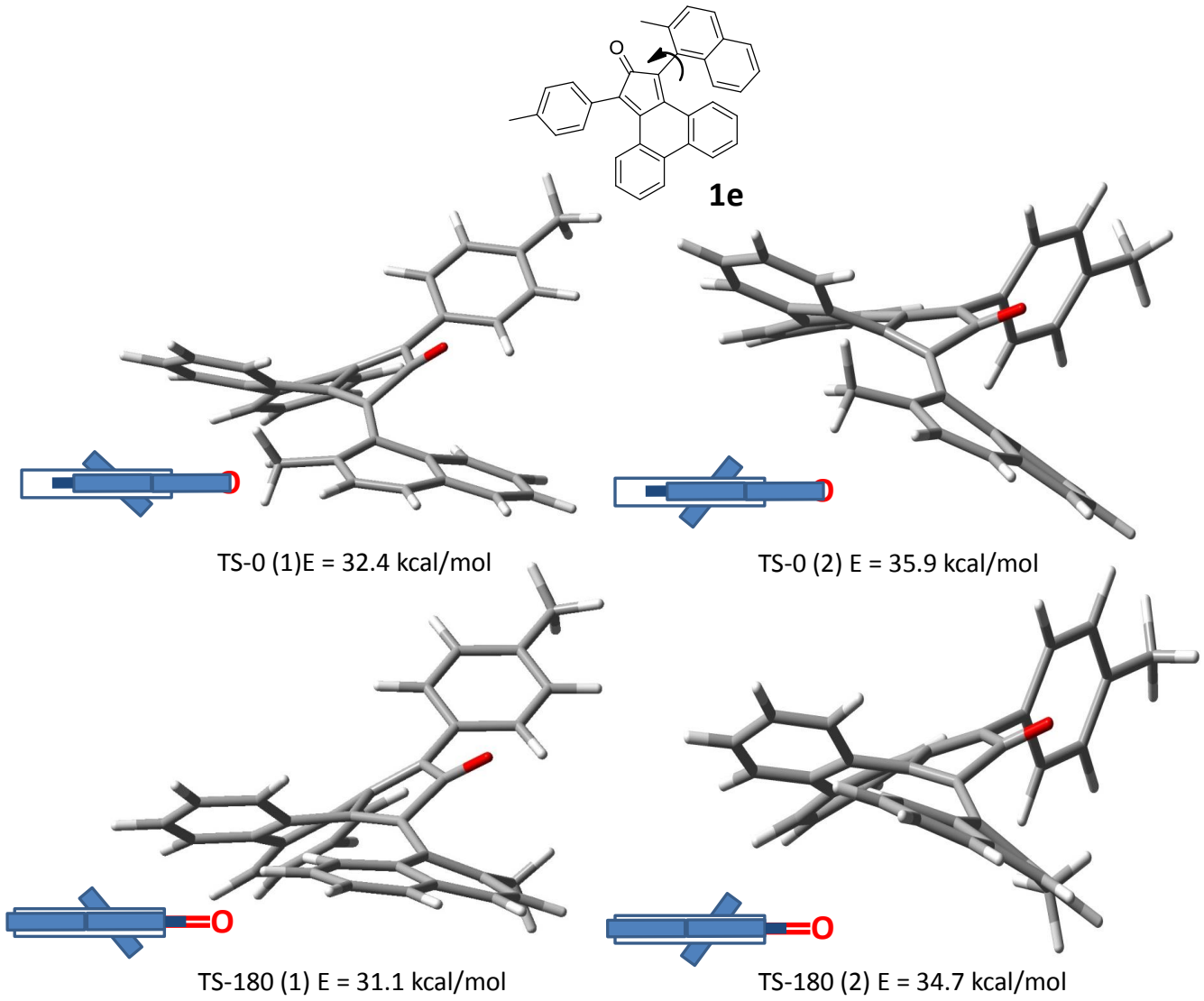
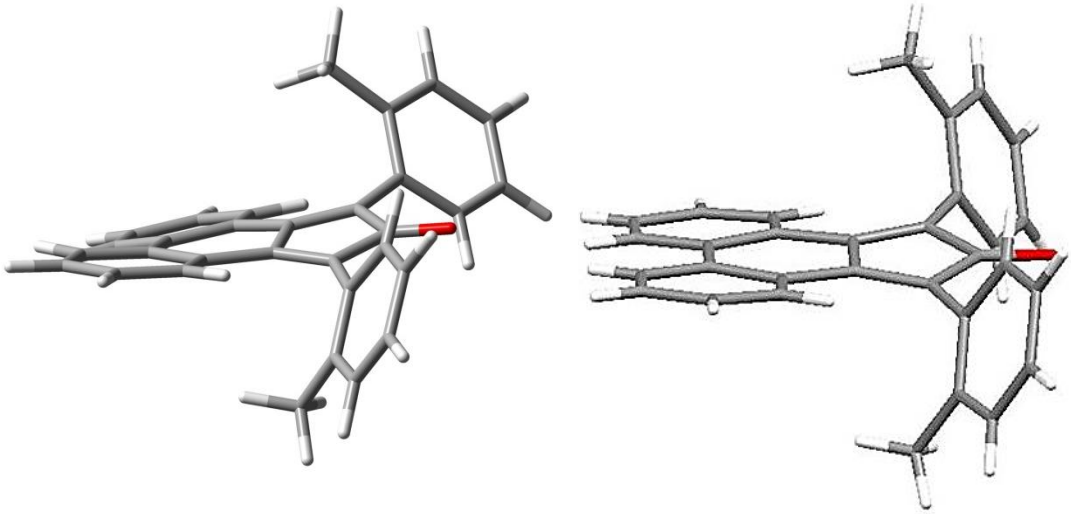


Figure S12 Transition states for the rotation of 2-Me-1-naphthyl of 1e optimized at B3LYP/6-31G(d) level



1a *P,P (anti) in-in*
calculated
E = 0.4 kcal/mol

1a *P,P (anti) in-in*
X-Ray

Figure S13 Compound 1a *P,P* (*anti*) *in-in*. Left: Calculated structure (B3LYP/6-31G(d)); Right: X-Ray structure

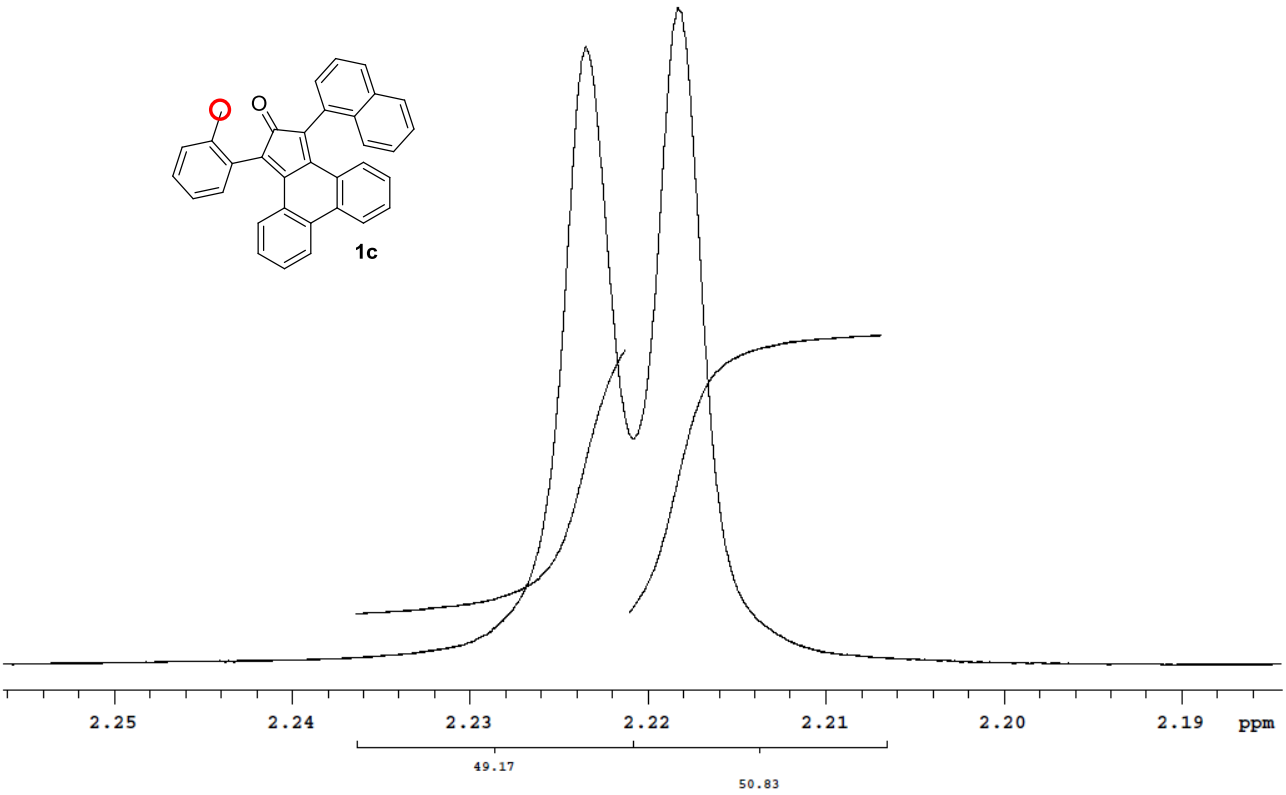


Figure S14: ^{1}H-NMR *o*-methyl signals of compound 1c (600 MHz in $CDCl_3$)

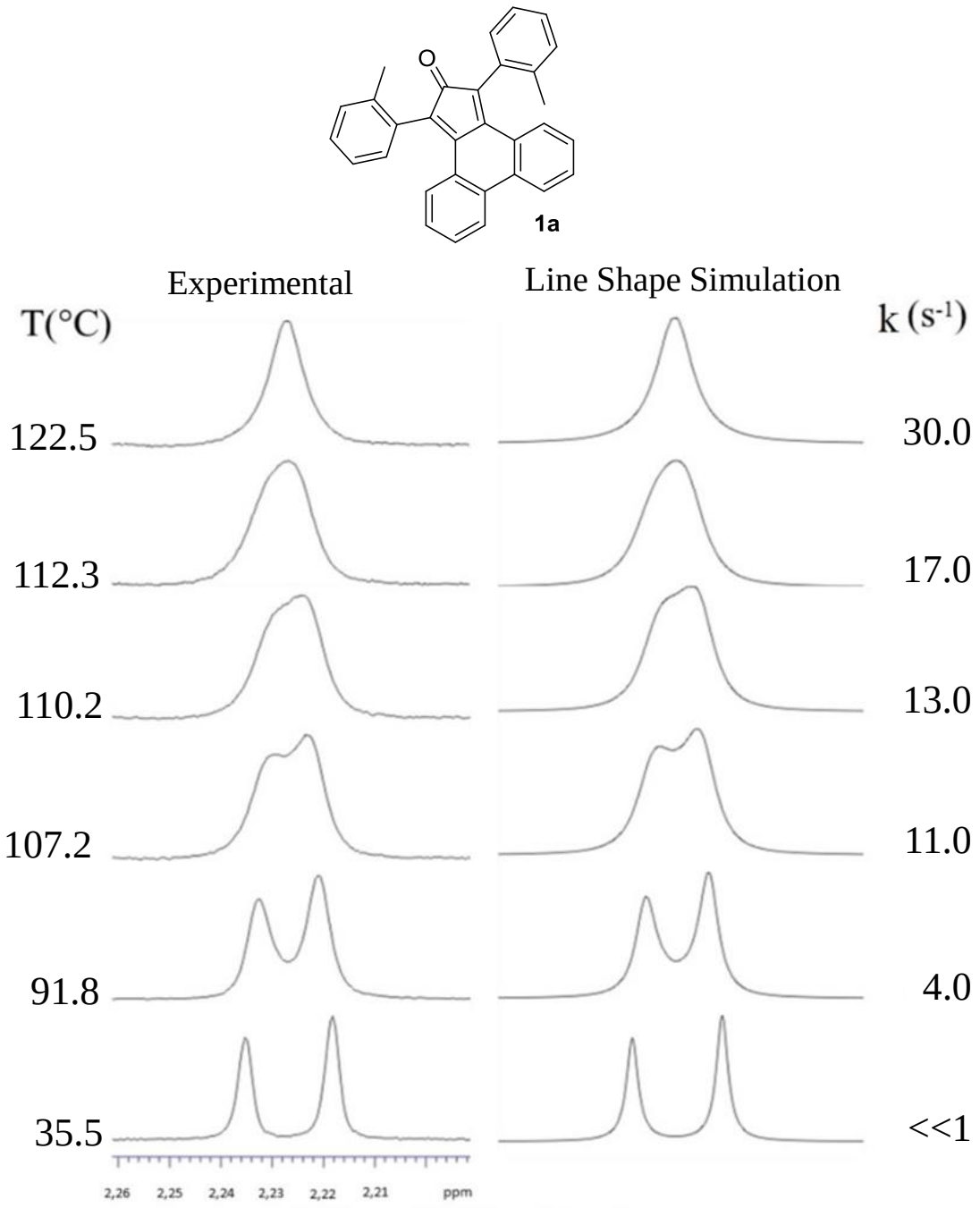


Figure S15. Left: ^{1}H-NMR *o*-methyl signals of compound 1a at different temperatures (600 MHz in DMSO-*d6*). Right: line shape simulations obtained with the corresponding rate constants.

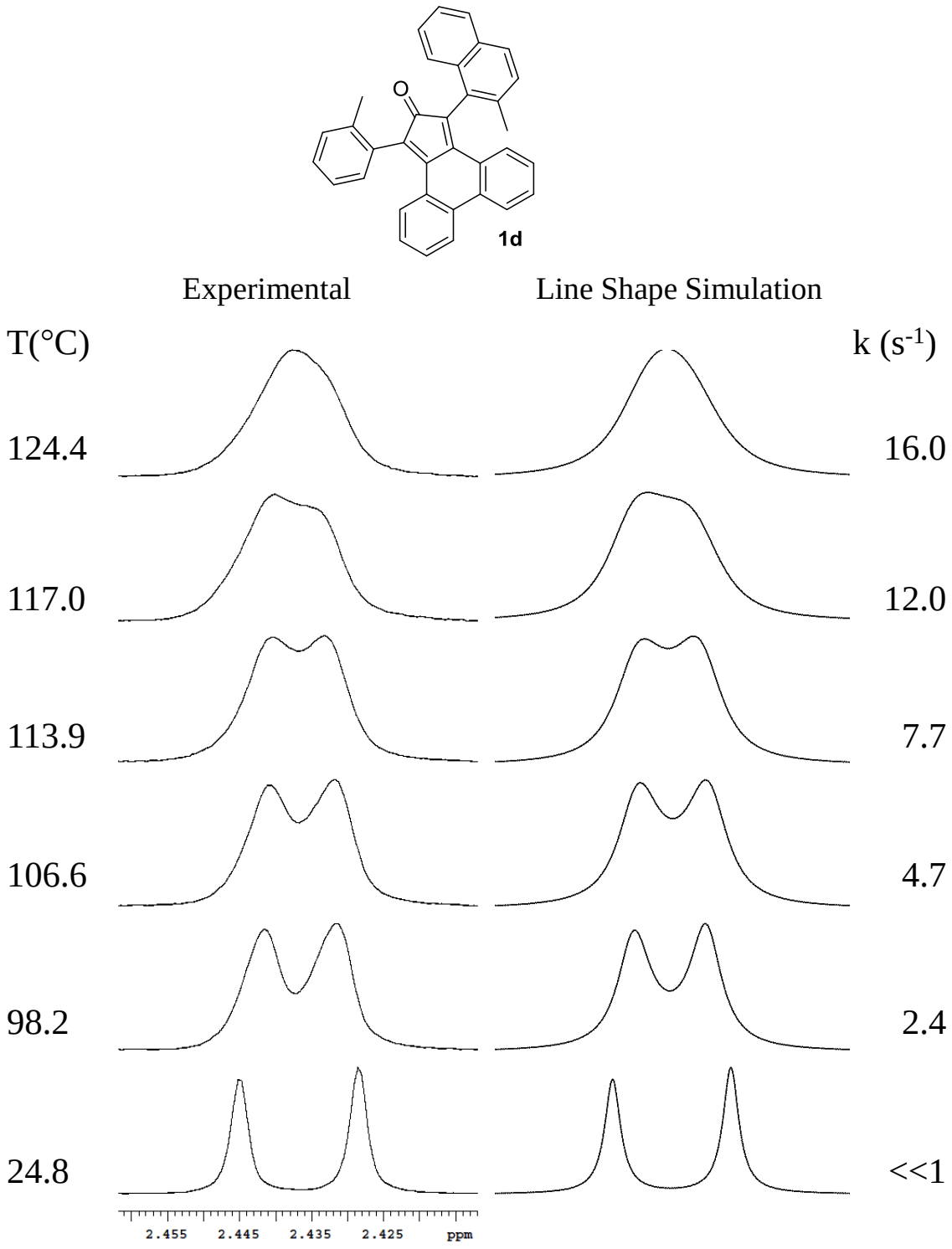


Figure S16. Left: ^{1}H-NMR *o*-methyl signals of compound 1d at different temperatures (600 MHz in $C_2D_2Cl_4$). Right: line shape simulations obtained with the corresponding rate constants.

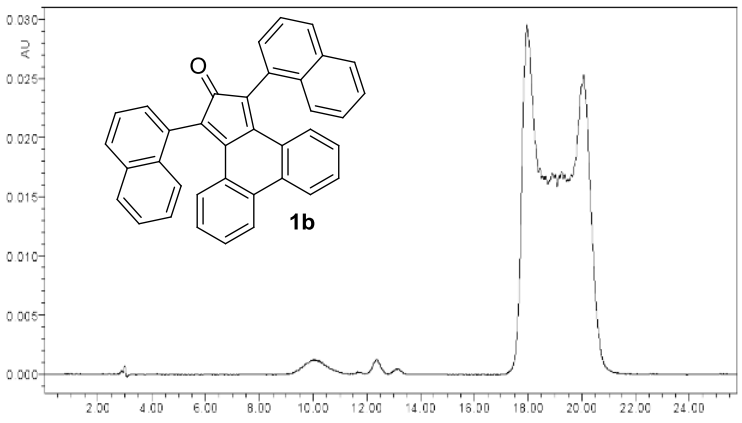


Figure S17. Chromatogram of 1b on Nova-Pak Silica column, 6 µm, 19 x 300 mm, *n*-hexane/CH_2Cl_2 92/8, 20 mL/min, +25 °C.

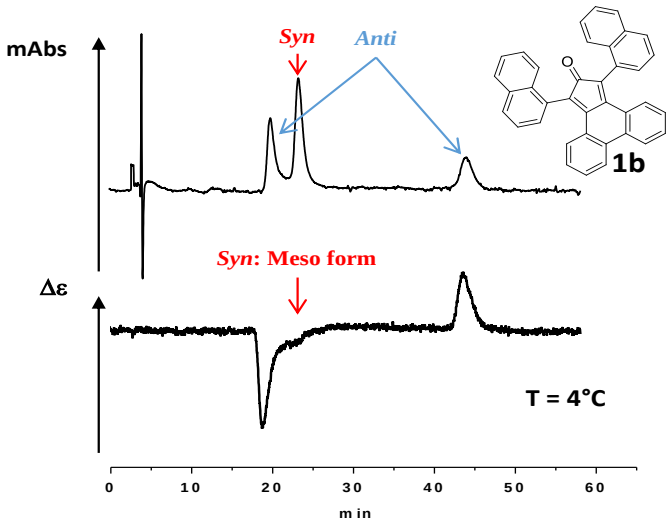


Figure S18. Top: CSP-HPLC of 1b at +4 °C (Chiralpak IA 250 x 4.6 mm eluent *n*-hexane/CH_2Cl_2 98/2 0.05% EtOH, 1.0 mL/min, UV detection at 280 nm). Bottom: ECD detection at 280 nm.

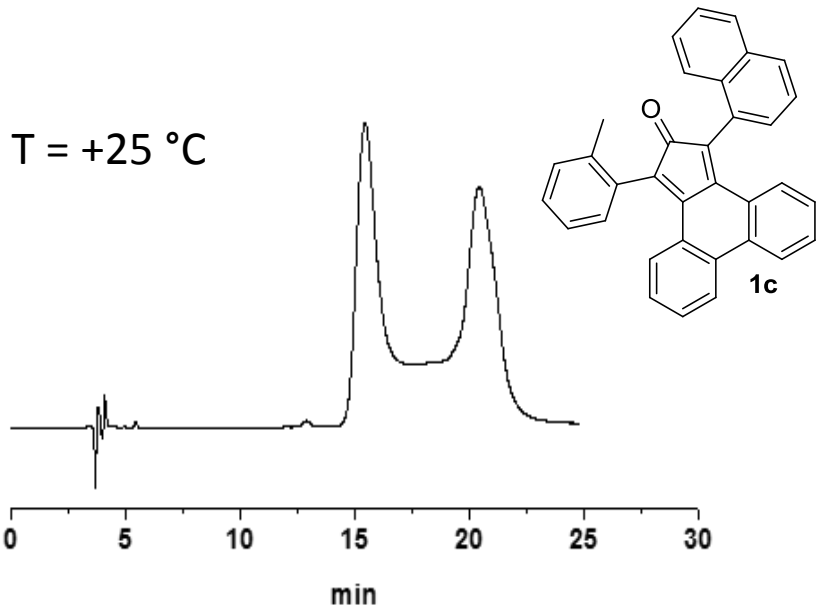


Figure S19. CSP-HPLC chromatogram of 1c recorded at + 25 °C. Chiralpak IA 250 x 4.6 mm eluent *n*-hexane/CH_2Cl_2, 98/2 + 0.05% EtOH, 1.0 mL/min, UV detection at 280 nm.

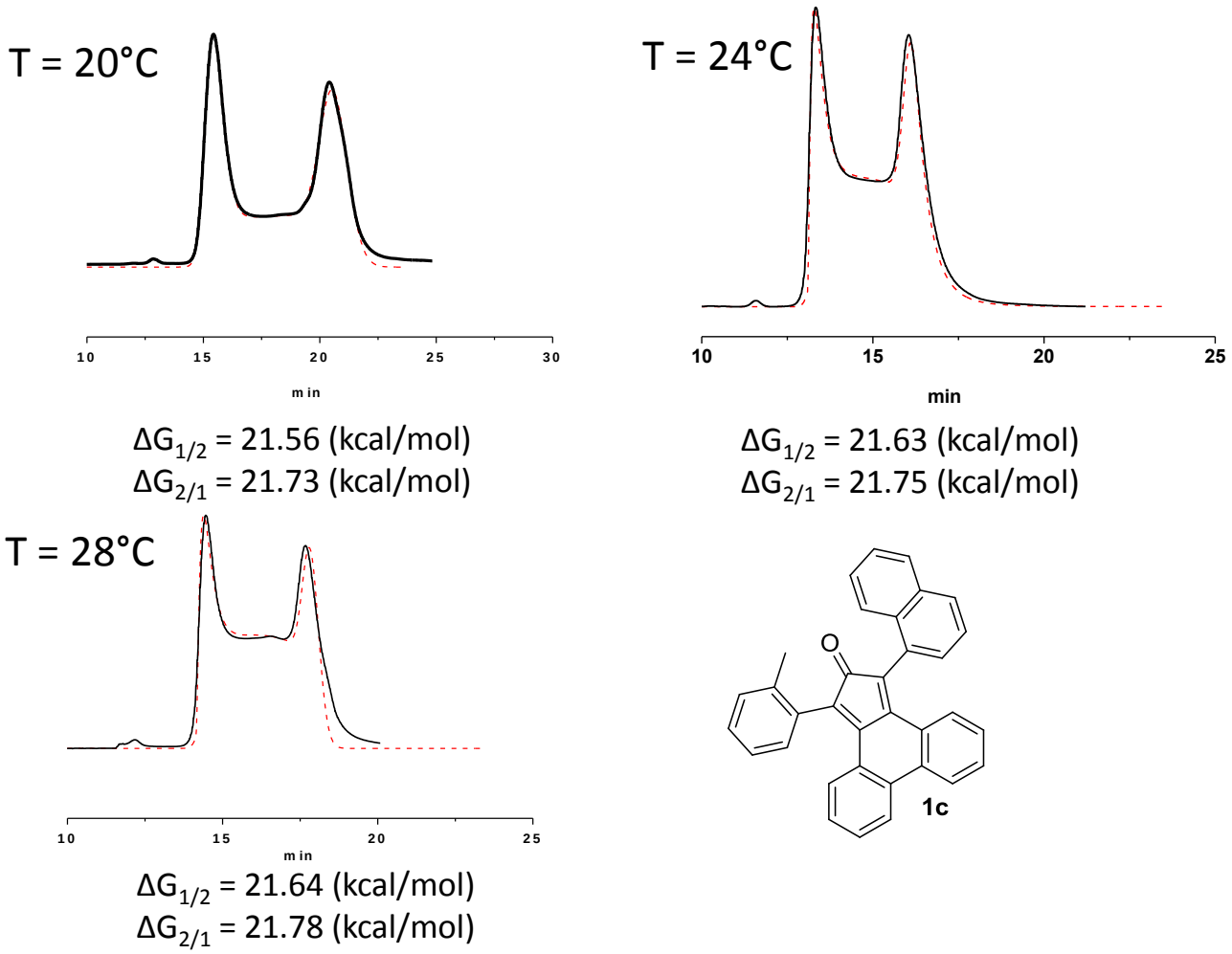


Figure S20. D-CSP-HPLC chromatogram of 1c recorded at different temperatures (Black solid line) and line shape simulation (red dotted line). Chiralpak IA 250 x 4.6 mm eluent *n*-hexane/CH_2Cl_2, 98/2 + 0.05% EtOH, 1.0 mL/min, UV detection at 280 nm $\Delta G_{1/2}$ is the rotational barrier to interconvert enantiomer 1 to enantiomer 2, while $\Delta G_{2/1}$ describes the reverse pathway.

Tables S2-S7. Experimental rotational barriers for 1a-1d determined by D-NMR and D-HPLC. The errors in the determination of $\Delta G^{\neq}$ were determined taking into account the maximum errors on the $\Delta G^{\neq}$ determination using modified temperatures (± 1 K or ± 0.5 K) and kinetic constant (± 30%).

1a D-NMR		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	*k* (s^{-1})	*k* and T	*k**1.3 and T-1 K	*k**0.7 and T+1 K
365.0	4	20.50	20.25	20.70
380.4	11	20.63	20.38	20.84
383.4	13	20.67	20.42	20.89
385.4	17	20.59	20.33	20.80
395.6	30	20.70	20.44	20.93
	mean value	20.62	20.36	20.83
		deviation	-0.26	0.21

1c D-NMR		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	*k* (s^{-1})	*k* and T	*k**1.3 and T-1 K	*k**0.7 and T+1 K
371.4	2	21.39	21.13	21.59
378.7	5.5	21.06	20.81	21.27
383.9	7.5	21.12	20.87	21.34
387.1	8	21.26	21.00	21.47
397.6	10	21.68	21.41	21.90
	mean value	21.30	21.04	21.51
		deviation	-0.26	0.21

1d D-NMR		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	*k* (s^{-1})	*k* and T	*k**1.3 and T-1 K	*k**0.7 and T+1 K
371.4	2.4	21.25	21.00	21.46
379.7	4.7	21.24	20.98	21.45
387.0	7.7	21.28	21.02	21.50
390.1	12	21.11	20.85	21.33
397.6	16	21.31	21.04	21.53
	mean value	21.24	20.98	21.45
		deviation	-0.26	0.21

1b D-HPLC 2 to 1		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	*k* (s^{-1})	*k* and T	*k**1.3 and T -0.5 K	*k**0.7 and T+ 0.5 K
278.15	0.00016	21.06	20.88	21.22
283.15	0.00033	21.05	20.86	21.21
288.15	0.00060	21.09	20.90	21.26
293.15	0.00092	21.22	21.03	21.39
	mean value	21.10	20.92	21.27
		deviation	-0.19	0.16

1b D-HPLC 1 to 2		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	k (s^{-1})	*k* and T	*k**1.3 and T -0.5 K	*k**0.7 and T+ 0.5 K
278.15	0.00018	21.02	20.83	21.17
283.15	0.00036	21.00	20.82	21.17
288.15	0.00064	21.05	20.86	21.22
293.15	0.00099	21.17	20.98	21.34
	mean value	21.06	20.88	21.23
		deviation	-0.18	0.17

1c D-HPLC 1 to 2		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	*k* (s^{-1})	*k* and T	*k**1.3 and T -0.5 K	*k**0.7 and T+ 0.5 K
293.15	0.00051	21.56	21.37	21.73
297.15	0.00076	21.63	21.44	21.80
301.15	0.00124	21.64	21.44	21.81
	mean value	21.61	21.42	21.78
		deviation	-0.19	0.17

1c D-HPLC 2 to 1		**$\Delta G^{\neq}$ (kcal/mol) derived using**		
T (K)	*k* (s^{-1})	*k* and T	*k**1.3 and T -0.5 K	*k**0.7 and T+ 0.5 K
293.15	0.00038	21.73	21.54	21.90
297.15	0.00062	21.75	21.56	21.92
301.15	0.00098	21.78	21.58	21.95
	mean value	21.75	21.56	21.93
		deviation	-0.19	0.18

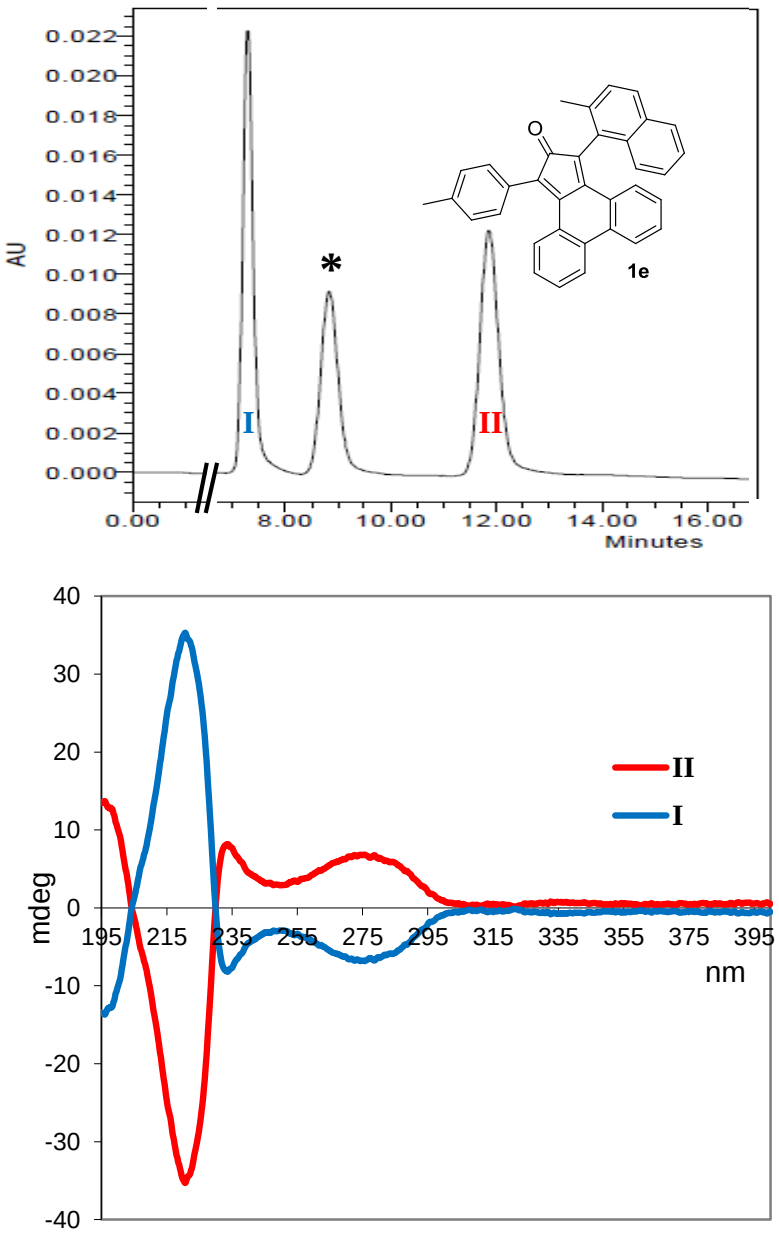


Figure S21. Top: Chromatogram of 1e in Chiral AD-H *n*-hexane/*i*-PrOH, 70/30 at +25 °C. Bottom: ECD spectra of the resolved atropoisomers. * impurity.

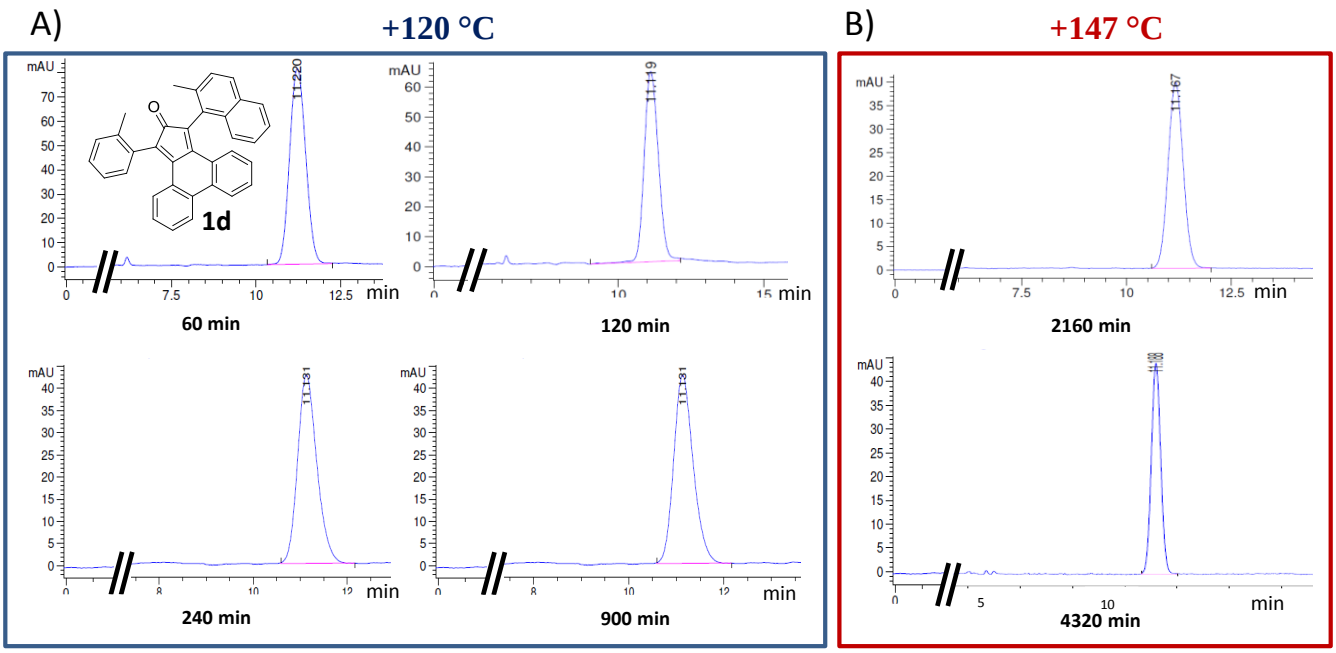


Figure S22. Chromatogram of enantiopure 1d eluted after have been left at A) +120 °C and B) +147 °C in $C_2D_2Cl_4$ for different times. (Chiral AD-H, *n*-hexane/*i*PrOH, 92/8, 1 mL/min).

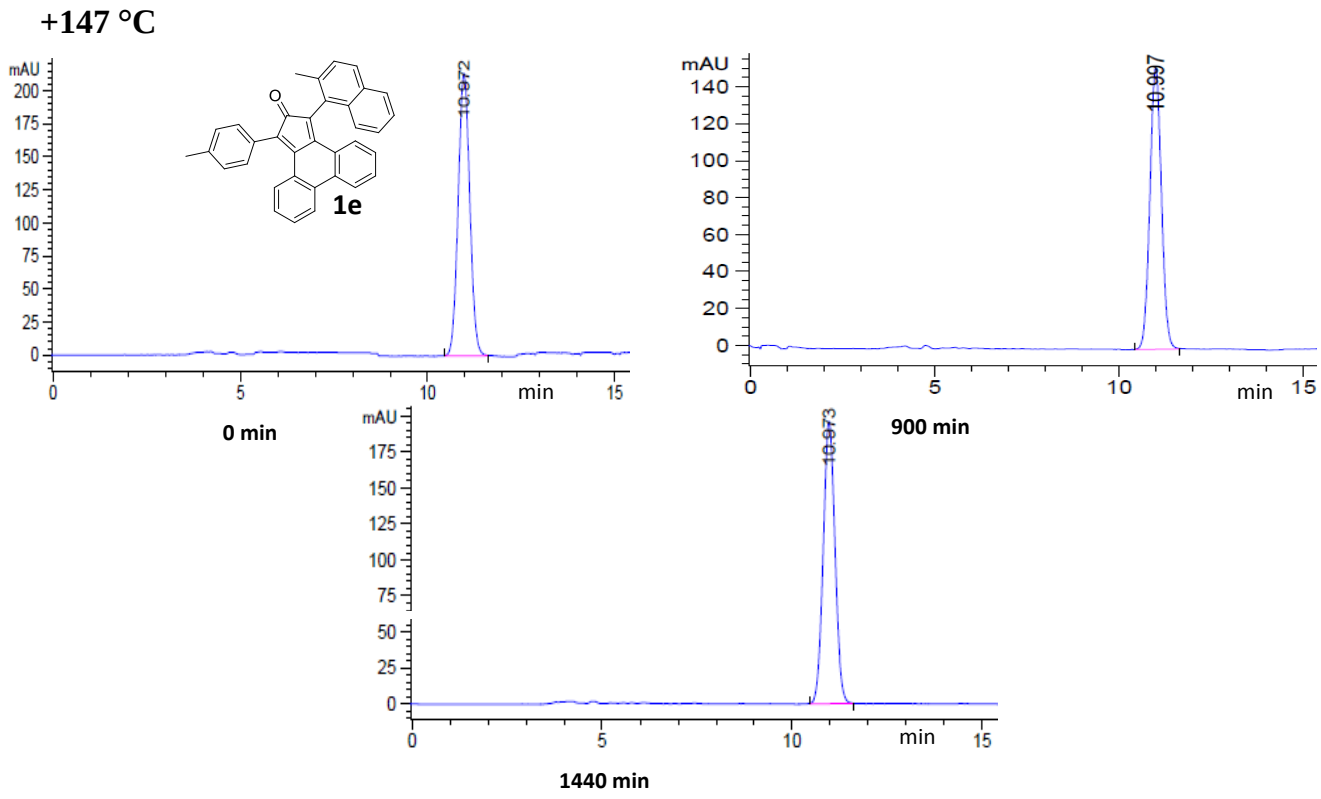


Figure S23. Chromatogram of enantiopure 1e eluted after have been left at +147 °C in $C_2D_2Cl_4$ for different times. (Chiral AD-H, *n*-hexane/*i*PrOH, 70/30, 0.8 mL/min).

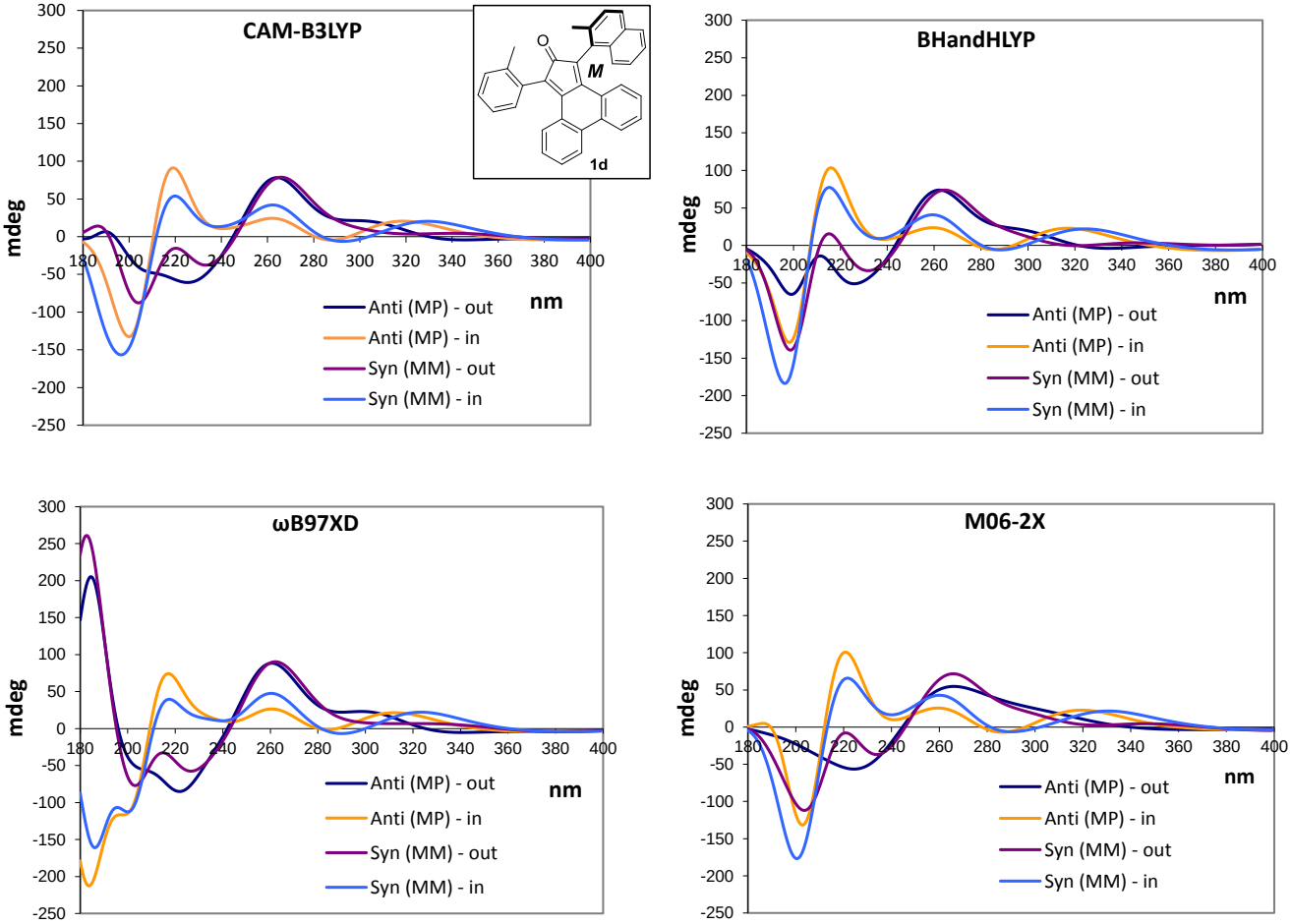


Figure S24. Computed spectra (at CAM-B3LYP/ BHandHLYP / M06-2X / ωB97XD // 6-311++G(2d,p) level of theory) for the four stable ground states of *M,P atropisomer of 1d.**

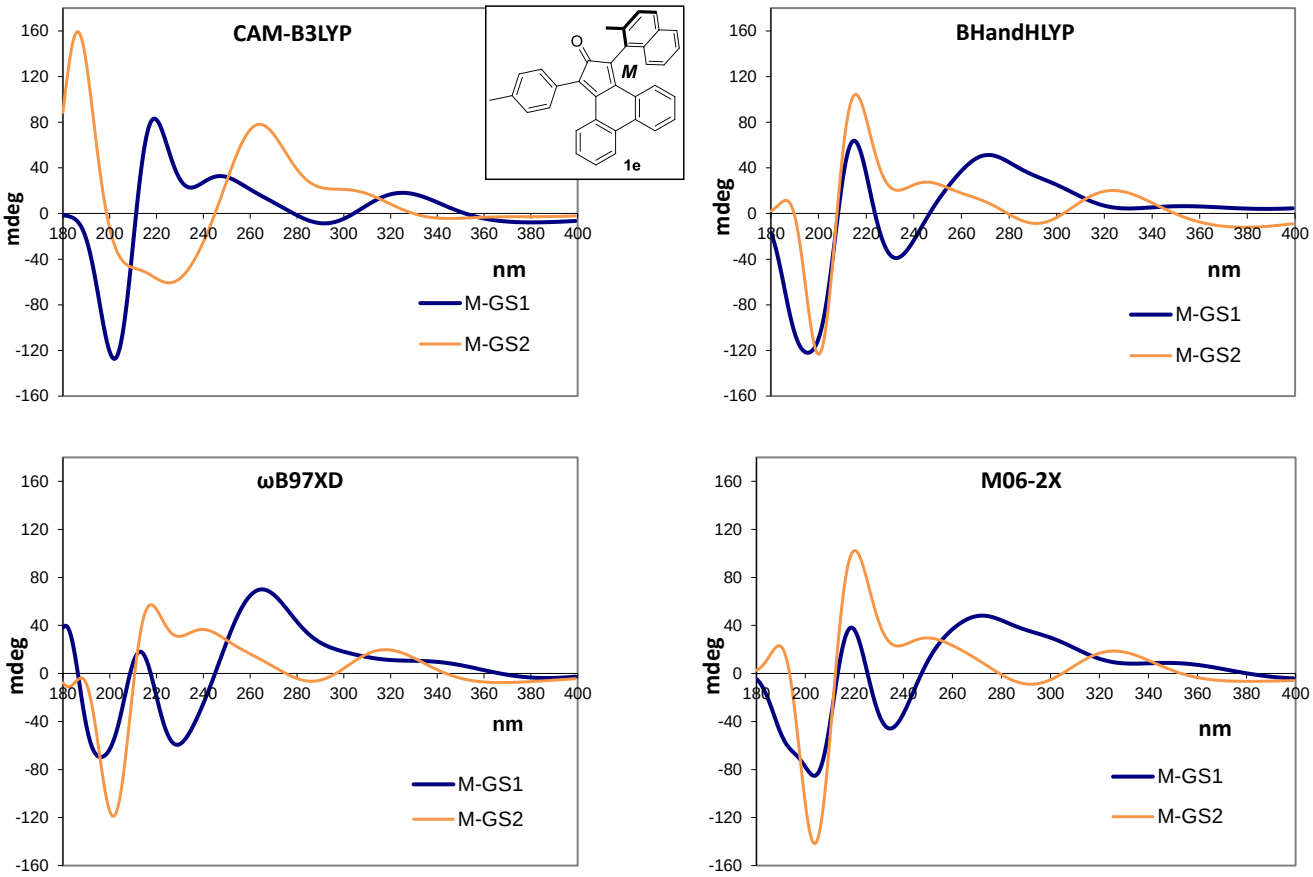


Figure S25. Computed spectra (at CAM-B3LYP/ BH&HLYP/ ωB97XD / M06-2X // 6-311++G(2d,p) level of theory) for the two stable ground states of *M* atropoisomer of 1e.

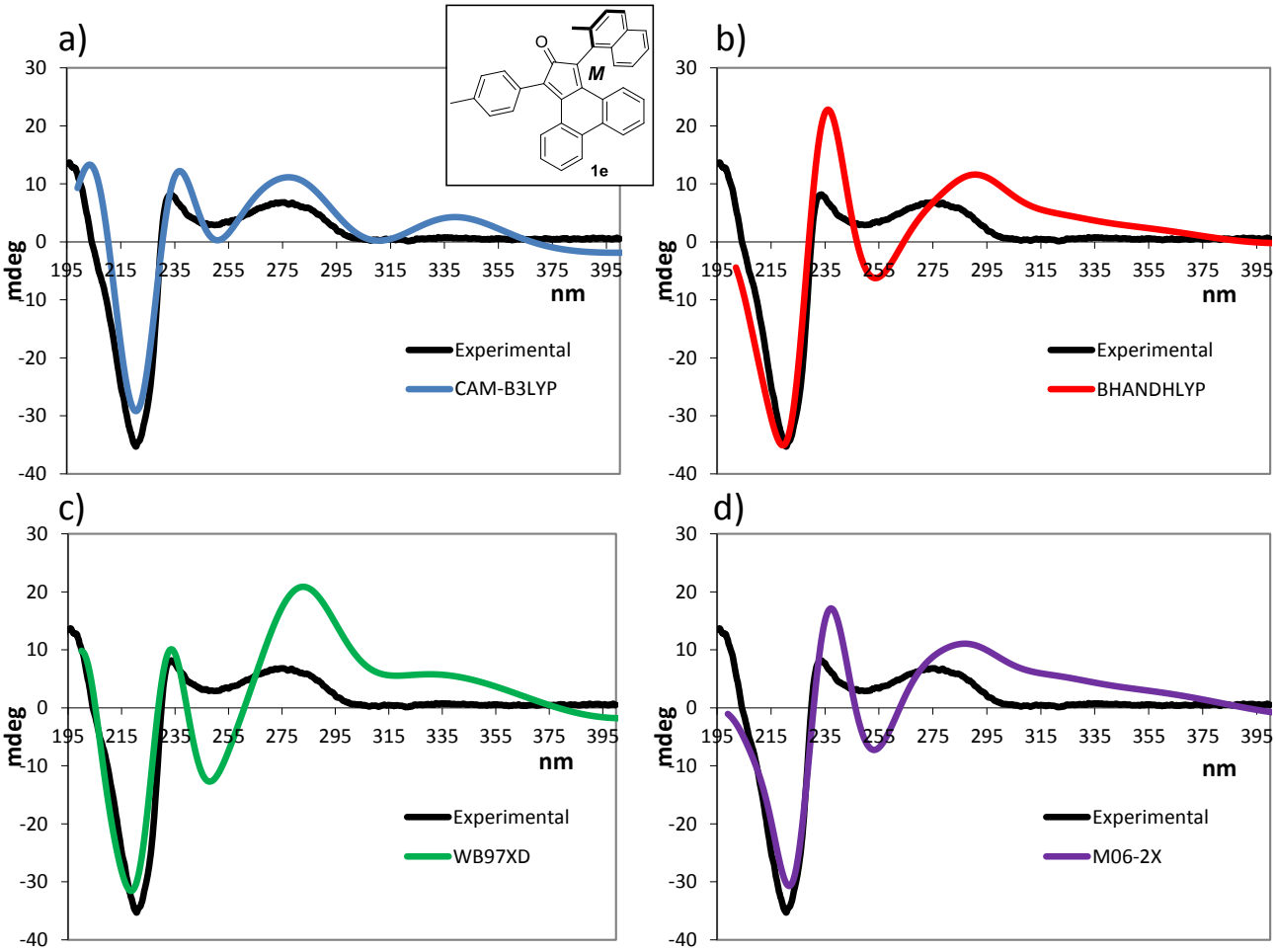


Figure S26. ECD spectra of compound 1e, experimental and calculated with different functionals: a) CAM-B3LYP, b) BH&HLYP, c) ωB97xD, d) M06-2x with the same 6-311++G(2d,p) basis set.

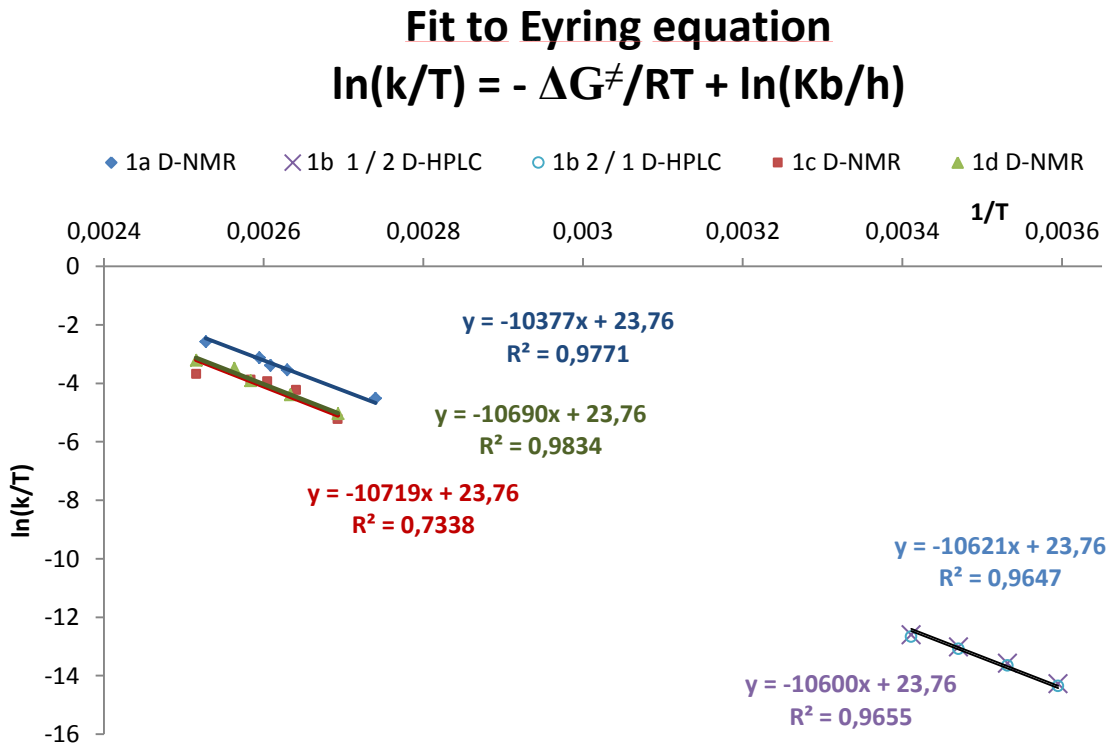
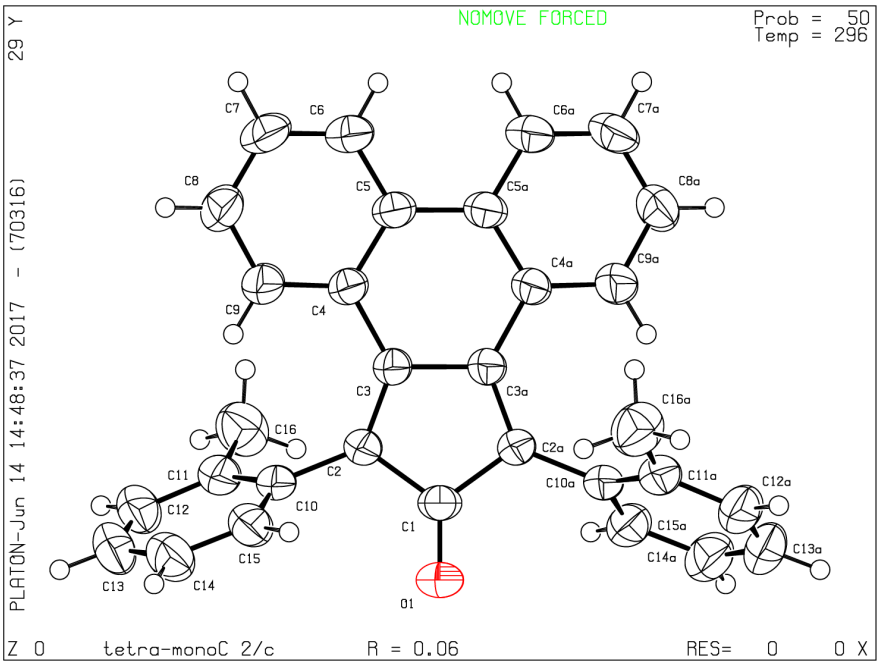


Fig.S27 Experimental data obtained with D-NMR and D-HPLC fitted into the Eyring equation.

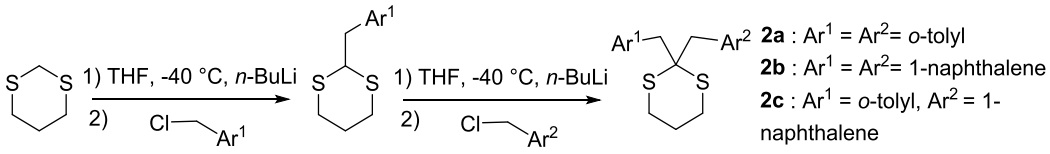
Crystal data for compound **1a**



Molecular formula: $C_{31}H_{22}O$; M_r = 410.48, monoclinic, space group *C*2/c, *a* = 19.134(10), *b* = 10.392(4), *c* = 10.998(5) Å, *β* = 98.116(10); *V* = 2164.9(17) Å^3, T = 298(2) K, *Z* = 4, ρ_c = 1.259 g cm^{-3}, *F*(000) = 864, graphite-monochromated $Mo_{K\alpha}$ radiation (λ = 0.71073 Å), μ($Mo_{K\alpha}$) = 0.078 mm^{-1}, colourless brick (0.3 × 0.1 × 0.05 mm^3), empirical absorption correction with SADABS (transmission factors: 0.978 – 0.996), 2400 frames, exposure time 15 s, 2.15 ≤ θ ≤ 26.00, –23≤ *h* ≤ 23, –12 ≤ *k* ≤ 12, –13 ≤ *l* ≤ 13, 10582 reflections collected, 2125 independent reflections (R_{int} = 0.0512), solution by intrinsic phasing method and subsequent Fourier syntheses, full-matrix least-squares on F_o^2 (SHELXL-2014/7), hydrogen atoms refined with a riding model, data / restraints / parameters = 2125/ 0/ 147, $S(F^2)$ = 1.068, $R(F)$ = 0.1144 and $wR(F^2)$ = 0.1423 on all data, $R(F)$ = 0.0627 and $wR(F^2)$ = 0.1208 for 1305 reflections with $I > 2\sigma(I)$, weighting scheme $w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 1.387P]$ where $P = (F_o^2 + 2F_c^2)/3$, largest difference peak and hole 0.150 and –0.224 e Å^{-3}. Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-1556103.

Preparation and spectroscopic characterization of the intermediates

General procedure (I) for the synthesis of dithianes (2a-2c)[1]



An oven dried three necked round bottomed flask kept under nitrogen atmosphere, was charged with a solution of 1,3-dithiane (15.4 mmol, 1.85 g, 1 eq.) in 60 mL of anhydrous THF (0.26 M). The resultant solution was brought to -40 °C and 15 mL of *n*-BuLi (15.4 mmol, 9.6 mL, 1 eq., 1.6 M in *n*-hexane) was added dropwise to the solution and left to stir at this temperature for 2 hours. In a separate two-necked flask flushed with N_2 was dissolved the appropriate halide (15.4 mmol, 1 eq.) in dry THF (1.2 M). This solution was then added dropwise at -40 °C to the solution of the lithiate: the resultant deep yellow solution was allowed to return to room temperature in 2.5 hours. The raw mixture of the mono-substituted 1,3-dithiane was cooled again to -40 °C and 15 mL of *n*-BuLi (15.4 mmol, 9.6 mL, 1 eq., 1.6 M in *n*-hexane) was added dropwise. The yellow solution became transparent, then turned from emerald to deep green, and left to stir at -40 °C for 2 hours. The second halide (15.4 mmol, 1 eq.) was diluted in THF (1.2 M) and added dropwise to the reaction at -40 °C. Then the mixture was allowed to return to ambient temperature overnight. Before working up the reaction, it was partially concentrated under reduced pressure and then quenched with a saturated solution of NH_4Cl. The organic phase was separated, and the water phase was washed three times with diethyl ether. The combined organic phase was dried over $MgSO_4$ and the solvent removed in vacuum. The crude 2,2-diarylmethylene-1,3-dithiane was then purified by crystallization, flash chromatography on silica gel or semi-preparative HPLC.

2,2-bis(2-methylbenzyl)-1,3-dithiane (2a)

Using the general procedure **I**, the crude product was purified through a precipitation in *n*-hexane/Et_2O 9:1. The sticky solid was used without further purification for the next step of the synthesis (2.00 g, yield = 39.5%). A small pure sample was obtained by a silica gel chromatography column (petroleum

[1] D. Seebach and E.J. Corey, *J. Org. Chem.*, 1975, **40**, 231

ether: EtOAc, 8:2 gradient to 6:4) and used for the characterization. **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 1.85 (m, 2H), 2.29 (s, 6H), 2.82 (m, 4H), 3.27 (s, 4H), 7.12-7.17 (m, 6H), 7.47 (m, 2H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 20.7 (CH_3), 24.1 (CH_2), 26.7 (CH_2), 42.3 (CH_2), 56.1 (Cq), 125.1 (CH), 126.9 (CH), 130.2 (CH), 131.9 (CH), 135.0 (Cq), 137.7 (Cq).

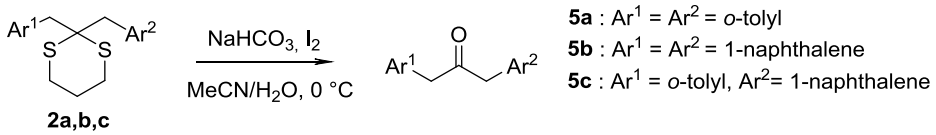
2,2-bis(naphthalen-1-yl-methyl)-1,3-dithiane (2b).

Using the general procedure **I**, the crude product was purified through a precipitation in *n*-hexane/Et_2O 9:1 as a sticky solid (1.54 g, yield = 25%). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 1.77 (m, 2H), 2.76 (m, 4H), 3.75 (s, 4H), 7.40-7.47 (m, 6H), 7.64 (d, *J* = 7.3 Hz, 2H), 7.78 (d, *J* = 8.1 Hz, 2H), 7.82 (d, *J* = 8.1 Hz, 2H), 7.82 (d, *J* = 8.5 Hz, 2H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 24.0 (CH_2), 27.0 (CH_2), 42.3 (CH_2), 55.9 (Cq), 124.7 (CH), 124.9 (CH), 125.2 (CH), 125.6 (CH), 127.8 (CH), 128.7 (CH), 129.9 (CH), 132.7 (Cq), 133.5 (Cq), 133.6 (Cq).

2-(2-methylbenzyl)-2-(naphthalen-1-ylmethyl)-1,3-dithiane (2c)

Using the general procedure **I**, the crude product was partially purified through multiple precipitation (in *n*-hexane : EtOAc, 9:1). The crude mixture was used without further purification for the next step of the synthesis (sticky solid, 2.53 g, yield 45%). A small pure sample for the characterization was obtained by a semi-preparative HPLC purification (Phenomenex Luna C18, 10µm, 100 Å, 250 x 21.2 mm, CH_3CN/H_2O, 90/10, 20 mL/min, 254 nm, t_R = 14.23 min). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 1.82 (m, 2H), 2.29 (s, 3H), 2.83 (m, 4H), 3.30 (s, 2H), 3.75 (s, 2H), 7.13-7.19 (m, 3H), 7.43-7.51 (m, 4H), 7.64 (d, *J* = 6.9 Hz, 1H), 7.79 (d, *J* = 7.7 Hz, 1H), 7.83 (m, 1H), 8.03 (m, 1H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 20.8 (CH_3), 24.0 (CH_2), 26.9 ($2CH_2$), 41.8 (CH_2), 42.8 (CH_2), 56.0 (Cq), 124.6 (CH), 124.8 (CH), 125.22 (CH), 125.2_3 (CH), $125._6$ (CH), 126.9 (CH), 127.7 (CH), 128.7 (CH), 129.8 (CH), 130.3 (CH), 132.0 (CH), 132.7 (Cq), 133.5 (Cq), 133.6 (Cq), 135.0 (Cq), 137.7 (Cq).

General procedure (II) for the deprotection of dithianes (2a-2c)[2]



[2] S. Geum and H-Y. Lee, *Org. Lett.*, 2014, **16**, 2466

To a solution of the appropriate 2,2-diarylmethylene-1,3-dithiane (1.13 mmol, 1 eq.) in acetonitrile (107 mL) and H_2O (5.4 mL) were added sodium bicarbonate (9.08 mmol, 0.762 g, 6.6 eq.) and iodine (4 mmol, 1.01 g, 2.9 eq.) at 0 °C. The reaction mixture was stirred for 1 hour and then quenched with a saturated solution of $Na_2S_2O_3$. The aqueous layer was extracted twice with diethyl ether (50 mL). The combined organic layer was collected and dried over $MgSO_4$. The crude was then purified by flash chromatography on silica gel or semi-preparative HPLC.

1,3-di-*o*-tolylpropan-2-one (5a)

Using the general procedure **II** the product was purified by flash chromatography on silica gel (petroleum ether : Et_2O, 9:1), as white solid (226 mg, yield = 84%). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 2.17 (s, 6H), 3.73 (s, 4H), 7.07 (d, *J* = 7.3 Hz, 2H), 7.14-7.20 (m, 6H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 19.5 ($2CH_3$), 47.3 ($2CH_2$), 126.2 (2CH), 127.4 (2CH), 130.4 (2CH), 130.5 (2CH), 132.9 (2Cq), 136.9 (2Cq), 205.6 (CO).

1,3-di(naphthalen-1-yl)propan-2one (5b)

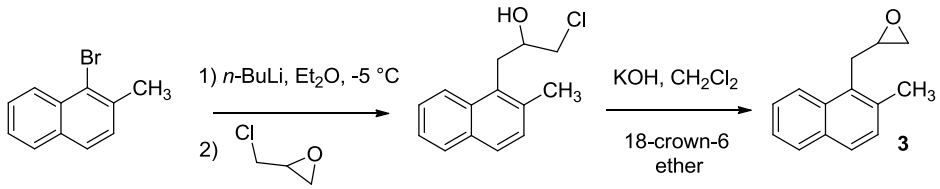
Using the general procedure **II** with longer (12 h) reaction time, the crude was pass through a plug of silica gel and the product was further purified by semi-preparative HPLC (Phenomenex Synergi Polar-RP, 4μm, 80 Å, 250 x 21.2 mm, CH_3CN/H_2O, 68/32, 20 mL/min, 254 nm, t_R = 13.05 min) in 54% yield as white solid (190 mg). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 4.14 (s, 4H), 7.29 (d, *J* = 6.5 Hz, 2H), 7.39-7.43 (m, 4H), 7.47 (dd, *J* = 8.6 Hz, *J* = 8.6 Hz, 2H), 7.70 (d, *J* = 8.6 Hz, 2H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.86 (d, *J* = 8.6 Hz, 2H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 47.1 ($2CH_2$), 123.9 (2CH), 125.5 (2CH), 125.8 (2CH), 126.4 (2CH), 128.1 (2CH), 128.5 (2CH), 128.7 (2CH), 130.8 (2Cq), 132.2 (2Cq), 133.9 (2Cq), 206.4 (CO).

1-(naphthalen-1-yl)-3-(*o*-tolyl)propan-2-one (5c)

Using the general procedure **II**, the crude was purified by flash chromatography on silica gel (petroleum ether : EtOAc, 8:2 gradient to 6:4). White solid (182 mg, yield = 59%). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS) δ 2.07 (s, 3H), 3.71 (s, 2H), 4.14 (s, 2H), 7.03 (d, *J* = 7.7 Hz, 1H), 7.10-7.18 (m, 3 H), 7.31 (d, *J* = 6.9 Hz, 1H), 7.42 (dd, *J* = 6.9 Hz, *J* = 7.0 Hz, 1H), 7.45-7.50 (m, 2H), 7.79 (m, 2H), 7.86 (m, 2H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS) δ 19.5 (CH_3), 46.9 (CH_2) 47.4 (CH_2), 123.8 (CH), 125.5 (CH), 125.8 (CH), 126.1 (CH), 126.5 (CH), 127.3 (CH), 128.0 (CH), 128.4

(CH), 128.7 (CH), 130.4$_1$ (CH), 130.4$_3$ (CH), 130.8 (Cq), 132.2 (Cq), 132.8 (Cq), 133.9 (Cq), 137.0 (Cq), 206.0 (CO).

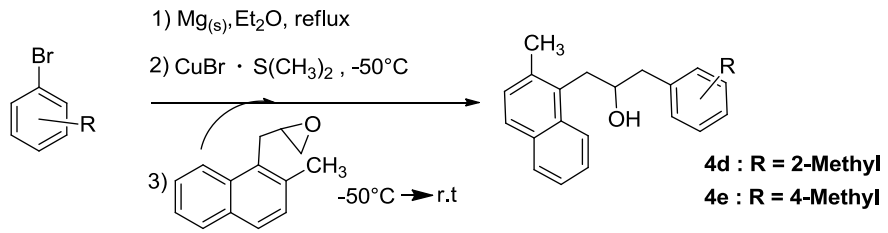
2-((2-methylnaphthalen-1-yl)methyl)oxirane (3)[3]



To an ice-cooled solution of 1-bromo-2-methylnaphthalene (16 mmol, 3.54 g, 1 eq.) in 130 mL of dry Et_2O (0.12 M) a solution of *n*-BuLi (16 mmol, 10 mL, 1 eq., 1.6M in *n*-hexane) was added over 7 minutes under N_2 atmosphere. The reaction was stirred at -5 °C for 40 minutes and 2-(chloromethyl)oxirane (16 mmol, 1.25 mL, 1 eq.) was added to the yellow solution. Stirring was continued for 90 minutes at -5 °C. The reaction was quenched with 150 mL of water, extracted with Et_2O (3 x 70 mL), and the collected organic layers washed with brine (50 mL). The product was dried under vacuum evaporation and used as crude for the subsequent reaction. The crude product was dissolved in CH_2Cl_2 (200 mL), and to the solution was added KOH (3.41 mmol, 1.91 g), and 18-crown-6 ether (0.98 mmol, 0.259 g). The reaction was stirred at room temperature overnight. Subsequent workup by filtration on a celite pad followed by evaporation of the solvent at low pressure. The product was purified by chromatography on silica gel (*n*-hexane:EtOAc, 8:2) as a yellow oil (2.57 g, overall yield = 81%). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 2.48 (dd, *J* = 4.9 Hz, *J* = 5.2 Hz, 1H), 2.49 (s, 3H), 2,68 (dd, *J* = 4.9 Hz, *J* = 5.2 Hz, 1H), 3.20 (m, 1H), 4.31 (dd, *J* = 15.0 Hz, *J* = 4.9 Hz, 1H), 4.36 (dd, *J* = 15.0 Hz, *J* = 4.9 Hz, 1H), 7.28 (d, *J* = 8.2 Hz, 1H), 7.39 (dd, *J* = 8.2 Hz, *J* = 8.2 Hz, 1H), 7.47 (dd, *J* = 8.2 Hz, *J* = 8.2 Hz, 1H), 7.63 (d, *J* = 8.2 Hz, 1H), 7.77 (d, *J* = 7.9 Hz, 1H), 8.01 (d, *J* = 8.5 Hz, 1H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS) δ 20.6 (CH_3), 30.5 (CH_2), 46.9 (CH_2), 51.7 (CH-O), 123.6 (CH), 124.6 (CH), 126.1 (CH), 126.9 (CH), 128.5 (CH), 129.0 (CH), 130.0 (Cq), 132.4 (Cq), 132.7 (Cq), 134.4 (Cq).

General procedure (III) for the synthesis of 1,3 di-substituted alcohols (4d-e)[4]

[3] H.D. Becker and K.A. Amin, *J. Org. Chem.*, 1989**, 54**, 3182
[4] E. Marotta, M. Baravelli, L. Maini, P. Righi and G. Rosini, *J. Org. Chem.*, 1998**, 63**, 8235



An oven dried double-necked round bottomed flask (50 mL) with internal stirring, was kept under nitrogen. The flask was charged with oven dried Mg turnings (5.125 mmol, 0.125 g, 2.05 eq.) and barely covered with dry diethyl ether (10 mL). The solution was stirred overnight in order to activate the Mg turnings. A catalytic amount of dibromoethane (0.25 mmol, 20 μL, 0.05 eq.) and iodine were added, the solution was refluxed until the brown colour disappear and then cooled to room temperature. The appropriate aryl bromide was added (5 mmol, 2 eq.) dropwise and the reaction was heated to reflux until most of the magnesium was consumed (1-2.5 hours). The aryl Grignard reagent so formed was cooled to room temperature and transferred into an oven dried three-necked round bottomed flask under nitrogen equipped with addition funnel, thermometer and septum. The solution was diluted with 10 mL of dry diethyl ether and cooled to -50 °C, then CuBr•$S(CH_3)_2$ (1.25 mmol, 0.257 g, 0.5 eq.) was added to the mixture that was kept stirring for 2 hours at -50 °C. A solution in Et_2O (0.33 M) of the epoxide **3** (2.5 mmol, 0.495 g, 1 eq.) was charged in the addition funnel and added dropwise at the mixture at -50 °C. The cooling bath was removed and the temperature was allowed to reach the room temperature in 1.5 hour. Subsequent work up with a saturated solution of NH_4Cl allowed to remove the copper as tetra ammonium complex (blue aqueous solution). The crude product was extracted several times with ethyl acetate and purified by flash chromatography.

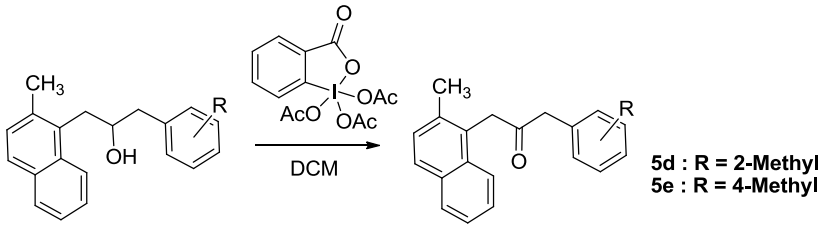
1-(2-methylnaphthalen-1-yl)-3-(*o*-tolyl)propan-2-ol (4d)

Using the general procedure **III** the product was obtained as yellowish oil (660 mg, yield = 91%) by flash chromatography on silica gel (*n*-hexane : EtOAc, 8:2). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 2.23 (s, 3H), 2.51 (s, 3H), 2.94 (m, 2H), 3.33-3.38 (m, 2H), 4.20 (m, 1H), 7.12-7.16 (m, 3H), 7.22 (m, 1H), 7.39 (dd, *J* = 8.4 Hz, *J* = 8.4 Hz, 1H), 7.43 (dd, *J* = 8.2 Hz, *J* = 8.0 Hz, 1H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.4 Hz, 1H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 19.5 (CH_3), 20.8 (CH_3), 35.7 (CH_2), 41.1 (CH_2), 72.7 (CH-OH), 123.7 (CH), 124.6 (CH), 125.9 (CH), 126.0 (CH), 126.6 (CH), 126.8 (CH), 128.6 (CH), 129.2 (CH), 130.2 (CH), 130.5 (CH), 131.9 (Cq), 132.6 (Cq), 132.7 (Cq), 134.6 (Cq), 136.5 (Cq), 136.8 (Cq).

1-(2-methylnaphthalen-1-yl)-3-(*p*-tolyl)propan-2-ol (4e)

Using the general procedure **III** the product was was obtained as yellowish oil (572 mg, yield = 79%) by flash chromatography on silica gel (hexane : EtOAc, 8:2). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C, TMS); δ 1.59 (d, *J* = 8.2, OH), 2.32 (s, 3H), 2.51 (s, 3H), 2.86-2.92 (m, 2H), 3.28-3.35 (m, 2H), 4.18 (m, 1H), 7.12 (m, 4H), 7.30 (d, *J* = 8.3 Hz, 1H), 7.39 (dd, *J* = 8.3 Hz, *J* = 8.3 Hz, 1H), 7.44 (dd, *J* = 8.5 Hz, *J* = 8.3 Hz, 1H), 7.64 (d, *J* = 8.5 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.93 (d, *J* = 8.3 Hz, 1H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 20.8 (CH_3), 21.0 (CH_3), 35.6 (CH_2), 43.5 (CH_2), 73.6 (CH-OH), 123.8 (CH), 124.6 (CH), 125.9 (CH), 126.7 (CH), 128.6 (CH), 129.2_2 (2CH), 129.2_3 (2CH), 129.3 (CH), 131.8 (Cq), 132.6 (Cq), 132.7 (Cq), 134.6 (Cq), 135.4 (Cq), 136.0 (Cq).

General procedure (IV) for the oxidation of 1,3 di-substituted alcohols (5)



To a solution of the appropriate alcohol (**4d-e**, 2.25 mmol, 1 eq) in DCM (0.01 M), was added in one portion the Dess-Martin periodinane (3.93 eq, 1.667 g, 1.75 eq). The solution was allowed to stir at room temperature for 36 hours. The reaction was quenched with 40 mL of aqueous NaOH 1.3 M and extracted three times with DCM. The combined organic layers were collected, dried with $MgSO_4$ and the solvent was removed under reduced pressure.

1-(2-methylnaphthalen-1-yl)-3-(*o*-tolyl)propan-2-one (5d)

Using the general procedure **IV**, the product was purified by flash chromatography on silica gel (*n*-hexane : EtOAc 9:1), as a white solid (337 mg, 52% yield). **^{1}H NMR** (400 MHz, $CDCl_3$, +25 °C, TMS); δ 2.08 (s, 3H), 2.36 (s, 3H), 3.69 (s, 2H), 4.19 (s, 2H), 7.04 (m, 1H), 7.11-7.20 (m, 3 H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.38-7.45 (m, 2H), 7.66 (m, 1H), 7.70 (m, 1H), 7.80 (m, 1H). **^{13}C NMR** (100.56 MHz, $CDCl_3$, +25 °C, TMS); δ 19.5 (CH_3), 20.5 (CH_3), 42.9 (CH_2), 47.2 (CH_2), 123.2 (CH), 124.8 (CH), 126.2 (CH), 126.5 (CH), 127.4 (CH), 127.6 (CH), 128.0 (Cq), 128.6 (CH), 129.0 (CH), 130.3 (CH), 130.4 (CH), 132.4 (Cq), 132.6 (Cq), 132.8 (Cq), 134.7 (Cq), 136.9 (Cq), 205.8 (CO).

1-(2-methylnaphthalen-1-yl)-3-(*p*-tolyl)propan-2-one (5e)

Using the general procedure **IV** the product was purified by flash chromatography on silica gel (*n*-hexane : EtOAc, 9:1) as a white solid (311 mg, 48% yield). **^{1}H NMR** (600 MHz, $CDCl_3$, +25 °C) δ = 2.33 (s, 3H), 2.36 (s, 3H), 3.65 (s, 2H), 4.19 (s, 2H), 7.01 (m, 2H), 7.11 (m, 2H), 7.31 (d, *J* = 8.1 Hz, 1H), 7.38-7.45 (m, 2H), 7.69 (m, 2H), 7.80 (m, 1H). **^{13}C NMR** (150.8 MHz, $CDCl_3$, +25 °C, TMS); δ 20.6 (CH_3), 21.1 (CH_3), 42.8 (CH_2), 48.7 (CH_2), 123.2 (CH), 124.7 (CH), 126.4 (CH), 127.5 (CH), 128.0 (Cq), 128.6 (CH), 129.1 (CH), 129.3 (2CH), 129.4 (2CH), 130.9 (Cq), 132.5 (Cq), 132.7 (Cq), 134.8 (Cq), 136.7 (Cq), 206.2 (CO).

Phencyclone-di-otolyl-CDCl3-1H-ATB Inova600-ATB H1-s2pul-cdcl3

Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
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INOVA-600 "i600"

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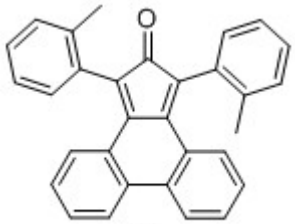
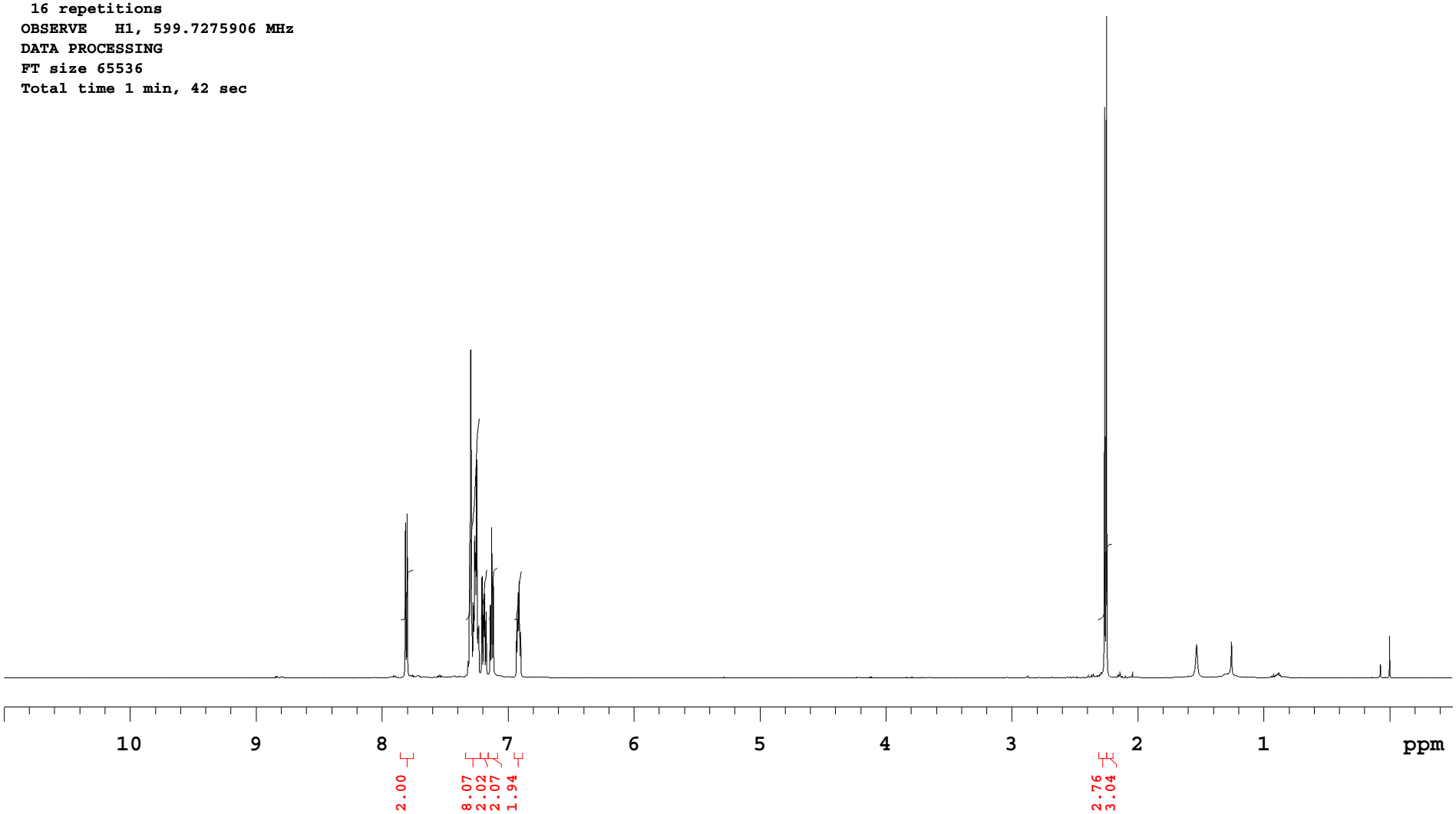


1a



Phencyclone-di-otolyl-CDCl3-1H-ATB Inova600-ATB H1-s2pul-cdcl3

Automation directory:

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INOVA-600 "i600"

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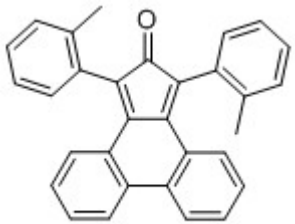
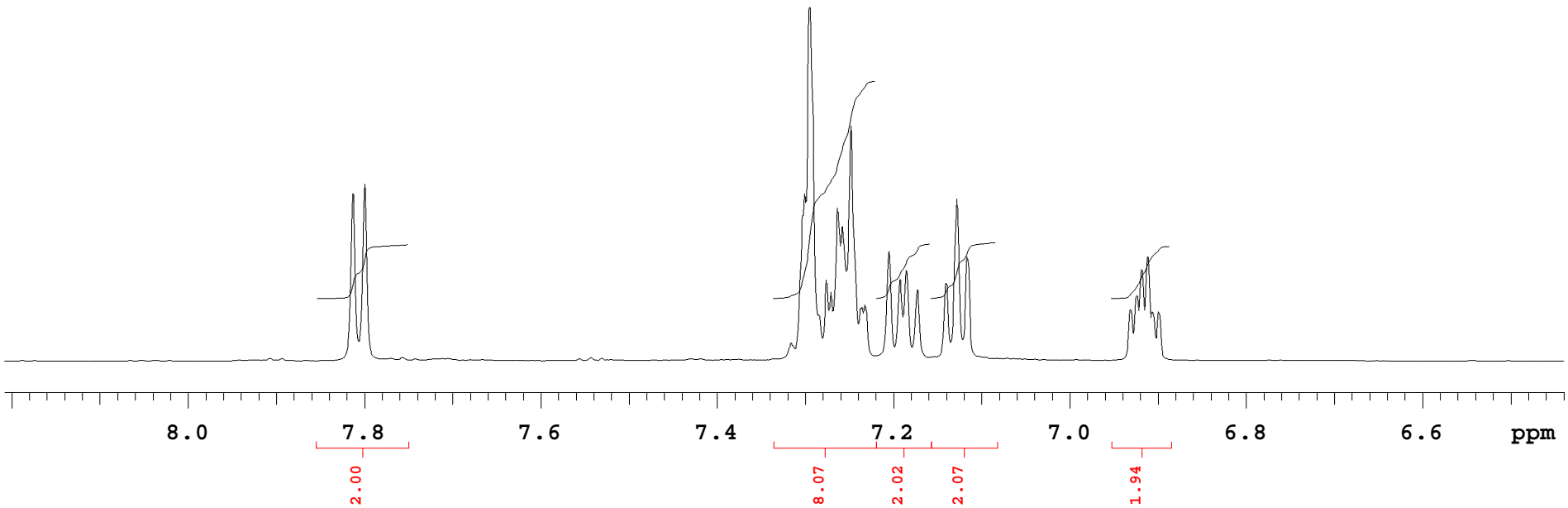


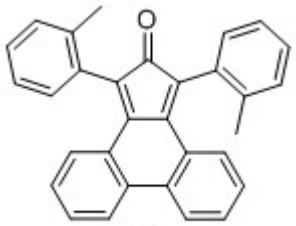
1a



Phencyclone-di-otolyl-CDCl3-13C-ATB Inova600-ATB C13-s2pul-cdcl3

Automation directory:

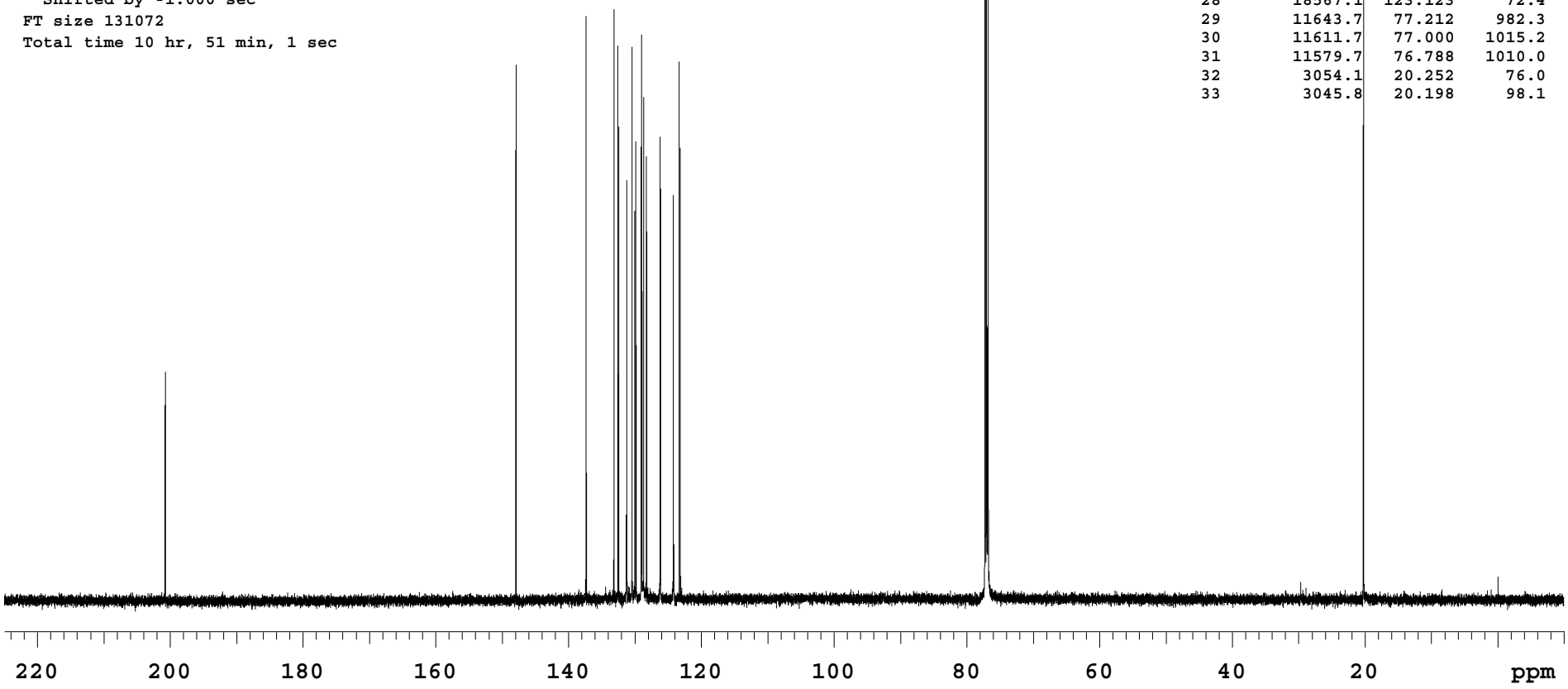
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-di-otolyl-CDCl3-13C-ATB
INOVA-600 "i600"



1a

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
6500 repetitions
OBSERVE C13, 150.8016240 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 10 hr, 51 min, 1 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	30277.3	200.776	31.2
2	30269.5	200.724	36.6
3	22309.3	147.938	72.1
4	22298.3	147.865	85.7
5	20714.8	137.365	80.7
6	20705.4	137.302	93.5
7	20078.2	133.143	78.6
8	20076.6	133.132	94.6
9	19983.8	132.517	88.7
10	19964.5	132.389	75.8
11	19790.6	131.236	56.8
12	19786.7	131.210	67.3
13	19665.3	130.405	88.6
14	19606.8	130.017	62.4
15	19575.3	129.808	73.4
16	19455.5	129.014	72.6
17	19451.1	128.984	65.3
18	19448.8	128.970	90.5
19	19438.9	128.904	49.5
20	19402.5	128.662	80.5
21	19341.7	128.259	71.1
22	19334.6	128.212	59.1
23	19030.9	126.198	74.2
24	19018.8	126.118	65.9
25	18725.0	124.170	57.6
26	18722.8	124.155	64.9
27	18595.8	123.313	86.2
28	18567.1	123.123	72.4
29	11643.7	77.212	982.3
30	11611.7	77.000	1015.2
31	11579.7	76.788	1010.0
32	3054.1	20.252	76.0
33	3045.8	20.198	98.1

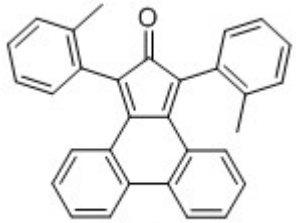


Phencyclone-di-otolyl-CDCl3-DEPT-ATB Inova600-ATB C13-DEPT-cdcl3

Automation directory:

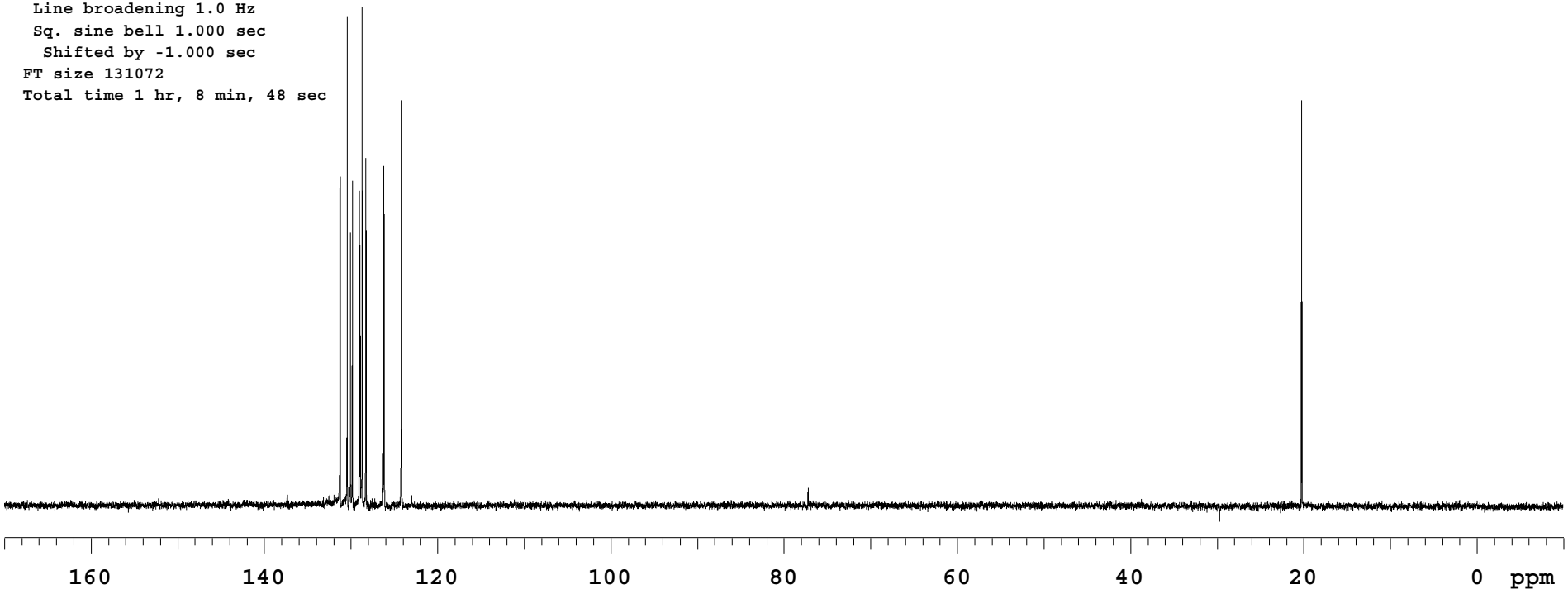
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-di-otolyl-CDCl3-DEPT-ATB
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
1024 repetitions
OBSERVE C13, 150.8016217 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 8 min, 48 sec



1a

INDEX	FREQUENCY	PPM	HEIGHT
1	19792.8	131.250	51.1
2	19789.0	131.225	52.9
3	19667.5	130.419	78.5
4	19609.0	130.031	43.9
5	19577.5	129.822	52.1
6	19453.3	128.999	50.6
7	19441.7	128.922	41.9
8	19404.7	128.676	80.0
9	19344.0	128.274	55.8
10	19337.4	128.230	44.2
11	19033.2	126.213	54.6
12	19021.0	126.132	46.8
13	18727.3	124.184	60.5
14	18725.1	124.170	65.0
15	3055.8	20.264	53.3
16	3048.1	20.212	65.0

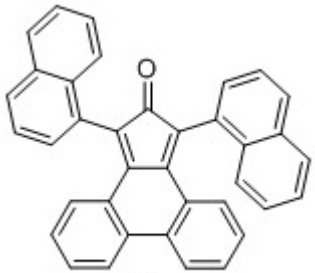


Phencyclone-di-napht-1H-Triple-CDCl3 Inova600-Triple H1-s2pul-cdcl3

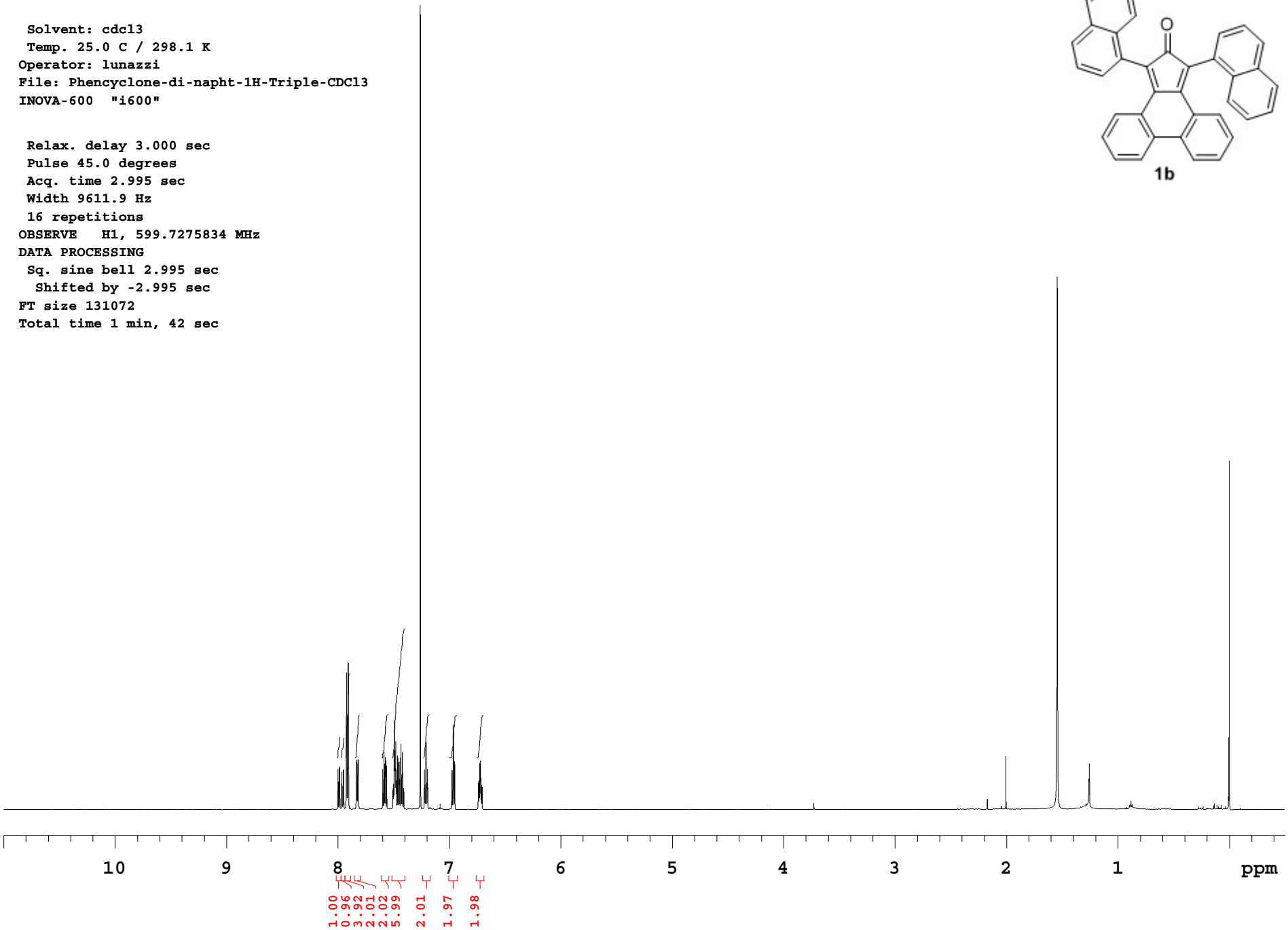
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-di-napht-1H-Triple-CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7275834 MHz
DATA PROCESSING
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 1 min, 42 sec



1b

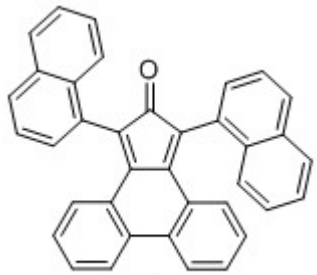


Phencyclone-di-napht-1H-Triple-CDCl3 Inova600-Triple H1-s2pul-cdcl3

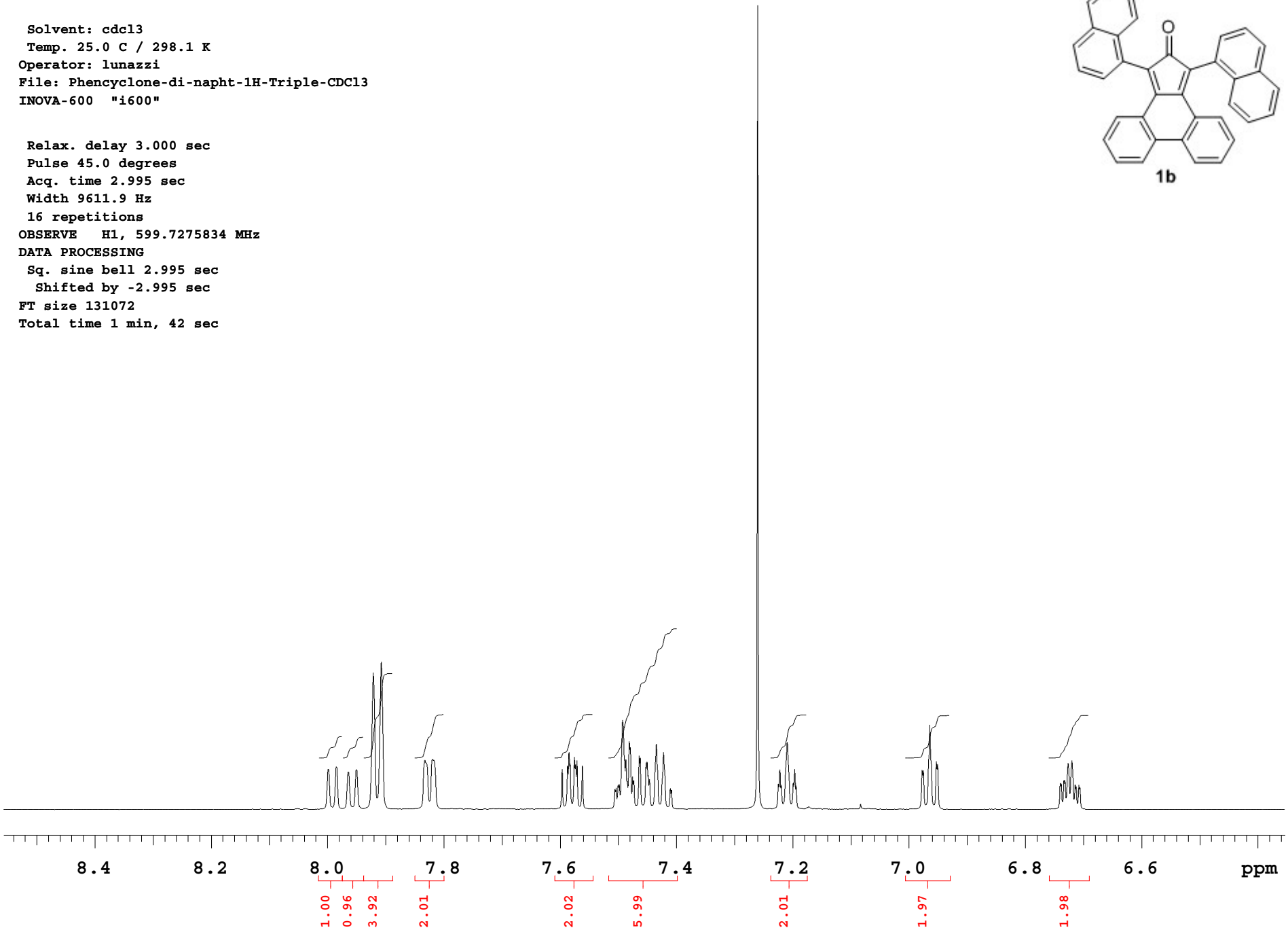
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-di-napht-1H-Triple-CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
16 repetitions
OBSERVE H1, 599.7275834 MHz
DATA PROCESSING
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 1 min, 42 sec

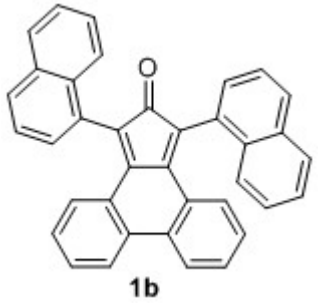


1b



Phencyclone-di-napht-13C-CDCl3 Inova600-ATB C13-s2pul-cdcl3

Automation directory:

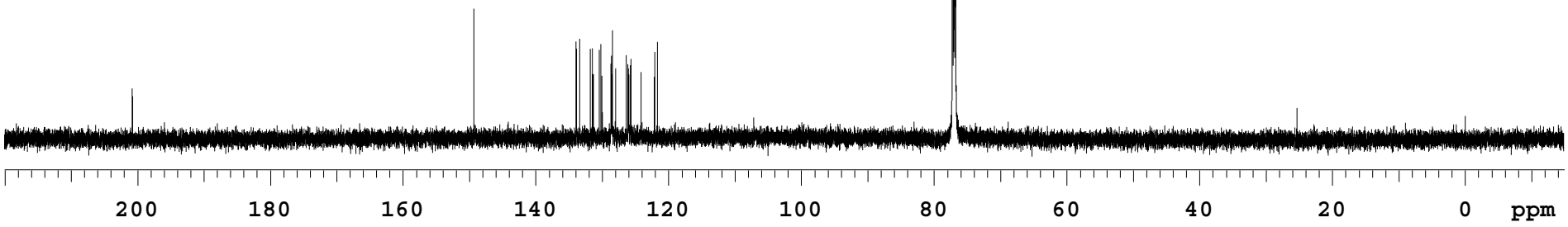


1b

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-di-napht-13C-CDCl3
INOVA-600 "i600"

Relax. delay 6.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
6144 repetitions
OBSERVE C13, 150.8016201 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 11 hr, 57 min, 45 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	30283.9	200.819	8.0
2	30270.1	200.728	6.8
3	22515.8	149.307	20.8
4	20192.5	133.901	15.6
5	20186.5	133.861	14.4
6	20116.3	133.396	16.0
7	20114.1	133.381	15.2
8	19878.4	131.818	14.4
9	19825.4	131.467	14.5
10	19808.3	131.353	10.3
11	19806.1	131.339	10.3
12	19672.5	130.453	14.2
13	19628.8	130.163	15.2
14	19604.5	130.002	10.1
15	19405.2	128.681	9.3
16	19400.8	128.651	12.0
17	19398.6	128.637	13.3
18	19395.3	128.615	11.3
19	19380.4	128.516	10.2
20	19373.8	128.472	17.3
21	19369.9	128.446	14.0
22	19302.5	128.000	11.3
23	19061.3	126.400	13.4
24	19059.1	126.385	12.4
25	19014.9	126.092	11.9
26	19005.0	126.026	11.3
27	19000.0	125.993	10.3
28	18992.8	125.946	8.9
29	18962.4	125.744	11.8
30	18954.7	125.693	12.8
31	18725.0	124.170	10.7
32	18413.7	122.105	13.9
33	18345.7	121.655	15.5
34	11643.7	77.212	1869.0
35	11611.7	77.000	1884.0
36	11579.7	76.788	1831.2

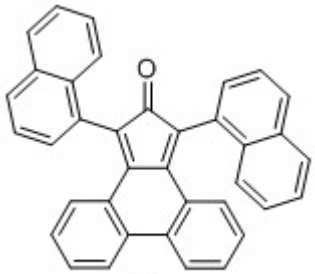


Phencyclone-di-napht-dept-CDCl3 Inova600-ATB C13-DEPT-cdcl3

Automation directory:

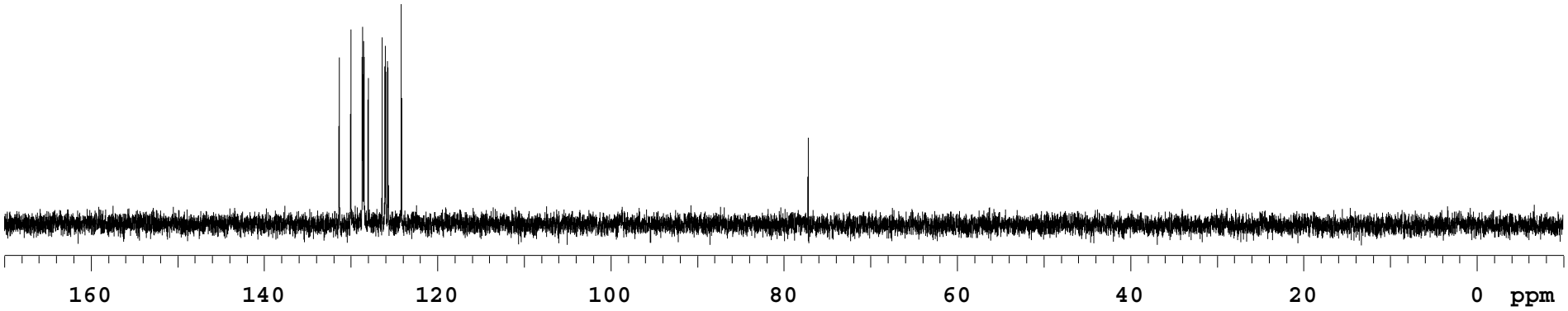
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-di-napht-dept-CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
1024 repetitions
OBSERVE C13, 150.8016201 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 8 min, 48 sec



1b

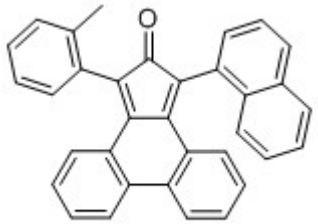
INDEX	FREQUENCY	PPM	HEIGHT
1	19808.3	131.353	26.8
2	19806.1	131.338	26.4
3	19605.1	130.006	31.2
4	19405.2	128.681	26.8
5	19398.6	128.637	31.5
6	19395.3	128.615	25.3
7	19380.4	128.516	29.4
8	19372.7	128.465	26.9
9	19302.5	128.000	23.5
10	19061.3	126.400	30.0
11	19059.1	126.385	29.7
12	19014.9	126.092	25.3
13	19005.0	126.026	28.6
14	19000.0	125.993	25.8
15	18993.4	125.949	24.5
16	18962.4	125.744	26.2
17	18954.7	125.693	25.1
18	18725.0	124.170	35.3



Phencyclone-onapht-otolyl-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

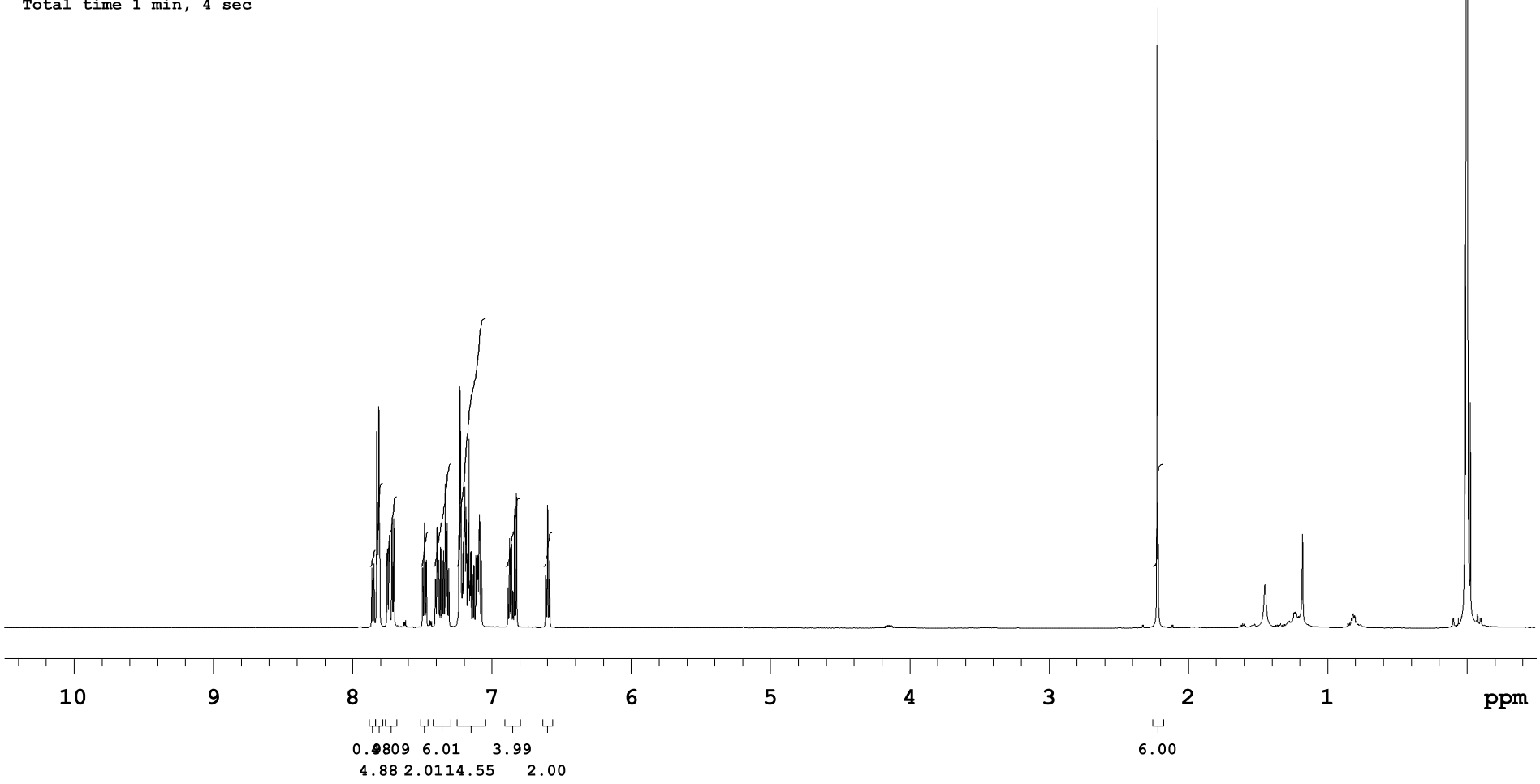
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-onapht-otolyl-1H-CDCl3
INOVA-600 "i600"



1c

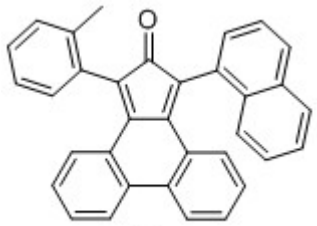
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE H1, 599.7276426 MHz
DATA PROCESSING
FT size 131072
Total time 1 min, 4 sec



Phencyclone_onapth-otolyl-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

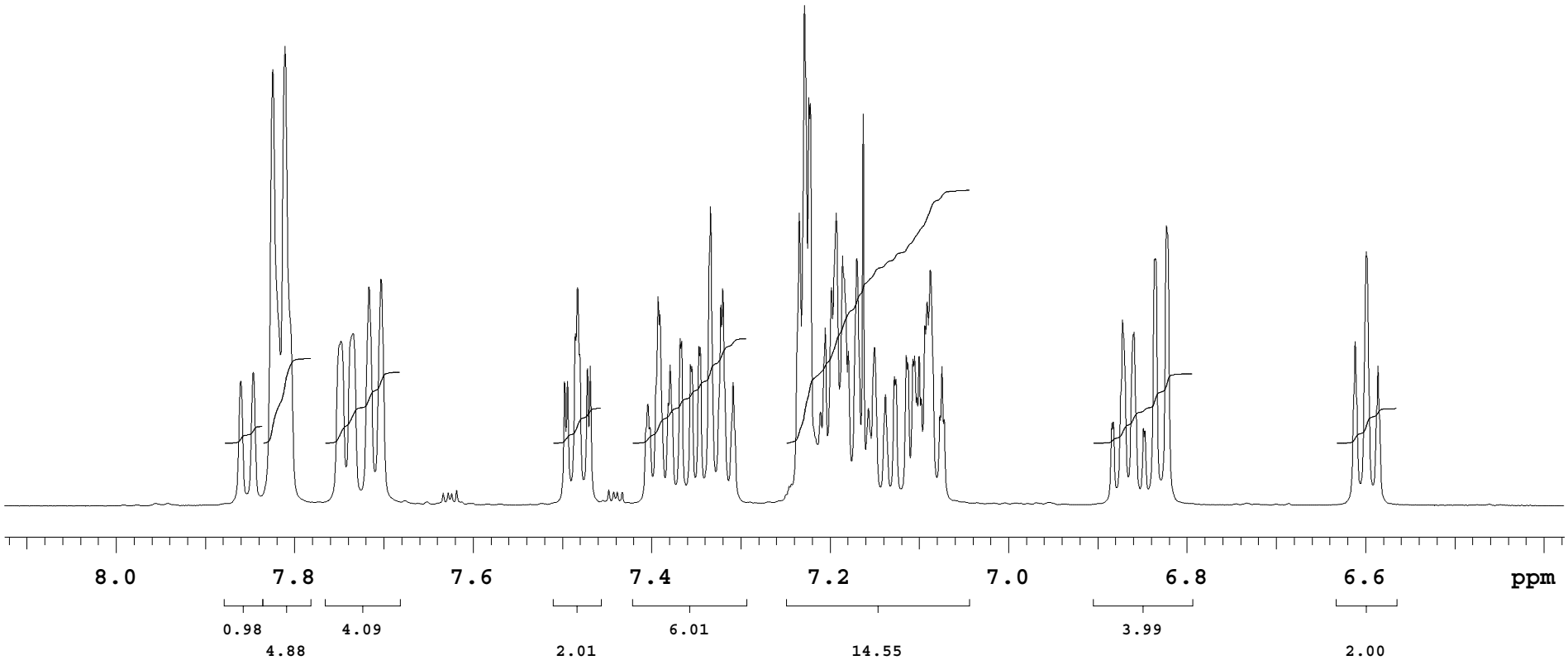
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone_onapth-otolyl-1H-CDCl3
INOVA-600 "i600"



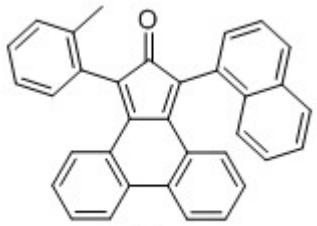
1c

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE H1, 599.7276426 MHz
DATA PROCESSING
FT size 131072
Total time 1 min, 4 sec



Phencyclone-onapht-otolyl-13C-CDCl3 Inova600-swbb 13C-s2pul-cdcl3

Automation directory:

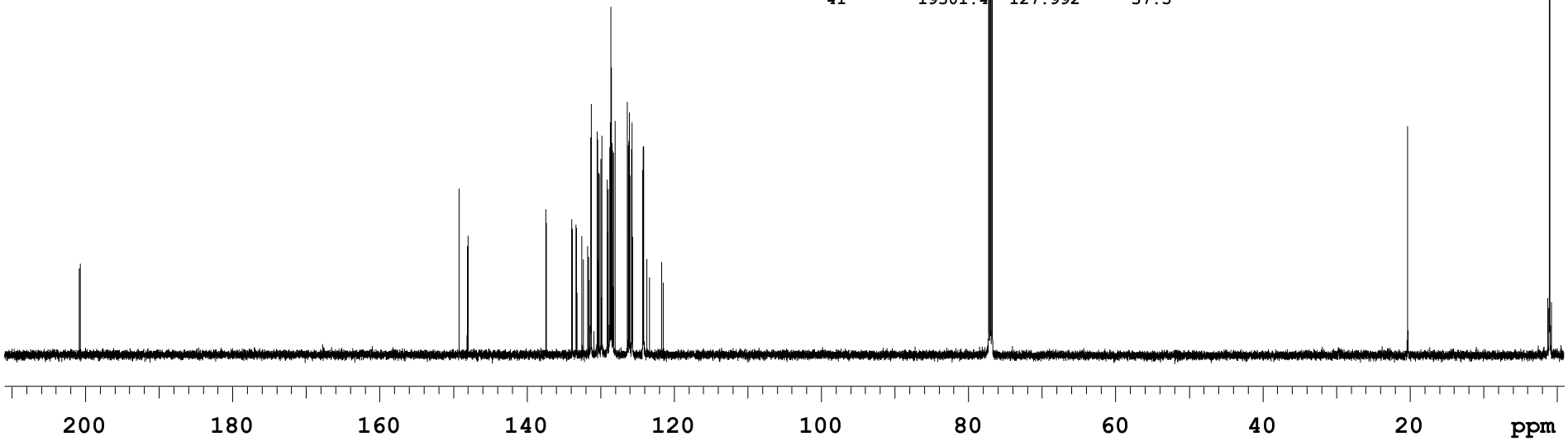


1c

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-onapht-otolyl-13C-CDCl3
INOVA-600 "i600"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
1024 repetitions
OBSERVE C13, 150.8016245 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 25 min, 30 sec

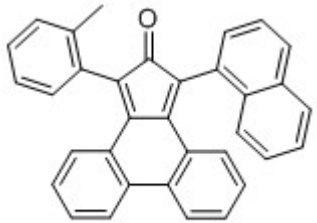
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	30283.3	200.816	13.9	42	19054.6	126.356	40.5
2	30267.3	200.710	14.6	43	19053.0	126.345	34.8
3	22499.8	149.201	26.7	44	19035.3	126.228	33.7
4	22331.9	148.088	17.4	45	19020.4	126.129	34.3
5	22314.8	147.975	19.2	46	19007.7	126.045	38.8
6	20725.3	137.434	23.4	47	19005.5	126.030	34.6
7	20708.2	137.321	21.1	48	18997.2	125.975	28.8
8	20189.2	133.879	21.7	49	18969.6	125.792	33.1
9	20184.8	133.850	20.2	50	18959.1	125.722	37.2
10	20102.5	133.305	19.3	51	18950.9	125.667	34.8
11	20099.8	133.286	20.9	52	18734.4	124.232	29.6
12	20091.5	133.231	20.3	53	18731.7	124.214	33.4
13	20089.3	133.217	19.7	54	18716.8	124.115	32.4
14	19986.0	132.532	19.1	55	18714.5	124.100	33.4
15	19955.7	132.331	15.3	56	18656.0	123.712	15.4
16	19866.8	131.741	17.5	57	18599.7	123.339	12.4
17	19839.7	131.562	15.7	58	18351.3	121.691	14.9
18	19805.5	131.335	29.6	59	18318.7	121.475	11.6
19	19797.8	131.284	34.9	60	11643.2	77.209	392.0
20	19795.0	131.265	40.2	61	11611.7	77.000	402.3
21	19671.9	130.449	35.8	62	11579.7	76.788	401.5
22	19665.3	130.405	34.6	63	3066.8	20.337	36.6
23	19647.1	130.284	15.1	64	3058.5	20.282	35.6
24	19633.8	130.196	29.2	65	152.2	1.010	362.4
25	19593.5	129.929	29.0				
26	19590.7	129.911	31.4				
27	19574.2	129.801	35.2				
28	19466.0	129.083	28.1				
29	19456.6	129.021	19.7				
30	19444.4	128.940	18.8				
31	19441.7	128.922	26.6				
32	19411.9	128.724	28.4				
33	19408.5	128.702	33.2				
34	19394.7	128.611	38.1				
35	19393.1	128.600	55.8				
36	19373.8	128.472	46.1				
37	19372.1	128.461	34.0				
38	19352.2	128.329	32.8				
39	19339.5	128.245	32.4				
40	19334.6	128.212	31.9				
41	19301.4	127.992	37.5				



Phencyclone-onapht-otolyl-DEPT-CDCl3 Inova600-ATB ●1?-DEPT-●d●1?

Automation directory:

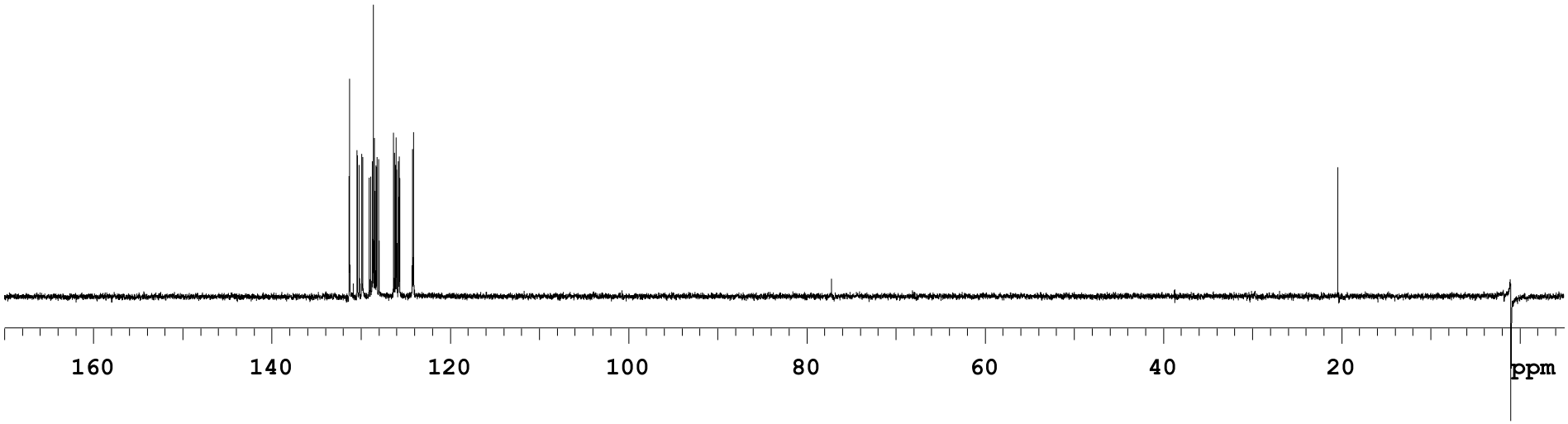
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-onapht-otolyl-DEPT-CDCl3
INOVA-600 "i600"



1c

Relax. delay 4.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
256 repetitions
OBSERVE C13, 150.8016252 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 21 min, 36 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	19805.4	131.334	19.3
2	19795.5	131.268	34.9
3	19671.3	130.445	23.4
4	19665.2	130.404	22.6
5	19633.2	130.192	21.1
6	19592.9	129.925	21.2
7	19590.1	129.907	22.9
8	19573.6	129.797	22.4
9	19465.3	129.079	19.1
10	19440.5	128.914	19.2
11	19411.2	128.720	20.7
12	19408.5	128.702	21.7
13	19392.5	128.596	46.7
14	19372.0	128.460	25.3
15	19373.1	128.468	25.4
16	19351.6	128.325	21.0
17	19338.9	128.241	20.7
18	19334.5	128.211	22.3
19	19300.8	127.988	22.0
20	19054.0	126.352	25.9
21	19052.4	126.341	26.3
22	19034.7	126.223	23.1
23	19019.8	126.125	21.1
24	19007.1	126.040	24.9
25	19004.9	126.026	25.5
26	18996.6	125.971	20.3
27	18969.0	125.788	21.7
28	18959.1	125.722	22.5
29	18950.8	125.667	21.7
30	18733.8	124.228	23.1
31	18731.0	124.210	23.6
32	18716.1	124.111	25.6
33	18714.5	124.100	26.4
34	3066.8	20.337	19.3
35	3058.5	20.282	19.3
36	151.6	1.005	-24.2

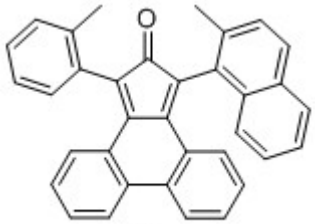


Phencyclone_2Menaph_otolyl_1H_CDCl3 Inova600-Triple H1-s2pul-cdcl3

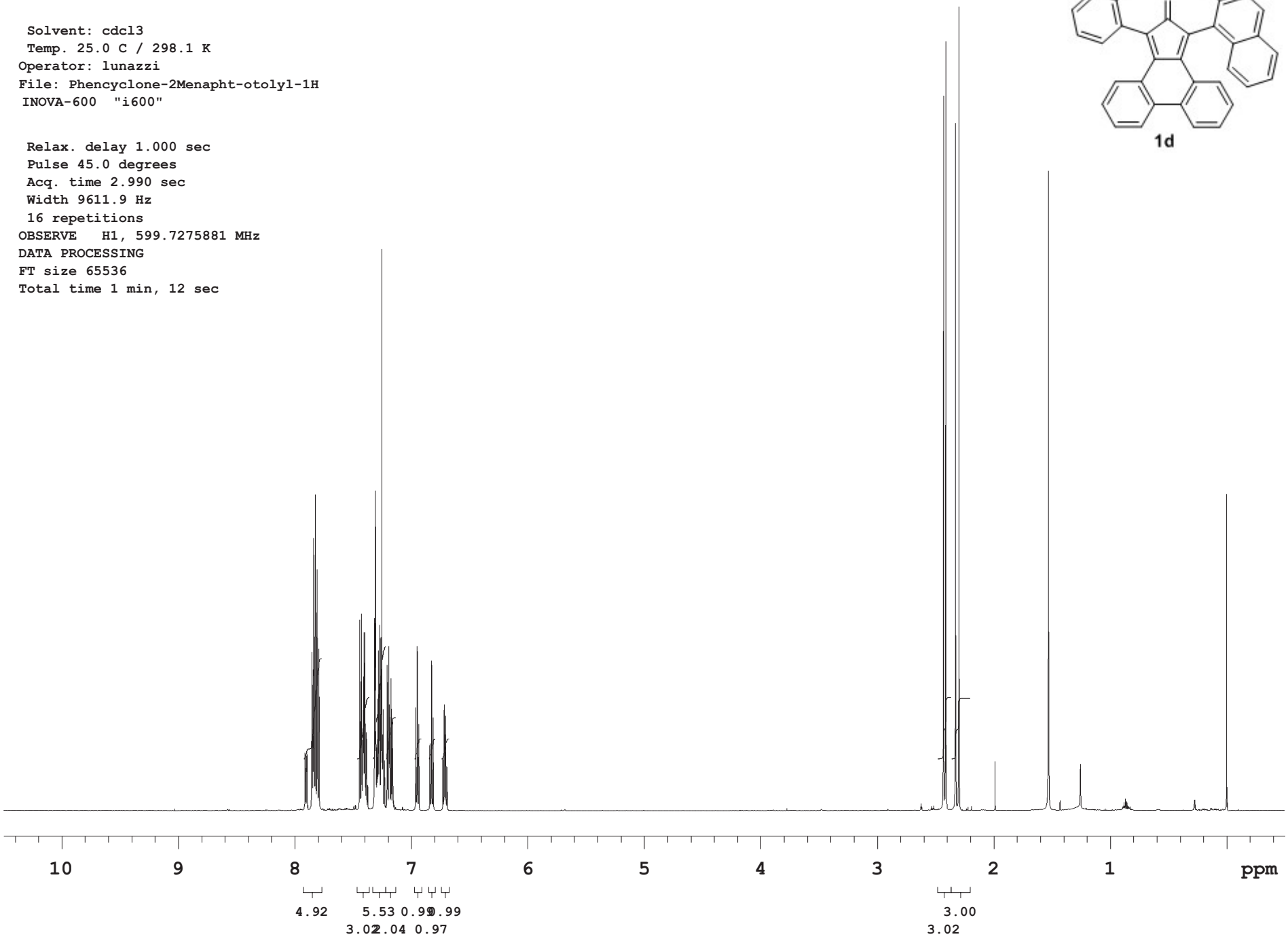
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone-2Menapht-otolyl-1H
INOVA-600 "i600"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7275881 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 12 sec



1d

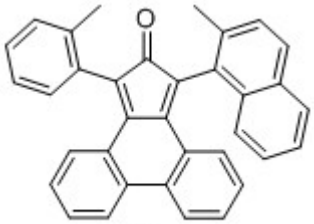


Phencyclone_2Menaph_otolyl_1H_CDCl3 Inova600-Triple H1-s2pul-cdcl3

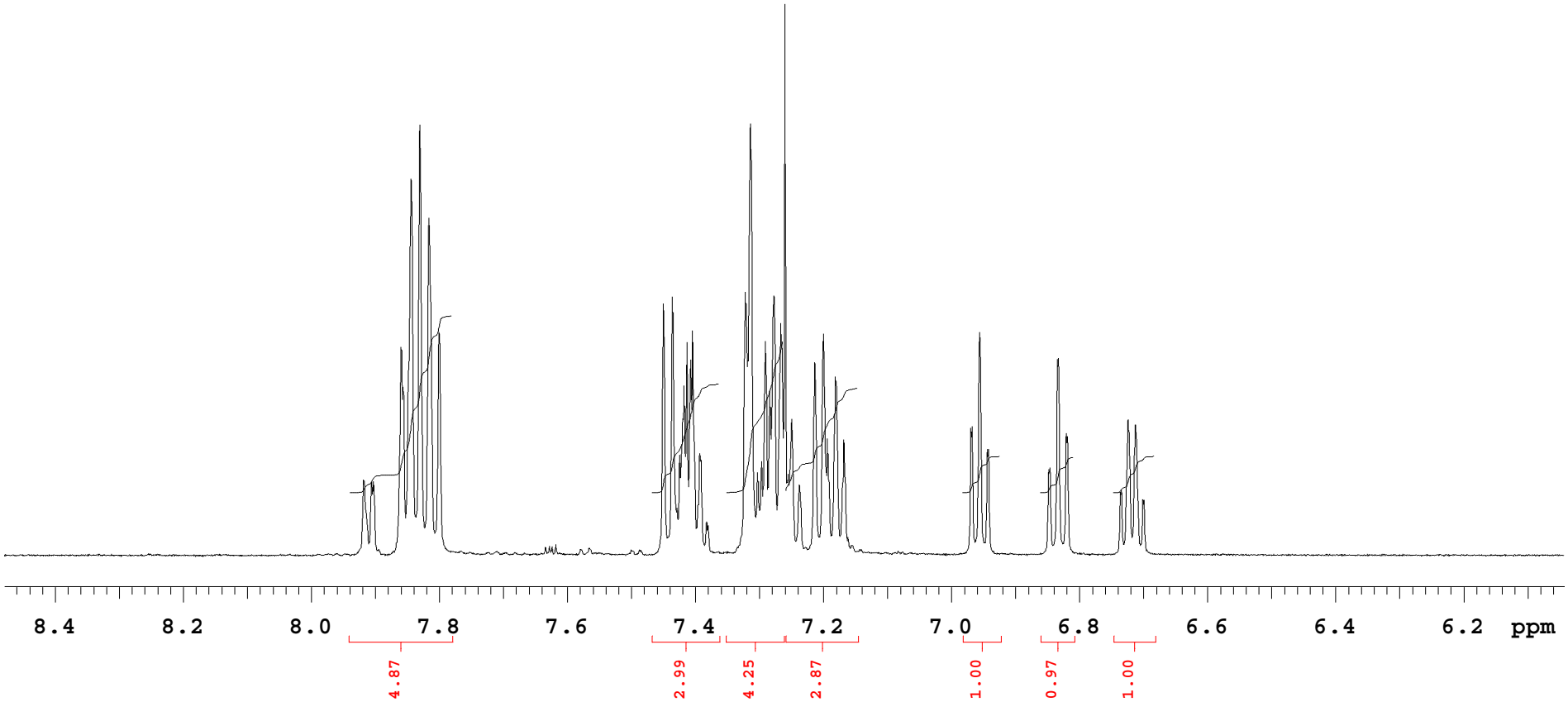
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone_2Menaph_otolyl_1H_CDCl3
INOVA-600 "i600"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
4 repetitions
OBSERVE H1, 599.7275833 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 24 sec

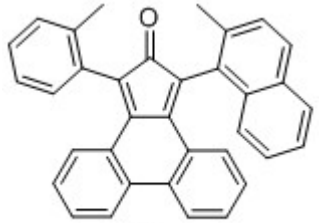


1d



Phencyclone-2Me-napht-otolyl-13C-CDCl3 Inova600-ATB C13-s2pul-cdcl3

Automation directory:

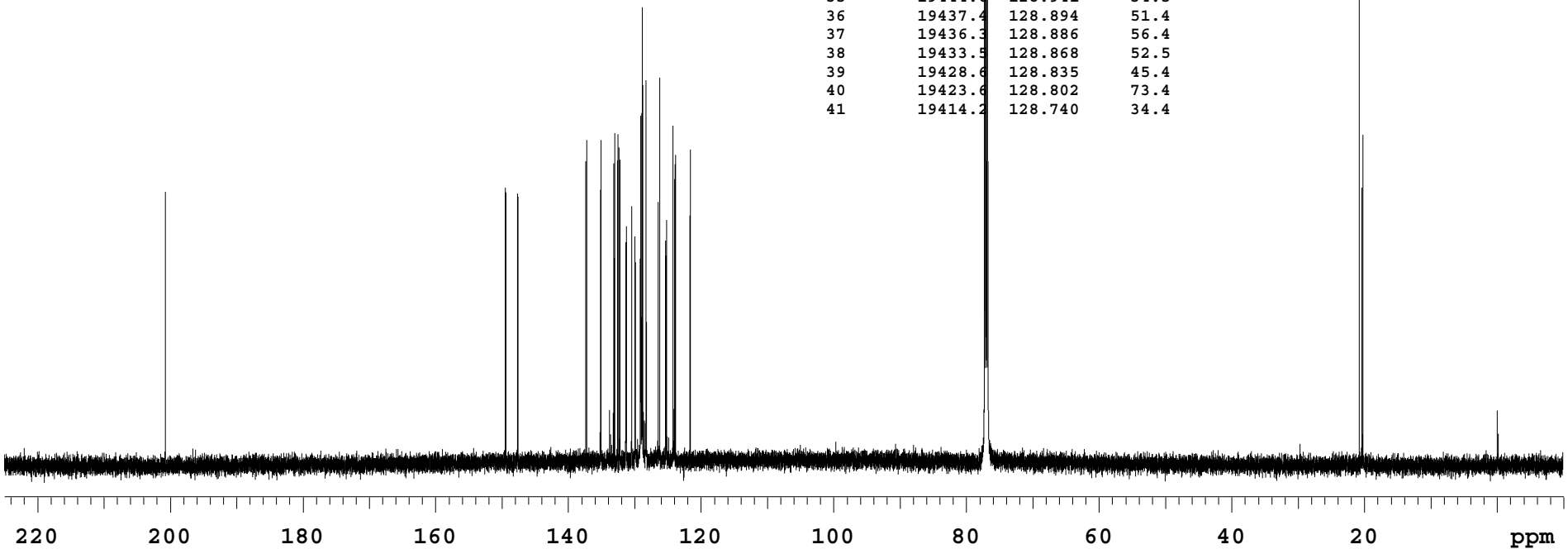


1d

Solvent: cdcl3
Ambient temperature
Operator: lunazzi
File: Phencyclone-2Me-napht-otolyl-13C-CDCl3
INOVA-600 "i600"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
9000 repetitions
OBSERVE C13, 150.8016194 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 15 hr, 1 min, 24 sec

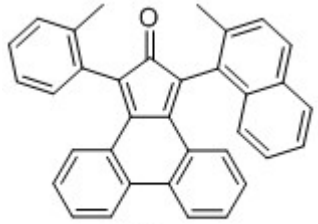
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	30270.2	200.729	43.8	42	19412.0	128.725	32.2
2	30266.9	200.707	35.0	43	19349.1	128.308	37.4
3	22537.5	149.451	44.5	44	19346.8	128.293	61.7
4	22532.5	149.418	43.7	45	19343.0	128.268	35.4
5	22263.6	147.635	43.5	46	19340.2	128.249	56.7
6	22254.2	147.573	43.0	47	19073.0	126.477	42.2
7	20716.1	137.373	48.7	48	19070.8	126.463	36.2
8	20694.0	137.227	52.1	49	19034.9	126.225	62.1
9	20383.7	135.169	44.1	50	18899.1	125.324	36.0
10	20371.6	135.088	52.1	51	18882.0	125.211	39.3
11	20083.4	133.177	46.1	52	18877.0	125.178	36.1
12	20080.6	133.159	48.4	53	18872.0	125.145	31.0
13	20060.2	133.024	53.2	54	18731.8	124.215	54.4
14	20059.1	133.016	44.7	55	18700.9	124.010	45.8
15	19989.5	132.555	48.8	56	18687.6	123.922	48.2
16	19987.8	132.544	53.1	57	18674.9	123.838	49.7
17	19959.1	132.354	48.9	58	18348.1	121.670	39.9
18	19957.5	132.343	50.9	59	18339.8	121.615	50.6
19	19953.1	132.313	47.6	60	11646.1	77.228	2355.7
20	19930.4	132.163	49.0	61	11614.6	77.019	2382.7
21	19801.2	131.306	34.3	62	11582.6	76.807	2388.5
22	19799.6	131.295	35.7	63	3131.5	20.766	101.3
23	19795.2	131.266	38.3	64	3069.1	20.352	44.4
24	19673.1	130.457	39.0	65	3053.7	20.250	52.9
25	19670.4	130.439	41.5				
26	19598.1	129.959	36.7				
27	19583.1	129.860	32.5				
28	19477.1	129.157	33.1				
29	19470.5	129.113	34.9				
30	19468.9	129.102	56.0				
31	19466.1	129.084	48.7				
32	19462.8	129.062	37.2				
33	19460.6	129.048	32.7				
34	19446.8	128.956	32.2				
35	19444.6	128.941	34.5				
36	19437.4	128.894	51.4				
37	19436.3	128.886	56.4				
38	19433.5	128.868	52.5				
39	19428.6	128.835	45.4				
40	19423.6	128.802	73.4				
41	19414.2	128.740	34.4				



Phencyclone-2Me-napht-otolyl-DEPT-CDCl3 Inova600-ATB C13-DEPT-cdcl3

Automation directory:

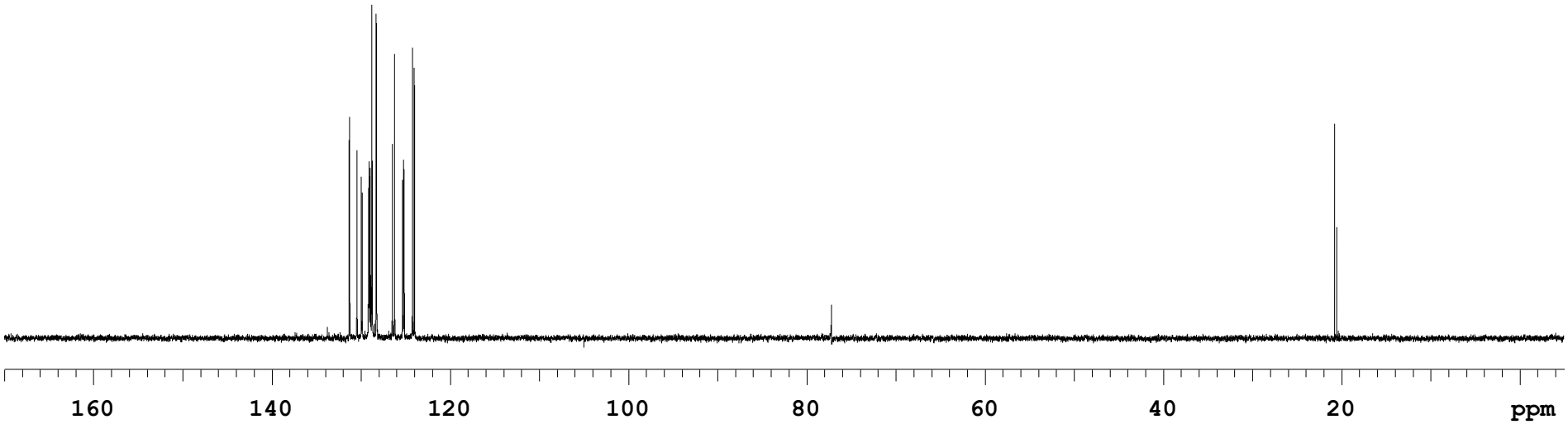
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazziOperator: lunazzi
File: Phencyclone-2Me-napht-otolyl-DEPT-CDCl3
INOVA-600 "i600"



1d

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
2048 repetitions
OBSERVE C13, 150.8016194 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 2 hr, 17 min, 28 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	19801.3	131.307	31.6
2	19799.6	131.296	31.8
3	19795.7	131.270	35.4
4	19673.2	130.457	27.1
5	19670.4	130.439	30.1
6	19598.1	129.959	25.8
7	19583.2	129.860	23.3
8	19477.2	129.158	24.1
9	19470.5	129.114	23.6
10	19462.8	129.062	28.3
11	19460.6	129.048	25.9
12	19446.8	128.956	25.8
13	19444.6	128.942	27.2
14	19423.6	128.802	53.4
15	19414.2	128.740	28.4
16	19412.0	128.726	26.6
17	19346.9	128.294	51.9
18	19342.5	128.264	34.1
19	19340.2	128.250	50.4
20	19073.0	126.478	31.1
21	19070.8	126.463	28.1
22	19034.9	126.225	45.5
23	18899.1	125.324	25.3
24	18882.0	125.211	28.5
25	18877.0	125.178	27.0
26	18872.1	125.145	22.9
27	18731.8	124.215	46.5
28	18700.9	124.010	43.3
29	3131.5	20.766	50.6
30	3069.1	20.352	26.3
31	3053.7	20.250	23.4

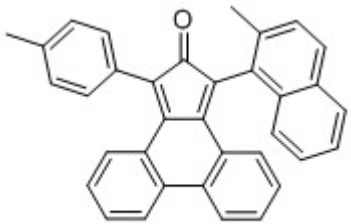


Phencyclone_2Menaph_ptolyl_1H_CDCl3 Inova600-Triple H1-s2pul-cdcl3

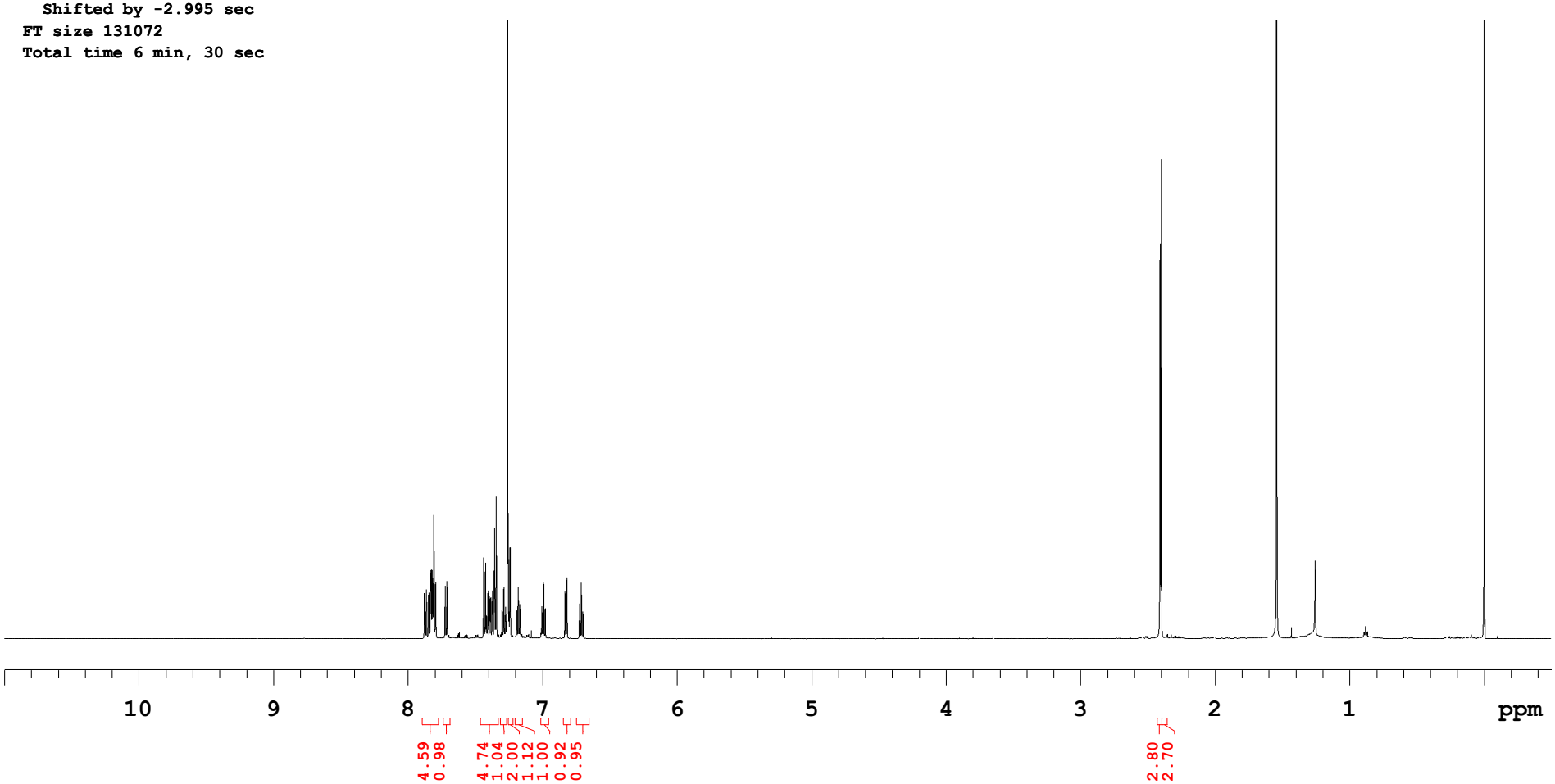
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone_2Menaph_ptolyl_1H_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
64 repetitions
OBSERVE   H1, 599.7275850 MHz
DATA PROCESSING
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 6 min, 30 sec



1e

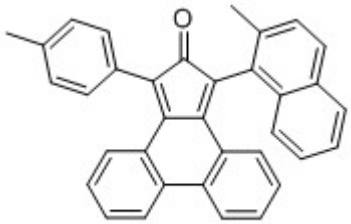


Phencyclone_2Menaph_ptolyl_1H_CDCl3 Inova600-Triple H1-s2pul-cdcl3

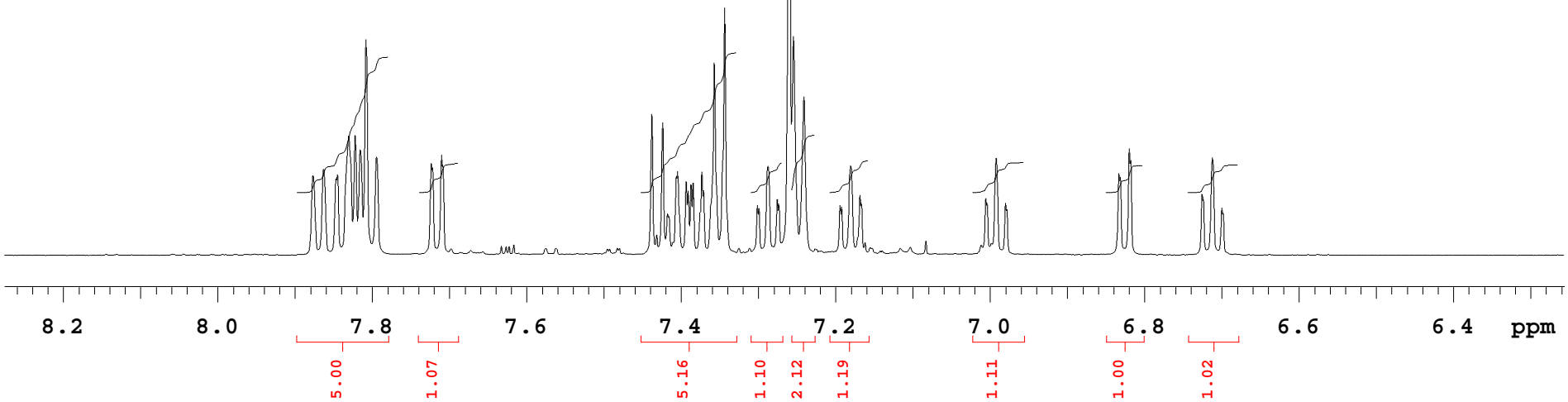
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone_2Menaph_ptolyl_1H_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
64 repetitions
OBSERVE   H1, 599.7275846 MHz
DATA PROCESSING
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 6 min, 30 sec

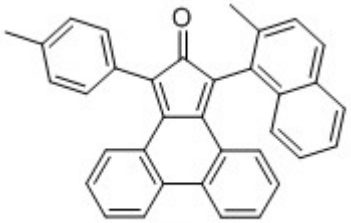


1e



Phencyclone_2Menapht_ptolil-13C_CDCl3 Inova600-ATB C13-s2pul-cdcl3

Automation directory:

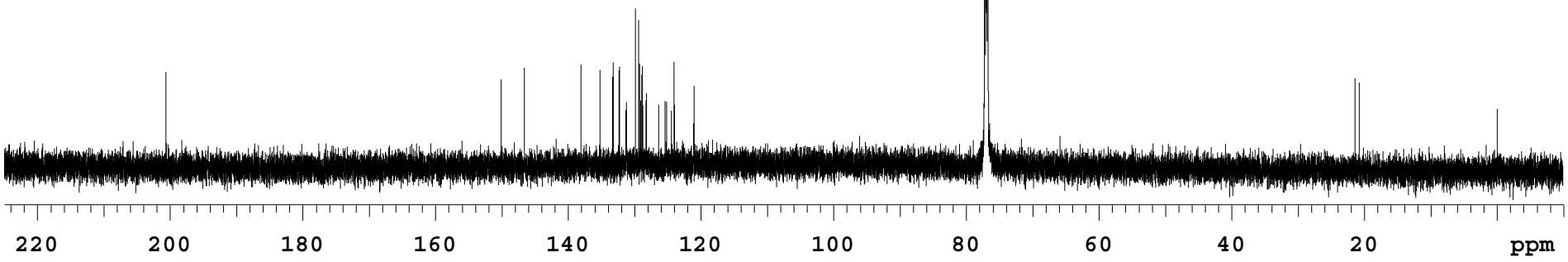


1e

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone_2Menapht_ptolil-13C_CDCl3
INOVA-600 "i600"

Relax. delay 6.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
32 repetitions
OBSERVE C13, 150.8016207 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Resol. enhancement -0.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 11 hr, 57 min, 45 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	30255.7	200.633	16.2
2	22641.7	150.142	15.1
3	22110.0	146.616	16.9
4	20821.4	138.071	17.4
5	20394.6	135.241	16.6
6	20100.9	133.294	15.5
7	20090.4	133.224	17.8
8	19951.8	132.305	16.5
9	19950.2	132.294	17.1
10	19795.0	131.265	10.1
11	19785.6	131.203	11.5
12	19579.7	129.837	26.4
13	19507.9	129.361	24.6
14	19486.9	129.222	17.6
15	19467.6	129.094	11.1
16	19459.3	129.039	11.7
17	19434.5	128.875	15.8
18	19428.4	128.834	16.5
19	19423.5	128.801	17.2
20	19336.2	128.223	12.0
21	19331.3	128.190	12.9
22	19059.1	126.385	11.0
23	18907.2	125.378	11.6
24	18873.0	125.151	11.5
25	18764.8	124.434	10.1
26	18709.6	124.068	17.9
27	18693.6	123.961	11.0
28	18256.3	121.062	14.1
29	11643.2	77.209	3023.0
30	11611.2	76.996	3005.7
31	11579.7	76.788	3008.4
32	3231.9	21.431	15.3
33	3137.5	20.805	14.6
34	-2.9	-0.019	10.3

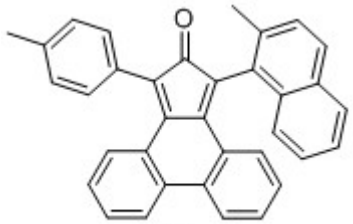


Phencyclone_2Menapht_ptolil-DEPT_CDCl3 Inova600-ATB C13-DEPT-cdcl3

Automation directory:

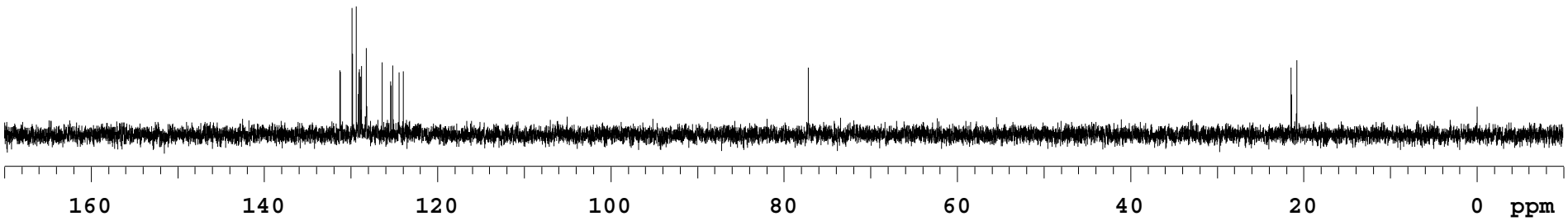
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Phencyclone_2Menapht_ptolil-DEPT_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
1440 repetitions
OBSERVE C13, 150.8016207 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 36 min, 42 sec



1e

INDEX	FREQUENCY	PPM	HEIGHT
1	19795.0	131.265	10.5
2	19785.6	131.203	10.2
3	19579.7	129.837	20.5
4	19507.9	129.362	20.7
5	19467.6	129.094	10.1
6	19459.3	129.039	10.6
7	19436.7	128.889	9.4
8	19421.2	128.787	11.1
9	19336.8	128.227	14.0
10	19331.3	128.190	11.4
11	19059.1	126.385	11.7
12	18907.2	125.378	8.6
13	18873.0	125.151	11.2
14	18764.2	124.430	10.1
15	18693.6	123.961	10.3
16	11642.1	77.201	10.8
17	3231.9	21.431	10.8
18	3138.0	20.809	12.0

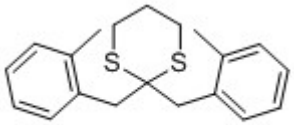


di-otolyl-dithiane-1H-CDCl3_ATB Inova600-ATB H1-s2pul-cdcl3

Automation directory:

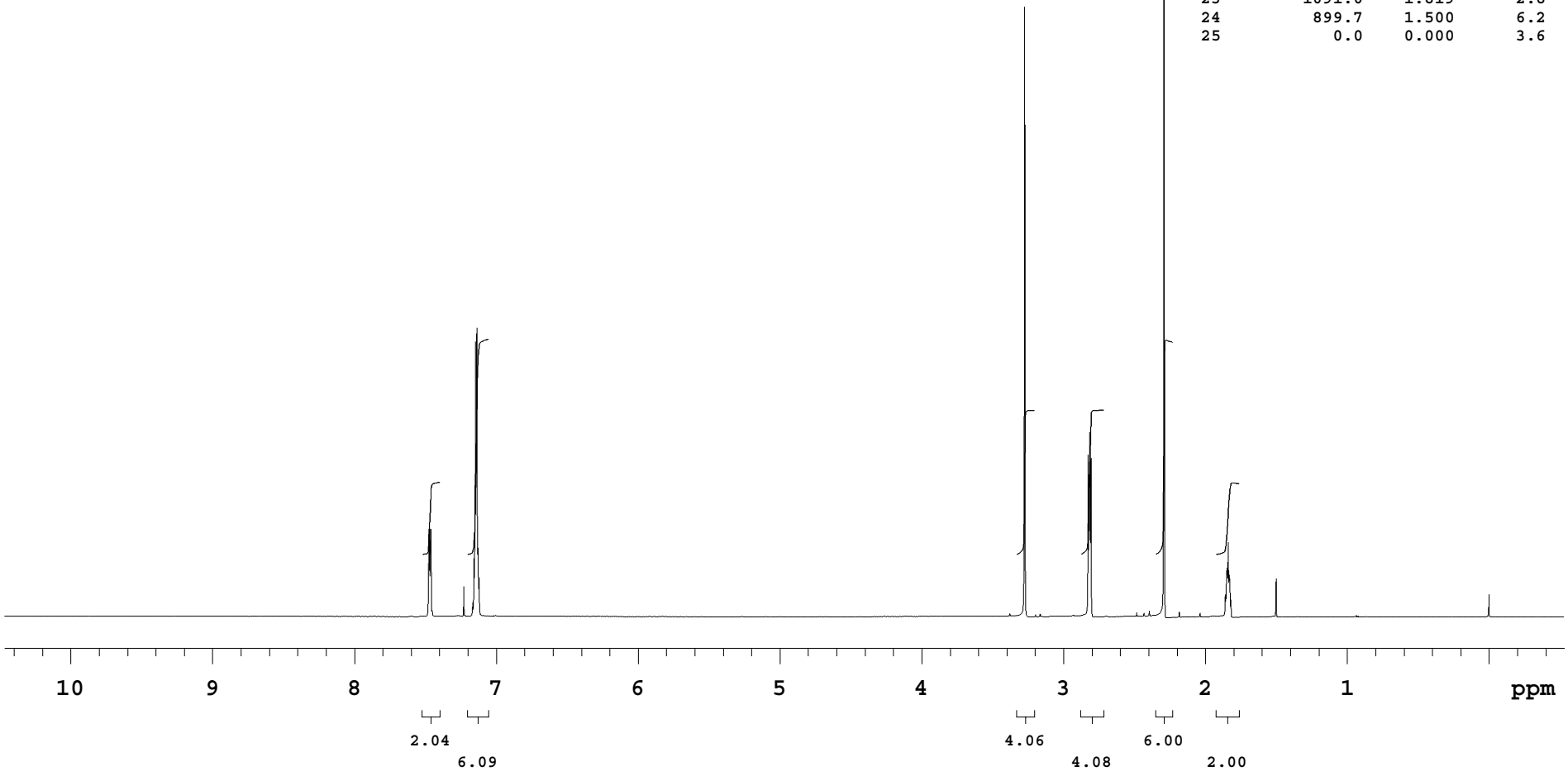
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-otolyl-ditiano-1H-CDCl3_ATB
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.986 sec
Width 10810.8 Hz
16 repetitions
OBSERVE   H1, 599.7276021 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 42 sec



2a

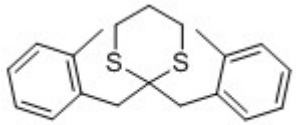
INDEX	FREQUENCY	PPM	HEIGHT
1	4481.6	7.473	14.1
2	4476.7	7.465	14.1
3	4474.0	7.460	14.1
4	4335.1	7.229	5.0
5	4296.9	7.165	2.4
6	4291.6	7.156	9.4
7	4285.7	7.146	44.2
8	4283.0	7.142	27.8
9	4279.7	7.136	46.3
10	4274.8	7.128	11.1
11	4270.8	7.121	6.4
12	1963.0	3.273	97.7
13	1694.5	2.825	26.0
14	1688.5	2.815	29.6
15	1682.6	2.806	25.5
16	1373.5	2.290	151.2
17	1114.5	1.858	3.7
18	1108.5	1.848	7.5
19	1106.2	1.845	7.9
20	1102.6	1.838	12.0
21	1099.3	1.833	7.5
22	1097.0	1.829	6.3
23	1091.0	1.819	2.8
24	899.7	1.500	6.2
25	0.0	0.000	3.6



di-otolyl-dithiane-13C-CDCl3_ATB Inova600-ATB H1-s2pul-cdcl3

Automation directory:

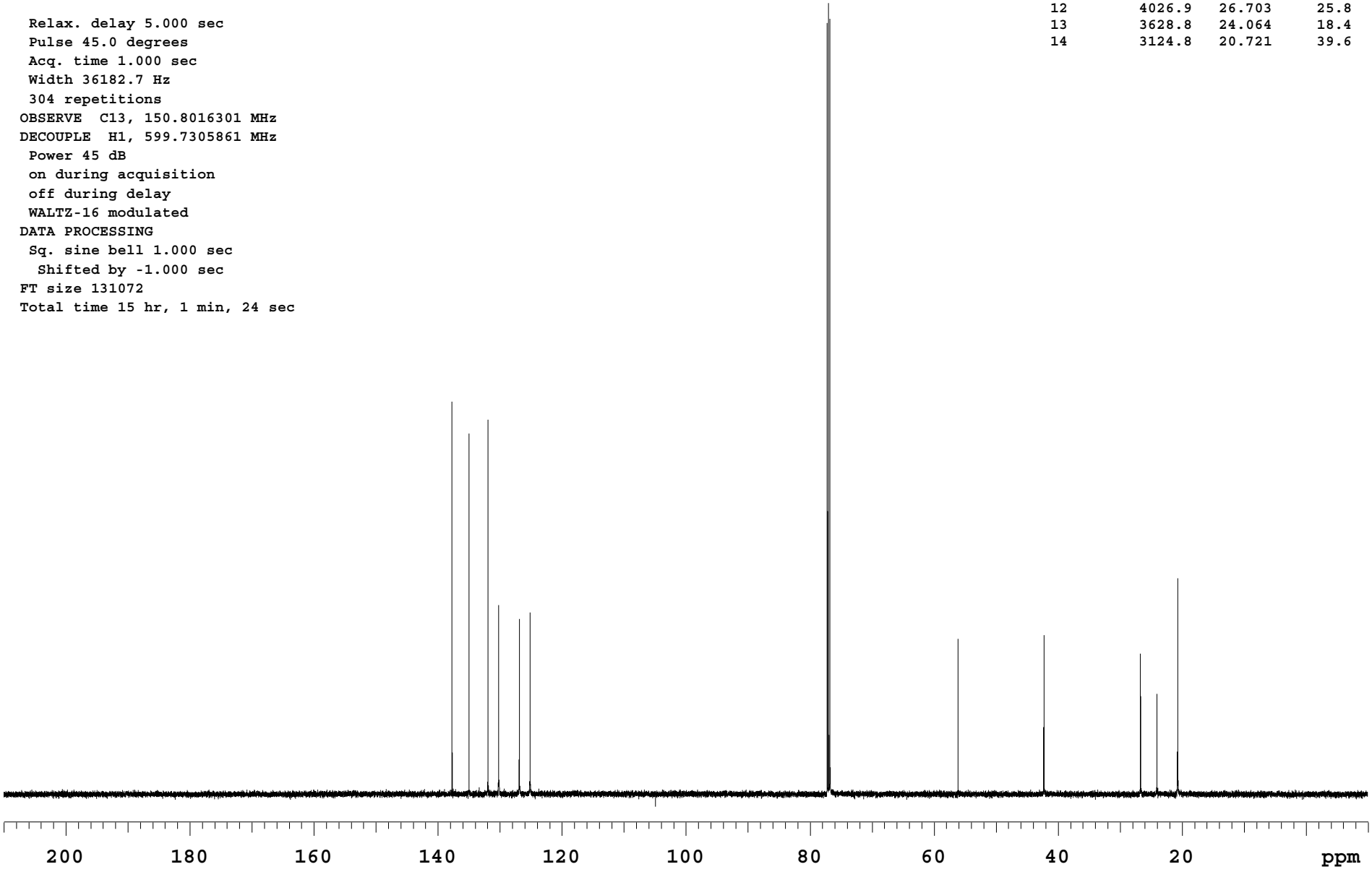
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-otolil-ditiano-13C-CDCl3_ATB
INOVA-600 "i600"



2a

INDEX	FREQUENCY	PPM	HEIGHT
1	20766.7	137.709	72.0
2	20356.0	134.985	66.1
3	19897.2	131.943	68.7
4	19633.3	130.193	34.7
5	19133.0	126.876	32.2
6	18871.3	125.140	33.4
7	11643.7	77.212	141.3
8	11611.7	77.000	145.0
9	11579.7	76.788	142.1
10	8463.6	56.124	28.5
11	6376.7	42.285	29.3
12	4026.9	26.703	25.8
13	3628.8	24.064	18.4
14	3124.8	20.721	39.6

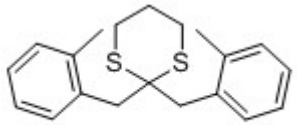
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
304 repetitions
OBSERVE C13, 150.8016301 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 15 hr, 1 min, 24 sec



di-tolyl-dithiane-DEPT-CDCl3 Inova600-Triple C13-DEPT-cdcl3

Automation directory:

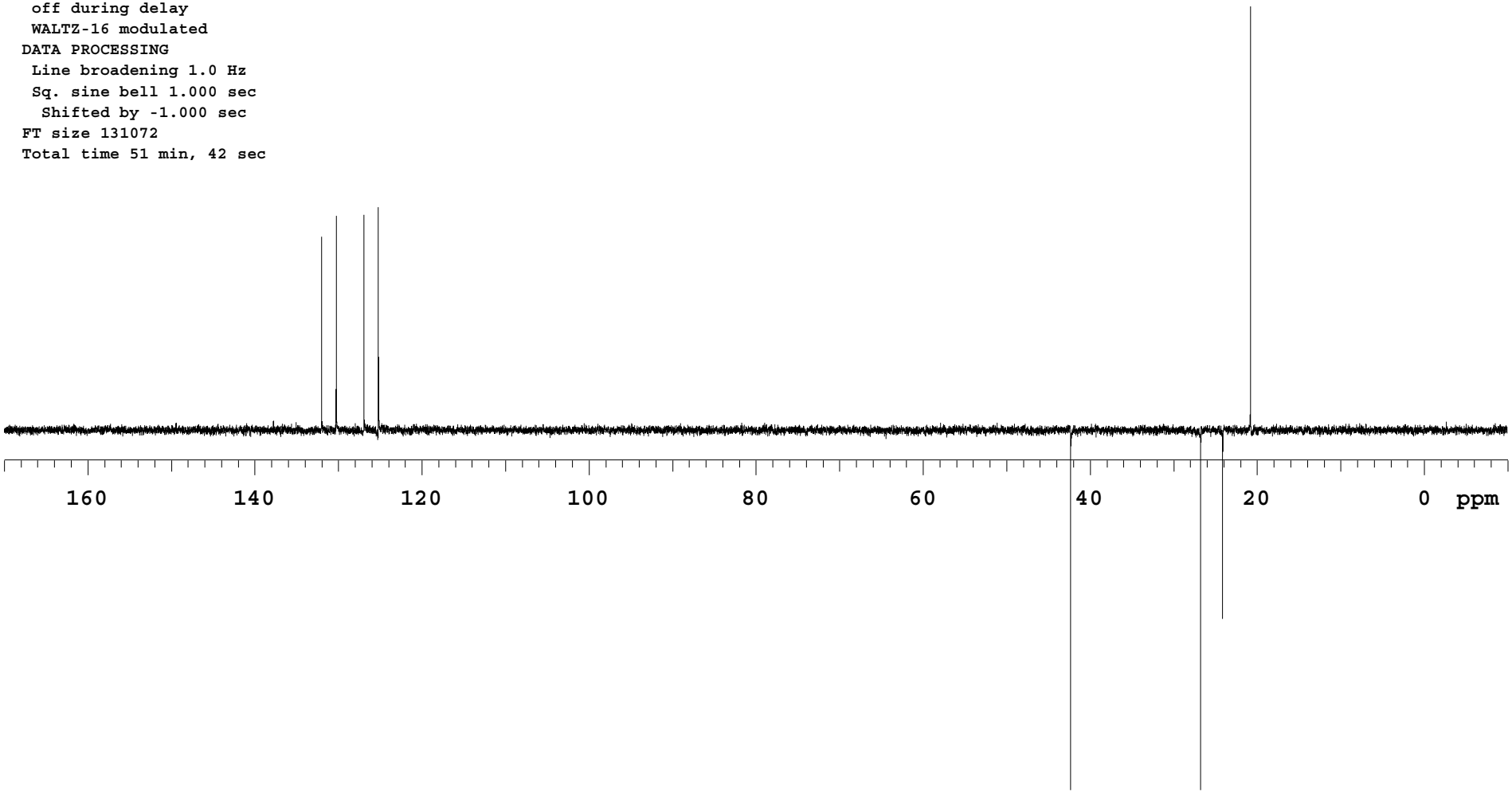
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-tolyl-ditiano-DEPT-CDCl3
INOVA-600 "i600"



2a

INDEX	FREQUENCY	PPM	HEIGHT
1	19906.5	132.005	32.1
2	19642.1	130.251	35.5
3	19142.4	126.938	35.7
4	18880.7	125.202	37.0
5	6384.9	42.340	-61.2
6	4035.7	26.762	-62.7
7	3638.2	24.126	-31.4
8	3134.1	20.783	70.4

Relax. delay 2.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
80 repetitions
OBSERVE C13, 150.8016218 MHz
DECOUPLE H1, 599.7305861 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 51 min, 42 sec

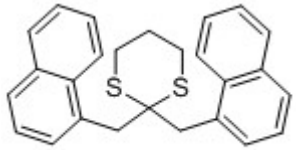


di-napht-dithiane-1H-CDCl3 Inova600-Triple H1-s2pul-cdcl3

Automation directory:

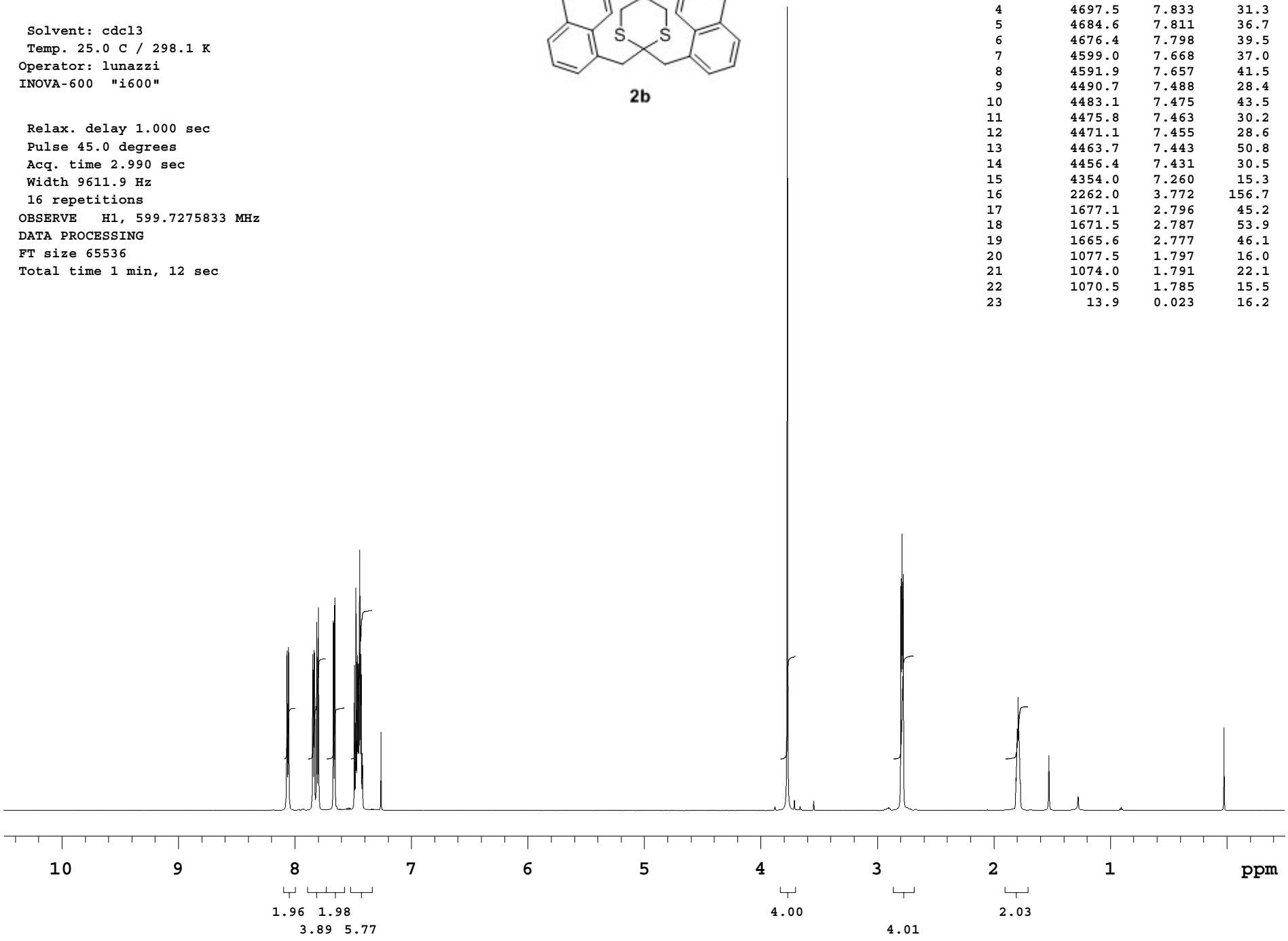
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7275833 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 12 sec



2b

INDEX	FREQUENCY	PPM	HEIGHT
1	4837.7	8.067	31.2
2	4829.5	8.053	31.9
3	4704.8	7.845	30.5
4	4697.5	7.833	31.3
5	4684.6	7.811	36.7
6	4676.4	7.798	39.5
7	4599.0	7.668	37.0
8	4591.9	7.657	41.5
9	4490.7	7.488	28.4
10	4483.1	7.475	43.5
11	4475.8	7.463	30.2
12	4471.1	7.455	28.6
13	4463.7	7.443	50.8
14	4456.4	7.431	30.5
15	4354.0	7.260	15.3
16	2262.0	3.772	156.7
17	1677.1	2.796	45.2
18	1671.5	2.787	53.9
19	1665.6	2.777	46.1
20	1077.5	1.797	16.0
21	1074.0	1.791	22.1
22	1070.5	1.785	15.5
23	13.9	0.023	16.2

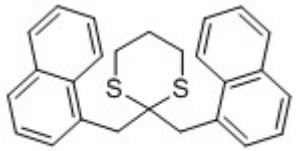


di-napht-dithiane-13C-CDCl3 Inova600-Triple C13-DEPT-cdcl3

Automation directory:

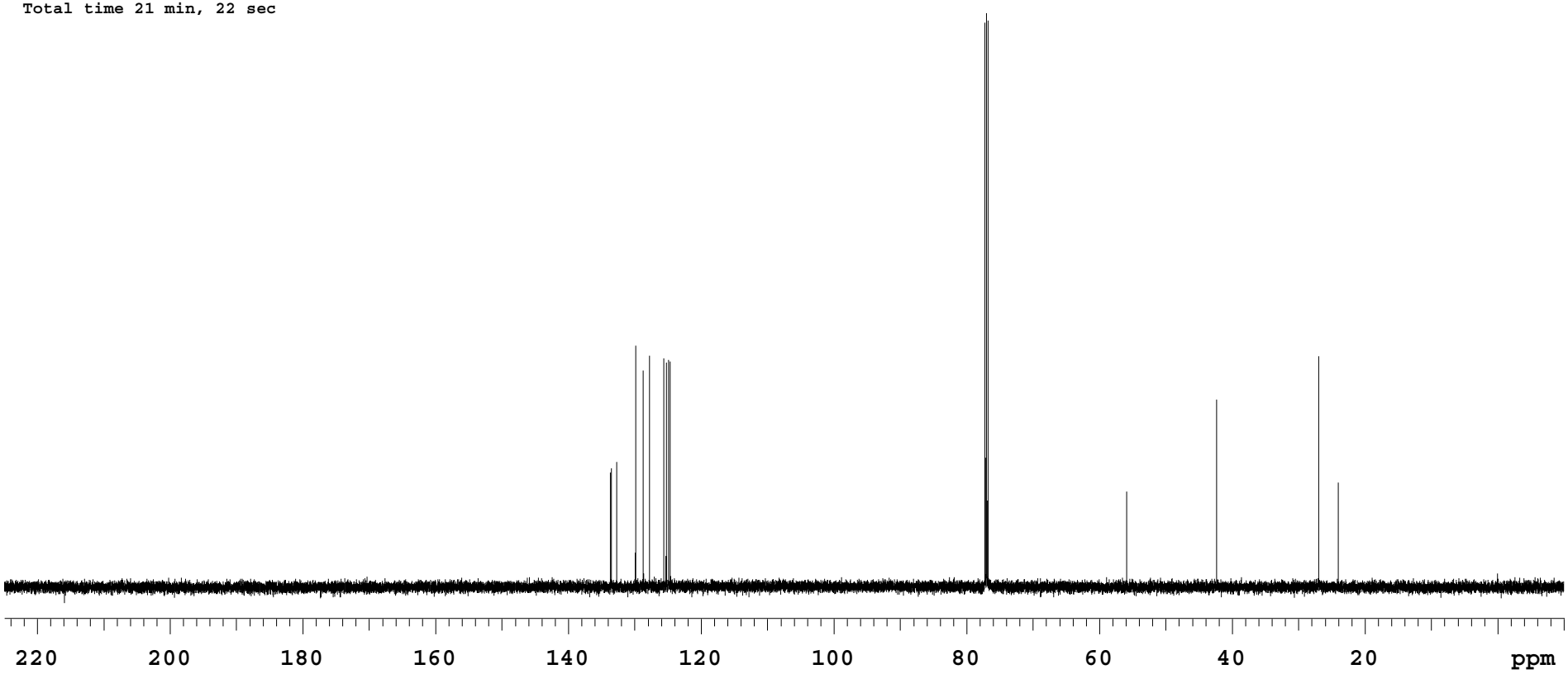
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-napht-ditiano-13C-CDCl3
INOVA-600 "i600"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
256 repetitions
OBSERVE C13, 150.8016256 MHz
DECOUPLE H1, 599.7305861 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 21 min, 22 sec



2b

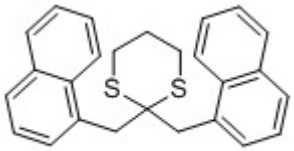
INDEX	FREQUENCY	PPM	HEIGHT
1	20153.3	133.641	18.4
2	20129.0	133.480	19.1
3	20007.6	132.675	20.1
4	19584.7	129.870	38.7
5	19411.3	128.721	34.8
6	19267.8	127.769	37.1
7	18942.6	125.612	36.7
8	18887.4	125.246	36.0
9	18835.5	124.902	36.4
10	18797.9	124.653	36.1
11	11643.7	77.212	90.5
12	11611.7	77.000	92.0
13	11579.7	76.788	90.8
14	8428.3	55.890	15.3
15	6383.8	42.333	30.1
16	4065.0	26.956	37.0
17	3617.3	23.987	16.7



di-napht-dithiane-DEPT-CDCl3 Inova600-Triple C13-DEPT-cdcl3

Automation directory:

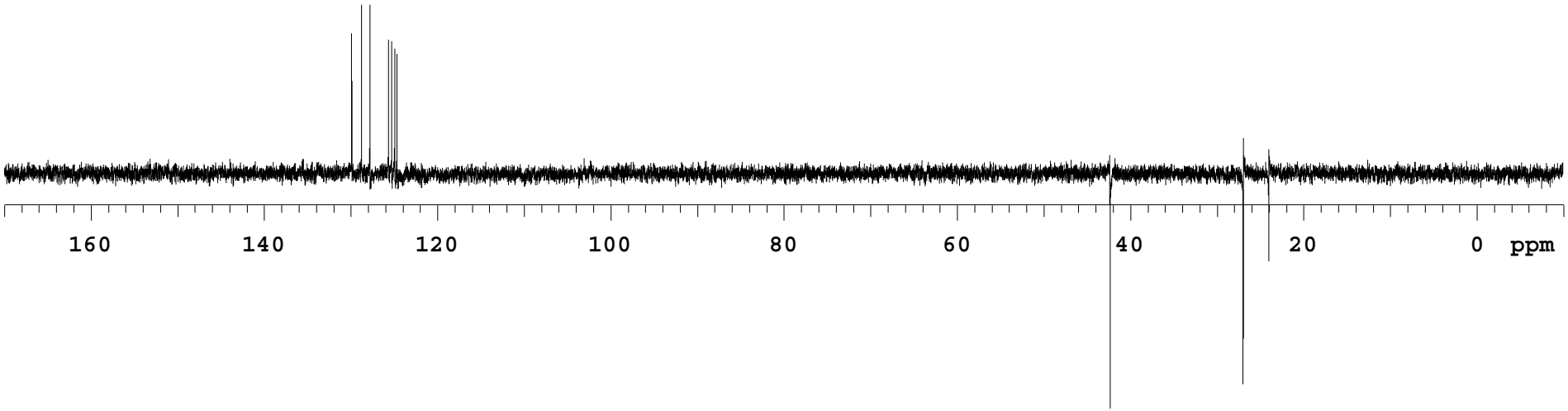
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"



2b

INDEX	FREQUENCY	PPM	HEIGHT
1	19588.5	129.896	22.4
2	19415.7	128.750	27.1
3	19272.2	127.798	27.0
4	18947.0	125.642	21.4
5	18891.2	125.272	21.2
6	18839.3	124.928	20.0
7	18801.8	124.679	19.1
8	6387.1	42.355	-37.6
9	4069.4	26.985	-33.7
10	3621.6	24.016	-14.1

Relax. delay 2.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
64 repetitions
OBSERVE C13, 150.8016218 MHz
DECOUPLE H1, 599.7305861 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 13 min, 0 sec

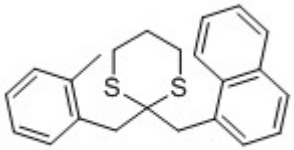


napht-o-tolyl-dithiane-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

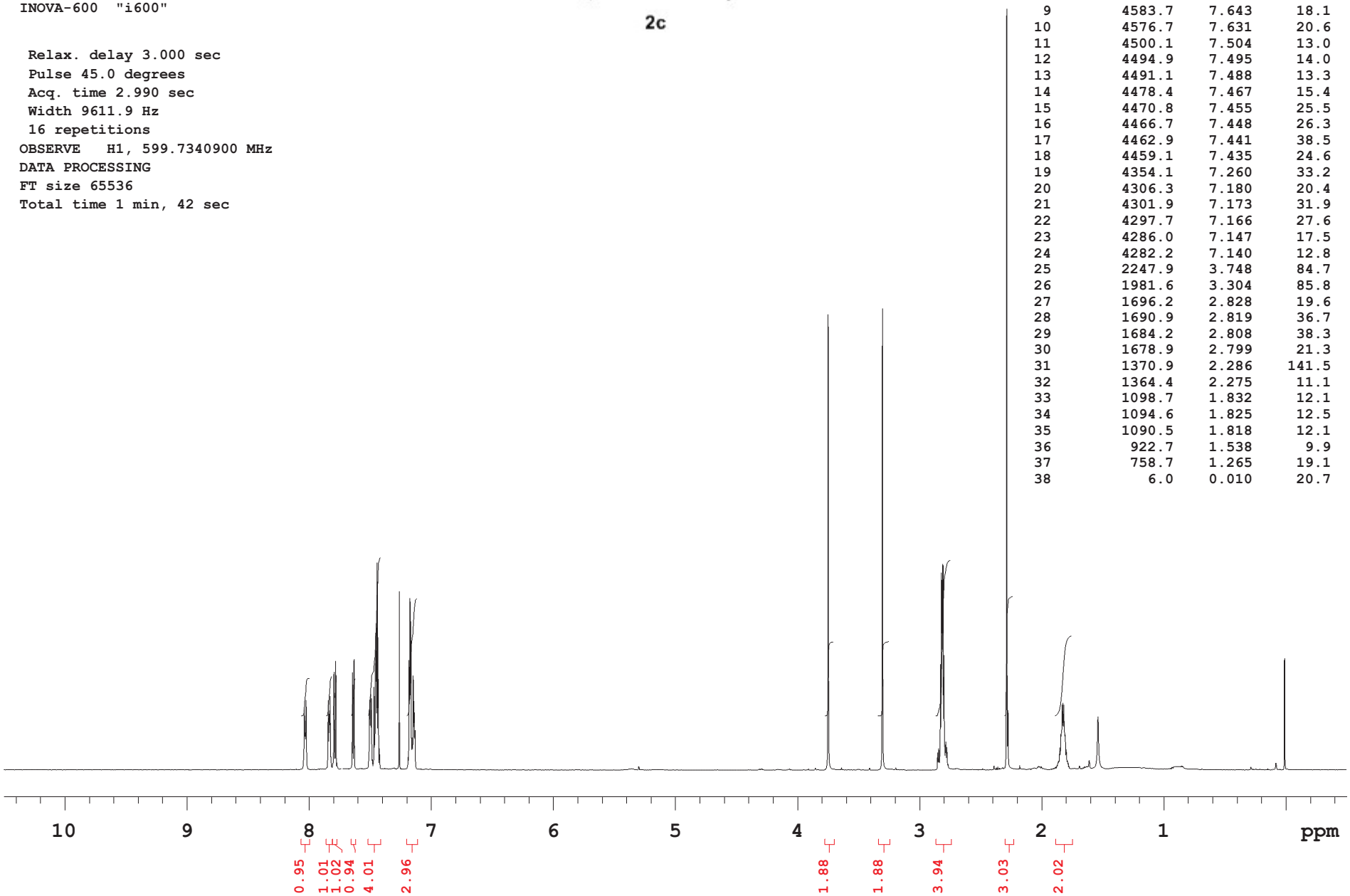
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7340900 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 42 sec



2c

INDEX	FREQUENCY	PPM	HEIGHT
1	4821.3	8.039	11.2
2	4819.0	8.035	9.4
3	4812.0	8.023	13.0
4	4703.4	7.843	13.0
5	4697.0	7.832	11.0
6	4694.0	7.827	13.7
7	4675.0	7.795	18.2
8	4666.8	7.781	20.3
9	4583.7	7.643	18.1
10	4576.7	7.631	20.6
11	4500.1	7.504	13.0
12	4494.9	7.495	14.0
13	4491.1	7.488	13.3
14	4478.4	7.467	15.4
15	4470.8	7.455	25.5
16	4466.7	7.448	26.3
17	4462.9	7.441	38.5
18	4459.1	7.435	24.6
19	4354.1	7.260	33.2
20	4306.3	7.180	20.4
21	4301.9	7.173	31.9
22	4297.7	7.166	27.6
23	4286.0	7.147	17.5
24	4282.2	7.140	12.8
25	2247.9	3.748	84.7
26	1981.6	3.304	85.8
27	1696.2	2.828	19.6
28	1690.9	2.819	36.7
29	1684.2	2.808	38.3
30	1678.9	2.799	21.3
31	1370.9	2.286	141.5
32	1364.4	2.275	11.1
33	1098.7	1.832	12.1
34	1094.6	1.825	12.5
35	1090.5	1.818	12.1
36	922.7	1.538	9.9
37	758.7	1.265	19.1
38	6.0	0.010	20.7

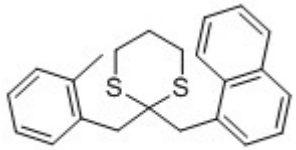


napht-o-tolyl-dithiane-13C-CDCl3 Inova600-ATB C13-s2pul-cdcl3

Automation directory:

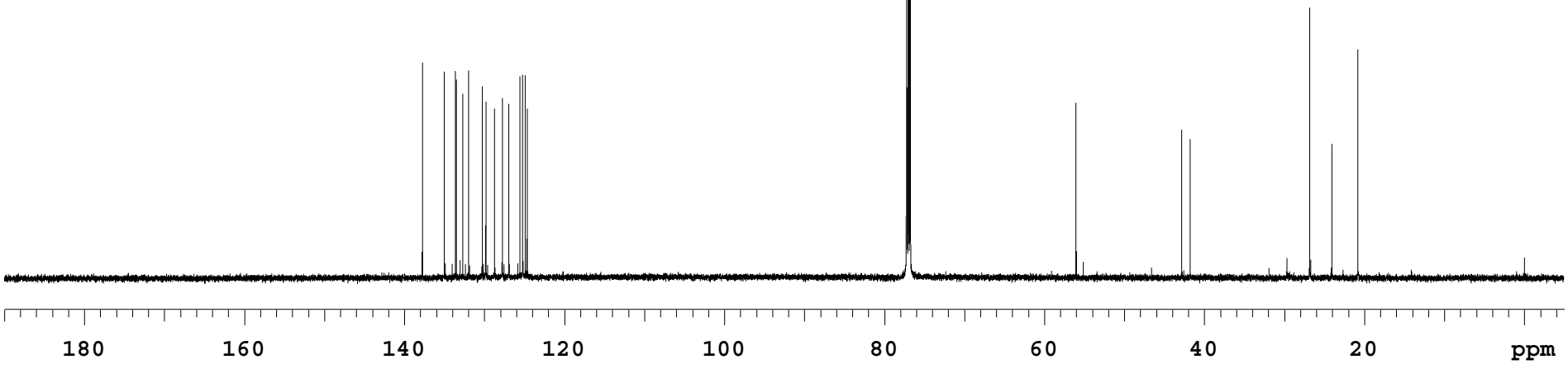
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
6000 repetitions
OBSERVE C13, 150.8032587 MHz
DECOUPLE H1, 599.7371004 MHz
Power 46 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 10 hr, 56 sec



2c

INDEX	FREQUENCY	PPM	HEIGHT
1	20772.9	137.748	34.5
2	20360.5	135.014	33.1
3	20151.3	133.626	33.2
4	20133.6	133.509	31.8
5	20011.0	132.696	29.5
6	19904.5	131.990	33.3
7	19646.1	130.276	30.7
8	19577.1	129.819	28.3
9	19409.8	128.709	27.2
10	19263.5	127.739	28.9
11	19143.1	126.941	28.0
12	18937.2	125.575	32.3
13	18885.3	125.231	32.6
14	18883.1	125.217	32.5
15	18832.3	124.880	32.5
16	18799.1	124.660	27.2
17	11643.9	77.212	613.8
18	11611.9	77.000	613.4
19	11580.4	76.791	601.2
20	8449.9	56.033	28.1
21	6458.5	42.827	23.8
22	6300.6	41.780	22.2
23	4049.1	26.850	43.4
24	3627.3	24.053	21.5
25	3137.0	20.802	36.7

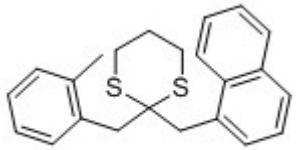


napht-o-tolyl-DEPT-CDCl3 Inova600-ATB C13-DEPT-cdcl3

Automation directory:

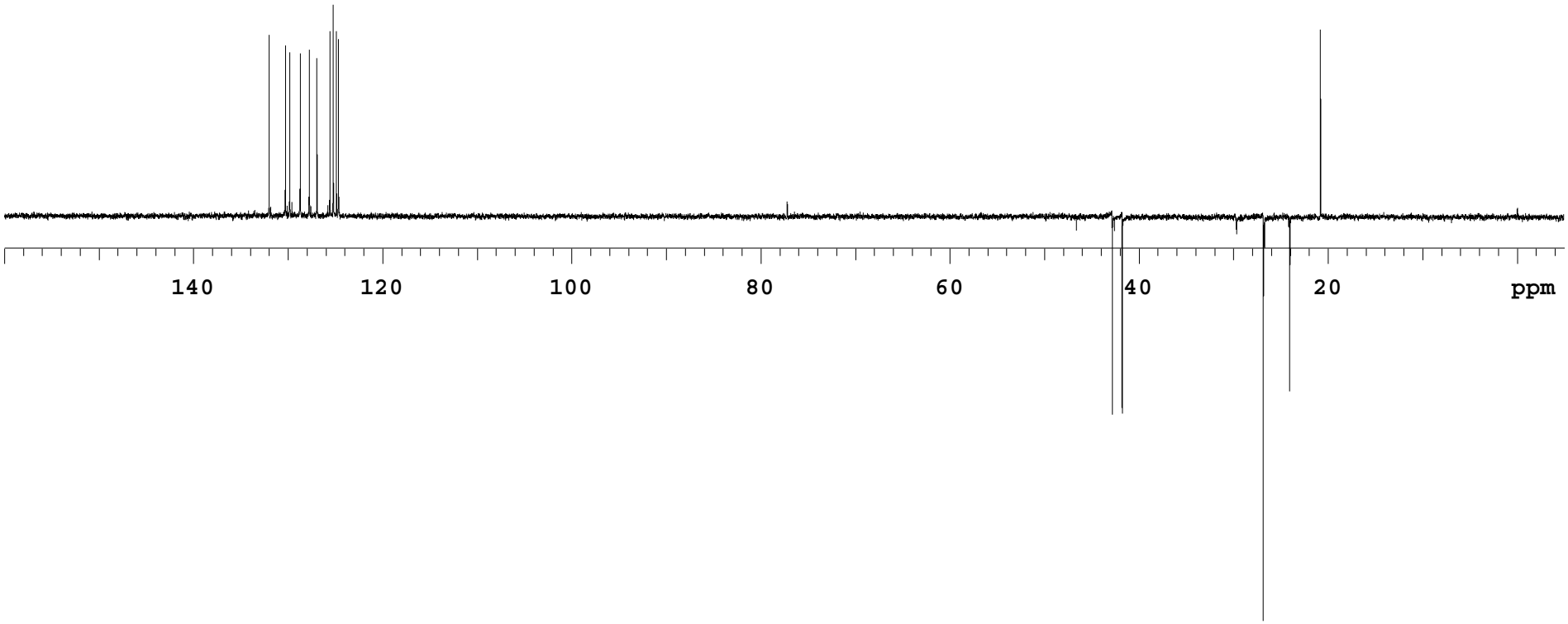
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
1024 repetitions
OBSERVE C13, 150.8032598 MHz
DECOUPLE H1, 599.7371004 MHz
Power 46 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 8 min, 48 sec



2c

INDEX	FREQUENCY	PPM	HEIGHT
1	19903.4	131.982	29.1
2	19645.0	130.269	27.4
3	19576.0	129.811	26.4
4	19408.7	128.702	26.2
5	19262.4	127.732	26.8
6	19142.0	126.934	25.4
7	18936.1	125.568	29.7
8	18884.2	125.224	34.0
9	18882.0	125.209	30.5
10	18831.2	124.873	29.8
11	18798.1	124.653	28.4
12	6457.4	42.820	-31.7
13	6299.0	41.769	-31.5
14	4048.0	26.843	-64.7
15	3625.7	24.042	-28.0
16	3135.4	20.791	30.0



Epox_2Me-napht_ATB_1H_CDCl3 Inova600-ATB H1-s2pul-cdcl3

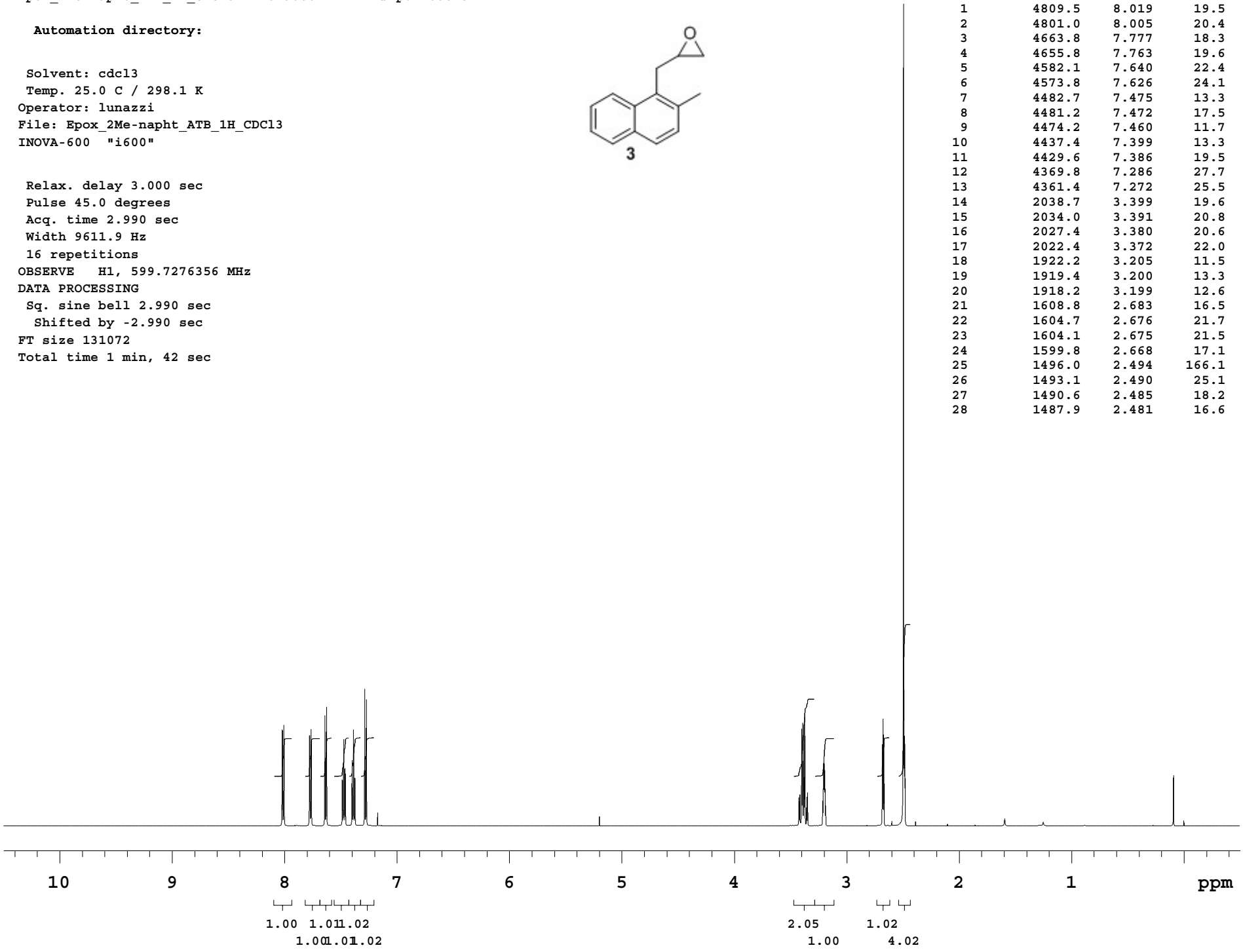
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Epox_2Me-napht_ATB_1H_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7276356 MHz
DATA PROCESSING
Sq. sine bell 2.990 sec
Shifted by -2.990 sec
FT size 131072
Total time 1 min, 42 sec

3

INDEX	FREQUENCY	PPM	HEIGHT
1	4809.5	8.019	19.5
2	4801.0	8.005	20.4
3	4663.8	7.777	18.3
4	4655.8	7.763	19.6
5	4582.1	7.640	22.4
6	4573.8	7.626	24.1
7	4482.7	7.475	13.3
8	4481.2	7.472	17.5
9	4474.2	7.460	11.7
10	4437.4	7.399	13.3
11	4429.6	7.386	19.5
12	4369.8	7.286	27.7
13	4361.4	7.272	25.5
14	2038.7	3.399	19.6
15	2034.0	3.391	20.8
16	2027.4	3.380	20.6
17	2022.4	3.372	22.0
18	1922.2	3.205	11.5
19	1919.4	3.200	13.3
20	1918.2	3.199	12.6
21	1608.8	2.683	16.5
22	1604.7	2.676	21.7
23	1604.1	2.675	21.5
24	1599.8	2.668	17.1
25	1496.0	2.494	166.1
26	1493.1	2.490	25.1
27	1490.6	2.485	18.2
28	1487.9	2.481	16.6

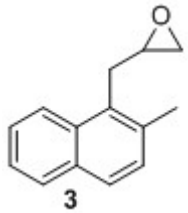


Epox_2Me-napht_ATB_13C_CDCl3 Inova600-ATB C13-s2pul-cdcl3

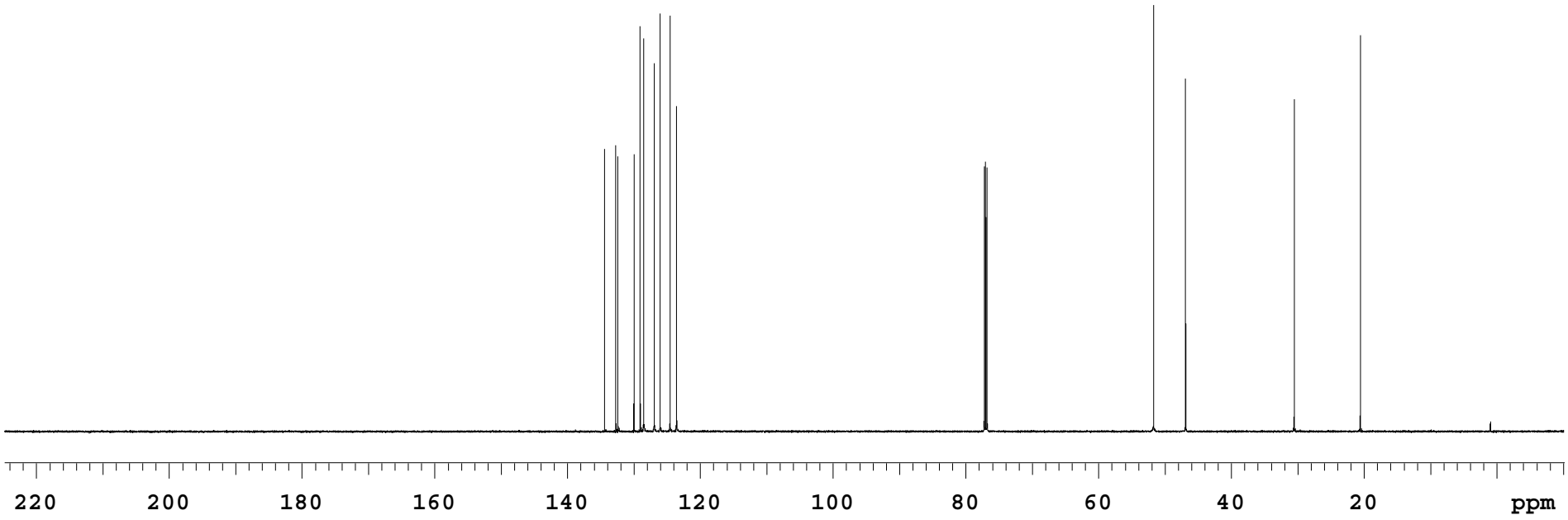
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Epox_2Me-napht_ATB_13C_CDCl3
INOVA-600 "i600"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
512 repetitions
OBSERVE C13, 150.8016444 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 42 min, 45 sec



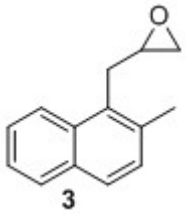
INDEX	FREQUENCY	PPM	HEIGHT
1	20270.4	134.418	45.3
2	20013.1	132.711	45.8
3	19965.1	132.393	44.0
4	19601.8	129.984	44.4
5	19460.4	129.047	64.9
6	19379.3	128.508	62.9
7	19141.9	126.934	59.0
8	19009.4	126.055	66.9
9	18784.1	124.562	66.6
10	18632.8	123.559	52.1
11	11643.7	77.212	42.4
12	11611.7	77.000	43.2
13	11579.7	76.788	42.3
14	7798.3	51.713	68.3
15	7069.0	46.876	56.5
16	4603.3	30.526	53.2
17	3101.0	20.564	63.5



Epox_2Me-napht_ATB_DEPT_CDCl3 Inova600-ATB C13-DEPT-cdcl3

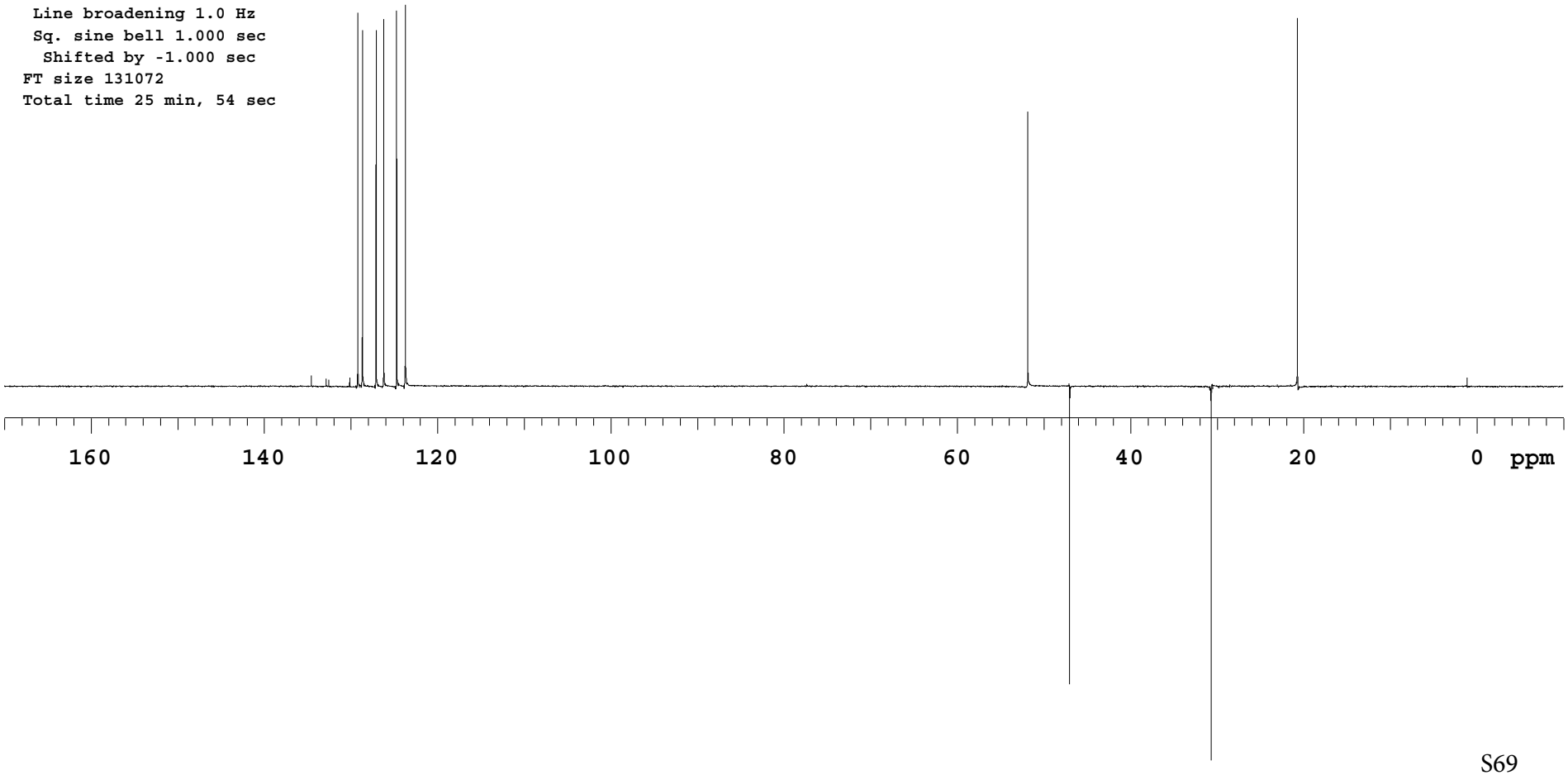
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: Epox_2Me-napht_ATB_DEPT_CDCl3
INOVA-600 "i600"



INDEX	FREQUENCY	PPM	HEIGHT
1	19483.1	129.197	59.9
2	19401.9	128.658	57.1
3	19164.5	127.084	57.0
4	19032.0	126.205	58.9
5	18806.7	124.712	60.3
6	18655.5	123.709	61.2
7	7821.0	51.863	44.0
8	7091.6	47.026	-47.7
9	4625.9	30.676	-79.5
10	3123.6	20.714	59.1

Relax. delay 2.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
512 repetitions
OBSERVE C13, 150.8016218 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 25 min, 54 sec

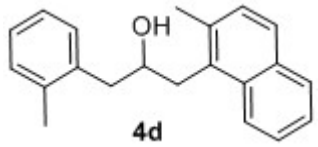


Hydroxy_2Menapht_o-tolil Inova600-Triple H1-s2pul-cdcl3

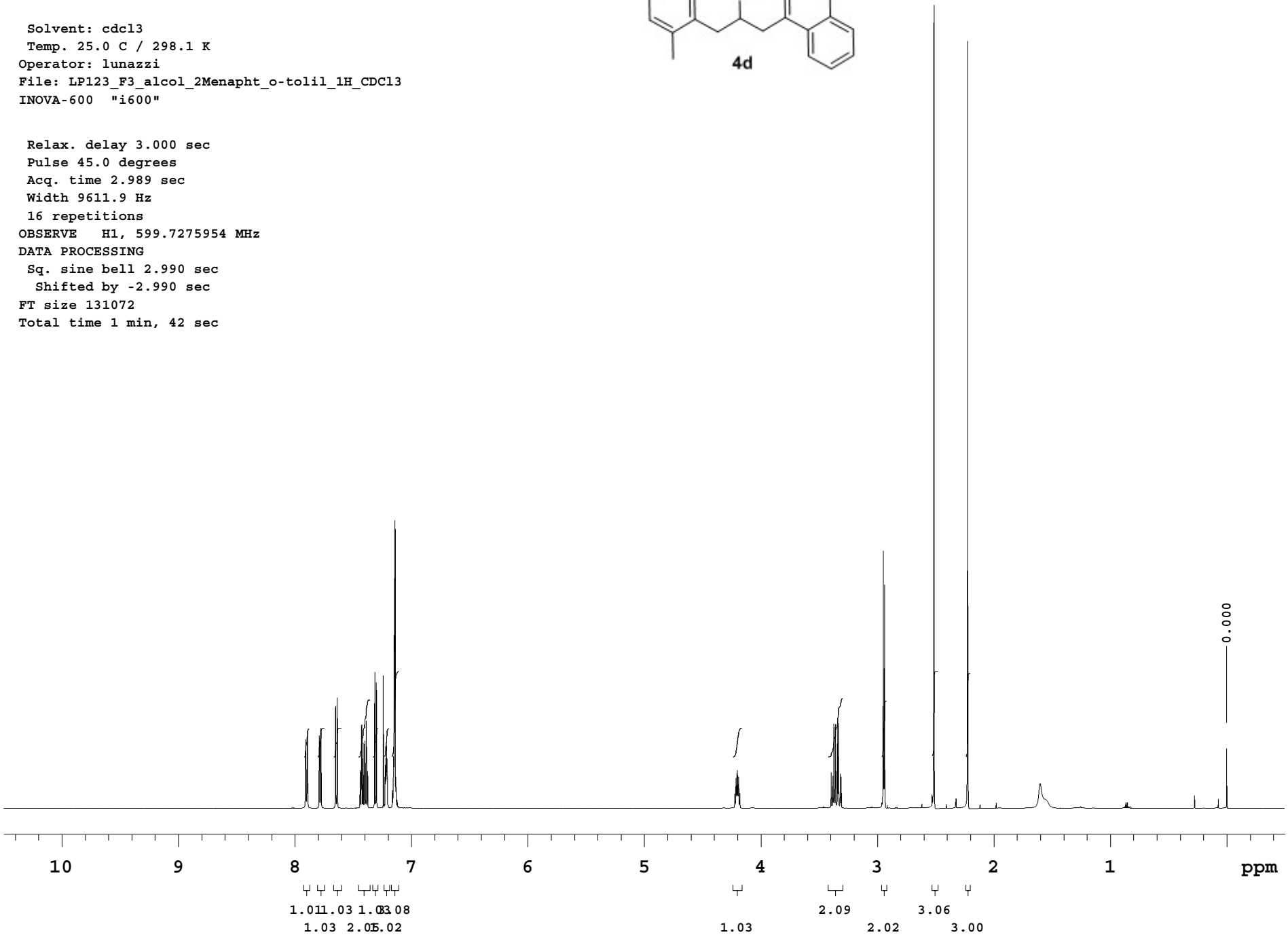
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: LP123_F3_alcol_2Menapht_o-tolil_1H_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.989 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7275954 MHz
DATA PROCESSING
Sq. sine bell 2.990 sec
Shifted by -2.990 sec
FT size 131072
Total time 1 min, 42 sec

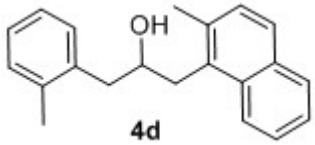


4d



Hydroxy_2Menapht_otolil_13C_CDCL3 Inova600-ATB C13-s2pul-cdcl3

Automation directory:

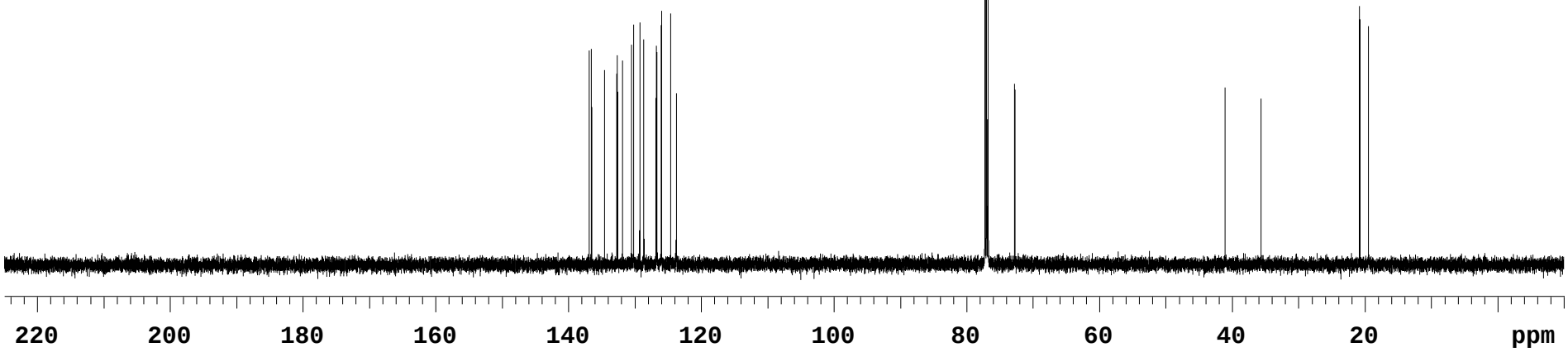


4d

Solvent: cdcl3
Ambient temperature
Operator: lunazzi
File: LP123_F3_alcol_2Menapht_otolil_13C_CDCL3
INOVA-600 "i600"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
288 repetitions
OBSERVE C13, 150.8016245 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 42 min, 45 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	20634.8	136.834	34.4
2	20584.0	136.497	34.6
3	20291.4	134.557	31.3
4	20007.0	132.671	30.7
5	19995.4	132.594	33.6
6	19884.5	131.858	32.8
7	19676.3	130.478	35.3
8	19628.3	130.160	38.6
9	19489.7	129.241	38.9
10	19395.3	128.615	36.2
11	19117.6	126.773	35.1
12	19097.7	126.641	34.1
13	19000.5	125.997	38.4
14	18989.5	125.924	40.7
15	18785.8	124.573	40.3
16	18660.4	123.742	27.5
17	11643.7	77.212	272.8
18	11611.7	77.000	276.6
19	11579.7	76.788	271.3
20	10970.2	72.746	29.1
21	6195.6	41.084	28.4
22	5377.4	35.659	26.7
23	3135.3	20.791	41.5
24	2936.0	19.469	38.3

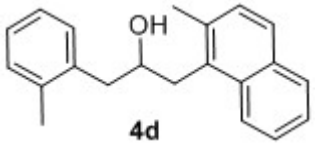


Hydroxy_2Me-napht-o-tolyl-DEPT-CDCl3 Inova600-ATB C13-DEPT-cdcl3

Automation directory:

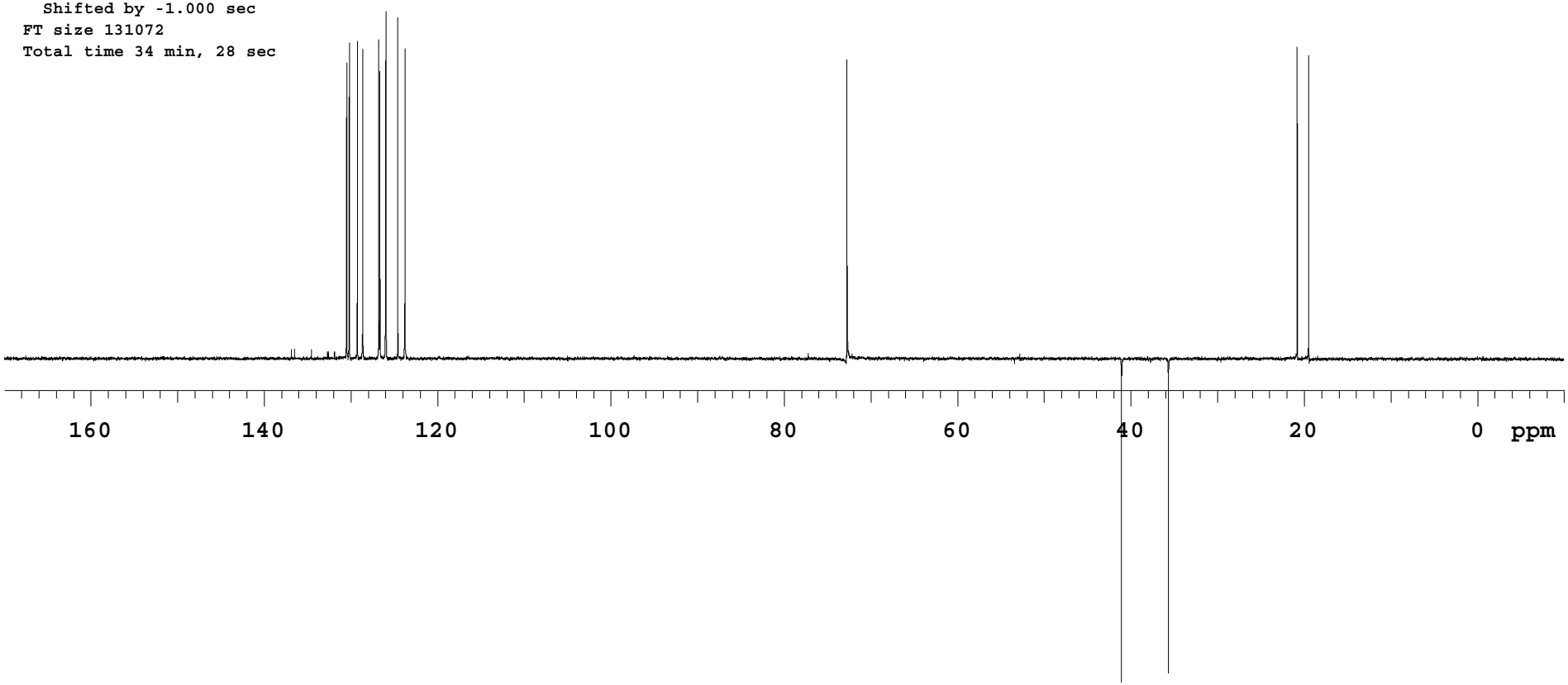
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
512 repetitions
OBSERVE C13, 150.8016262 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 34 min, 28 sec



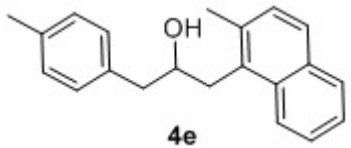
4d

INDEX	FREQUENCY	PPM	HEIGHT
1	19675.8	130.474	47.6
2	19629.4	130.167	50.7
3	19489.7	129.241	51.0
4	19395.3	128.615	49.7
5	19117.0	126.769	51.2
6	19097.2	126.638	46.2
7	19000.0	125.993	47.8
8	18989.5	125.924	55.8
9	18785.8	124.573	54.8
10	18661.6	123.749	49.8
11	10969.6	72.742	48.0
12	6195.0	41.081	-51.8
13	5376.8	35.655	-50.4
14	3135.3	20.791	50.1
15	2936.0	19.469	48.7



alcol_2Menapht_ptolil_1H_CDCl3 Inova600-ATB H1-s2pul-cdcl3 Mar 11 2016

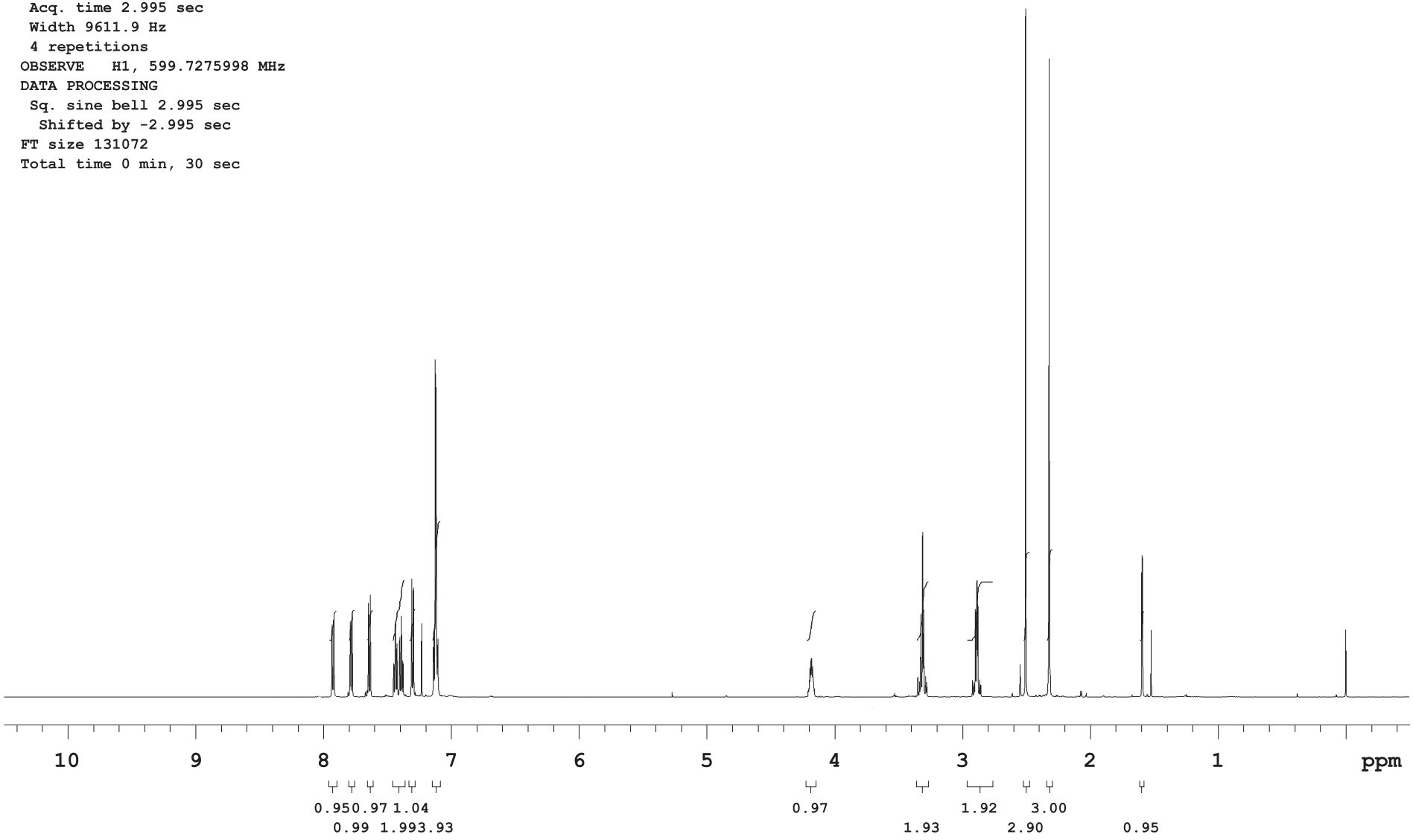
Automation directory:



4e

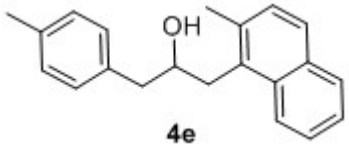
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: alcol_2Menapht_ptolil_1H_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
4 repetitions
OBSERVE   H1, 599.7275998 MHz
DATA PROCESSING
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 0 min, 30 sec



Hydroxy_2Menapht_ptolyl_13C_CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

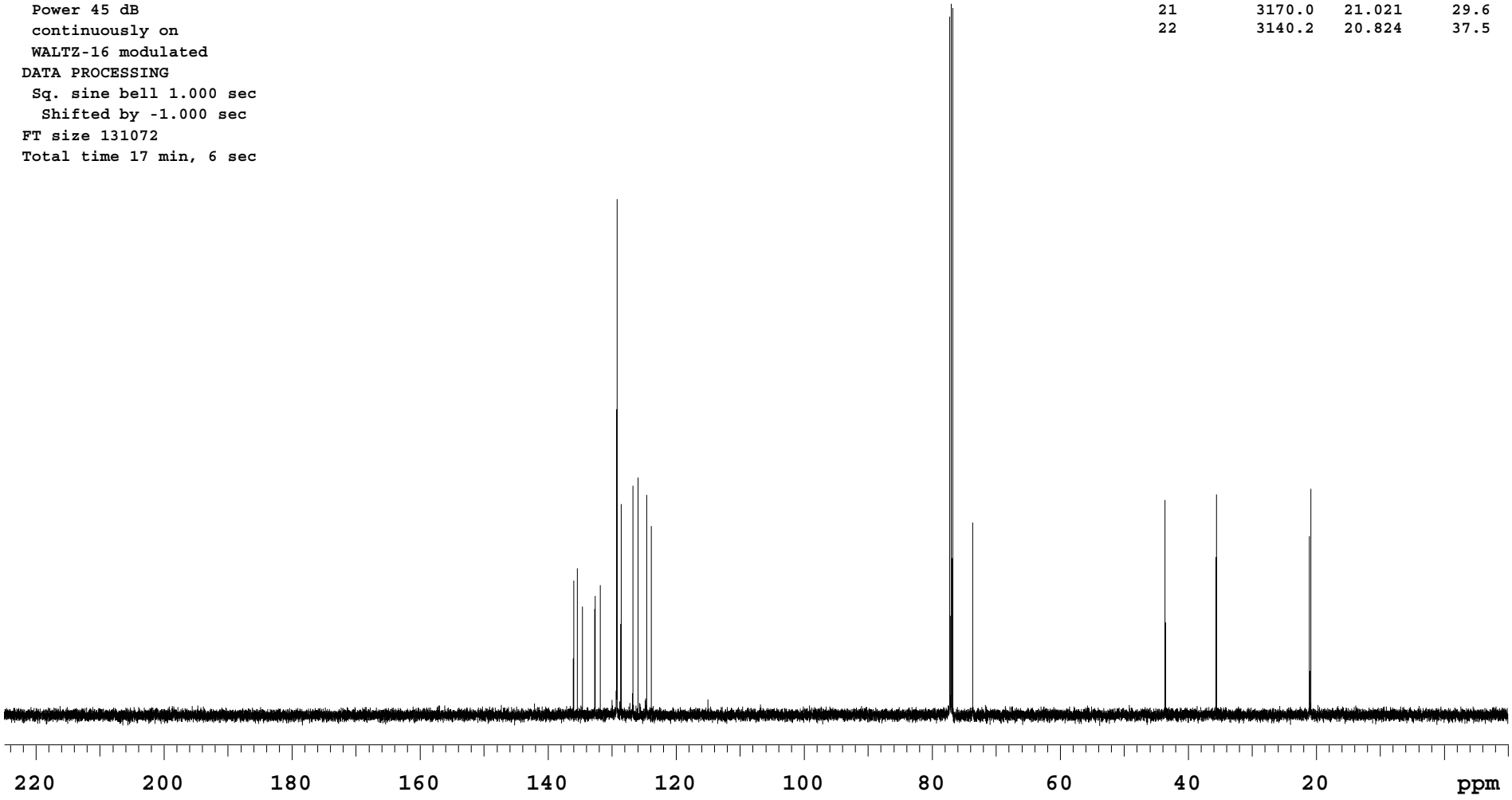


4e

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: alcol_2Menapht_ptolil_13C_CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
64 repetitions
OBSERVE C13, 150.8016262 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 17 min, 6 sec

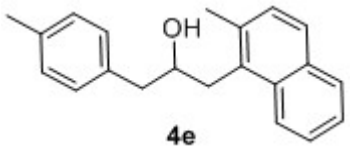
INDEX	FREQUENCY	PPM	HEIGHT
1	20510.0	136.006	22.3
2	20417.2	135.391	24.3
3	20302.4	134.630	18.0
4	20013.7	132.715	17.5
5	19994.9	132.591	19.7
6	19877.8	131.814	21.5
7	19492.5	129.259	41.1
8	19488.6	129.233	85.6
9	19487.5	129.226	75.4
10	19392.0	128.593	35.0
11	19114.8	126.755	38.0
12	18989.5	125.924	39.4
13	18784.7	124.565	36.5
14	18675.9	123.844	31.4
15	11643.2	77.209	116.0
16	11611.7	77.000	118.1
17	11579.7	76.788	117.4
18	11104.9	73.639	31.9
19	6570.5	43.570	35.6
20	5368.0	35.596	36.6
21	3170.0	21.021	29.6
22	3140.2	20.824	37.5



Hydroxy_2Menapht_ptolyl_dept_CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

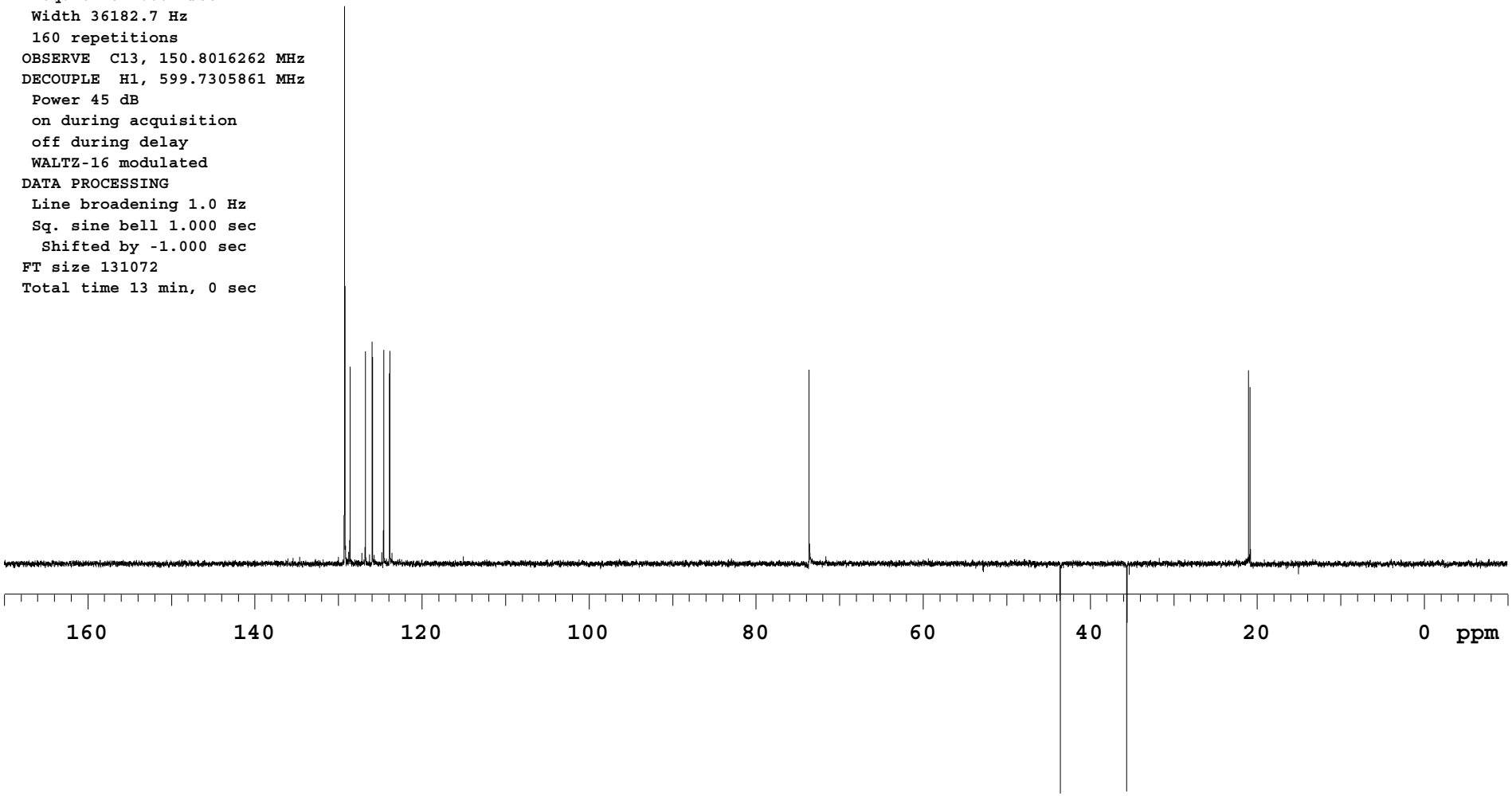
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: alcol_2Menapht_ptolil_dept_CDCl3
INOVA-600 "i600"



4e

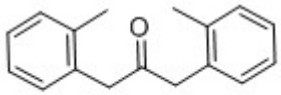
INDEX	FREQUENCY	PPM	HEIGHT
1	19492.5	129.259	41.8
2	19488.0	129.230	92.7
3	19392.0	128.593	32.8
4	19114.8	126.755	35.3
5	18989.5	125.924	36.9
6	18784.7	124.565	35.6
7	18675.9	123.844	35.4
8	11104.3	73.635	32.2
9	6570.5	43.570	-38.2
10	5368.0	35.596	-37.8
11	3170.0	21.021	32.2
12	3140.2	20.824	29.4

Relax. delay 2.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
160 repetitions
OBSERVE C13, 150.8016262 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 13 min, 0 sec



di-tolyl-ketone-ATB-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

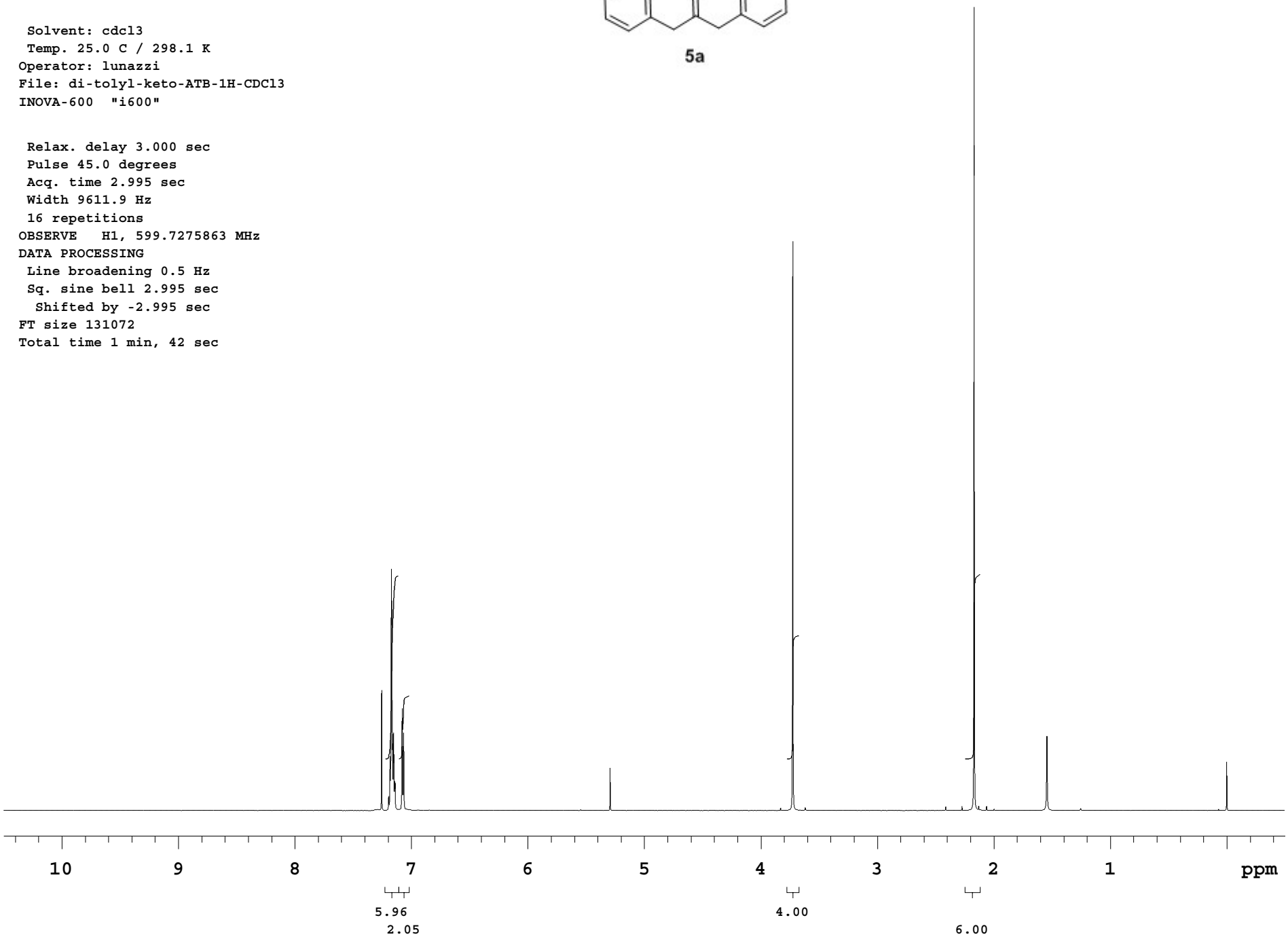
Automation directory:



5a

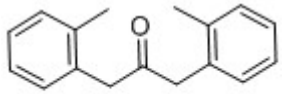
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-tolyl-keto-ATB-1H-CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7275863 MHz
DATA PROCESSING
Line broadening 0.5 Hz
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 1 min, 42 sec



di-tolyl-ketone-ATB-13C-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

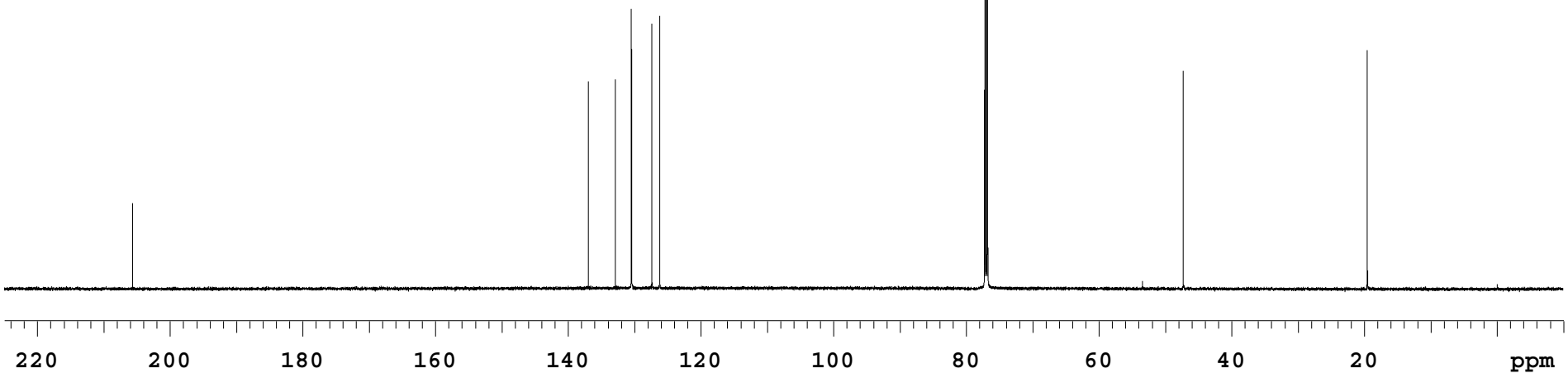


5a

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-tolyl-keto-ATB-13C-CDCl3
INOVA-600 "i600"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
6144 repetitions
OBSERVE C13, 150.8016218 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 10 hr, 15 min, 21 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	31006.1	205.608	13.8
2	20649.7	136.933	33.3
3	20041.3	132.898	33.6
4	19675.2	130.471	44.9
5	19664.2	130.398	38.5
6	19207.6	127.370	42.5
7	19029.8	126.191	43.7
8	11643.2	77.209	300.8
9	11611.7	77.000	305.9
10	11579.7	76.788	306.7
11	7130.8	47.286	35.0
12	2946.4	19.539	38.2

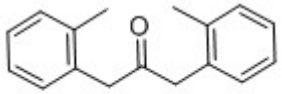


di-tolyl-ketone-ATB-dept-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

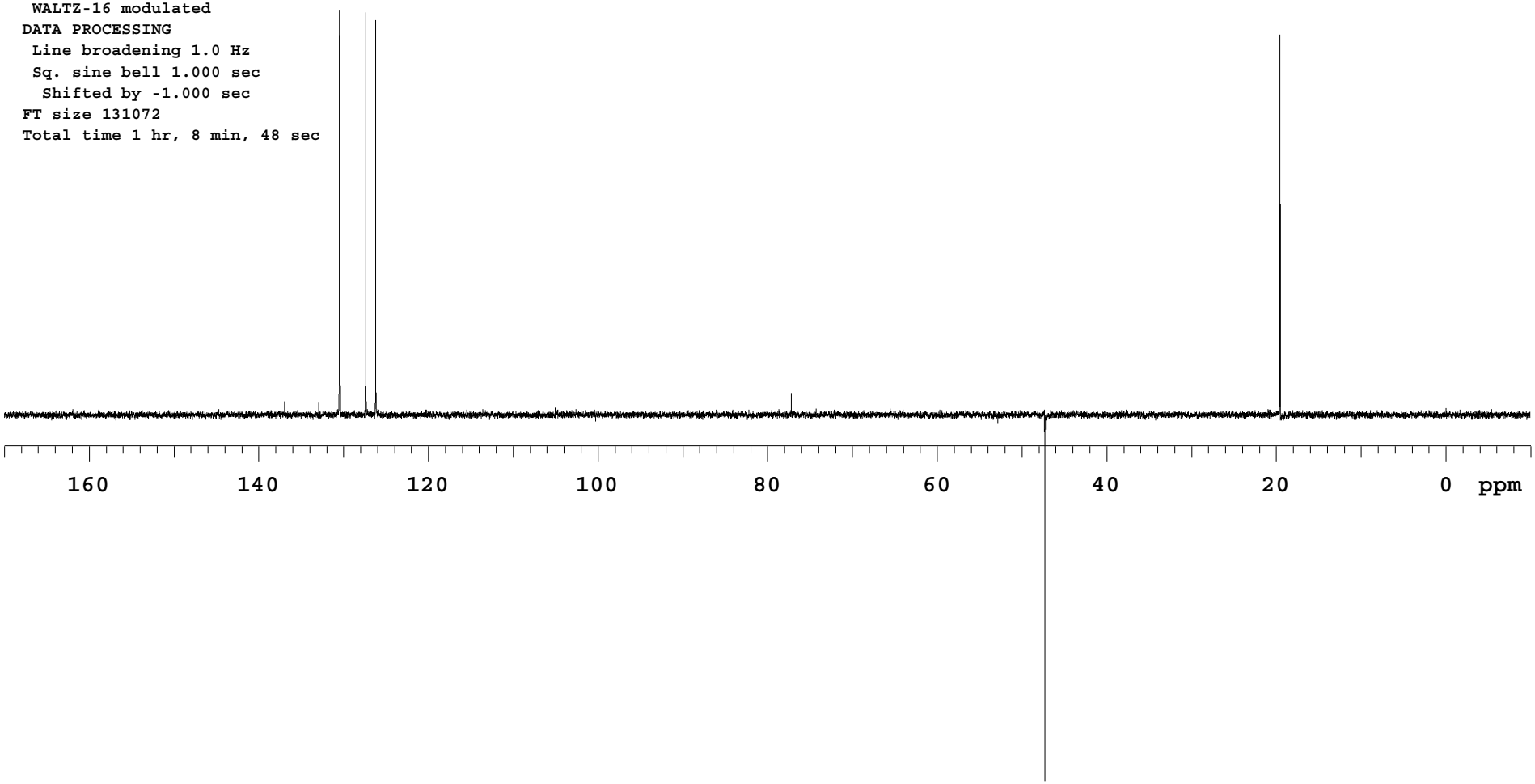
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: di-tolyl-keto-ATB-dept-CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
1024 repetitions
OBSERVE C13, 150.8016218 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 8 min, 48 sec



5a

INDEX	FREQUENCY	PPM	HEIGHT
1	19675.2	130.471	66.4
2	19664.1	130.397	62.2
3	19207.6	127.370	66.0
4	19029.8	126.191	64.7
5	7130.8	47.286	-80.5
6	2946.4	19.538	62.4

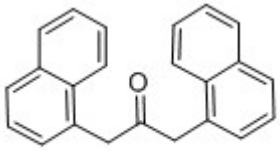


ketone-dinapht-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

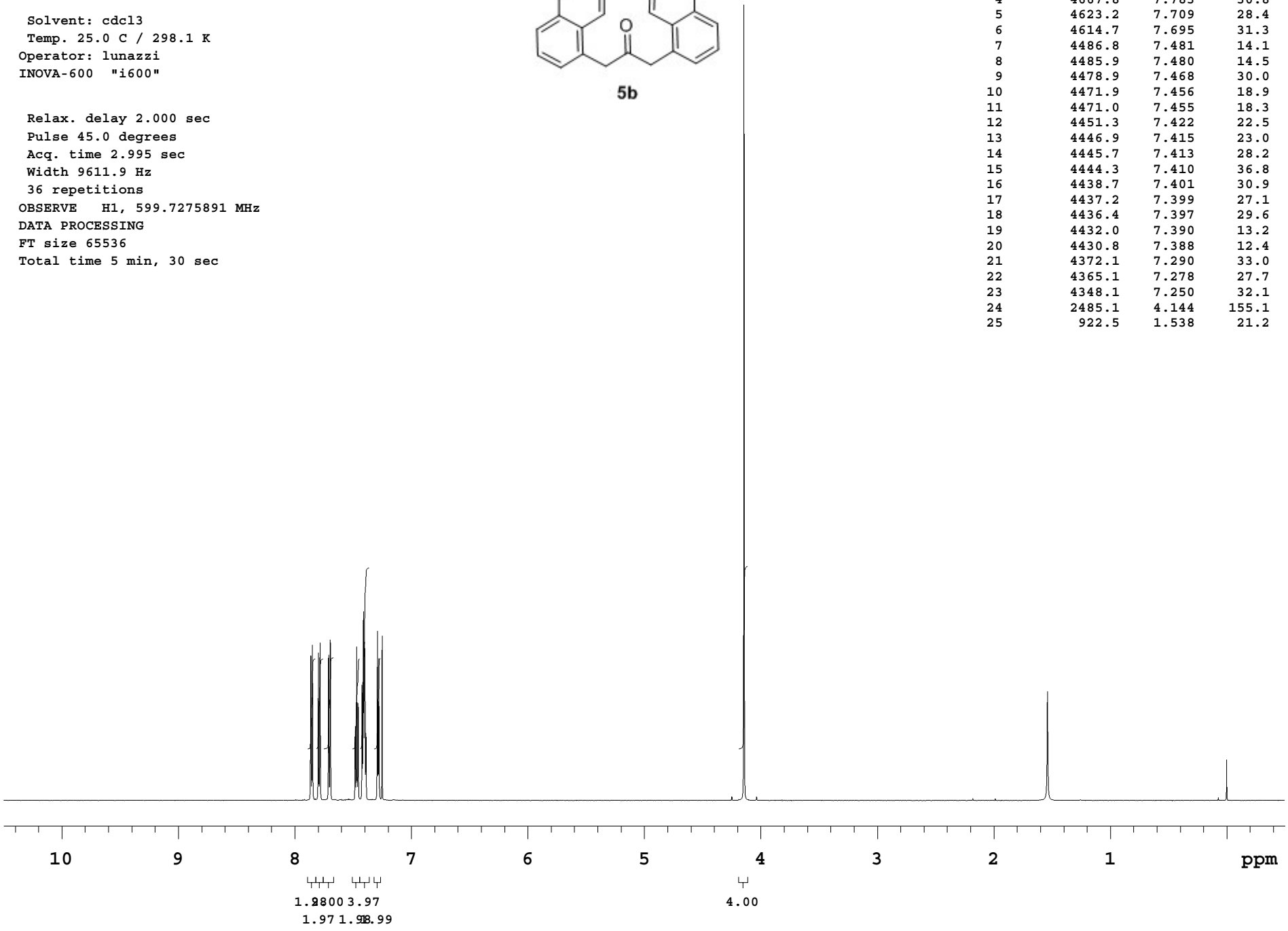
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
36 repetitions
OBSERVE   H1, 599.7275891 MHz
DATA PROCESSING
FT size 65536
Total time 5 min, 30 sec



5b

INDEX	FREQUENCY	PPM	HEIGHT
1	4715.3	7.862	28.2
2	4707.1	7.849	30.3
3	4676.0	7.797	28.8
4	4667.8	7.783	30.8
5	4623.2	7.709	28.4
6	4614.7	7.695	31.3
7	4486.8	7.481	14.1
8	4485.9	7.480	14.5
9	4478.9	7.468	30.0
10	4471.9	7.456	18.9
11	4471.0	7.455	18.3
12	4451.3	7.422	22.5
13	4446.9	7.415	23.0
14	4445.7	7.413	28.2
15	4444.3	7.410	36.8
16	4438.7	7.401	30.9
17	4437.2	7.399	27.1
18	4436.4	7.397	29.6
19	4432.0	7.390	13.2
20	4430.8	7.388	12.4
21	4372.1	7.290	33.0
22	4365.1	7.278	27.7
23	4348.1	7.250	32.1
24	2485.1	4.144	155.1
25	922.5	1.538	21.2

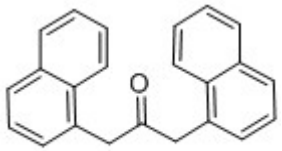


ketone-dinapht-13C-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

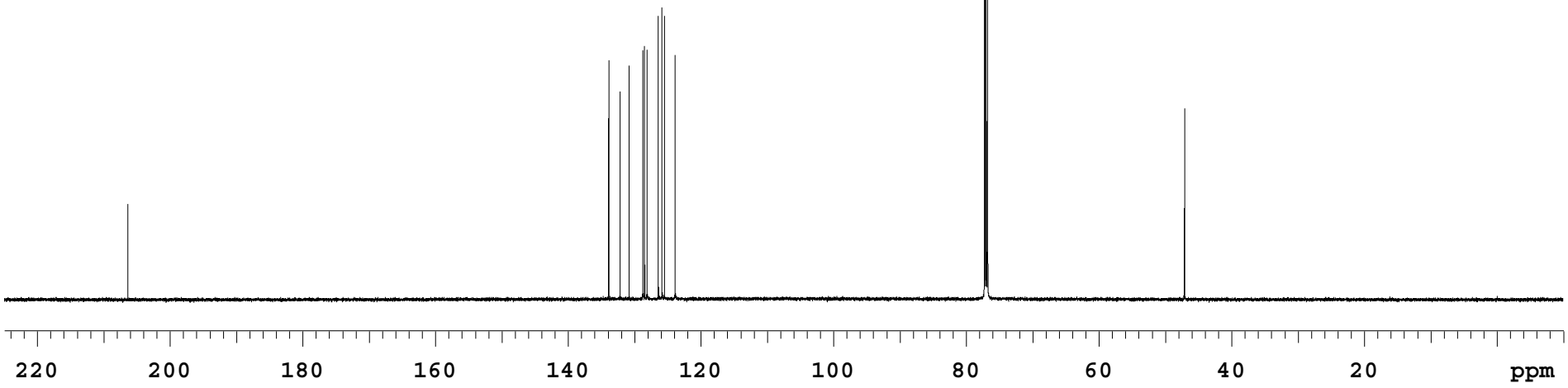
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
6144 repetitions
OBSERVE C13, 150.8016223 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 10 hr, 15 min, 21 sec



5b

INDEX	FREQUENCY	PPM	HEIGHT
1	31118.7	206.355	15.3
2	20189.2	133.879	38.4
3	19934.1	132.188	33.4
4	19723.2	130.789	37.6
5	19412.4	128.728	40.0
6	19373.2	128.468	40.7
7	19316.3	128.091	40.1
8	19061.8	126.403	45.5
9	18978.5	125.850	46.8
10	18921.0	125.470	45.5
11	18678.1	123.859	39.2
12	11643.7	77.212	300.0
13	11611.7	77.000	302.0
14	11579.7	76.788	292.8
15	7102.7	47.100	30.7

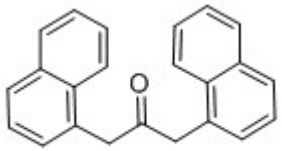


ketone-dinapht-dept-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

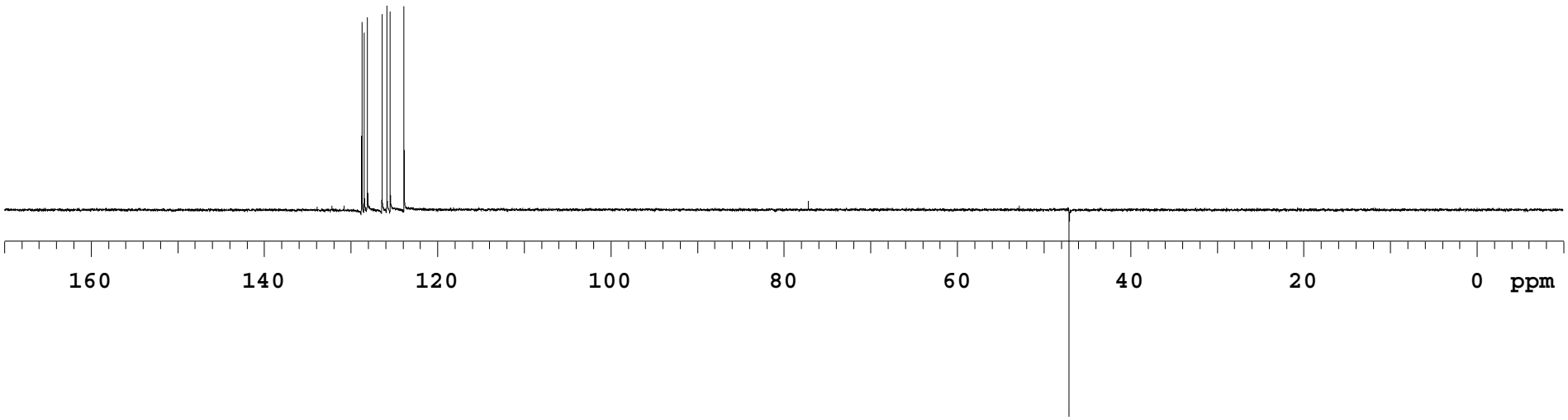
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
1024 repetitions
OBSERVE C13, 150.8016223 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 1 hr, 8 min, 48 sec



5b

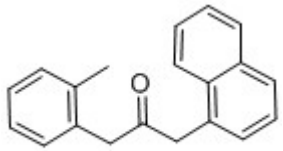
INDEX	FREQUENCY	PPM	HEIGHT
1	19412.4	128.728	30.1
2	19373.2	128.468	28.4
3	19316.3	128.091	30.8
4	19061.8	126.403	31.4
5	18978.4	125.850	32.7
6	18921.0	125.470	31.8
7	18678.1	123.859	32.6
8	7102.7	47.099	-33.1



ketone-naph-otolyl-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

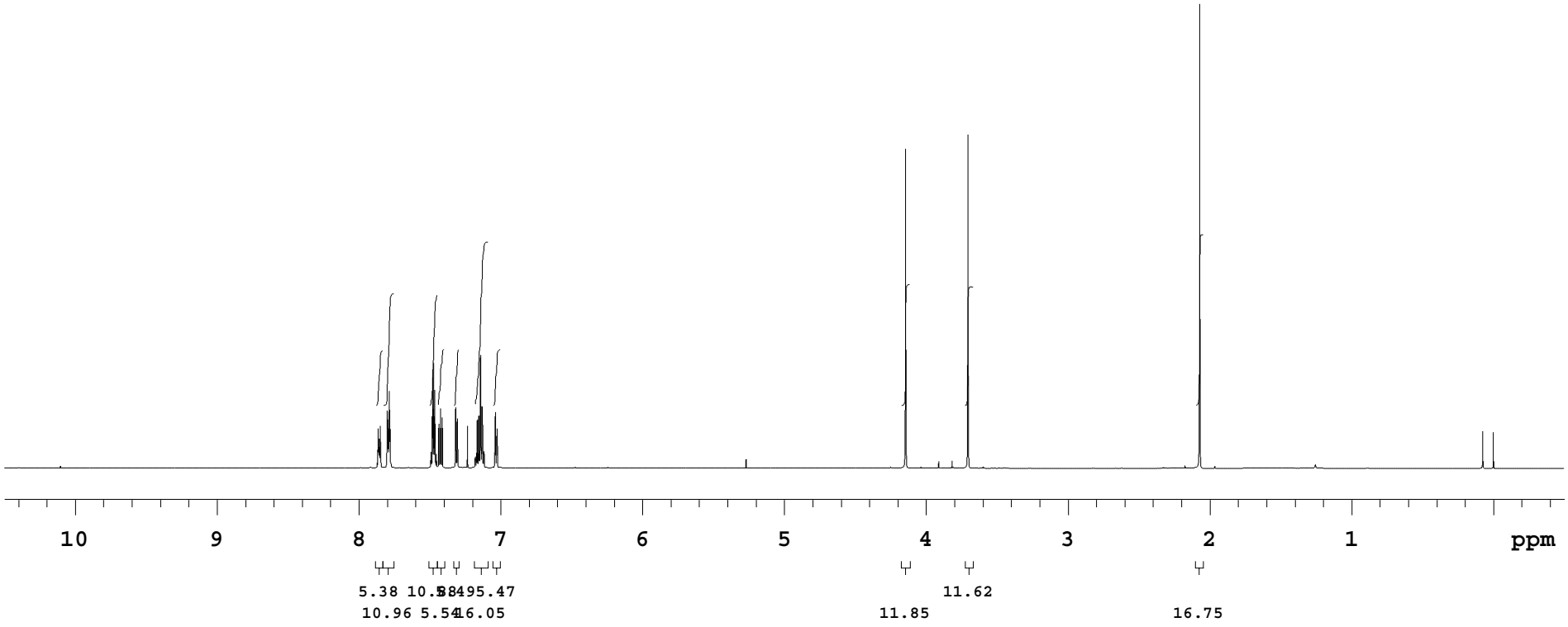
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: keto-naph-otolyl-1H-CDCl3
INOVA-600 "i600"



5c

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.990 sec
Width 9611.9 Hz
16 repetitions
OBSERVE   H1, 599.7275983 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 12 sec

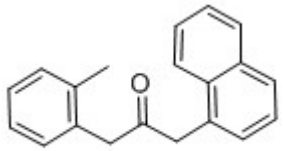


ketone-napht-o-tolyl-13C-ATB-CDCL3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

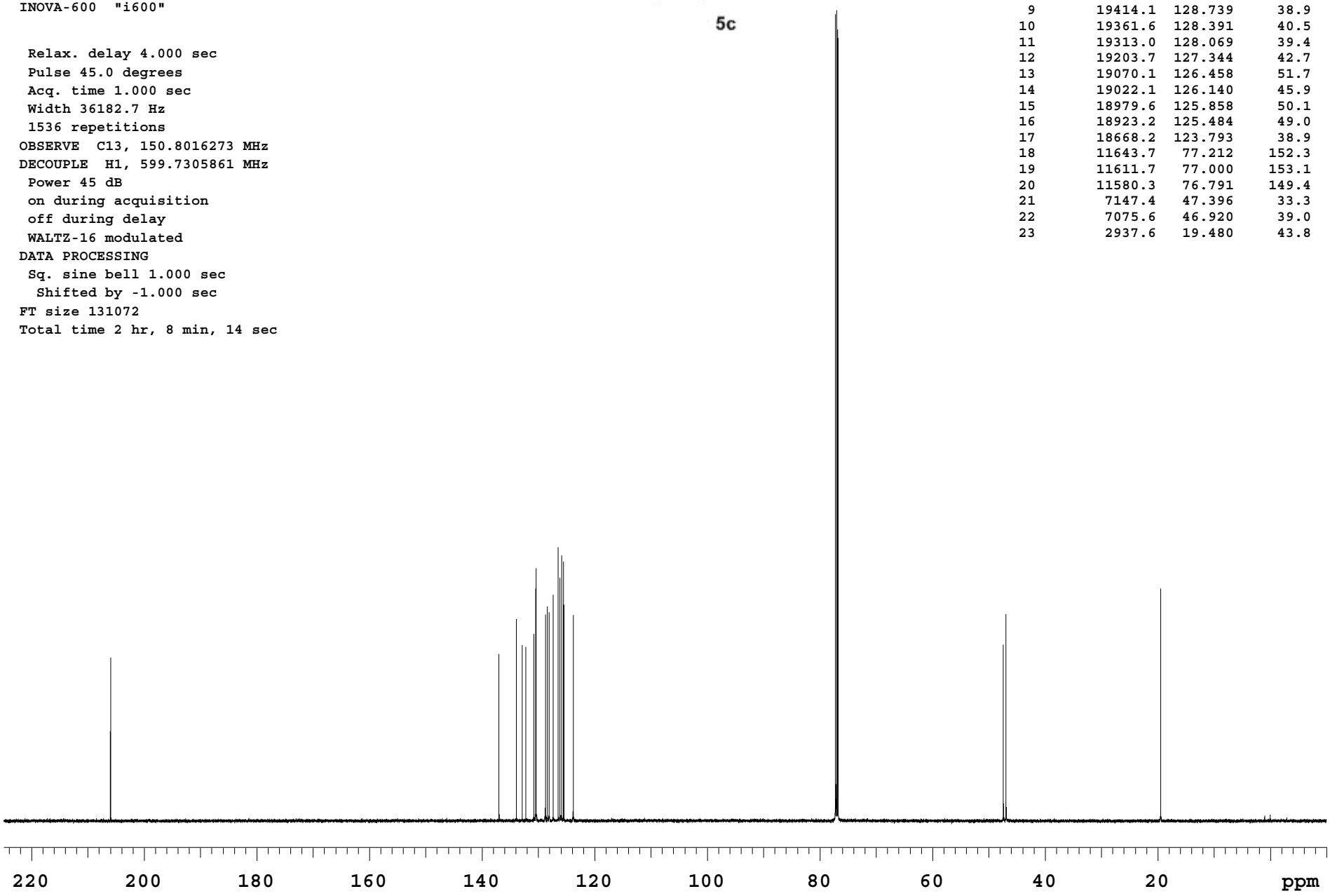
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
1536 repetitions
OBSERVE C13, 150.8016273 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 2 hr, 8 min, 14 sec



5c

INDEX	FREQUENCY	PPM	HEIGHT
1	31059.6	205.963	30.8
2	20654.6	136.966	31.5
3	20188.1	133.872	38.1
4	20032.4	132.840	33.2
5	19934.7	132.192	32.9
6	19725.5	130.804	35.4
7	19668.6	130.427	43.9
8	19665.8	130.409	47.7
9	19414.1	128.739	38.9
10	19361.6	128.391	40.5
11	19313.0	128.069	39.4
12	19203.7	127.344	42.7
13	19070.1	126.458	51.7
14	19022.1	126.140	45.9
15	18979.6	125.858	50.1
16	18923.2	125.484	49.0
17	18668.2	123.793	38.9
18	11643.7	77.212	152.3
19	11611.7	77.000	153.1
20	11580.3	76.791	149.4
21	7147.4	47.396	33.3
22	7075.6	46.920	39.0
23	2937.6	19.480	43.8

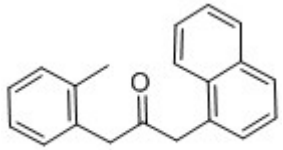


ketone-napht-o-tolyl-dept-ATB-CDCL3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

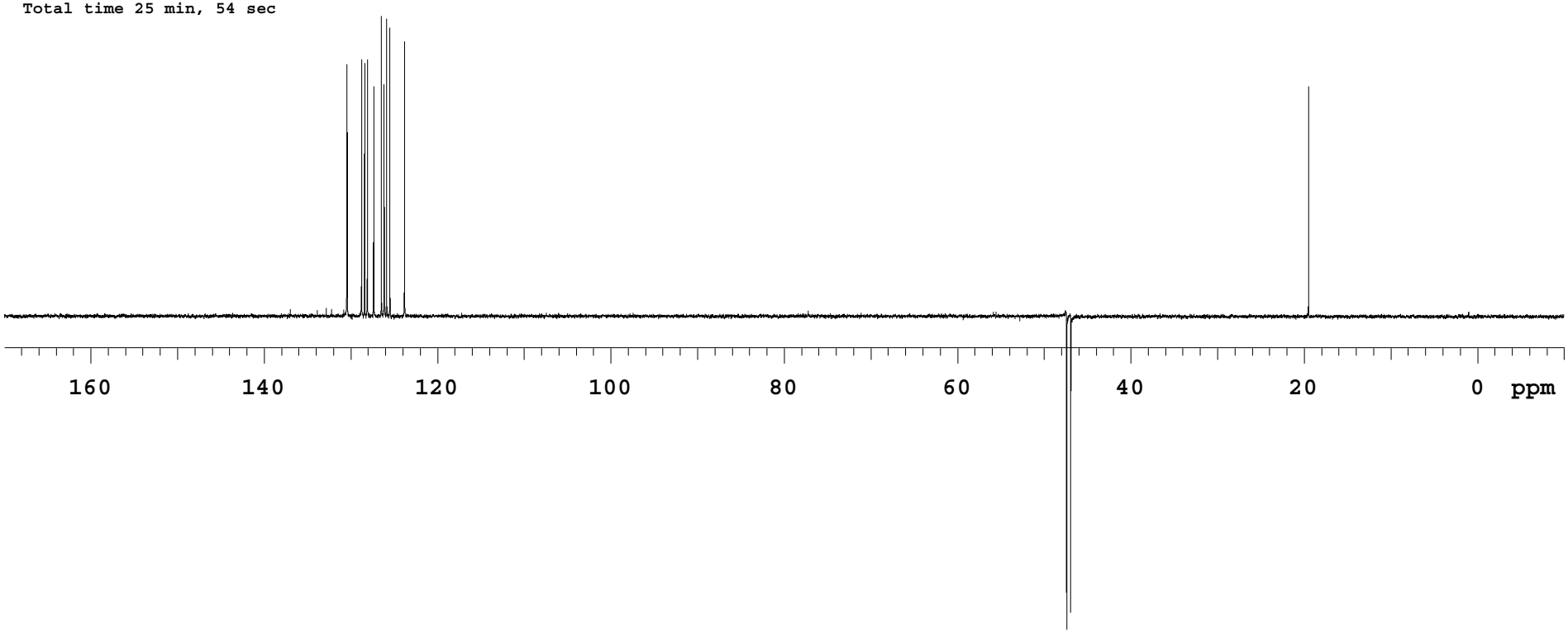
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
INOVA-600 "i600"

Relax. delay 2.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
512 repetitions
OBSERVE C13, 150.8016273 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 25 min, 54 sec



5c

INDEX	FREQUENCY	PPM	HEIGHT
1	19668.6	130.427	39.9
2	19665.8	130.409	40.4
3	19414.6	128.743	41.2
4	19361.6	128.391	40.6
5	19313.6	128.073	41.2
6	19203.7	127.344	36.8
7	19070.1	126.458	48.1
8	19022.1	126.140	37.2
9	18979.6	125.858	47.7
10	18923.3	125.484	46.3
11	18668.2	123.793	44.1
12	7146.9	47.392	-50.2
13	7075.1	46.917	-47.4
14	2937.6	19.480	36.8

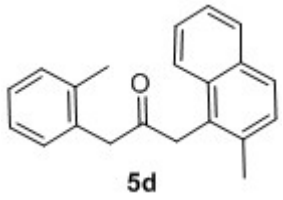


2Me-napht-otolyl-ketone-1H-CDCl3 Mercury400-ATB H1-s2pul-cdcl3

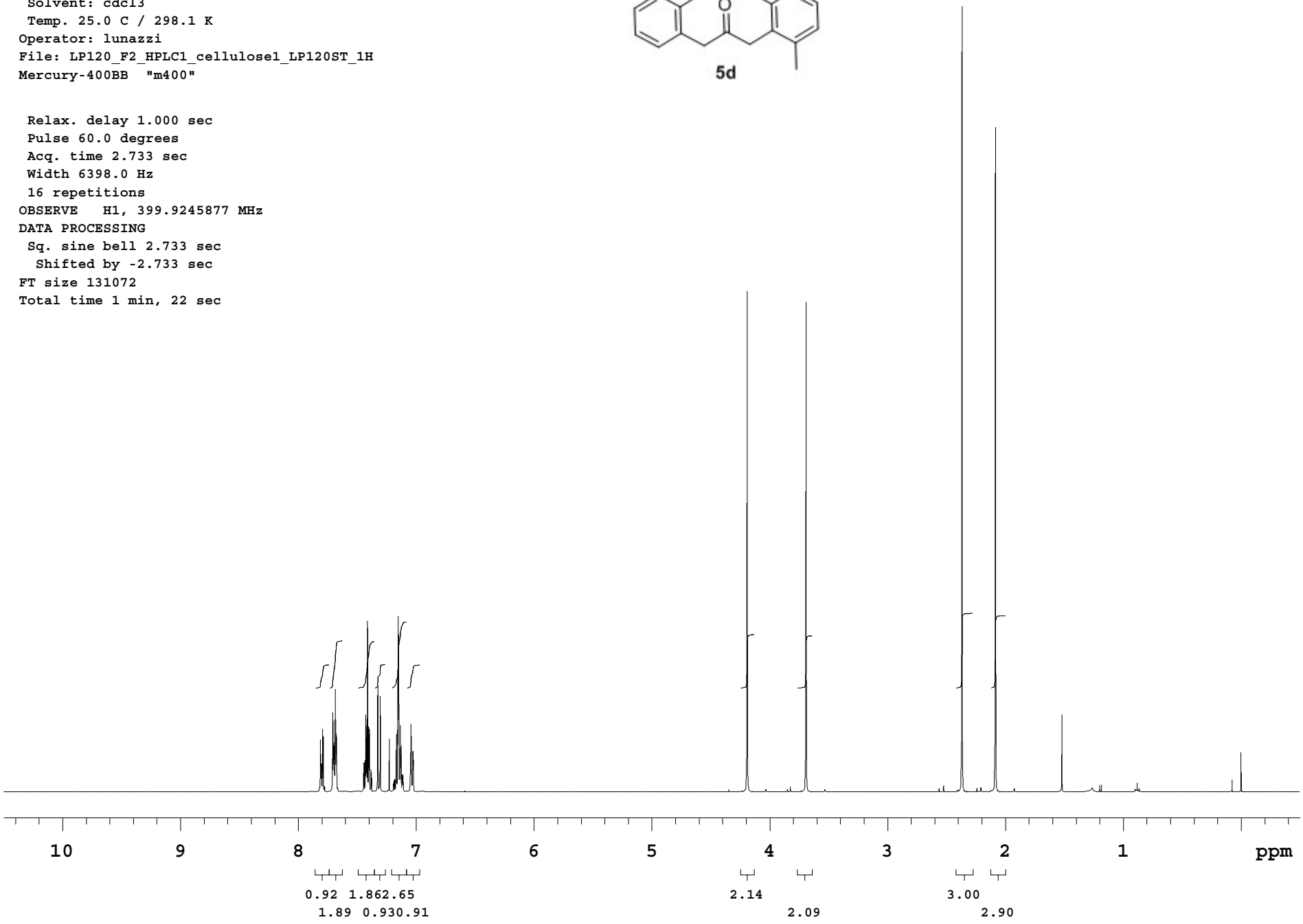
Automation directory:

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: LP120_F2_HPLC1_cellulose1_LP120ST_1H
Mercury-400BB "m400"

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
16 repetitions
OBSERVE H1, 399.9245877 MHz
DATA PROCESSING
Sq. sine bell 2.733 sec
Shifted by -2.733 sec
FT size 131072
Total time 1 min, 22 sec



5d

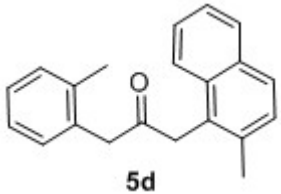


2Me-napht-otolyl-ketone-13C-CDCl3 Mercury400-ATB C13-s2pul-cdcl3

Automation directory:

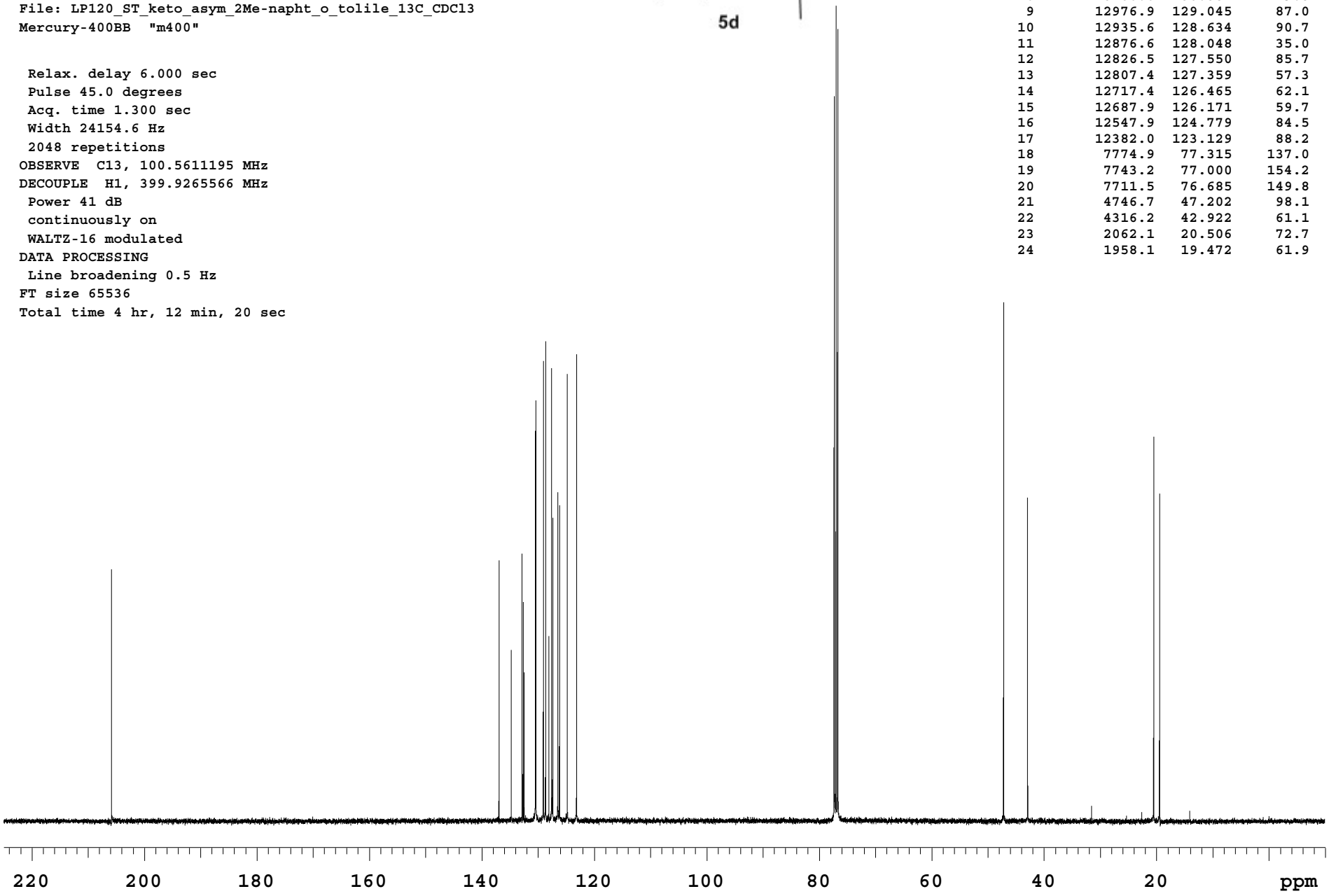
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: LP120_ST_keto_asym_2Me-napht_o_tolile_13C_CDCl3
Mercury-400BB "m400"

Relax. delay 6.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
2048 repetitions
OBSERVE C13, 100.5611195 MHz
DECOUPLE H1, 399.9265566 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 4 hr, 12 min, 20 sec



5d

INDEX	FREQUENCY	PPM	HEIGHT
1	20699.2	205.837	47.6
2	13769.3	136.925	49.3
3	13549.7	134.740	32.3
4	13356.5	132.820	50.6
5	13337.4	132.629	41.4
6	13319.7	132.453	28.1
7	13117.0	130.438	73.7
8	13108.8	130.357	79.5
9	12976.9	129.045	87.0
10	12935.6	128.634	90.7
11	12876.6	128.048	35.0
12	12826.5	127.550	85.7
13	12807.4	127.359	57.3
14	12717.4	126.465	62.1
15	12687.9	126.171	59.7
16	12547.9	124.779	84.5
17	12382.0	123.129	88.2
18	7774.9	77.315	137.0
19	7743.2	77.000	154.2
20	7711.5	76.685	149.8
21	4746.7	47.202	98.1
22	4316.2	42.922	61.1
23	2062.1	20.506	72.7
24	1958.1	19.472	61.9

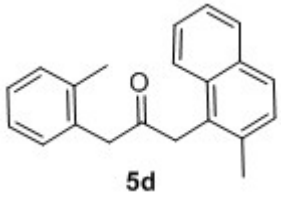


2Me-napht-otolyl-ketone-DEPT-CDCl3 Mercury400-ATB C13-DEPT-cdcl3

Automation directory:

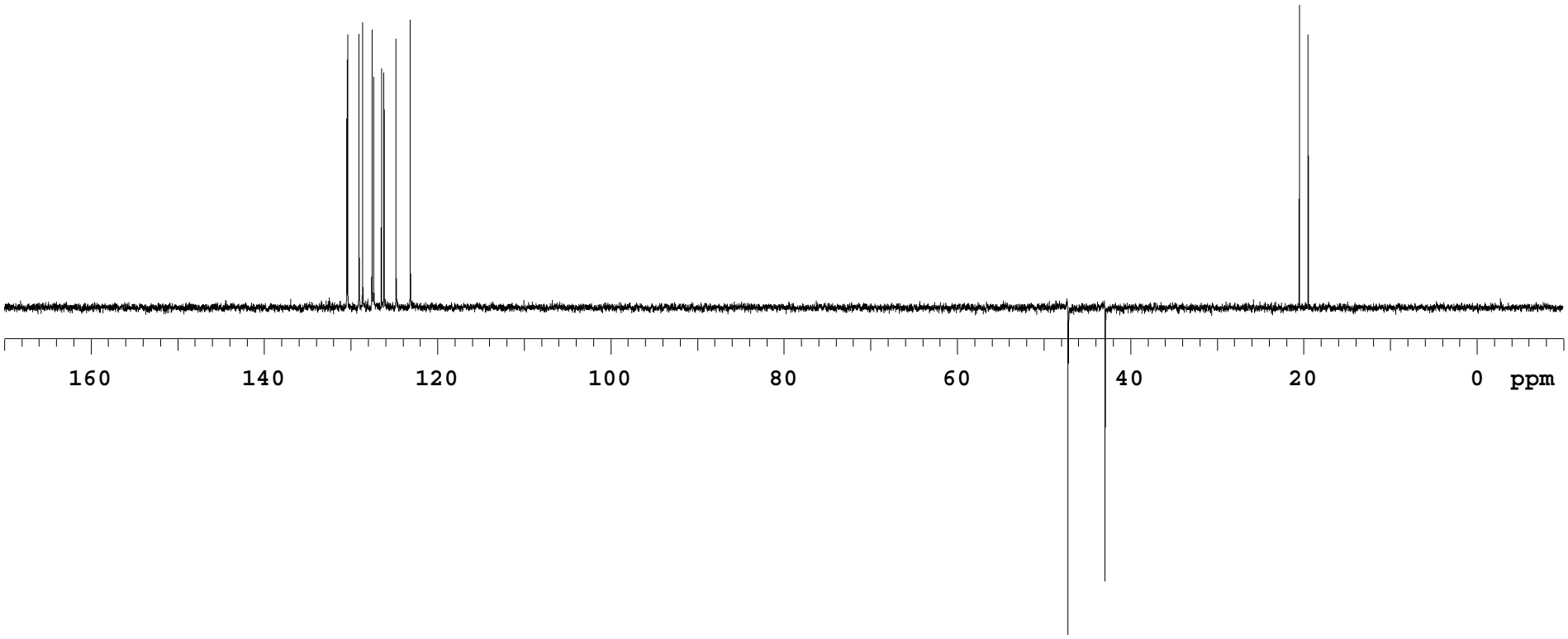
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: LP120_ST_keto_asym_2Me-napht_o_tolile_DEPT_CDCL3
Mercury-400BB "m400"

Relax. delay 4.000 sec
Pulse 90.0 degrees
Acq. time 1.000 sec
Width 24154.6 Hz
256 repetitions
OBSERVE C13, 100.5611195 MHz
DECOUPLE H1, 399.9265566 MHz
Power 41 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 22 min, 37 sec



5d

INDEX	FREQUENCY	PPM	HEIGHT
1	13116.9	130.438	39.6
2	13108.8	130.357	43.7
3	12976.9	129.045	43.7
4	12935.6	128.634	45.6
5	12826.5	127.549	44.5
6	12807.3	127.359	36.9
7	12717.4	126.465	38.3
8	12687.9	126.171	37.6
9	12547.9	124.779	43.0
10	12382.0	123.129	46.1
11	4746.7	47.202	-52.5
12	4315.5	42.914	-43.8
13	2062.1	20.506	48.5
14	1958.1	19.472	43.7

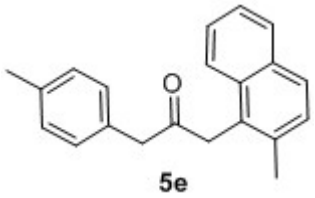


ketone_2Menapht_ptolil-1H-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

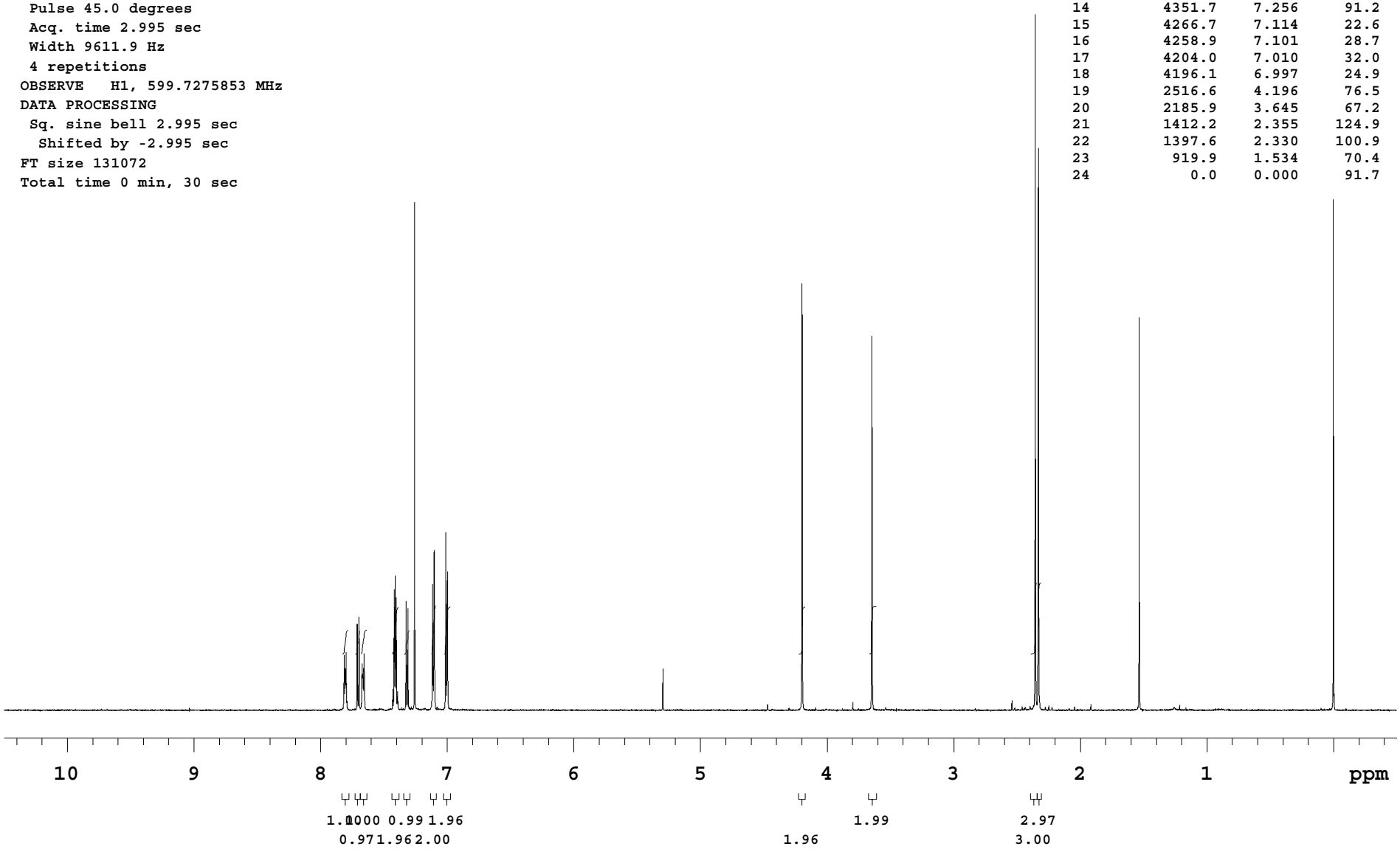
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: keto_2Menapht_ptolil-1H-CDCl3
INOVA-600 "i600"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
4 repetitions
OBSERVE   H1, 599.7275853 MHz
DATA PROCESSING
Sq. sine bell 2.995 sec
Shifted by -2.995 sec
FT size 131072
Total time 0 min, 30 sec



5e

INDEX	FREQUENCY	PPM	HEIGHT
1	4685.4	7.813	9.9
2	4676.0	7.797	10.4
3	4623.8	7.710	15.5
4	4615.4	7.696	16.8
5	4600.6	7.671	8.4
6	4591.1	7.655	10.1
7	4449.3	7.419	13.0
8	4447.2	7.415	21.7
9	4443.4	7.409	24.2
10	4439.6	7.403	20.2
11	4438.0	7.400	12.5
12	4391.9	7.323	19.6
13	4383.6	7.309	18.3
14	4351.7	7.256	91.2
15	4266.7	7.114	22.6
16	4258.9	7.101	28.7
17	4204.0	7.010	32.0
18	4196.1	6.997	24.9
19	2516.6	4.196	76.5
20	2185.9	3.645	67.2
21	1412.2	2.355	124.9
22	1397.6	2.330	100.9
23	919.9	1.534	70.4
24	0.0	0.000	91.7

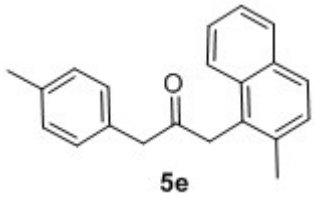


ketone_2Menapht_ptolil-13C-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

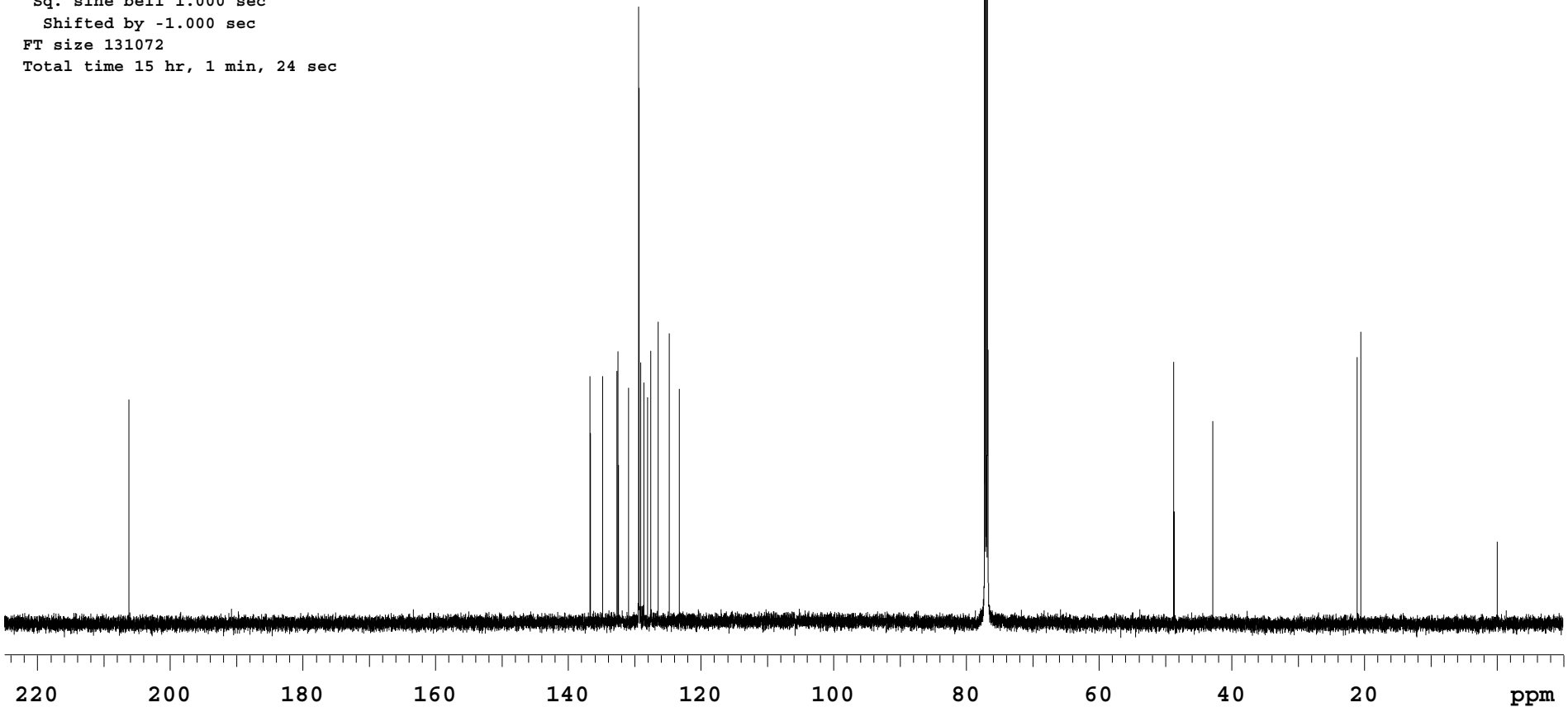
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: keto_2Menapht_ptolil-13C-CDCl3
INOVA-600 "i600"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 36182.7 Hz
9000 repetitions
OBSERVE C13, 150.8016207 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 15 hr, 1 min, 24 sec



5e

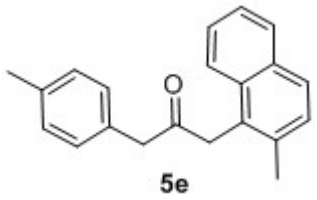
INDEX	FREQUENCY	PPM	HEIGHT
1	31092.2	206.179	35.9
2	20612.1	136.684	39.7
3	20328.9	134.806	39.6
4	20004.8	132.657	40.5
5	19975.0	132.459	43.6
6	19740.9	130.906	37.8
7	19510.1	129.376	98.8
8	19500.7	129.314	85.8
9	19461.5	129.054	41.8
10	19396.4	128.622	38.6
11	19309.7	128.047	36.2
12	19233.5	127.542	43.7
13	19065.1	126.425	48.3
14	18812.8	124.752	46.5
15	18584.3	123.236	37.6
16	11643.7	77.212	1682.0
17	11611.7	77.000	1675.0
18	11580.3	76.791	1668.9
19	7345.1	48.707	41.9
20	6459.5	42.834	32.5
21	3176.7	21.065	42.7
22	3099.9	20.556	46.7
23	-2.3	-0.016	13.1



ketone_2Menapht_ptolil-dept-CDCl3 Inova600-ATB H1-s2pul-cdcl3

Automation directory:

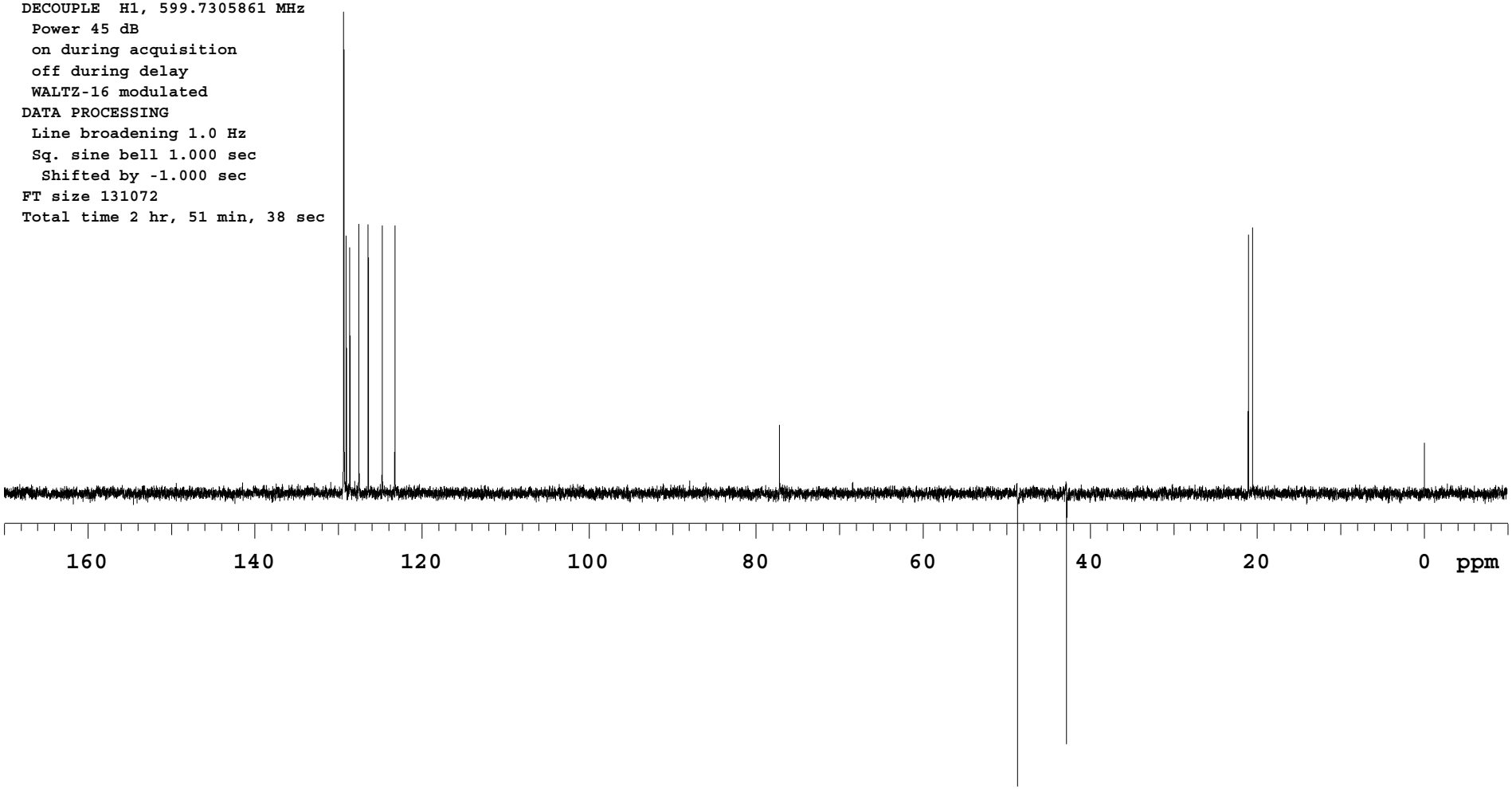
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: lunazzi
File: keto_2Menapht_ptolil-dept-CDCl3
INOVA-600 "i600"



5e

INDEX	FREQUENCY	PPM	HEIGHT
1	19509.0	129.369	79.9
2	19499.6	129.306	73.7
3	19460.4	129.047	42.7
4	19395.3	128.614	40.9
5	19232.4	127.534	44.7
6	19064.0	126.418	44.6
7	18811.7	124.745	44.5
8	18583.1	123.229	44.4
9	7343.9	48.699	-48.7
10	6457.8	42.823	-41.7
11	3175.5	21.058	42.9
12	3098.8	20.549	44.1

Relax. delay 4.000 sec
Pulse 90.0 degrees
Acq. time 1.001 sec
Width 36182.7 Hz
2048 repetitions
OBSERVE C13, 150.8016218 MHz
DECOUPLE H1, 599.7305861 MHz
Power 45 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Sq. sine bell 1.000 sec
Shifted by -1.000 sec
FT size 131072
Total time 2 hr, 51 min, 38 sec

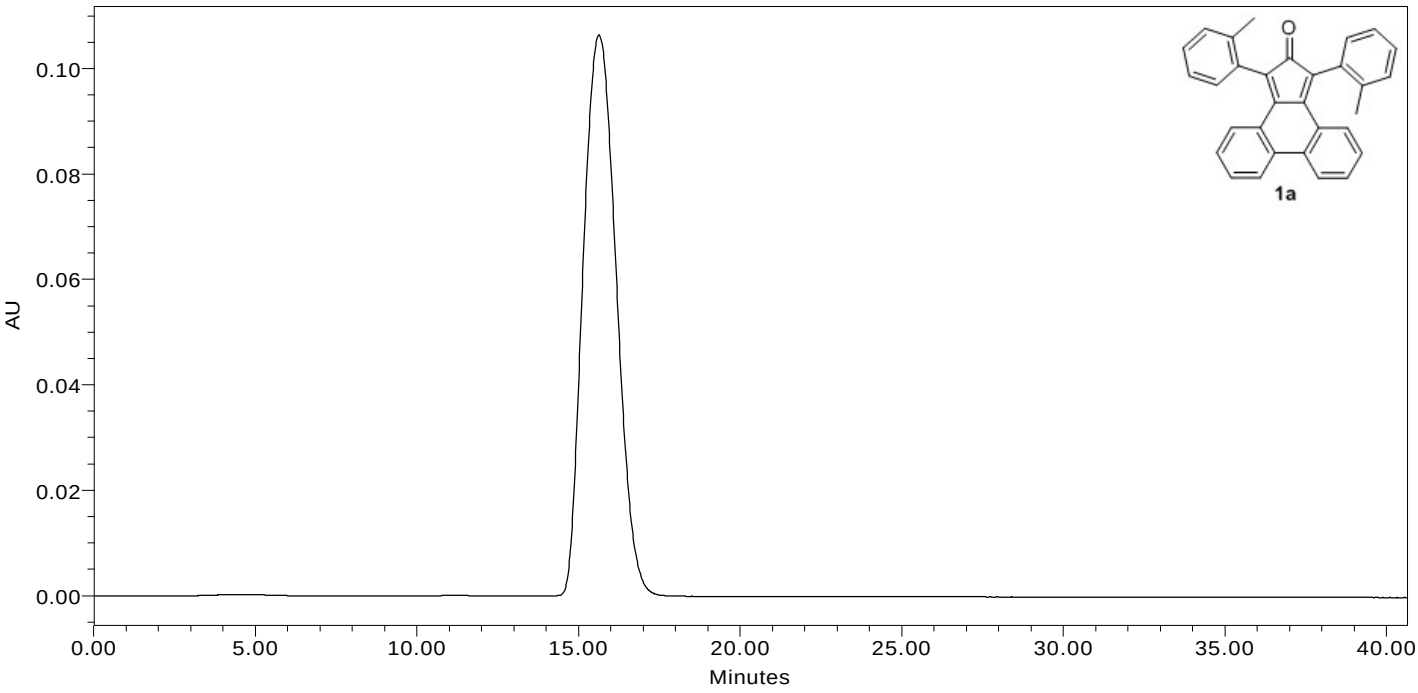


Reported by User: System

Project Name: Kinetex1

SAMPLE INFORMATION

Sample Name:	phenc-di-otolyl-gemini-anal-art	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	7/25/2017 2:50:39 AM
Vial:	13	Acq. Method Set:	azeo_0_1
Injection #:	1	Date Processed:	****
Injection Volume:	10.00 ul	Processing Method	****
Run Time:	60.0 Minutes	Channel Name:	****
Sample Set Name		Proc. Chnl. Descr.:	****



Basic LC Peaks Table group contains no data.

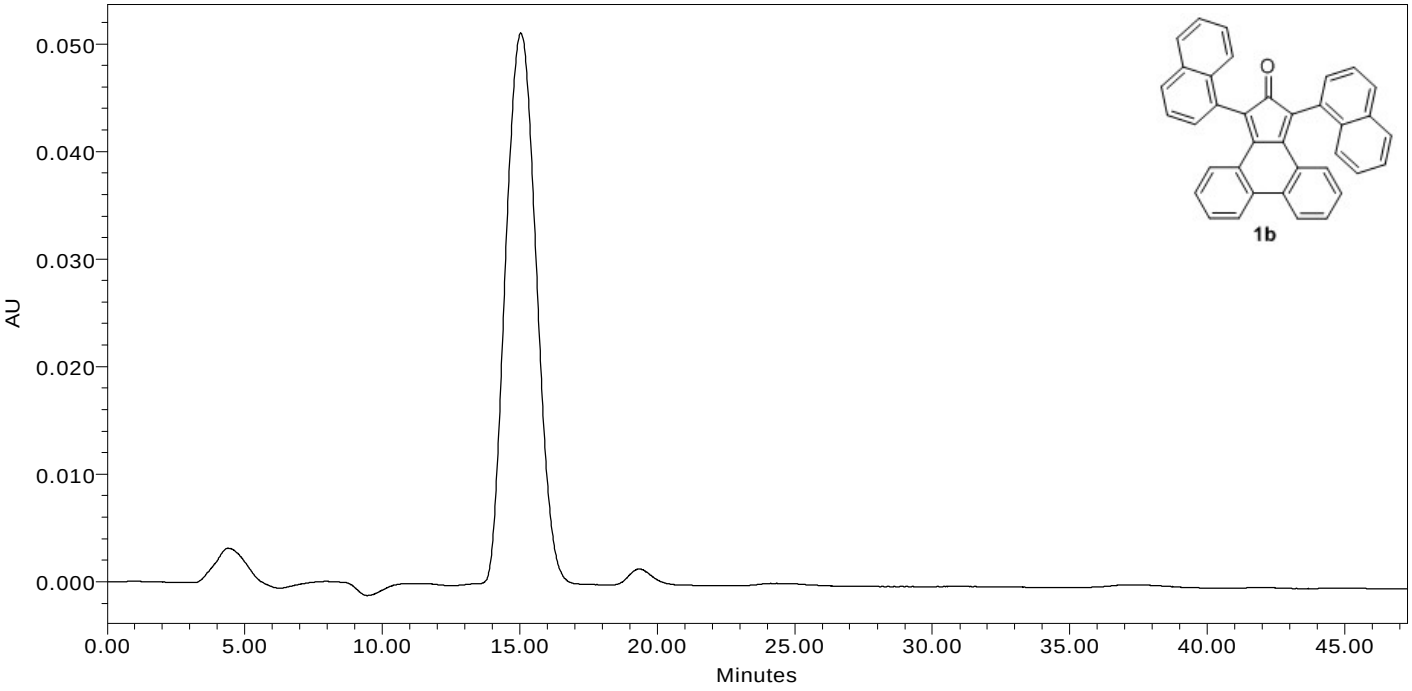
Gemini-NX-5u-C18-110 A, 250 x 4.6 mm, CH_3CN : H_2O 90:10, 1 mL/min, UV: 254 nm

Reported by User: System

Project Name: Kinetex1

SAMPLE INFORMATION

Sample Name:	phenc-di-napht-gemini-anal-art	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	7/25/2017 12:37:24 AM
Vial:	8	Acq. Method Set:	azeo_0_1
Injection #:	1	Date Processed:	****
Injection Volume:	10.00 ul	Processing Method	****
Run Time:	60.0 Minutes	Channel Name:	****
Sample Set Name		Proc. Chnl. Descr.:	****



Basic LC Peaks Table group contains no data.

Gemini-NX-5u-C18-110 A, 250 x 4.6 mm, CH_3CN : H_2O 90:10, 1 mL/min, UV: 254 nm

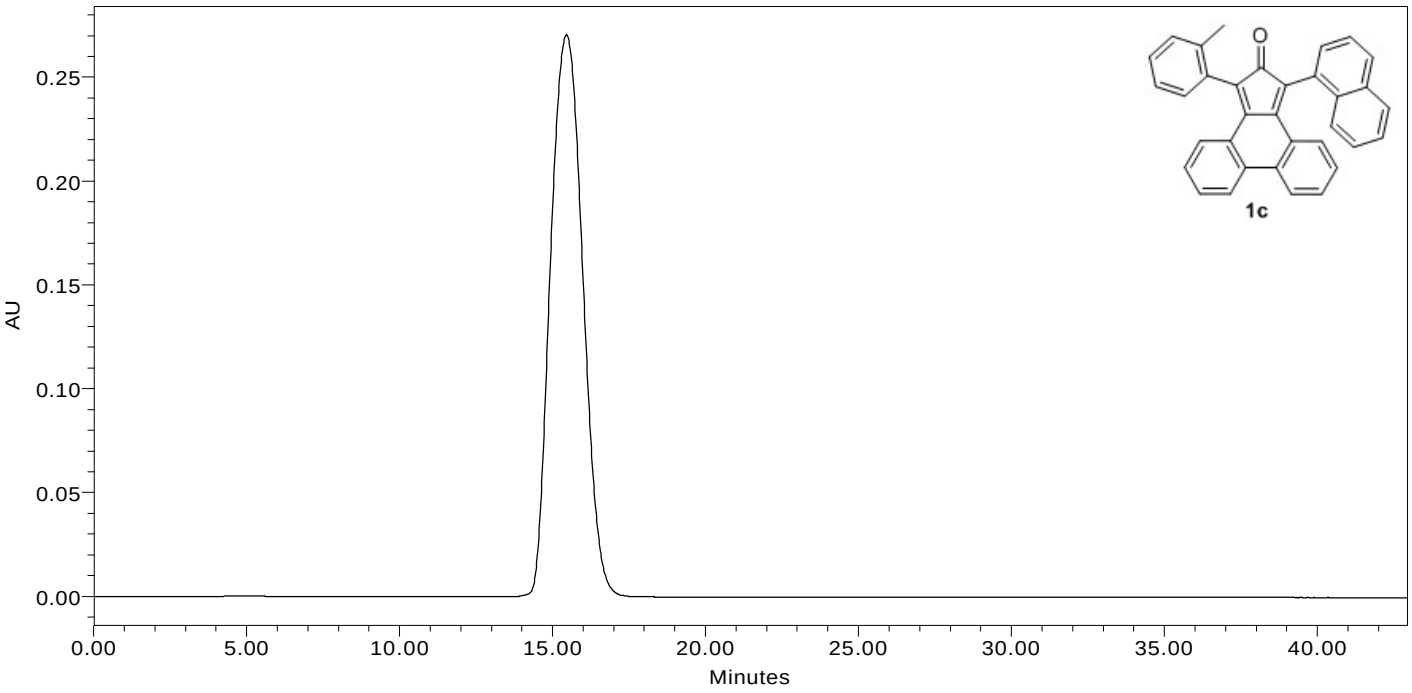
Default Individual Report

Reported by User: System

Project Name: Kinetex1

SAMPLE INFORMATION

Sample Name:	phenc-napht-otolygemini-anal-art	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	7/25/2017 1:46:30 AM
Vial:	11	Acq. Method Set:	azeo_0_1
Injection #:	1	Date Processed:	****
Injection Volume:	10.00 ul	Processing Method	****
Run Time:	60.0 Minutes	Channel Name:	****
Sample Set Name		Proc. Chnl. Descr.:	****



Basic LC Peaks Table group contains no data.

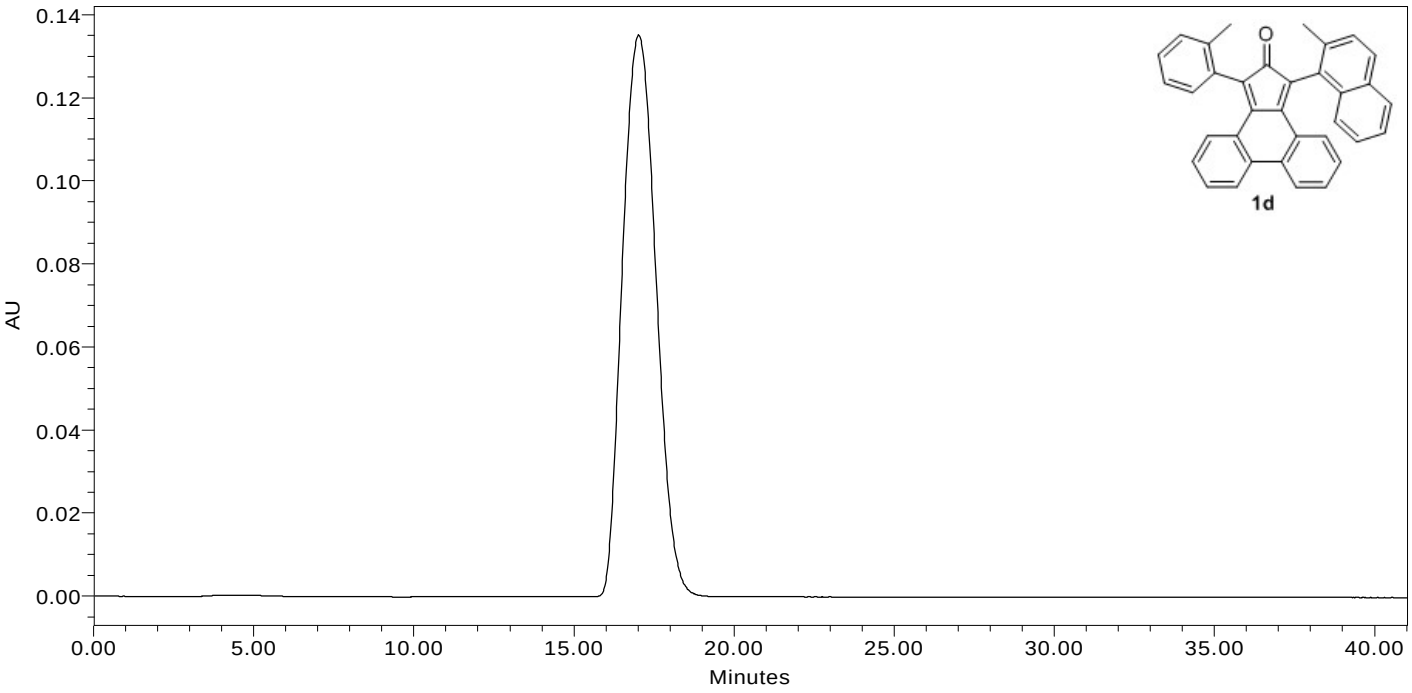
Gemini-NX-5u-C18-110 A, 250 x 4.6 mm, CH_3CN : H_2O 90:10, 1 mL/min, UV: 254 nm

Reported by User: System

Project Name: Kinetex1

SAMPLE INFORMATION

Sample Name:	phenc-2Me-otolyl-gemini-anal-art	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	7/25/2017 3:55:00 AM
Vial:	15	Acq. Method Set:	azeo_0_1
Injection #:	1	Date Processed:	****
Injection Volume:	10.00 ul	Processing Method	****
Run Time:	60.0 Minutes	Channel Name:	****
Sample Set Name		Proc. Chnl. Descr.:	****



Basic LC Peaks Table group contains no data.

Gemini-NX-5u-C18-110 A, 250 x 4.6 mm, CH_3CN : H_2O 90:10, 1 mL/min, UV: 254 nm

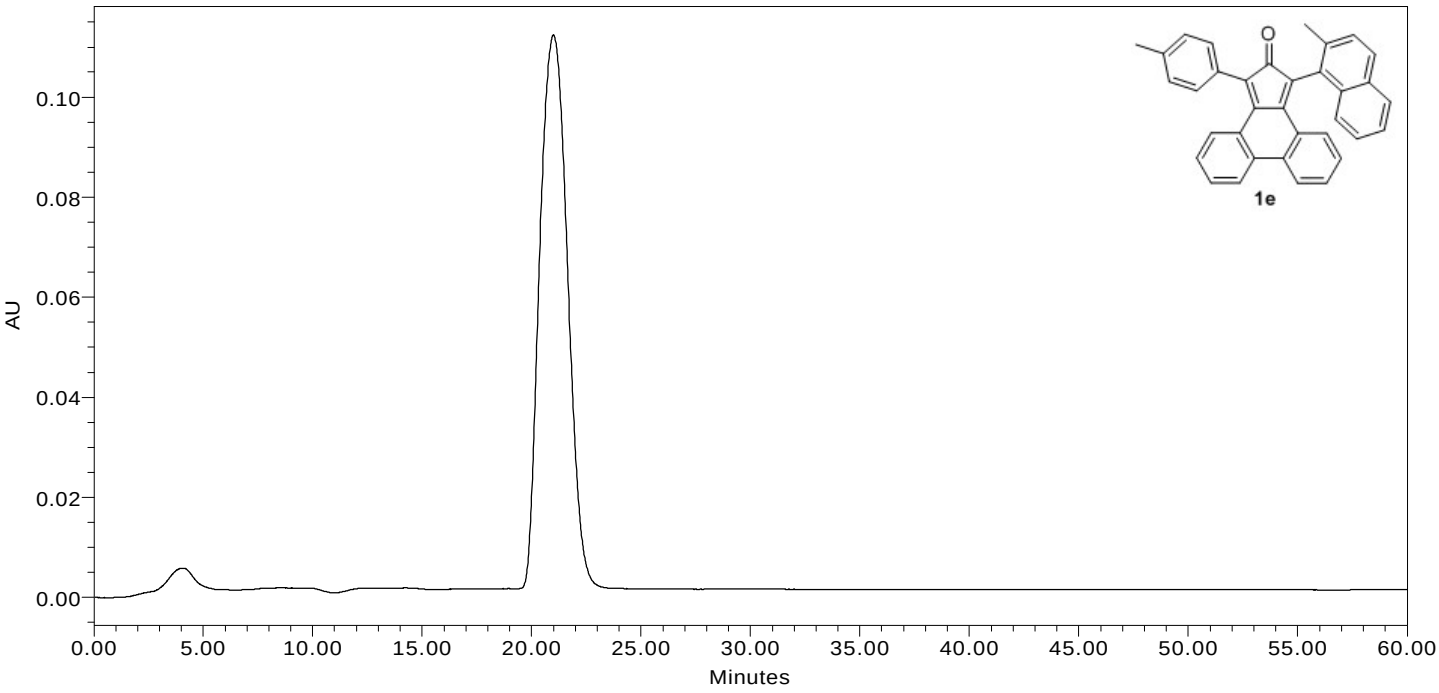
Default Individual Report

Reported by User: System

Project Name: Kinetex1

SAMPLE INFORMATION

Sample Name:	phenc-2Me-ptolyl-gemini-anal-art	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	7/25/2017 4:49:27 AM
Vial:	16	Acq. Method Set:	azeo_0_1
Injection #:	1	Date Processed:	****
Injection Volume:	10.00 ul	Processing Method	****
Run Time:	60.0 Minutes	Channel Name:	****
Sample Set Name		Proc. Chnl. Descr.:	****



Basic LC Peaks Table group contains no data.

Gemini-NX-5u-C18-110 A, 250 x 4.6 mm, CH_3CN : H_2O 90:10, 1 mL/min, UV: 254 nm