Electronic Supplementary Information

Stereoselective synthesis of enantiomerically pure bowl-shaped hydroxytribenzotriquinacenes

*Philipp Rommelmann,[a] Beate Nachtigall,[a] Tim Guntelmann,[a] Harald Gröger,[a,b]** and *Dietmar Kuck[a,b]**

[a]Department of Chemistry, Bielefeld University, Universitätsstraße 25, and
[b]Center for Molecular Materials (CM_2), Bielefeld University,
Universitätsstraße 25, 33615 Bielefeld, Germany

harald.groeger@uni-bielefeld.de
dietmar.kuck@uni-bielefeld.de

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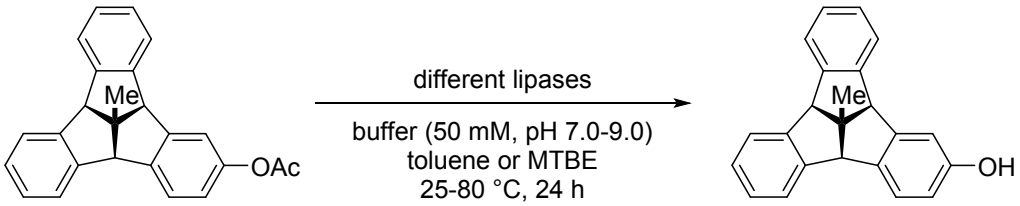
Parameter optimisation of the enzymatic ester hydrolysis

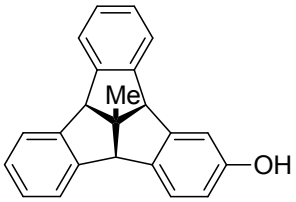


rac-**11** **12**

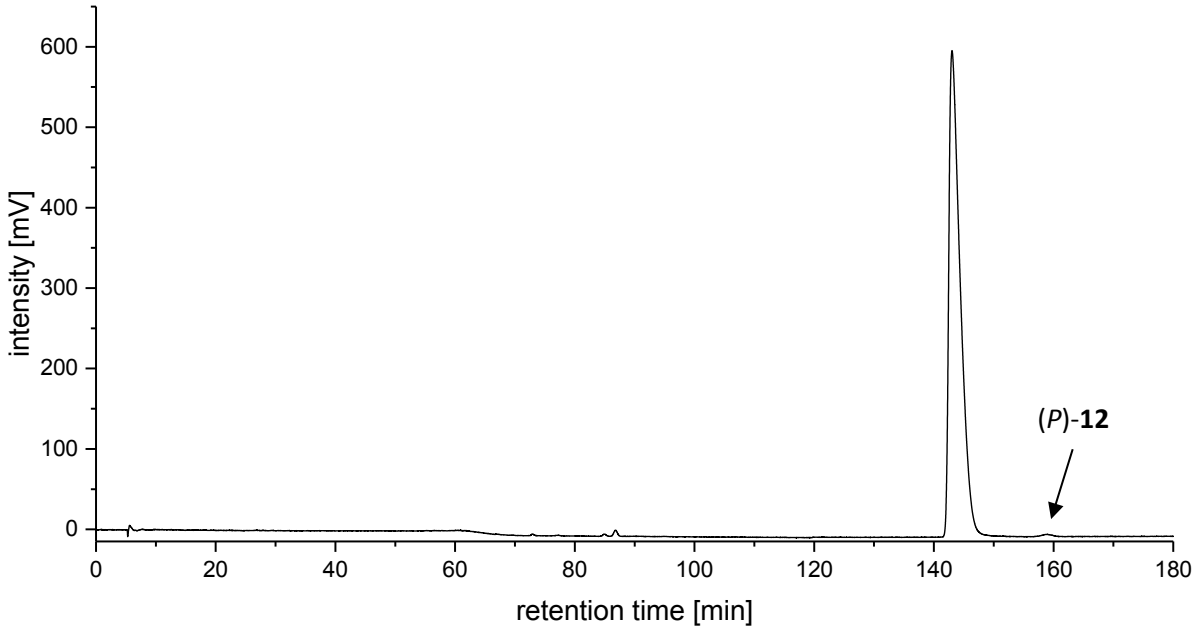
lipase	amount enzyme [mg]	amount *rac*-11 [mg]	solvent ([mL])	buffer[e] (pH/amount [mL])	temp. [° C]	conv. to 12 [%]	*ee* [%]	E-value
CAL-B[a]	25	50	MTBE (7)	KP_i (7.0, 7)	50	3	88	17
CAL-B[a]	50	9	toluene (0.05)	AMP (9.0, 0.5)	25	16	84	15
CAL-B[a]	52	52	toluene (7)	KP_i (7.0, 7)	80	6	94	36
CAL-B[a]	50	9	toluene (0.05)	KP_i (7.4, 0.5)	25	9	74	8
CAL-B[b]	5	9	toluene (0.05)	KP_i (7.4, 0.5)	25	20	38	2
D 20[c]	5	9	toluene (0.05)	KP_i (7.4, 0.5)	25	7	38	2
CAL-B[d]	49	9	toluene (0.05)	KP_i (7.0, 0.5)	25	23	88	25
CAL-B[d]	51	9	toluene (0.05)	AMP (9.0, 0.5)	25	25	92	40
CAL-A[d]	52	9	toluene (0.05)	KP_i (7.0, 0.5)	25	52	48	5
CAL-A[d]	30	30	toluene (10)	KP_i (7.0, 10)	25	40	92	45

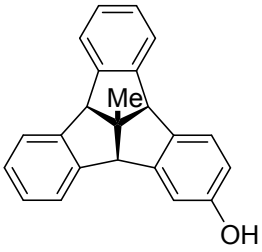
[a]: *C-LEcta* (immobilised); [b]: *C-LEcta* (lyophilised); [c]: *Amano*; [d]: *Sigma Aldrich* (immobilised);
[e]: KP_i: phosphate buffer; AMP: 2-amino-2-methyl-1-propanol buffer.

HPLC chromatograms

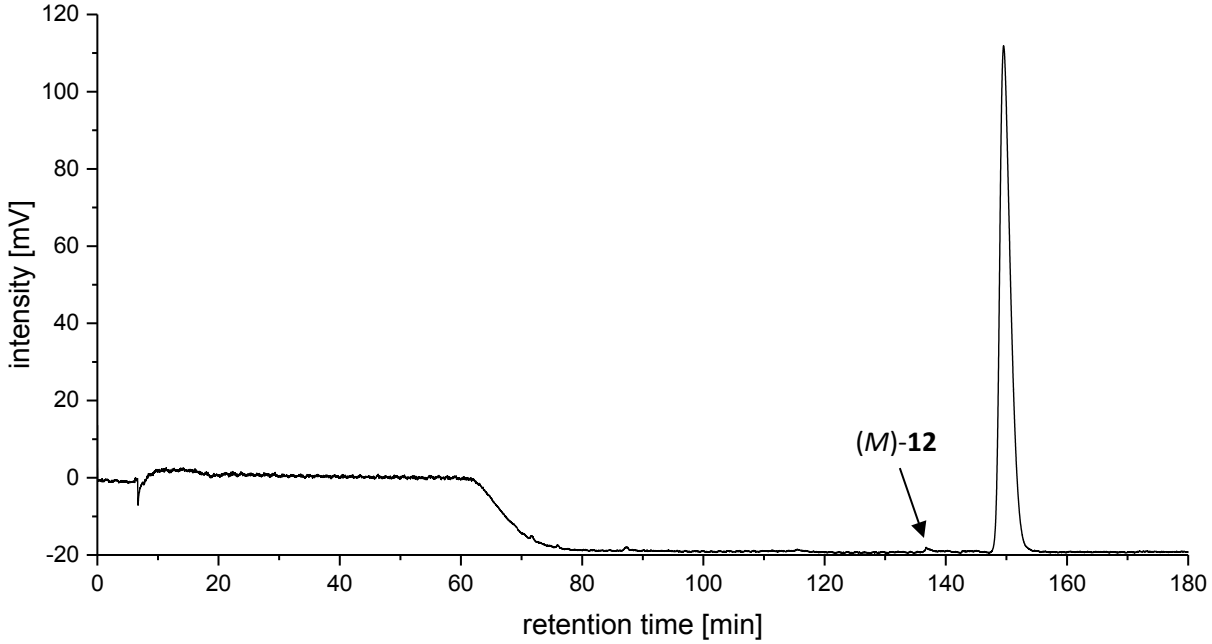


HPLC chromatogram of compound (*M*)-**12** (>99% *ee*)

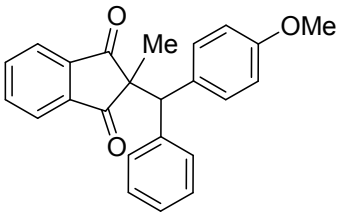




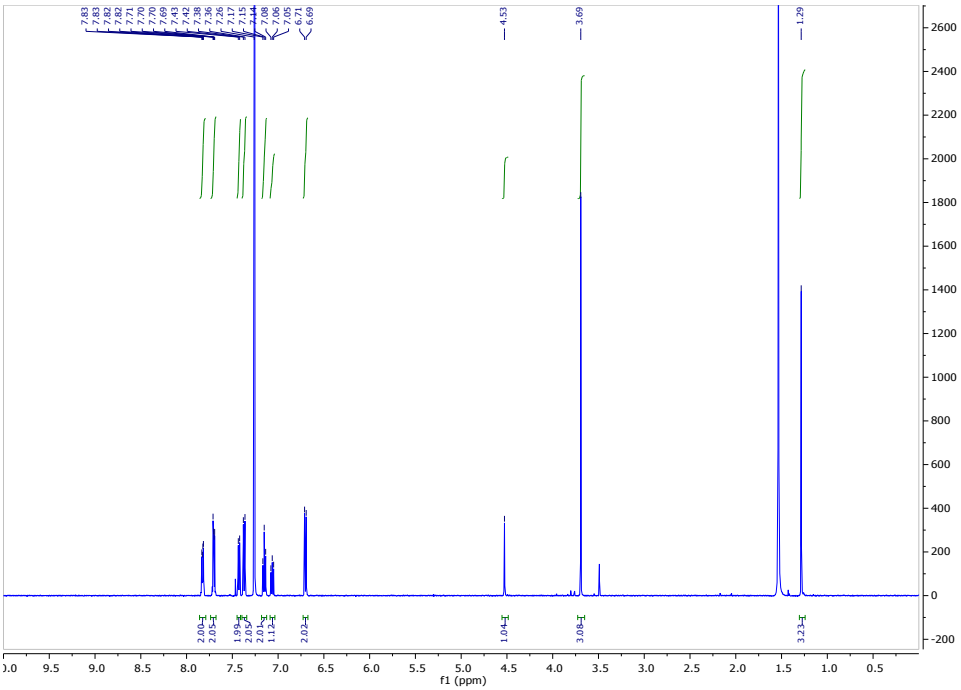
HPLC chromatogram of compound (*P*)-**12** (>99% *ee*)



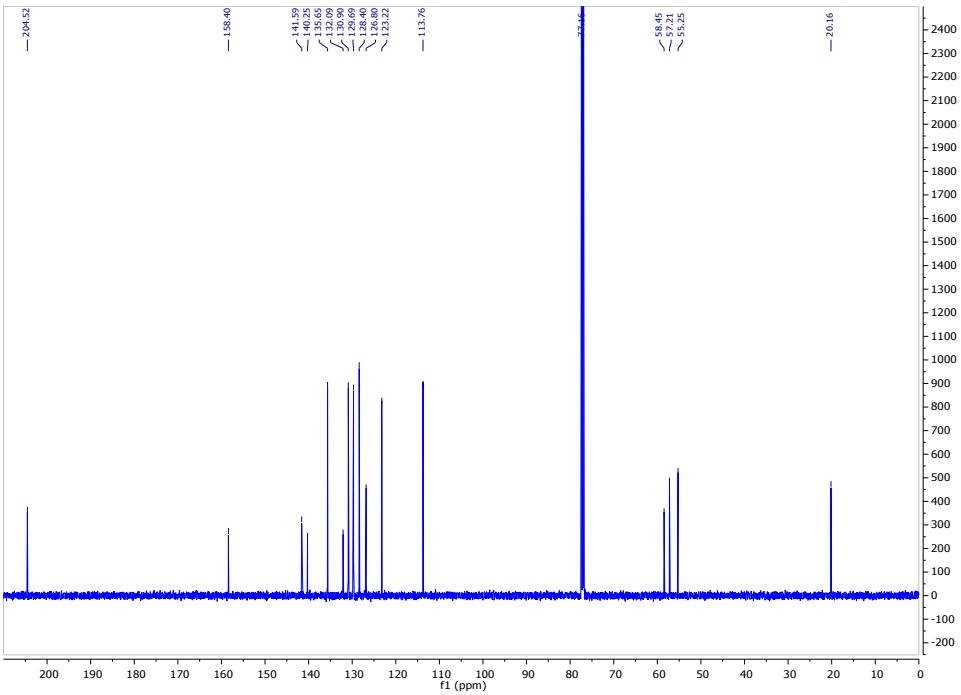
^{1}H and ^{13}C NMR spectra

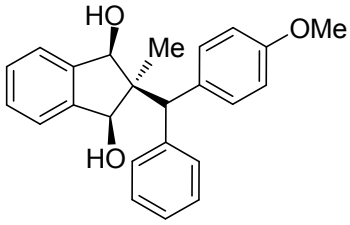


^{1}H NMR spectrum of compound **15** (500 MHz, $CDCl_3$)

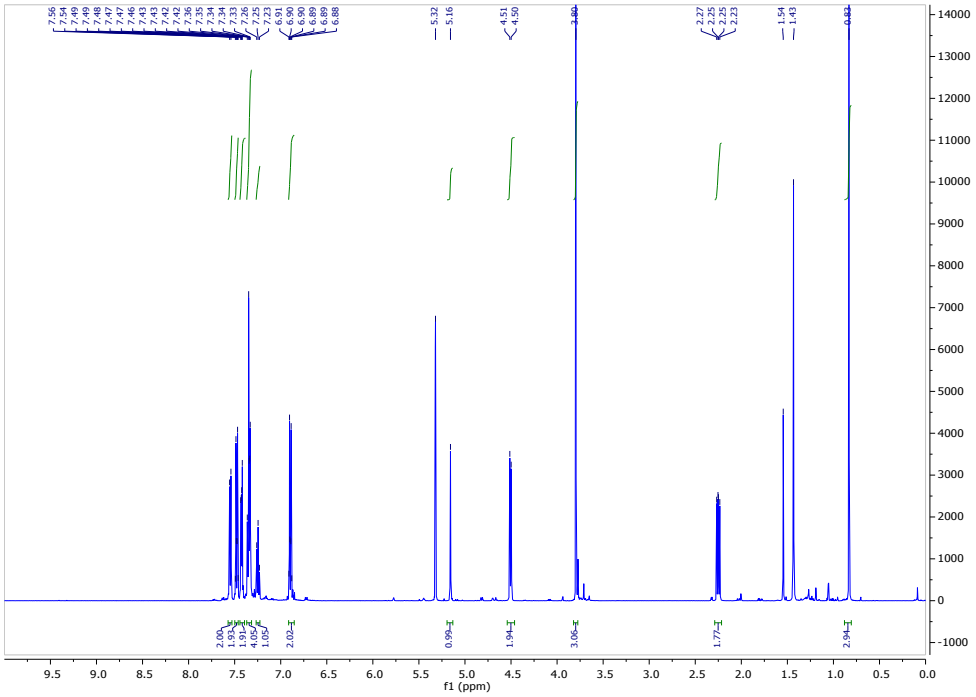


^{13}C NMR spectrum of compound **15** (126 MHz, $CDCl_3$)

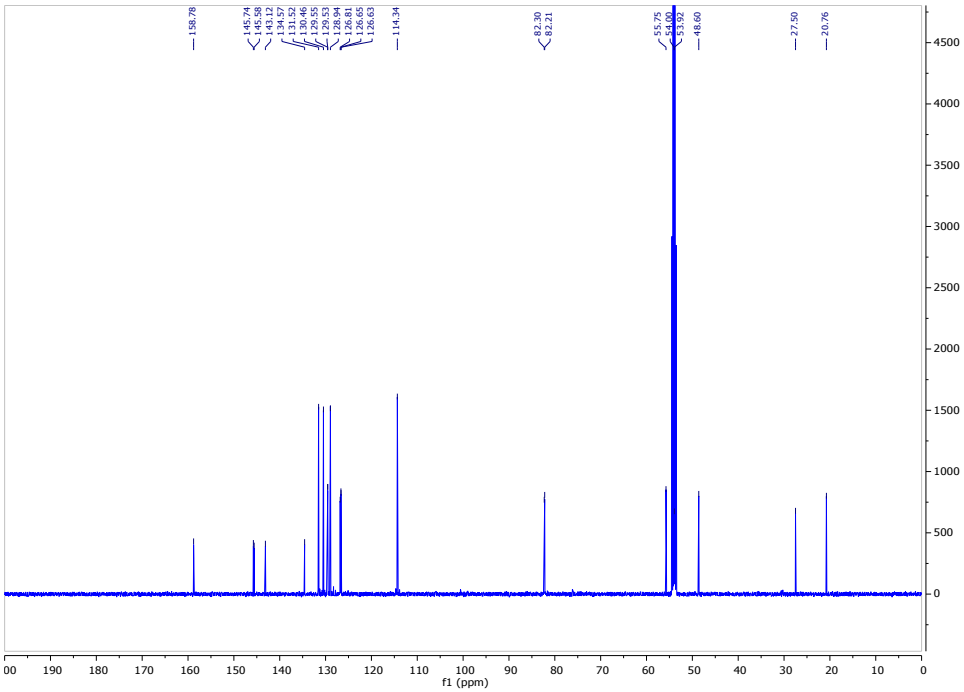


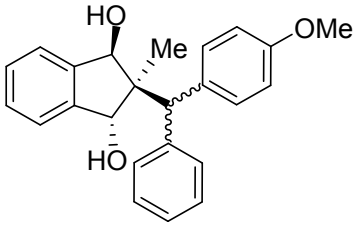


^{1}H NMR spectrum of compound **16a** (all-*cis*-isomer) (500 MHz, $CDCl_3$)

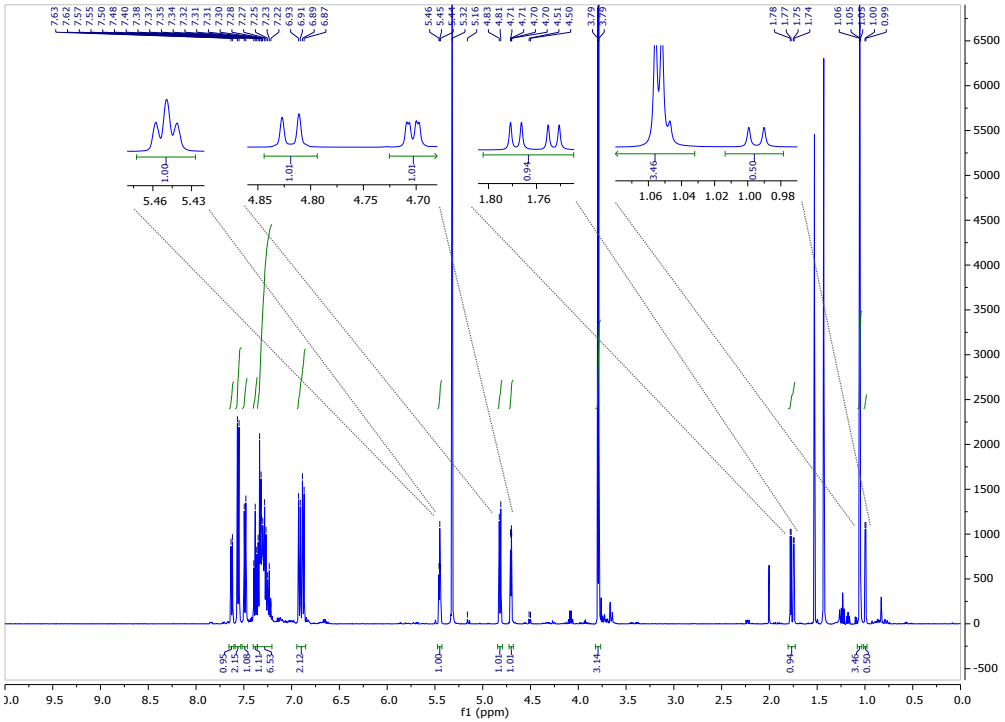


^{13}C NMR spectrum of compound **16a** (all-*cis*-isomer) (126 MHz, $CDCl_3$)

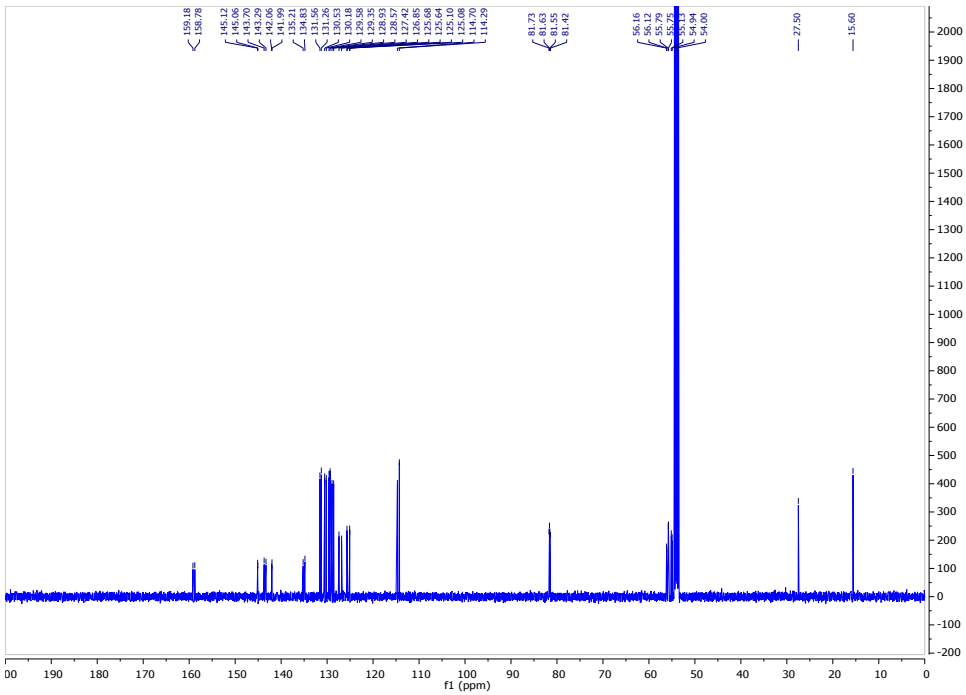


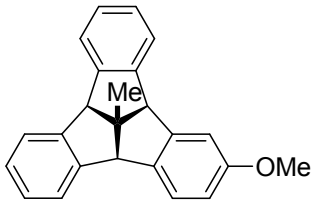


^{1}H NMR spectrum of compound **16b** (*cis*,*trans*-isomer, ~1:1 mixture of two diastereomers) (500 MHz, $CDCl_3$)

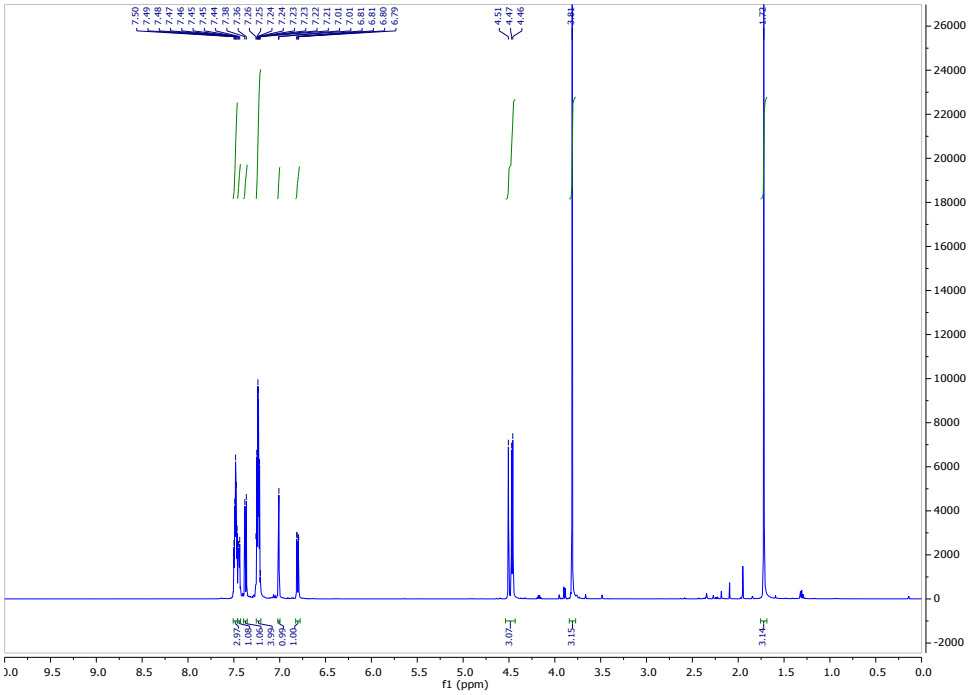


^{13}C NMR spectrum of compound **16b** (*cis*,*trans*-isomer, ~1:1 mixture of two diastereomers) (126 MHz, $CDCl_3$)

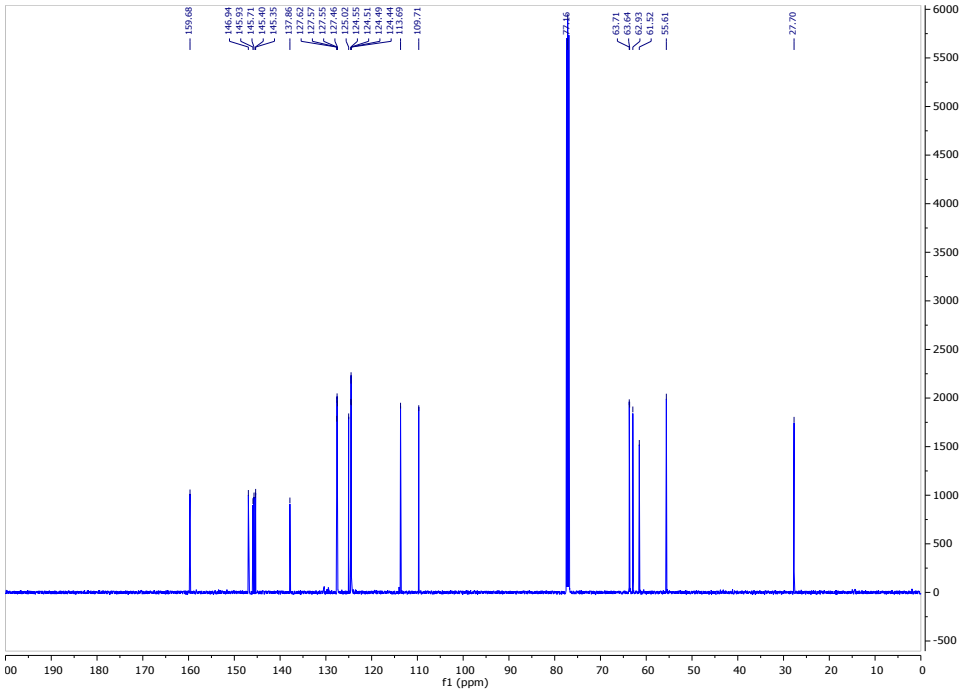


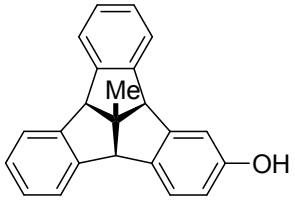


^{1}H NMR spectrum of compound **17** (500 MHz, $CDCl_3$)

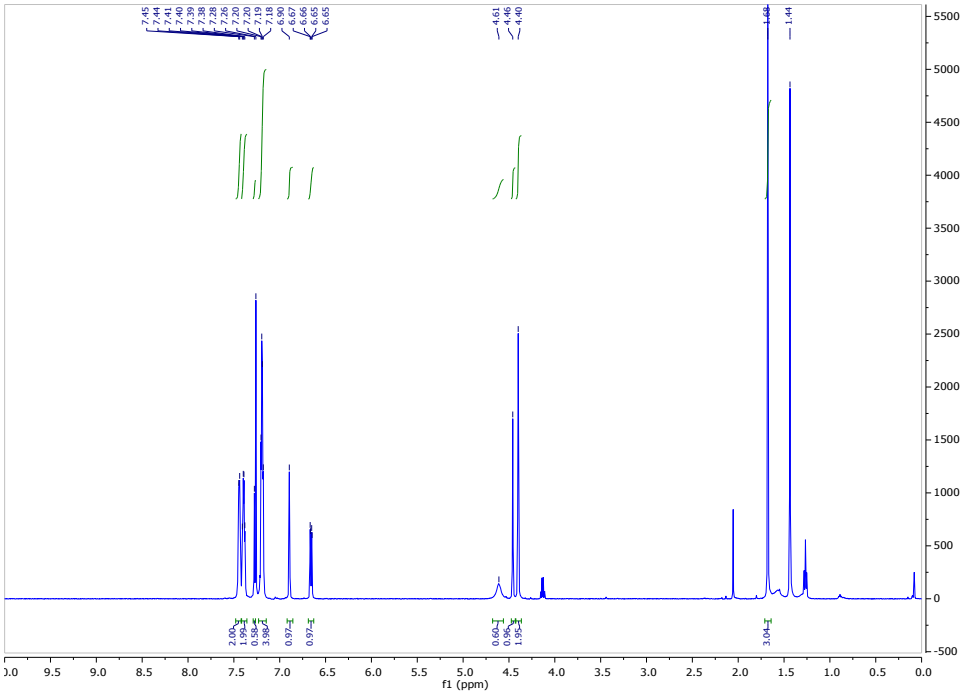


^{13}C NMR spectrum of compound **17** (126 MHz, $CDCl_3$)

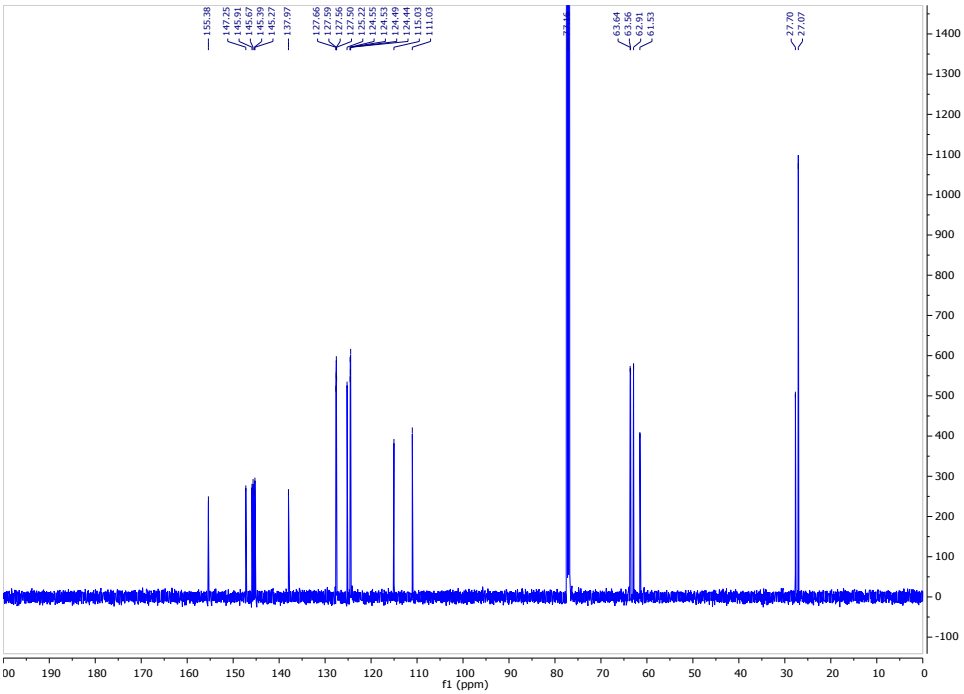


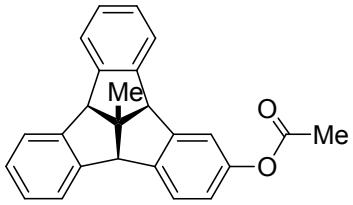


^{1}H NMR spectrum of compound **12** (500 MHz, $CDCl_3$)

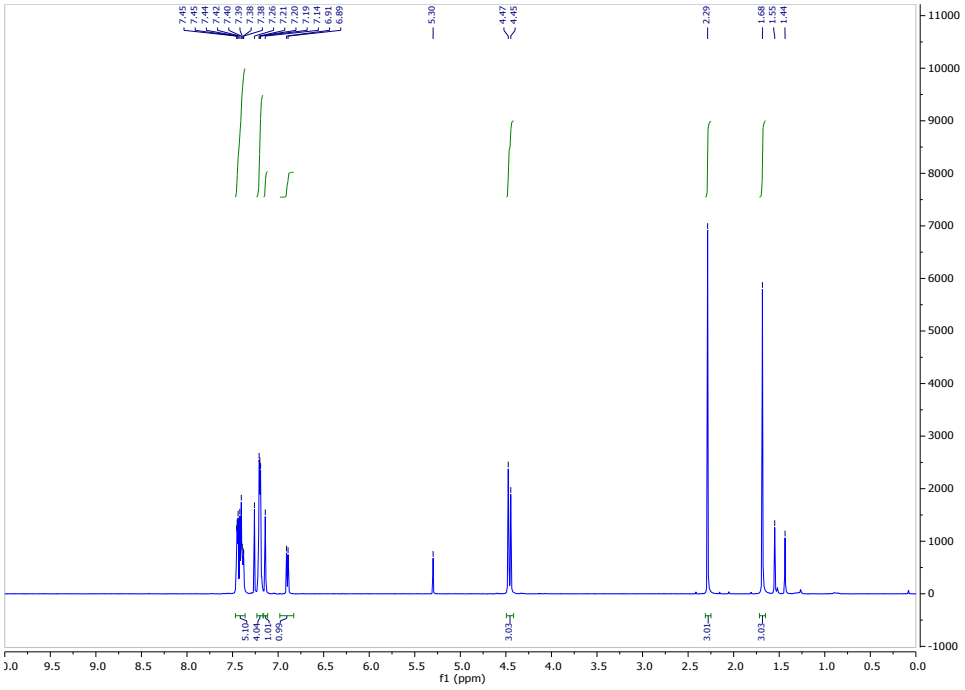


^{13}C NMR spectrum of compound **12** (126 MHz, $CDCl_3$)

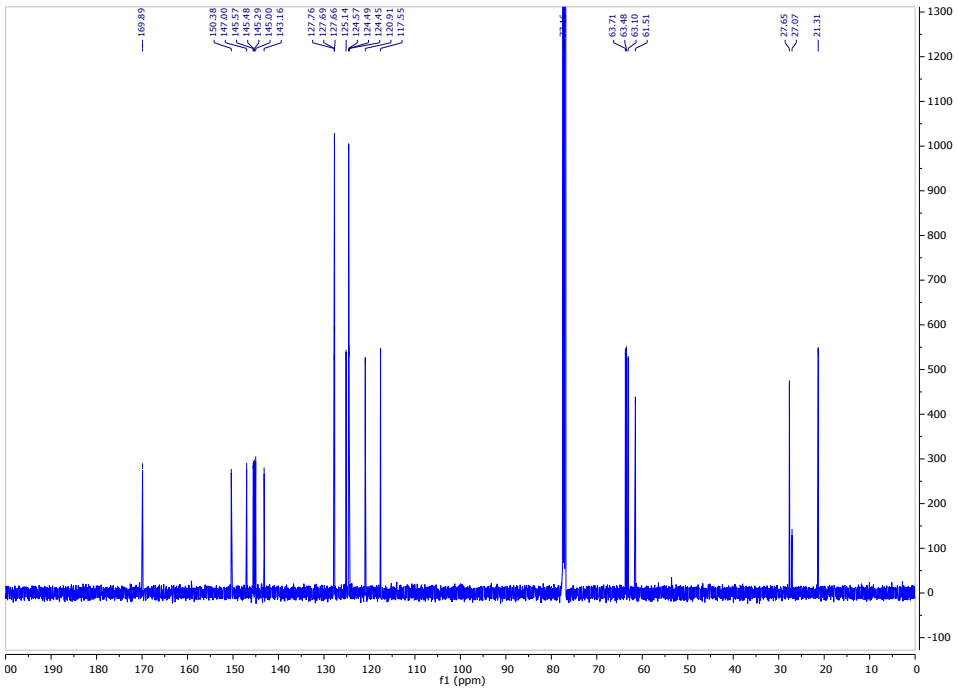


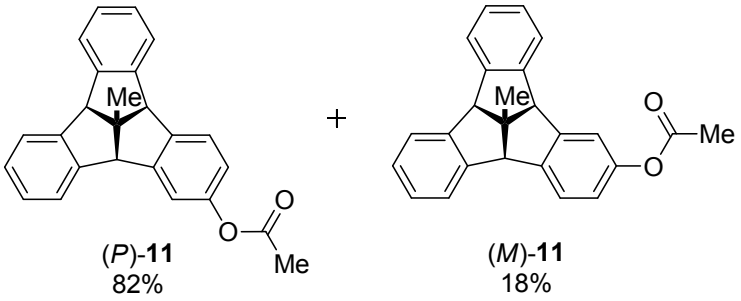


^{1}H NMR spectrum of compound **11** (500 MHz, $CDCl_3$); residual solvent signals: 5.30 ppm (dichloromethane), 1.55 ppm (water), 1.44 ppm (cyclohexane).

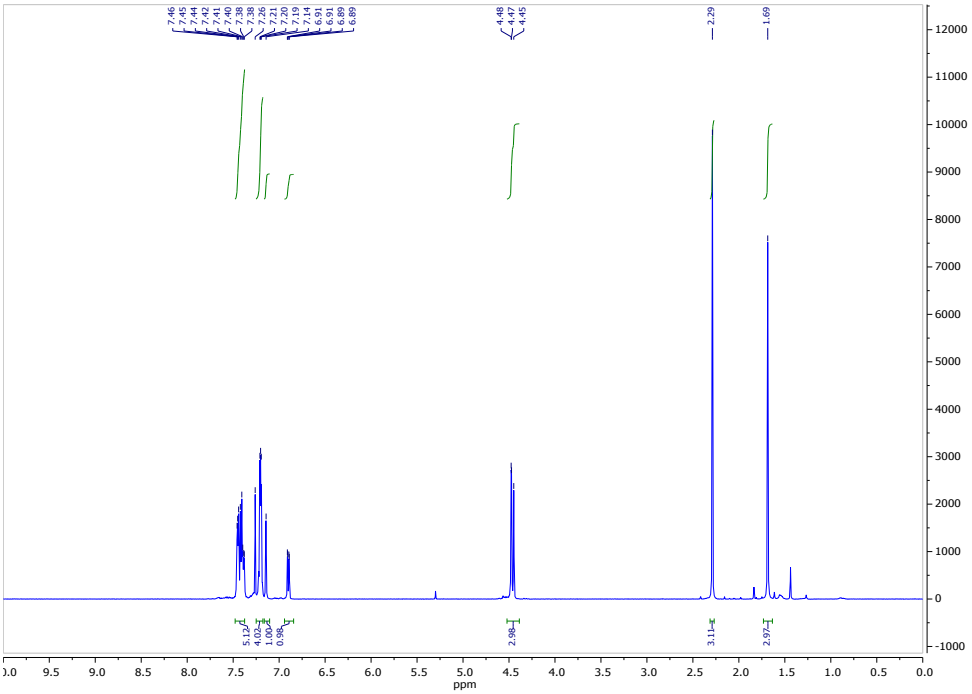


^{13}C NMR spectrum of compound **11** (126 MHz, $CDCl_3$); residual solvent signals: solvent residuals: 27.07 ppm (cyclohexane).

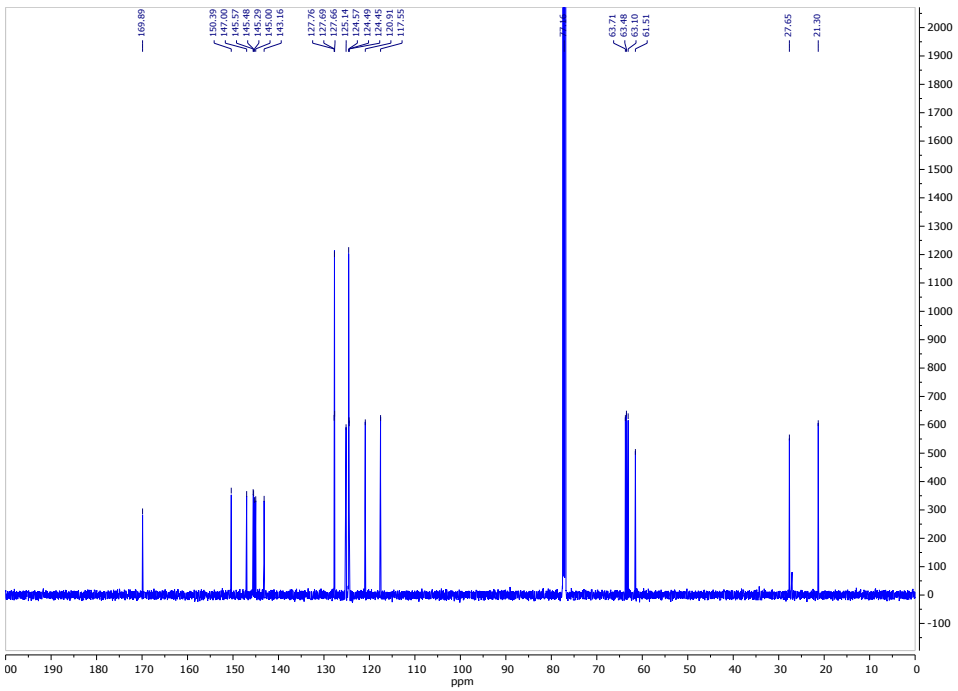


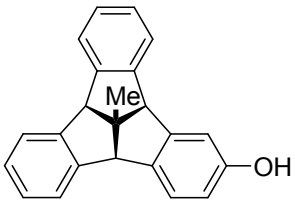


^{1}H NMR spectrum of compound (*P*)-**11** (64% *ee*) (500 MHz, $CDCl_3$)

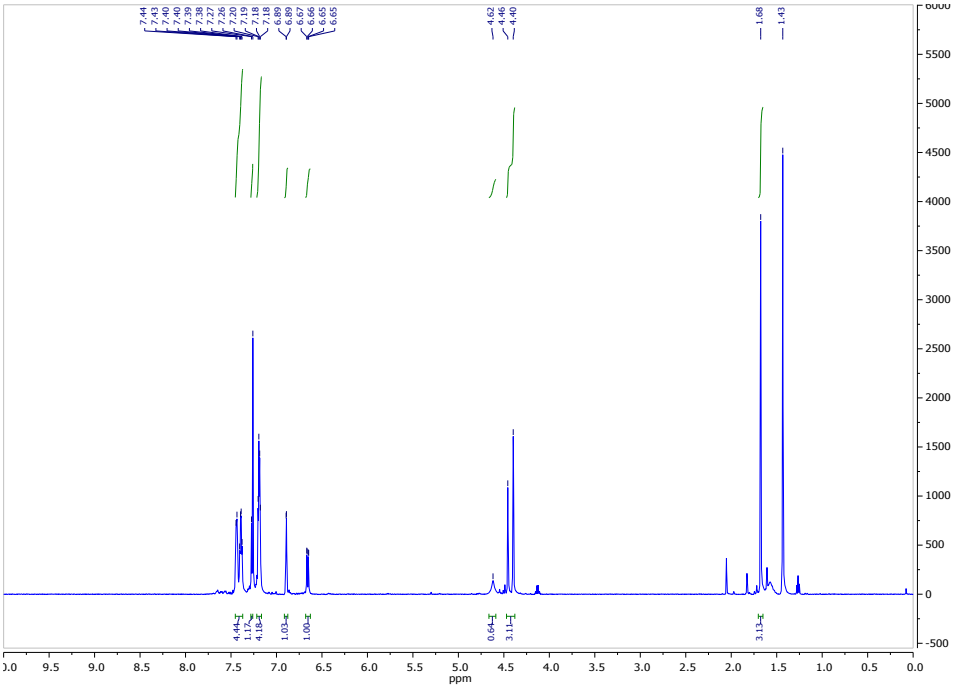


^{13}C NMR spectrum of compound (*P*)-**11** (64% *ee*) (126 MHz, $CDCl_3$)

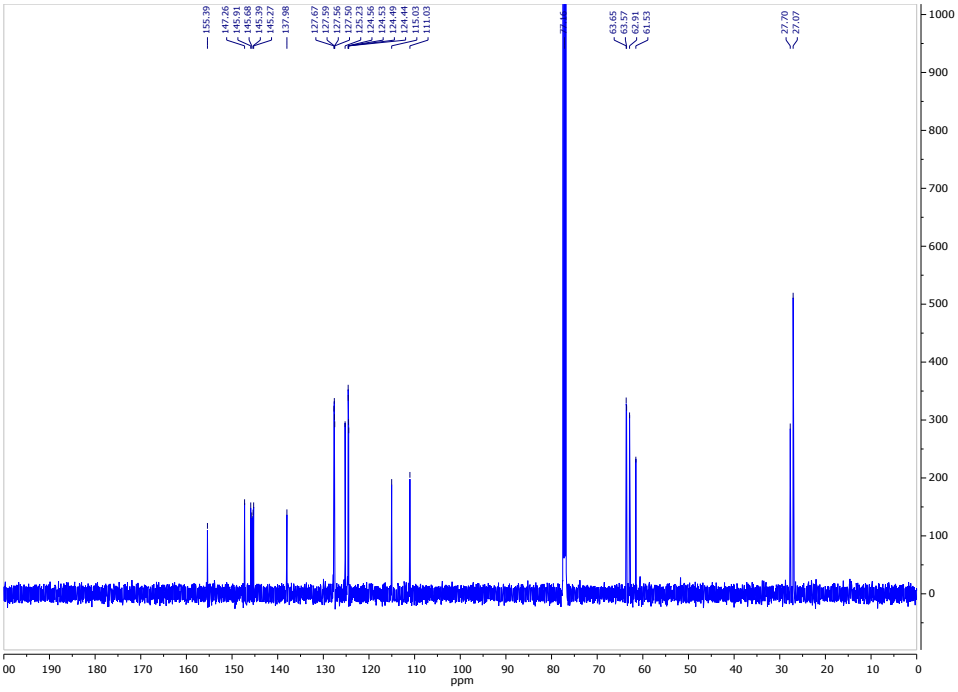


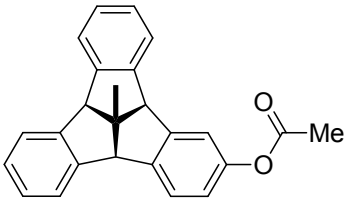


^{1}H NMR spectrum of compound (*M*)-**12** (>99% *ee*) (500 MHz, $CDCl_3$)

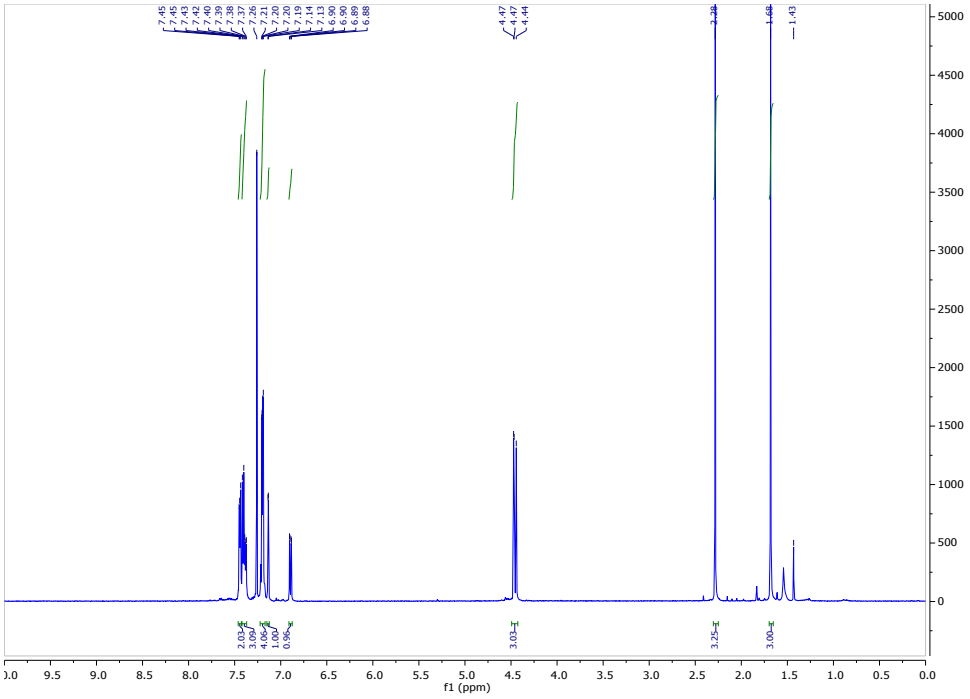


^{13}C NMR spectrum of compound (*M*)-**12** (>99% *ee*) (126 MHz, $CDCl_3$)

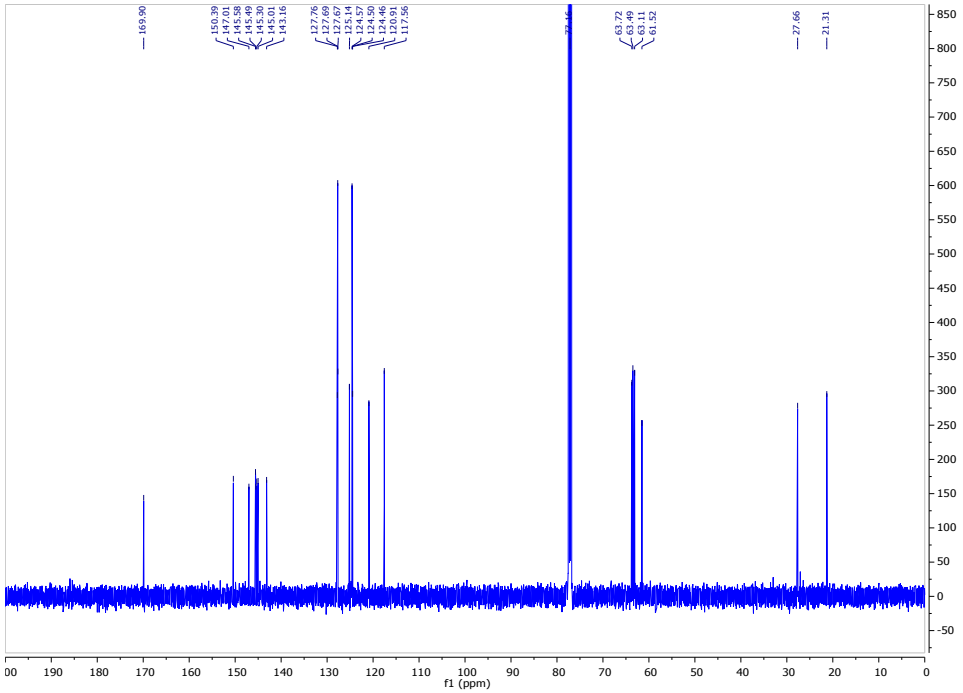


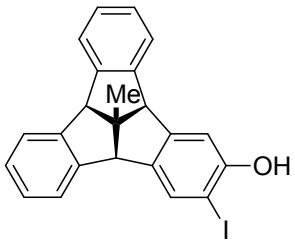


^{1}H NMR spectrum of compound (*M*)-**11** (500 MHz, $CDCl_3$)

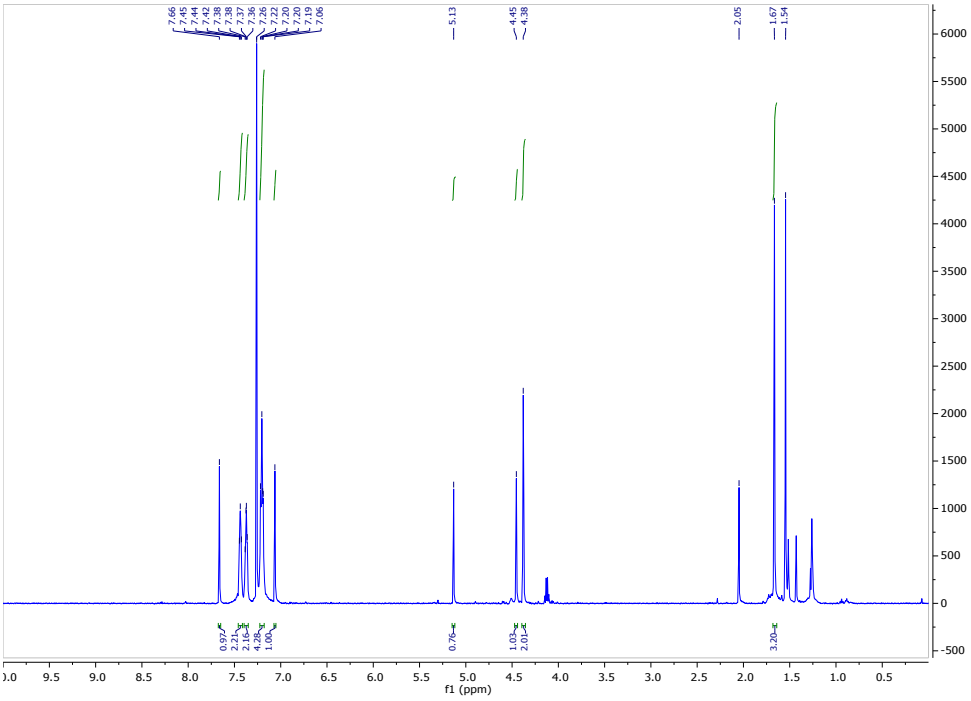


^{13}C NMR spectrum of compound (*M*)-**11** (126 MHz, $CDCl_3$)

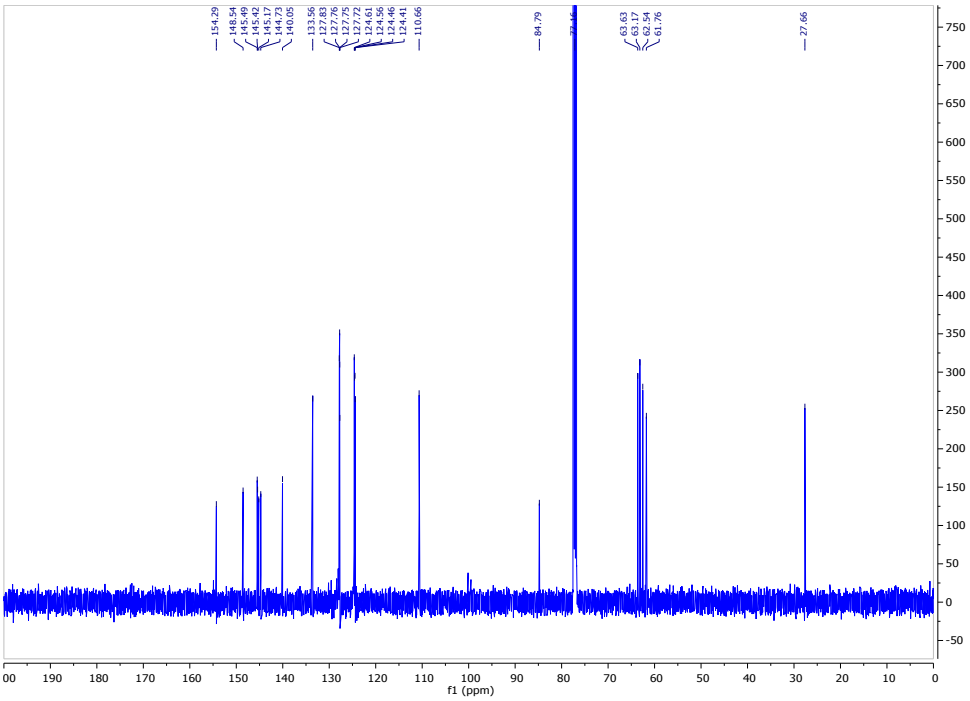


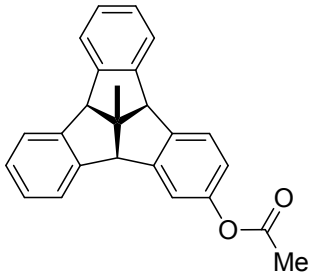


^{1}H NMR spectrum of compound (*P*)-**13** (500 MHz, $CDCl_3$)

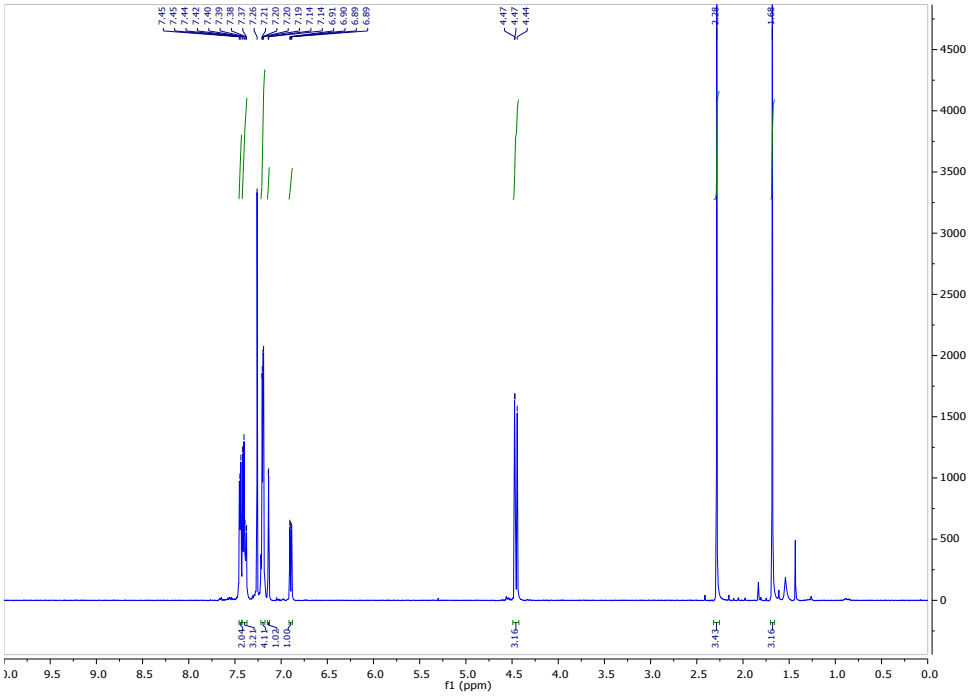


^{13}C NMR spectrum of compound (*P*)-**13** (126 MHz, $CDCl_3$)





^{1}H NMR spectrum of compound (*P*)-**11** (500 MHz, $CDCl_3$)



^{13}C NMR spectrum of compound (*P*)-**11** (126 MHz, $CDCl_3$)