

Iminium ion catalysis: the enantioselective Friedel-Crafts alkylation-acetalization cascade of naphthols with α,β -unsaturated cyclic ketones

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Supplementary Information

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General informations.

The ^1H and ^{13}C NMR spectra were recorded on a Varian inova 300, at 300 MHz and 75 MHz respectively, Varian mercury 400, at 400 MHz and 100 MHz respectively, or Varian inova 600, at 600 MHz and 150 MHz respectively. The chemical shifts (δ) for ^1H and ^{13}C are given in ppm relative to residual signals of the solvents (CDCl_3 , $\text{DMSO}-d_6$). The following abbreviations are used to indicate the multiplicity: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; bs, broad signal. CDCl_3 was passed over a short pad of alumina before use. Coupling constants are given in Hz. When 2D-NMR were not performed, the carbon types were determined from DEPT ^{13}C NMR experiments. NOE spectra were recorded using the DPFGE-NOE sequence¹ using a mixing time of 2.00 s and “rsnob” 20 - 50 Hz wide selective pulses, depending on the crowding of the spectra region. High Resolution Mass spectra (HRMS) were obtained from the Department of Organic Chemistry “A. Mangini” Mass Spectroscopy facility, on a Thermo-Finnigan MAT 95 XP spectrometer. X-ray data were acquired at the Department of Physical and Inorganic Chemistry X-ray Crystallography facility, on a Bruker APEX-2 diffractometer. Optical rotations were measured on a Perkin-Elmer 341 polarimeter and reported as follow: $[\alpha]_D^t$ (c in g per 100 mL, solvent). Thin Layer Chromatography (TLC) was performed on commercially available Fluka TLC plates on aluminium or PET foils with fluorescent indicator at 254 nm, using UV light as the visualizing agent and an acidic mixture of ceric ammonium molybdate or basic aqueous potassium permanganate (KMnO_4), and heat as developing agents.

Purification of the products was carried out by flash chromatography (FC) on silica gel (Aldrich, 230-400 mesh) according to the method of Still². Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator.

Materials

All the commercially available reagents and solvents were used without any further purifications; otherwise, where necessary, they were purified as recommended³. Chiral primary amine catalysts 9-amino(9-deoxy)*epi*-quinine **A** and its *pseudo*-enantiomer 9-amino(9-deoxy)*epi*-quinidine **ent-A** were synthesized according to literature procedures⁴.

¹ (a) K. Stott, J. Stonehouse, J. Keeler, T.-L. Hwand and A. Shaka, *J. Am. Chem. Soc.* 1995, **117**, 4199. (b) K. Stott, J. Keeler, Q. N. Van and A. J. Shaka, *J. Magn. Resonance* 1997, **125**, 302. (c) Q. N. Van, E. M. Smith and A. J. Shaka, *J. Magn. Resonance* 1999, **141**, 191. (d) See also: Claridge, T.D.W. *High Resolution NMR Techniques in Organic Chemistry*; Pergamon: Amsterdam, 1999.

² W. C. Still, M. Kahn and A. J. Mitra, *J. Org. Chem.* 1978, **43**, 2923.

³ W. L. F. Armarego and D. D. Perrin, In *Purification of Laboratory Chemicals*, 4th ed.; Butterworth Heinemann: Oxford, 1996.

⁴ (a) S. H.; McCooey and S. J.; *Connon Org. Lett.* 2007, **9**, 599; (b) W. Chen, W. Du, Y.-Z. Duan, Y. Wu, S.-Y. Yang and Y.-C. Chen, *Angew. Chem. Int. Ed.* 2007, **46**, 7667.

All the ketones and the naphthols were purchased from Sigma-Aldrich or Alfa Aesar and used as received. Compounds **1c-d**⁵ and **9a-d**⁶ were prepared following the literature procedures *Tert-butyl (3-hydroxynaphthalen-2-yl)carbamate 2d* was synthesized according to the literature procedure⁷. *Tert-butyl (4-hydroxynaphthalen-1-yl)carbamate 4d* was synthesized as follows:

In a flame-dried flask equipped with a condenser and a magnetic stirring bar, triethylamine (0.344 ml, 1 eq.) was added to a 0.5 M solution of 4-amino-1-naphthol hydrochloride (2.56 mmol, 0.5 g, 1 eq.) in anhydrous THF (5.11 ml). The reaction mixture was left to stir at r.t. for 5 min. Then di-*tert*-butyl dicarbonate (0.558 g, 1 eq.) was added as a solid and the reaction mixture was stirred and refluxed for three days. Then it was cooled to room temperature and the solvent was removed in vacuo. The crude product was purified through flash chromatography on silica gel (Hex/EtOAc 70:30) to give a violet solid, which was crystallized from Et₂O/Hex to afford **4d** in 57% yield as a pink solid.

¹H NMR (300 MHz, CDCl₃)⁸: δ 1.58 (s, 9H), 6.41 (d, 1H, *J* = 7.8 Hz), 6.49 (bs, 1H), 6.71 (bs, 1H), 7.22 (d, 1H, *J* = 8.1 Hz), 7.40 (t, 1H, *J* = 8.2 Hz), 7.51 (dt, 1H, *J*_a = 8.2 Hz, *J*_b = 1.5 Hz), 7.84 (d, 1H, *J* = 8.4 Hz), 8.05 (d, 1H, *J* = 8.4 Hz).

Determination of diastereomeric ratios and enantiomeric purity.

Diastereomeric ratios was determined by ¹H NMR spectroscopy of the crude product. Enantiomeric excesses were determined, after purification, through HPLC analysis on chiral stationary phase performed on an Agilent 1100-series instrumentation using Daicel Chiralpak AD-H, Daicel Chiralpak AS-H, Daicel Chiralcel OD-H, Daicel Chiralcel OJ-H, Phenomenex Lux-Amilose 2 and Phenomenex Lux-Cellulose 2 columns. Racemic samples of compounds **3ab**, **3ac**, **5ab**, **5ac**, **10aa** were obtained performing the reaction with *p*-anisidine 30 mol% and 5-nitrosalicylic acid 60 mol% as catalyst combination. All the other racemic samples were prepared by mixing the two product antipodes obtained performing the reaction with catalyst 9-amino(9-deoxy)*epi*-quinine **A** and its *pseudo*-enantiomer 9-amino(9-deoxy)*epi*-quinidine **ent-A** separately.

⁵(a) B.-D. Chong, Y.-I. Ji, S.-S. Oh, J.-D. Yang, W. Baik and S. Koo *J. Org. Chem.* 1997, **62**, 9323; (b) Y. Ergün, N. Bayraktar, S. Patir and G. Okay, *J. Heterocyclic Chem.* 2000, **37**, 11.

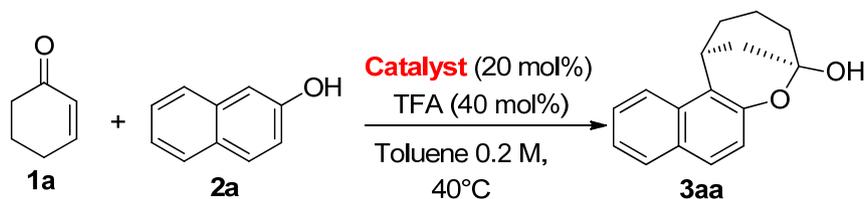
⁶ a) L. Minuti, A. Taticchi, E. Gacs-Baitz and A. Marrocchi, *Tetrahedron*, 1995, **51**, 8953; b) E. Zimmerman and V. Suryanarayan, *Eur. J. Org. Chem.* 2007, 4091.

⁷ S. Kumar, D. Hernandez, B. Hoa, Y. Lee, J.-S. Yang and A. McCurdy, *Org. Lett.*, 2008, **10**, 3761.

⁸ Bachir Latli, *J. Label Compd. Radiopharm.* 2004; **47**, 847.

Optimization data for ketone 1a and β -naphthol 2a

Table 1. Catalyst screening



Entry	1a:2a	Cat. (mol%)	Acid (mol%)	t (h)	Yield %	ee %
1	1.1:1	A (20)	TFA (40)	96	87	49
2	1.1:1	B (20)	TFA (40)	48	12.5	45
3	1.1:1	C (20)	TFA (40)	96	89	45
4	1.1:1	-	-	48	0	-
5	1.1:1	-	TFA (40)	48	0	-
	1.1:1	<i>ent</i> -A (20)	-	48	-	-
7	1.1:1	<i>ent</i> -A (20)	TFA (40)	96	42	45
8	1.1:1	<i>ent</i> -C (20)	TFA (40)	48	60	45
9	1.1:1	<i>ent</i> -A (20)	TFA (60%)	96	96	33
10	3:1	A (20)	TFA (40)	96	63	53

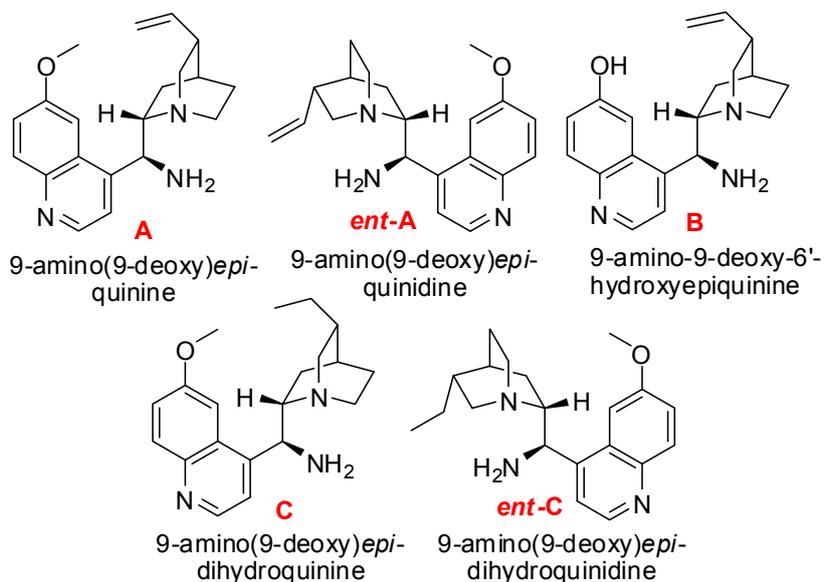
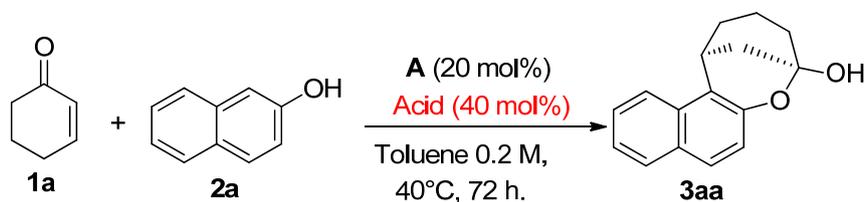
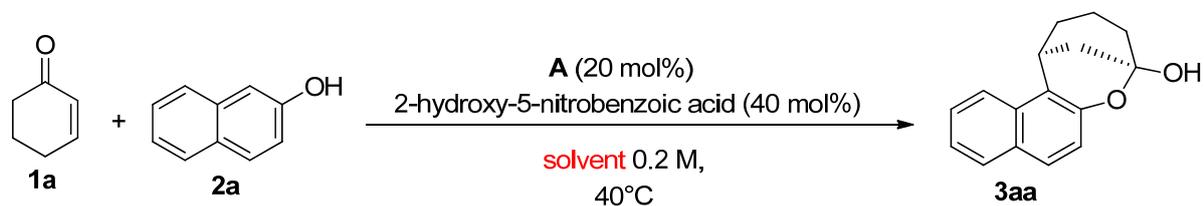


Table 2. Acid screening



Entry	Acid	Yield (%)	ee (%)
1	benzoic acid	29	32
2	<i>p</i> -nitrobenzoic acid	58	71
3	3,5-dinitrobenzoic acid	28	56
4	<i>o</i> -nitrobenzoic acid	82	74
5	<i>o</i> -hydroxybenzoic acid	45	74
6	<i>o</i> -fluorobenzoic acid	61	57
7	2-hydroxy-5-nitrobenzoic acid	78	82
8	1-hydroxy-2-naphtoic acid	81	76
9	2-hydroxy-3-nitrobenzoic acid	61	73
10	2-hydroxy-1-naphtoic acid	58	78
11	diphenyl hydrogen phosphate	43	19
12	(<i>R</i>)-BINOL-hydrogenphosphate	12	n.d.
13	<i>p</i> -TsOH	0	-
14	2-mercaptobenzoic acid	57	53
15	Anthranilic acid	63	0

Table 3. Solvent screening



Entry	Solvent	t (d)	Yield (%)	ee (%)
1	Toluene	3	78	82
2	dry Toluene	3	82	84
3	Water	3	43	46
4	Et ₂ O	3	6	n.d.
5	MeOH	3	0	-
6	EtOAc	3	16	n.d.
7	THF	3	0	-
8	CHCl ₃	2	52	80
9	Hexane	2	18	52
10	dioxane	2	12	n.d.
11	MTBE	2	5	n.d.
12	HFIP	2	0	-
13	Fluorobenzene	2	46	80
14	DCM	3	47	80
15	p-Xylene	3	43	68
16	Clorobenzene	3	89	79
17	1,2-DCE	3	24	76
18	Toluene/Brine 1:1	3	83	73

Conformational analysis and absolute configuration determination.

Compounds 10

Good crystals suitable for X-ray diffraction were obtained for compound **10de** by slow evaporation of a methanol solution. The anomalous scattering determination of the absolute configuration was possible thanks to the presence of the chlorine atom. The *S* configuration was determined for the selected crystal, and its relationship to the major enantiomer obtained with ent-A catalyst, was confirmed by means of enantioselective HPLC analysis of the very same crystal used for X-ray analysis (this was not straightforward, since the crystals were obtained from a 81% ee mixture of enantiomers). The crystal cell contained two conformations of the *S* enantiomers, that were different in the orientation of the OMe group on the naphthalene ring (see below for refinement details).

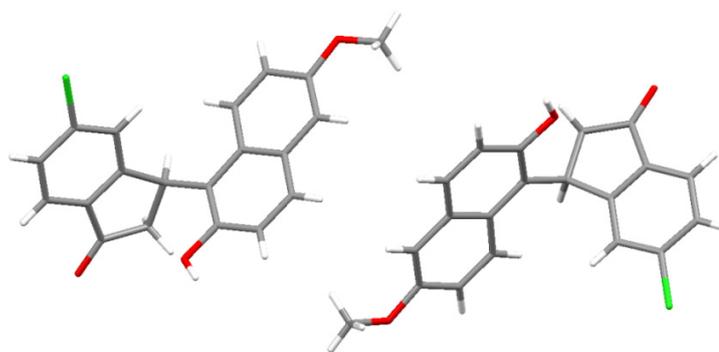


Figure S1: X-Ray structure of **10de**. Two different conformations with the same *S* absolute configuration represent the asymmetric unity.

Since the prepared compounds belong to four different classes, a straight relationship of the stereochemical course of the reaction is likely, but in principle it could not be safely assumed. However, despite many attempts, enantiopure crystals of other compounds containing a suitable heavy atom could not be obtained. In the remaining cases, the X-ray analysis could not determine the absolute configuration. For these reason we switched to a different approach based on conformational analysis and chiroptical methods.

The determination of the absolute configurations (AC) of chiral molecules using chiroptical techniques like optical rotation (OR), electronic circular dichroism (ECD), and vibrational circular dichroism (VCD) has gained feasibility and reliability because of the development of reliable methods for the prediction of these properties based on density functional theory (DFT) and on its Time-Dependent formalism (TD-DFT).⁹ In the present case the theoretical calculation of ECD spectra was

⁹ For recent examples: H. Hussain, K. Krohn, I. Ahmed, S. Draeger, B. Schulz, S. Di Pietro and G. Pescitelli *Eur. J. Org. Chem.* 2012, 1783; X-F Hou, S. Yao, A. Mándi, T. Kurtán, C-P. Tang, C-Q. Ke, X-Q. Li and Y. Ye. *Org. Lett.* 2012, **14**, 460; Y-S. Cai, T. Kurtán, Z-H. Miao, A. Mándi, I. Komáromi, H-L. Liu, J. Ding, and Y-W Guo *J. Org. Chem.* 2011, **76**, 1821; M, Woźnica, A. Butkiewicz, A. Grzywacz, P. Kowalska, M. Masnyk, K. Michalak, R. Luboradzki, F. Furche, H. Kruse, S. Grimme and J. Frelek.

selected for the absolute configuration assignment. From a conformational point of view, the rigidity of the scaffold of compounds **3**, **5**, **10** and **11** reduces the number of conformations to be considered and thus simplify the conformational analysis.¹⁰ In addition to this, the configuration assignment by chiroptical methods of a sample compound of the **10** series can provide information on the reliability of this method applied to the present compounds.

A preliminary conformational search on **10aa** was carried out using Monte Carlo method together with the MMFF94 molecular mechanics force field (as implemented in Titan 1.0.5, Wavefunction inc.). All the conformations within a 10 kcal/mol window were then optimized using DFT at the B3LYP/6-31G(d) level^{10,11}, the harmonic vibrational frequencies of each conformation were calculated at the same level to confirm their stability (no imaginary frequencies were observed) and to evaluate the free energy of each conformation.

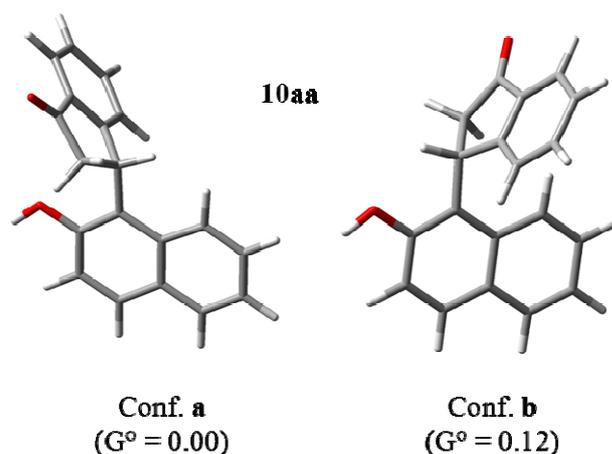


Figure S2: 3D view of the two most stable conformations of **10aa**, calculated at the B3LYP/6-31G(d) level. Energy differences are in kcal/mol and represent ZPE-corrected free energies in standard conditions.

After DFT minimization only two conformations were found to be enclosed in the 10 kcal/mol

J. Org. Chem. 2011, **76**, 3306. For reviews see: G. Bringmann, T. Bruhn, K. Maksimenka and Y. Hemberger. *Eur. J. Org. Chem.* 2009, 2717. T.D. Crawford; M.C. Tam and M.L. Abrams, *J. Chem. Phys. A* 2007, **111**, 12057. For a review on conformational analysis for the AC determination see: A. Mazzanti and D. Casarini, *D. WIREs Comput. Mol. Sci.* 2012, **2**, 613.

¹⁰ P.L. Polavarapu; E.A. Donahue; G. Shanmugam; G. Scalmani; E.K. Hawkins; C. Rizzo; I. Ibrusaud; G. Thomas; D. Habel and D. Sebastian; *J Phys Chem A* 2011, **115**, 5665.

¹¹ Program Gaussian 09, rev A.02. M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, Ö. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

window (Figure S2). These correspond to two conformational diastereoisomers due to the rotation on the indane ring that is almost perpendicular to the naphthol ring in the ground states. The electronic excitation energies and rotational strengths have been calculated in the gas phase for the two conformations of **10aa** using TD-DFT with four different methods (functionals) to ascertain if different calculations provide different shapes of the simulated spectra. The simulation were performed with the hybrid functionals BH&HLYP¹² and M06-2X,¹³ the Long-range Correlated LC- ω B97XD that includes empirical dispersion,¹⁴ and CAM-B3LYP that includes long range correction using the Coulomb Attenuating Method.¹⁵ All the calculations employed the 6-311++G(2d,p) basis set, that proved to be sufficiently accurate at a reasonable computational cost.¹⁶

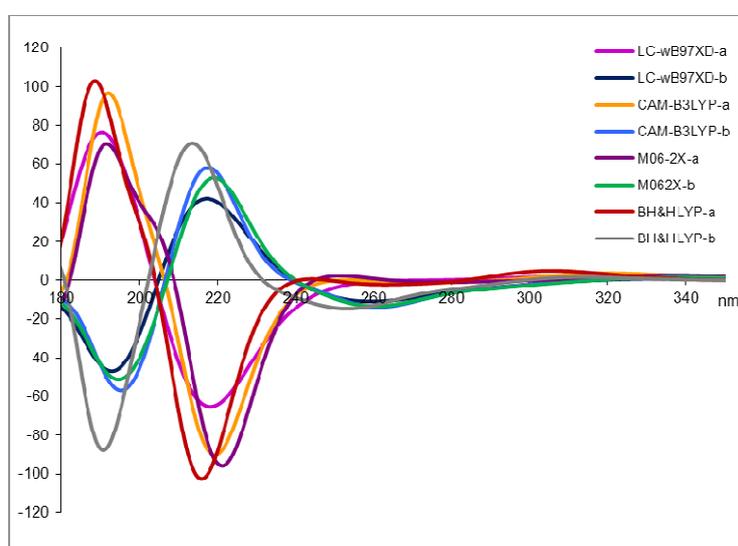


Figure S3: ECD simulations for the two conformations of **10aa**, obtained with the four different functionals and the same 6-311++G(2d,p) basis set.

The rotational strengths were calculated in both length and velocity representation with the resulting values being very similar. For this reason the errors due to basis set incompleteness should be considered very small, or negligible.¹⁷ All the calculations were performed supposing *S* Absolute Configuration, with the results shown in Figure S3: ECD simulations for the two conformations of **10aa**, obtained with the four different functionals and the same 6-311++G(2d,p) basis set. The eight simulated spectra are divided into two set of opposite shaped spectra (reddish lines and

¹² In Gaussian 09 the BH&HLYP functional has the form: $0.5 * E_x^{HF} + 0.5 * E_x^{LSDA} + 0.5 * \Delta E_x^{Becke88} + E_C^{LYP}$

¹³ Y. Zhao and D.G. Truhlar, *Theor. Chem. Acc.* 2008, **120**, 215.

¹⁴ J-D. Chai and M. Head-Gordon. *Phys. Chem. Chem. Phys.* 2008, **10**, 6615.

¹⁵ T. Yanai; D. Tew and N. Handy. *Chem. Phys. Lett.* 2004, **393**, 51.

¹⁶ G. Cera, M. Chiarucci, A. Mazzanti, M. Mancinelli and M. Bandini. *Org. Lett.* 2012, **14**, 1350-1353. A. Mazzanti, T. Calbet, M. Font-Bardia, A. Moyano and R. Rios. *Org. Biomol. Chem.* 2012, **10**, 1645-1652. S. Duce, F. Pescioli, L. Gramigna, L. Bernardi, A. Mazzanti and G. Bencivenni. *Adv. Synth. Cat.* 2011, **353**, 860.

¹⁷ P.J. Stephens, D.M. McCann, F.J. Devlin, J.R. Cheeseman and M.J. Frisch *J. Am. Chem. Soc.* 2004, **126**, 7514.

bluish/greenish lines), each one corresponding to one of the two conformations of **10aa**. The opposite pattern can be attributed to the excitonic coupling¹⁸ to the two chromophores, i.e. the naphthyl and the indane ring, that have opposite helicity in the two conformations (*R*M** in conformation **a** and *R*P** in conformation **b**). Therefore the shape of the simulated spectrum that should be compared with the experimental one strongly depends on the population ratio employed. Unfortunately, the calculated energies of the two conformations are very similar ($\Delta G^\circ = 0.12$ kcal/mol), thus a correct evaluation of the populations¹⁹ is completely unreliable. This implies that the AC assignment is unfeasible without further experimental data.

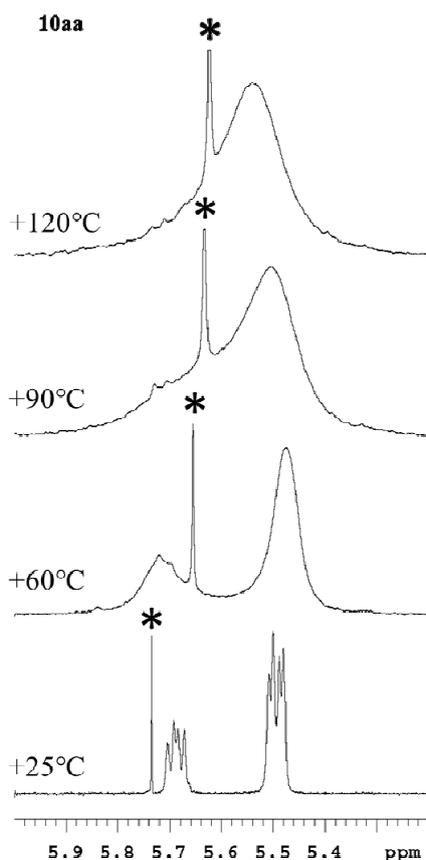


Figure S4: variable temperature spectra (¹H at 400 MHz in DMSO-d₆) of compound **10aa**. The signal of the CH of the indanone ring is showed. The asterisk marks a solvent impurity.

However, the conformational analysis of **10aa** showed that the indane ring is forced to be perpendicular to the naphthol ring by the steric hindrance exerted by the OH and the *peri* hydrogen (H-

¹⁸ a) N. Harada and K. Nakanishi, *Acc. Chem. Res.* 1972, **5**, 257; (b) N. Harada and K. Nakanishi, *Circular Dichroic Spectroscopy: Exciton Coupling in Organic Stereochemistry*; University Science Books: Mill Valley, CA, 1983; (c) K. Nakanishi and N. Berova, In *Circular Dichroism: Principle and Applications*; Berova, N.; Nakanishi, K.; Woody, R.W. Eds; VCH: New York, 2000; Chapter 12, p 337.

¹⁹ G. Mazzeo, E. Giorgio, R. Zanasi, N. Berova and C. Rosini *J. Org. Chem.* 2010, **75**, 4600.

8) If the interconversion barrier is sufficiently high, two observable conformational diastereoisomers could be generated. This is what actually happened. ^1H NMR showed two sets of signals in a 69:31 ratio due to the frozen rotation around the $\text{C}_\alpha\text{-CH}$ bond. When the temperature is raised the two multiplets broadens and show a single peak at $+120^\circ\text{C}$, when the rotation is fast in the NMR time scale, (Figure S4). An energy barrier of 17.8 kcal/mol was derived at the coalescence temperature ($+90^\circ\text{C}$).

NOE spectra were then acquired to determine which conformational diastereoisomer is the more populated. In the **a** diastereoisomer the CH hydrogen of indane is close to the *peri* H-8 hydrogen of the naphthol ring (1.87 Å), whereas in the **b** conformation the CH points towards the OH and it is far from H-8. DPGSE-NOE²⁰ spectra obtained on saturation of the two CH signals showed that the major conformation has the CH close to H-8 and the minor has the CH close to OH (Figure S5: DPGSE-NOE spectra of **10aa** (600 MHz in DMSO- d_6). Bottom: control spectrum. Middle trace. NOE obtained on saturation of the major CH. Top: NOE spectrum obtained on saturation of the minor CH. (in both NOEs the small inverted peak of the second diastereoisomer was due to saturation transfer effects)).

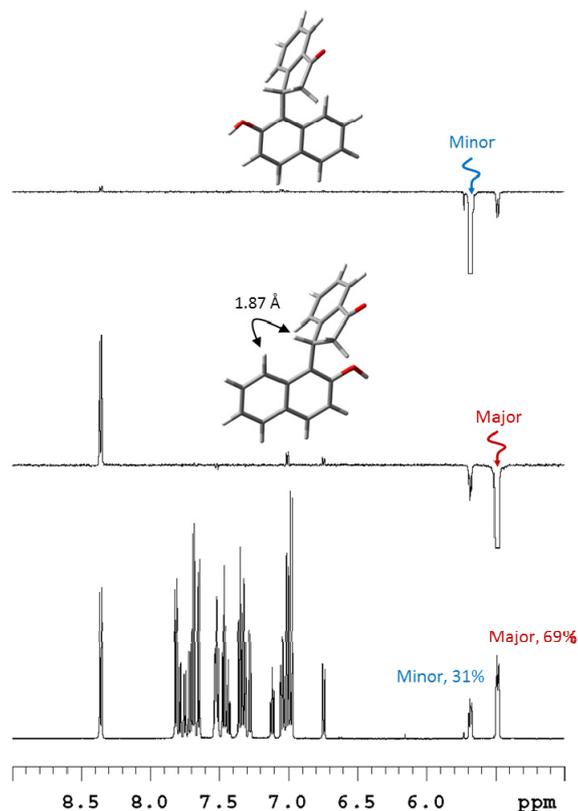


Figure S5: DPGSE-NOE spectra of **10aa** (600 MHz in DMSO- d_6). Bottom: control spectrum. Middle trace. NOE obtained on saturation of the major CH. Top: NOE spectrum obtained on saturation of the

²⁰ (a) K. Stott, J. Stonehouse, J. Keeler, T. L. Hwand and A. J. Shaka, *J. Am. Chem. Soc.* 1995, **117**, 4199; (b) K. Stott, J. Keeler, Q. N. Van and A. J. Shaka, *J. Magn. Resonance* 1997, **125**, 302. See also: T. D. W. Claridge, *High Resolution NMR Techniques in Organic Chemistry*; Pergamon: Amsterdam, 1999.

minor CH. (in both NOEs the small inverted peak of the second diastereoisomer was due to saturation transfer effects).

In the present case the integration of the NMR spectra and the NOE spectra provide the exact ratio of the two conformational diastereoisomers to be used in the simulation of the experimental ECD spectrum. The simulated ECD spectrum was thus obtained for each model of calculation by taking into account the 69:31 populations ratio experimentally determined by NMR. All the four simulations obtained (Figure S6. Simulations of the experimental ECD spectrum of **10aa** (black traces) obtained with different methods of calculation (functionals). Each simulated spectrum (red, blue, green and purple lines) was obtained starting from the spectra obtained for the two conformations weighted using the experimental NMR data. The experimental spectrum of **10aa** was obtained in acetonitrile solution (1 10^{-4} M, 0.2 cm path length). $\Delta\epsilon$ are expressed in Mol L⁻¹ cm⁻¹. The simulated spectra were vertically scaled to match the experimental intensity, and red shifted to match the experimental peak at 238 nm.) display now a good agreement with the experimental spectrum and the best simulation was obtained with the BH&HLYP functional. Provided the experimental ratio of the two conformations,²¹ the ECD simulations showed to be able to tackle the absolute configuration of **10aa**.

²¹ D. Casarini, L. Lunazzi, M. Mancinelli, A. Mazzanti and P. Scafato *Chirality* 2009, **21**, 16.

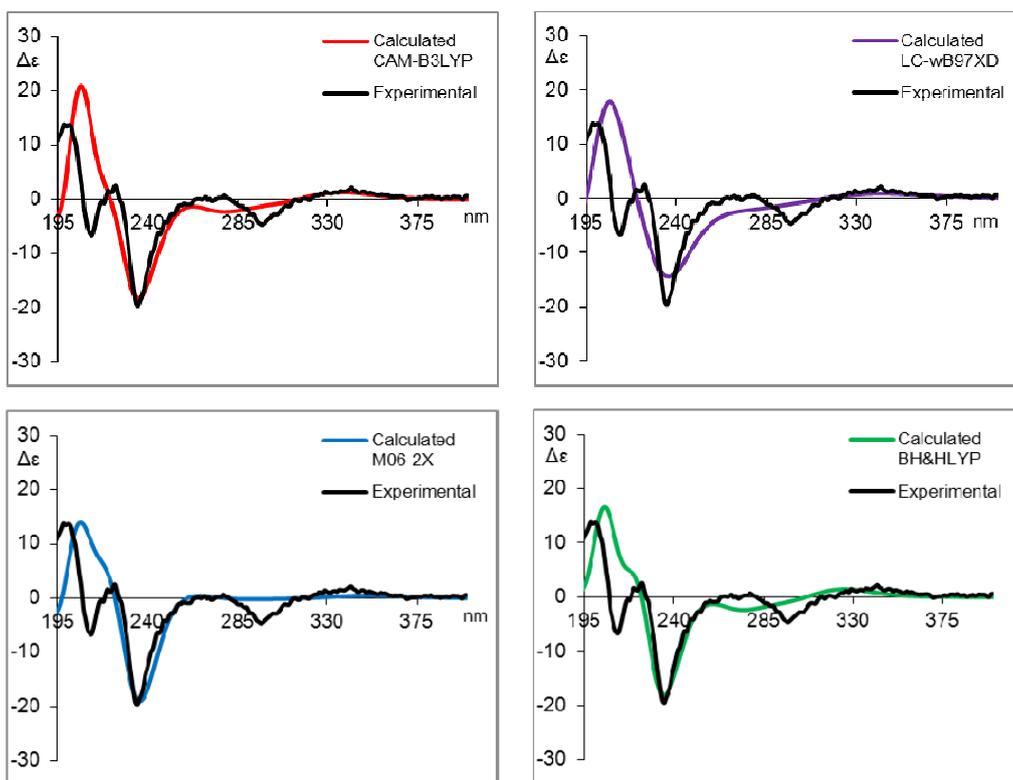


Figure S6. Simulations of the experimental ECD spectrum of **10aa** (black traces) obtained with different methods of calculation (functionals). Each simulated spectrum (red, blue, green and purple lines) was obtained starting from the spectra obtained for the two conformations weighted using the experimental NMR data. The experimental spectrum of **10aa** was obtained in acetonitrile solution (1×10^{-4} M, 0.2 cm path length). $\Delta\epsilon$ are expressed in $\text{Mol L}^{-1} \text{cm}^{-1}$. The simulated spectra were vertically scaled to match the experimental intensity, and red shifted to match the experimental peak at 238 nm.

Compounds 11

No compound of the **11** series could be crystallized as single crystals and the assignment of **11bb** was performed by means of same chiroptical methods used for **10aa**.

As Before, also in the case of **11bb** the two conformations found by conformational analysis and subsequent DFT optimizations have the CH pointing towards the OH (**a** conformation) and the CH in position 3 (**b** conformation), respectively. In this case, however, the steric hindrance to the rotation of the indane ring is smaller and the ^1H NMR spectrum recorded at room temperature does not show the signals corresponding to the two conformational diastereoisomers. However, since calculations estimated a very similar energy they should be both populated. For this reason, variable temperature NMR spectra were recorded down to -100°C . Below -60°C the signal of the CH broadened and split at -100°C into two signals with a 90:10 ratio. Following the trend observed for **10aa**, the lower field signal

(90%) can be attributed to the conformation in which the CH hydrogen is close to OH. Comfortably, this is also the lowest energy conformation, and also the evaluation of the energy difference matched well the experimental ratio (calculated ΔG^\ddagger : 0.56 kcal/mol; experimental: 0.75 kcal/mol at -100°C). By applying Boltzmann statistics, the ratio at room temperature is 78:22.

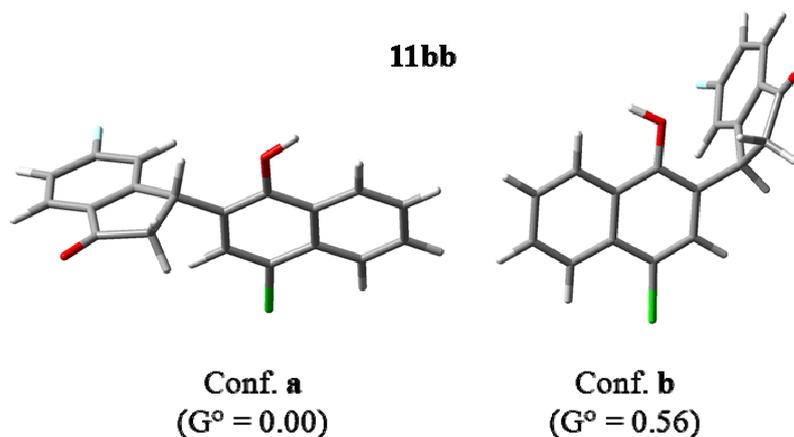


Figure S7: 3D view of the two stable conformations of **11bb**, calculated at the B3LYP/6-31G(d) level. Energy differences are in kcal/mol and represent ZPE-corrected free energies in standard conditions.

The rotational strengths and electronic excitation energies were calculated in the gas phase for the two conformations of **11bb** using TD-DFT and the same four different methods used for **10aa**. All the calculations employed the 6-311++G(2d,p) basis set and supposing *S* Absolute Configuration (Figure S8. TD-DFT simulations for the two conformations of 11bb, obtained with the four functionals and the 6-311++G(2d,p) basis set.).

As for **10aa**, the opposite helicity generated by the out-of-plane disposition of the indane ring yields two set of calculated ECD spectra with opposite pattern. However, when the final simulated spectra are obtained using the population ratio determined by low-temperature NMR, the agreement with the experimental trace is very good (Figure S9), and the *S* absolute configuration can be assigned to compound **11bb**. In addition to this, the agreement of the four methods and the similarity of the spectra enhances the reliability of the assignment.

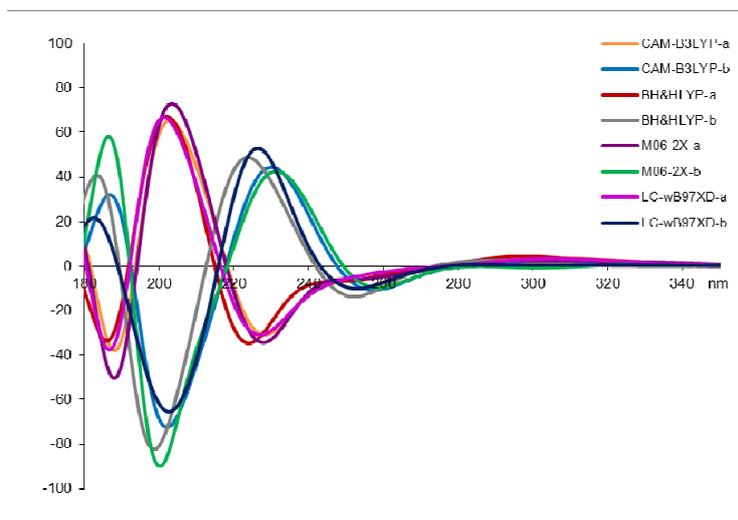


Figure S8. TD-DFT simulations for the two conformations of **11bb**, obtained with the four functionals and the 6-311++G(2d,p) basis set.

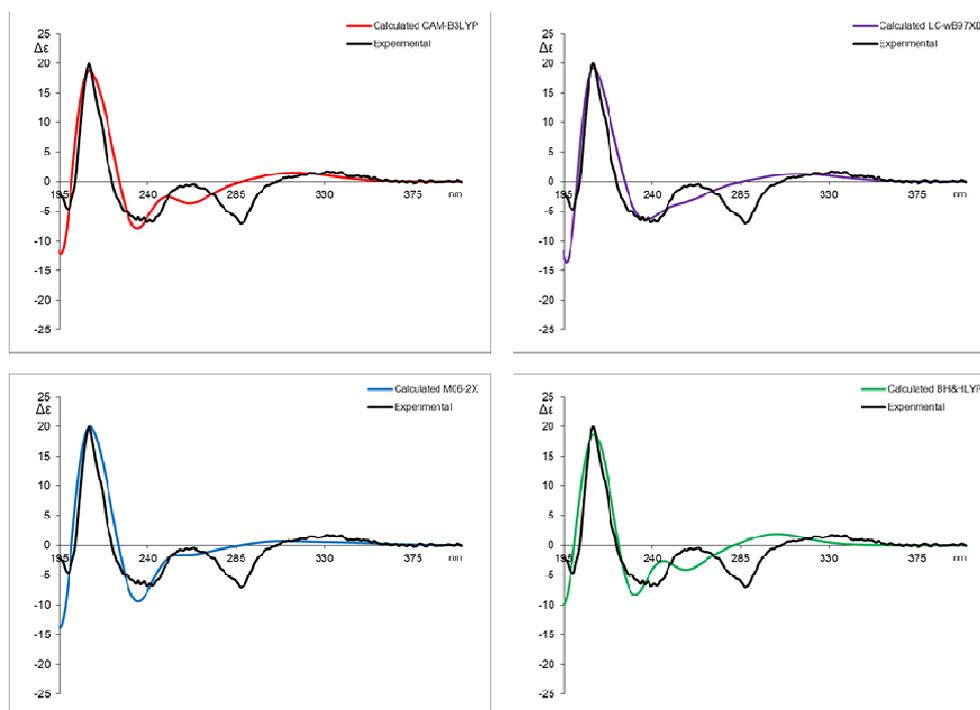


Figure S9. Simulations of the experimental ECD spectrum of **11bb** (black traces) obtained with different methods of calculation. Each simulated spectrum (red, blue, green and purple lines) was obtained starting from the spectra obtained for the four conformations weighted using Boltzmann statistics at +25°C. The experimental spectrum was obtained in acetonitrile solution (1×10^{-4} M, 0.2 cm path length). $\Delta\epsilon$ are expressed in $\text{Mol L}^{-1} \text{cm}^{-1}$. The simulated spectra were vertically scaled to match the experimental intensity, and red shifted to match the experimental maxima.

Compounds 5

As reported in the text, compounds **5** correspond to the hemiacetalic form. From a conformational point of view, these structures are completely blocked, and only one conformation is supposed to be populated. This corresponds to the chair conformation of the cyclohexane part of the bicyclic system. Compound **5da**, containing a phenyl group in position 4 was selected for the spectroscopic analysis because the presence of a second chromophore that could couple with the naphthalene ring to cause a stronger ECD spectrum.

The analysis of the ^1H spectrum of compound **5da** showed that the phenyl ring in position **4** occupies the equatorial position. The signal of benzylic CH shows a trans-diaxial coupling constant of 12.8 Hz and an axial-equatorial constant of 4.6 Hz. The $2R^*,4S^*,6S^*$ relative configuration can be therefore assumed in the following discussions (Figure S10. Left: 3D view of the optimized conformation of **5da**, calculated at the B3LYP/6-31G(d) level.). It is worth to note that the second diastereoisomer produced by the reaction (**6da**) does not evolve to the hemiacetalic form because in this compound the phenyl ring should occupy the axial position. Although the absolute configuration of **6da** could not be established by anomalous scattering, the X-ray structure confirmed that the two chiral carbons have opposite chirality (thus $3R^*,5S^*$).

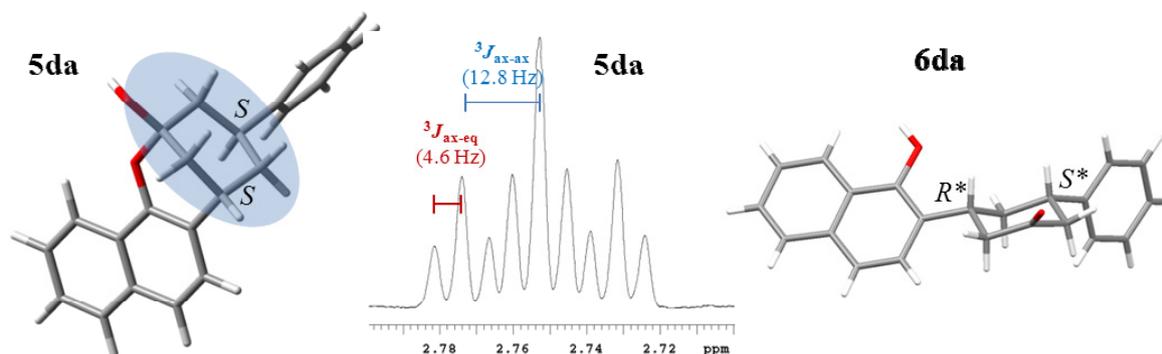


Figure S10. Left: 3D view of the optimized conformation of **5da**, calculated at the B3LYP/6-31G(d) level. The chair-shaped cyclohexane ring is shaped. Center: ^1H multiplet of the CH in position 4 showing the trans-diaxial and axial-equatorial coupling constants. Right: X-ray structure of **6da**.

To exclude the presence of other low-energy conformations, the conformational search by MM methods was performed as above, but no other conformations were found. As in the previous cases, DFT optimization at the B3LYP/6-31G(d) level provided the final geometry to be used in the TD-DFT simulations. The rotational strengths and electronic excitation energies were calculated in the gas phase by TD-DFT and the same four different methods used for **10aa** and **11bb**. All the calculations employed the 6-311++G(2d,p) basis set and supposed *S* Absolute Configuration at the reaction center. (thus $2R, 4S, 6S$) (Figure S11). In this case the simulation of the experimental spectrum is straightforward, since no conformational averaging is needed and the four calculated spectra are very

similar. As shown in Figure S11, the experimental trace is well matched by all the simulation obtained for the 2*R*, 4*S*, 6*S* absolute configuration.

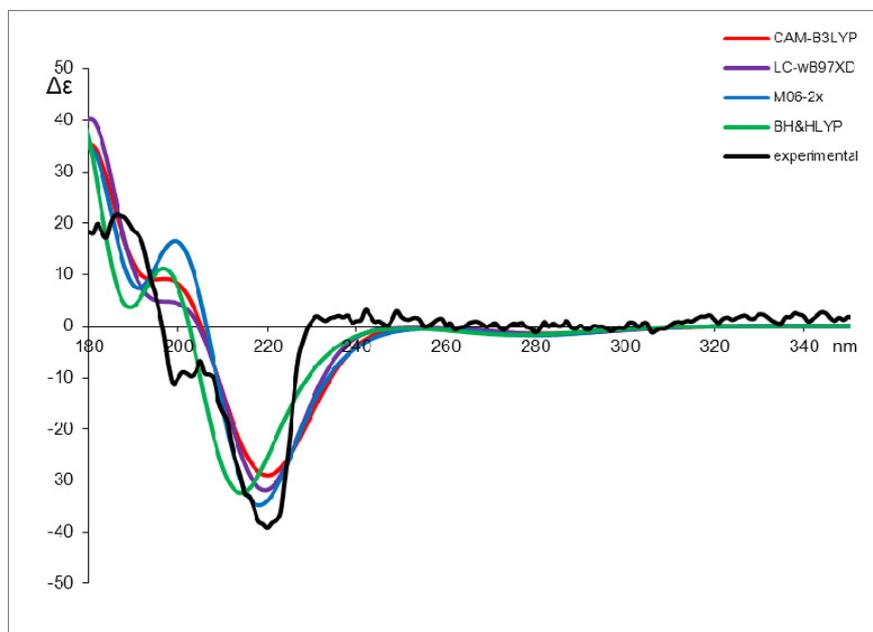


Figure S11. TD-DFT simulations for **5da**, obtained with the four different functionals and the 6-311++G(2d,p) basis set. The black line corresponds to the experimental spectrum obtained in acetonitrile solution (1×10^{-4} M, 0.05 cm path length). $\Delta\epsilon$ are expressed in $\text{Mol L}^{-1} \text{cm}^{-1}$. The simulated spectra were vertically scaled to match the experimental intensity, and red shifted to match the experimental maxima.

Compounds 3

Compounds **3** has the same hemiacetalic scaffold of compounds **5**, thus a second chiral carbon is generated with complete stereocontrol during the cyclization. With respect to the cyclohexane scaffold the two substituents must occupy the 1-3 diaxial positions (Figure S12). Compound **3ab** was selected for the stereochemical assignment.

As in the case of **5**, the bicyclic structure is very rigid, and only a single conformation was found by MM search. In this case, however, a second conformation with a boat-shaped cyclohexane could not be excluded in principle. When optimized by DFT, this conformation showed to be about 5 kcal/mol higher in energy with respect to the conformation found by conformational search, and it can be neglected in the ECD simulations. Compound **3ab** lacks a second chromophore on the cyclohexane ring and the experimental ECD is rather weak. However, the TD-DFT simulations performed assuming the 1*S*, 5*R* absolute configuration satisfactorily followed the experimental trend (Figure S12). It should be pointed out that the same *S* configuration at the reaction center was assigned as in the three previous cases.

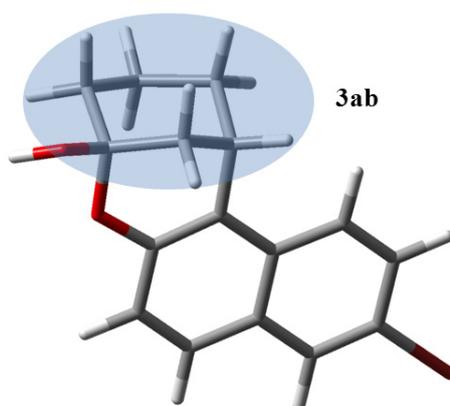


Figure S12 3D view of the optimized conformation of **3ab**, calculated at the B3LYP/6-31G(d) level.

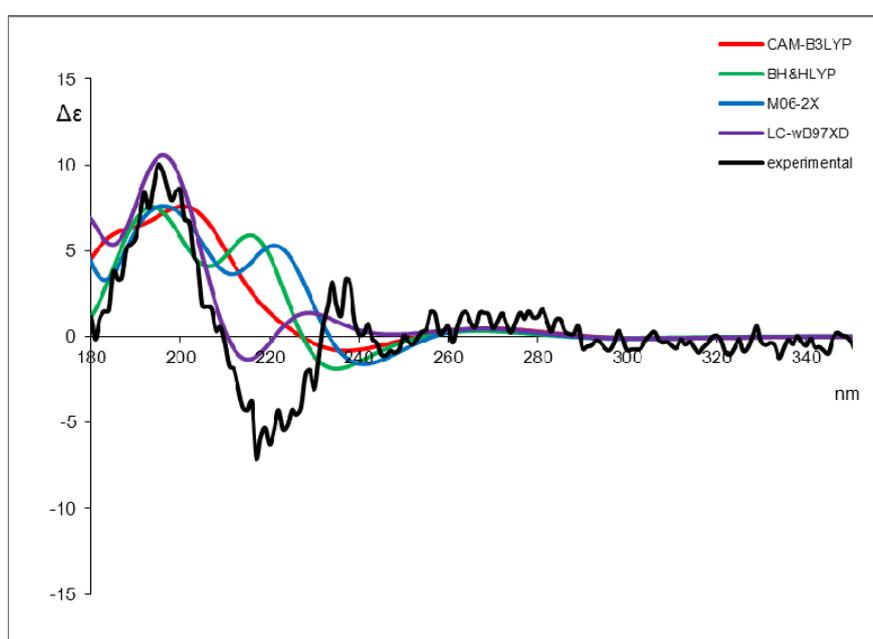
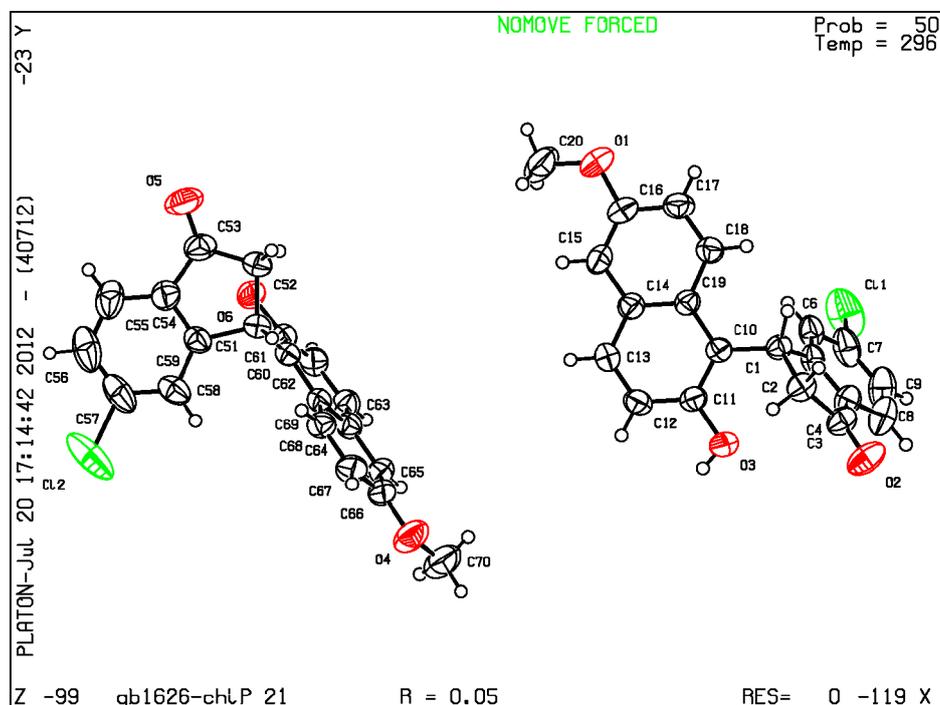


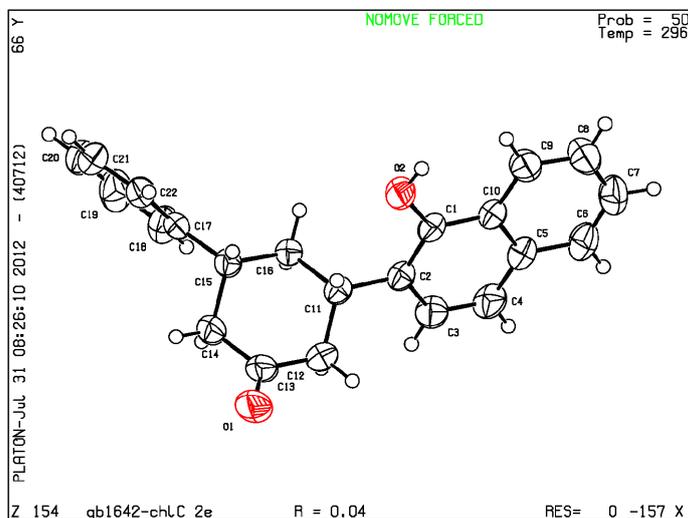
Figure S13. TD-DFT simulations for **3ab**, obtained with the four different functionals and the same 6-311++G(2d,p) basis set. The black line corresponds to the experimental spectrum obtained in acetonitrile solution (5×10^{-5} M, 0.2 cm path length). $\Delta\epsilon$ are expressed in $\text{Mol L}^{-1} \text{cm}^{-1}$. The simulated spectra were vertically scaled to match the experimental intensity, and red shifted to match the experimental maxima

Crystal data for 10de



Molecular formula: $C_{20}H_{15}ClO_4$, MW 338.77. Monoclinic, space group $P2_1$, $a = 8.6325(13)$, $b = 8.6693(13)$, $c = 22.017(3)$, $\beta = 92.146(2)$. $V = 1646.6(4) \text{ \AA}^3$, $T = 298(2) \text{ }^\circ\text{K}$, $Z = 4$, $\rho_c = 1.367 \text{ g cm}^{-3}$, $F(000) = 2762$, graphite-monochromated $Mo_{K\alpha}$ radiation ($\lambda = 0.71073 \text{ \AA}$), $\mu(Mo_{K\alpha}) = 0.247 \text{ mm}^{-1}$, colorless sticks ($0.40 \times 0.15 \times 0.10 \text{ mm}^3$), empirical absorption correction with SADABS (transmission factors: $0.9078 - 0.9758$), 2400 frames, exposure time 20 s, $1.85 \leq \theta \leq 27.40$, $-11 \leq h \leq 11$, $-11 \leq k \leq 11$, $-28 \leq l \leq 28$, 7373 reflections collected, 5444 independent ($R_{int} = 0.0277$), solution by direct methods (SHELXS97) and subsequent Fourier syntheses, full-matrix least-squares on F_o^2 (SHELX97), hydrogen atoms refined with a riding model except for the hydroxyl hydrogen that was experimentally located; data / restraints / parameters = 7373/ 1 / 441, $S(F^2) = 1.044$, $R(F) = 0.0634$ and $wR(F^2) = 0.1258$ on all data, $R(F) = 0.0454$ and $wR(F^2) = 0.1124$ for 5444 reflections with $F_o > 4\sigma(F_o)$, weighting scheme $w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.0000P]$ where $P = (F_o^2 + 2F_c^2)/3$, largest difference peak and hole 0.186 and $-0.293 \text{ e \AA}^{-3}$. Flack parameter: $0.02(6)$. The unit cell contains two different conformation belonging to the same chirality, that are different because of the different disposition of the OMe group. CCDC-893970 CIF file contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

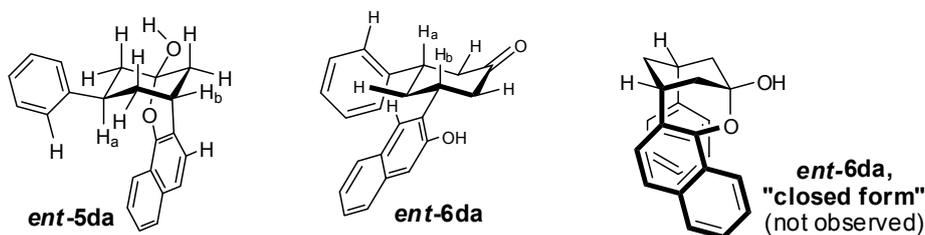
Crystal data for 6da



Molecular formula: $C_{22}H_{20}ClO_2$, MW 316.38. Monoclinic, space group $C2$, $a = 19.582(3)$, $b = 6.4957(10)$, $c = 15.898(2)$, $\beta = 123.3630(10)$. $V = 1688.9(4) \text{ \AA}^3$, $T = 298(2) \text{ }^\circ\text{K}$, $Z = 4$, $\rho_c = 1.244 \text{ g cm}^{-3}$, $F(000) = 672$, graphite-monochromated $Mo_{K\alpha}$ radiation ($\lambda = 0.71073 \text{ \AA}$), $\mu(Mo_{K\alpha}) = 0.078 \text{ mm}^{-1}$, colorless plates ($0.40 \times 0.40 \times 0.20 \text{ mm}^3$), empirical absorption correction with SADABS (transmission factors: 0.9845 – 0.9694), 2400 frames, exposure time 15 s, $1.53 \leq \theta \leq 27.49$, $-25 \leq h \leq 25$, $-8 \leq k \leq 8$, $-20 \leq l \leq 20$, 9738 reflections collected, 3846 independent ($R_{int} = 0.0201$), solution by direct methods (SHELXS97) and subsequent Fourier syntheses, full-matrix least-squares on F_o^2 (SHELX97), hydrogen atoms refined with a riding model except for the hydroxyl hydrogen that was experimentally located; data / restraints / parameters = 3846/ 1 / 221, $S(F^2) = 1.025$, $R(F) = 0.0440$ and $wR(F^2) = 0.0879$ on all data, $R(F) = 0.0360$ and $wR(F^2) = 0.827$ for 3296 reflections with $F_o > 4\sigma(F_o)$, weighting scheme $w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.3168P]$ where $P = (F_o^2 + 2F_c^2)/3$, largest difference peak and hole 0.127 and $-0.125 \text{ e \AA}^{-3}$. Flack parameter: $-0.6(11)$. CCDC-894320 CIF file contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Relative Configuration assignment of compounds *ent-5da* and *ent-6da*

The relative stereochemistry of compounds *ent-5da* and *ent-6da* was established by means of NOE experiments. It was found that in *ent-5da* the phenyl group is *trans* to the naphthalene moiety, thus adopting an equatorial position and that in compound *ent-6da* the phenyl group is *cis* to the naphthalene moiety adopting again an equatorial position. This may also explain why structure *ent-6da*, which has the *cis* relative disposition, is only obtained as the “open form”. In fact, in the corresponding “closed form” the *cis*-phenyl group would give rise to a strained all-axial structure where the two aromatic moieties will be in close contact.



Compound *ent-5da*

Irradiation of axial proton H_a generated a visible NOE effect on the two equatorial protons of the vicinal carbons of the cyclohexane ring and on the ortho-protons of the phenyl group.

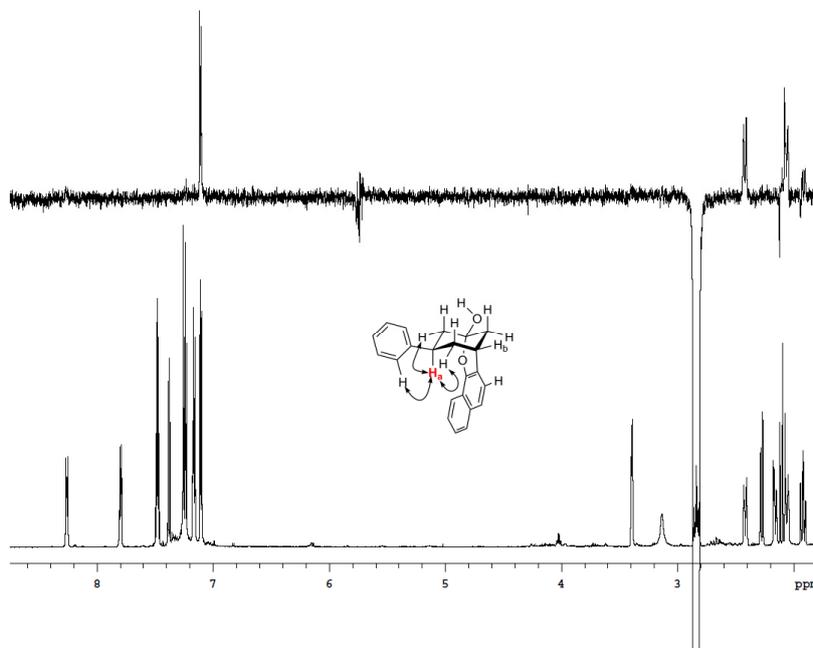


Figure S14. DPFGE-NOE spectra obtained for *ent-5da* (25°C, 600 MHz, CDCl₃) on saturation of proton H_a . Full spectrum.

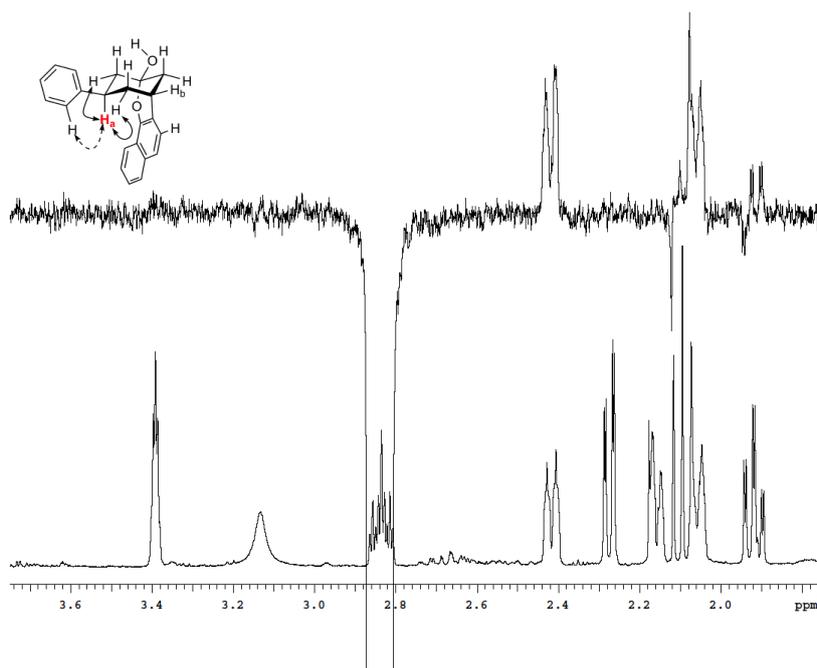


Figure S15. DPGSE-NOE spectra obtained for *ent-5da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_a . Detail of the aliphatic region.

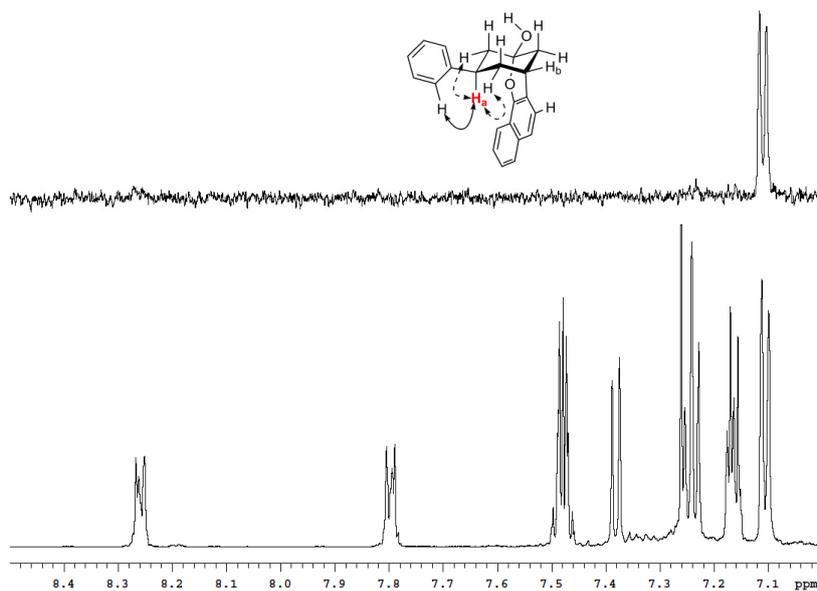


Figure S16. DPGSE-NOE spectra obtained for *ent-5da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_a . Detail of the aromatic region.

Irradiation of equatorial proton H_b produced a NOE effect on all four protons of the two adjacent methylene groups on the cyclohexane ring and on the C3H of the naphthalene ring.

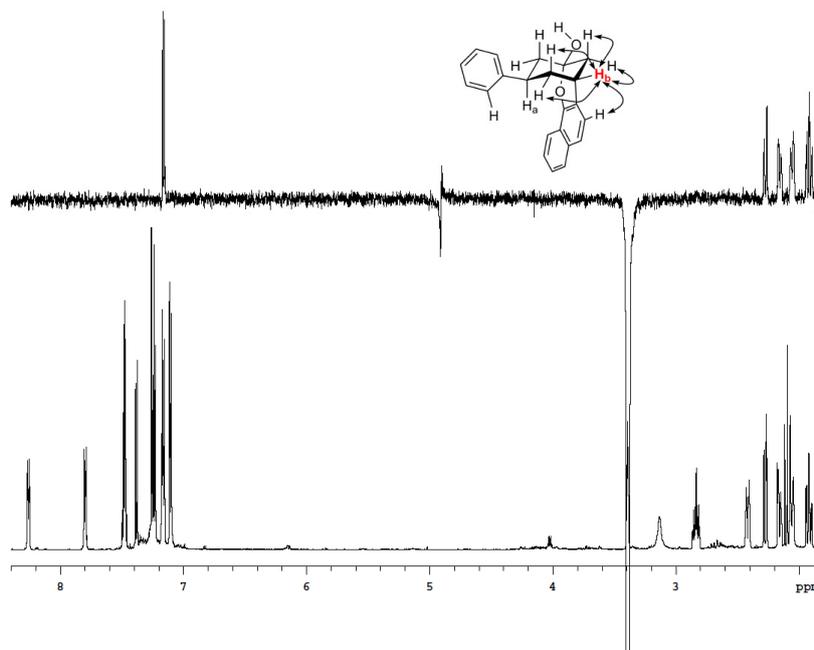


Figure S17. DPGSE-NOE spectra obtained for *ent-5da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_b .

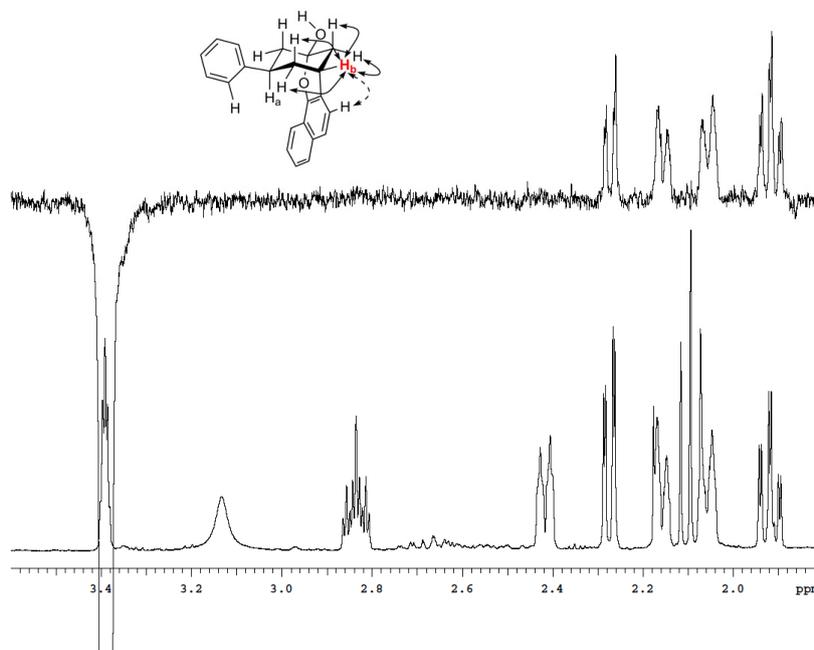


Figure S18. DPGSE-NOE spectra obtained for *ent-5da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_b . Detail of the aliphatic region.

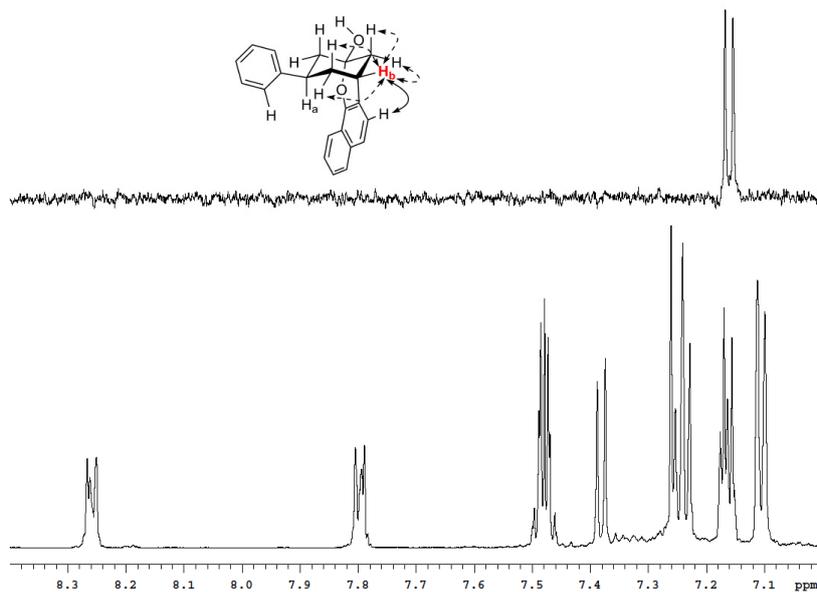


Figure S19. DPGSE-NOE spectra obtained for *ent*-5da (25°C, 600 MHz, CDCl₃) on saturation of proton H_b. Detail of the aromatic region.

Compound *ent-6da*

Irradiation of axial proton H_a at 3.13 ppm generated a strong NOE effect on H_b , thus confirming the 1,3-*cis*-diaxial relative disposition of these two protons, and additional NOE effects on the equatorial protons only of the vicinal methylene groups of the cyclohexane ring and on the ortho-protons of the phenyl group.

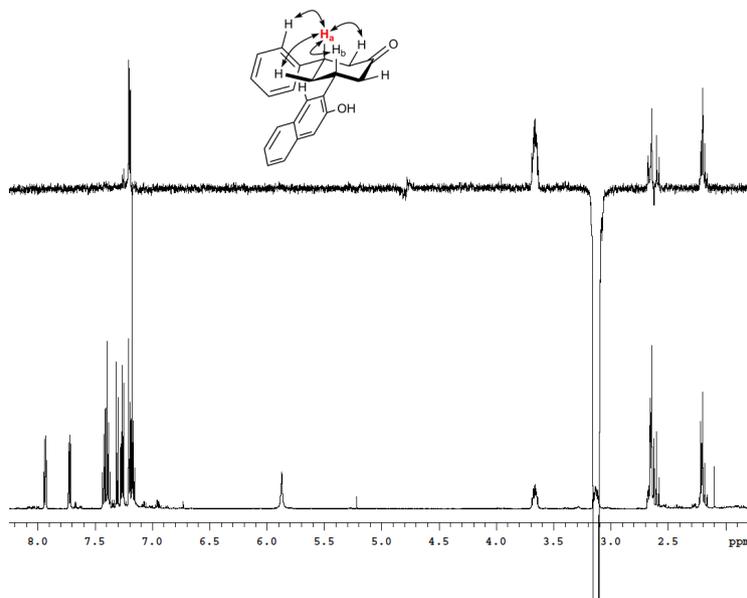


Figure S20. DPGSE-NOE spectra obtained for *ent-6da* (25°C, 600 MHz, CDCl₃) on saturation of proton H_a . Full spectrum.

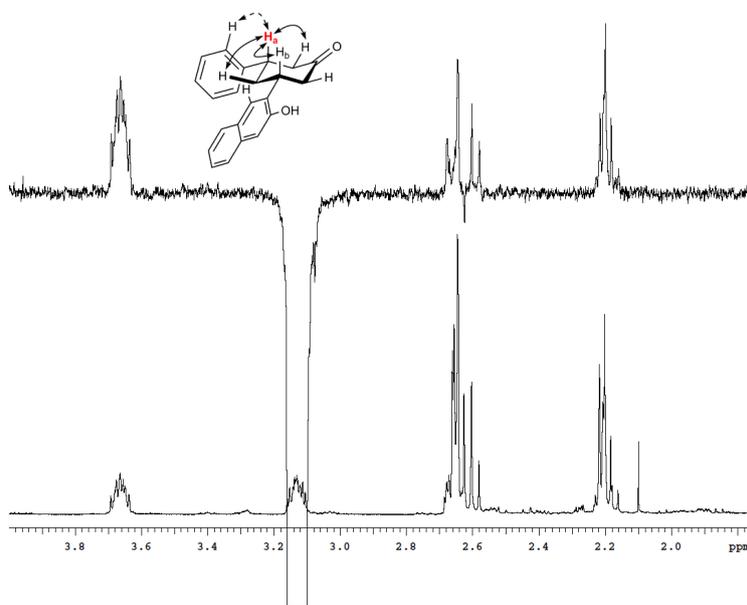


Figure S21. DPGSE-NOE spectra obtained for *ent-6da* (25°C, 600 MHz, CDCl₃) on saturation of proton H_a . Detail of the aliphatic region.

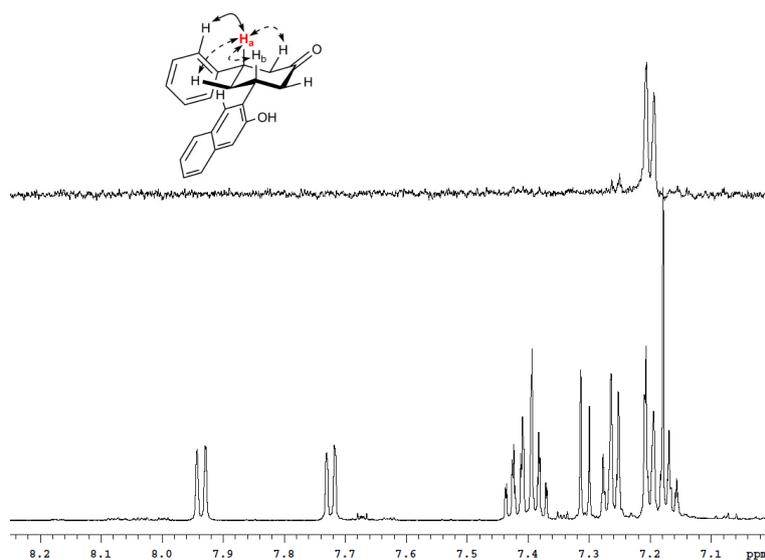


Figure S22. DPGSE-NOE spectra obtained for *ent-6da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_a . Detail of the aromatic region.

Irradiation of the axial proton H_b at 3.66 ppm produced visible NOE effect on proton H_a , on the equatorial protons of the vicinal methylene groups of the cyclohexane ring, and on OH and C3H protons of the naphthalene moiety.

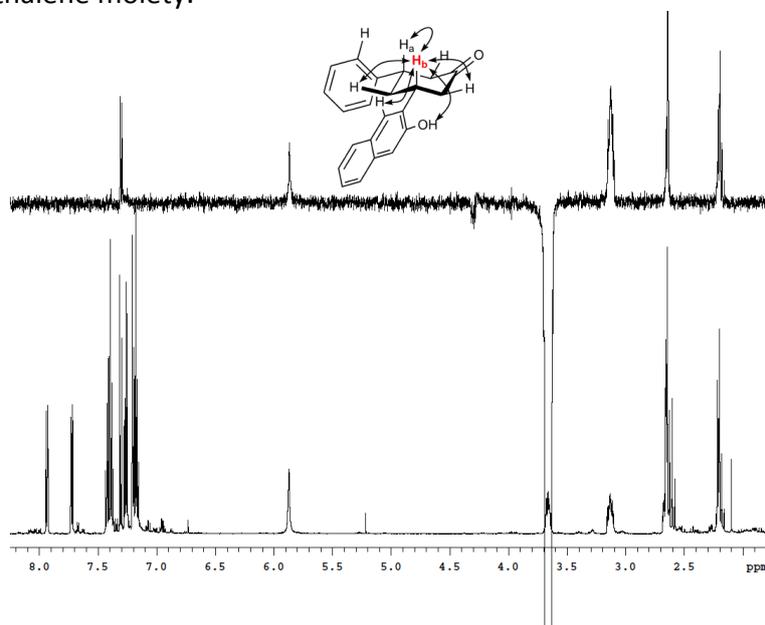


Figure S23. DPGSE-NOE spectra obtained for *ent-6da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_b . Full spectrum.

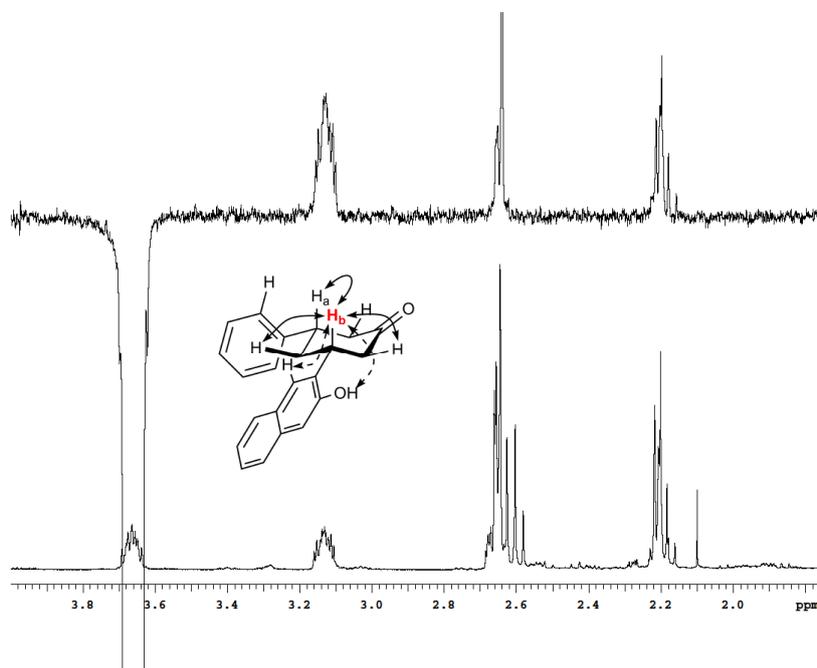


Figure S24. DPGSE-NOE spectra obtained for *ent-6da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_b . Detail of the aliphatic region.

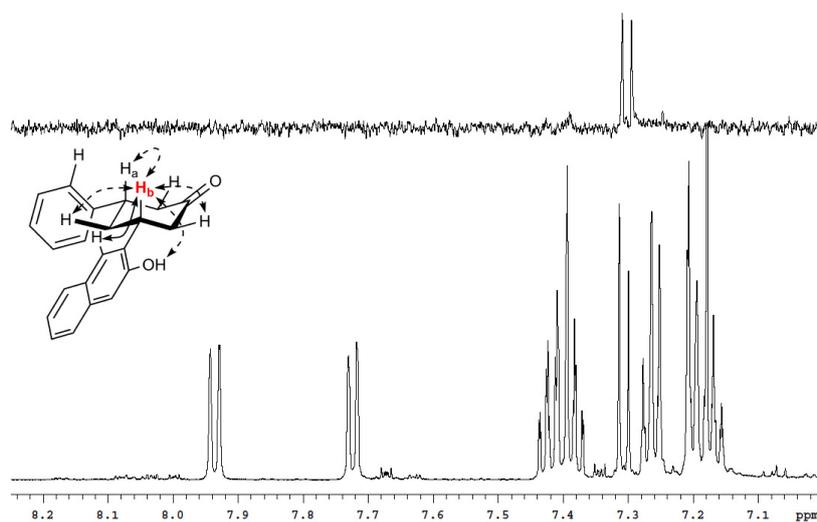
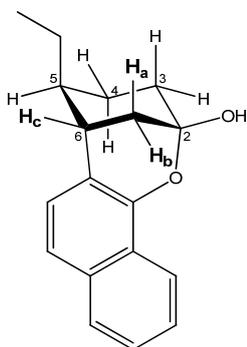


Figure S25. DPGSE-NOE spectra obtained for *ent-6da* (25°C, 600 MHz, $CDCl_3$) on saturation of proton H_b . Detail of the aromatic region.

Relative Configuration assignment of compound *ent*-5ca



The relative stereochemistry of compound *ent*-5ca was established by means of NOE experiments. Irradiation of axial proton H_a generated a strong NOE effect on geminal proton H_b as well as a visible NOE on H_c , on one of the two protons on C3 and on the methylene of the ethyl group. As a control, irradiation on equatorial proton H_b in addition to the strong NOE effect on geminal proton H_a , produced only an additional visible NOE effect on H_c . Neither H_a nor H_b show visible NOE effects on C5H. According to these experiments the C5 ethyl group should be in an axial position, trans with respect to the C6-Ar bond.

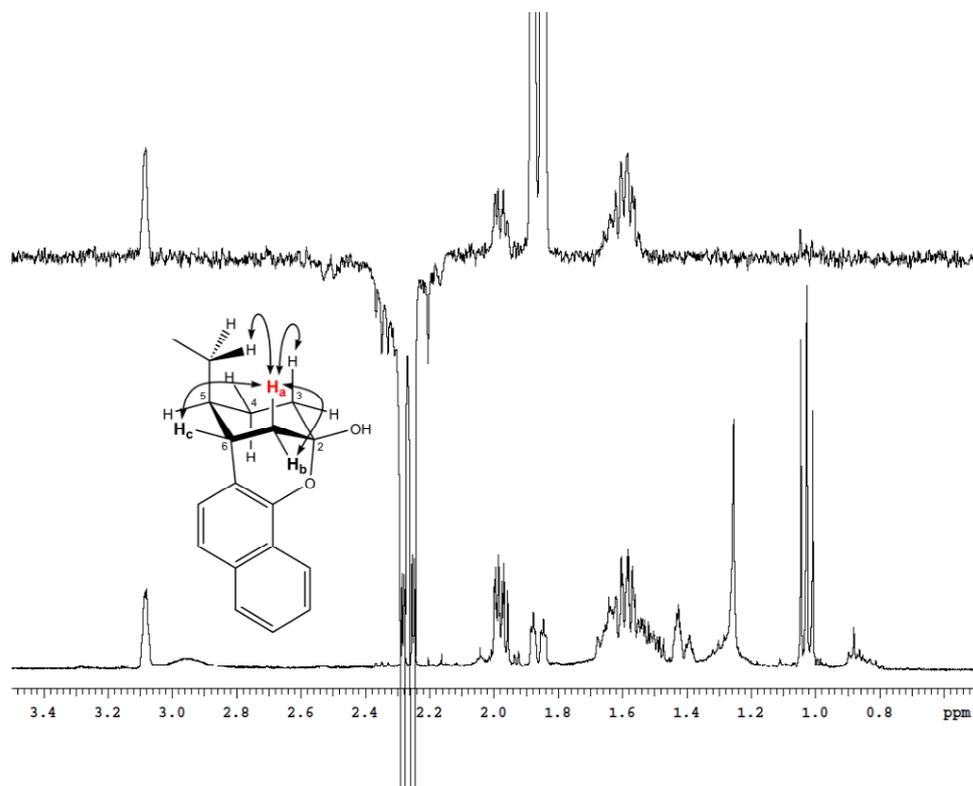


Figure S26. DPGSE-NOE spectra obtained for *ent*-5ca (25°C, 600 MHz, CDCl₃) on saturation of proton H_a . Detail of the aliphatic region.

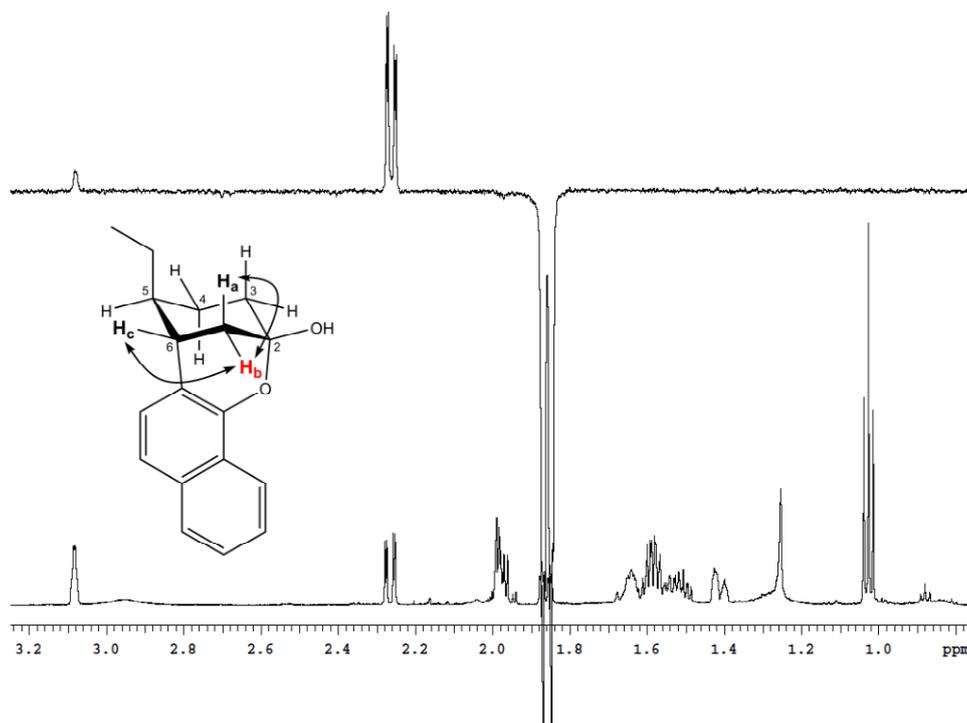
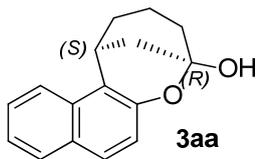


Figure S27. DPGSE-NOE spectra obtained for *ent*-5ca (25°C, 600 MHz, CDCl₃) on saturation of proton *H_b*. Detail of the aliphatic region.

General procedure for the Friedel-Crafts alkylation-acetalization cascade of naphthols with α,β -unsaturated cyclic ketones

In a screw-capped vial equipped with a Teflon coated magnetic stir bar an anhydrous toluene solution of 9-amino(9-deoxy)*epi*-quinine **A** (0.04 mmol, 0.2 eq., 20 mol%) and 2-hydroxy-5-nitrobenzoic acid (0.08 mmol, 0.4 eq., 40 mol%) was prepared under argon. After 5 min α,β -unsaturated ketone (0.2 mmol, 1 eq.) was added. The resulting yellow solution was stirred for further 5 minutes then β - or α -naphthol (0.22 mmol, 1.1 eq) was added and stirring was continued for 72 hours at 40 °C. Then the septum was replaced, the vial was refilled with argon, and quickly closed with the screw-cap. The vial was placed at 40°C in a pre-heated oil bath and stirring was continued for 72 hours. Subsequently the reaction mixture was diluted with an 1:1 mixture of Et₂O/DCM, passed through a short plug of silica gel and solvent was evaporated in vacuo to give the crude product which was purified through flash chromatography on silica gel.

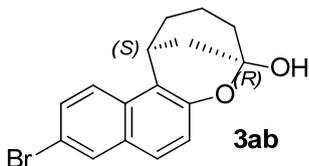
(1S,5R)-2,3,4,5-tetrahydro-1H-1,5-methanonaphtho[2,1-b]oxocin-5-ol 3aa (Table 2, entry 1): The



reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 80:20) as a white solid in 82% yield and 84% ee. **HPLC analysis:** Phenomenex Lux-Cellulose 2 column; Hex/*i*-PrOH 95:5, flow rate 0.65 mL/min, T = 20 °C, λ = 230 nm, τ_{minor} = 20.0 min

τ_{major} = 30.1 min. $[\alpha]_D^{30}$: +106.7 (c 3.96, CHCl₃, 84% ee). **HRMS** calculated for C₁₆H₁₆O₂: 240.11503, found 240.11537. **¹H-NMR (300 MHz, CDCl₃):** δ 1.43 (tt, J_a = 13.7 Hz, J_b = 4.8 Hz, 1H), 1.55-1.66 (m, 1H), 1.74 (tt, J_a = 13.1 Hz, J_b = 3.8 Hz, 1H), 1.86 (td, J_a = 14.4 Hz, J_b = 5.5 Hz, 1H), 1.89-7.99 (m, 1H), 2.00-2.09 (m, 1H), 2.11-2.20 (m, 1H), 2.19 (dd, J_a = 12.4 Hz, J_b = 2.9 Hz, 1H), 2.92 (bs, 1H), 3.86 (m, 1H), 7.07 (d, J = 8.2, 1H), 7.31 (td, J_a = 7.5 Hz, J_b = 1.1 Hz, 1H), 7.47 (td, J_a = 7.7 Hz, J_b = 1.5 Hz, 1H), 7.63 (d, J = 8.9, 1H), 7.76 (d, J = 8 Hz, 1H), 7.84 (d, J = 8.5 Hz, 1H). **¹³C-NMR (75 MHz, CDCl₃):** δ 19.4 (CH₂), 29.8 (CH₂), 30.6 (CH), 36.3 (CH₂), 39.4 (CH₂), 98.3 (C), 116.3 (C), 117.7 (CH), 121.5 (CH), 123.0 (CH), 126.4 (CH), 128.1 (CH), 128.6 (CH), 128.9 (C), 131.4 (C), 153.0 (C).

(1S,5R)-10-bromo-2,3,4,5-tetrahydro-1H-1,5-methanonaphtho[2,1-b]oxocin-5-ol 3ab (Table 2, entry

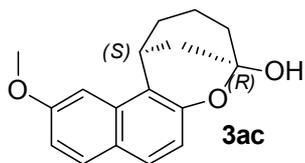


2): The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 80:20) as a white solid in 58% yield and 82% ee. **HPLC analysis:** Phenomenex Lux-Cellulose 2 column; Hex/*i*-PrOH 90:10, flow rate 0.65 mL/min, T = 20 °C, λ = 230 nm,

τ_{minor} = 12.9 min τ_{major} = 15.1 min. $[\alpha]_D^{30}$: +76.8 (c 0.6, CHCl₃, 82% ee). **HRMS** calculated for C₁₆H₁₅BrO₂: 318.02554, found 318.02526. **¹H-NMR (600 MHz, CDCl₃):** δ 1.38 (tq, J_a = 14.1 Hz, J_b = 4.4 Hz, 1H), 1.58-

1.67 (m, 1H), 1.75 (tt, $J_a = 13.2$ Hz, $J_b = 4.4$ Hz, 1H), 1.84-1.93 (m, 2H), 2.02-2.08 (m, 1H), 2.17 (d, $J = 13.5$ Hz, 1H), 2.20 (dd, $J_a = 12.3$ Hz, $J_b = 2.9$ Hz, 1H), 2.9 (bs, 1H), 3.8 (m, 1H), 7.08 (d, $J = 9.2$, 1H), 7.51-7.56 (m, 2H), 7.71 (d, $J = 9.0$ Hz, 1H), 7.90 (d, $J = 2.0$ Hz, 1H). $^{13}\text{C-NMR}$ (150 MHz, CDCl_3): δ 19.3 (CH_2), 29.8 (CH_2), 30.6 (CH), 36.1 (CH_2), 39.3 (CH_2), 98.4 (C), 116.5 (C), 116.6 (C), 118.8 (CH), 123.3 (CH), 127.2 (CH), 129.6 (CH), 130.0 (CH), 132.1 (C), 130.5 (C), 153.3 (C).

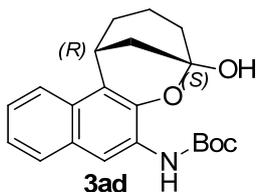
(1S,5R)-11-methoxy-2,3,4,5-tetrahydro-1H-1,5-methanonaphtho[2,1-b]oxocin-5-ol 3ac (Table 2, entry 3):



The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/ Et_2O 80:20) as a white solid in 82% yield and 78% ee. **HPLC analysis:** Phenomenex Lux-Cellulose 2 column; Hex/*i*-PrOH 9:1, flow rate 0.65 mL/min, $T = 20$ °C, $\lambda = 230$ nm, $\tau_{\text{minor}} = 15.97$ min $\tau_{\text{major}} = 24.92$ min. $[\alpha]_D^{30}$: +105 (c 1.10, CHCl_3 , 78% ee). **HRMS** calculated for $\text{C}_{17}\text{H}_{18}\text{O}_3$: 270.125595, found 270.12562. $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.34-1.49 (m, 1H), 1.55-1.67 (m, 1H), 1.73 (tt, $J_a = 13.0$ Hz, $J_b = 3.8$ Hz, 1H), 1.85 (dt, $J_a = 13.4$ Hz, $J_b = 5.3$ Hz, 1H), 1.90-1.99 (m, 1H), 2.0-2.09 (m, 1H), 2.11-2.22 (m, 2H), 3.06 (brs, 1H), 3.73-3.78 (m, 1H), 3.91 (s, 3H), 6.92 (d, $J = 8.7$ Hz, 1H), 6.99 (dd, $J_a = 8.7$ Hz, $J_b = 2.5$ Hz, 1H), 7.11 (brd, $J = 2.4$ Hz, 1H), 7.54 (d, $J = 8.8$ Hz, 1H), 7.66 (d, $J = 8.8$ Hz, 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ 19.4 (CH_2), 29.4 (CH_2), 30.8 (CH), 36.4 (CH_2), 39.4 (CH_2), 55.3 (CH_3), 98.2 (C), 101.0 (CH), 114.7 (CH), 115.2 (CH), 115.3 (C), 124.2 (C), 127.8 (CH), 130.1 (CH), 132.7 (C), 153.6 (C), 158.4 (C).

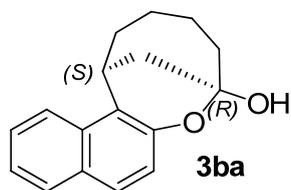
tert-butyl

((1R,5S)-5-hydroxy-2,3,4,5-tetrahydro-1H-1,5-methanonaphtho[2,1-b]oxocin-7-yl)carbamate 3ad (Table 2, entry 4):



The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/ EtOAc 9:1) as a pink solid in 94% yield and 73% ee. **HPLC analysis:** Daicel Chiralpak AD-H column; Hex/*i*-PrOH 9:1, flow rate 0.7 mL/min, $T = 25$ °C, $\lambda = 230$ nm, $\tau_{\text{minor}} = 10.9$ min $\tau_{\text{major}} = 15.3$ min. $[\alpha]_D^{29}$: -56.6 (c 1.39, CHCl_3 , 73% ee). **HRMS** calculated for $\text{C}_{21}\text{H}_{25}\text{NO}_4$ 355.17836, found 355.17882. $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.37 (tt, $J_a = 13.6$ Hz, $J_b = 5.0$ Hz, 1H), 1.58 (s, 10H), 1.72 (tt, $J_a = 13.0$ Hz, $J_b = 3.8$ Hz, 1H), 1.85 (td, $J_a = 13.4$ Hz, $J_b = 5.2$ Hz, 1H), 1.85-1.96 (m, 1H), 2.01-2.10 (m, 1H), 2.14-2.22 (m, 1H), 2.21 (dd, $J_a = 12.0$ Hz, $J_b = 3.2$ Hz, 1H), 3.67 (bs, 1H), 3.78 (m, 1H), 7.27-7.41 (m, 3H), 7.66-7.77 (m, 2H), 8.41 (bs, 1H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 19.3 (CH_2), 28.4 (CH_3), 29.6 (CH_2), 30.7 (CH), 36.4 (CH_2), 39.2 (CH_2), 80.6 (C), 99.5 (C), 113.1 (CH), 116.1 (C), 121.0 (CH), 123.6 (CH), 124.6 (CH), 126.5 (C), 126.8 (C), 128.3 (CH), 129.2 (C), 143.0 (C), 152.8 (C).

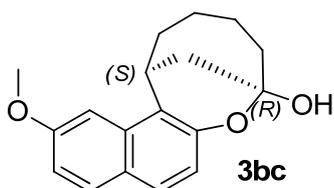
(8R,13S)-8,9,10,11,12,13-hexahydro-8,13-methanonaphtho[2,1-b]oxonin-8-ol 3ba (Table 2, entry 5):



The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 85:15) to give a yellow solid in 57% yield and 73% ee. **HPLC analysis:** Phenomenex Lux-Cellulose 2 column; Hex/*i*-PrOH 95:5, flow rate 0.65 mL/min, T = 20 °C, λ =

230 nm, τ_{minor} = 20.8 min τ_{major} = 33.1 min. [α]_D²⁸: +20.9 (c 0.54, CHCl₃, 73% ee). **HRMS** calculated for C₁₇H₁₈O₂ 254.13068, found 254.13044. **¹H-NMR (300 MHz, CDCl₃):** δ 1.42-1.68 (m, 4H), 2.00-2.31 (m, 5H), 2.58 (dd, J_a = 13.8 Hz, J_b = 1.7 Hz, 1H), 2.79 (s, 1H), 3.73 (m, 1H), 7.07 (d, J = 8.9 Hz, 1H), 7.34 (dt, J_a = 7.5 Hz, J_b = 1.1 Hz, 1H), 7.49 (dt, J_a = 8.3 Hz, J_b = 1.5 Hz, 1H), 7.65 (d, J = 8.9 Hz, 1H), 7.79 (d, J = 8 Hz, 1H), 7.84 (d, J = 8.5 Hz, 1H). **¹³C-NMR (75 MHz, CDCl₃):** δ 22.3 (CH₂), 25.1 (CH₂), 28.7 (CH), 33.8 (CH₂), 37.0 (CH₂), 43.0 (CH₂), 100.4 (C), 117.8 (C), 118.7 (CH), 122.3 (CH), 123.1 (CH), 126.3 (CH), 128.3(CH), 128.7(CH), 129.4 (C), 131.6 (C), 151.2 (C).

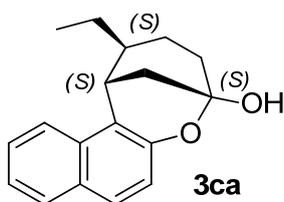
(8R,13S)-2-methoxy-8,9,10,11,12,13-hexahydro-8,13-methanonaphtho[2,1-b]oxonin-8-ol 3bc (Table 2, entry 6):



The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 85:15) to give a yellow solid in 79% yield and 76% ee. **HPLC analysis:** Phenomenex Lux-Cellulose 2 column; Hex/*i*-PrOH 9:1, flow rate 0.7

mL/min, T = 23 °C, λ = 230 nm, τ_{minor} = 16.0 min τ_{major} = 33.7 min. [α]_D³⁰: + 41.5 (c 0.54, CHCl₃, 76% ee). **HRMS** calculated for C₁₈H₂₀O₃ 284.14125, found 284.14163. **¹H-NMR (400 MHz, CDCl₃):** δ 1.54 (m, 4H), 2.04 (m, 2H), 2.19, (m, 3H), 2.54 (d, J = 14.4 Hz, 1H), 2.90 (br. s, 1H), 3.59, (m, 1H), 3.91 (s, 3H), 6.91 (d, J = 8.8 Hz, 1H), 7.00 (dd, J_a = 8.8 Hz, J_b = 2.6 Hz, 1H), 7.10 (d, J = 2.1 Hz, 1H), 7.55 (d, J = 8.8 Hz, 1H), 7.67 (d, J = 9.0 Hz, 1H). **¹³C-NMR (100 MHz, CDCl₃):** δ 22.6 (CH₂), 25.4 (CH₂), 29.1 (CH), 33.5 (CH₂), 37.3 (CH₂), 43.3 (CH₂), 55.5 (CH₃), 100.6 (C), 102.4 (CH), 114.9 (CH), 116.5 (CH), 117.2 (C), 124.9 (C), 128.3 (CH), 130.5 (CH), 152.1 (C), 158.5 (C).

(1S,2S,5S)-2-ethyl-2,3,4,5-tetrahydro-1H-1,5-methanonaphtho[2,1-b]oxocin-5-ol 3ca (Table 2, entry 7):

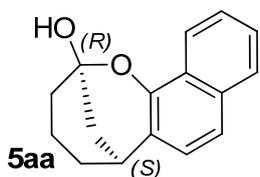


The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 85:15) to give a yellow oil in 16% yield and 60% ee. **HPLC analysis:** Phenomenex Lux-Amilose 2 column; Hex/*i*-PrOH 85:15, flow rate 0.4 mL/min, T = 23°C, λ = 230 nm, τ_{minor} = 13.9 min τ_{major} = 15.7 min. [α]_D³²: + 6.7 (c

0.285; CHCl₃, 60% ee). **HRMS** calculated for C₁₈H₂₀O₂ 268.14633, found 268.14658. **¹H-NMR (600 MHz,**

CDCl₃): δ 1.11 (t, $J = 7.6$ Hz, 3H), 1.43 (m, 1H), 1.67, (m, 3H), 1.84 (m, 1H), 1.96 (dd, $J_a = 4.0$ Hz, $J_b = 1.4$ Hz, 1H), 2.00 (dd, $J_a = 5.34$ Hz, $J_b = 2.5$ Hz, 1H), 2.34 (dd, $J_a = 12.7$ Hz, $J_b = 2.9$ Hz, 1H), 2.88 (s, 1H), 3.69 (s, 1H), 7.07 (d, $J = 9.1$ Hz, 1H), 7.31 (m, 1H), 7.47 (m, 1H), 7.63, (d, $J = 9.0$ Hz, 1H), 7.76 (d, $J = 8.23$ Hz, 1H), 7.83 (d, $J = 8.5$ Hz, 1H). **¹³C-NMR (150 MHz, CDCl₃)**: δ 12.6 (CH₃), 23.1 (CH₂), 24.2 (CH₂), 30.8 (CH₂), 34.0 (CH), 35.1 (CH₂), 39.0 (CH), 99.5 (C), 117.7 (CH), 117.8 (C), 121.3 (CH), 122.9 (CH), 126.4 (CH), 128.1 (CH), 128.7 (CH), 128.9 (C), 131.3 (C), 152.9 (C).

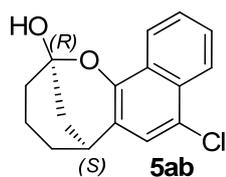
(2R,6S)-3,4,5,6-tetrahydro-2H-2,6-methanonaphtho[1,2-b]oxocin-2-ol 5aa (Table 3, entry 1): The



reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 85:15) to give a white solid in 73% yield and 96% ee. **HPLC analysis**: Phenomenex Lux-Amilose 2 column; Hex/*i*-PrOH 95:5, flow rate 0.65 mL/min, T = 20 °C, $\lambda = 230$ nm, $\tau_{minor} =$

14.5 min $\tau_{major} = 16.1$ min. **[α]_D³⁰**: -28.9 (c 3.60, CHCl₃, 96% ee). **HRMS** calculated for C₁₆H₁₆O₂ 240.11503, found 240.11537. **¹H-NMR (600 MHz, CDCl₃)**: δ 1.33-1.47 (m, 1H), 1.54-1.63 (m, 1H), 1.67-1.81 (m, 2H) 1.85 (dt, 1H, $J_a = 13.7$ Hz, $J_b = 5.5$ Hz), 2.05 (m, 1H), 2.14 (dd, 1H, $J_a = 12.2$ Hz, $J_b = 2.6$ Hz), 2.18 (d, 1H, $J = 13.3$ Hz), 2.98 (bs, 1H), 3.28 (m, 1H), 7.11 (d, 1H, $J = 8.2$), 7.33 (d, 1H, $J = 8.2$), 7.44 (m, 2H), 7.75 (m, 1H), 8.21 (m, 1H). **¹³C-NMR (150 MHz, CDCl₃)**: δ 18.7 (CH₂), 31.5 (CH₂), 35.3 (CH), 36.5 (CH₂), 39.1 (CH₂), 99.1 (C), 118.5 (C), 119.4 (CH), 121.7 (CH), 123.8 (C), 125.7 (CH), 125.7 (CH), 126.1 (CH), 127.4 (CH), 133.5 (C), 150.3 (C).

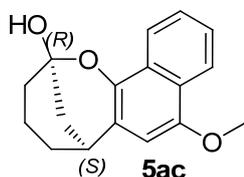
(2R,6S)-8-chloro-3,4,5,6-tetrahydro-2H-2,6-methanonaphtho[1,2-b]oxocin-2-ol 5ab (Table 3, entry



2): The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 80:20) to give a white solid in 91% yield and 93% ee. **HPLC analysis**: Daicel Chiralpak AD-H column; Hex/*i*-PrOH 95:5, flow rate 0.7 mL/min $\lambda = 214$ nm, $\tau_{major} = 18.27$ min, $\tau_{minor} = 19.37$ min.

[α]_D³⁰: -14.99 (c 0.99, CHCl₃, 93% ee). **HRMS** calculated for C₁₆H₁₅O₂Cl 274.076059, found 274.07611. **¹H-NMR (400 MHz, CDCl₃)**: δ 1.29-1.46 (m, 1H), 1.53-1.64 (m, 1H), 1.64-1.78 (m, 2H), 1.84 (dt, 1H, $J_a = 13.6$ Hz, $J_b = 5.3$ Hz), 2.00-2.23 (m, 3H), 3.17-3.23 (m, 1H), 3.23-3.25 (brs, 1H), 7.16 (s, 1H), 7.49 (m, 1H), 7.55 (m, 1H), 8.15 (m, 1H), 8.22 (m, 1H). **¹³C-NMR (100 MHz, CDCl₃)**: δ 18.6 (CH₂), 31.3 (CH₂), 35.1 (CH), 36.2 (CH₂), 39. (CH₂), 99.3 (C), 118.9 (C), 122.0 (C), 122.1 (CH), 124.1 (CH), 124.7 (C), 125.7 (CH), 125.8 (CH), 126.7 (CH), 130.2 (C), 149.4 (C).

(2R,6S)-8-methoxy-3,4,5,6-tetrahydro-2H-2,6-methanonaphtho[1,2-b]oxocin-2-ol 5ac (Table 3, entry

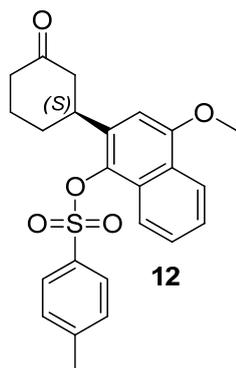


3): The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 70:30) to give a white solid in 95% yield and 92% ee. **HPLC analysis**: Phenomenex Lux-Cellulose 2 column; Hex/*i*-PrOH 87:13, flow rate 0.7 mL/min, T = 15 °C, λ = 230 nm, τ_{minor} = 10.63 min τ_{major} = 17.68 min. **HRMS** calculated for C₁₇H₁₈O₃ 270.125595, found 270.12566.

¹H-NMR (400 MHz, CDCl₃): δ 1.25-1.40 (m, 1H), 1.43-1.52 (m, 1H), 1.55-1.80 (m, 3H), 1.93-2.02 (m, 2H), 2.02-2.07 (m, 1H), 3.04-3.10 (m, 1H), 3.14 (brs, 1H), 3.84 (s, 3H), 6.31 (s, 1H), 7.31-7.42 (m, 2H), 8.03-8.10 (m, 2H).

¹³C-NMR (100 MHz, CDCl₃): δ 18.8 (CH₂), 31.3 (CH₂), 35.8 (CH), 36.6 (CH₂), 39.2 (CH₂), 55.7 (CH₃), 98.6 (C), 104.0 (CH), 117.4 (C), 121.4 (CH), 121.6 (CH), 124.4 (C), 125.0 (CH), 125.1 (C), 125.7 (CH), 144.0 (C), 148.8 (C).

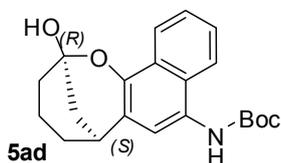
Compound **5ac** (50 mg, 0.185 mmol) has been reacted with TsCl (70.54 mg, 0.37 mmol, 2 equiv) in the presence of Et₃N (52 μL, 0.37 mmol, 2 equiv.) and DMAP (50% mol, 0.0925 mmol, 11.30 mg) in 4 ml of dichloromethane for 2 days. The reaction mixture was poured into water (10 ml) and extracted 2 times with 10 ml of dichloromethane. After evaporation of the solvent compound **12** was obtained in 50% yield.



[α]_D³¹: -40.5 (c 0.44, CHCl₃, 92% ee). **¹H-NMR (400 MHz, CDCl₃)**: δ 1.51-1.71 (m, 1H), 1.73-1.90 (m, 1H), 1.96-2.17 (m, 2H), 2.27-2.53 (m, 7H), 3.41 (m, 1H), 4.02 (s, 3H), 6.67 (s, 1H), 7.33-7.39 (m, 2H), 7.39-7.47 (m, 2H), 7.75-7.82 (m, 1H), 7.82-7.88 (m, 2H), 8.15-8.22 (m, 1H). **¹³C-NMR (100 MHz, CDCl₃)**: δ 21.7 (CH₃), 25.4 (CH₂), 31.8 (CH₂), 38.2 (CH), 41.1 (CH₂), 47.6 (CH₂), 55.7 (CH₃), 101.1 (CH), 121.9 (CH), 122.7 (CH), 125.3 (C), 125.7 (CH), 127.3 (CH), 128.2 (CH), 128.8 (C), 130.0 (CH), 133.7 (C), 134.1 (C), 135.9 (C), 145.5 (C), 154.6 (C), 209.9 (C).

tert-butyl

((2R,6S)-2-hydroxy-3,4,5,6-tetrahydro-2H-2,6-methanonaphtho[1,2-b]oxocin-8-

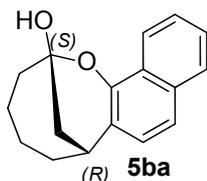


yl)carbamate 5ad (Table 3, entry 4): The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst and the title compound was obtained in 58% yield determined by ¹H-NMR using CH₂Br₂ as internal standard. Compound **5ad** was purified by flash chromatography on silica gel (eluent mixture Hex/Et₂O 60:40) to give a pink solid in 87% ee. Further purification was carried out through preparative HPLC (AD-H column, Hex/*i*-PrOH 8:2, flow rate 20 mL/min) to give a white solid. **HPLC analysis**: Daicel Chiralpak AD-H column; Hex/*i*-PrOH 8:2, flow rate 1 mL/min, T = 25 °C, λ = 230 nm, τ_{major} = 8.6 min, τ_{minor} = 13.3 min. [α]_D³⁰: -

11.0 (c 0.82, CHCl₃, 87% ee). **HRMS** calculated for C₂₁H₂₅NO₄ 355.17836, found 355.17882. **¹H-NMR**

(600 MHz, CDCl₃): δ 1.42 (tt, 1H, $J_a = 13.5$ Hz, $J_b = 4.5$ Hz), 1.54 (s, 10H), 1.70 (tt, 1H, $J_a = 13.5$ Hz, $J_b = 3.7$ Hz), 1.79-1.86 (m, 1H), 1.84 (td, 1H, $J_a = 13.1$ Hz, $J_b = 5.3$ Hz), 2.01-2.06 (m, 1H), 2.11-2.19 (m, 2H), 2.96 (s, 1H), 3.28 (m, 1H), 6.56 (s, 1H), 7.39-7.54 (m, 3H), 7.80 (d, 1H, $J = 8.7$ Hz), 8.24 (d, 1H, $J = 8.1$ Hz). ¹³C-NMR (100 MHz, CDCl₃): δ 17.8 (CH₂), 27.4 (CH₃), 30.3 (CH₂), 34.3 (CH), 35.5 (CH₂), 38.1 (CH₂), 79.3 (C), 98.1 (C), 117.2 (C), 119.8 (C), 121.3 (CH), 122.4 (C), 123.0 (C), 123.9 (C), 124.2 (2×CH), 125.0 (2 CH), 146.9 (C).

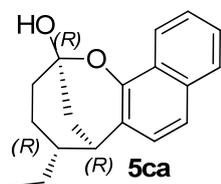
(2*S*,7*R*)-2,3,4,5,6,7-hexahydro-2,7-methanonaphtho[1,2-*b*]oxonin-2-ol and 3-(1-hydroxynaphthalen-2-yl)cycloheptanone **5ba** (Table 3, entry 5):



The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was obtained in 34% yield determined by ¹H-NMR using CH₂Br₂ as internal standard together with a 8% of Friedel-Crafts alkylation compound.

Compound **5ba** was purified from crude mixture by flash chromatography on silica gel (eluent mixture Hex/Et₂O 8:2) in 90% ee. **HPLC analysis:** Phenomenex Lux-Amilose 2 column; Hex/*i*-PrOH 95:5, flow rate 0.7 mL/min, T = 20 °C, $\lambda = 230$ nm, $\tau_{minor} = 30.3$ min $\tau_{major} = 34.5$ min. $[\alpha]_D^{30}$: +48.0 (c 0.375; CHCl₃, 90% ee). **HRMS calcd.** for C₁₆H₁₆O₂ 240.11503, found 240.11537. **¹H NMR (300 MHz, CDCl₃) of 5ca with traces of Friedel-Crafts alkylation compound:** δ 1.29-1.38 (m, 1H), 1.49-1.55 (m 2H), 1.61-1.71 (m, 1H), 1.83-1.93 (m 1H), 2.03-2.09 (m, 1H), 2.09-2.17 (m, 1H), 2.17-2.25 (m, 2H), 2.58 (d, 1H, $J = 13.6$ Hz), 2.99 (bs, 1H), 2.25 (m, 1H), 7.23 (d, 1H, $J = 8.1$ Hz), 7.40 (d, 1H, $J = 8.2$ Hz), 7.43-7.48 (m, 2H), 7.75-7.79 (m, 1H), 8.22-8.27 (m, 1H). **¹³C-NMR (150 MHz, CDCl₃) of 5ca with traces of Friedel-Crafts alkylation compound:** δ 22.0 (CH₂), 25.0 (CH₂), 32.3 (CH), 34.3 (CH₂), 37.0 (CH₂), 42.4 (CH₂), 100.3 (C), 118.5 (C), 119.1 (CH), 120.8 (CH), 124.1 (C), 124.2 (CH), 124.8 (C), 125.3 (CH), 126.3 (CH), 132.3 (C), 147.4 (C).

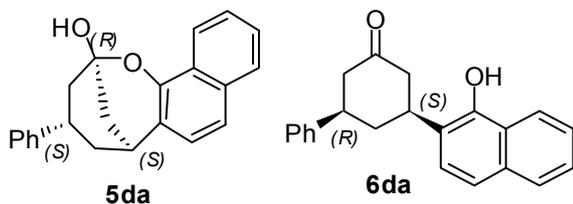
(2*R*,5*R*,6*R*)-5-ethyl-3,4,5,6-tetrahydro-2*H*-2,6-methanonaphtho[1,2-*b*]oxocin-2-ol **5ca** (Table 3, entry 6):



The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 9:1) to give a yellow oil in 27% and 89% ee. **HPLC analysis:** Daicel Chiralcel OJ-H; flow rate 0.7 ml/min, Hex/*i*-PrOH 9:1, $\lambda = 230$ nm, 23°C, $\tau_{minor} = 24.5$ min $\tau_{major} = 32.3$ min. $[\alpha]_D^{31}$: -60.1 (c 0.27; CHCl₃, 89% ee).

HRMS calculated for C₁₈H₂₀O₂ 268.14633, found 268.14658. **¹H-NMR (400 MHz, CDCl₃):** δ 1.04 (t, 3H, $J = 7.6$ Hz), 1.48-1.46 (m, 1H), 1.48-1.70 (m, 5H), 1.85-1.91 (m, 1H), 1.96-2.02 (m, 2H), 2.28 (dd, 1H, $J_a = 12.6$ Hz, $J_b = 2.7$ Hz), 2.95 (s, 1H), 3.10 (m, 1H), 7.14 (d, 1H, $J = 8.2$ Hz), 7.35 (d, 1H, $J = 8.2$ Hz), 7.41-7.49 (m, 2H), 7.74-7.79 (m, 1H), 8.20-8.25 (m, 1H). **¹³C-NMR (100 MHz, CDCl₃):** δ 12.7 (CH₃), 22.1 (CH₂), 24.2 (CH₂), 31.2 (CH₂), 34.7 (CH₂), 39.1 (CH), 41.2 (CH), 99.2 (C), 119.4 (CH), 120.2 (C), 121.6 (CH), 123.8 (C), 125.0 (CH), 125.6 (CH), 126.1 (CH), 127.4 (CH), 133.4 (C), 150.1 (C).

(2R,4S,6S)-4-phenyl-3,4,5,6-tetrahydro-2H-2,6-methanonaphtho[1,2-b]oxocin-2-ol 5da and (3S,5R)-3-(1-hydroxynaphthalen-2-yl)-5-phenylcyclohexanone 6da (Table 3, entries 7-8): The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. Compounds **5da** and **6da** were isolated after 5 days by flash chromatography on silica gel (eluent

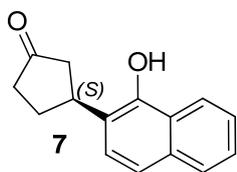


mixture Hex/Et₂O 70:30) in 26% yield and 93% ee and 31% yield and 98% ee respectively (57% overall yield).

HPLC analysis: for compound **5da** Daicel Chiralpak AD-H column; Hex/*i*-PrOH 95:5, flow rate 0.75 mL/min λ = 214 nm, τ_{major} = 32.69 min, τ_{minor} = 35.13 min; for compound **6da** Daicel Chiralpak AD-H column; Hex/*i*-

PrOH 80:20, flow rate 0.75 mL/min λ = 214 nm, τ_{major} = 14.39 min, τ_{minor} = 16.19 min. $[\alpha]_D^{30}$ for **5da**: -133.8 (*c* 0.447; CHCl₃, 93% ee); $[\alpha]_D^{30}$ for **6da**: +19.2 (*c* 0.47; CHCl₃, 98% ee). **HRMS calcd.** for C₂₂H₂₀O₂ 316.14633, found 316.14661. **¹H-NMR (400 MHz, CDCl₃) 5da:** δ 1.92 (dt, 1H, J_a = 12.7 Hz, J_b = 3.2 Hz), 2.02-2.19 (m, 3H), 2.27 (dd, 1H, J_a = 12.3 Hz, J_b = 2.8 Hz), 2.38-2.46 (m, 1H), 2.84 (m, 1H), 3.13 (brs, 1H), 3.36-3.42 (m, 1H), 7.08-7.13 (m, 2H), 7.14-7.19 (m, 2H), 7.21-7.25 (m, 2H), 7.38 (d, 1H, J = 8.3 Hz), 7.44-7.51 (m, 2H), 7.77-7.82 (m, 1H), 8.23-8.29 (m, 1H). **¹³C-NMR (100 MHz, CDCl₃) 5da:** δ 35.3 (CH), 36.1 (CH₂), 36.5 (CH), 39.6 (CH₂), 46.4 (CH₂), 99.3 (C), 118.9 (C), 119.7 (CH), 121.7 (CH), 123.8 (C), 125.2 (CH), 125.8 (CH), 126.0 (CH), 126.3 (CH), 127.0 (CH), 127.4 (CH), 128.4 (CH), 133.6 (C), 144.3 (C), 150.1 (C). **¹H-NMR (400 MHz, CDCl₃) 6da:** δ 2.20-2.35 (m, 2H), 2.60-2.80 (m, 4H), 3.21 (m, 1H), 3.74 (m, 1H), 5.93 (brs, 1H), 7.21-7.31 (m, 3H), 7.32-7.42 (m, 3H), 7.43-7.54 (m, 3H), 7.81 (m, 1H), 8.01 (m, 1H). **¹³C-NMR (100 MHz, CDCl₃) 6da:** δ 37.3 (CH), 39.3 (CH₂), 44.0 (CH), 47.3 (CH₂), 48.6 (CH₂), 120.1 (CH), 121.1 (CH), 123.9 (C), 124.1 (CH), 124.5 (C), 125.8 (CH), 125.9 (CH), 126.6 (CH), 126.9 (CH), 128.1 (CH), 128.8 (CH), 133.3 (CH), 143.9 (CH), 147.6 (CH), 210.8 (C).

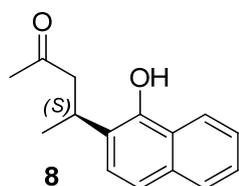
(S)-3-(1-hydroxynaphthalen-2-yl)cyclopentanone 7 (Table 3, entry 9): The reaction was performed following the general procedure using 9-amino(9-deoxy)*epi*-quinine **A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 70:30) to give a yellow solid in a 63% yield and 90%



ee. **HPLC analysis:** Daicel Chiralpak AS-H; Hex/*i*-PrOH 85:15, flow rate 0.7 ml/min, λ = 230 nm, 25°C, τ_{major} = 23.1 min, τ_{minor} = 29.2 min. $[\alpha]_D^{29}$: -56.0 (*c*

0.67; CHCl₃, 90% ee). **HRMS calcd.** for C₁₆H₁₆O₂ 226.09938, found 226.09952. **¹H-NMR (400 MHz, CDCl₃):** δ 2.06-2.30 (m, 2H), 2.30-2.60 (m, 4H), 2.75 (dd, 2H, J_a = 17.8 Hz, J_b = 7.8 Hz), 3.84-3.98 (m, 1H), 5.51 (s, 1H), 7.35 (d, 1H, J = 8.6 Hz), 7.44-7.57 (m, 3H), 7.84 (d, 1H, J = 8.1 Hz), 7.99 (dm, 1H, J = 8.1 Hz). **¹³C-NMR (100 MHz, CDCl₃):** δ 28.7 (CH₂), 34.2, 35.4 (CH₂), 35.9, 37.8 (CH₂), 37.9, 39.2, 43.8 (CH₂), 118.7 (CH), 120.0 (CH), 120.4 (C), 122.0 (C), 123.3 (C), 123.4 (CH), 124.1, 124.4, 124.6, 124.8 (CH), 126.5, 127.2 (CH), 132.9 (CH), 147.2 (C), 218.2 (C).

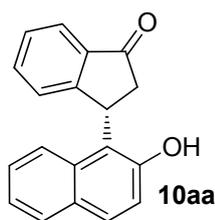
(S)-4-(1-hydroxynaphthalen-2-yl)pentan-2-one 8 (Table 3, entry 10): The reaction was performed



following the general procedure using 9-amino(9-deoxy)*epi*-quinidine *ent-A* as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hex/Et₂O 70:30) to give a yellow solid in 71% yield and 44% ee.

HPLC analysis: Daicel Chiralpak AD-H; flow rate 0.7 ml/min, Hex/*i*-PrOH 96:4, $\lambda = 230$ nm, 25°C, $\tau_{major} = 47.7$ min, $\tau_{minor} = 52.5$ min. $[\alpha]_D^{31} = +25$ (c 1.39; CHCl₃, 44% ee). **HRMS calcd.** for C₁₆H₁₆O₂ 228.11503, found 228.11533. **¹H-NMR (400 MHz, CDCl₃):** δ 1.39 (d, 3H, $J = 7.4$ Hz), 2.13 (s, 3H), 2.73 (dd, 1H, $J_a = 16.6$ Hz, $J_b = 8.7$ Hz), 2.89 (dd, 1H, $J_a = 16.5$ Hz, $J_b = 5.2$ Hz), 4.12 (m, 1H), 5.65 (s, 1H), 6.77 (d, 1H, $J_a = 7.6$ Hz), 7.18 (d, 1H, $J = 7.5$ Hz), 7.45-7.60 (m, 2H), 8.10 (d, 1H, $J = 8.5$ Hz), 8.26 (d, 1H, $J = 8.3$ Hz). **¹³C-NMR (100 MHz, CDCl₃):** δ 21.4 (CH₃), 29.3 (CH), 30.4 (CH₃), 51.8 (CH₂), 108.0 (CH), 122.4 (CH), 122.5 (CH), 122.6 (C), 122.9 (CH), 124.8 (CH), 124.9 (C), 126.5 (CH), 132.1 (C), 134.3 (C), 150.1 (C), 208.5 (C).

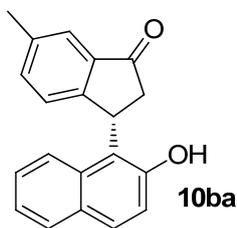
(S)-3-(2-hydroxynaphthalen-1-yl)-2,3-dihydro-1H-inden-1-one 10aa (Table 4, entry 1): The reaction



was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine *ent-A* as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/AcOEt 80:20) as a white solid in 63% yield and 95% ee. **HPLC analysis:** Daicel Chiralcel OD-H, Hex/*i*-PrOH 90:10, flow rate 0.7 ml/min, $\lambda = 254$ nm: $\tau_{major} = 16.32$ min, $\tau_{minor} = 21.18$ min.

HRMS calculated for C₁₉H₁₄O₂ 274.09938, found 274.09907. $[\alpha]_D^{30} = -118.9$ (c 0.45; DMSO, 95% ee). **¹H-NMR (400 MHz, DMSO *d*₆):** 70:30 mixture of conformational diastereoisomers *A* (major) and *B* (minor); δ 2.84 (dd, $J_a = 19.4$ Hz, $J_b = 5.0$ Hz, 1H_B), 2.95 (dd, $J_a = 18.6$ Hz, $J_b = 3.4$ Hz, 1H_A), 3.08 (dd, $J_a = 18.6$ Hz, $J_b = 8.2$ Hz, 1H_A), 3.19 (dd, $J_a = 19.2$ Hz, $J_b = 8.3$ Hz, 1H_B), 5.51 (m, 1H_A), 5.70 (m, 1H_B), 6.77 (d, $J = 9.0$ Hz, 1H_B), 7.0 (dd, $J_a = 16.7$ Hz, $J_b = 7.7$ Hz, 2H_A), 7.05-7.09 (m, 2H_B), 7.12-7.16 (m, 1H_B), 7.30 (d, $J = 9.1$ Hz, 1H_B), 7.32-7.41 (m, 2H_A), 7.43-7.52 (m, 1H_A + 1H_B), 7.53-7.58 (m, 1H_A + 1H_B), 7.67 (d, $J = 7.7$ Hz, 1H_A), 7.71 (d, $J = 8.7$ Hz, 1H_A), 7.75 (d, $J = 8.9$ Hz, 1H_B), 7.77 (d, $J = 8.1$ Hz, 1H_B), 7.81 (d, $J = 7.6$ Hz, 1H_B), 7.84 (d, $J = 8.2$ Hz, 1H_A), 8.38 (d, $J = 8.7$ Hz, 1H_A). 9.43 (s, 1H_A), 10.08 (s, 1H_B). **¹³C-NMR (100 MHz, DMSO *d*₆):** δ 34.55 (CH), 35.5 (CH), 42.8 (CH₂), 43.4 (CH₂), 117.6 (CH), 117.9 (C), 118.6 (CH), 119.6 (C), 122.0 (CH), 122.1 (CH), 122.2 (CH), 122.4 (CH), 122.4 (CH), 123.2 (CH), 125.3 (CH), 125.6 (CH), 125.8 (CH), 126.7 (CH), 126.8 (CH), 127.4 (CH), 128.1 (C), 124.4 (CH), 128.5 (CH), 128.9 (CH), 129.1 (CH), 131.6 (C), 133.6 (C), 134.4 (CH), 135.3 (CH), 136.3 (C), 136.6 (C), 152.6 (C), 153.6 (C), 158.8 (C), 159.6 (C), 205.2 (C), 206.3 (C).

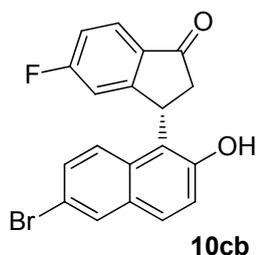
(S)-3-(2-hydroxynaphthalen-1-yl)-6-methyl-2,3-dihydro-1H-inden-1-one 10ba (Table 4, entry 3): The



reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/AcOEt 80:20) as a white solid in 97% yield and 90% ee. **HPLC analysis:** Daicel Chiralcel OJ-H, Hex/i-PrOH 90:10, flow rate 1.0 ml/min, $\lambda = 254$ nm: $\tau_{minor} = 26.63$ min, $\tau_{major} = 35.26$ min.

HRMS calculated for $C_{20}H_{16}O_2$ 288.11503, found 288.11535. $[\alpha]_D^{31} = -288.3$ (c 0.80; DMSO, 90% ee). **1H -NMR (400 MHz, DMSO- d_6):** 65:35 mixture of conformational diastereoisomers A (major) and B (minor); δ 2.35 (s, 3H_A), 2.38 (s, 3H_B), 2.83 (dd, $J_a = 19.3$ Hz, $J_b = 4.9$ Hz, 1H_B), 2.96 (dd, $J_a = 18.4$ Hz, $J_b = 3.6$ Hz, 1H_A), 3.06 (dd, $J_a = 18.6$ Hz, $J_b = 7.9$ Hz, 1H_A), 3.13-3.23 (m, 1H_B), 5.44 (m, 1H_A), 5.66 (m, 1H_B), 6.80 (d, $J = 8.4$ Hz, 1H_B), 6.91 (d, $J = 7.8$ Hz, 1H_A), 6.96 (d, $J = 7.8$ Hz, 1H_B), 7.00 (d, $J = 8.7$ Hz, 1H_A), 7.03-7.09 (m, 1H_B), 7.10-7.16 (m, 1H_B), 7.26-7.40 (m, 1H_A + 2H_B), 7.47 (s, 1H_A), 7.49-7.57 (m, 1H_A), 7.60 (s, 1H_B), 7.66-7.79 (m, 1H_A), 7.80-7.86 (m, 1H_A), 8.36 (d, $J = 8.5$ Hz, 1H_A), 9.41 (brs, 1H_A), 10.06 (brs, 1H_B). **^{13}C -NMR (100 MHz, DMSO- d_6):** mixture of conformers δ 20.5 (CH₃), 20.6 (CH₃), 30.7 (CH), 34.2 (CH), 43.1 (CH₂), 43.7 (CH₂), 117.7 (C), 117.9 (CH), 118.7 (CH), 119.8 (C), 122.0 (CH), 122.1 (CH), 122.2 (CH), 122.4 (CH), 122.5 (CH), 123.1 (CH), 125.0 (CH), 125.3 (CH), 125.8 (CH), 126.7 (CH), 128.1 (C), 128.3 (CH), 128.5 (CH), 128.8 (CH), 129.1 (CH), 131.7 (C), 133.6 (C), 135.4 (CH), 136.2 (C), 136.4 (CH), 136.5 (C), 136.9 (C), 153.0 (C), 153.6 (C), 156.2 (C), 157.0 (C), 205.1 (C), 206.3 (C).

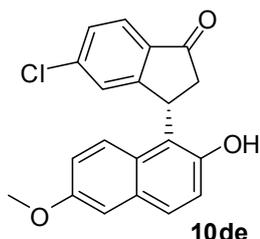
(S)-3-(6-bromo-2-hydroxynaphthalen-1-yl)-5-fluoro-2,3-dihydro-1H-inden-1-one 10cb (Table 4, entry



4): The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/AcOEt 70:30) as a white solid in 83% yield and 90% ee. **HPLC analysis:** Daicel Chiralcel OD-H, Hex/i-PrOH 95:5, flow rate 0.7 ml/min, $\lambda = 214$ nm: $\tau_{major} = 28.23$ min, $\tau_{minor} = 33.48$ min. **ESI-MS:** 393 (M + Na)⁺, 395 (M + Na)⁺. $[\alpha]_D^{30} = -205.4$ (c 0.91, DMSO, 90% ee). **1H -NMR (400 MHz, DMSO- d_6):** 73.5:26.5 mixture of conformational diastereoisomers A (major) and B (minor); δ 2.82 (dd, $J_a = 19.3$ Hz, $J_b = 5.1$ Hz, 1H_B), 2.90 (dd, $J_a = 18.5$ Hz, $J_b = 3.5$ Hz, 1H_A), 3.11 (dd, $J_a = 18.4$ Hz, $J_b = 8.1$ Hz, 1H_A), 3.21 (dd, $J_a = 19.2$ Hz, $J_b = 8.2$ Hz, 1H_B), 5.45 (m, 1H_A), 5.68 (m, 1H_B), 6.70 (d, $J = 9.3$ Hz, 1H_B), 6.74-6.84 (m, 1H_A + 1H_B), 7.07 (d, $J = 8.9$ Hz, 1H_A), 7.21 (m, 1H_A), 7.26-7.38 (m, 2H_B), 7.62 (dd, $J_a = 9.2$ Hz, $J_b = 2.2$ Hz, 1H_A), 7.70-7.78 (m, 2H_A + 1H_B), 7.88 (dd, $J_a = 8.6$ Hz, $J_b = 5.4$ Hz, 1H_B), 8.06 (d, $J = 2.1$ Hz, 1H_B), 8.10 (d, $J = 2.2$ Hz, 1H_A), 8.30 (d, $J = 9.4$ Hz, 1H_A), 9.75 (brs, 1H_A + 1H_B). **^{13}C -NMR (100 MHz, DMSO- d_6):** mixture of conformers δ 34.5 (CH), 35.6 (CH), 43.2 (CH₂), 43.7 (CH₂), 111.7 (d, CH, $J_{C-F} = 22.3$ Hz), 111.9 (d, CH, $J_{C-F} = 22.3$ Hz), 114.9 (d, CH, $J_{C-F} = 23.4$ Hz), 115.3 (C), 115.8 (d, CH, $J_{C-F} = 23.4$ Hz), 117.3 (C), 119.1 (CH), 119.4 (C), 119.8 (CH), 124.5 (CH), 124.7 (CH), 125.0 (d, CH, $J_{C-F} = 10.2$ Hz), 126.2 (d, CH, $J_{C-F} = 10.8$ Hz), 128.0 (CH), 128.7

(d, CH, $J_{C-F} = 34.6$ Hz), 129.4 (C), 129.5 (CH), 130.2 (CH), 130.3 (d, C, $J_{C-F} = 32.5$ Hz), 130.9 (C), 132.2 (C), 133.1 (C), 133.4 (d, C, $J_{C-F} = 1.5$ Hz), 153.6 (C), 154.3 (C), 161.9 (d, C, $J_{C-F} = 9.7$ Hz), 162.4 (d, C, $J_{C-F} = 9.6$ Hz), 165.1 (C), 165.6 (C), 167.6 (C), 168.1(C), 203.1(C), 204.2(C).

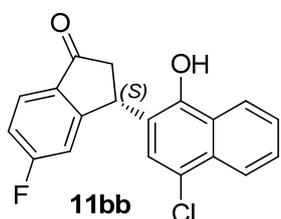
(S)-5-chloro-3-(2-hydroxy-6-methoxynaphthalen-1-yl)-2,3-dihydro-1H-inden-1-one 10de (Table 4, entry 5):



The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/Et₂O 70:30) as a white solid in 84% yield and 81% ee. **HPLC analysis:** Daicel Chiralpak AD-H, Hex/*i*-PrOH 90:10, flow rate 0.75 ml/min, $\lambda = 254$ nm: $\tau_{minor} = 23.19$ min, $\tau_{major} = 25.66$ min. **HRMS** calculated for C₂₀H₁₅O₃Cl 338.070974, found 338.07133. $[\alpha]_D^{30}$ determined on the product obtained in the reaction with 9-

amino(9-deoxy)*epi*-quinine **A** as catalyst = +141.5 (c 0.9; DMSO, 78% ee). **¹H-NMR (400 MHz, DMSO-*d*₆):** 75:25 mixture of conformational diastereoisomers *A* (major) and *B* (minor); δ 2.83 (dd, $J_a = 19.3$ Hz, $J_b = 4.9$ Hz, 1H_B), 2.93 (dd, $J_a = 18.6$ Hz, $J_b = 3.5$ Hz, 1H_A), 3.10 (dd, $J_a = 18.6$ Hz, $J_b = 8.1$ Hz, 1H_A), 3.21 (dd, $J_a = 19.4$ Hz, $J_b = 8.4$ Hz, 1H_B), 3.75 (s, 3H_B), 3.86 (s, 3H_A), 5.45 (m, 1H_A), 5.68 (m, 1H_B), 6.67 (d, $J = 9.3$ Hz, 1H_B), 6.81 (dd, $J_a = 9.3$ Hz, $J_b = 2.6$ Hz, 1H_B), 6.95-7.04 (m, 2H_A + 1H_B), 7.17-7.32 (m, 2H_A + 2H_B), 7.43 (dd, $J_a = 8.2$ Hz, $J_b = 1.9$ Hz, 1H_A), 7.51 (m, 1H_B), 7.60-7.71 (m, 2H_A + 1H_B), 7.81 (d, $J = 8.3$ Hz, 1H_B), 8.26 (d, $J = 9.5$ Hz, 1H_A), 9.26 (s, 1H_A), 9.88 (s, 1H_B). **¹³C-NMR (100 MHz, DMSO-*d*₆):** mixture of conformers δ 34.5 (CH), 35.6 (CH), 43.1 (CH₂), 43.6 (CH₂), 54.9 (CH), 55.1 (CH), 107.0 (CH), 108.0 (CH), 117.2 (C), 118.1 (CH), 118.4 (CH), 119.0 (CH), 119.1 (CH), 119.4 (C), 123.6 (C), 123.7 (CH), 124.0 (CH), 125.0 (CH), 125.1 (C), 125.2 (CH), 126.5 (C), 127.3 (CH), 127.4 (CH), 127.9 (CH), 128.0 (CH), 128.7 (C), 129.1 (C), 130.2 (C), 135.2 (C), 135.4 (C), 139.2 (C), 140.2 (C), 151.2 (C), 152.0 (C), 154.4 (C), 154.9 (C), 160.8 (C), 161.4 (C), 203.8 (C), 204.9 (C).

(S)-3-(4-chloro-1-hydroxynaphthalen-2-yl)-5-fluoro-2,3-dihydro-1H-inden-1-one 11bb (Table 4, entry 6):

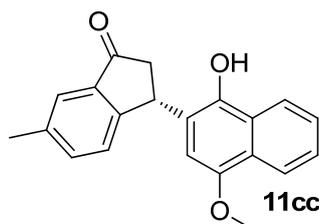


The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/Et₂O 60:40) as a white solid in 60% yield and 90% ee. **HPLC analysis:** Daicel Chiralcel OD-H, Hex/*i*-PrOH 95:5, flow rate 0.75 ml/min, $\lambda = 214$ nm: $\tau_{minor} = 21.63$ min, $\tau_{major} = 24.11$ min. **ESI-MS:** 327 (M + 1)⁺, 329 (M + 1)⁺, 349 (M + Na)⁺, 351 (M + Na)⁺.

$[\alpha]_D^{30} = -119.6$ (c 0.78; DMSO, 90% ee). **¹H-NMR (400 MHz, DMSO-*d*₆):** δ 2.78 (dd, $J_a = 9.0$ Hz, $J_b = 4.0$ Hz, 1H), 3.24 (dd, $J_a = 19$ Hz, $J_b = 8.2$ Hz, 1H), 5.12 (m, 1H), 7.09 (dd, $J_a = 8.9$ Hz, $J_b = 2.1$ Hz, 1H), 7.24-7.34 (m, 2H), 7.55-7.68 (m, 2H), 7.80 (dd, $J_a = 8.5$ Hz, $J_b = 5.3$ Hz, 1H), 8.06 (m, 1H), 8.30 (m, 1H), 9.83 (s, 1H). **¹³C-NMR (100 MHz, DMSO-*d*₆):** δ 38.4 (CH), 44.7 (CH₂), 119.9 (d, CH, $J_{C-F} =$

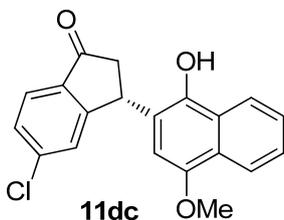
22.1 Hz), 115.9 (d, CH, $J_{C-F} = 23.7$ Hz), 121.4 (C), 122.8 (CH), 123.6 (CH), 124.7 (C), 125.4 (d, CH, $J_{C-F} = 10.7$ Hz), 126.1 (CH), 126.6 (CH), 126.7 (C), 127.3 (CH), 129.8 (C), 133.3 (d, C, $J_{C-F} = 1.3$ Hz), 149.5 (C), 161.0 (d, C, $J_{C-F} = 9.7$ Hz), 165.2 (C), 167.8 (C), 203.5 (C).

(S)-3-(1-hydroxy-4-methoxynaphthalen-2-yl)-6-methyl-2,3-dihydro-1H-inden-1-one 11cc (Table 4, entry 7): The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/Et₂O 75:25) as a white solid in 92% yield and 94% ee. **HPLC analysis:** Daicel Chiralpak AD-H, Hex/i-PrOH 90:10, flow rate 0.75 ml/min, $\lambda = 214$ nm: $\tau_{major} = 18.33$ min, $\tau_{minor} = 20.09$ min. **HRMS** calculated for C₂₁H₁₈O₃ 318.12559, found 318.12592. **¹H-NMR (400 MHz, DMSO-*d*₆):** mixture of conformers δ 2.78



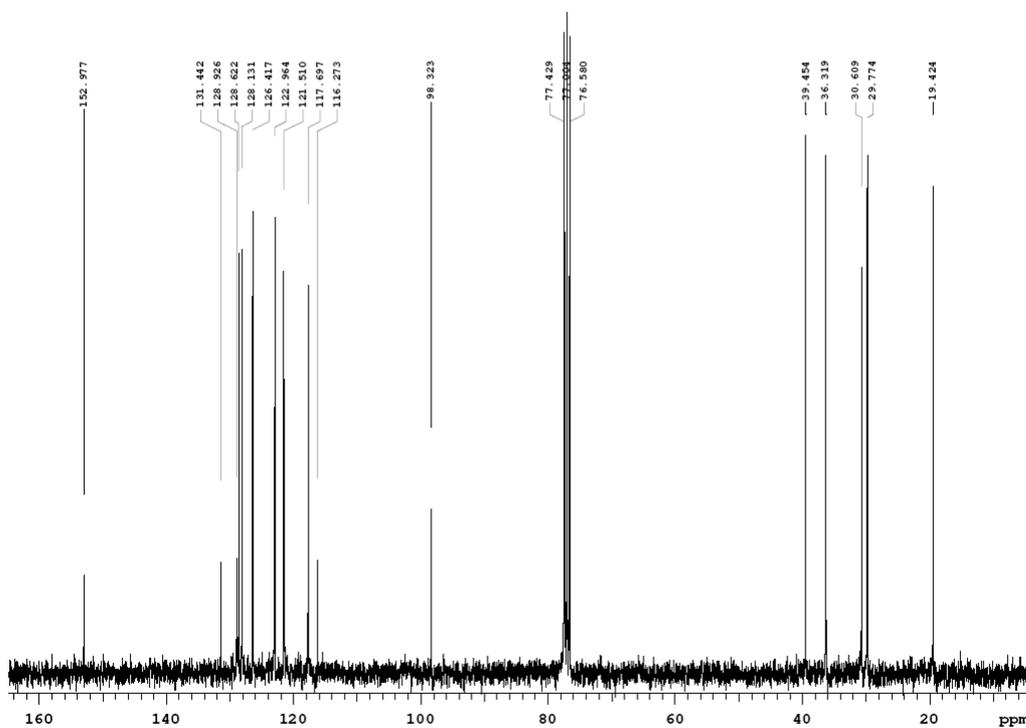
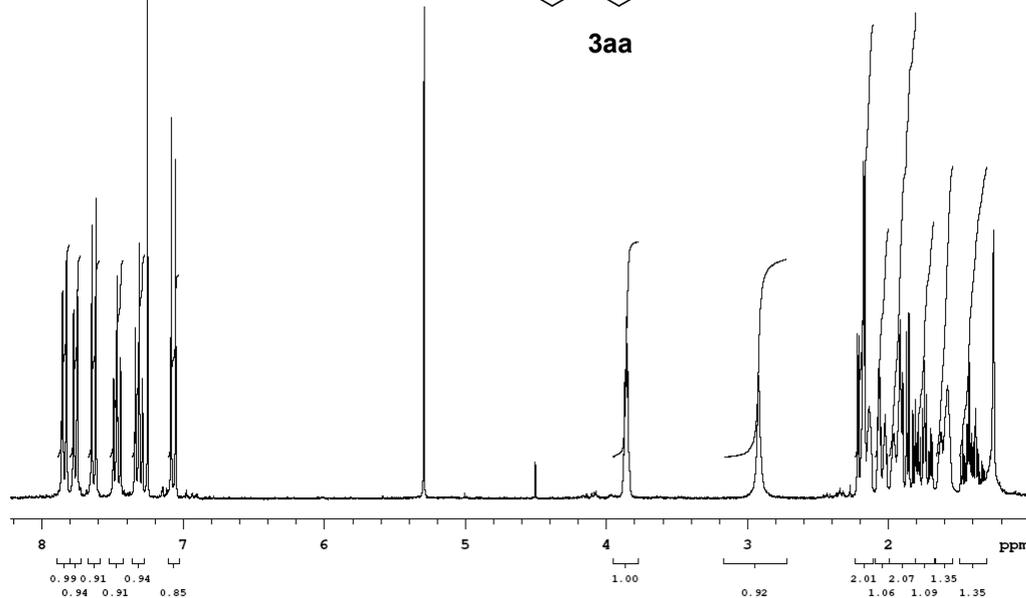
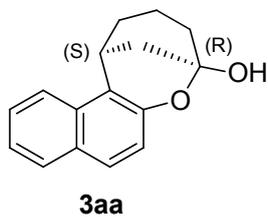
(dd, $J_a = 18.8$ Hz, $J_b = 3.9$ Hz, 1H), 3.18 (dd, $J_a = 19.0$ Hz, $J_b = 8.0$ Hz, 1H), 3.75 (s, 3H), 5.11 (m, 1H), 6.45 (s, 1H), 7.17 (d, $J = 7.8$ Hz, 1H), 7.42-7.54 (m, 4H), 8.04 (m, 1H), 8.16 (m, 1H), 8.85 (s, 1H). **¹³C-NMR (100 MHz, DMSO-*d*₆):** δ 20.6 (CH₃), 38.1 (CH), 44.9 (CH₂), 55.5 (CH₃), 104.3 (CH), 121.3 (CH), 122.1 (CH), 122.5 (CH), 124.4 (C), 124.5 (C), 125.0 (CH), 125.7 (CH), 126.3 (CH), 126.7 (C), 136.0 (CH), 136.6 (C), 137.0 (C), 143.0 (C), 148.6 (C), 155.7 (C), 205.6 (C).

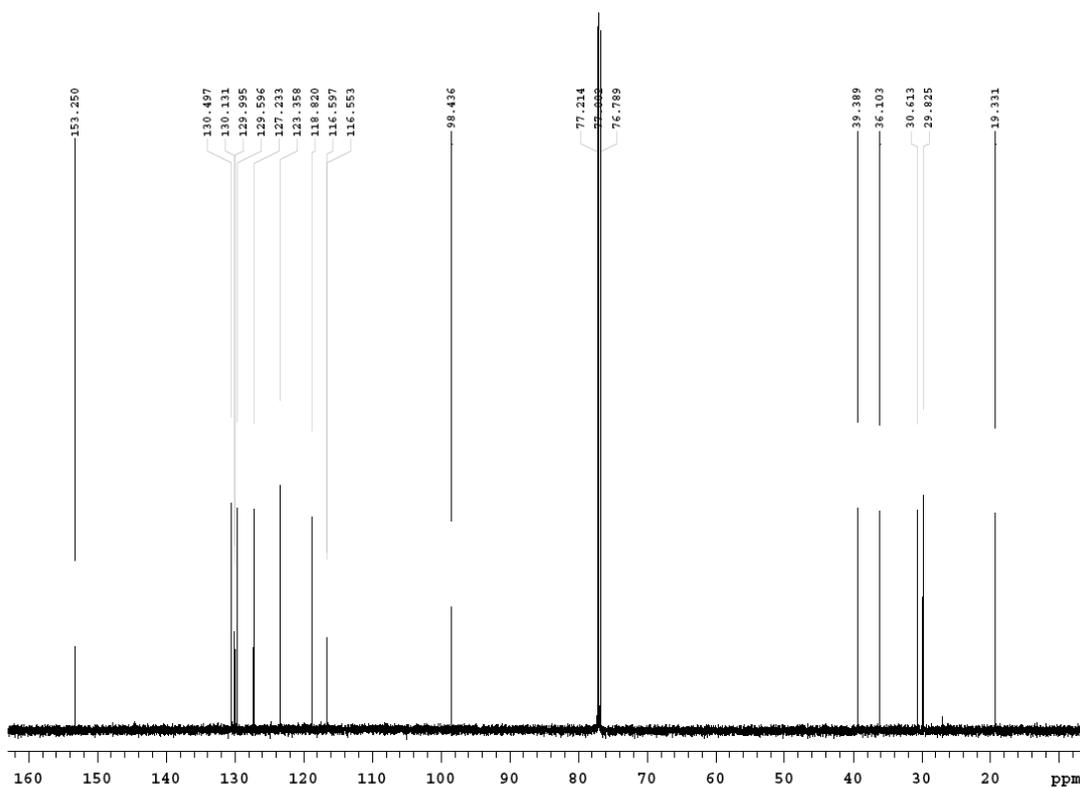
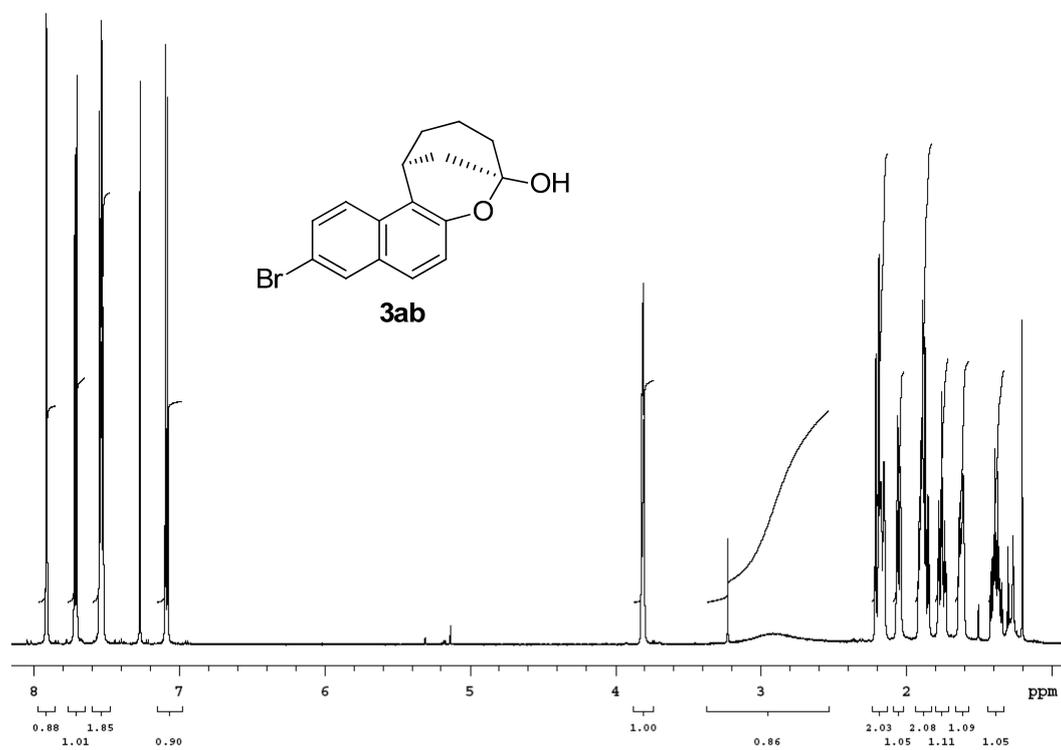
(S)-5-chloro-3-(1-hydroxy-4-methoxynaphthalen-2-yl)-2,3-dihydro-1H-inden-1-one 11dc (Table 4 entry 8): The reaction was performed following the general procedure on using 9-amino(9-deoxy)*epi*-quinidine **ent-A** as catalyst. The title compound was isolated by flash chromatography on silica gel (eluent mixture Hexane/Et₂O 70:30) as a white solid in 83% yield and 90% ee. **HPLC analysis:** Daicel Chiralcel OD-H, Hex/i-PrOH 95:5, flow rate 0.75 ml/min, $\lambda = 214$ nm: $\tau_{minor} = 32.18$ min, $\tau_{major} = 38.27$ min. **ESI-MS:** 361 (M + Na)⁺, 363 (M + Na)⁺.



¹H-NMR (400 MHz, DMSO-*d*₆): δ 2.90 (dd, $J_a = 19.0$ Hz, $J_b = 4.0$ Hz, 1H), 3.20 (dd, $J_a = 19.0$ Hz, $J_b = 8.1$ Hz, 1H), 3.81 (s, 3H), 5.11 (m, 1H), 6.59 (s, 1H), 7.40-7.58 (m, 3H), 7.72 (d, $J = 8.2$ Hz, 1H), 8.06 (d, $J = 8.2$ Hz, 1H), 8.15 (d, $J = 8.3$ Hz, 1H), 8.88 (s, 1H). **¹³C-NMR (100 MHz, DMSO-*d*₆):** δ 44.3 (CH₂), 38.1 (CH), 55.6 (CH₃), 104.8 (CH), 121.4 (CH), 122.1 (CH), 123.8 (C), 124.4 (CH), 124.7 (C), 125.2 (CH), 126.2 (CH), 126.8 (C), 128.0 (CH), 135.2 (C), 139.7 (C), 143.1 (C), 148.7 (C), 160.3 (C), 204.4 (C).

^1H - and ^{13}C -NMR-Traces



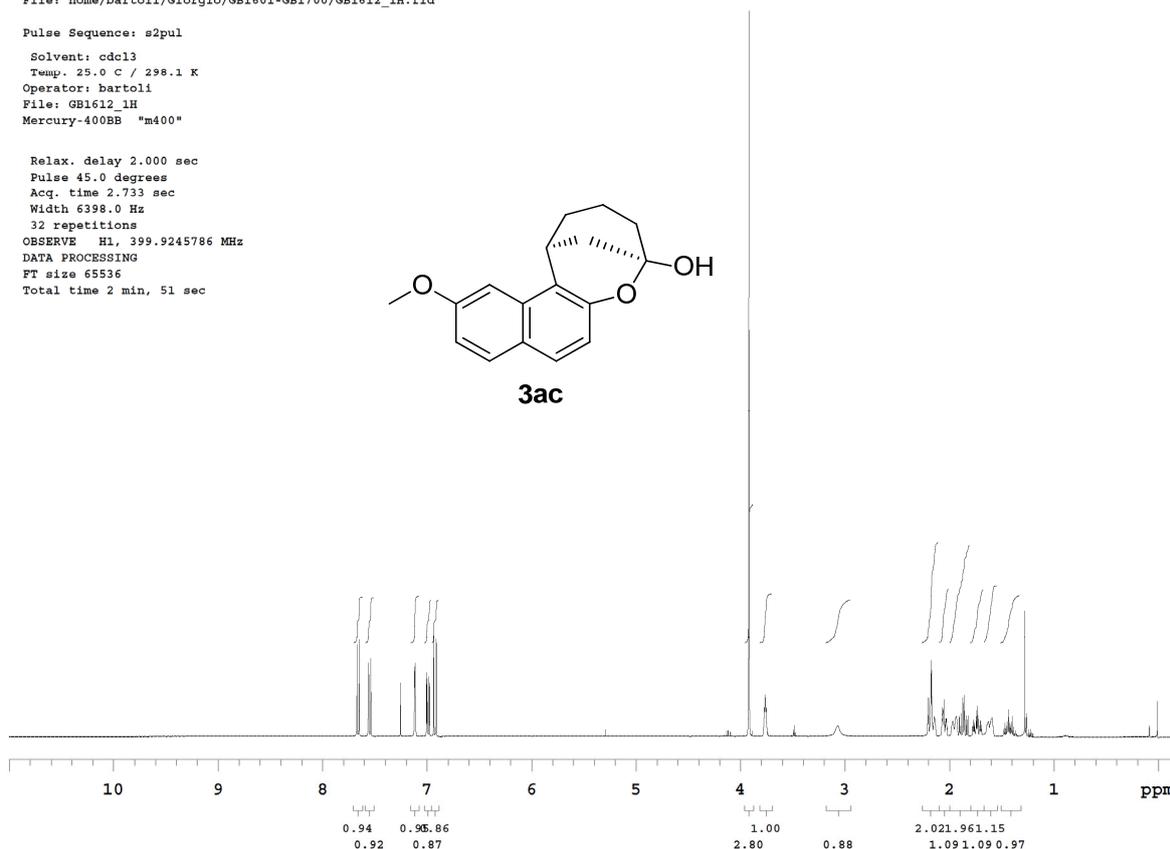
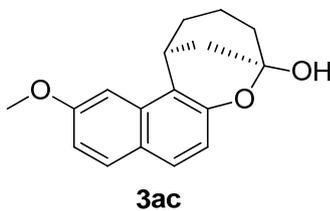


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1612_1H.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1612_1H
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.9245786 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 51 sec

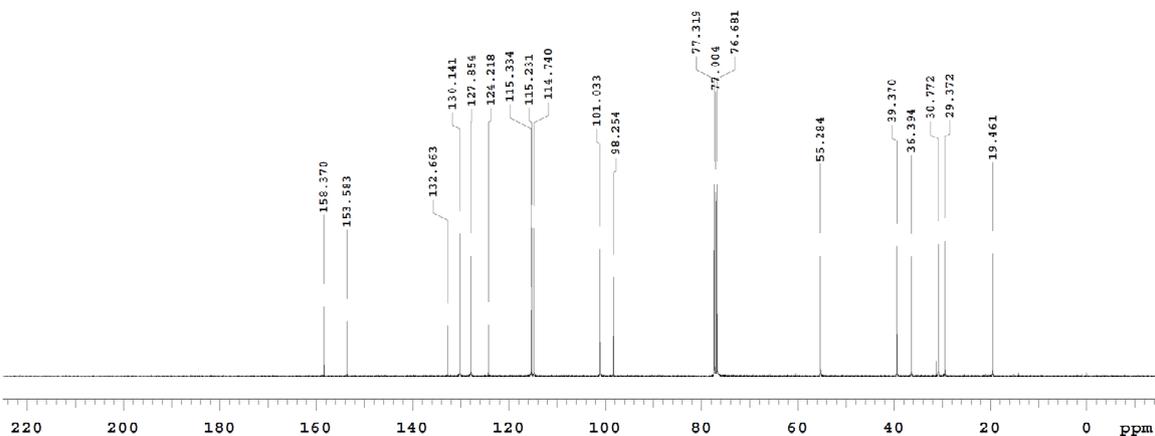


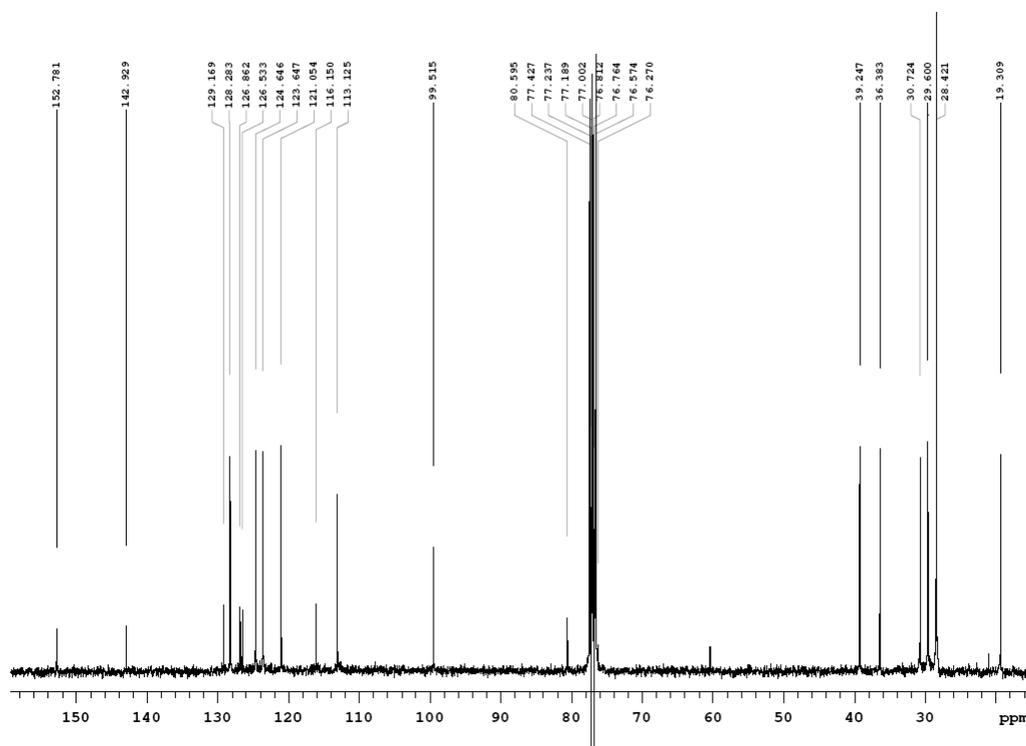
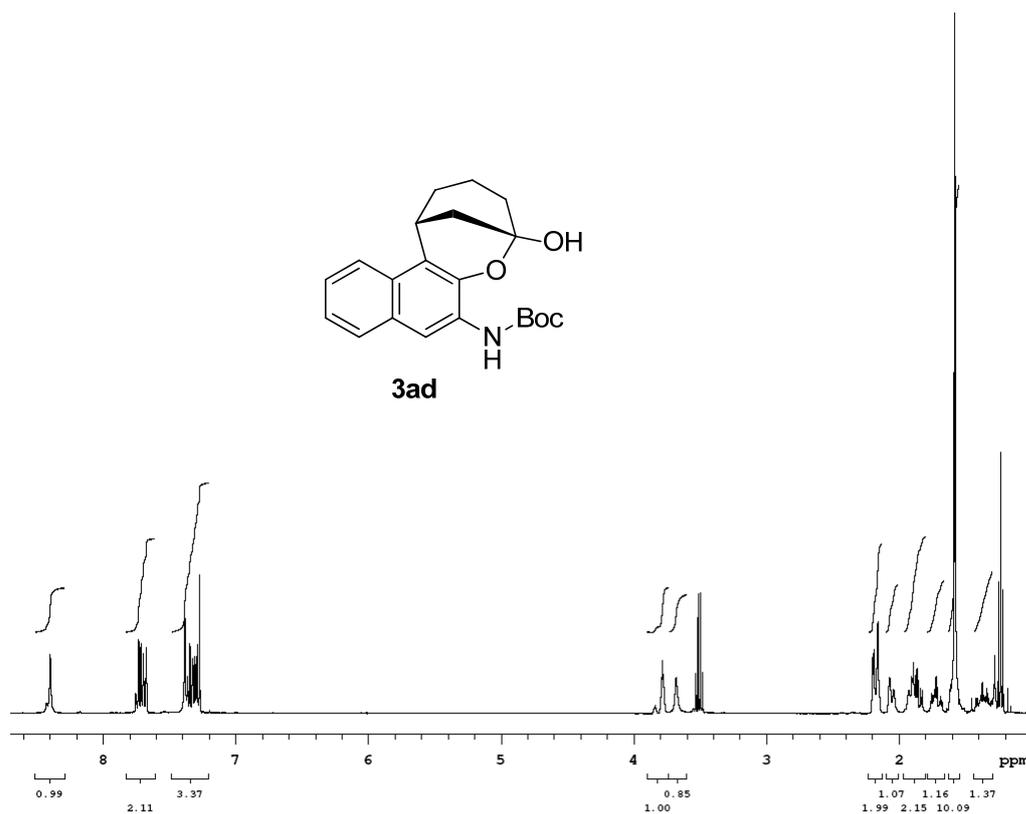
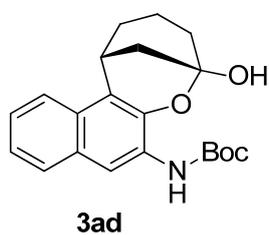
Std Carbon experiment

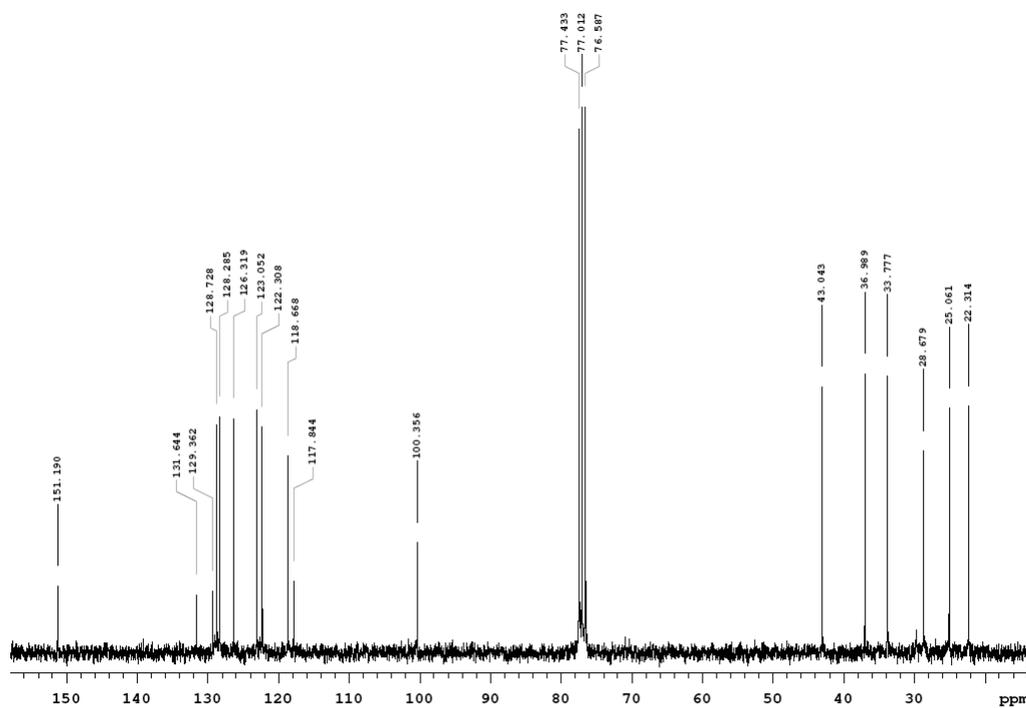
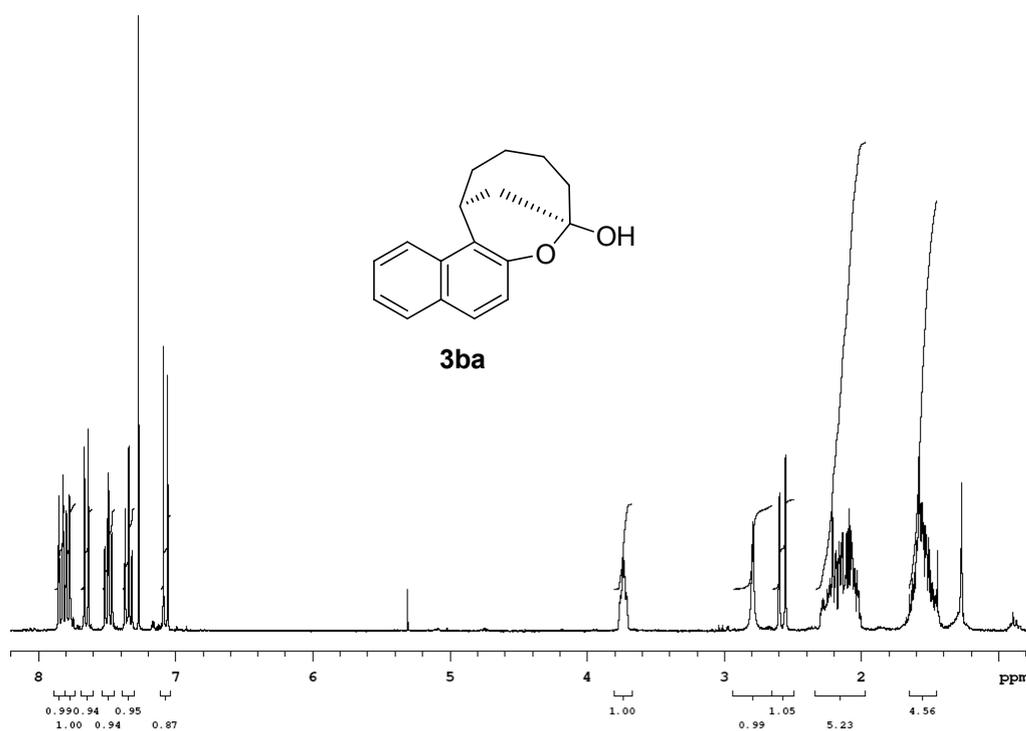
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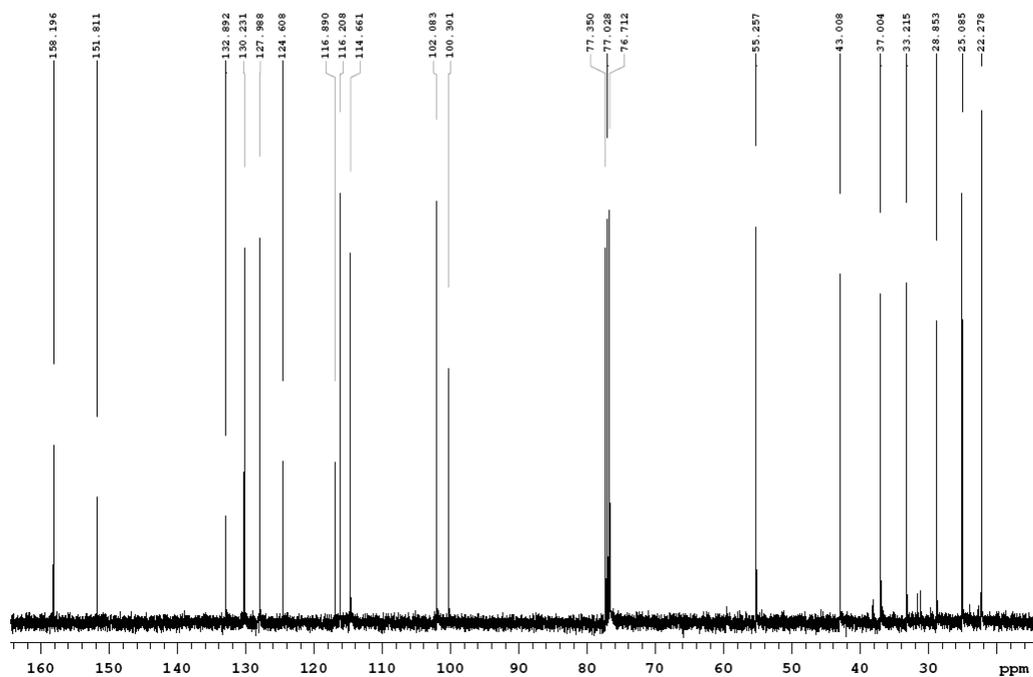
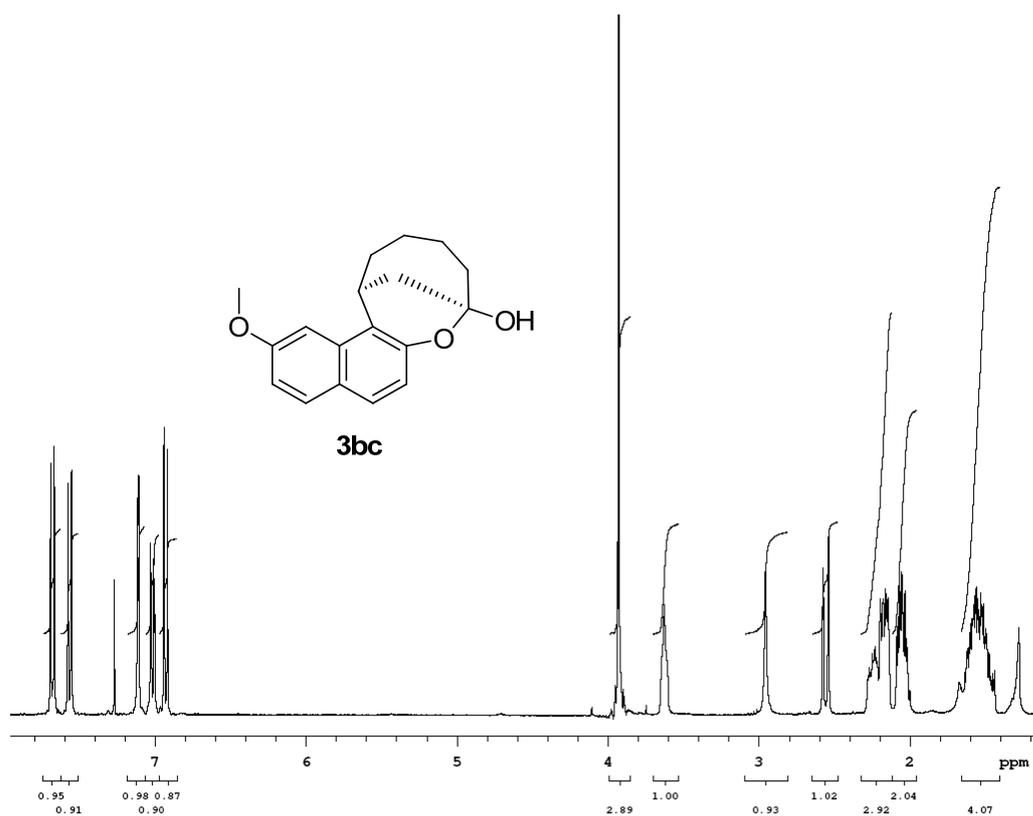
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1612_13C
Mercury-400BB "m400"

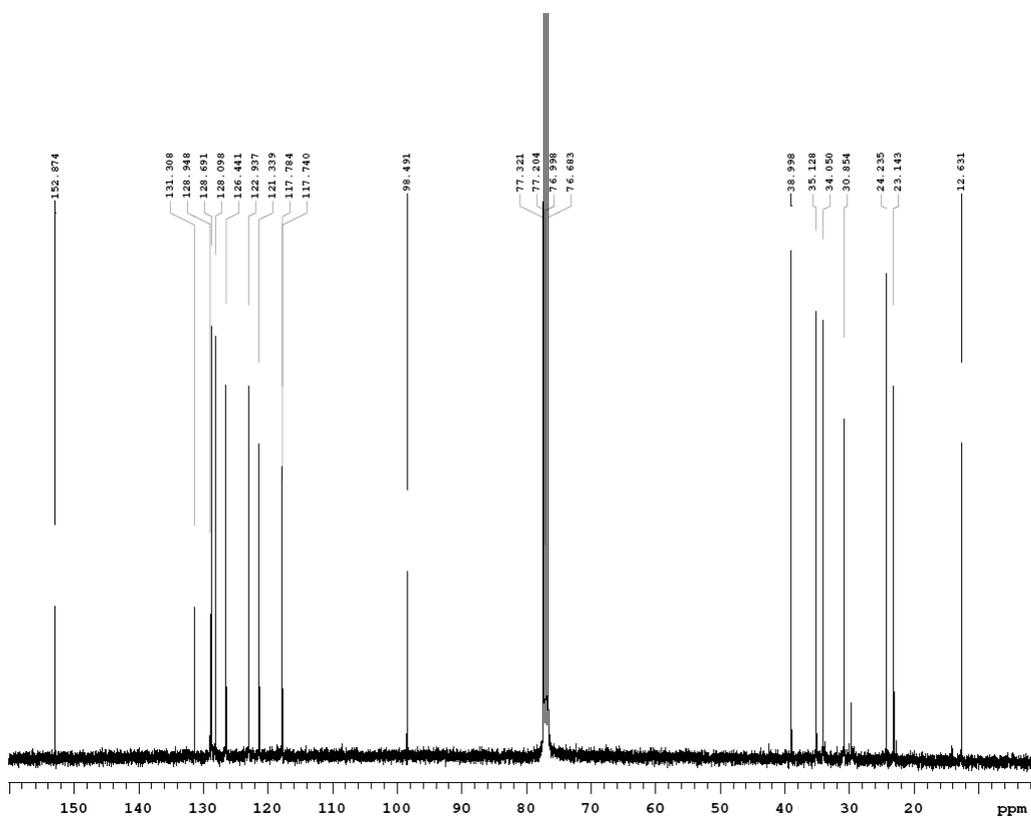
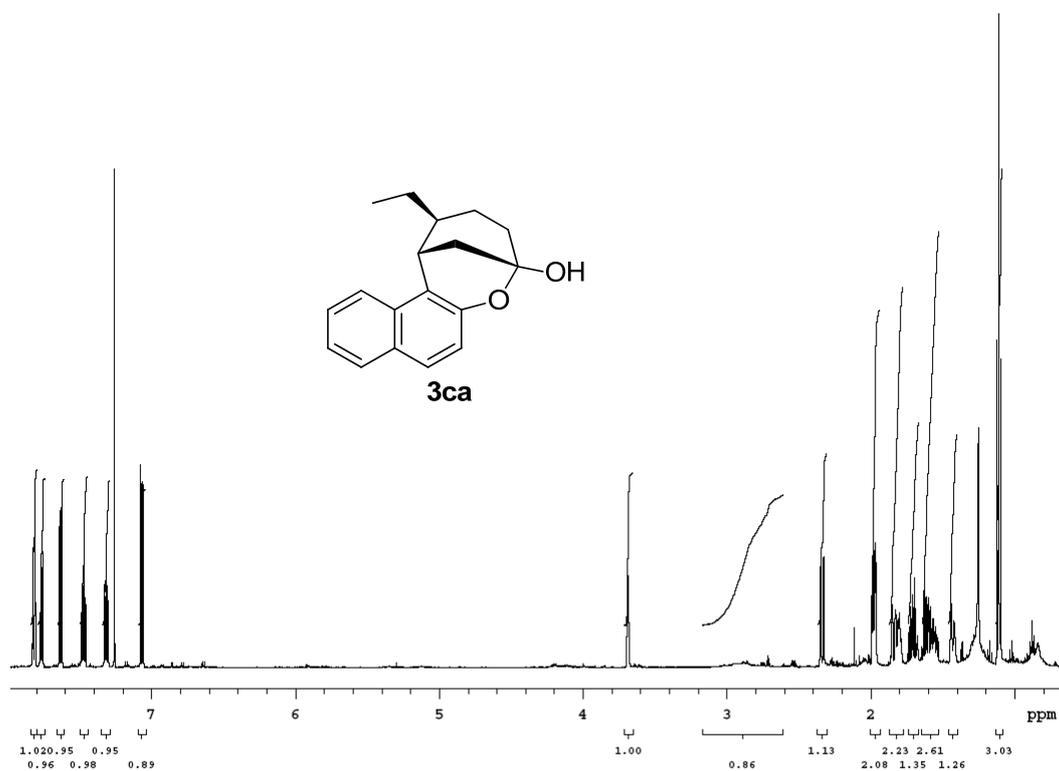
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
15728 repetitions
OBSERVE C13, 100.5611169 MHz
PROBHDPR H1, 399.9265566 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 hr, 52 min, 19 sec

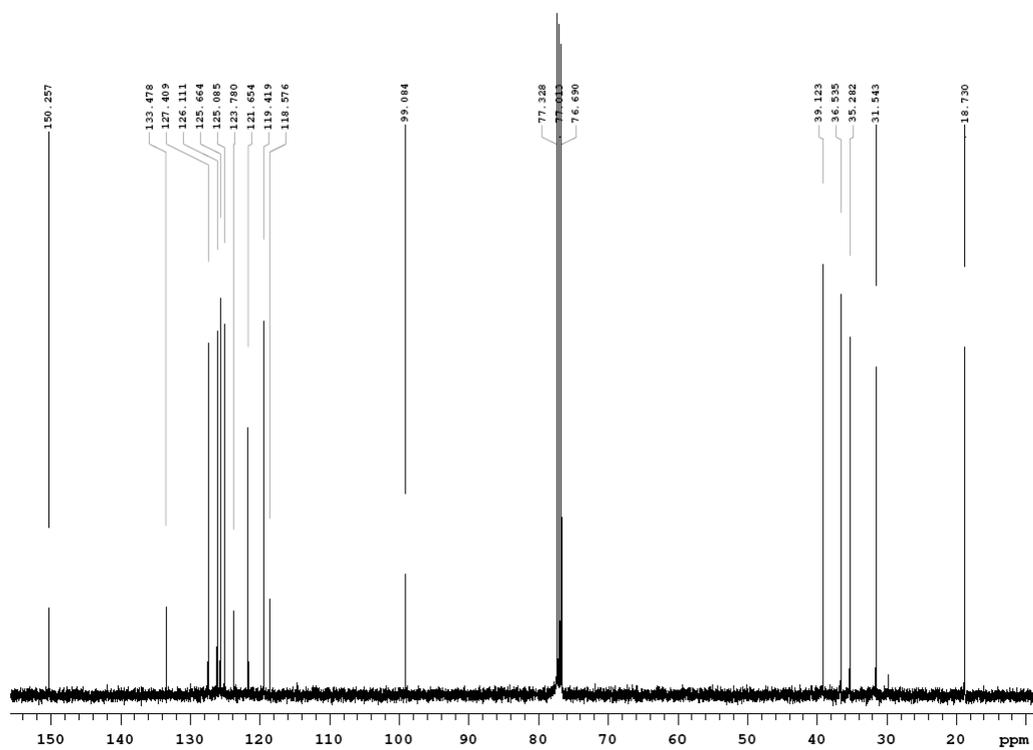
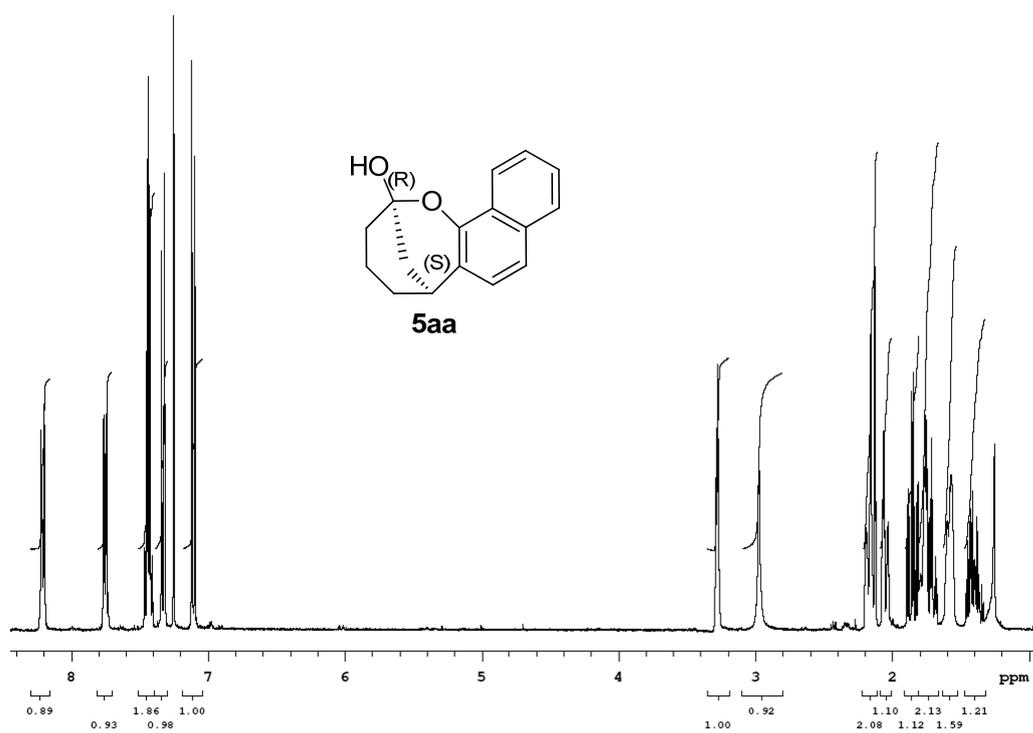










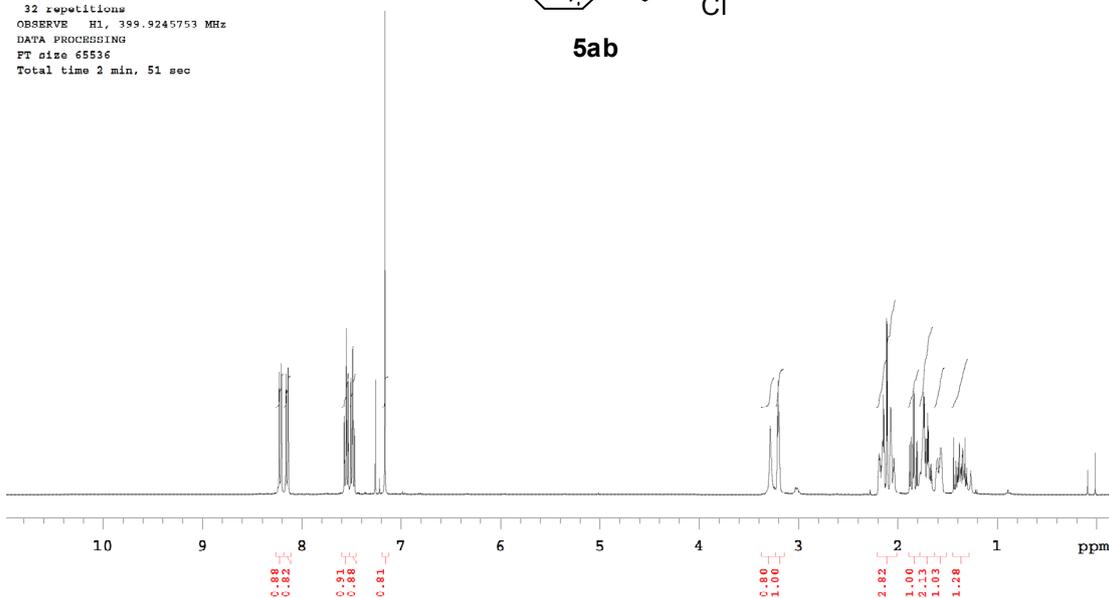
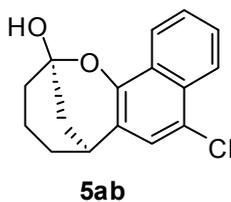


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1613_1H.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1613_1H
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.9245753 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 51 sec

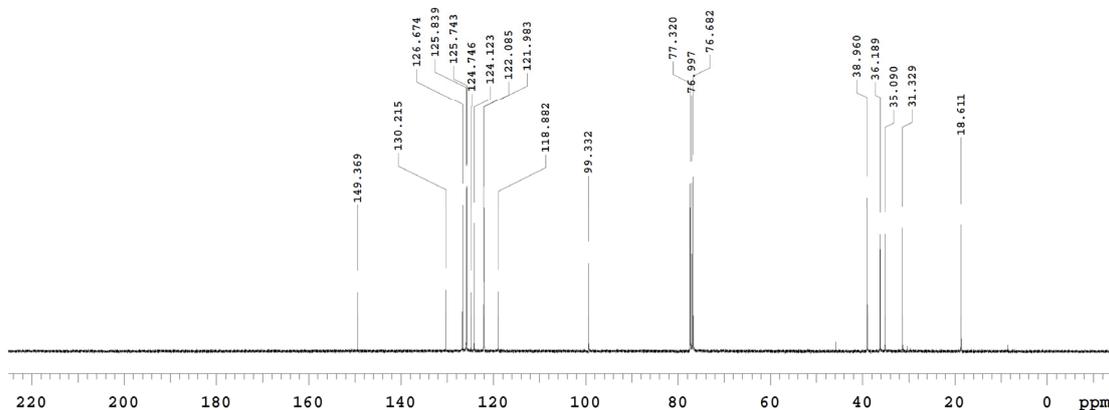


Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1613_13C.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1613_13C
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1392 repetitions
OBSERVE C13, 100.5611176 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 9 hr, 56 min, 10 sec



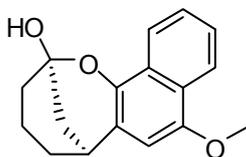
Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1611_1H.fid

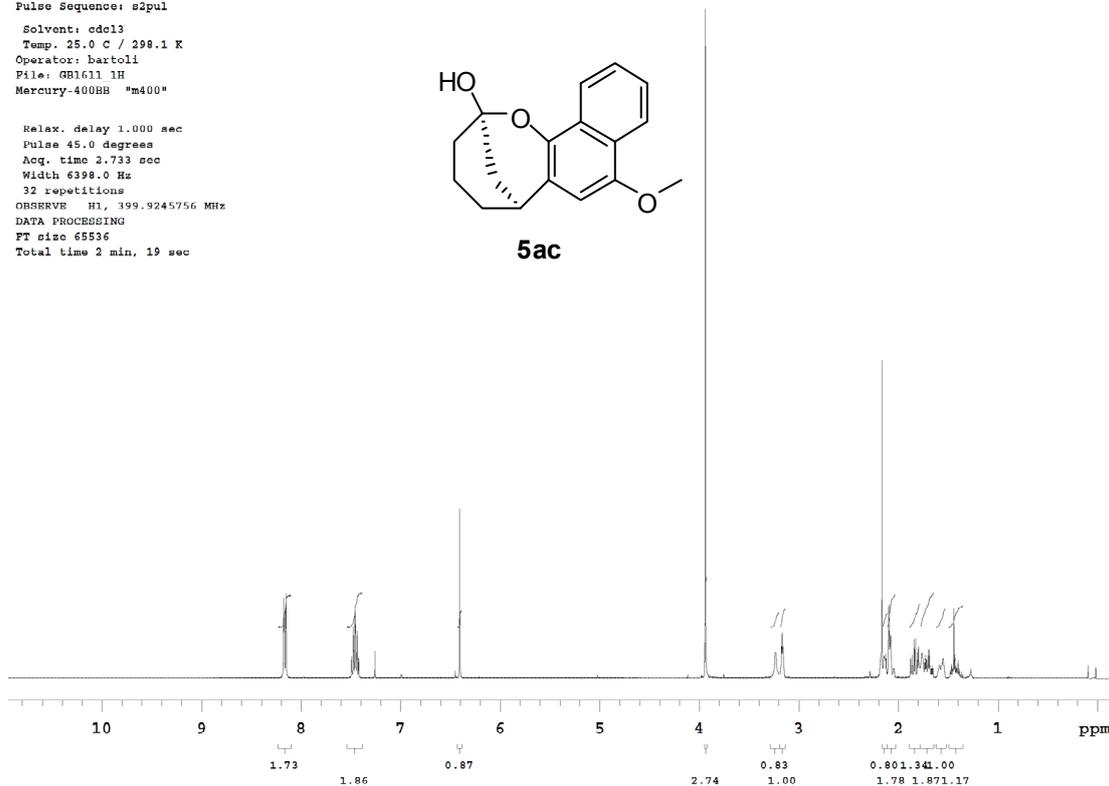
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1611_1H
Mercury-400BB "m400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.9245756 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 19 sec



5ac



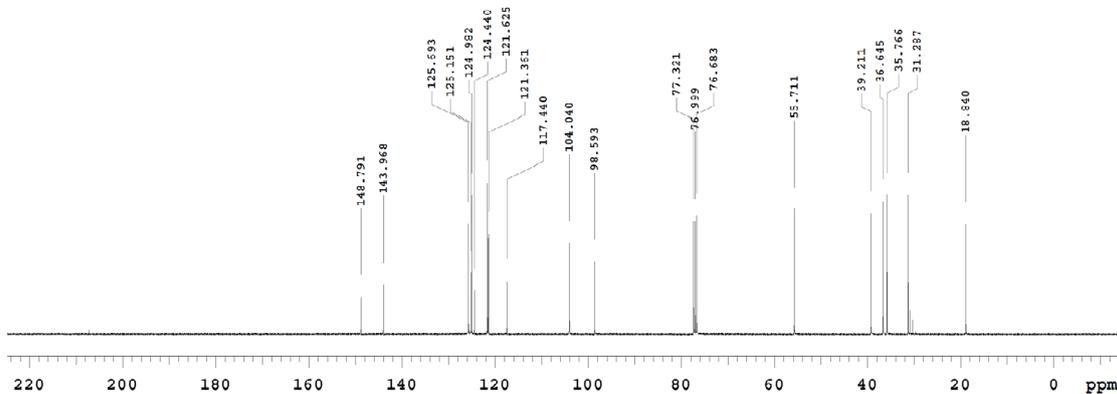
Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1611_13C.fid

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1611_13C
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
2080 repetitions
OBSERVE C13, 100.5611188 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 9 hr, 56 min, 10 sec

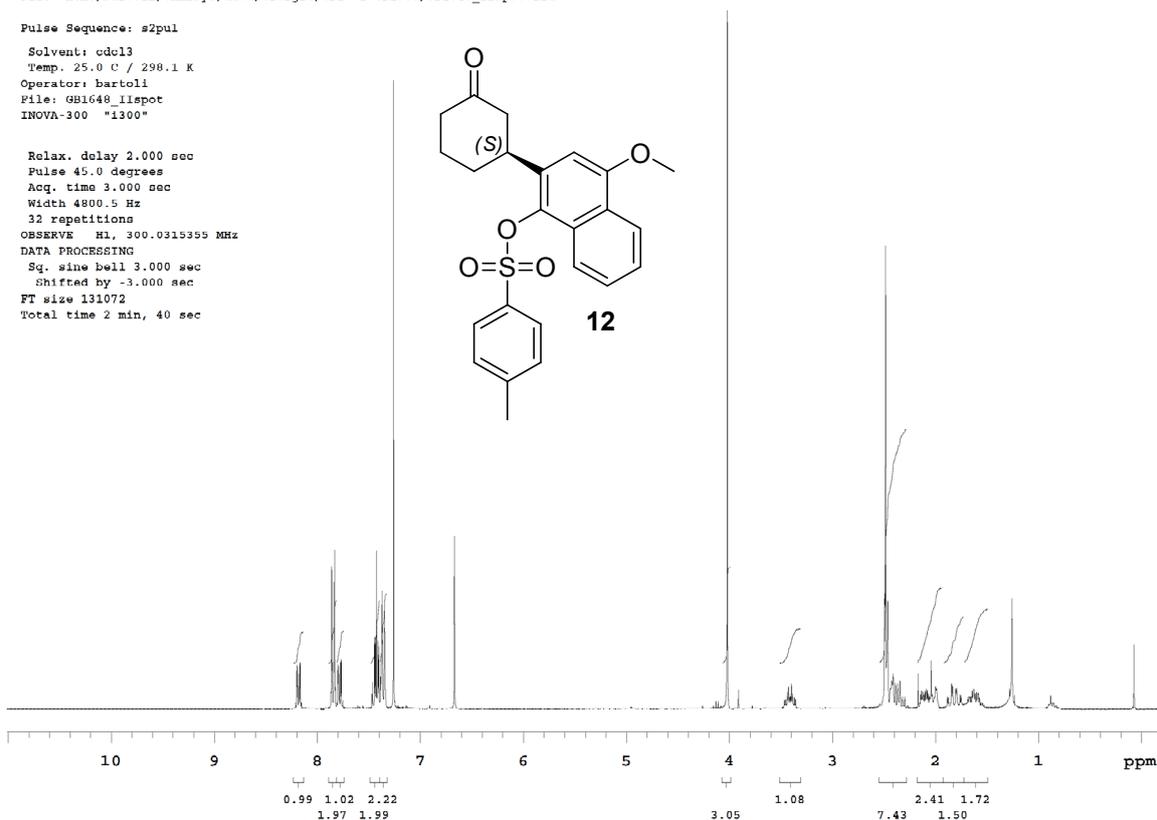


Standard Proton Parameters - i300@fci.unibo.it

File: home/bartoli/vnmrsvs/data/Giorgio/GB1601-GB1700/GB1648_IIspot.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1648_IIspot
INOVA-300 "i300"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 4800.5 Hz
32 repetitions
OBSERVE H1, 300.0315395 MHz
DATA PROCESSING
Sq. sine bell 3.000 sec
Shifted by -3.000 sec
FT size 131072
Total time 2 min, 40 sec

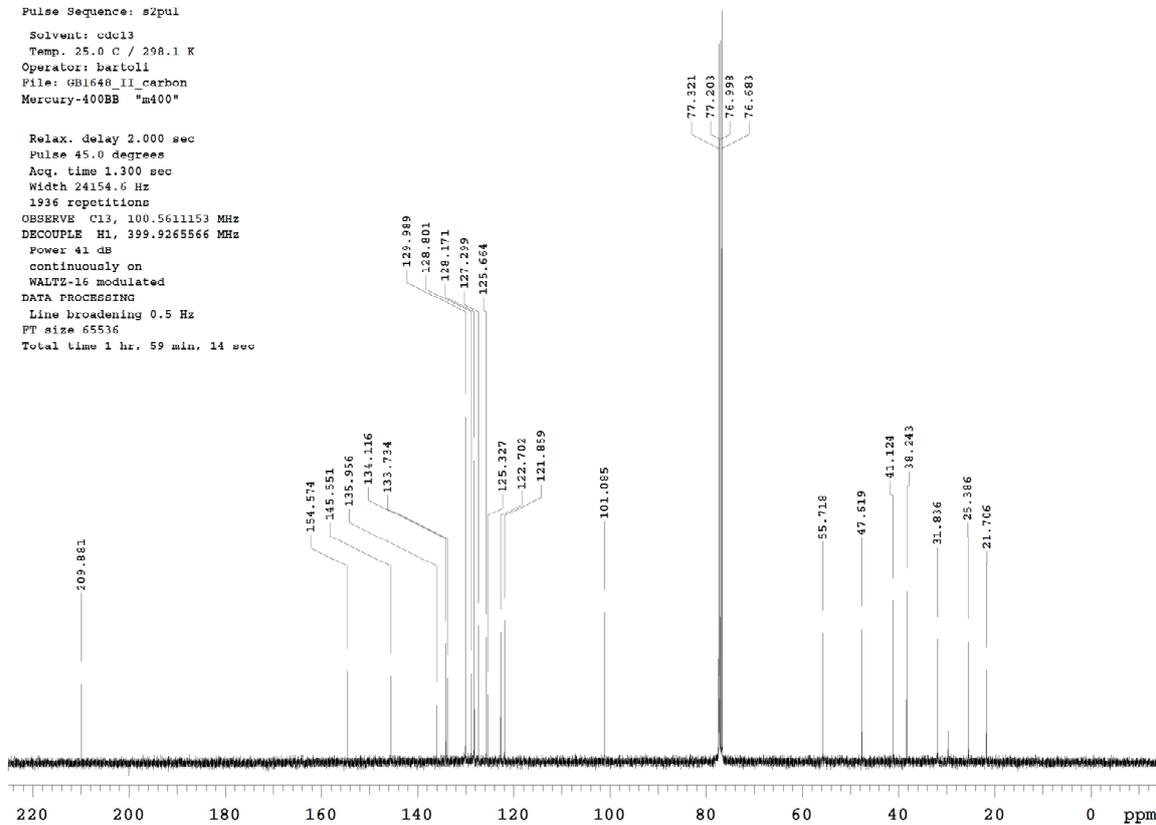


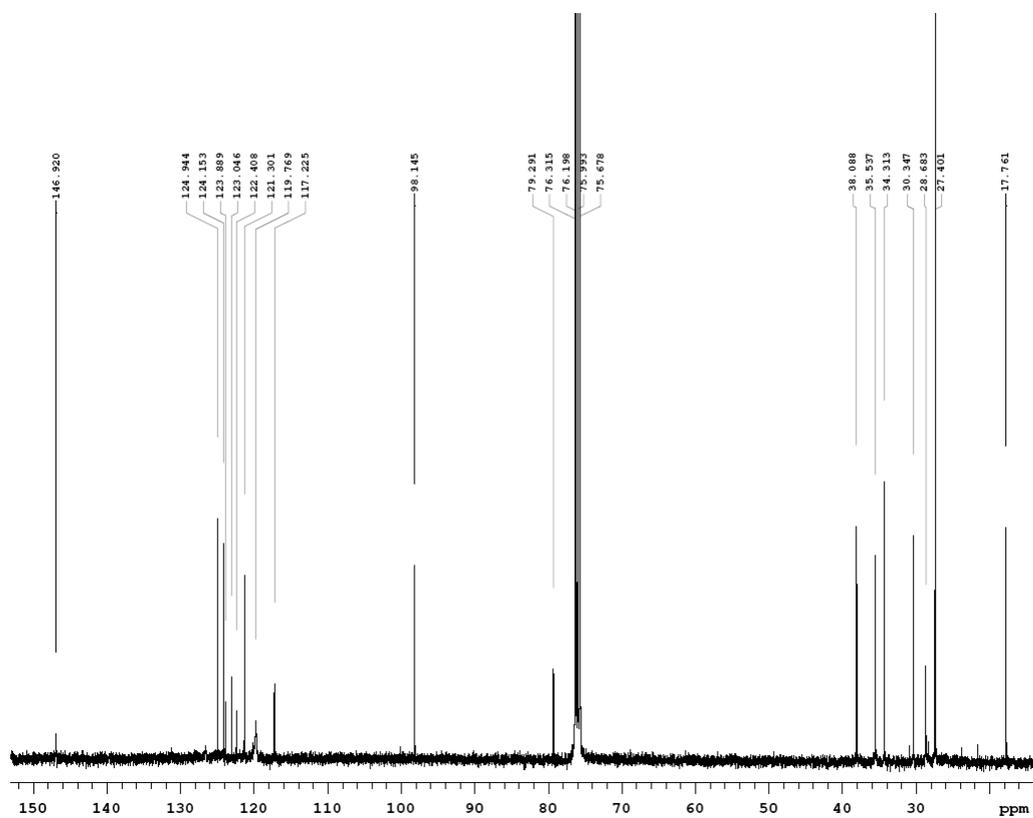
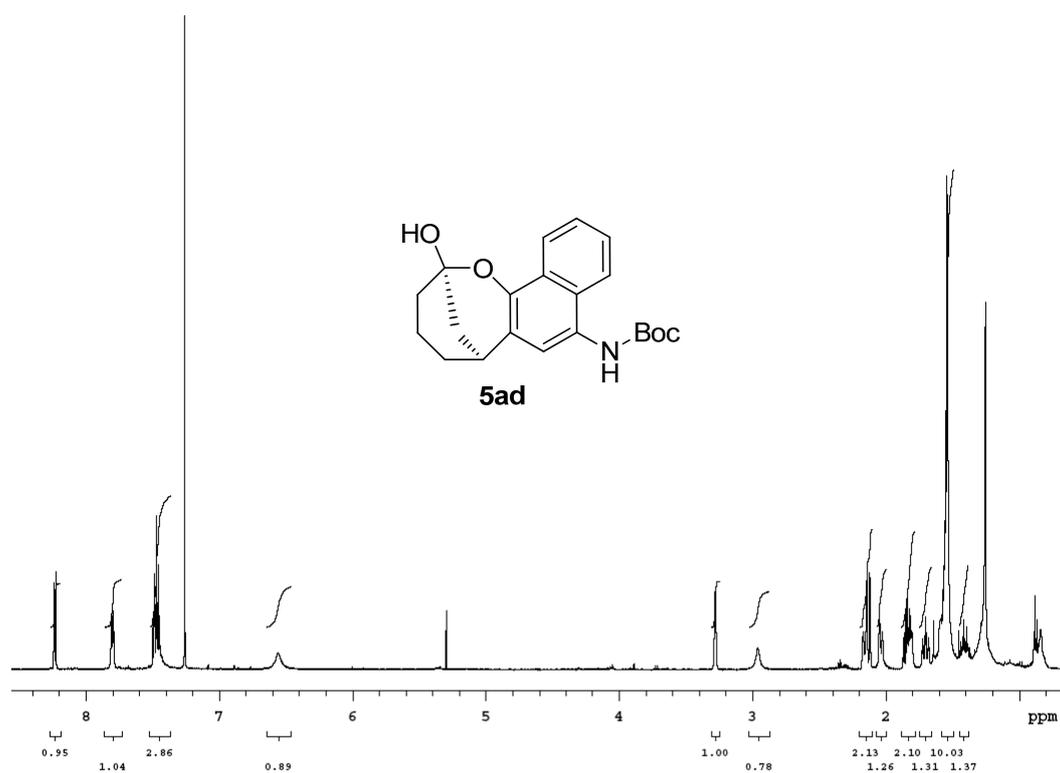
Std Carbon experiment

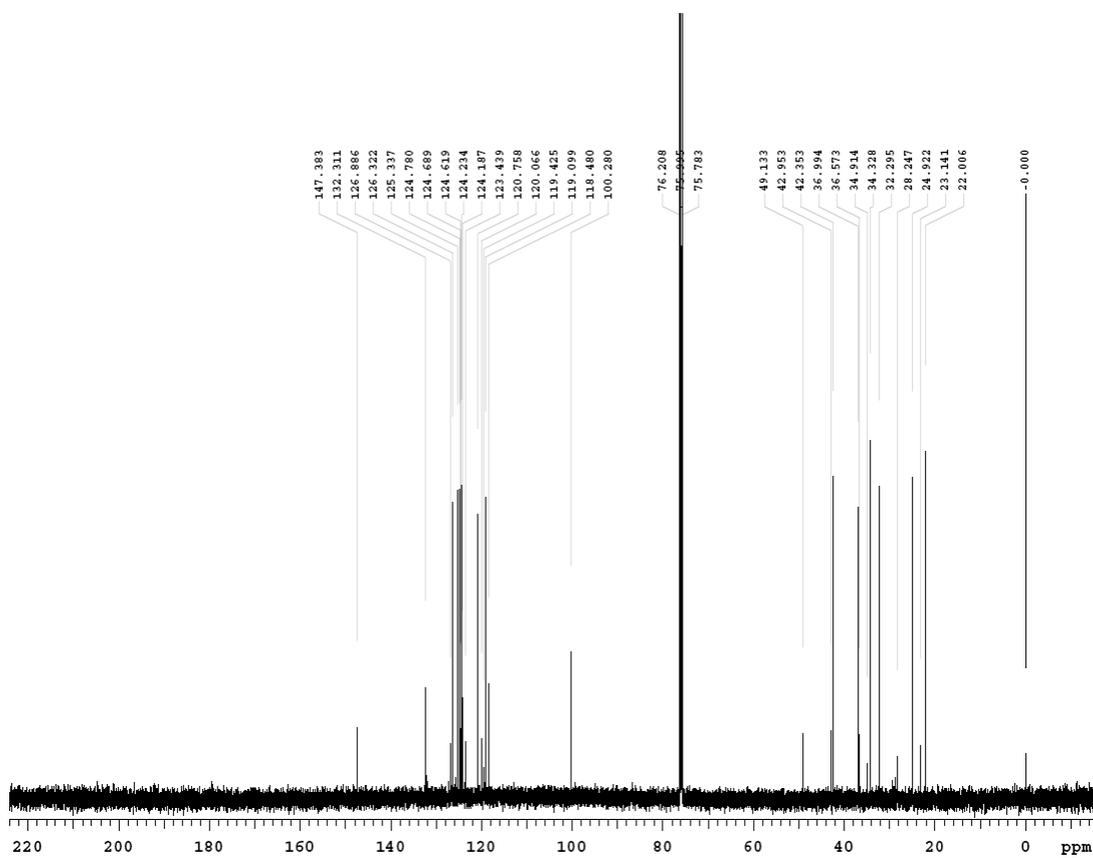
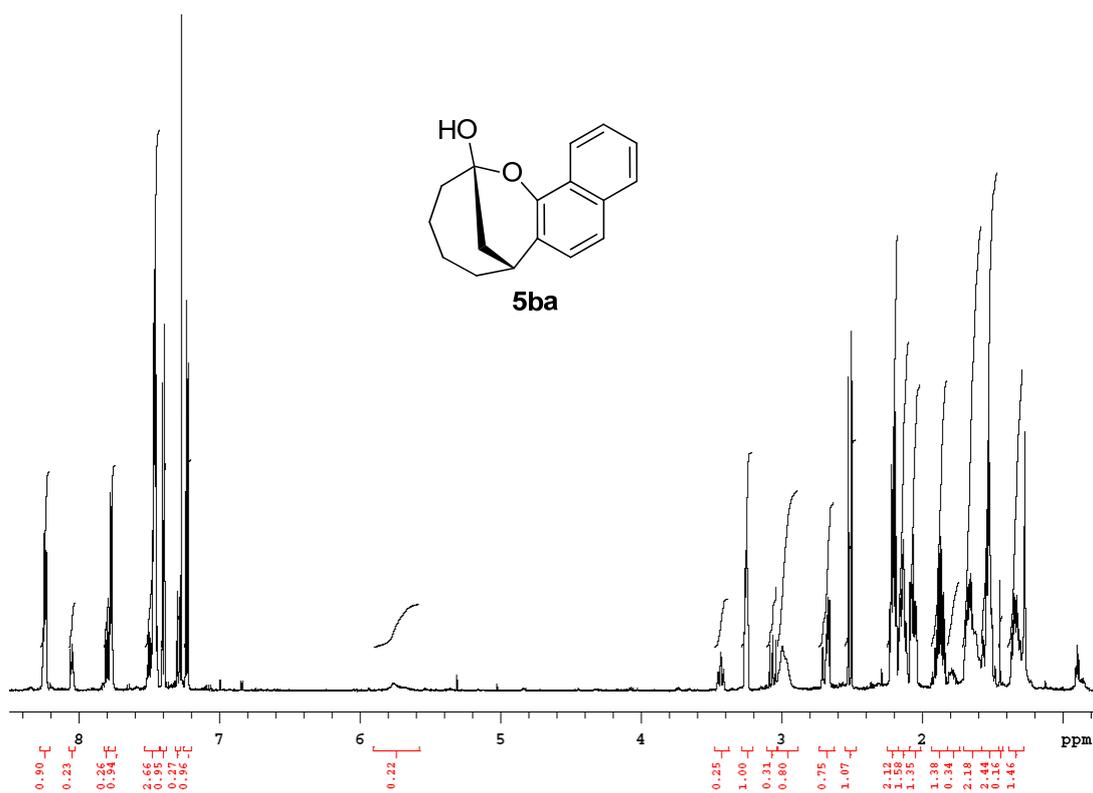
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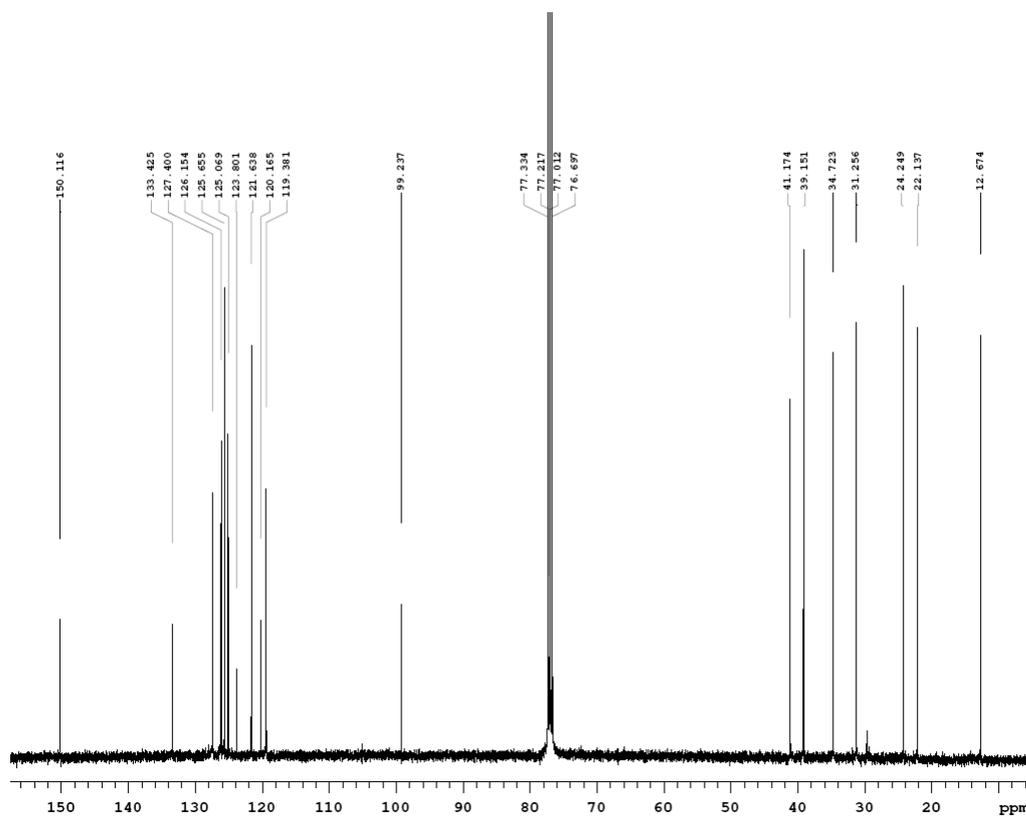
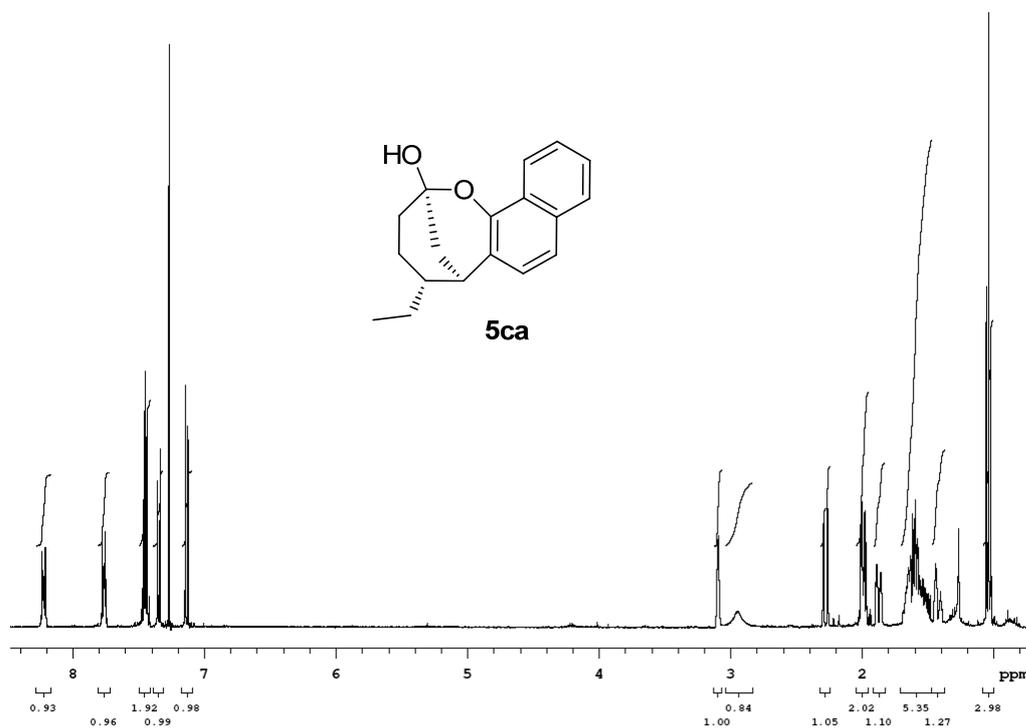
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1648_II_carbon
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1936 repetitions
OBSERVE C13, 100.5611153 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 1 hr, 59 min, 14 sec







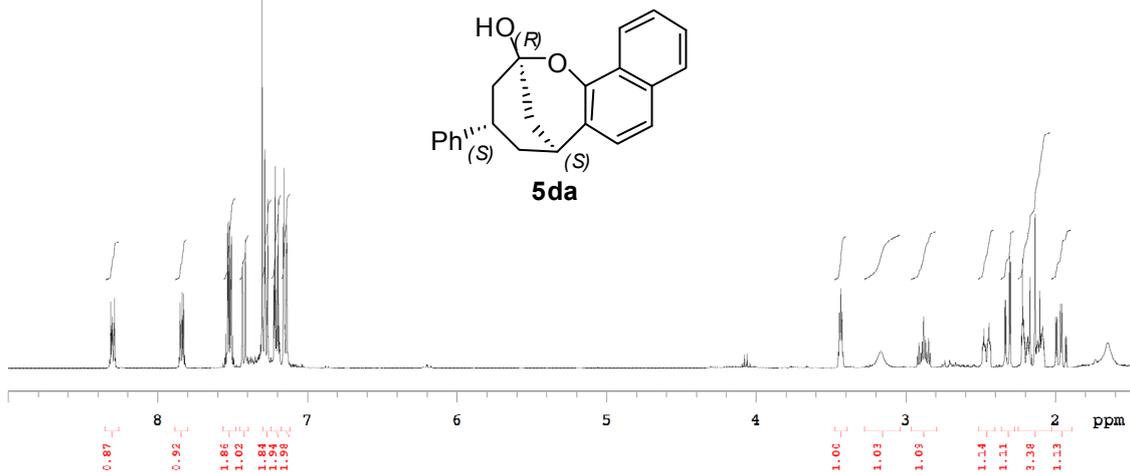


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1642_I_1H.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1642_I_1H
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
22 repetitions
OBSERVE H1, 399.9245580 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 9 sec

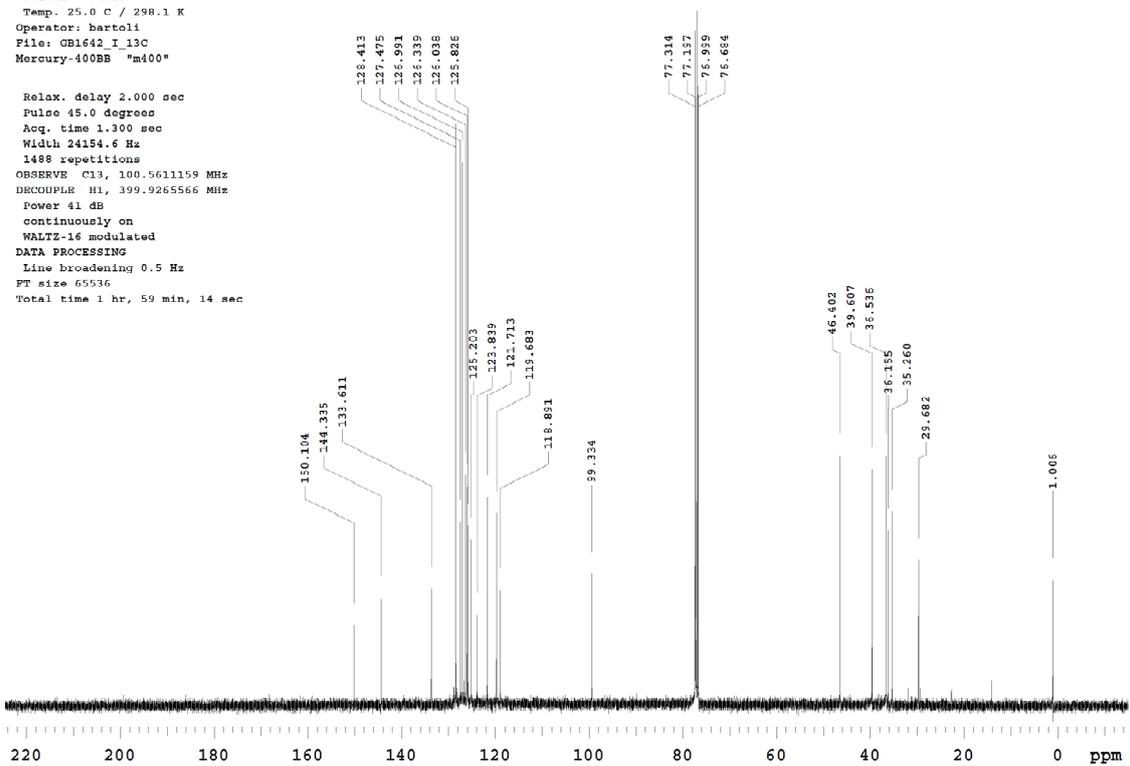


Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1642_I_13C.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1642_I_13C
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
1488 repetitions
OBSERVE C13, 100.5611159 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 1 hr, 59 min, 14 sec

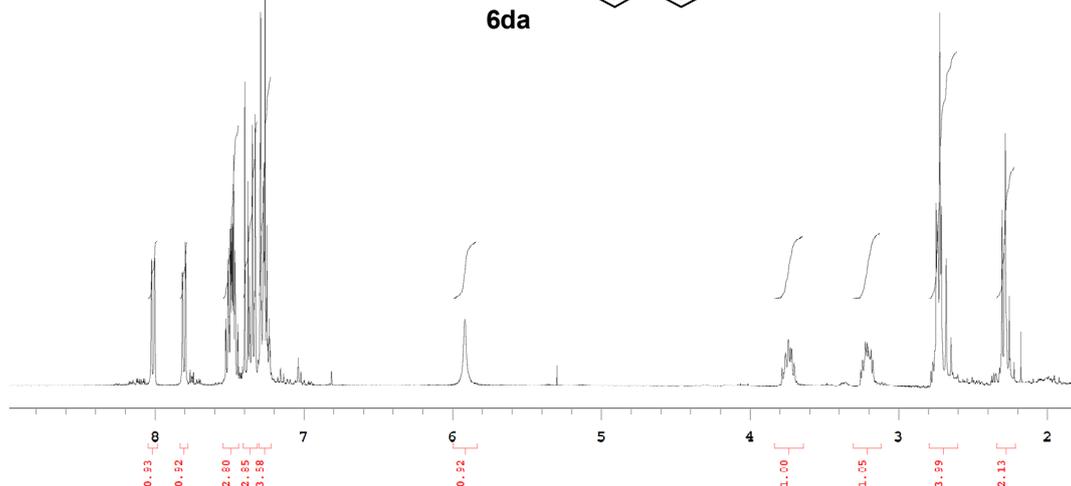
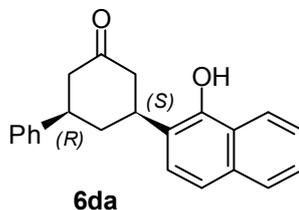


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1642 II 1H.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1642 II 1H
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.9245753 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 37 sec

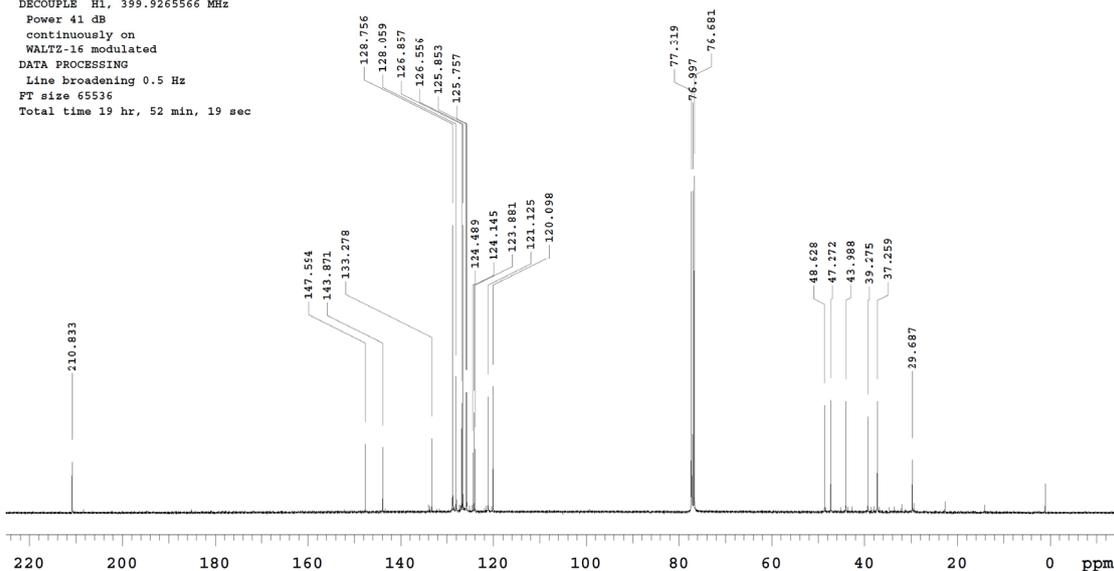


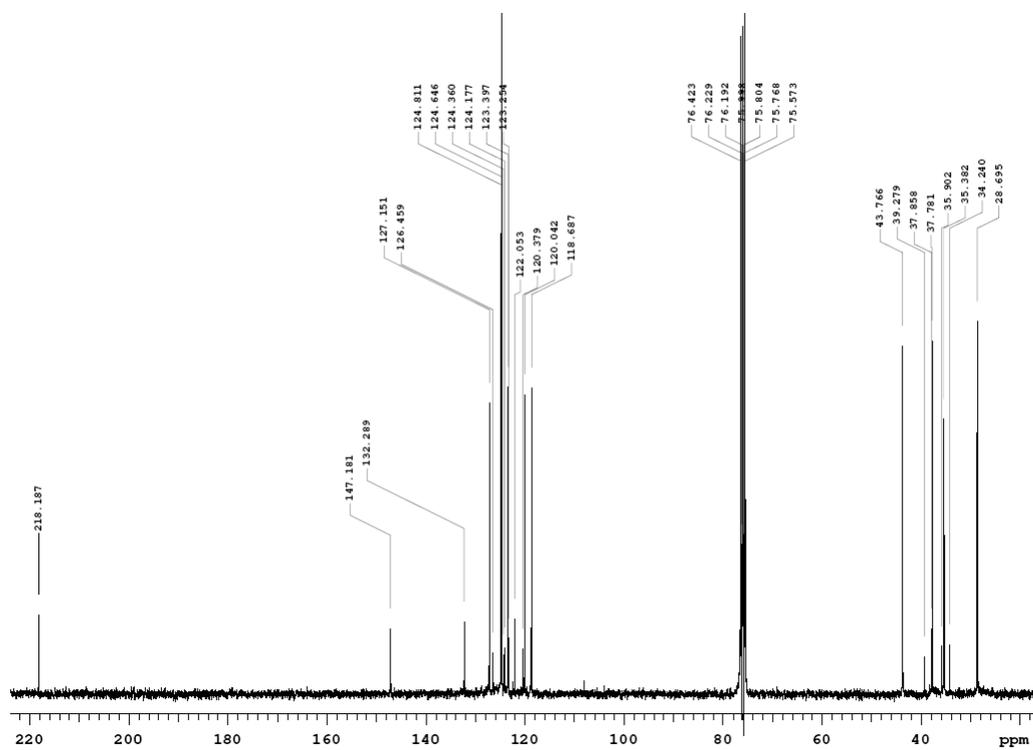
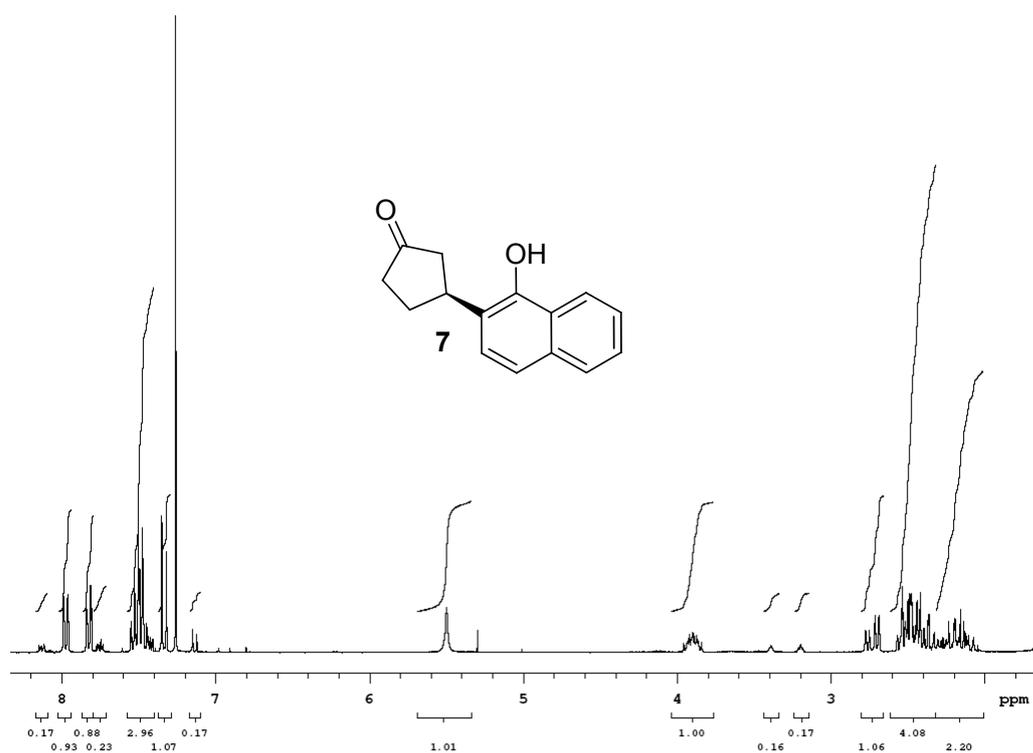
Std Carbon experiment

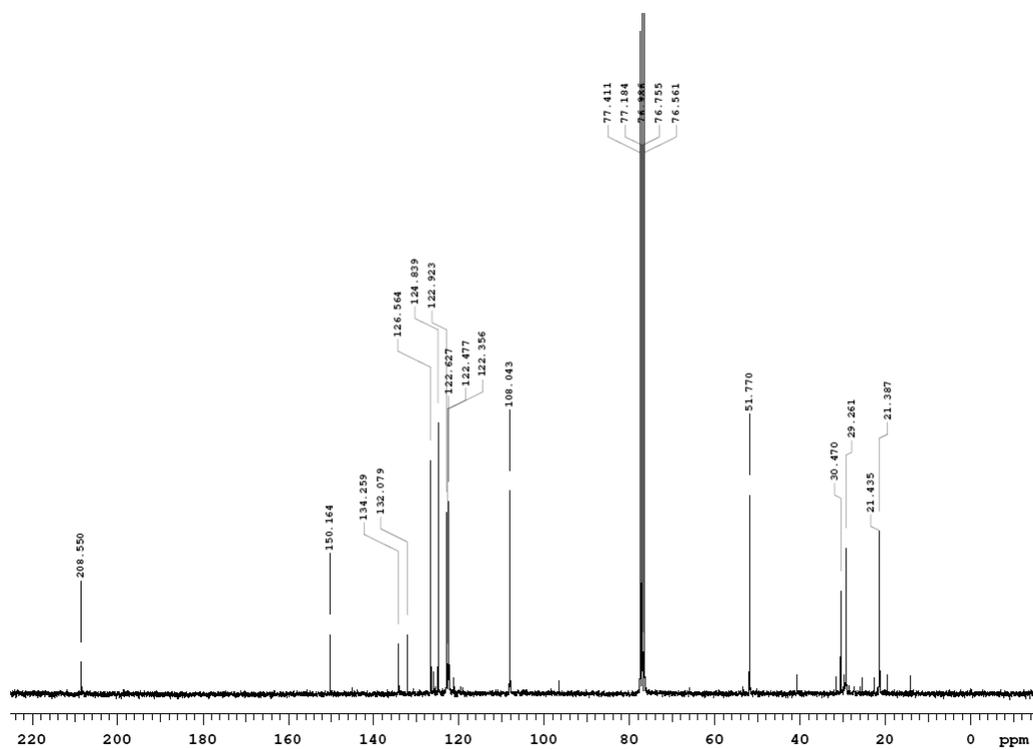
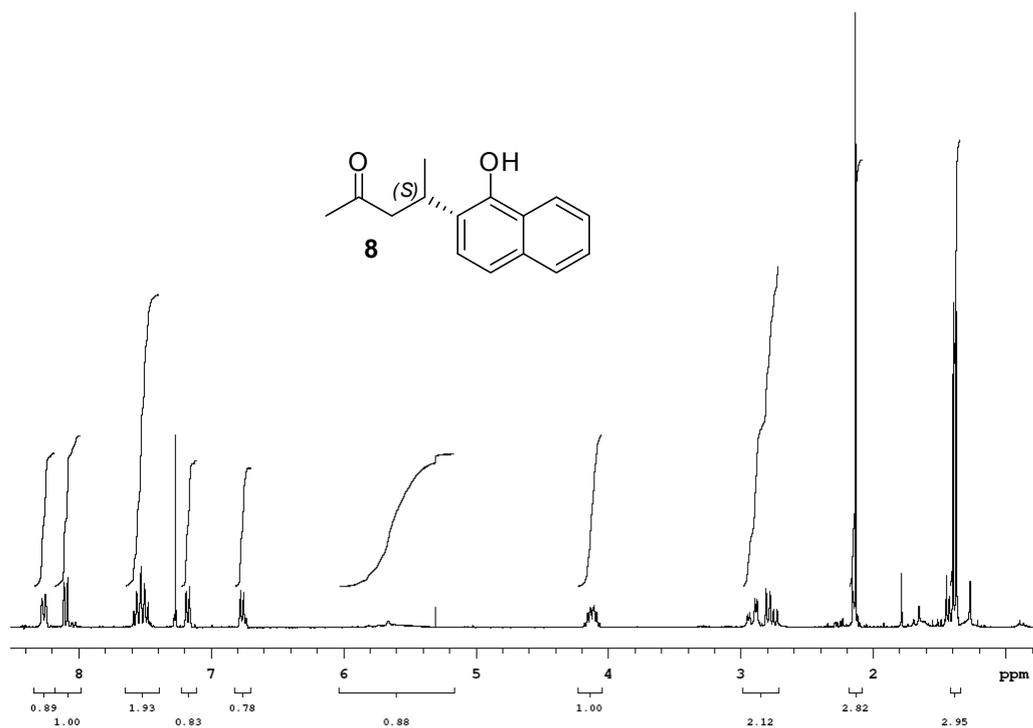
File: home/bartoli/Giorgio/GB1601-GB1700/GB1642 II 13C.fid

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1642 II 13C
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
16768 repetitions
OBSERVE C13, 100.5611154 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 19 hr, 52 min, 19 sec



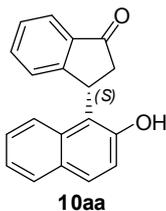




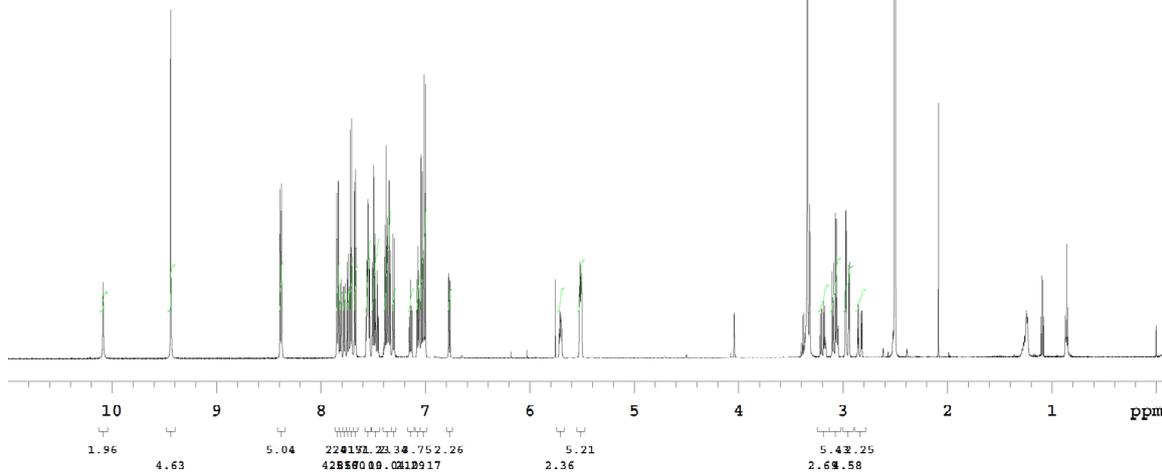
GB-1597 Inova600-Triple H1-s2pul-dmsco Jun 4 2012

Sample: code2_colonna
 File: home/lunazzi/GB-1597/gb1597-protone-setup.fid

Pulse Sequence: s2pul
 Solvent: dmsco
 Temp: 25.0 C / 298.1 K
 Operator: lunazzi
 File: gb1597-protone-setup
 INOVA-600 "1600"



Pulse 45.0 degrees
 Acq. time 2.990 sec
 Width 9611.9 Hz
 2 repetitions
 OBSERVE H1, 599.7304201 MHz
 DATA PROCESSING
 Sg. sine bell 2.990 sec
 Shifted by -2.990 sec
 FT size 131072
 Total time 0 min, 12 sec

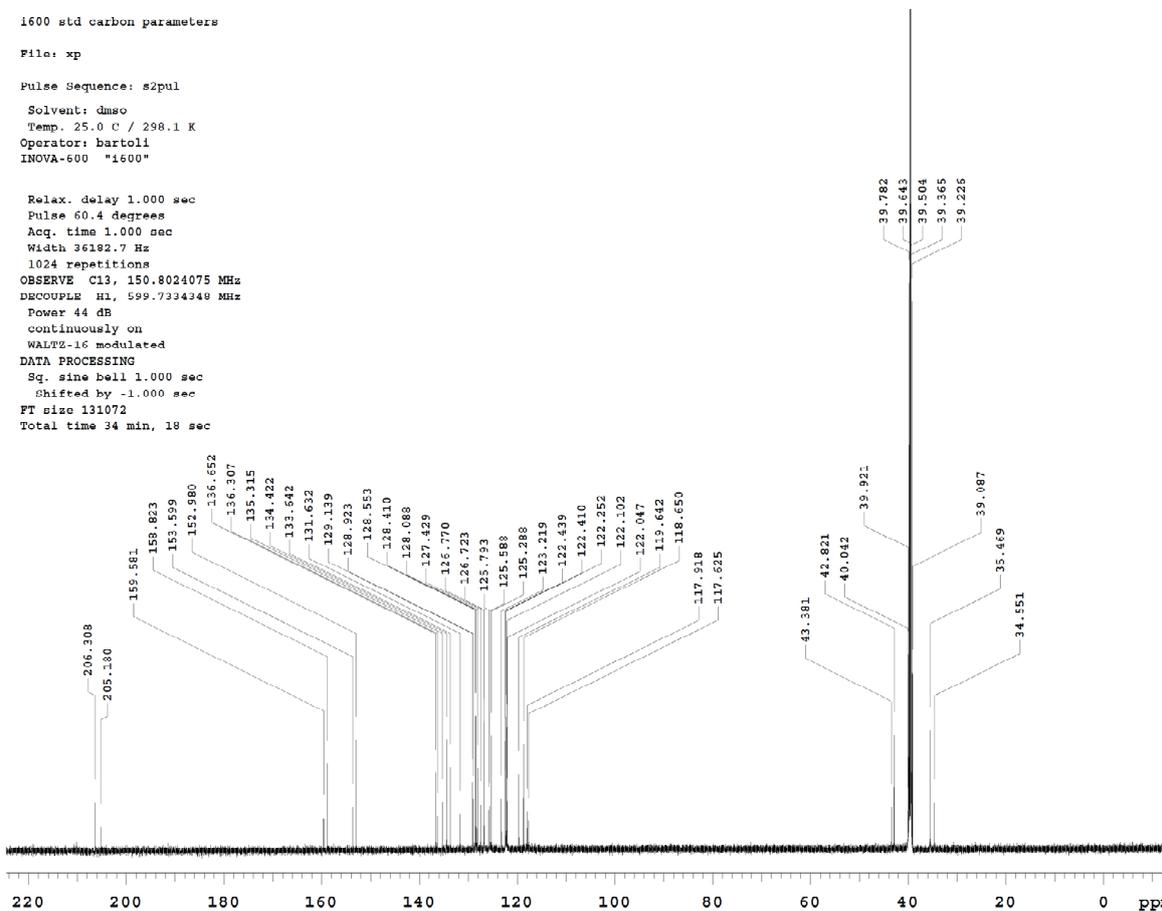


1600 std carbon parameters

File: xp

Pulse Sequence: s2pul
 Solvent: dmsco
 Temp: 25.0 C / 298.1 K
 Operator: bartoli
 INOVA-600 "1600"

Relax. delay 1.000 sec
 Pulse 60.4 degrees
 Acq. time 1.000 sec
 Width 36182.7 Hz
 1024 repetitions
 OBSERVE C13, 150.8024075 MHz
 DECOUPLE H1, 599.7334349 MHz
 Power 44 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Sg. sine bell 1.000 sec
 Shifted by -1.000 sec
 FT size 131072
 Total time 34 min, 18 sec

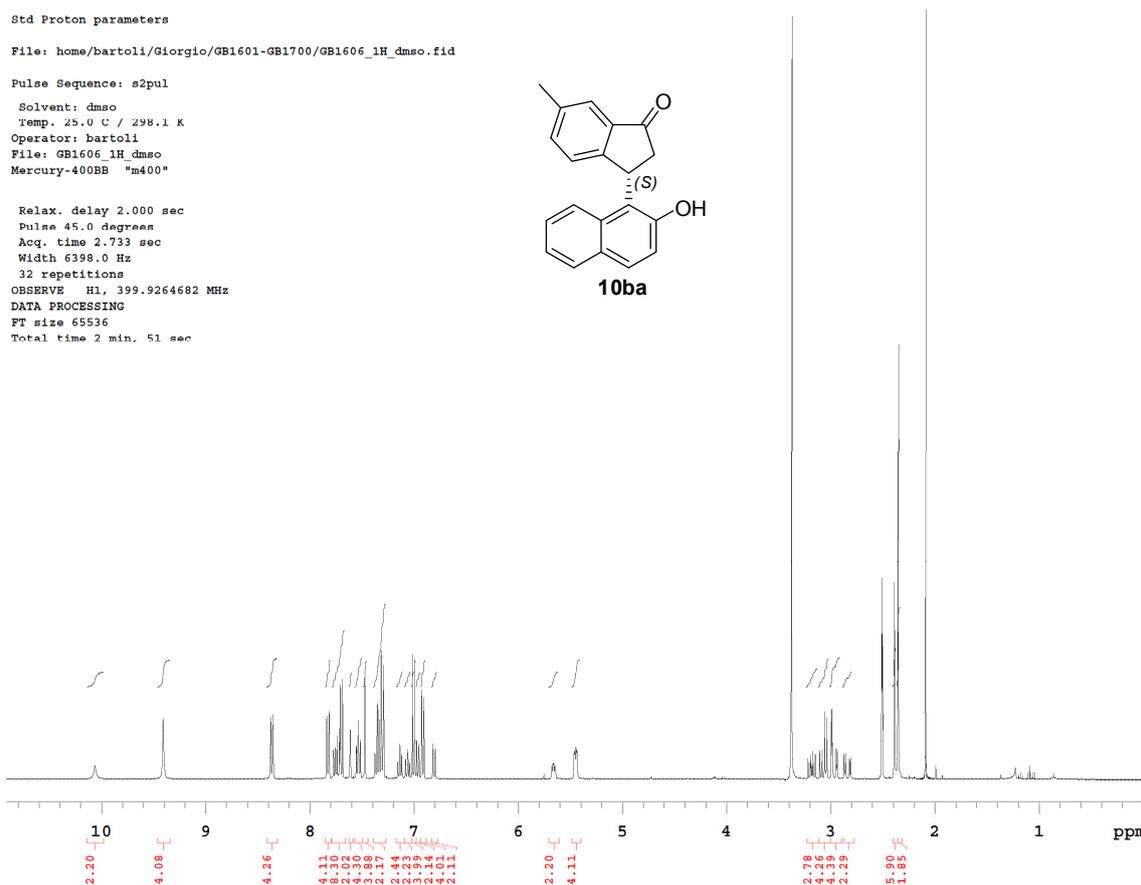
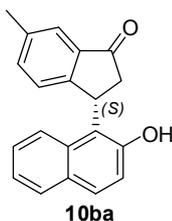


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1606_1H_dms0.fid

Pulse Sequence: s2pul
 Solvent: dms0
 Temp: 25.0 C / 298.1 K
 Operator: bartoli
 File: GB1606_1H_dms0
 Mercury-400BB "m400"

Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.733 sec
 Width 6398.0 Hz
 32 repetitions
 OBSERVE H1, 399.9264682 MHz
 DATA PROCESSING
 FT size 65536
 Total time 2 min, 51 sec

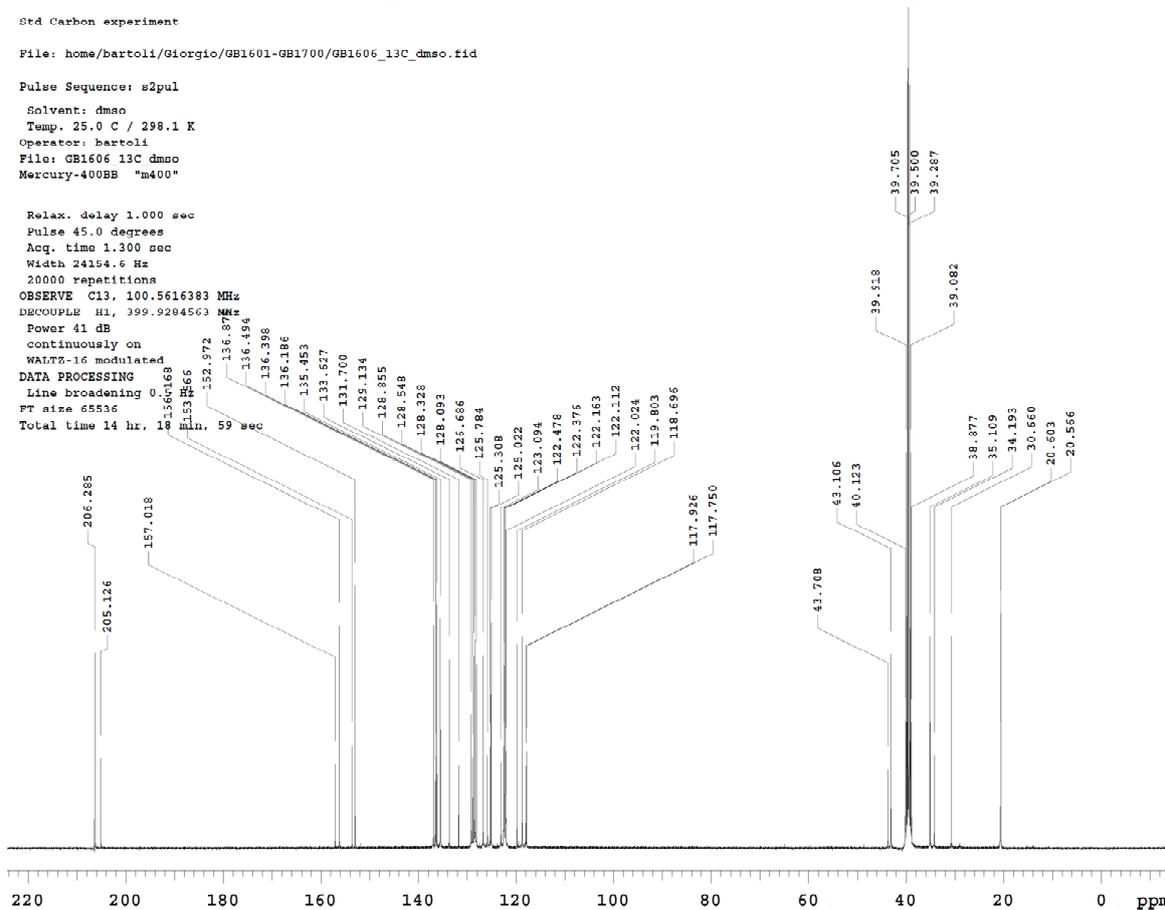


Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1606_13C_dms0.fid

Pulse Sequence: s2pul
 Solvent: dms0
 Temp: 25.0 C / 298.1 K
 Operator: bartoli
 File: GB1606_13C_dms0
 Mercury-400BB "m400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 20000 repetitions
 OBSERVE C13, 100.5616383 MHz
 DECOUPLE H1, 399.9284563 MHz
 Power 41 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3
 FT size 65536
 Total time 14 hr, 18 min, 59 sec

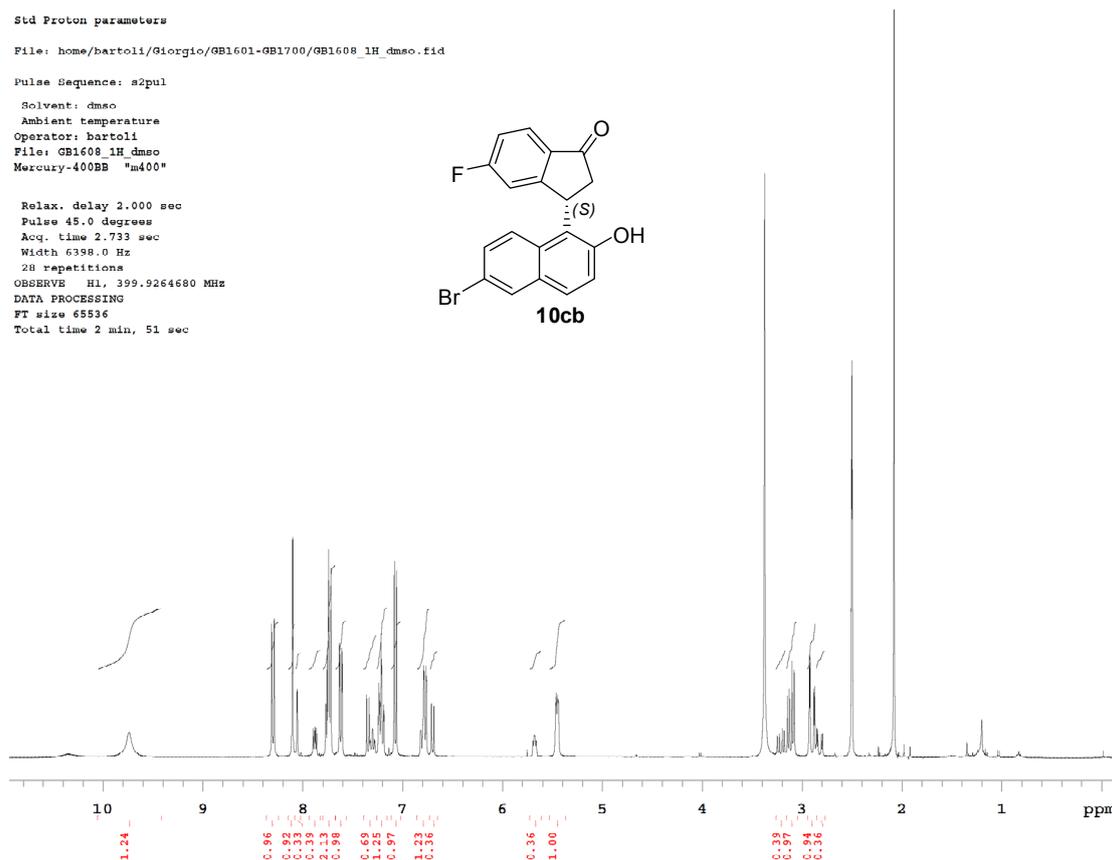
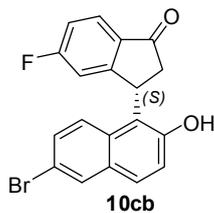


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1608_1H_dmsc.fid

Pulse Sequence: s2pul
 Solvent: dmsc
 Ambient temperature
 Operator: bartoli
 File: GB1608_1H_dmsc
 Mercury-400BB "m400"

Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.733 sec
 Width 6398.0 Hz
 28 repetitions
 OBSERVE H1, 399.9264680 MHz
 DATA PROCESSING
 FT size 65536
 Total time 2 min, 51 sec

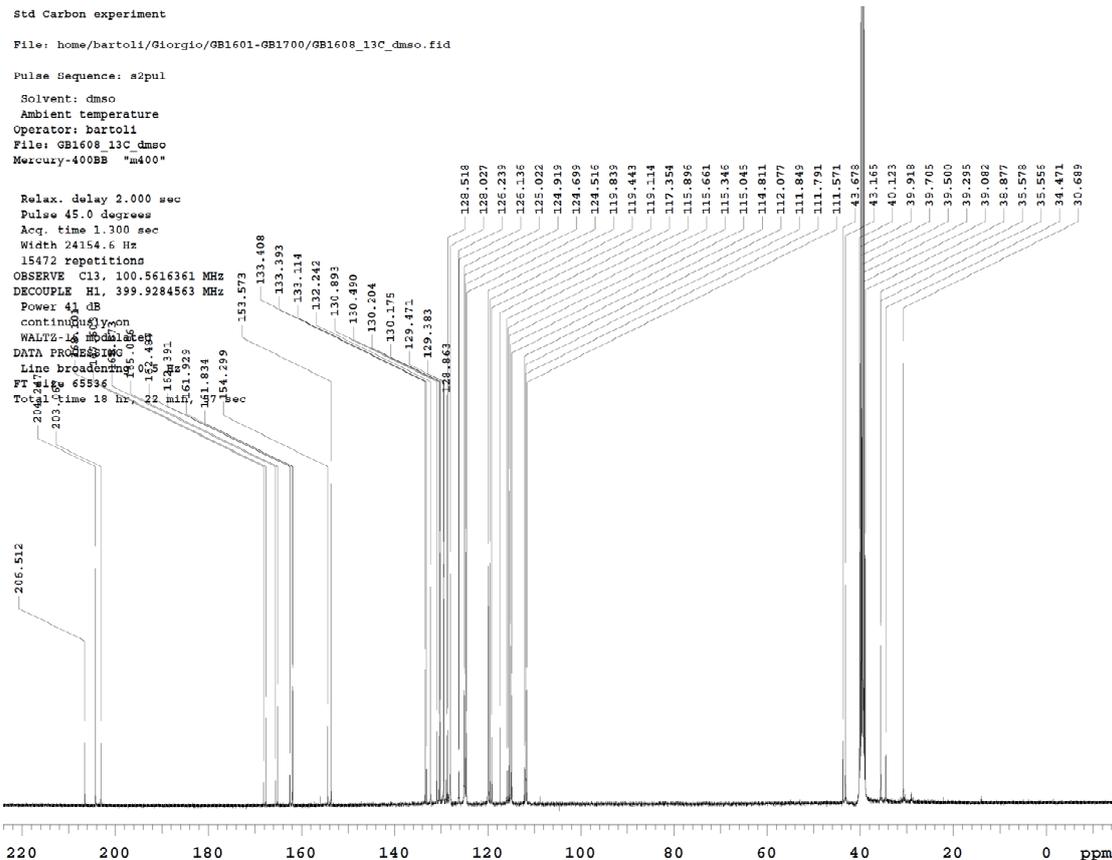


Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1608_13C_dmsc.fid

Pulse Sequence: s2pul
 Solvent: dmsc
 Ambient temperature
 Operator: bartoli
 File: GB1608_13C_dmsc
 Mercury-400BB "m400"

Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 15472 repetitions
 OBSERVE C13, 100.5616361 MHz
 DECOUPLE H1, 399.9284563 MHz
 Power 41 dB
 continuous on
 WALTZ-16 modulation
 DATA PROCESSING
 Line broadening 0.5
 FT size 65536
 Total time 18 hr, 22 min, 54 sec

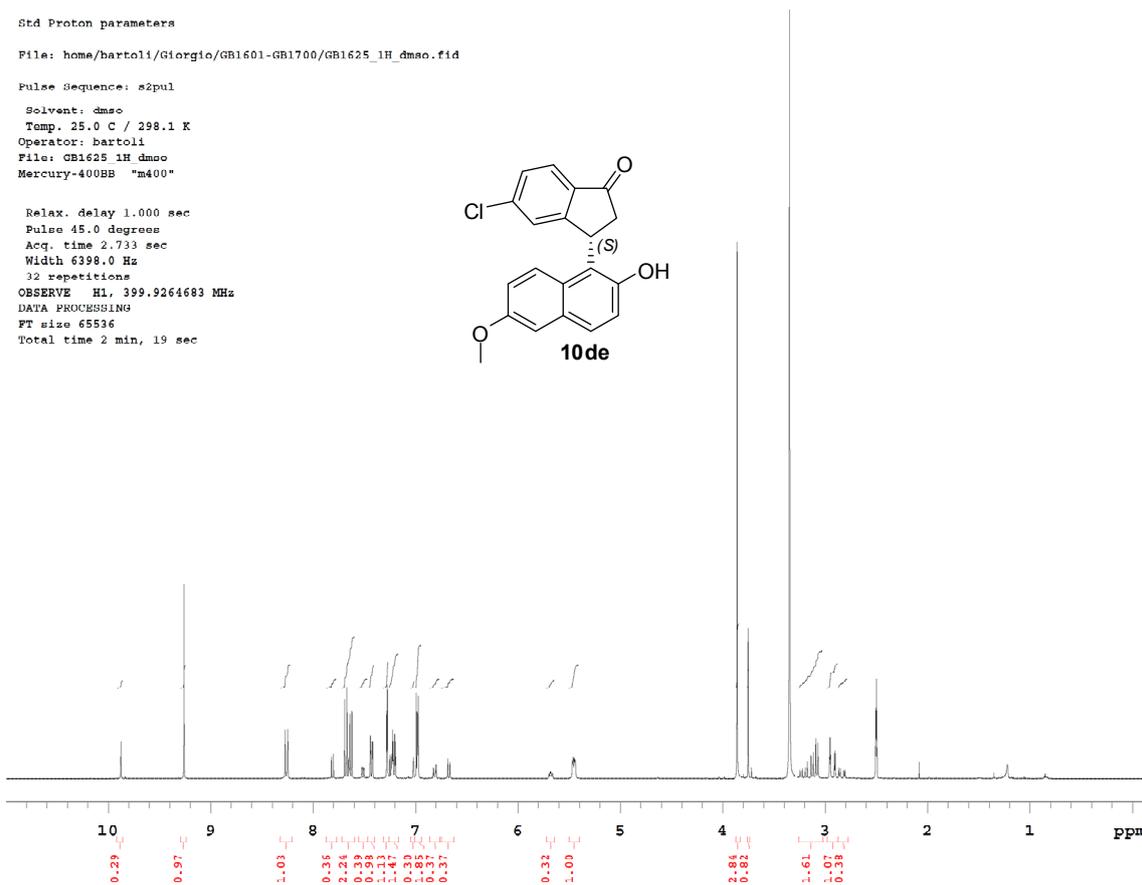
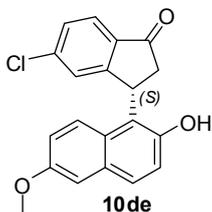


Std Proton parameters

File: home/bartol1/Giorgio/GB1601-GB1700/GB1625_1H_dmao.fid

Pulse Sequence: s2pul
 Solvent: dmao
 Temp. 25.0 C / 298.1 K
 Operator: bartol1
 File: GB1625_1H_dmao
 Mercury-400BB "m400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.733 sec
 Width 6398.0 Hz
 32 repetitions
 OBSERVE H1, 399.9264683 MHz
 DATA PROCESSING
 FT size 65536
 Total time 2 min, 19 sec

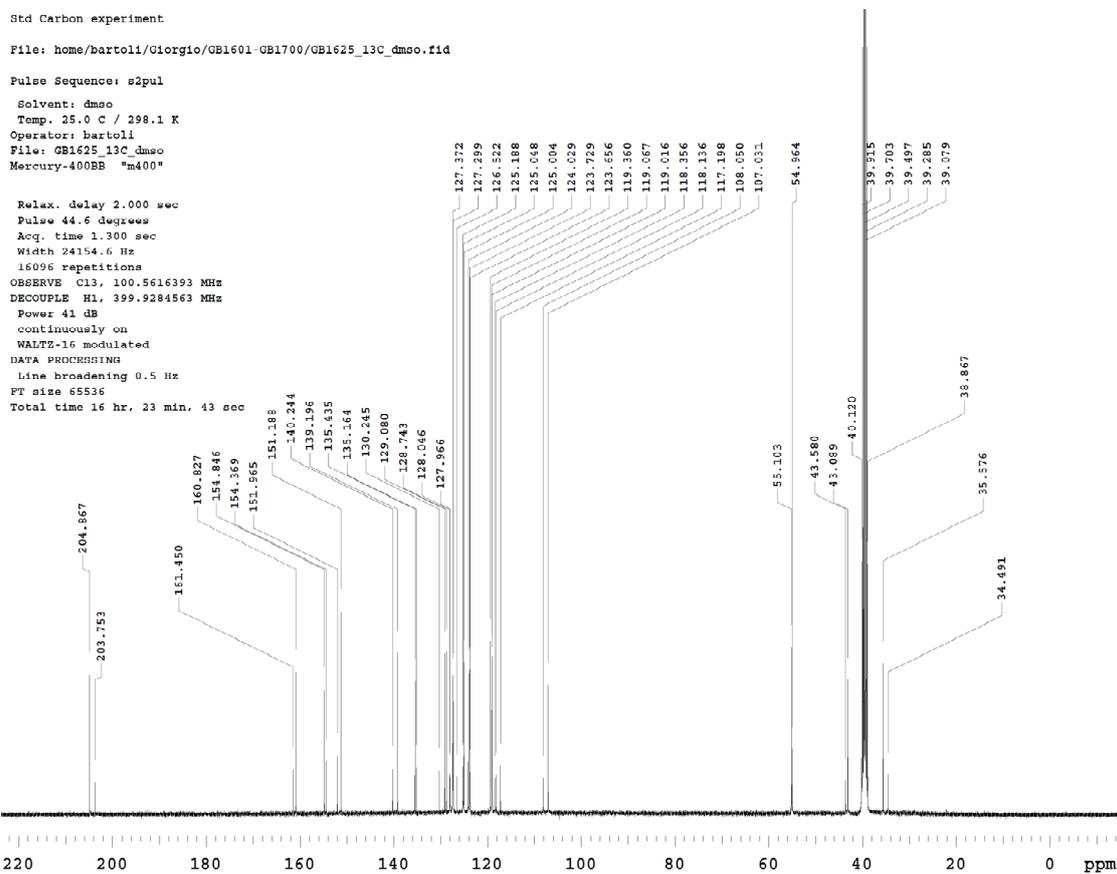


Std Carbon experiment

File: home/bartol1/Giorgio/GB1601-GB1700/GB1625_13C_dmao.fid

Pulse Sequence: s2pul
 Solvent: dmao
 Temp. 25.0 C / 298.1 K
 Operator: bartol1
 File: GB1625_13C_dmao
 Mercury-400BB "m400"

Relax. delay 2.000 sec
 Pulse 44.6 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 16096 repetitions
 OBSERVE C13, 100.5616393 MHz
 DECOUPLE H1, 399.9284563 MHz
 Power 41 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 16 hr, 23 min, 43 sec

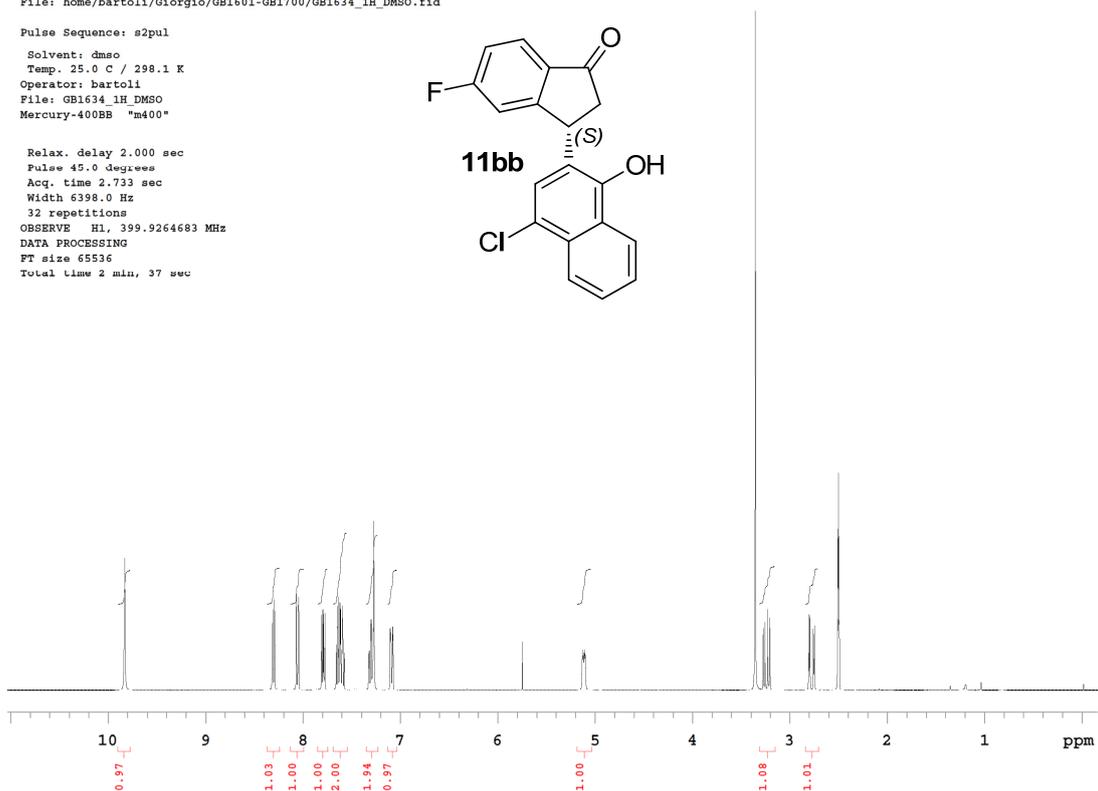
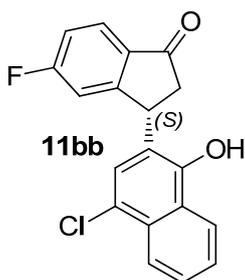


Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1634_1H_DMSO.fid

Pulse Sequence: s2pul
Solvent: dmsd
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1634_1H_DMSO
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.9264683 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 37 sec

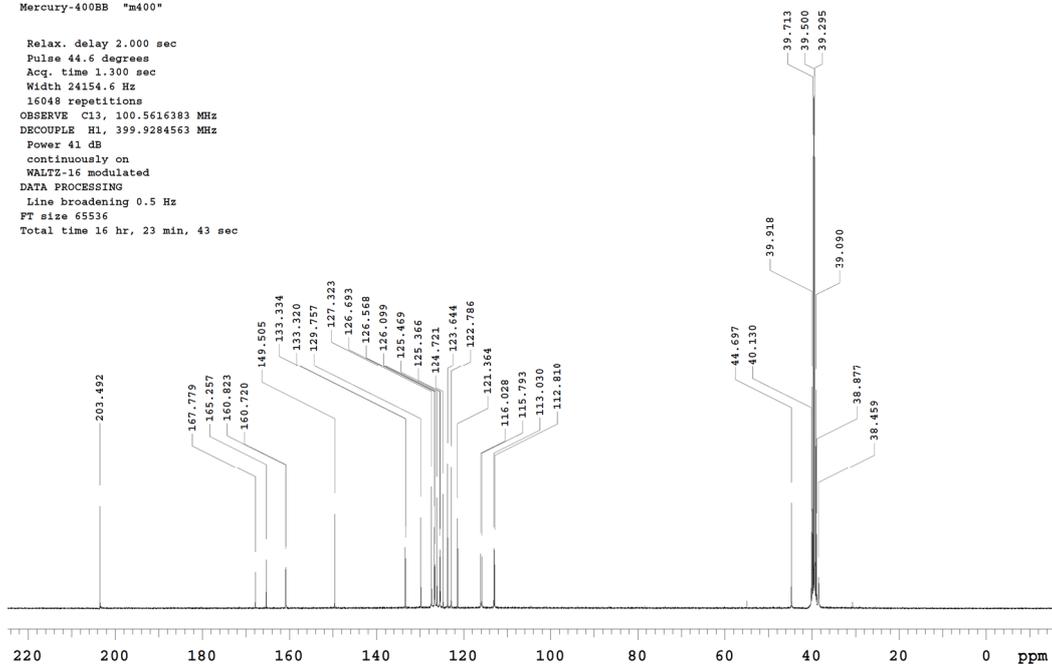


Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1634_13C_DMSO.fid

Pulse Sequence: s2pul
Solvent: dmsd
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1634_13C_DMSO
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 44.6 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
16048 repetitions
OBSERVE C13, 100.5616383 MHz
DECOUPLE H1, 399.9284563 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 16 hr, 23 min, 43 sec



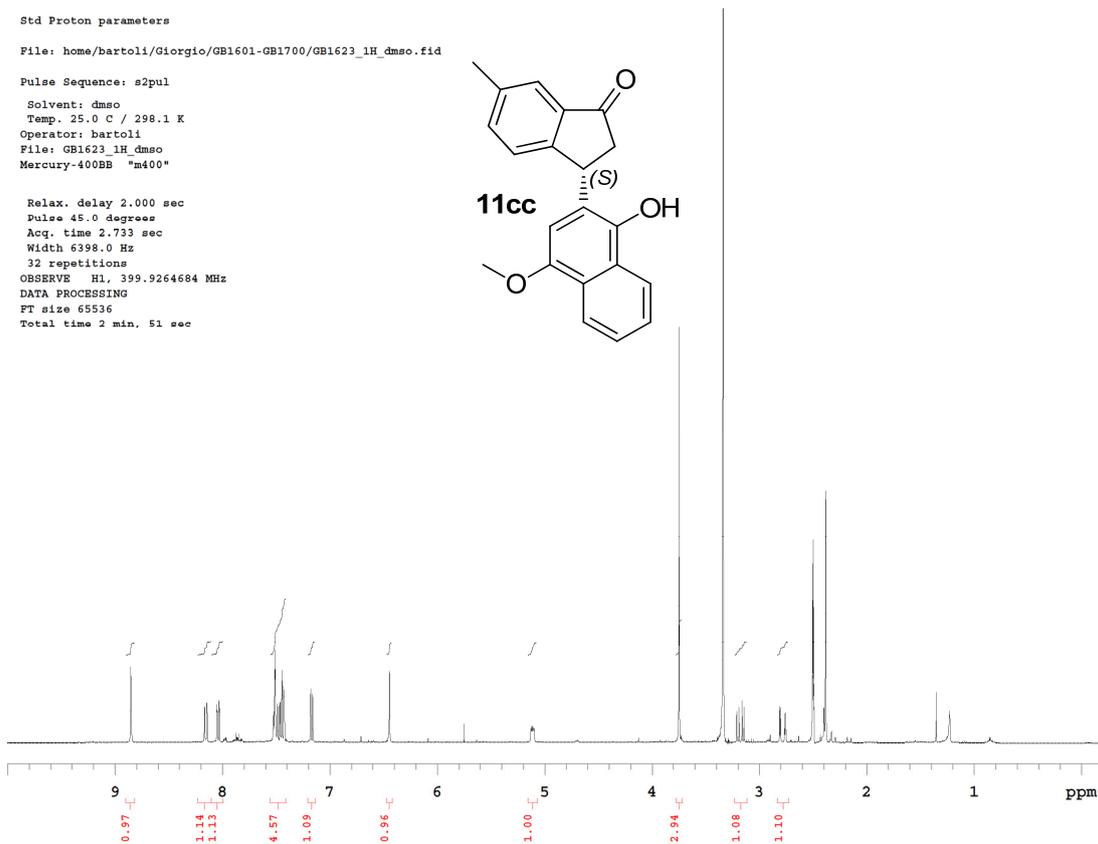
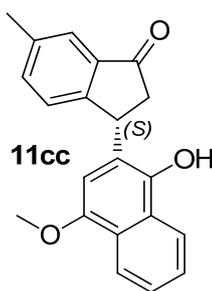
Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1623_1H_dmsso.fid

Pulse Sequence: s2pul

Solvent: dmsco
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1623_1H_dmsco
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 6398.0 Hz
32 repetitions
OBSERVE H1, 399.9264684 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 51 sec



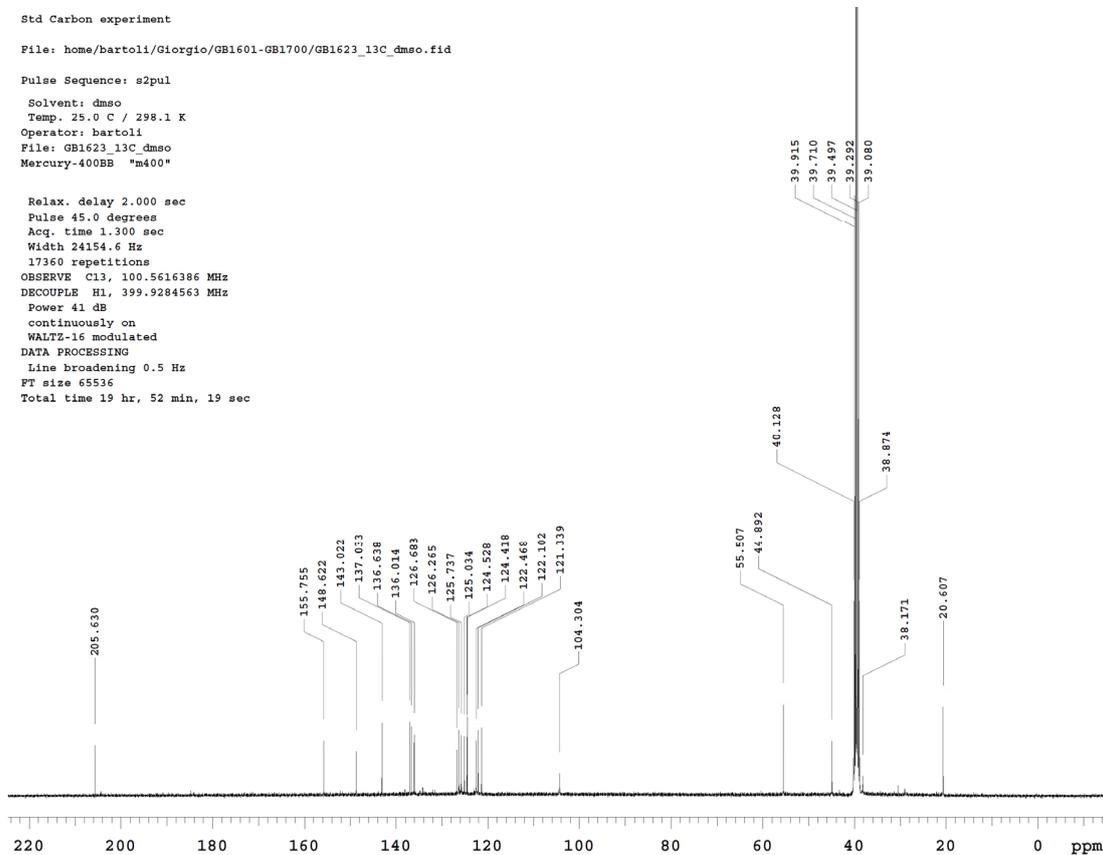
Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1623_13C_dmsso.fid

Pulse Sequence: s2pul

Solvent: dmsco
Temp. 25.0 C / 298.1 K
Operator: bartoli
File: GB1623_13C_dmsco
Mercury-400BB "m400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24154.6 Hz
17360 repetitions
OBSERVE C13, 100.5616386 MHz
DECOUPLE H1, 399.9284563 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 hr, 52 min, 19 sec



Std Proton parameters

File: home/bartoli/Giorgio/GB1601-GB1700/GB1630_1H_DMSO.fid

Pulse Sequence: s2pul

Solvent: dmsc

Temp. 25.0 C / 298.1 K

Operator: bartoli

File: GB1630_1H_DMSO

Mercury-400BB "m400"

Relax. delay 2.000 sec

Pulse 4E.0 degrees

Acq. time 2.733 sec

Width 6398.0 Hz

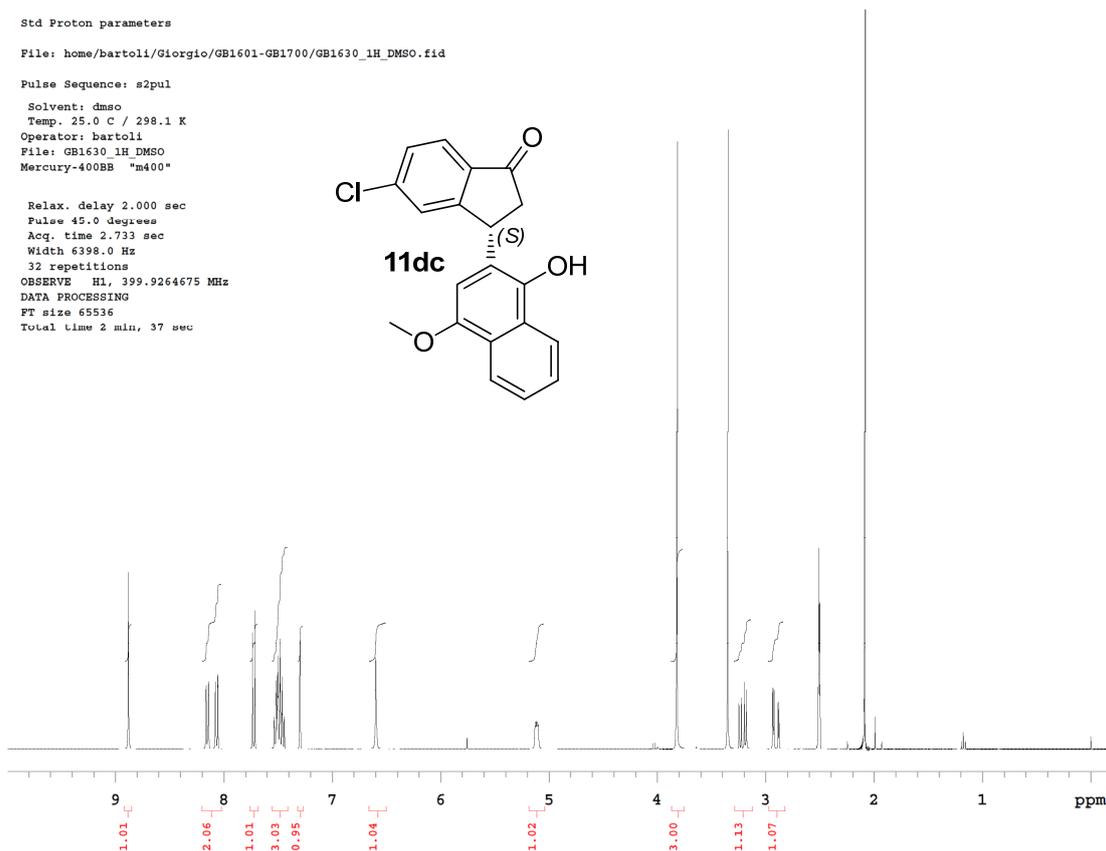
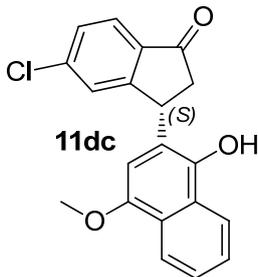
32 repetitions

OBSERVE H1, 399.9264675 MHz

DATA PROCESSING

FT size 65536

Total time 2 min, 37 sec



Std Carbon experiment

File: home/bartoli/Giorgio/GB1601-GB1700/GB1630_13C_DMSO.fid

Pulse Sequence: s2pul

Solvent: dmsc

Temp. 25.0 C / 298.1 K

Operator: bartoli

File: GB1630_13C_DMSO

Mercury-400BB "m400"

Relax. delay 2.000 sec

Pulse 4E.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

2432 repetitions

OBSERVE C13, 100.5615922 MHz

DECOUPLE H1, 399.9284563 MHz

Power 41 dB

continuously on

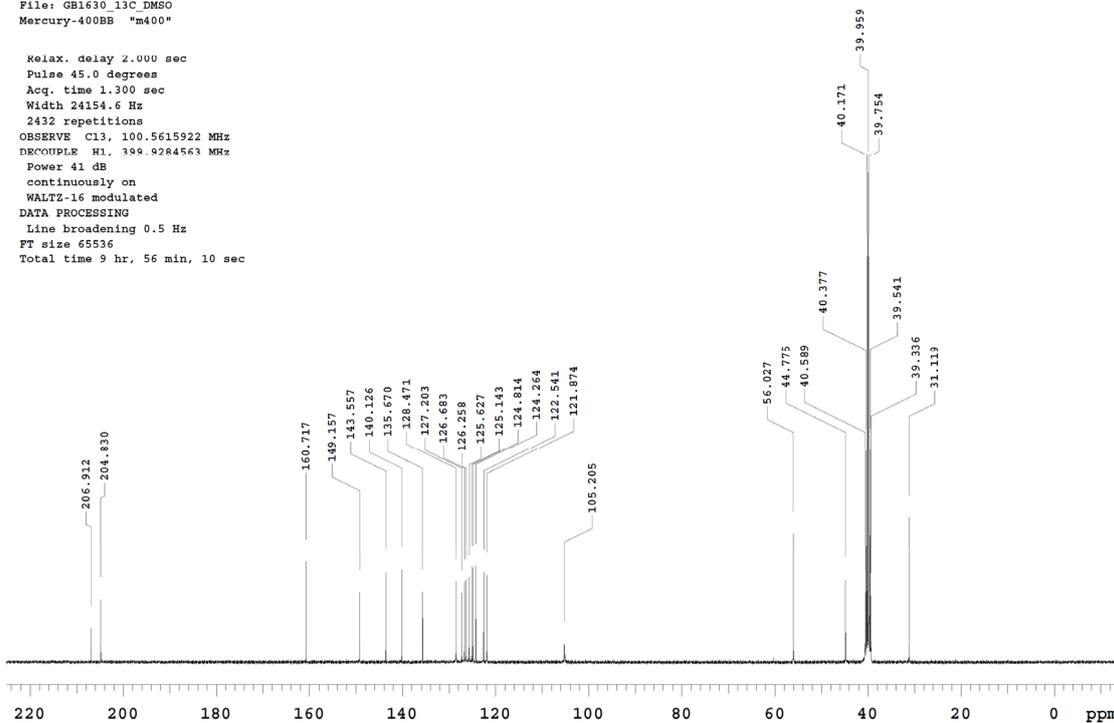
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 9 hr, 56 min, 10 sec



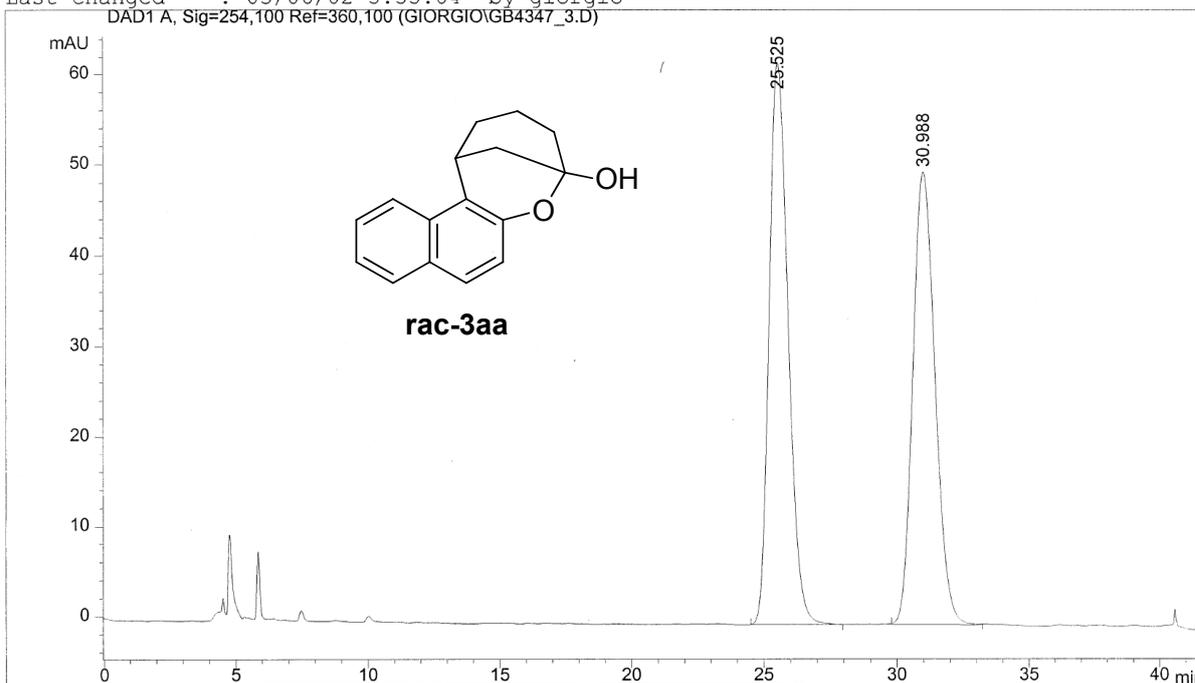
HPLC traces

Data File D:\HPCHEM\2\DATA\GIORGIO\GB4347_3.D

Sample Name: gb4347

Racemic Sample
OD-H, 95:5 n-Hexane:isopropanol, 0.650 ml/min

=====
Injection Date : 25/02/02 23.29.53
Sample Name : gb4347 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 25/02/02 23.07.05 by giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/06/02 5.55.04 by giorgio
DAD1 A, Sig=254,100 Ref=360,100 (GIORGIO\GB4347_3.D)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

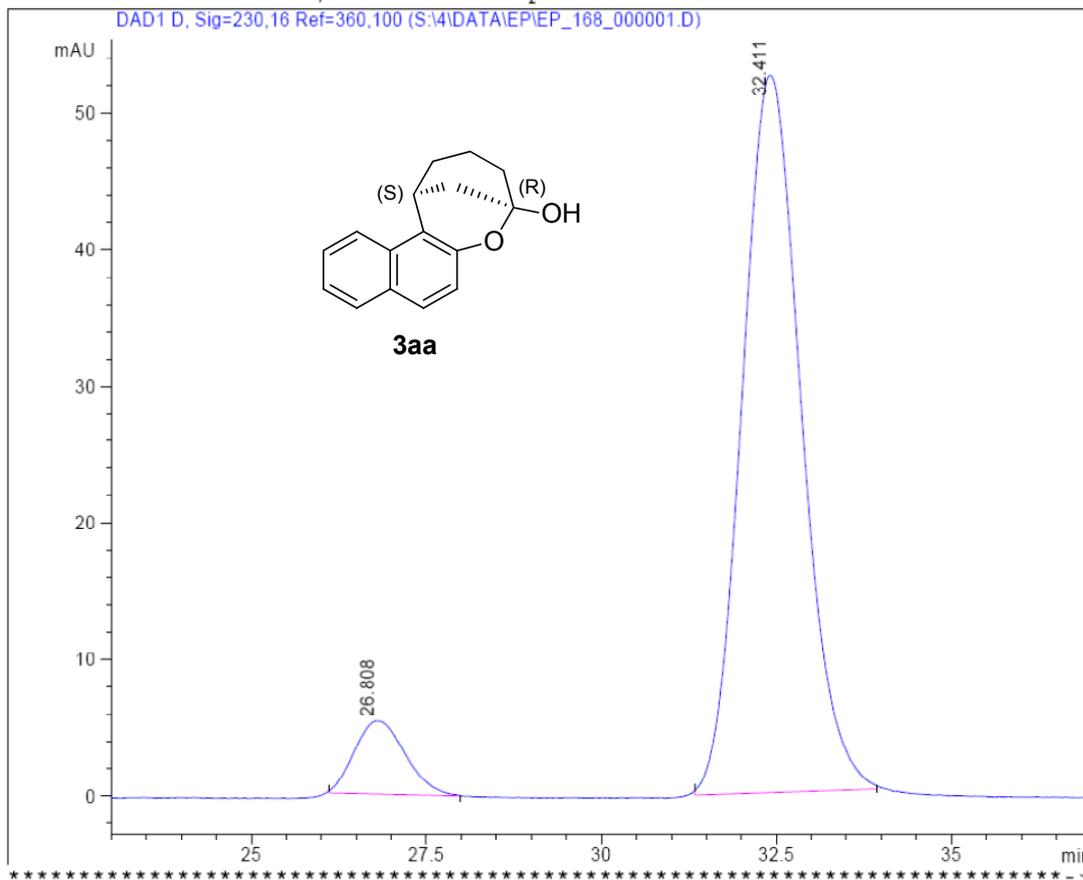
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.525	PP	0.7811	3094.95581	62.13245	51.4726
2	30.988	BB	0.9192	2917.87183	50.12292	48.5274

Totals : 6012.82764 112.25537

Results obtained with enhanced integrator!

=====
*** End of Report ***

Data File: S:\4\DATA\EP\EP_168_000001.D
Sample name: EP_168_
Sample Info: OD-H, 0.65 ml/min, 95:5 Hex/i-PrOH, 20°, 230 nm. ->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.650 mL/min Temp.: 20°C



*****-->

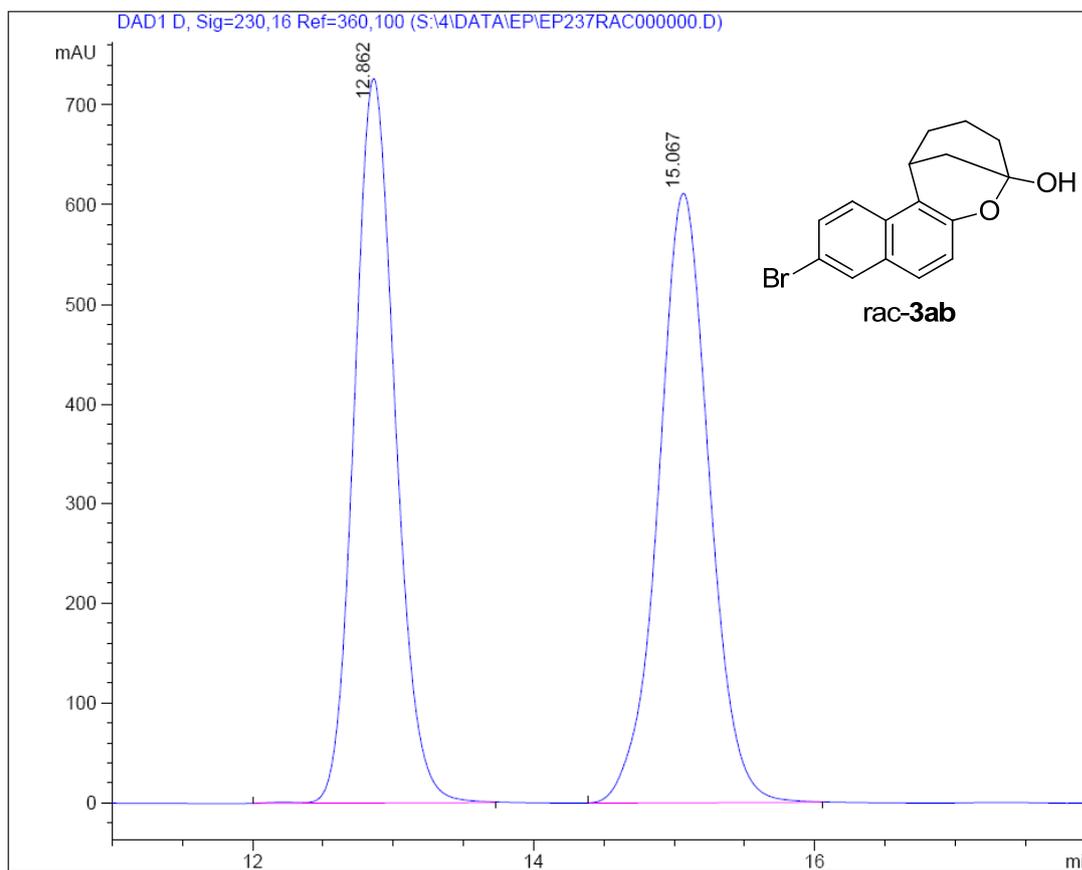
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	26.808	BB	0.601	5.351	269.460	8.058	0.000	
2	32.411	BB	0.875	52.498	3074.665	91.942	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP237RAC000000.D
Sample name: EP237rac
Sample Info: Lux-cellulose 2, Hex/i-PrOH 9:1, 0.65 ml/min, 230 nm, 2
0°C ->
Acquired by: EP on: 02/05/2012 18.14.50
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: 5uL
Column: Chiralpak AD-H 250 mm x 4.6 mm
Flow: 0.650 mL/min Temp.: 19.99°C

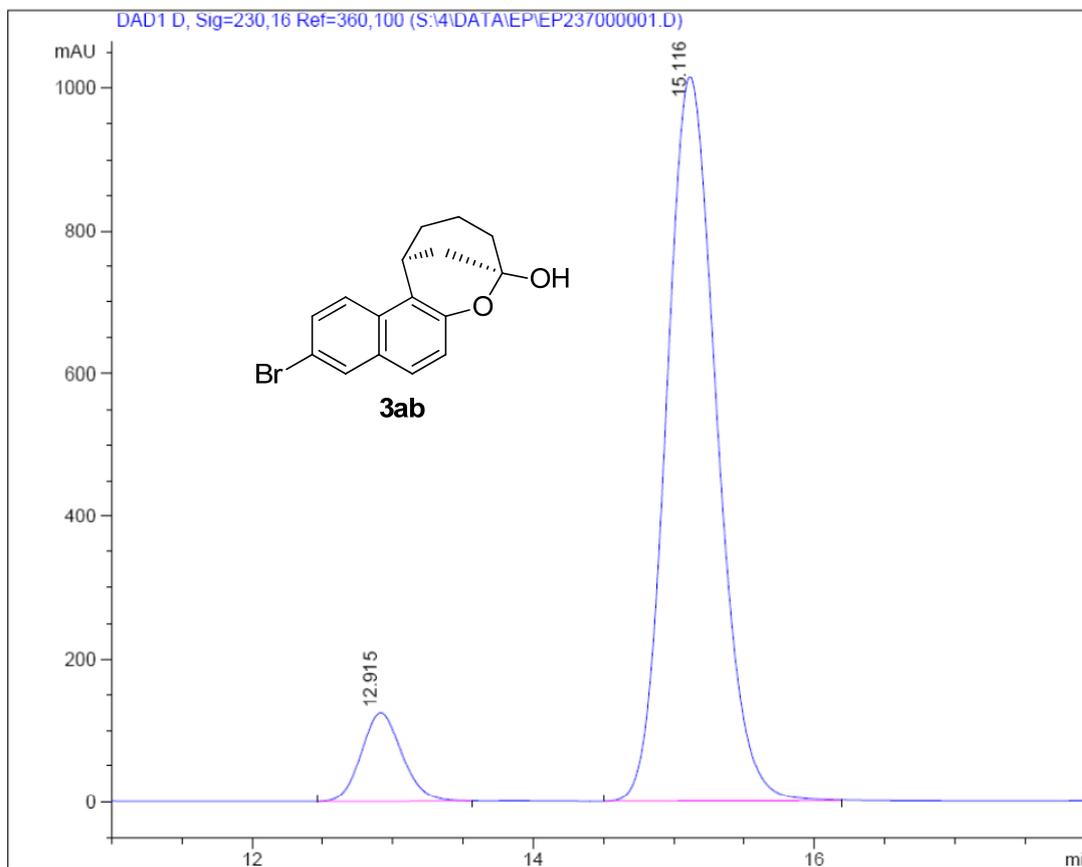


***** ->
Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	12.862	BB	0.315	726.557	14751.840	48.925	0.000	
2	15.067	BB	0.384	611.287	15400.125	51.075	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP237000001.D
 Sample name: EP237
 Sample Info: Lux-cellulose 2, Hex/i-PrOH 9:1, 0.65 ml/min, 230 nm, 20°C
 Acquired by: EP on: 02/05/2012 18.41.08
 Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
 Location: Vial 1 Volume: 5uL
 Column: Chiralpak AD-H 250 mm x 4.6 mm
 Flow: 0.650 mL/min Temp.: 20.01°C

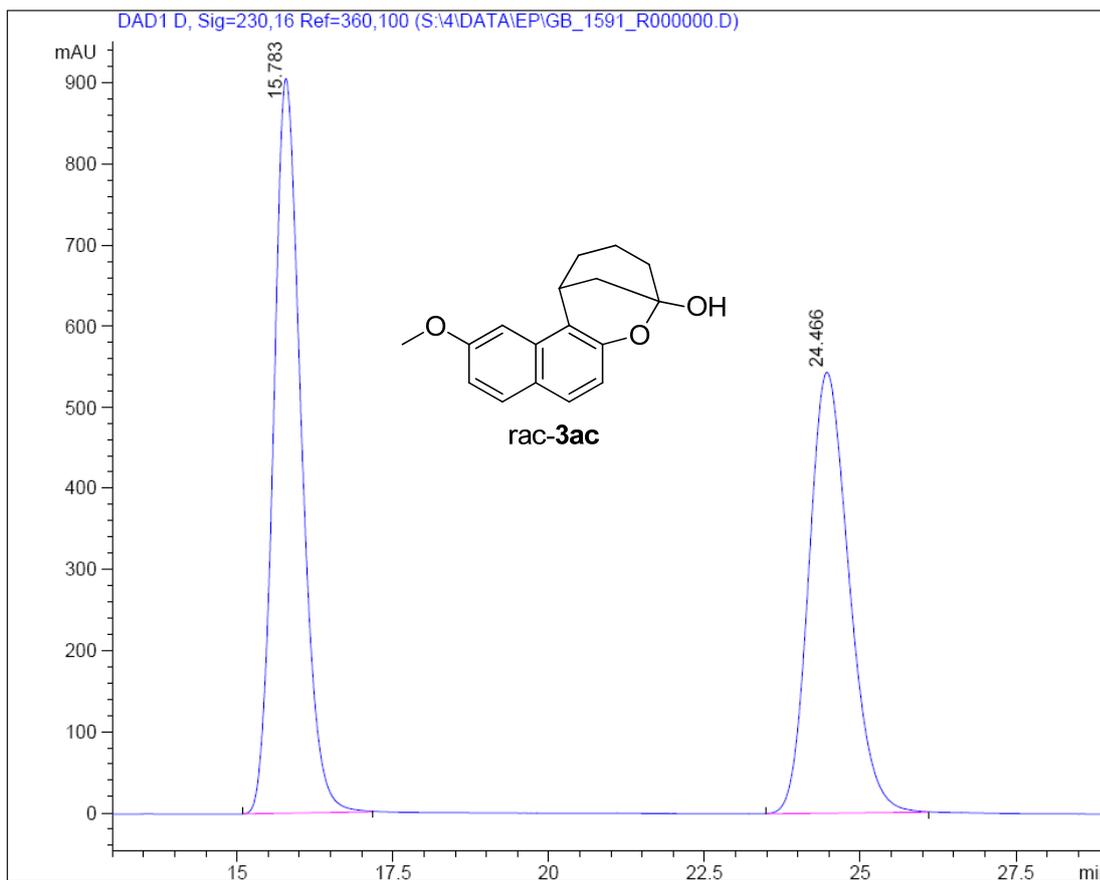


***** ->
 Area Percent Report
 ***** ->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	12.915	BB	0.310	124.175	2513.833	9.069	0.000	
2	15.116	BB	0.386	1015.170	25204.023	90.931	0.000	
							0.000	

Data File: S:\4\DATA\EP\GB_1591_R000000.D
Sample name: GB_1591_r
Sample Info: Lux-Cellulose 2, 0.7 ml/min, Hex/i-PrOH 9:1, 230 nm, 20 °C.
Acquired by: EP on: 04/05/2012 18.18.03
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: 5uL
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 20°C



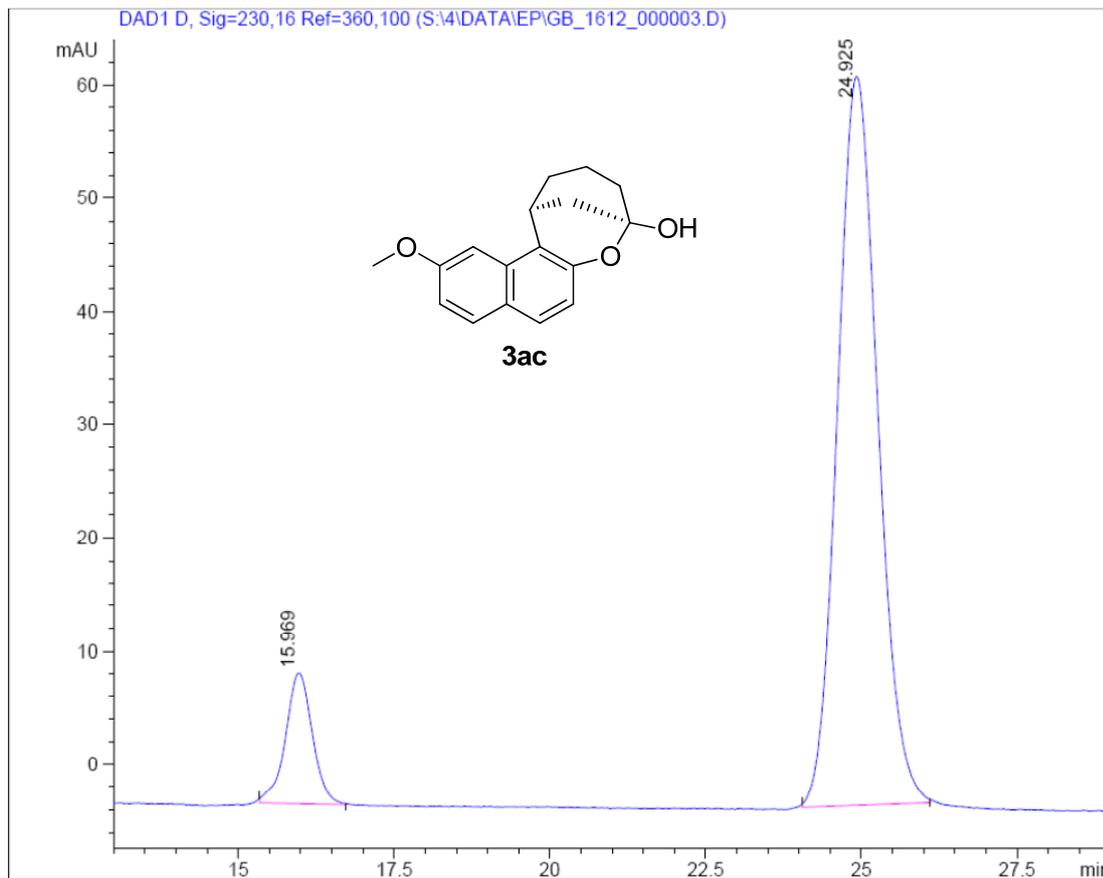
*****-->
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	15.783	BB	0.471	905.739	28018.012	53.471	0.000	
2	24.466	BB	0.691	543.923	24380.063	46.529	0.000	
							0.000	

Data File: S:\4\DATA\EP\GB_1612_000003.D
Sample name: GB_1612_
Sample Info: Lux-Cellulose2; Hex-Ipa 9:1; T=20°; flusso=0,7ml/min; 2
30 nm; ->
Acquired by: EP on: 23/05/2012 17.42.41
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 25 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 20°C



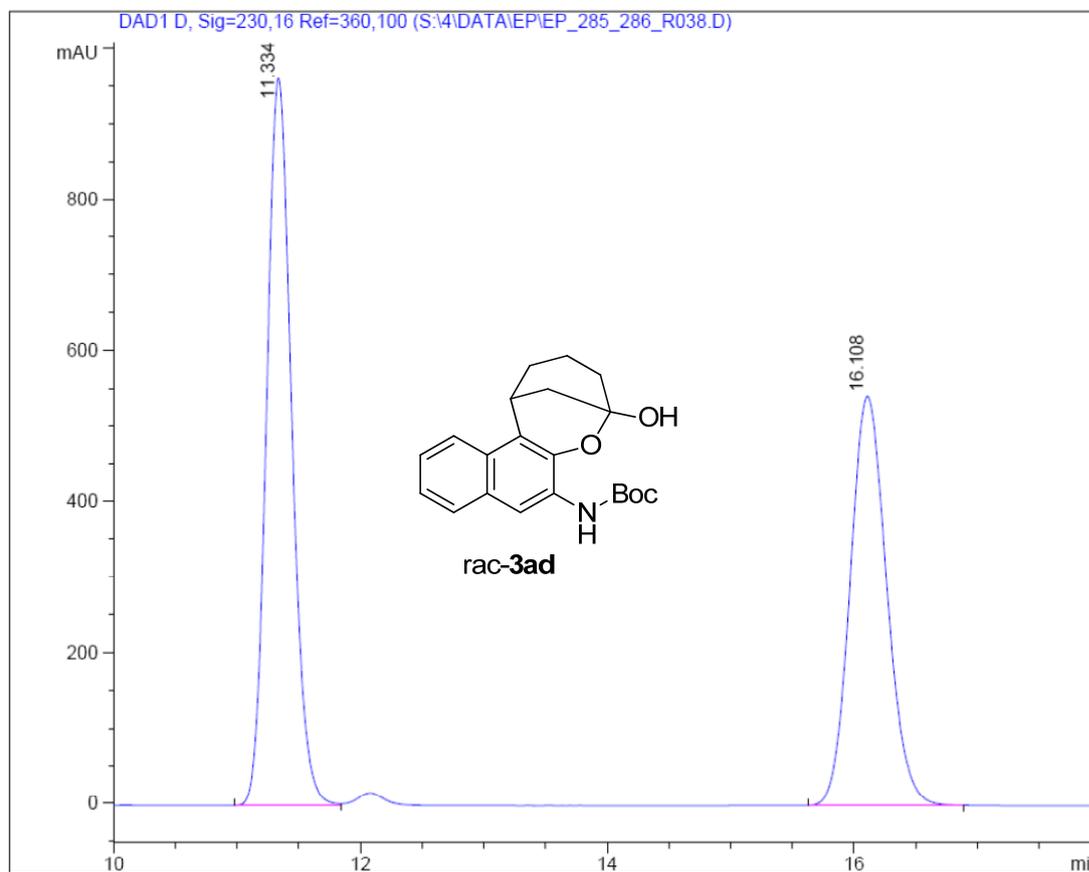
*****-->
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	15.969	BB	0.444	11.535	345.854	10.698	0.000	
2	24.925	BB	0.659	64.366	2887.069	89.302	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_285_286_R038.D
Sample name: EP_285_286_R
Sample Info: AD-H, 0.7 ml/min, 9:1 Hex/i-PrOH, 230 nm, 25°
x rac rac prima prova ->
Acquired by: EP on: 17/07/2012 18.10.21
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 41 Volume: 10uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 25°C



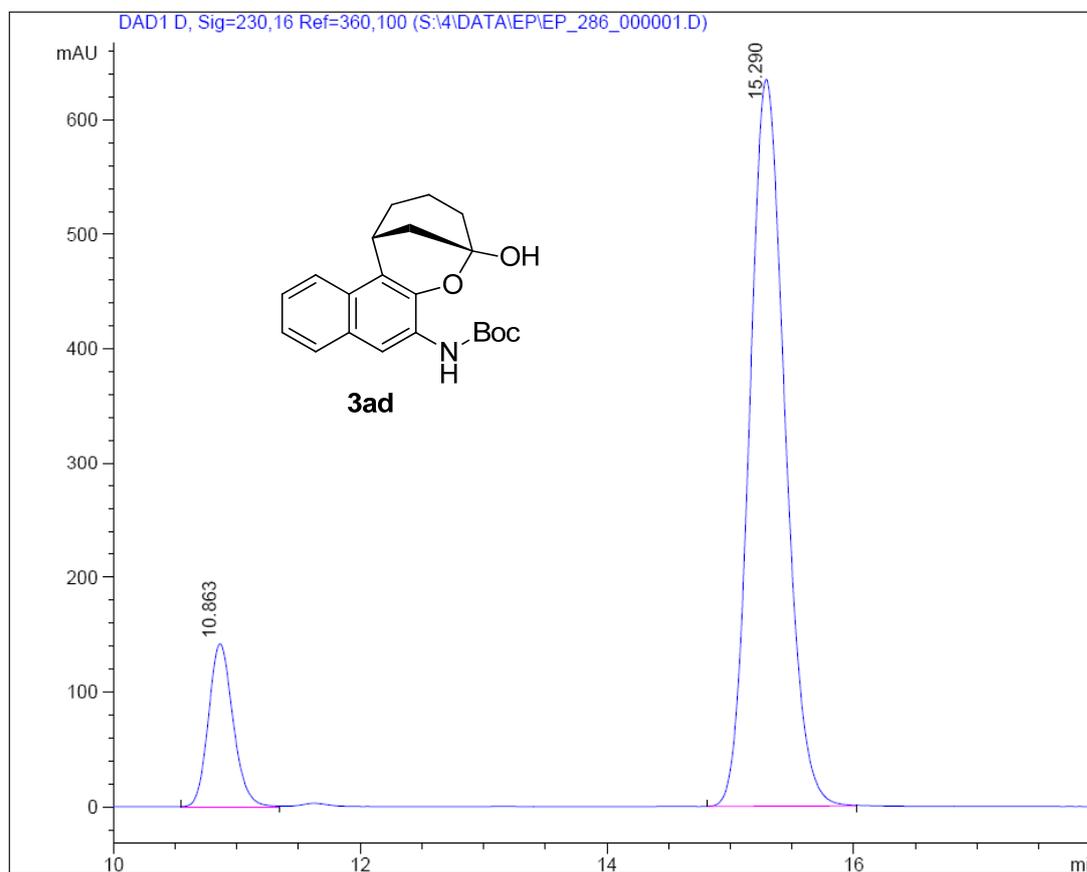
*****->
Area Percent Report

*****->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	11.334	BV	0.219	963.370	13636.094	55.012	0.000	
2	16.108	BB	0.320	542.180	11151.326	44.988	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_286_000001.D
Sample name: EP_286_
Sample Info: AD-H 0.7 ml-min, Hex/i-PrOH 9:1 230 nm, 25°C.
3 (Boc-amino)-2-naphthol + cicloesenone cat QD-NH2 ->
Acquired by: EP on: 28/06/2012 14.13.30
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 41 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 25.01°C



*****-->

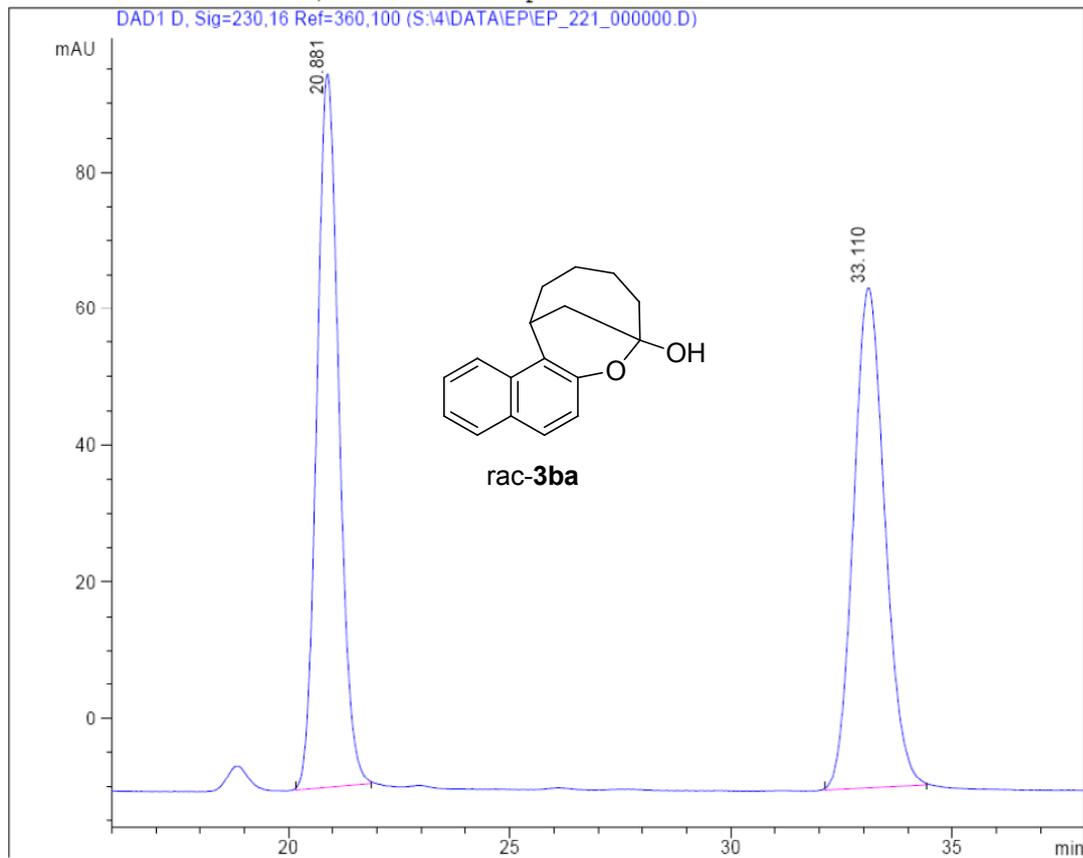
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	10.863	BB	0.213	142.021	1965.635	13.511	0.000	
2	15.290	BB	0.307	635.167	12582.741	86.489	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_221_000000.D
Sample name: EP_221_
Sample Info: Lux-Cellulose 2; Hex-Ipa 95:5; T=20°C; 0,65ml/min; 230n ->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.650 mL/min Temp.: 20°C



*****-->

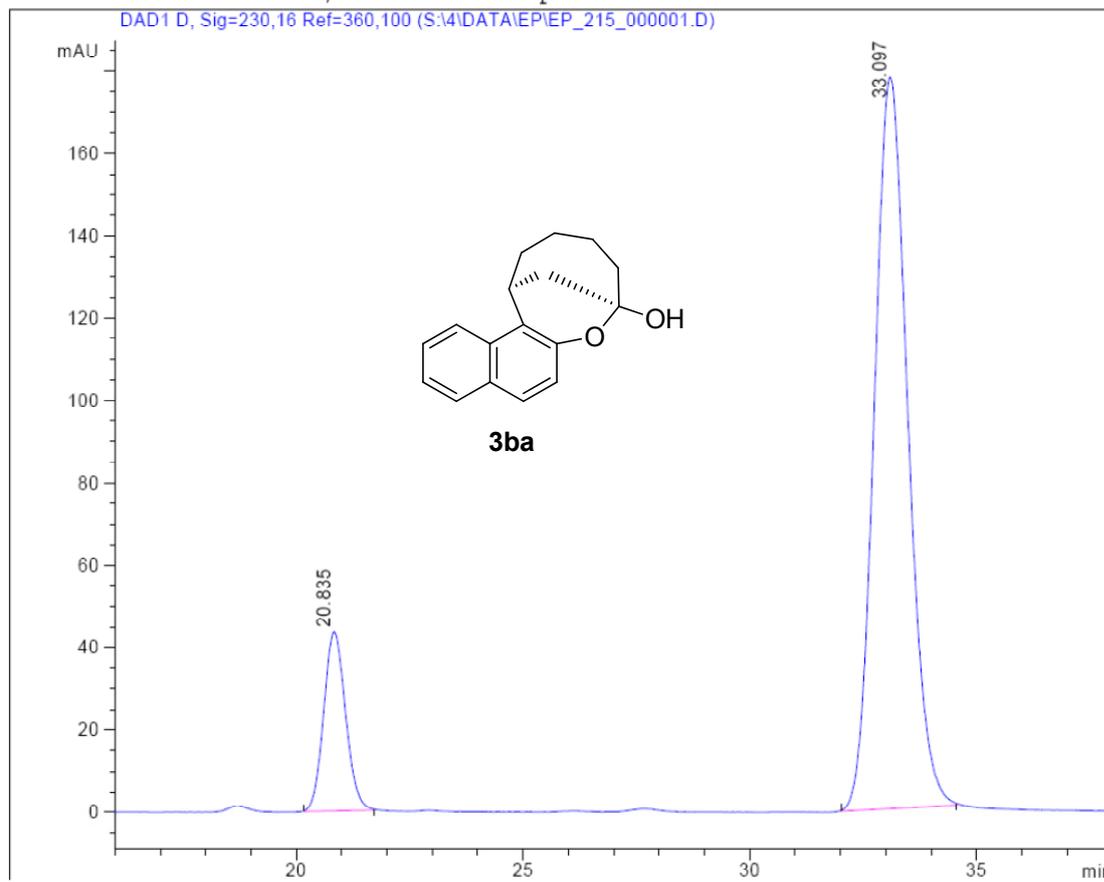
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	20.881	BB	0.532	104.389	3615.333	50.042	0.000	
2	33.110	BB	0.747	73.205	3609.264	49.958	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_215_000001.D
Sample name: EP_215_
Sample Info: Lux-Cellulose 2; Hex-Ipa 95:5; T=20°C; 0,65ml/min; 230n ->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.650 mL/min Temp.: 19.99°C

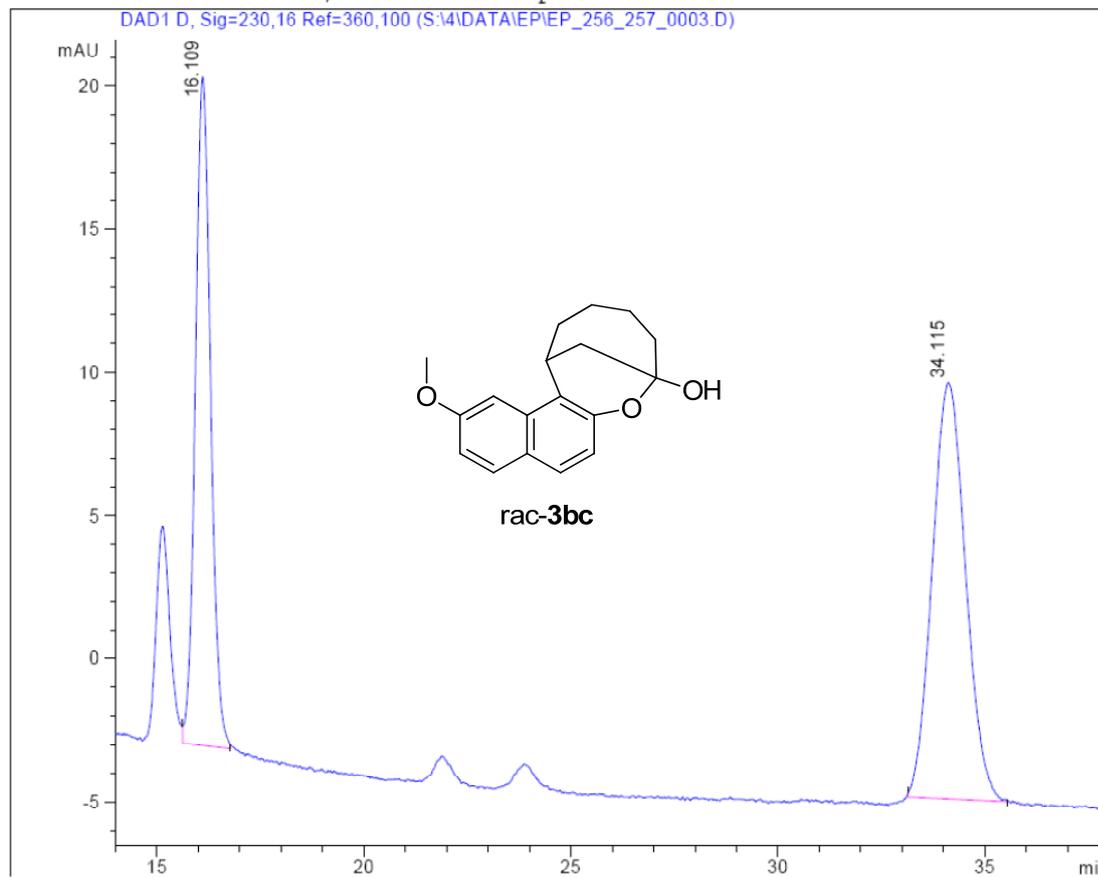


Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	20.835	BB	0.511	43.537	1465.176	13.833	0.000	
2	33.097	BB	0.797	177.685	9126.369	86.167	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_256_257_0003.D
Sample name: EP_256_257_
Sample Info: Lux-Cellulose-2, 0.7 ml/min, 9:1 Hex/i-PrOH, 230 nm, 23 ->
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 23°C

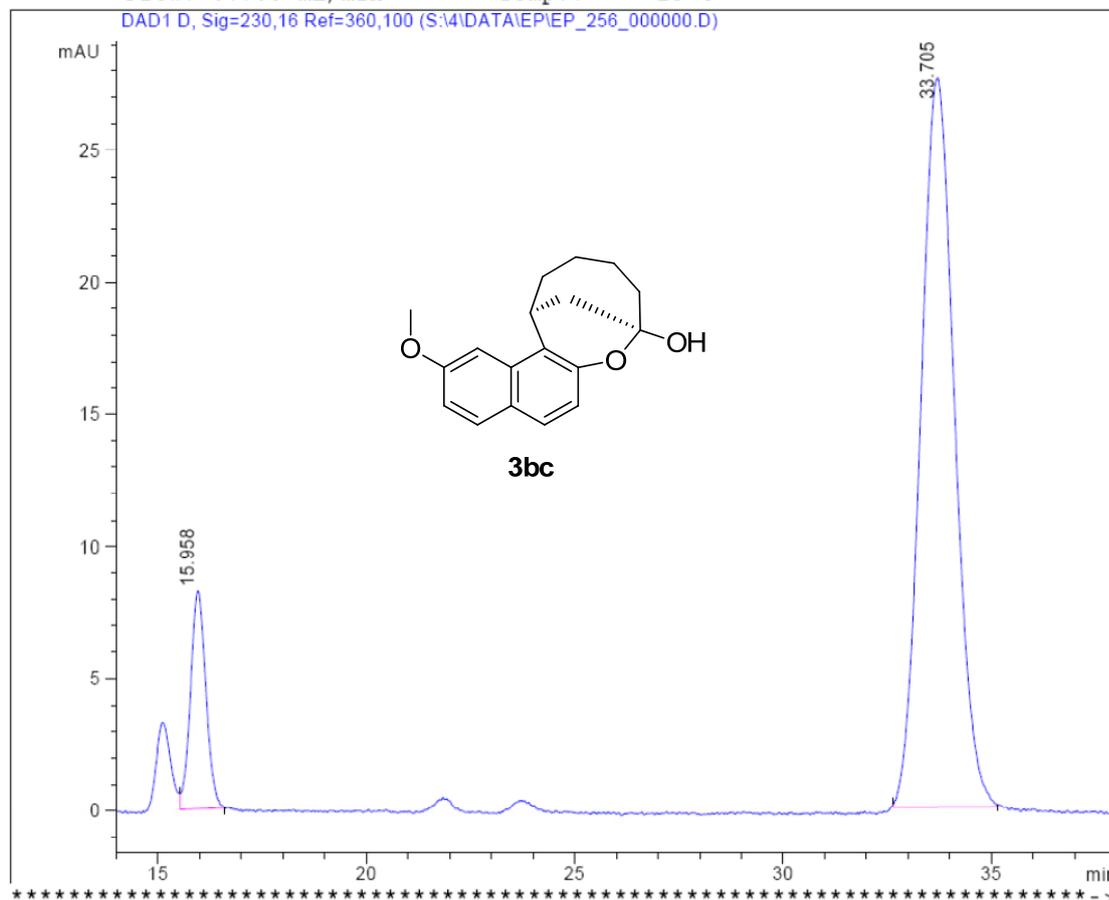


Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	16.109	VB	0.398	23.358	597.232	42.447	0.000	
2	34.115	BB	0.675	14.521	809.773	57.553	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_256_000000.D
Sample name: EP_256_
Sample Info: Lux-Cellulose-2, 0.7 ml/min, 9:1 Hex/i-PrOH, 230 nm, 23 ->
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 23°C

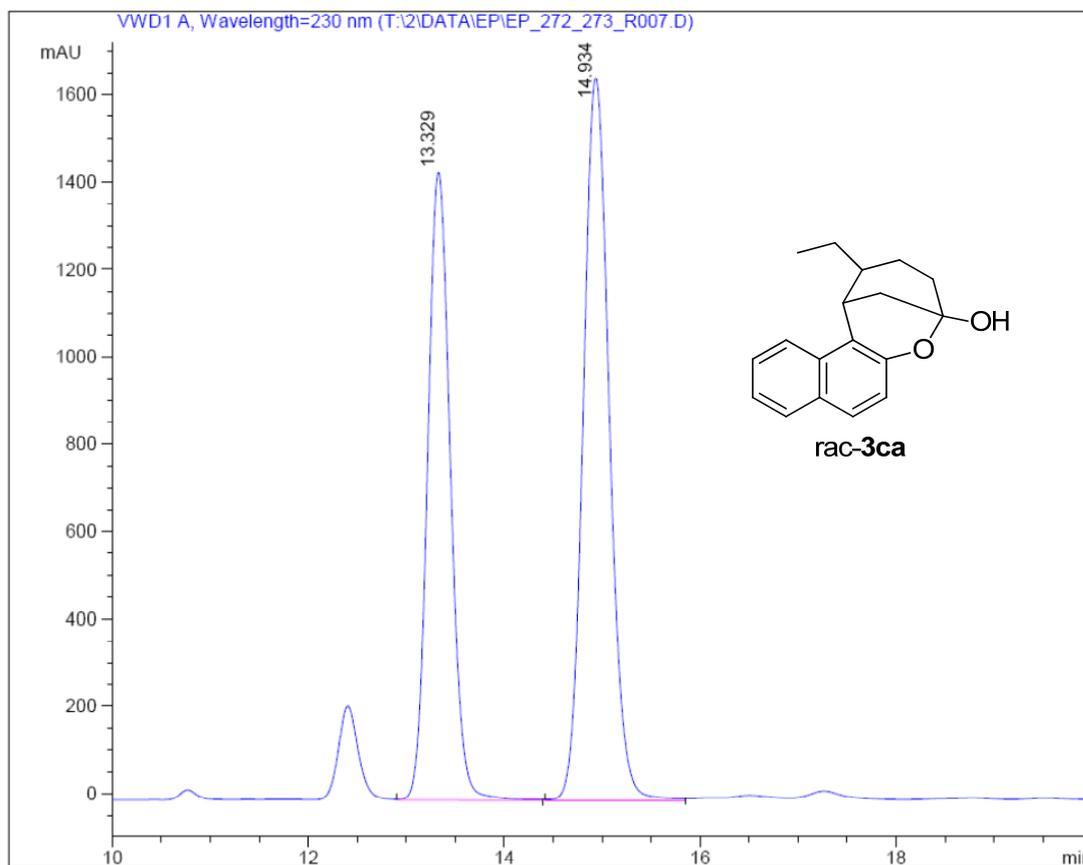


Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	15.958	VB	0.376	8.225	211.433	12.013	0.000	
2	33.705	BB	0.695	27.585	1548.619	87.987	0.000	
							0.000	

Data File: T:\2\DATA\EP\EP_272_273_R007.D
Sample name: EP_272_273_r
Sample Info: Lux cellulose 2, 0.4, 85:15, 230, not controlled.
x rac rac ->
Acquired by: EP on: 16/07/2012 14.19.48
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: Injecti->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.400 mL/min Temp.: 34.4°C

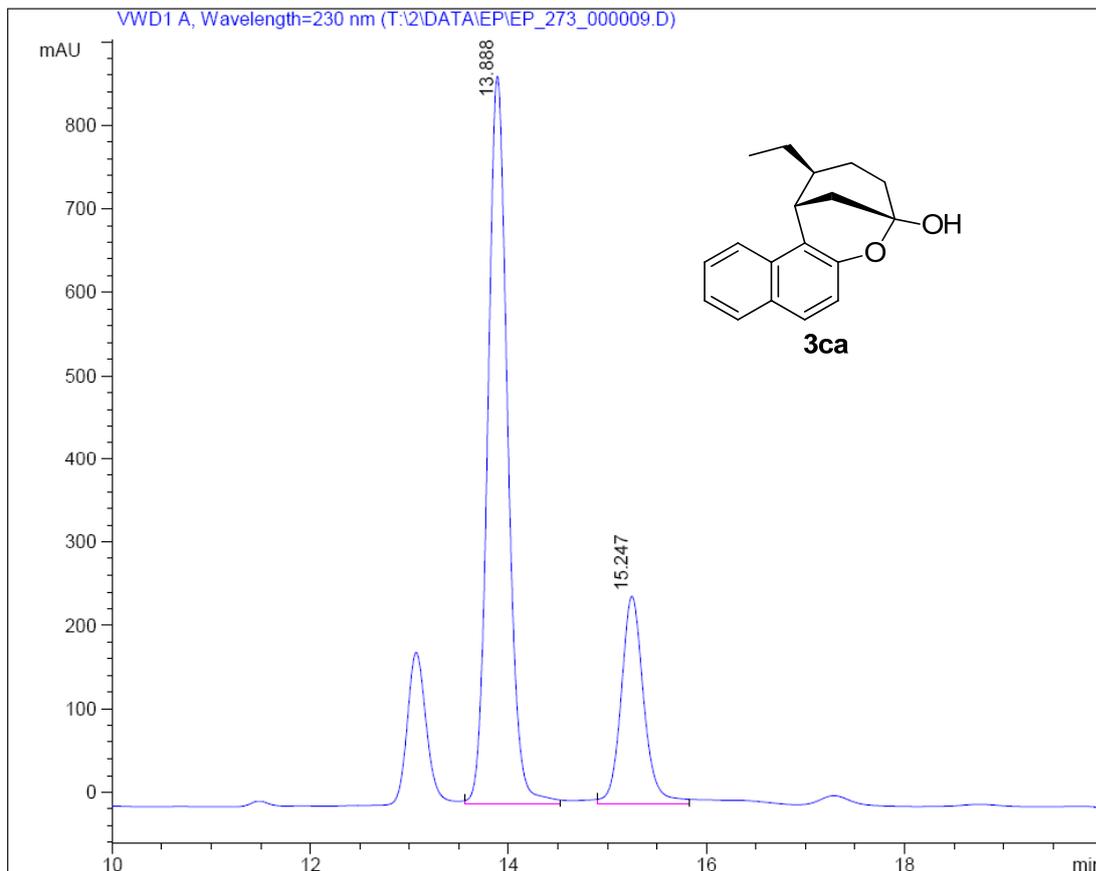


*****-->
Area Percent Report
*****-->
Signal: VWD1 A, Wavelength=230 nm

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	13.329	VB	0.247	1438.575	23124.908	43.087	0.000	
2	14.934	BV	0.287	1654.724	30544.869	56.913	0.000	
							0.000	

Data File: T:\2\DATA\EP\EP_273_000009.D
Sample name: EP_273_
Sample Info: Lux cellulose 2, 0.4, 85:15, 230, not controlled (33°C)

Acquired by: EP on: 16/07/2012 15.26.07
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: Injecti->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.400 mL/min Temp.: 34.68°C



*****-->

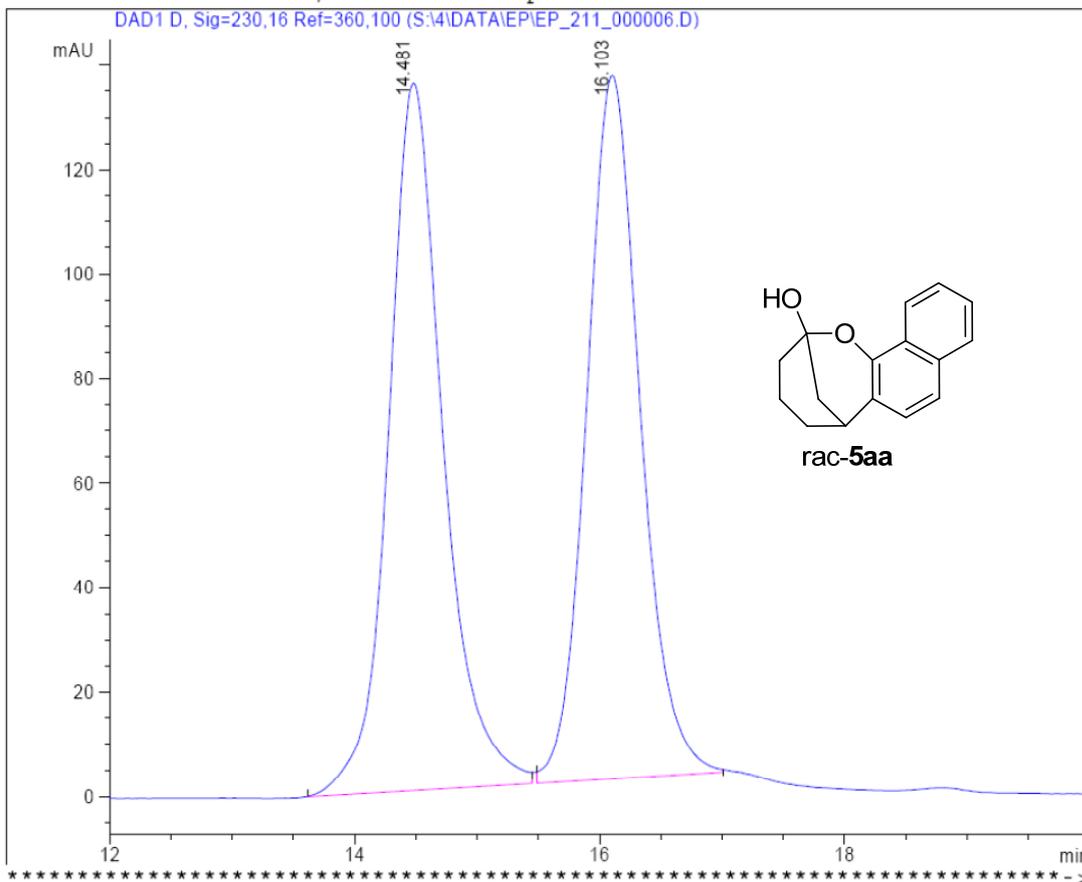
Area Percent Report

*****-->

Signal: VWD1 A, Wavelength=230 nm

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	13.888	MM	0.232	873.267	12157.461	75.357	0.000	
2	15.247	MM	0.266	249.149	3975.663	24.643	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_211_000006.D
Sample name: EP_211_
Sample Info: Lux-Amilose 2; Hex-i-PrOH 95:5; T=20°C; 0,65ml/min; 230 ->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.650 mL/min Temp.: 20.01°C



*****-->

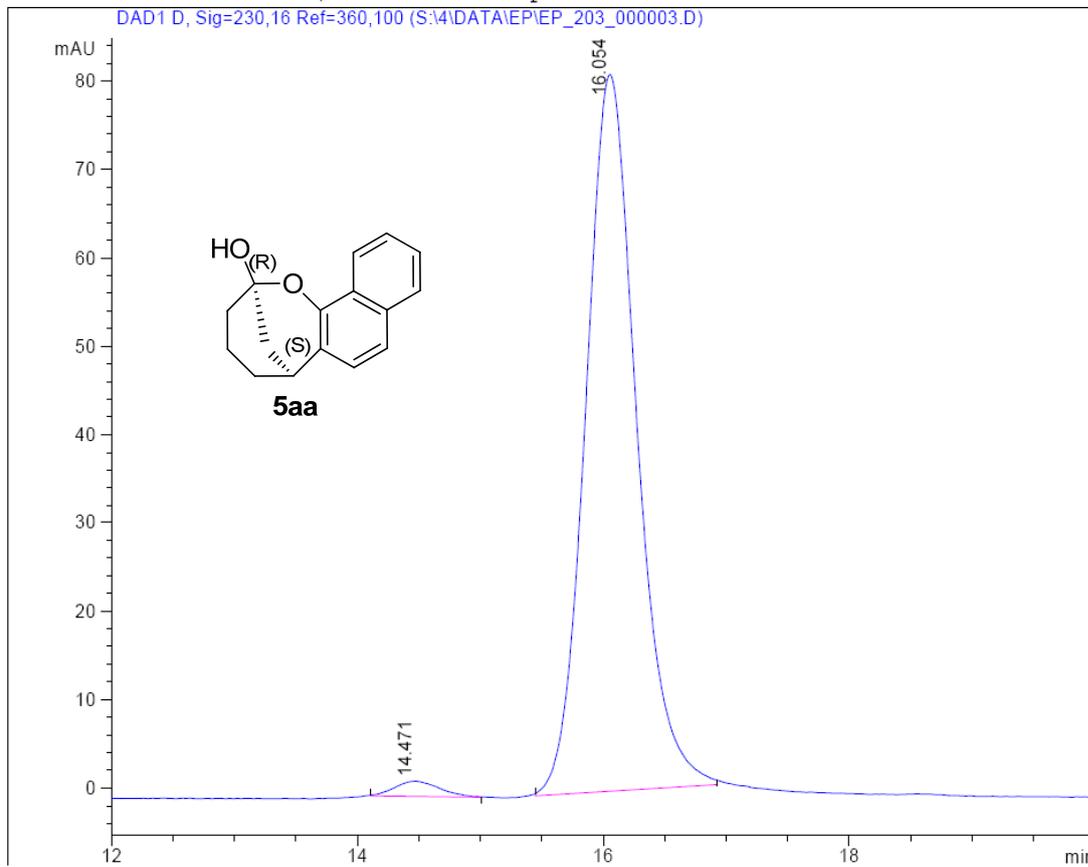
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	14.481	BB	0.464	135.230	4194.784	50.693	0.000	
2	16.103	BB	0.466	134.426	4080.022	49.307	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_203_000003.D
Sample name: EP_203_
Sample Info: Lux amilose-2 ---Hex-ipa 95:5 0.65 ml/min; 230nm 20° C ->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.650 mL/min Temp.: 19.99°C



*****-->

Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	14.471	BB	0.312	1.703	42.952	1.808	0.000	
2	16.054	BB	0.438	81.102	2332.375	98.192	0.000	

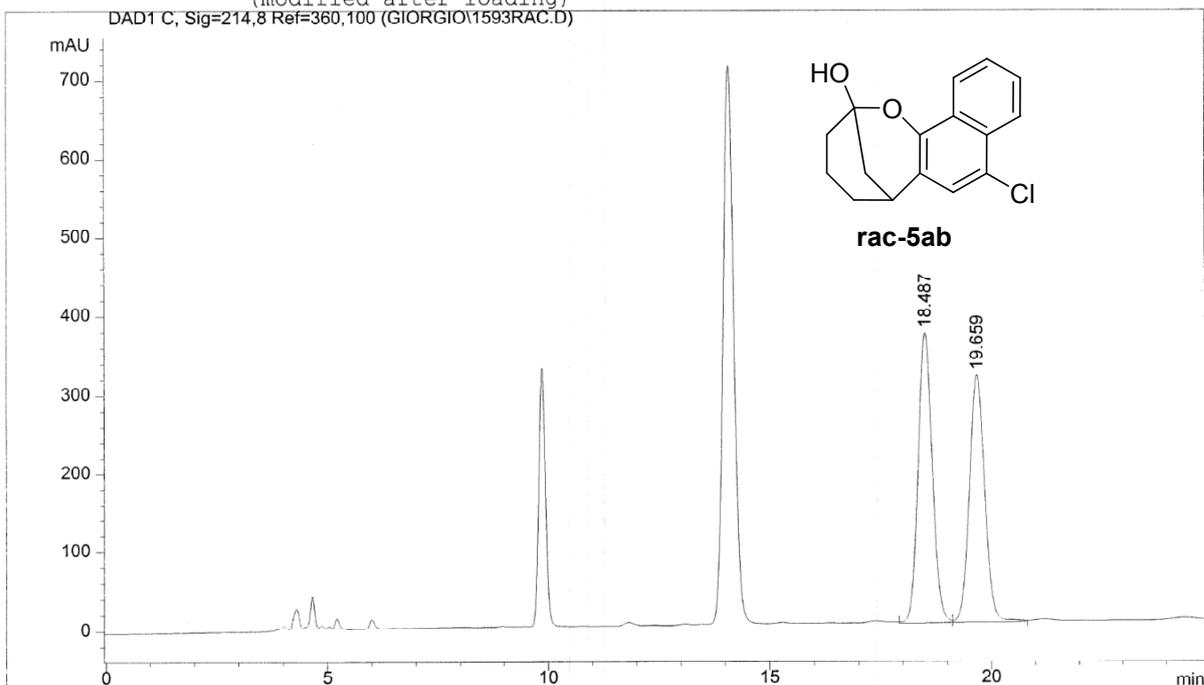
0.000

Data File D:\HPCHEM\2\DATA\GIORGIO\1593RAC.D

Sample Name: gb1593

4Cl-alfa-naftolo, ciclooesone, racemo
AD-H 95:5 0.70 ml/min

=====
Injection Date : 30/04/02 21.06.49
Sample Name : gb1593 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 30/04/02 20.43.19 by chiara
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.19.51 by Nicolas
(modified after loading)
=====



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Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.487	VV	0.3477	8195.54688	368.16284	51.8729
2	19.659	VV	0.3782	7603.72363	314.30829	48.1271

Totals : 1.57993e4 682.47113

Results obtained with enhanced integrator!

=====
*** End of Report ***

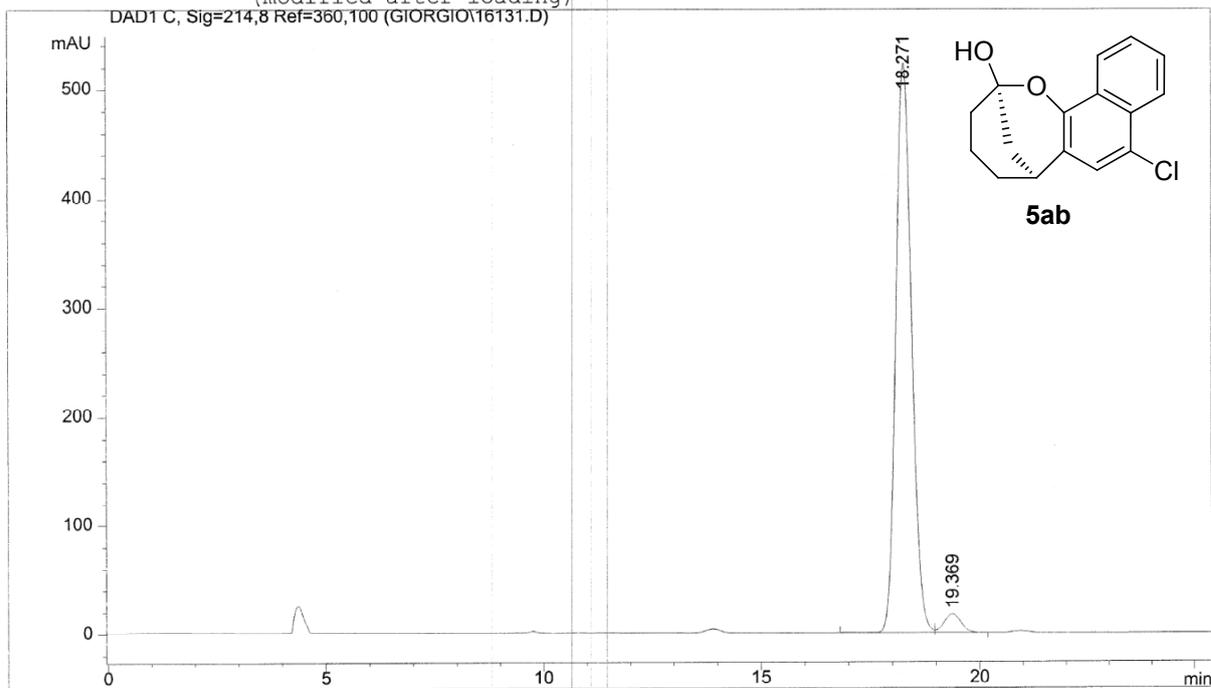
Data File D:\HPCHEM\2\DATA\GIORGIO\16131.D

Sample Name: gb1613

4Cl-alfa-naftolo, ciclooesenone, quinine
AD-H 95:5 0.70 ml/min

```
=====
Injection Date   : 30/04/02 22.34.45
Sample Name     : gb1613
Acq. Operator   : giorgio
Acq. Method     : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed    : 30/04/02 20.43.19 by chiara
                  (modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed    : 03/07/02 4.19.51 by Nicolas
                  (modified after loading)
=====
```

Location : Vial 1



Area Percent Report

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Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
```

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

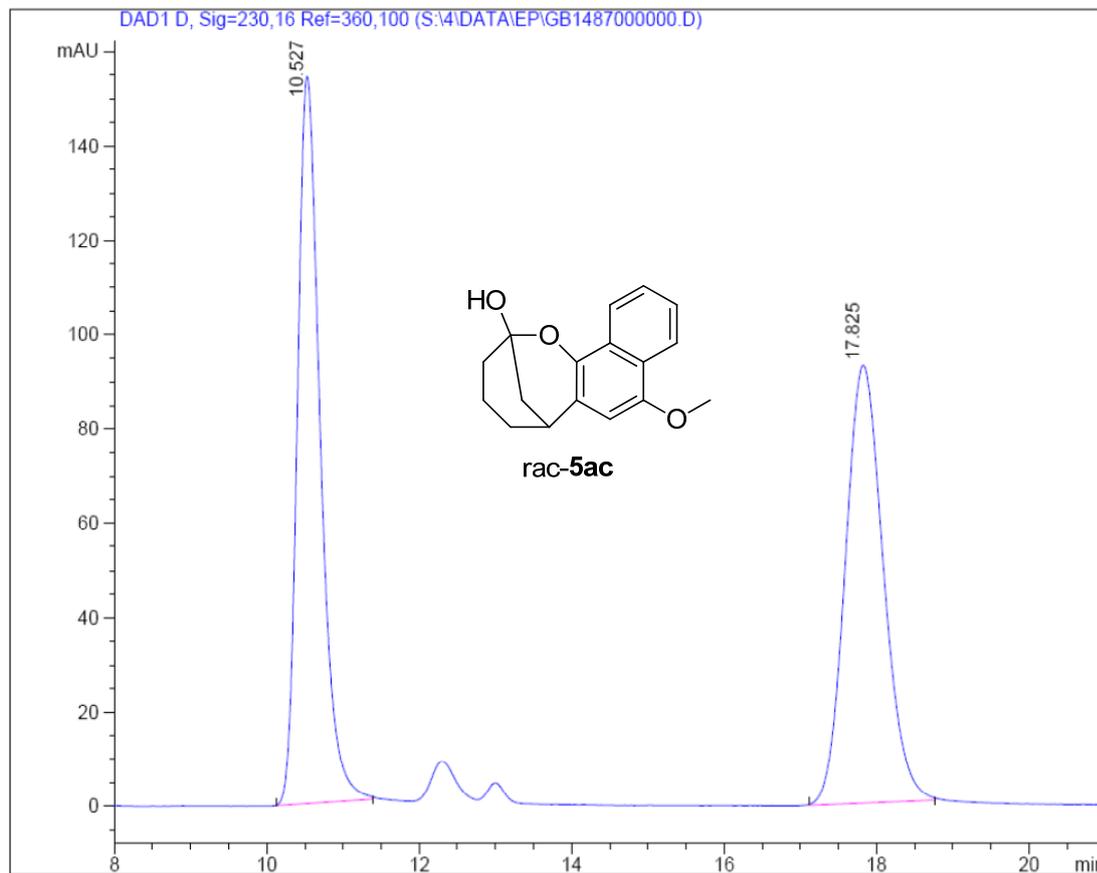
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.271	BV	0.3914	1.30591e4	522.79858	96.6567
2	19.369	VB	0.4131	451.70193	17.04720	3.3433

Totals : 1.35108e4 539.84579

Results obtained with enhanced integrator!

*** End of Report ***

Data File: S:\4\DATA\EP\GB1487000000.D
Sample name: GB1487
Sample Info: Lux-Cellulose 2, 0.7 ml/min, Hex/i-PrOH 87:13, 230 nm,
15°C. ->
Acquired by: EP on: 07/05/2012 15.11.13
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: 5uL
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 14.99°C

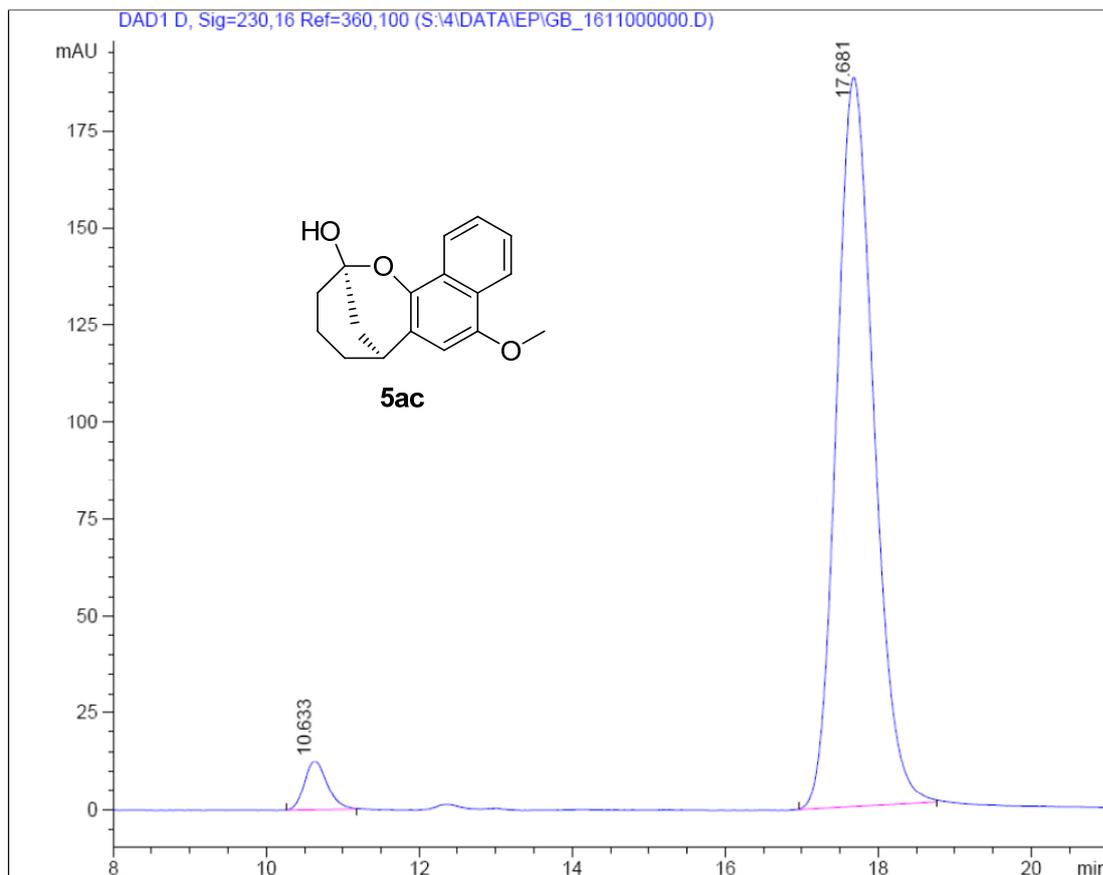


*****-->
Area Percent Report
*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	10.527	BB	0.317	154.231	3207.071	49.897	0.000	
2	17.825	BB	0.537	92.849	3220.300	50.103	0.000	
							0.000	

Data File: S:\4\DATA\EP\GB_1611000000.D
Sample name: GB_1611
Sample Info: Lux-Cellulose2; Hex-Ipa 87:13; T=15°; flusso=0,7ml/min;
230 nm; ->
Acquired by: EP on: 22/05/2012 18.25.53
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 25 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 14.99°C

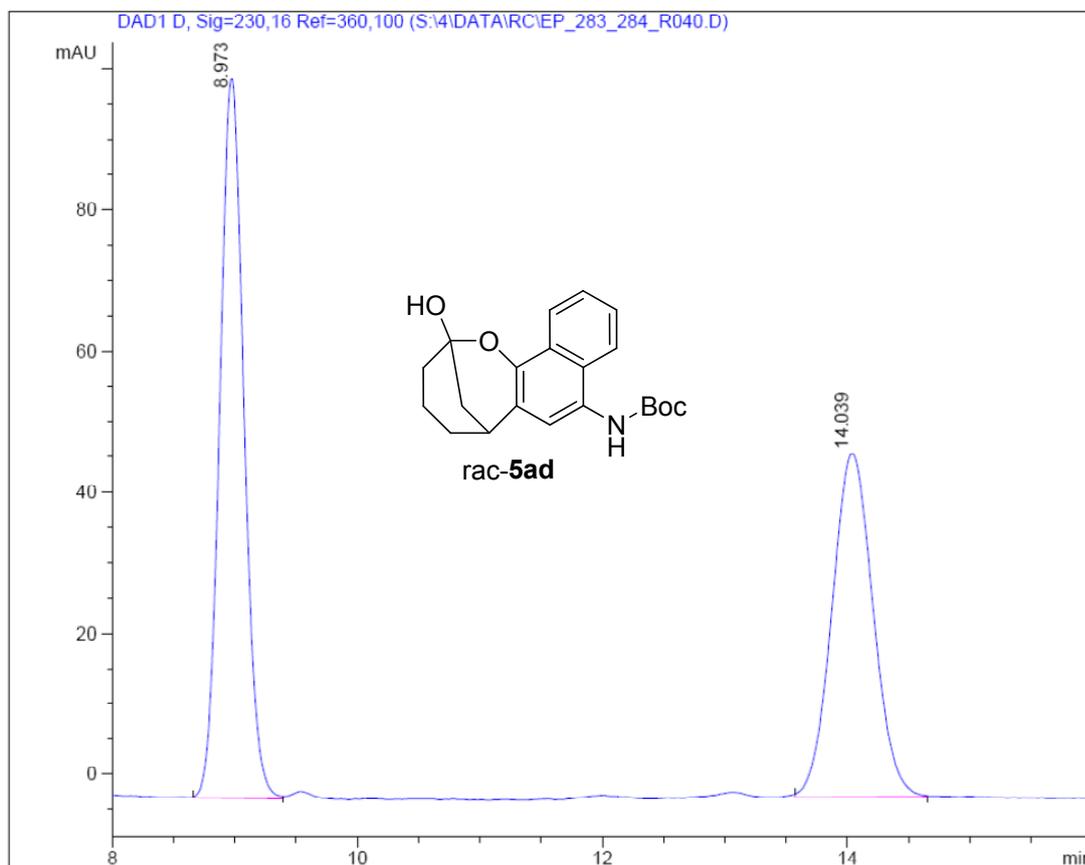


*****-->
Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	10.633	BB	0.311	12.460	257.209	3.880	0.000	
2	17.681	BB	0.526	187.866	6371.189	96.120	0.000	
							0.000	

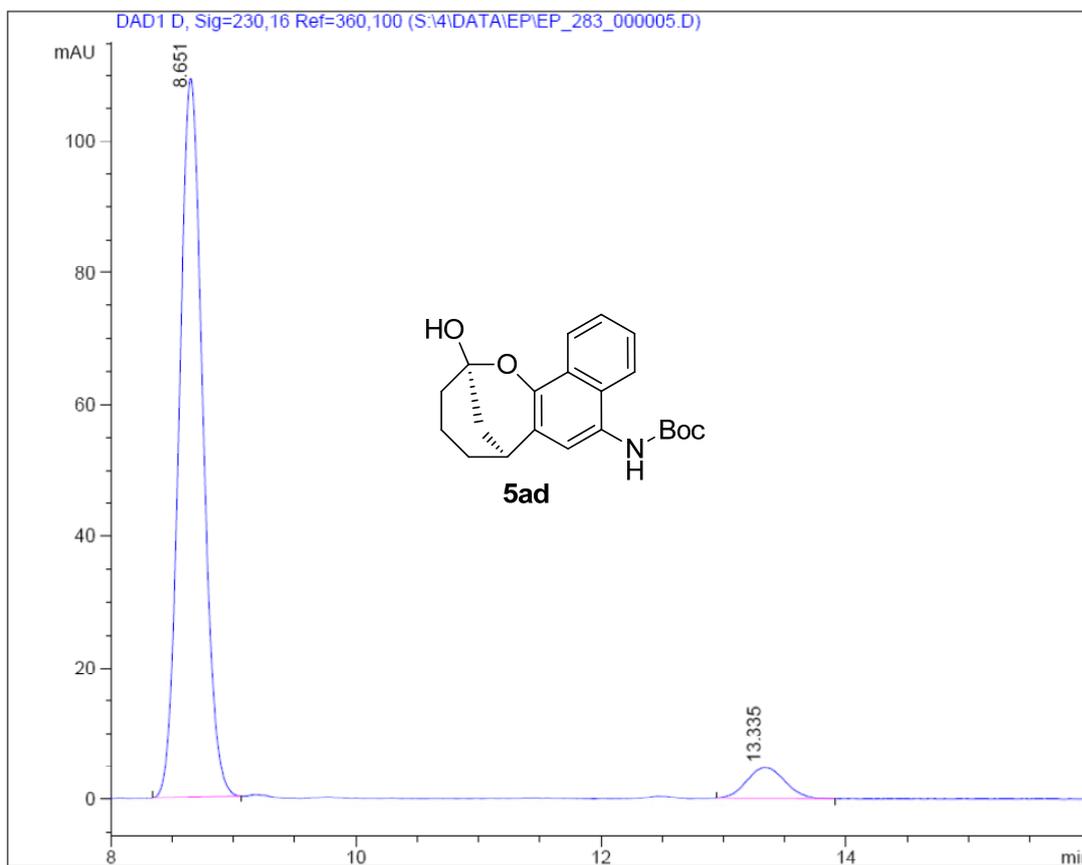
Data File: S:\4\DATA\RC\EP_283_284_R040.D
Sample name: EP_283_284_R
Sample Info: AD-H, 1 ml/min, 8:2 Hex/i-PrOH, 230 nm, 25°
x rac rac seconda prova ->
Acquired by: EP on: 18/07/2012 12.02.23
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 41 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 1.000 mL/min Temp.: 24.99°C



*****->
Area Percent Report
*****->
Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	8.973	BB	0.212	102.162	1402.210	55.212	0.000	
2	14.039	BB	0.362	48.710	1137.494	44.788	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_283_000005.D
 Sample name: EP_283_
 Sample Info: AD-H 1ml-min, Hex/i-PrOH 80:20, 230 nm, 25°C,
 4-(boc-amino)-1-naphthol + ciclohexenone cat Q-NH2 ->
 Acquired by: EP on: 29/06/2012 12.55.50
 Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
 Location: Vial 41 Volume: 5uL
 Column: Lux 5u Amylose-2 250 mm x 4.6 mm
 Flow: 1.000 mL/min Temp.: 24.99°C

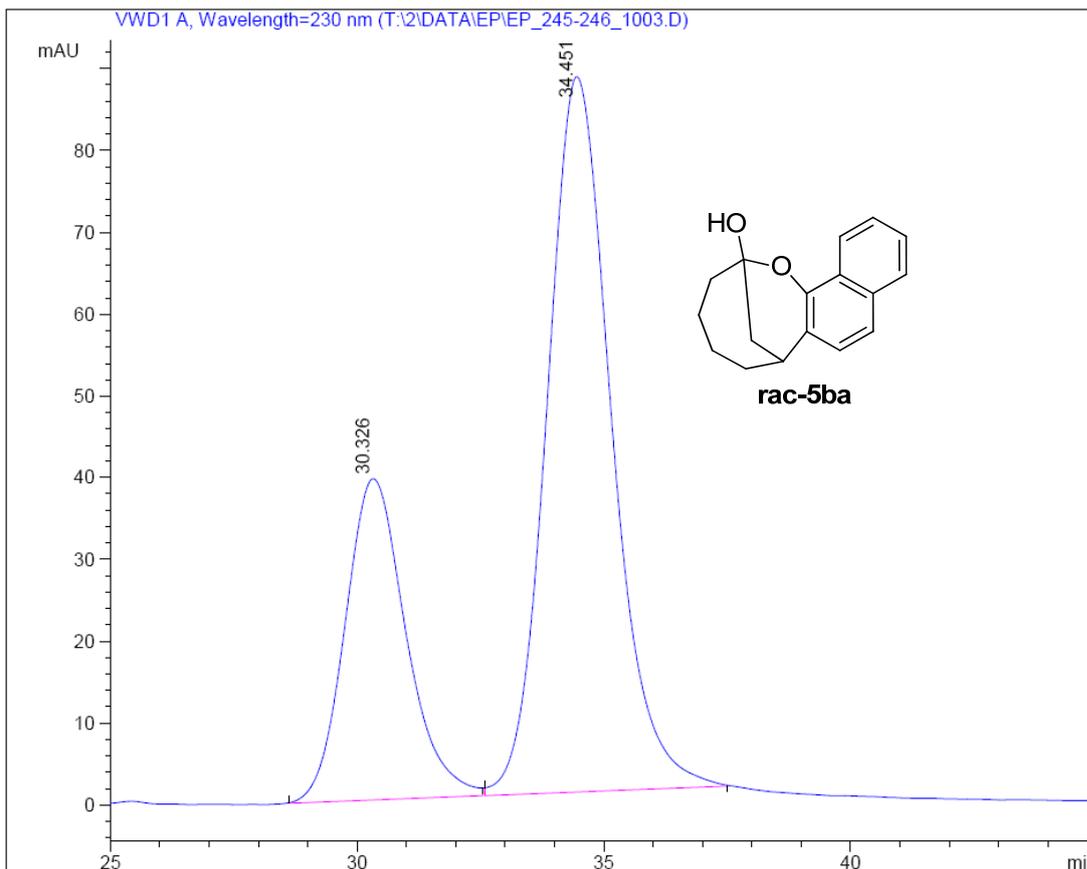


*****-->
 Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	8.651	BB	0.207	109.338	1454.041	93.287	0.000	
2	13.335	BB	0.302	4.735	104.638	6.713	0.000	
							0.000	

Data File: T:\2\DATA\EP\EP_245-246_1003.D
Sample name: EP_245-246_1
Sample Info: Lux amilose-2 Hex/i-PrOH 95:5 0.7ml/Min 20°, 230 nm
Friedel craft 1-naphthol + cycloheptenone RACEMO ->
Acquired by: EP on: 10/05/2012 10.10.37
Method: S:\4\METHODS\RGMO01-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: Injecti->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 19.99°C



*****-->

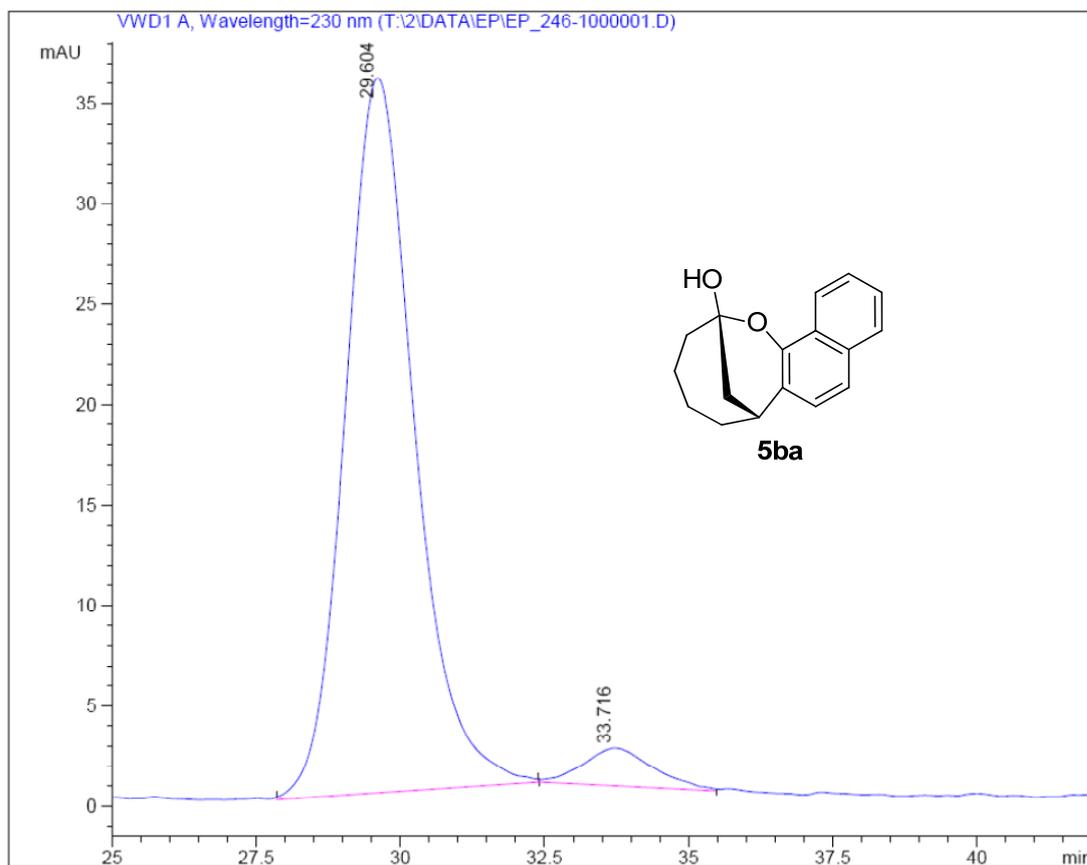
Area Percent Report

*****-->

Signal: VWD1 A, Wavelength=230 nm

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	30.326	BB	1.272	39.202	3355.477	29.696	0.000	
2	34.451	BB	1.376	87.416	7944.121	70.304	0.000	
							0.000	

Data File: T:\2\DATA\EP\EP_246-1000001.D
Sample name: EP_246-1
Sample Info: Lux amilose-2 Hex/i-PrOH 95:5 0.7ml/Min 20°, 230 nm
Friedel craft 1-naphthol + cycloheptenone cat QD-NH2 ->
Acquired by: EP on: 10/05/2012 12.04.00
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 1 Volume: Injecti->
Column: Lux Cellulose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 19.99°C

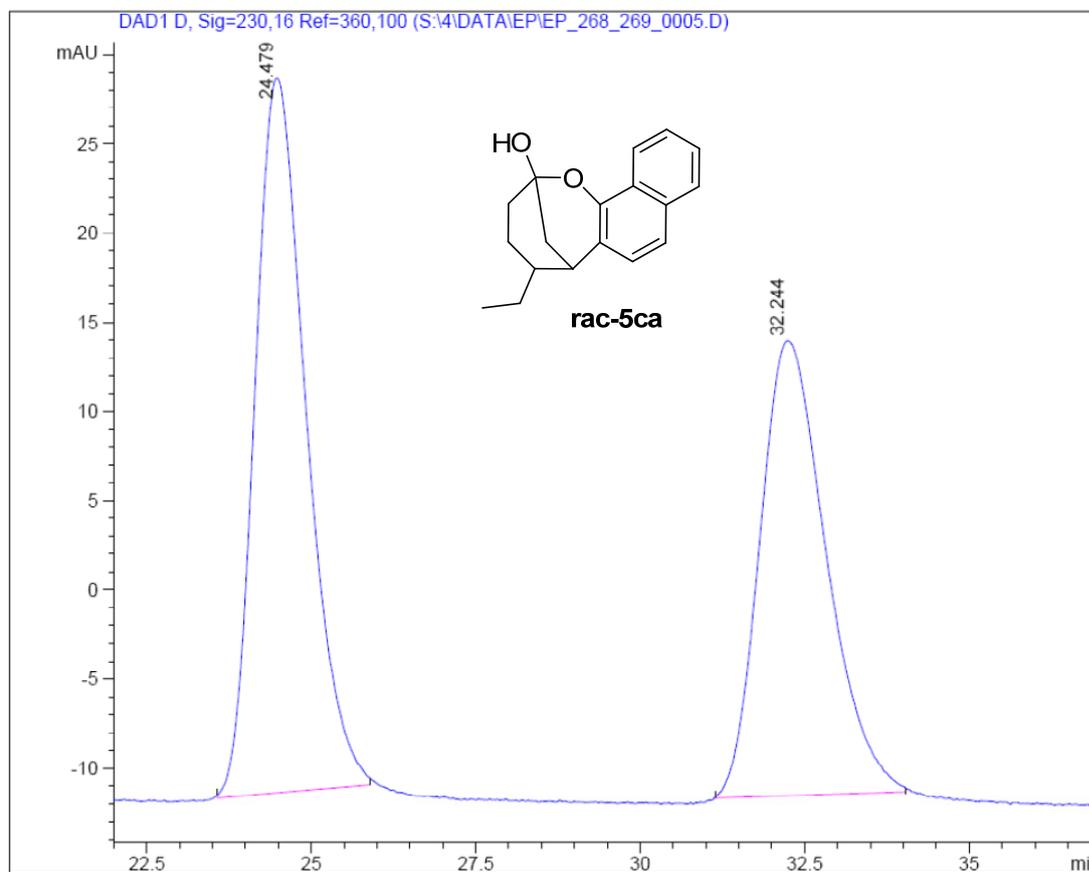


*****-->
Area Percent Report

Signal: VWD1 A, Wavelength=230 nm

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	29.604	MM R	1.390	35.635	2971.501	94.839	0.000	
2	33.716	MM T	1.416	1.903	161.696	5.161	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_268_269_0005.D
Sample name: EP_268_269_
Sample Info: OJ-H, 0.7 ml/min, 9:1 Hex/i-PrOH, 230 nm, 23°C.
1-naphthol + 4-methyl-cyclohexenone rac. ->
Acquired by: EP on: 12/06/2012 15.35.14
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 26 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 23°C



*****-->

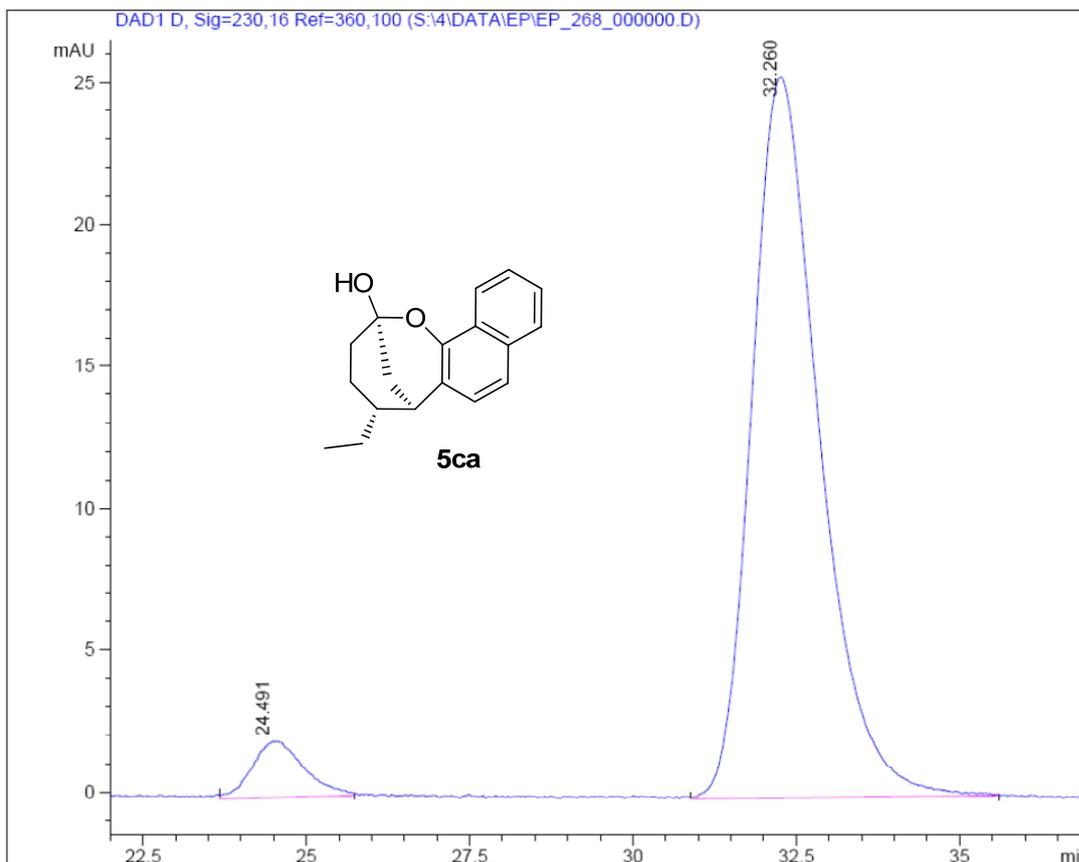
Area Percent Report

*****-->

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	24.479	BB	0.762	40.068	2172.226	54.914	0.000	
2	32.244	BB	0.859	25.531	1783.493	45.086	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_268_000000.D
Sample name: EP_268_
Sample Info: OJ-H, 0.7 ml/min, 9:1 Hex/i-PrOH, 230 nm, 23°C.
1-naphthol + 4-methyl-cyclohexenone Q-NH2. ->
Acquired by: EP on: 12/06/2012 16.30.41
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 27 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 22.99°C



*****->
Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

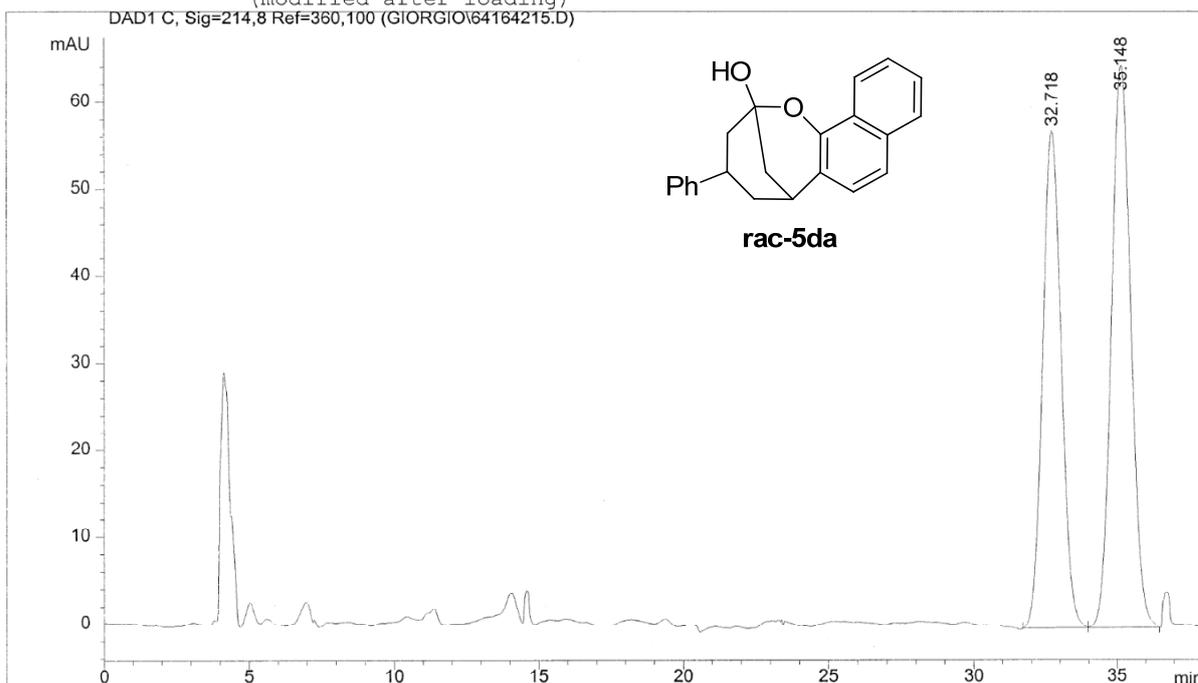
#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	24.491	MM	0.925	1.981	109.916	5.661	0.000	
2	32.260	MM	1.201	25.428	1831.670	94.339	0.000	
							0.000	

Data File D:\HPCHEM\2\DATA\GIORGIO\64164215.D

Sample Name: 1641+1642first

5Ph-cyclohexen-2-one, alfa-naftolo, rac, first spot
AD-H 95:5 Hexane:IPA 0.750 ml/min

=====
Injection Date : 30/05/02 3.12.52
Sample Name : 1641+1642first Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 30/05/02 1.02.34 by Riccardo
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.38.55 by Riccardo
(modified after loading)
=====



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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.718	PV	0.7033	2583.63062	57.15831	45.6109
2	35.148	VP	0.7395	3080.87646	64.70591	54.3891

Totals : 5664.50708 121.86422

Results obtained with enhanced integrator!

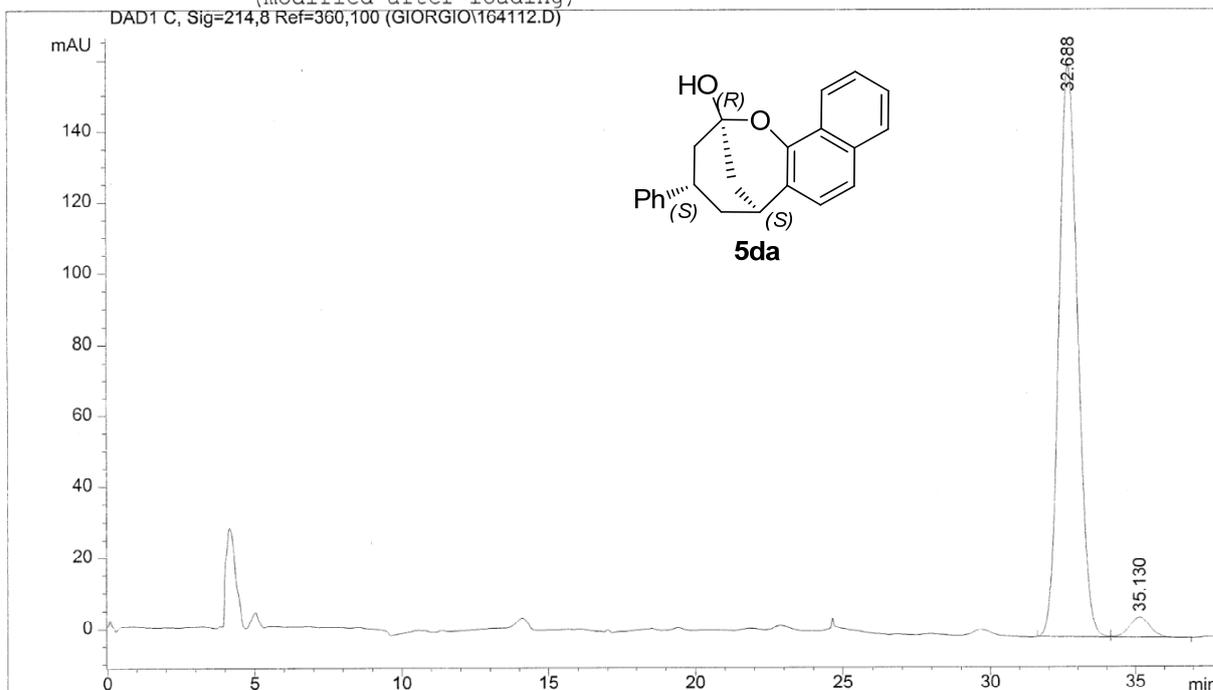
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\164112.D

Sample Name: 1641first

5Ph-cyclohexen-2-one, alfa-naftolo, quinine, first spot
AD-H 95:5 Hexane:IPA 0.750 ml/min

=====
Injection Date : 30-May-02, 04:11:29
Sample Name : 1641first Location : Vial 1
Acq. Operator : giorgio
Acq. Method : PAOLO.M
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.07.30 by Riccardo
(modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.688	BV	0.7064	7256.47656	160.82423	96.4294
2	35.130	VP	0.7296	268.69434	5.50345	3.5706

Totals : 7525.17090 166.32769

Results obtained with enhanced integrator!

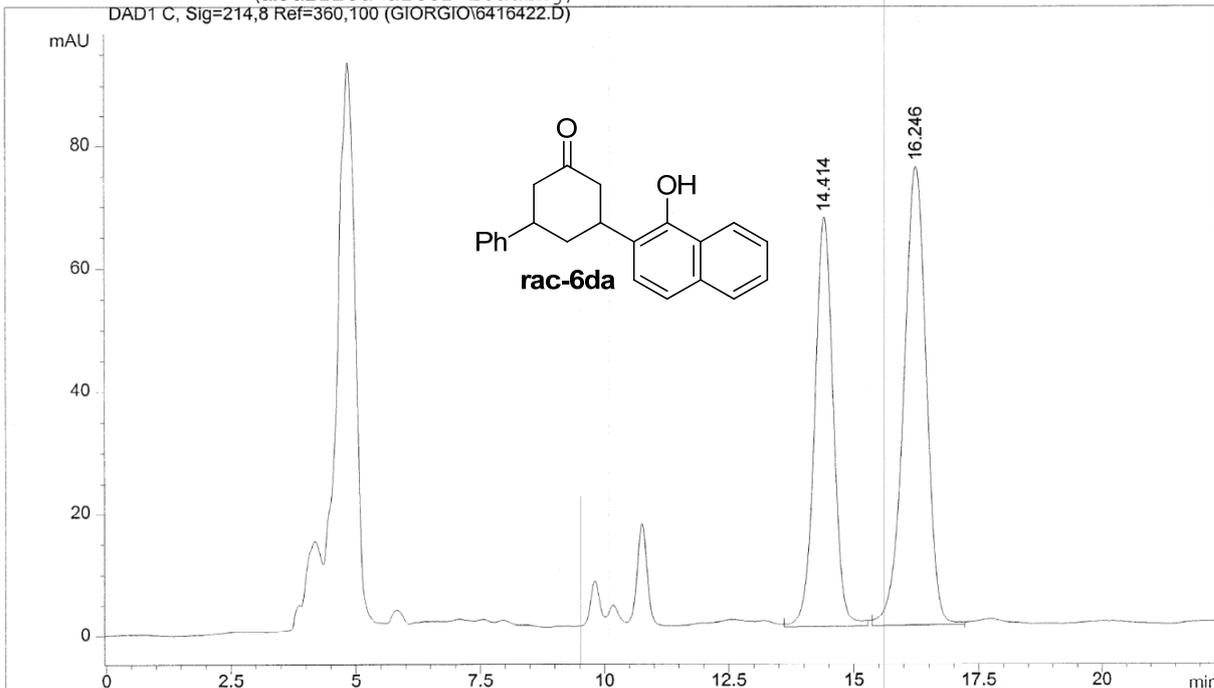
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*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\6416422.D

Sample Name: 1641+1642second

5Ph-cyclohexen-2-one, alfanaphthol, rac
AD-H, 8:2 hex/isopr 0,75 ml/min

=====
Injection Date : 02/06/02 1.09.47
Sample Name : 1641+1642second Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 02/06/02 0.27.15 by chiara
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.44.28 by Riccardo
(modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.414	VB	0.3981	1729.10950	66.77161	42.3313
2	16.246	BB	0.4868	2355.59766	74.92651	57.6687

Totals : 4084.70715 141.69811

Results obtained with enhanced integrator!

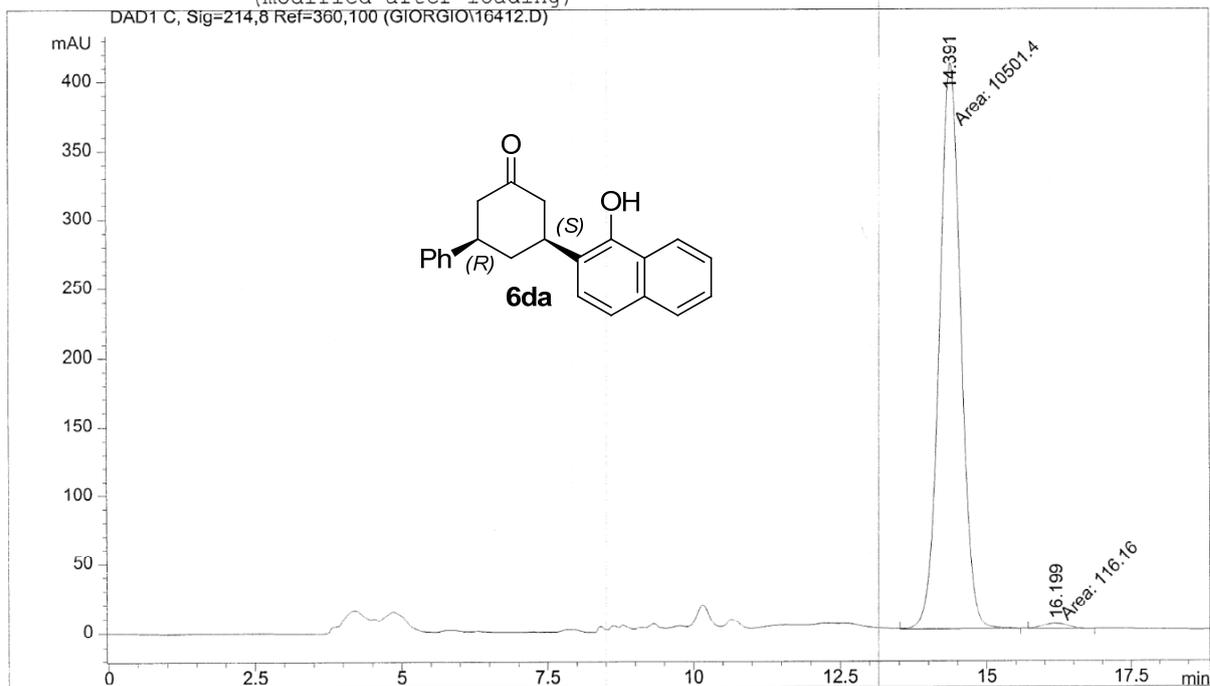
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\16412.D

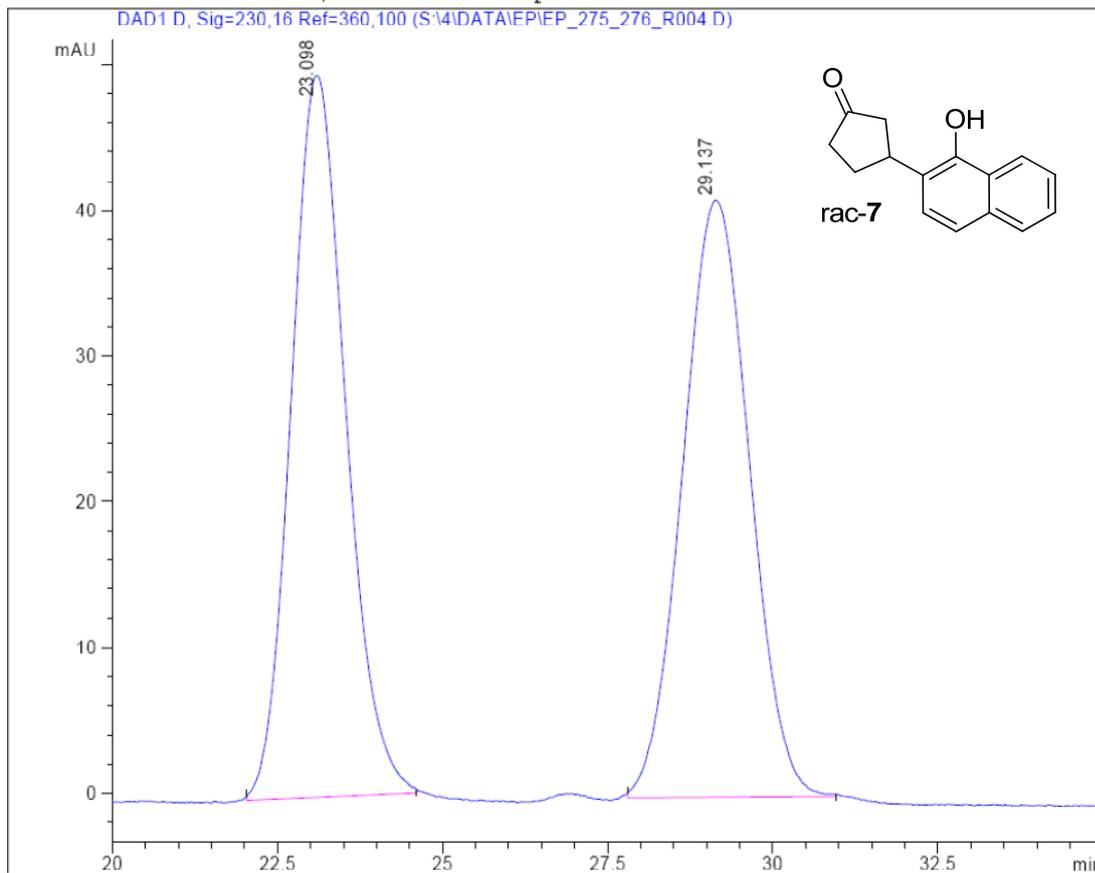
Sample Name: 1641second

5Ph-cyclohexen-2-one, alfanaphthol, quinine
AD-H, 8:2 hex/isopr 0,75 ml/min

=====
Injection Date : 02/06/02 1.33.39
Sample Name : 1641second Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 02/06/02 0.27.15 by chiara
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.44.28 by Riccardo
(modified after loading)



Data File: S:\4\DATA\EP\EP_275_276_R004.D
Sample name: EP_275_276_R
Sample Info: AS-H 0.7 ml-min, Hex/i-PrOH 85:15, 230 nm, 25°C. ->
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 24.99°C

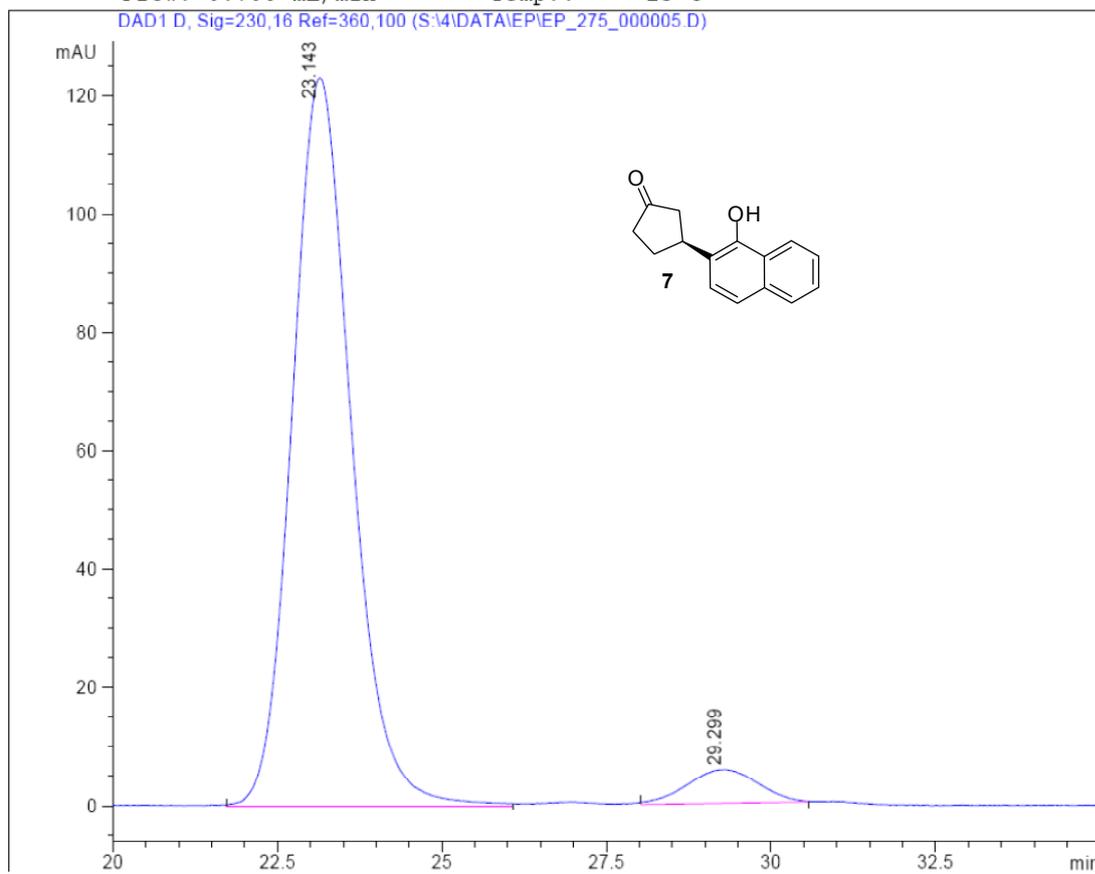


Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	23.098	BB	0.796	49.478	2842.634	49.775	0.000	
2	29.137	BB	0.915	40.950	2868.383	50.225	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_275_000005.D
Sample name: EP_275_
Sample Info: AS-H 0.7 ml-min, Hex/i-PrOH 85:15, 230 nm, 25°C. ->
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 25°C

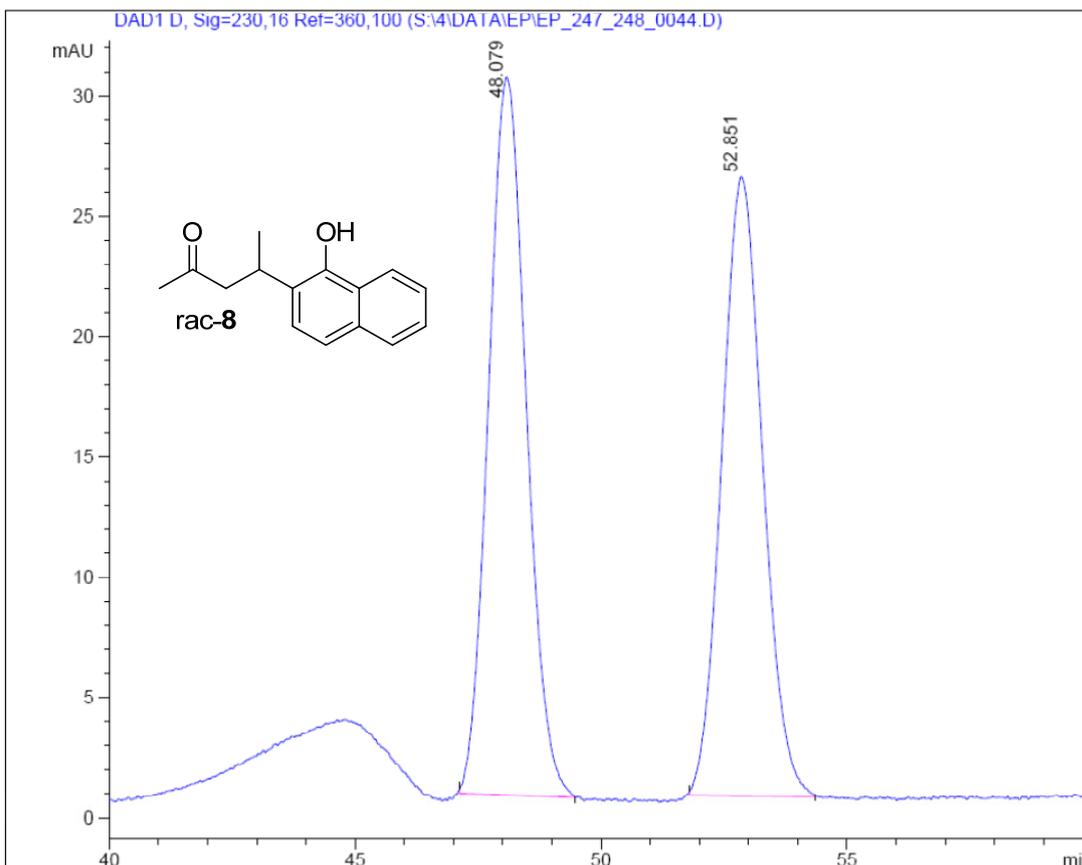


Area Percent Report

Signal: DAD1 D, Sig=230,16 Ref=360,100

#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	23.143	MM	1.043	123.185	7712.532	94.848	0.000	
2	29.299	MM	1.245	5.610	418.966	5.152	0.000	
							0.000	

Data File: S:\4\DATA\EP\EP_247_248_0044.D
Sample name: EP_247_248_
Sample Info: AD-H, 0.7 ml/min, 96:4 Hex/i-PrOH, 230 nm, 25°
rac per vedere se i picchi si separano. ->
Acquired by: EP on: 18/07/2012 15.51.08
Method: S:\4\METHODS\RGM001-C2-A90-B10-0.75-20C-S20.M
Location: Vial 41 Volume: 5uL
Column: Lux 5u Amylose-2 250 mm x 4.6 mm
Flow: 0.700 mL/min Temp.: 25°C



*****->
Area Percent Report
*****->

Signal: DAD1 D, Sig=230,16 Ref=360,100

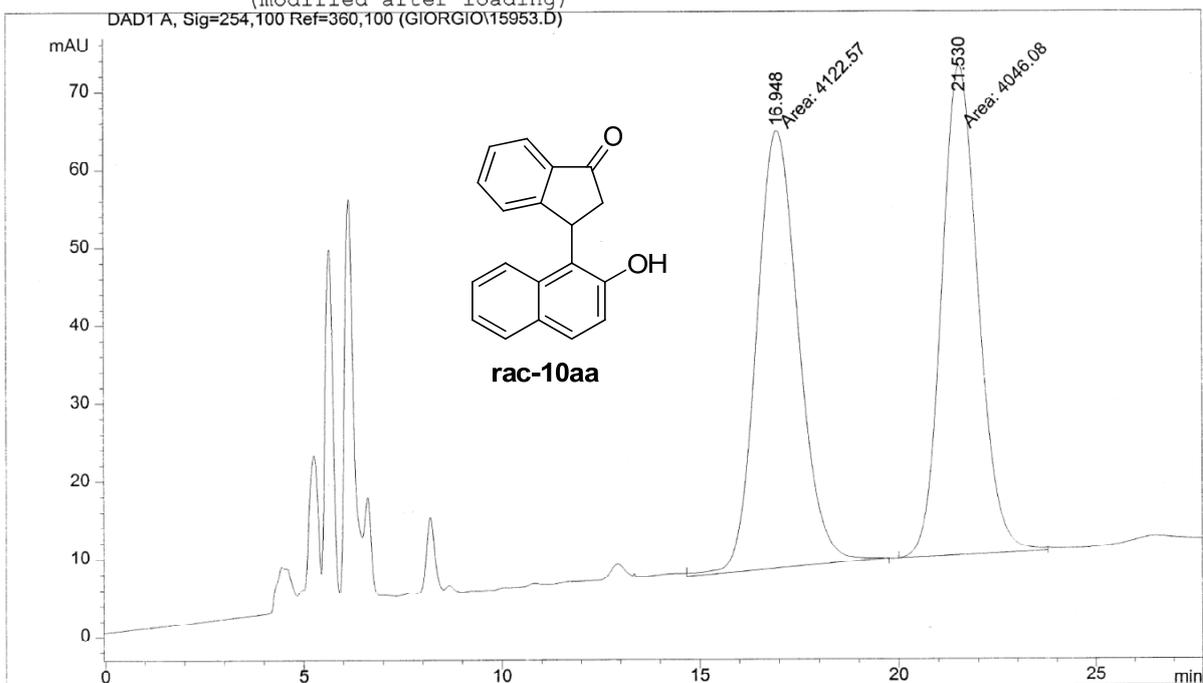
#	Time	Type	Width	Height	Area	Area %	Amount	Compound Nam
1	48.079	BB	0.679	29.825	1592.888	51.755	0.000	
2	52.851	BB	0.738	25.716	1484.838	48.245	0.000	
							0.000	

Data File D:\HPCHEM\2\DATA\GIORGIO\15953.D

Sample Name: GB1595

fc beta naftolo indenone, racemo
OD-H 9:1 0.70ml/min

=====
Injection Date : 15/04/02 21.41.35
Sample Name : GB1595 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 15/04/02 21.08.23 by giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.33.08 by Nicolas
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.948	MM	1.2213	4122.57178	56.25970	50.4682
2	21.530	MM	1.0714	4046.07593	62.94114	49.5318

Totals : 8168.64771 119.20084

Results obtained with enhanced integrator!

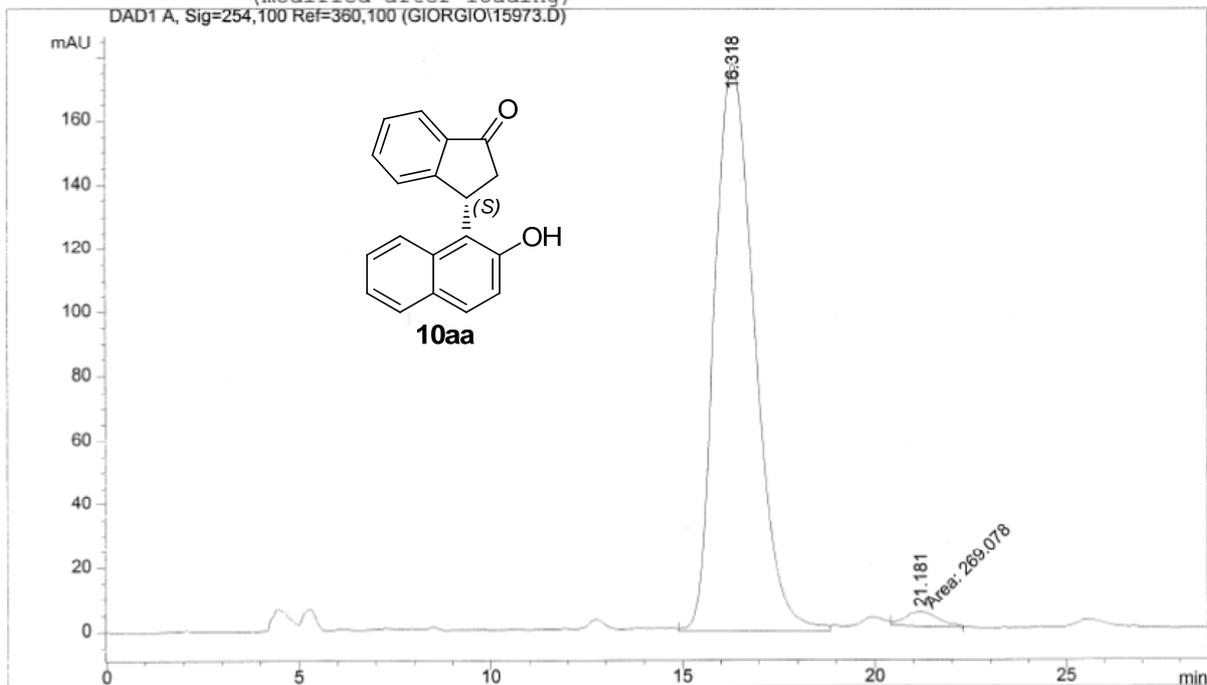
=====
*** End of Report ***
=====

Data File D:\HPCHEM\2\DATA\GIORGIO\15973.D

Sample Name: GB1597

fc beta naftolo indenone, QD-NH2
OD-H 9:1 0.70 ml/min

=====
Injection Date : 15/04/02 22.22.36
Sample Name : GB1597 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 15/04/02 21.08.23 by giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.33.08 by Nicolas
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.318	VV	1.0954	1.28053e4	177.98105	97.9420
2	21.181	MM	1.0221	269.07751	4.38784	2.0580

Totals : 1.30744e4 182.36889

Results obtained with enhanced integrator!

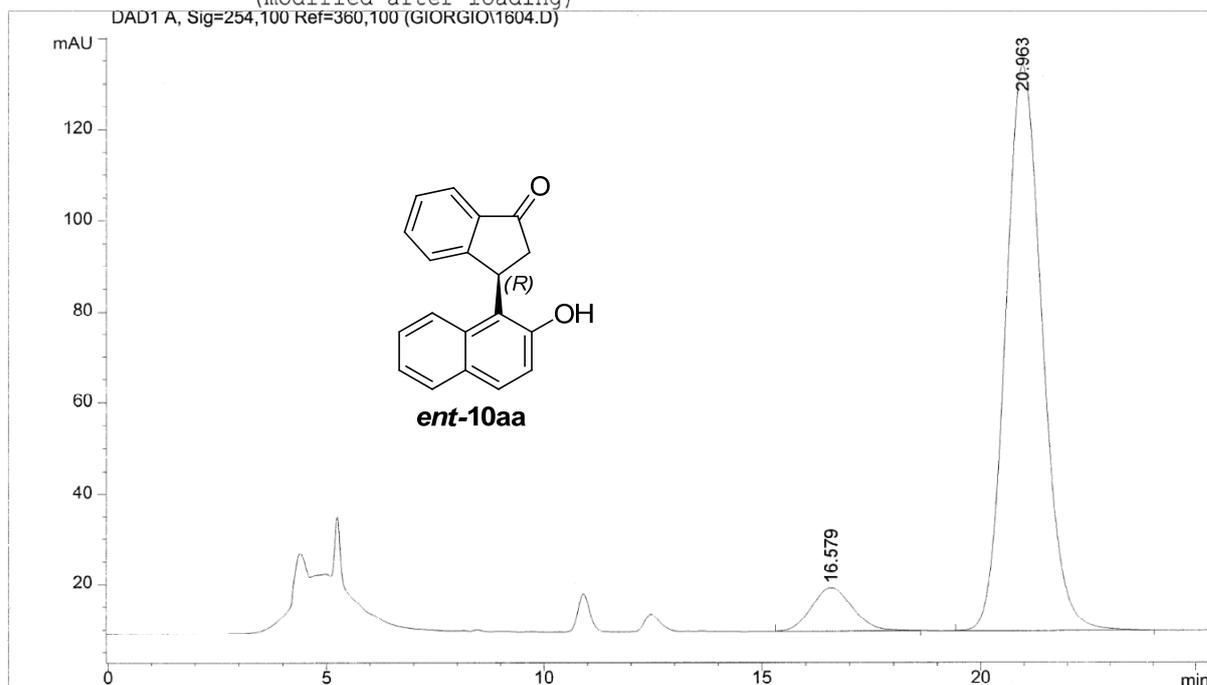
=====
*** End of Report ***
=====

Data File D:\HPCHEM\2\DATA\GIORGIO\1604.D

Sample Name: gb1604

FC, beta-naftolo, ciclooesenone, quinine
OD-H 90:10 0.700 ml/min

```
=====
Injection Date   : 21/04/02 5.21.44
Sample Name     : gb1604
Location        : Vial 1
Acq. Operator   : giorgio
Acq. Method     : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed    : 21/04/02 4.14.55 by simone
                  (modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed    : 03/07/02 4.48.29 by Nicolas
                  (modified after loading)
=====
```



=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
```

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.579	BB	1.0184	614.63019	9.40573	7.7303
2	20.963	BB	0.9201	7336.30273	124.38314	92.2697

Totals : 7950.93292 133.78887

Results obtained with enhanced integrator!

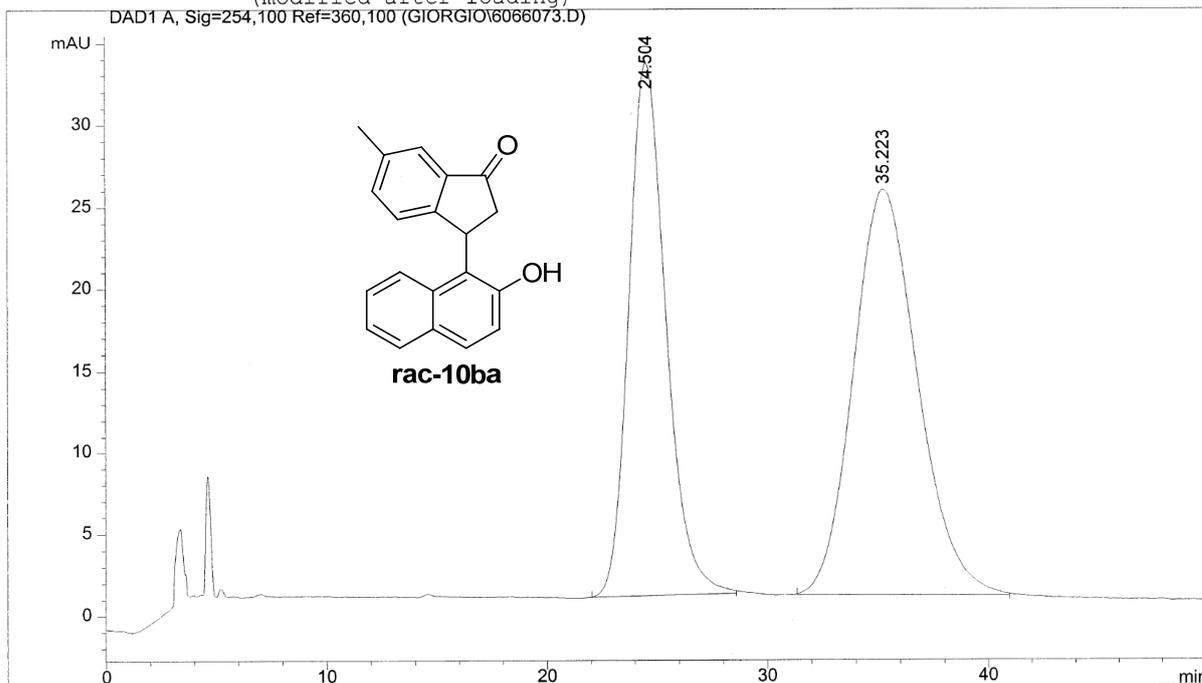
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\6066073.D

Sample Name: gb1606+gb1607

FC, beta-naftolo, 5-Me-indenone, racemo
Oj-H 90:10 1.0 ml/min

```
=====
Injection Date   : 23/04/02 2.23.17
Sample Name      : gb1606+gb1607           Location : Vial 1
Acq. Operator    : giorgio
Acq. Method      : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed     : 23/04/02 2.19.41 by giorgio
                  (modified after loading)
Analysis Method  : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed     : 03/07/02 4.33.08 by Nicolas
                  (modified after loading)
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.504	BB	1.8248	3819.77979	32.55754	43.2494
2	35.223	BB	2.8588	5012.20068	24.83272	56.7506

Totals : 8831.98047 57.39026

Results obtained with enhanced integrator!

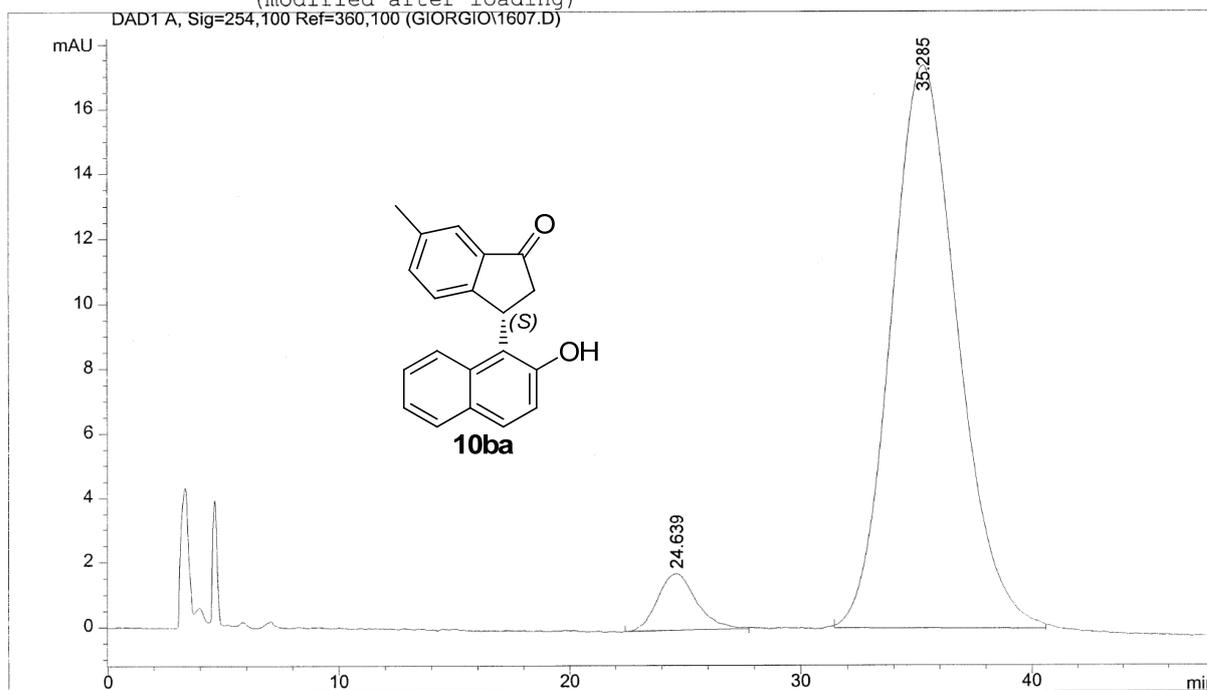
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\1607.D

Sample Name: gb1607

FC, beta-naftolo, 5-Me-indenone, quinidine
Oj-H 90:10 1.0 ml/min

=====
Injection Date : 23/04/02 3.21.50
Sample Name : gb1607 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 23/04/02 2.19.41 by giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.33.08 by Nicolas
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.639	PB	1.4577	203.73454	1.73536	5.5507
2	35.285	BB	2.6142	3466.68213	17.38621	94.4493

Totals : 3670.41667 19.12158

Results obtained with enhanced integrator!

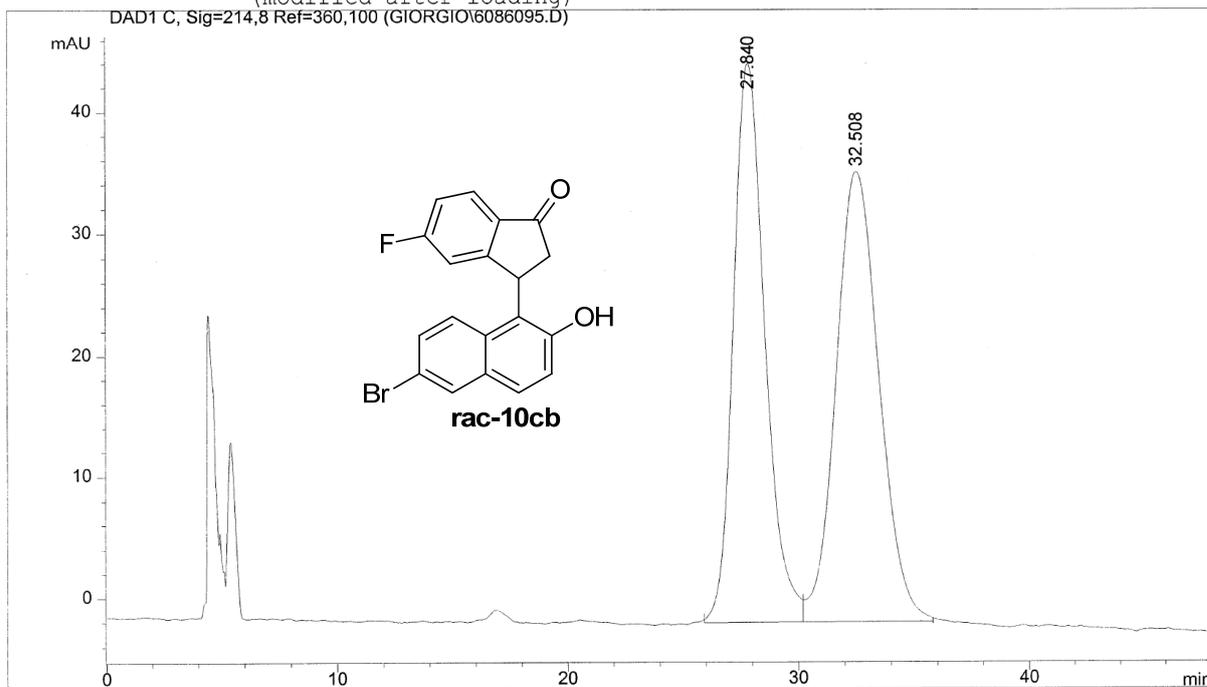
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\6086095.D

Sample Name: GB1608+GB1609

5F-indenone, 6-bromonaftolo, racemo
OD-H 95:5 0.70 ml/min

=====
Injection Date : 29/04/02 23.43.54
Sample Name : GB1608+GB1609 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 29/04/02 20.48.01 by simone
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.28.50 by Nicolas
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.840	BV	1.4147	4369.47607	46.06099	47.6912
2	32.508	VB	1.9677	4792.54785	37.08383	52.3088

Totals : 9162.02393 83.14482

Results obtained with enhanced integrator!

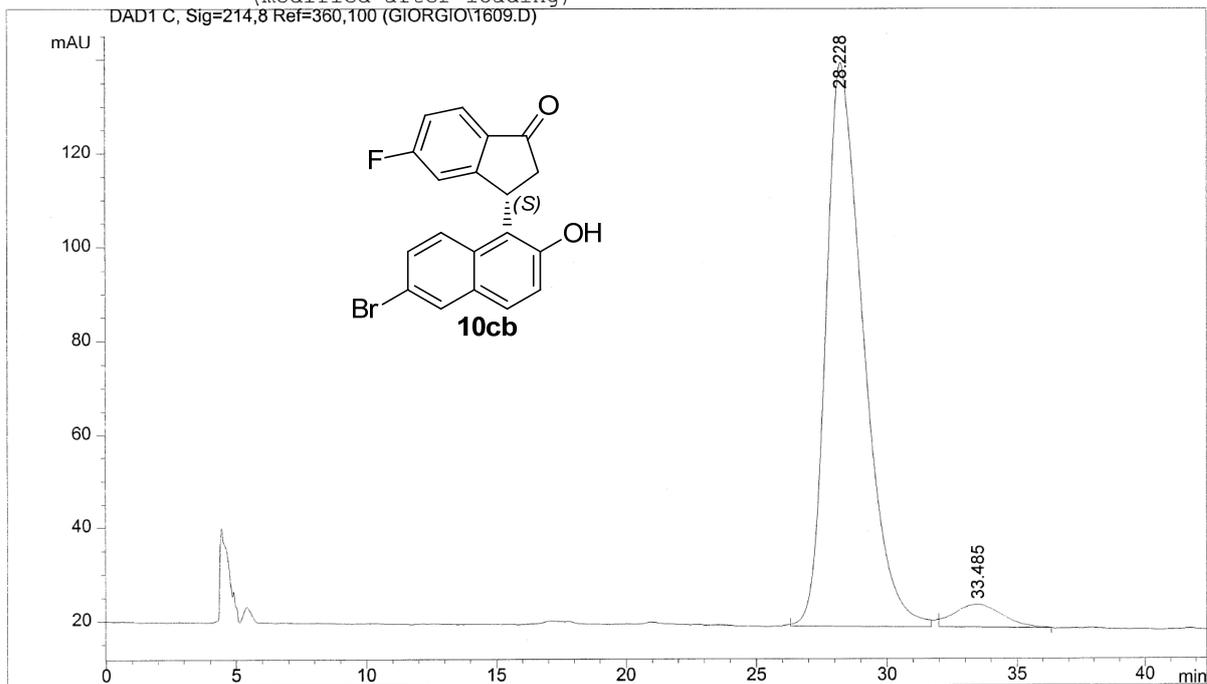
=====
*** End of Report ***
=====

Data File D:\HPCHEM\2\DATA\GIORGIO\1609.D

Sample Name: GB1609

5F-indenone, 6-bromonaftolo, quinidine
OD-H 95:5 0.70 ml/min

=====
Injection Date : 29/04/02 22.16.03
Sample Name : GB1609 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 29/04/02 20.48.01 by simone
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.28.50 by Nicolas
(modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.228	BB	1.5203	1.19491e4	120.56570	94.9233
2	33.485	BB	1.5463	639.06958	4.93662	5.0767

Totals : 1.25882e4 125.50232

Results obtained with enhanced integrator!

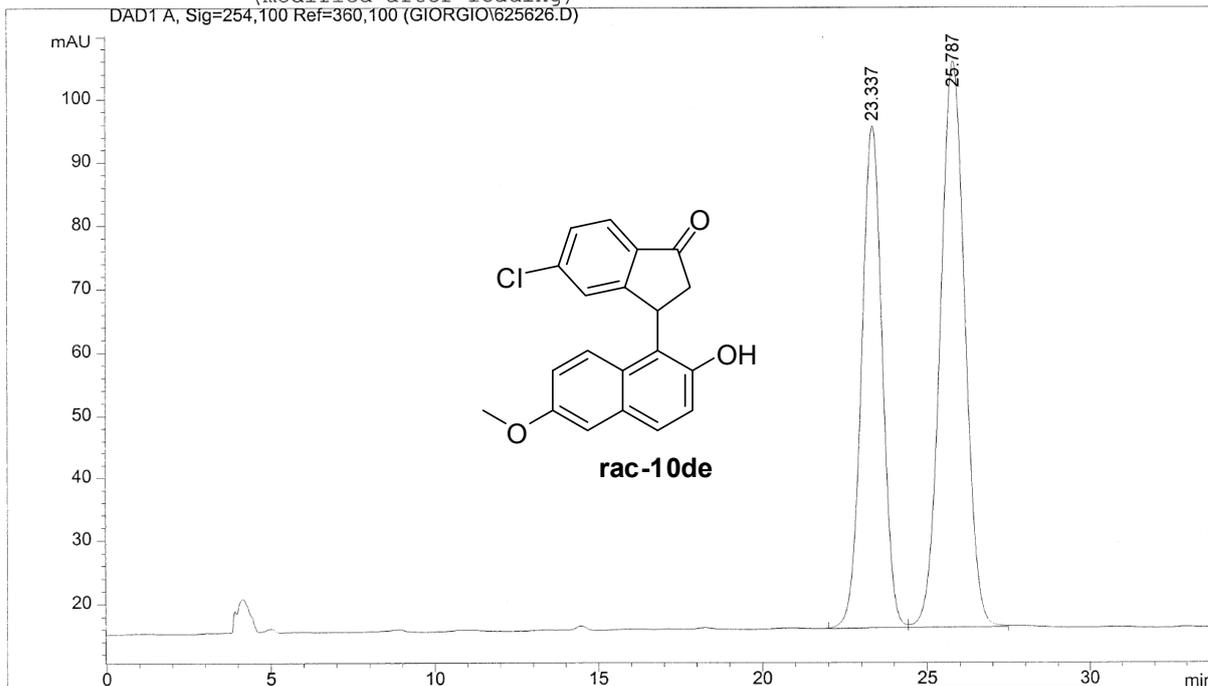
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\625626.D

Sample Name: GB1624+GB1623

6MeO-betanaftolo, 5Cl-indenone, racemo
AD-H 90:10 Hexane:IPA 0.750 ml/min

=====
Injection Date : 15/05/02 4.28.35
Sample Name : GB1624+GB1623 Location : Vial 1
Acq. Operator : Giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 15/05/02 3.22.48 by Giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.44.28 by Riccardo
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.337	BV	0.6775	3474.31787	79.55318	42.5144
2	25.787	VB	0.8193	4697.77051	89.65218	57.4856

Totals : 8172.08838 169.20535

Results obtained with enhanced integrator!

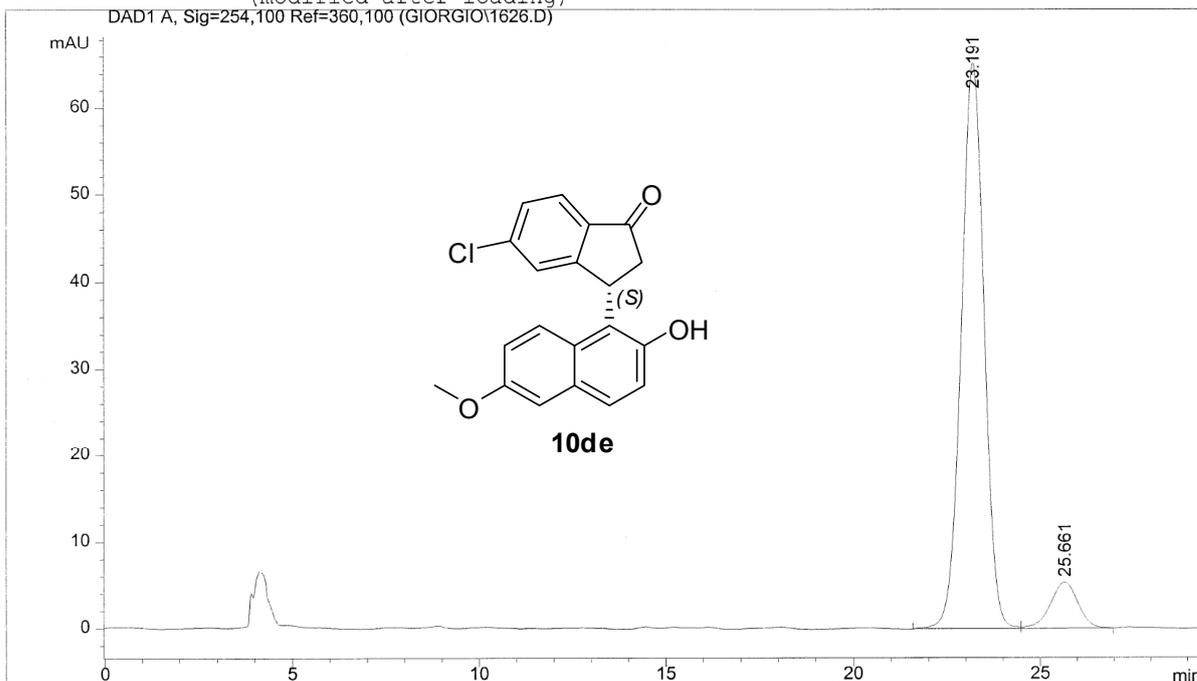
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*** End of Report ***
=====

Data File D:\HPCHEM\2\DATA\GIORGIO\1626.D

Sample Name: gb1626

6MeO-betanaftolo, 5Cl-indenone, quinidine nh2
AD-H 90:10 Hexane:IPA 0.750 ml/min

=====
Injection Date : 15/05/02 5.39.05
Sample Name : gb1626 Location : Vial 1
Acq. Operator : Giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 15/05/02 3.22.48 by Giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.44.28 by Riccardo
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.191	PV	0.6575	2784.74219	65.29891	90.8451
2	25.661	VV	0.8020	280.63330	5.36762	9.1549

Totals : 3065.37549 70.66653

Results obtained with enhanced integrator!

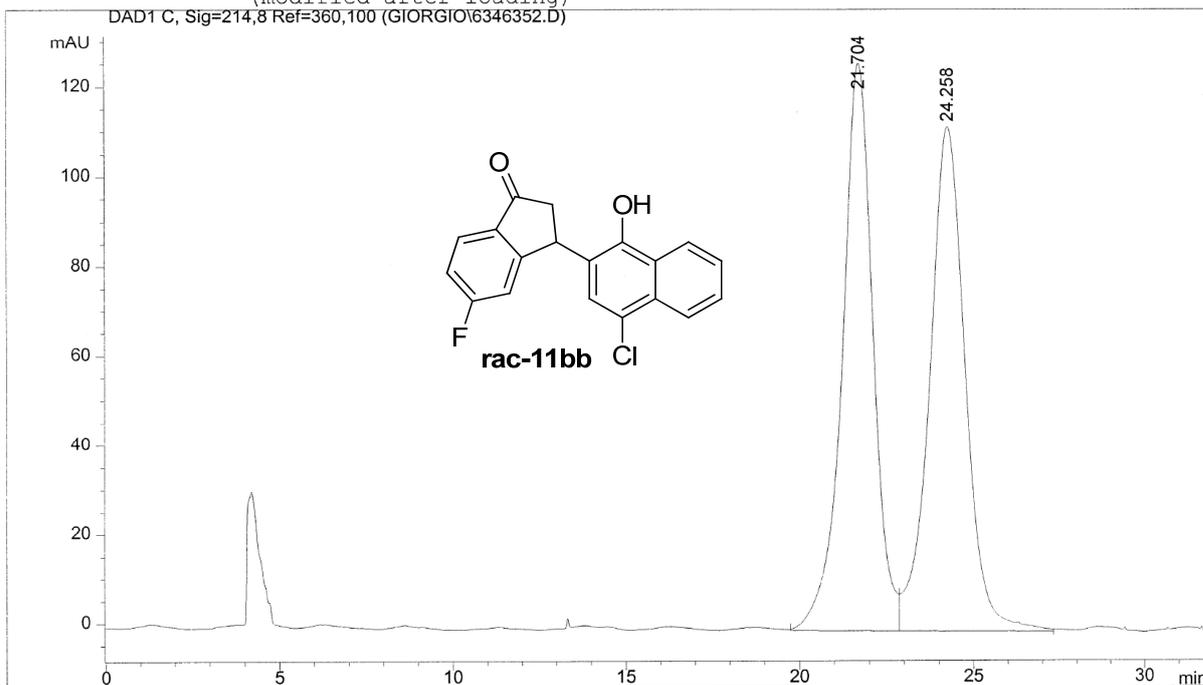
=====
*** End of Report ***
=====

Data File D:\HPCHEM\2\DATA\GIORGIO\6346352.D

Sample Name: GB1634+GB1635

5f-indenone, 4Cl-Alfanaftolo, rac
OD-H 95:5 Hexane:IPA 0.750 ml/min

```
=====
Injection Date   : 26/05/02 3.56.58
Sample Name     : GB1634+GB1635           Location : Vial 1
Acq. Operator   : giorgio
Acq. Method     : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed    : 26/05/02 3.42.18 by giorgio
                  (modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed    : 03/07/02 3.44.28 by Riccardo
                  (modified after loading)
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.704	BV	0.9242	7802.30908	127.09318	49.1728
2	24.258	VB	1.0811	8064.82031	112.93558	50.8272

Totals : 1.58671e4 240.02876

Results obtained with enhanced integrator!

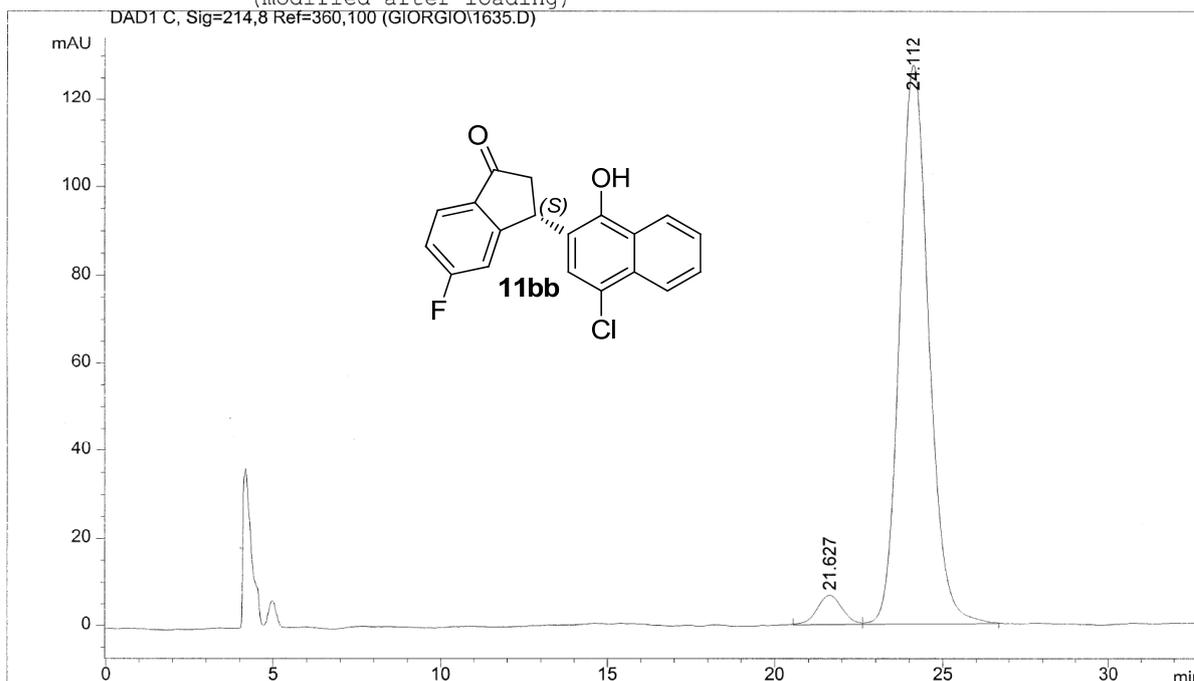
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*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\1635.D

Sample Name: GB1635

5f-indenone, 4Cl-Alfanaftolo, qd
OD-H 95:5 Hexane:IPA 0.750 ml/min

=====
Injection Date : 27/05/02 3.16.27
Sample Name : GB1635 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 27/05/02 3.06.17 by giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.44.28 by Riccardo
(modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.627	BV	0.8042	356.17905	6.78778	4.4170
2	24.112	VB	0.9281	7707.62939	127.71667	95.5830

Totals : 8063.80844 134.50445

Results obtained with enhanced integrator!

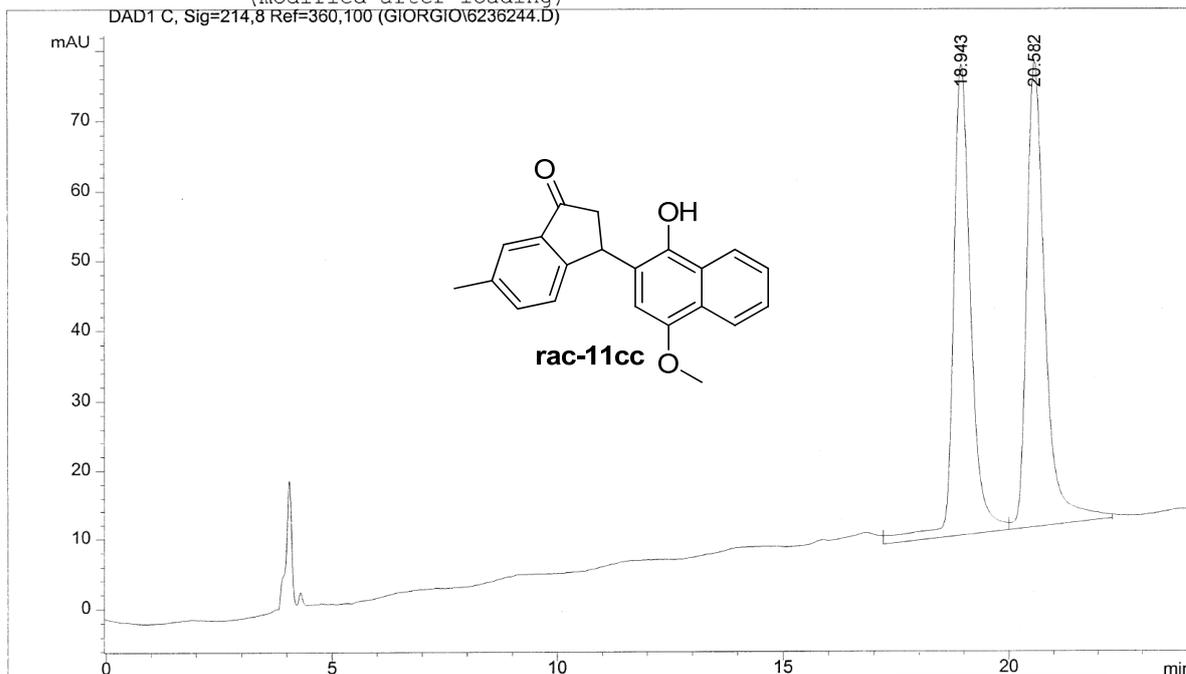
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*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\6236244.D

Sample Name: GB1624+GB1623

4MeO-alfanaftolo, 6-Meindenone, racemo
AD-H 90:10 Hexane:IPA 0.750 ml/min

=====
Injection Date : 15/05/02 2.27.01
Sample Name : GB1624+GB1623 Location : Vial 1
Acq. Operator : Giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 15/05/02 2.12.16 by Giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.16.51 by Nicolas
(modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.943	VV	0.4136	1840.32117	67.60520	48.8476
2	20.582	VB	0.4407	1927.15125	66.77039	51.1524

Totals : 3767.47241 134.37559

Results obtained with enhanced integrator!

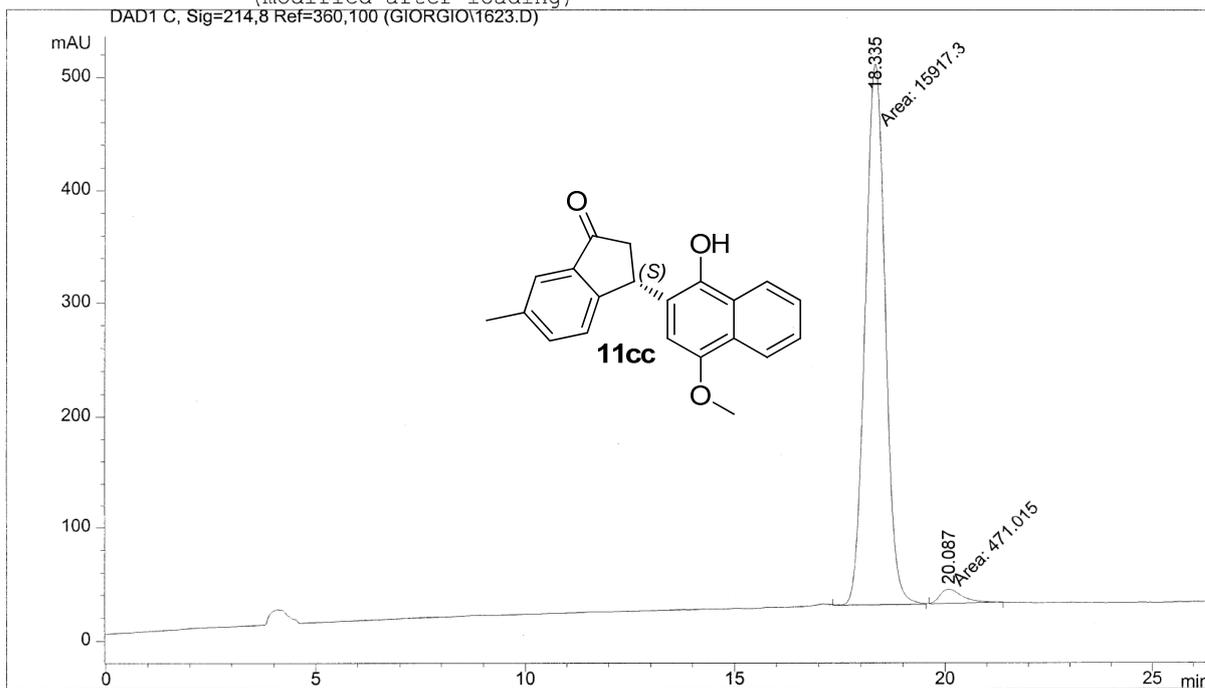
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\1623.D

Sample Name: GB1623

4MeO-alfanaftolo, 6-Meindenone, QD-NH2
AD-H 90:10 Hexane:IPA 0.750 ml/min

=====
Injection Date : 14/05/02 22.06.00
Sample Name : GB1623 Location : Vial 1
Acq. Operator : Giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 14/05/02 21.09.37 by Giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 4.16.51 by Nicolas
(modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.335	MM	0.5515	1.59173e4	481.03955	97.1259
2	20.087	MM	0.6272	471.01508	12.51645	2.8741

Totals : 1.63883e4 493.55600

Results obtained with enhanced integrator!

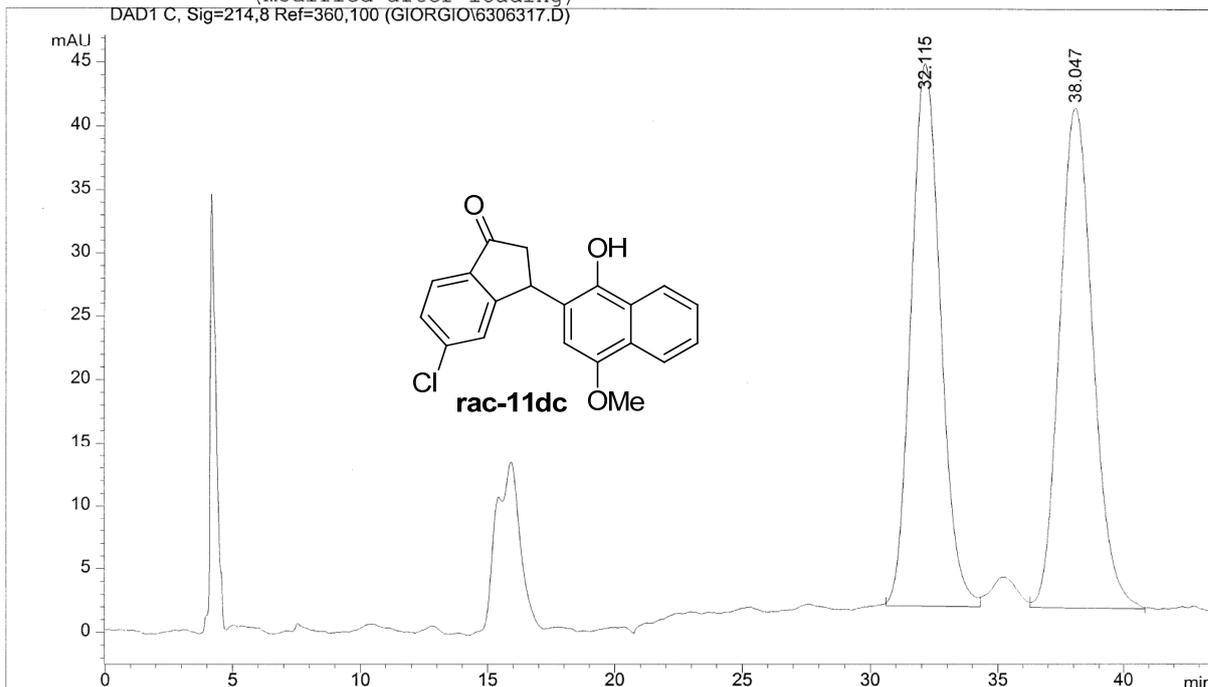
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\6306317.D

Sample Name: GB1631+GB1630

5Cl-indenone, 4Meo-Alfanaftolo, rac
OD-H 95:5 Hexane:IPA 0.750 ml/min

=====
Injection Date : 27/05/02 0.45.28
Sample Name : GB1631+GB1630 Location : Vial 1
Acq. Operator : giorgio
Acq. Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 26/05/02 21.15.03 by giorgio
(modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed : 03/07/02 3.44.28 by Riccardo
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.115	BB	1.2359	3444.74634	42.94129	48.4821
2	38.047	VB	1.3987	3660.44580	39.58815	51.5179

Totals : 7105.19214 82.52944

Results obtained with enhanced integrator!

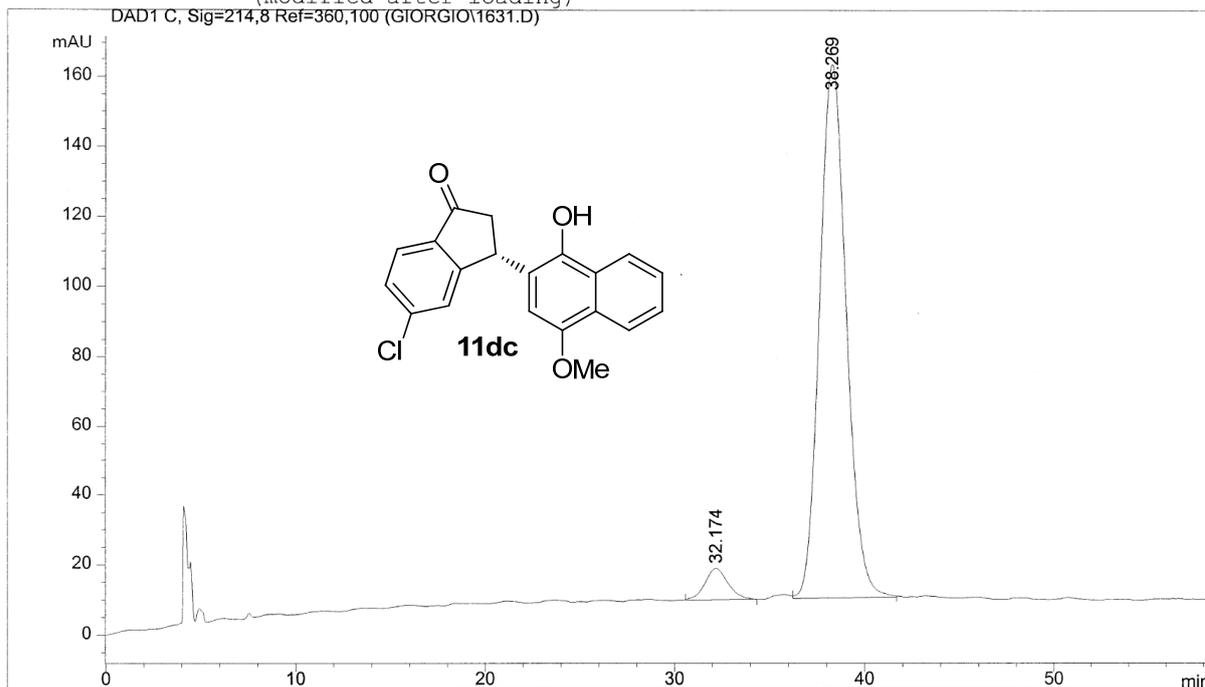
=====
*** End of Report ***

Data File D:\HPCHEM\2\DATA\GIORGIO\1631.D

Sample Name: GB1631

5Cl-indenone, 4Meo-Alfanaftolo, qd
OD-H 95:5 Hexane:IPA 0.750 ml/min

```
=====
Injection Date   : 26/05/02 22.03.37
Sample Name     : GB1631                      Location : Vial 1
Acq. Operator  : giorgio
Acq. Method    : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed   : 26/05/02 21.15.03 by giorgio
                (modified after loading)
Analysis Method : D:\HPCHEM\2\METHODS\PAOLO.M
Last changed   : 03/07/02 3.44.28 by Riccardo
                (modified after loading)
=====
```



=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
```

Signal 1: DAD1 C, Sig=214,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.174	BB	1.2154	754.26141	8.91163	4.8269
2	38.269	VB	1.4934	1.48720e4	153.07890	95.1731

Totals : 1.56263e4 161.99053

Results obtained with enhanced integrator!

=====
*** End of Report ***