Electronic Supplementary Material (ESI) for Chemical Communications
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Amine/*N*-Heterocyclic Carbene Cascade Catalysis for Asymmetric Synthesis of Fused Indane Derivatives with Multiple Chiral Centres

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Supplementary Information

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1. General methods

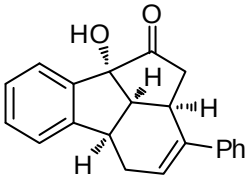
NMR data were obtained for ^{1}H at 400 MHz, and for ^{13}C at 100 MHz. Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard in $CDCl_3$ solution. ESI-HRMS was recorded on a Bruker Apex-2. In each case, enantiomeric excess was determined by HPLC analysis on Chiralpak AS, IC, AD and Chiralcel OD columns in comparison with authentic racemic samples. UV detection was monitored at 220 nm. Optical rotation data were examined at 589 nm in $CHCl_3$ solution at 20 ºC. Column chromatography was performed on silica gel (200-300 mesh) eluting with ethyl acetate and petroleum ether. TLC was performed on glass-backed silica plates. UV light and I_2 were used to visualize products. All chemicals were used without purification as commercially available unless otherwise noted. All types of 3-bromo-1-indanone were prepared from substituted benzaldehyde and Meldrum's acid according to literature procedures.[1] The secondary amine catalysts were also synthesized according to the literature procedures.[2]

(1) N. Griebenow, T. Flessner, A. Buchmueller, M. Raabe, H. Bischoff and P. Kolkhof, *Bioorg. Med. Chem. Lett.*, 2011, **21**, 2554.

(2) (*a*) M. Marigo, T. C. Wabnitz, D. Fielenbach and K. A. Jørgensen, *Angew. Chem., Int. Ed.*, 2005, **44**, 794; (*b*) Y. Hayashi, H. Gotoh, T. Hayashi and M. Shoji, *Angew. Chem., Int. Ed.*, 2005, **44**, 4212; (*c*) Y.-K. Liu, C. Ma, K. Jiang, T.-Y. Liu and Y.-C. Chen, *Org. Lett.*, 2009, **11**, 2848.

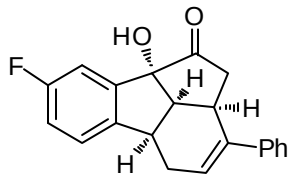
2. General procedure for cascade Diels–Alder and benzoin reaction

The reactions were performed with 3-bromo-1-indanone **3** (0.2 mmol), 2,4-dienal **4** (0.1 mmol), amine catalyst **1b** (0.02 mmol), carbene precursor **2** (0.02 mmol) and PhCOONa (0.3 mmol) in $CHCl_3$ (1 mL) at 55 °C for a specified reaction time. After the reaction completed, the mixture was concentrated and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate) to afford fused indane derivative **5.**

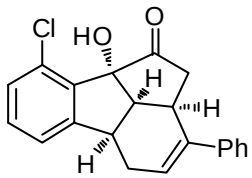


(2a*R*,2a^{1}*S*,5a*R*,9b*R*)-9b-hydroxy-3-phenyl-2,2a,2a^{1},5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5a): 4 h; 57% yield; $[\alpha]_D^{20}$ = -160.2 (*c* = 0.60 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*hexane/*i*PrOH = 70/30, 1.0 mL/min, λ = 254 nm, t (major) = 12.02 min, t (minor) = 9.49 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39-7.35 (m, 1H), 7.30-7.19 (m, 8H), 6.08 (t, *J* = 4.8 Hz, 1H), 3.87 (dd, *J* = 13.6, 7.2 Hz, 1H), 3.65 (dd, *J* = 20.4, 10.0 Hz, 1H), 3.28 (t, *J* = 9.2

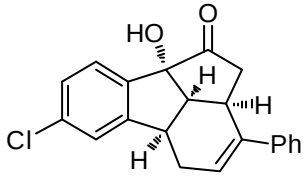
Hz, 1H), 2.89-2.81 (m, 1H), 2.60-2.54 (m, 2H), 2.08 (dd, *J* = 17.6, 11.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 215.0, 148.2, 142.2, 141.1, 140.3, 130.1, 128.3, 127.8, 127.1, 125.9, 123.7, 123.5, 123.3, 89.0, 48.8, 41.5, 39.4, 31.2, 26.9 ppm; ESI-HRMS: calcd. for $C_{21}H_{18}O_2$+Na: 325.1204, found 325.1202.



(2a*R*,2a^1*S*,5a*R*,9b*R*)-8-fluoro-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5b): 3 h; 52% yield; $[\alpha]_D^{20}$ = -183.0 (*c* = 0.20 in $CHCl_3$); 90% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*hexane/*i*PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 20.53 min, t (minor) = 12.72 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31-7.20 (m, 6H), 7.05 (td, *J* = 8.8, 2.4 Hz, 1H), 6.91 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.08 (t, *J* = 4.8 Hz, 1H), 3.86-3.83 (m, 1H), 3.66 (dd, *J* = 20.0, 10.0 Hz, 1H), 3.30 (t, *J* = 9.2 Hz, 1H), 2.87-2.82 (m, 1H), 2.59 (dd, *J* = 18.0, 8.8 Hz, 1H), 2.54 (dt, *J* = 17.2, 5.6 Hz, 1H), 2.11 (dd, *J* = 18.0, 12.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.7, 163.8, 161.3, 143.9, 143.7, 141.0, 140.4, 135.6, 130.1, 128.4, 127.2, 125.9, 125.0, 124.9, 123.3, 117.5, 117.2, 110.3, 110.1, 88.1, 49.3, 41.6, 38.9, 31.3, 27.1 ppm; ESI-HRMS: calcd. for $C_{21}H_{17}FO_2$+H: 321.1291, found 321.1293.

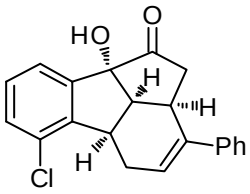


(2a*R*,2a^1*S*,5a*R*,9b*R*)-9-chloro-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5c): 4 h; 36% yield; $[\alpha]_D^{20}$ = -57.5 (*c* = 0.12 in $CHCl_3$); 91% ee, determined by HPLC analysis [Daicel Chiralpak IC, *n*hexane/*i*PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 13.08 min, t (minor) = 20.92 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33-7.20 (m, 8H), 6.06 (t, *J* = 4.4 Hz, 1H), 3.89 (dd, *J* = 13.2, 6.8 Hz, 1H), 3.57 (dd, *J* = 20.4, 10.0 Hz, 1H), 3.20 (dd, *J* = 10.4, 8.8 Hz, 1H), 2.86-2.81 (m, 1H), 2.64-2.56 (m, 2H), 2.13 (dd, *J* = 16.8, 12.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.8, 150.9, 140.9, 140.3, 139.6, 139.0, 130.9, 128.7, 128.4, 127.2, 126.0, 123.1, 122.2, 89.1, 48.3, 41.3, 39.5, 30.6, 26.5 ppm; ESI-HRMS: calcd. for $C_{21}H_{17}ClO_2$+Na: 359.0815, found 359.0816.

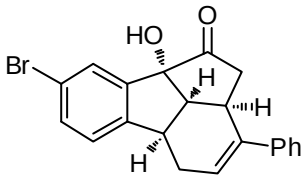


(2a*R*,2a^1*S*,5a*R*,9b*R*)-7-chloro-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5d): 3 h; 49% yield; $[\alpha]_D^{20}$ =

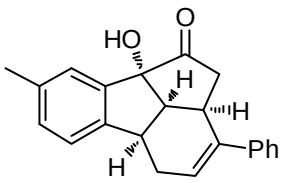
-282.6 (*c* = 0.14 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel Chiralpak IC, *n*hexane/*i*PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 8.08 min, t (minor) = 11.17 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30-7.20 (m, 7H), 7.14 (d, *J* = 8.4 Hz, 1H), 6.07 (t, *J* = 4.0 Hz, 1H), 3.87 (dd, *J* = 13.2, 7.2 Hz, 1H), 3.65 (dd, *J* = 20.4, 10.0 Hz, 1H), 3.29 (t, *J* = 9.6 Hz, 1H), 2.86-2.82 (m, 1H), 2.62-2.54 (m, 2H), 2.05 (dd, *J* = 17.6, 12.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.6, 150.2, 140.7, 136.2, 133.7, 130.1, 128.4, 128.2, 127.2, 125.9, 124.5, 124.1, 123.1, 88.4, 48.9, 41.4, 39.3, 31.0, 26.6 ppm; ESI-HRMS: calcd. for $C_{21}H_{17}ClO_2$+Na: 359.0815, found 359.0816.



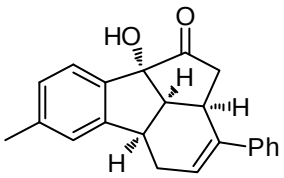
(2a*R*,2a^1*S*,5a*R*,9b*R*)-6-chloro-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5e): 2 h; 61% yield; $[\alpha]_D^{20}$ = -263.0 (*c* = 0.60 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel Chiralpak IC, *n*hexane/*i*PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 6.87 min, t (minor) = 7.60 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.41-7.22 (m, 8H), 6.38 (dd, *J* = 7.2, 2.8 Hz, 1H), 3.83-3.71 (m, 2H), 3.39-3.34 (m, 2H), 2.82 (dd, *J* = 18.0, 8.0 Hz, 1H), 2.46 (dd, *J* = 18.4, 12.8 Hz, 1H), 2.08-2.01 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 216.0, 145.8, 143.7, 141.3, 140.6, 133.7, 130.2, 129.5, 128.6, 127.3, 125.1, 124.3, 122.3, 89.1, 51.5, 42.6, 41.3, 34.1, 27.3 ppm; ESI-HRMS: calcd. for $C_{21}H_{17}ClO_2$+Na: 359.0815, found 359.0814.



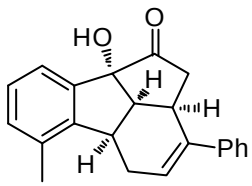
(2a*R*,2a^1*S*,5a*R*,9b*R*)-8-bromo-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5f): 3 h; 55% yield; $[\alpha]_D^{20}$ = -95.3 (*c* = 0.32 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*hexane/*i*PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t (major) = 23.31 min, t (minor) = 15.04 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48 (dd, *J* = 8.0, 5.6 Hz, 1H), 7.34 (d, *J* = 1.6 Hz, 1H), 7.30-7.16 (m, 6H), 6.07 (t, *J* = 4.4 Hz, 1H), 3.82 (dd, *J* = 13.2, 7.2 Hz, 1H), 3.65 (dd, *J* = 20.4, 10.4 Hz, 1H), 3.28 (t, *J* = 9.6 Hz, 1H), 2.88-2.82 (m, 1H), 2.64-2.52 (m, 1H), 2.09 (dd, *J* = 18.0, 12.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.4, 147.1, 144.3, 141.0, 140.4, 133.1, 130.1, 128.4, 127.2, 126.6, 125.9, 125.3, 123.2, 88.6, 48.9, 41.5, 39.1, 31.1, 26.7 ppm; ESI-HRMS: calcd. for $C_{21}H_{17}BrO_2$+H: 381.0490, found 381.0490.



(2a*R*,2a^1*S*,5a*R*,9b*R*)-8-methyl-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5g): 3 h; 65% yield; $[\alpha]_D^{20}$ = -140.0 (*c* = 0.20 in $CHCl_3$); 90% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*hexane/*i*PrOH = 70/30, 1.0 mL/min, λ = 254 nm, t (major) = 13.11 min, t (minor) = 9.31 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.30-7.17 (m, 7H), 7.02 (s, 1H), 6.08 (t, *J* = 4.8 Hz, 1H), 3.84 (dd, *J* = 13.6, 7.2 Hz, 1H), 3.64 (dd, *J* = 20.0, 10.0 Hz, 1H), 3.26 (t, *J* = 9.2 Hz, 1H), 2.86-2.80 (m, 1H), 2.59-2.52 (m, 2H), 2.31 (s, 3H), 2.09 (dd, *J* = 17.6, 12.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 215.2, 145.3, 142.2, 141.2, 140.2, 137.7, 131.0, 128.3, 127.1, 125.9, 123.6, 123.5, 123.4, 89.0, 49.0, 41.5, 39.0, 31.2, 27.0, 21.2 ppm; ESI-HRMS: calcd. for $C_{22}H_{20}O_2$+Na: 339.1361, found 339.1352.

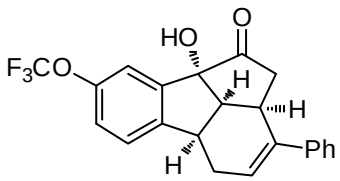


(2a*R*,2a^1*S*,5a*R*,9b*R*)-7-methyl-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5h): 4 h; 55% yield; $[\alpha]_D^{20}$ = -186.9 (*c* = 0.16 in $CHCl_3$); 87% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*hexane/*i*PrOH = 70/30, 1.0 mL/min, λ = 254 nm, t (major) = 14.99 min, t (minor) = 9.58 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32-7.26 (m, 3H), 7.23-7.21 (m, 2H), 7.11-7.04 (m, 3H), 6.08 (t, *J* = 4.8 Hz, 1H), 3.84 (dd, *J* = 14.0, 7.2 Hz, 1H), 3.65 (dd, *J* = 20.4, 10.0 Hz, 1H), 3.27 (t, *J* = 9.2 Hz, 1H), 2.85-2.80 (m, 1H), 2.60-2.52 (m, 2H), 2.38 (s, 3H), 2.09 (dd, *J* = 17.6, 12.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.8, 148.4, 141.0, 140.0, 139.2, 128.6, 128.1, 126.9, 125.7, 124.1, 123.3, 122.8, 88.7, 48.9, 41.3, 39.1, 31.0, 26.6, 21.3 ppm; ESI-HRMS: calcd. for $C_{22}H_{20}O_2$+Na: 339.1361, found 339.1352.

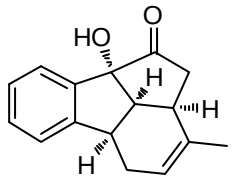


(2a*R*,2a^1*S*,5a*R*,9b*R*)-6-methyl-9b-hydroxy-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5i): 4 h; 57% yield; $[\alpha]_D^{20}$ = -208.1 (*c* = 0.16 in $CHCl_3$); 88% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*hexane/*i*PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t (major) = 21.70 min, t (minor) = 19.54 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.42-7.15 (m, 8H), 6.37 (dd, *J* = 7.6, 3.2 Hz, 1H), 3.79-3.70 (m, 2H), 3.33 (t, *J* = 9.6 Hz, 1H), 3.12 (dt, *J* = 16.4, 7.2 Hz, 1H), 2.77 (ddd, *J* = 18.0, 8.0, 1.2 Hz, 1H), 2.47 (dd, *J* = 18.0, 13.2 Hz, 1H), 2.40 (s, 3H), 2.03 (ddd, *J* = 16.4, 10.4, 3.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 216.5, 147.0, 141.6, 141.4, 140.7, 134.4, 131.2, 128.6, 128.2, 127.3, 125.1, 124.2, 121.2, 89.0, 51.7, 42.6, 41.0, 34.3, 28.2, 19.1 ppm; ESI-HRMS:

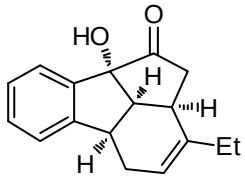
calcd. for $C_{22}H_{20}O_2$+Na: 339.1361, found 339.1353.



(2a*R*,2a^1*S*,5a*R*,9b*R*)-9b-hydroxy-3-phenyl-8-(trifluoromethoxy)-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5j): 4 h; 57% yield; $[\alpha]_D^{20}$ = -161.2 (*c* = 0.30 in $CHCl_3$); 85% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*hexane/*i*PrOH = 70/30, 1.0 mL/min, λ = 254 nm, t (major) = 9.39 min, t (minor) = 7.50 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48 (t, *J* = 8.0 Hz, 1H), 7.36 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.29-7.21 (m, 6H), 6.10 (t, *J* = 4.4 Hz, 1H), 3.87 (dd, *J* = 14.0, 6.4 Hz, 1H), 3.67 (dd, *J* = 20.0, 10.0 Hz, 1H), 3.35-3.31 (m, 1H), 2.90-2.84 (m, 1H), 2.63 (dd, *J* = 18.0, 8.4 Hz, 1H), 2.56 (dt, *J* = 17.2, 5.6 Hz, 1H), 2.17-2.05 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.7, 151.9, 147.0, 143.5, 140.9, 140.5, 133.7, 130.2, 128.5, 128.4, 127.3, 125.8, 124.5, 123.2, 117.8, 88.6, 49.4, 41.6, 39.2, 31.5, 27.0 ppm; ESI-HRMS: calcd. for $C_{22}H_{17}F_3O_3$+K: 425.0767, found 425.0769.

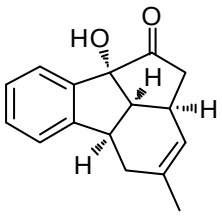


(2a*R*,2a^1*S*,5a*R*,9b*R*)-9b-hydroxy-3-methyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5k): 5 h; 66% yield; $[\alpha]_D^{20}$ = -226.3 (*c* = 0.16 in $CHCl_3$); 88% ee, determined by HPLC analysis [Daicel Chiralpak IC, *n*hexane/*i*PrOH = 95/5, 1.0 mL/min, λ = 220 nm, t (major) = 27.31 min, t (minor) = 36.38 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.35 (td, *J* = 8.0, 1.2 Hz, 1H), 7.26-7.16 (m, 3H), 5.46 (d, *J* = 1.2 Hz, 1H), 3.86 (dd, *J* = 10.4, 6.8 Hz, 1H), 3.07 (dd, *J* = 11.2, 8.0 Hz, 1H), 2.89 (dd, *J* = 20.4, 10.4 Hz, 1H), 2.59-2.56 (m, 2H), 2.52 (dd, *J* = 16.8, 8.8 Hz, 1H), 2.10 (dd, *J* = 16.8, 11.2 Hz, 1H), 1.58 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.9, 148.2, 142.7, 134.7, 130.0, 127.7, 123.5, 122.9, 119.3, 89.0, 47.3, 40.1, 39.0, 32.4, 25.1, 22.2 ppm; ESI-HRMS: calcd. for $C_{16}H_{16}O_2$+K: 279.0787, found 279.0787.

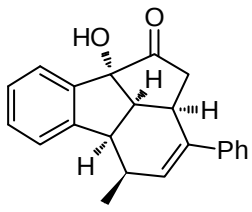


(2a*R*,2a^1*S*,5a*R*,9b*R*)-9b-hydroxy-3-ethyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5l): 4 h; 72% yield; $[\alpha]_D^{20}$ = -178.3 (*c* = 0.24 in $CHCl_3$); 86% ee, determined by HPLC analysis [Daicel Chiralpak IC, *n*hexane/*i*PrOH = 95/5, 1.0 mL/min, λ = 220 nm, t (major) = 23.63 min, t (minor) = 32.62 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34 (td, *J* = 7.2, 1.6 Hz, 1H), 7.26-7.20 (m, 3H), 5.51 (s, 1H), 3.81 (dd, *J* = 12.4, 7.2 Hz, 1H), 3.09 (dd, *J* = 10.4, 8.4 Hz, 1H), 3.00 (dd, *J* = 20.0,

10.0 Hz, 1H), 2.68-2.62 (m, 1H), 2.53 (dd, *J* = 17.2, 8.8 Hz, 1H), 2.46 (dt, *J* = 17.2, 4.4 Hz, 1H), 2.14 (dd, *J* = 17.2, 11.6 Hz, 1H), 1..92 (qd, *J* = 15.6, 8.0 Hz, 1H),0.97 (t, *J* = 15.6 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 215.4, 148.5, 142.4, 140.8, 130.0, 127.7, 123.7, 123.1, 117.7, 89.0, 48.0, 40.4, 39.6, 31.8, 28.7, 25.8, 12.1 ppm; ESI-HRMS: calcd. for $C_{17}H_{18}O_2$+K: 293.0944, found 293.0946.

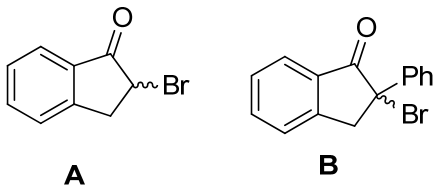


(2a*R*,2a^1*S*,5a*R*,9b*R*)-9b-hydroxy-4-methyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5m): 4 h; 42% yield; $[\alpha]_D^{20}$ = -176.7 (*c* = 0.06 in $CHCl_3$); 86% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*hexane/*i*PrOH = 95/5, 1.0 mL/min, λ = 220 nm, t (major) = 27.16 min, t (minor) = 23.59 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34 (td, *J* = 8.0, 1.6 Hz, 1H), 7.21 (dd, *J* = 14.4, 3.6 Hz, 1H, 3H), 5.20 (s, 1H), 3.94-3.93 (m, 1H), 3.06-3.00 (m, 2H), 2.62 (dd, *J* = 18.0, 9.2 Hz, 1H), 2.50 (d, *J* = 3.2 Hz, 2H), 2.08 (dd, *J* = 18.0, 8.0 Hz, 1H), 1.66 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 215.8, 148.0, 142.6, 133.6, 130.1, 127.6, 123.7, 123.1, 123.0, 88.7, 47.1, 42.3, 40.1, 29.6, 28.3, 24.5 ppm; ESI-HRMS: calcd. for $C_{16}H_{16}O_2$+K: 279.0787, found 279.0789.



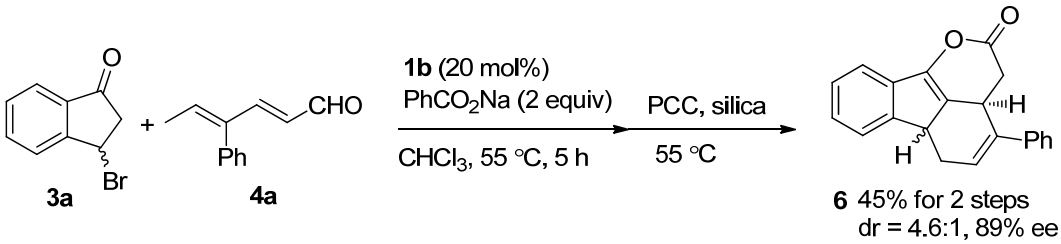
(2a*R*,2a^1*S*,5*R*,5a*R*,9b*R*)-9b-hydroxy-5-methyl-3-phenyl-2,2a,2a^1,5,5a,9b-hexahydro-1*H*-cyclopenta[*jk*]fluoren-1-one (5n): 4 h; 29% yield; $[\alpha]_D^{20}$ = -75.5 (*c* = 0.20 in $CHCl_3$); 91% ee, determined by HPLC analysis [Daicel Chiralpak IC, *n*hexane/*i*PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 28.31 min, t (minor) = 45.71 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53 (d, *J* = 7.6 Hz, 1H), 7.30 (td, *J* = 7.6, 1.2 Hz, 1H), 7.21-7.10 (m, 5H), 7.00 (d, *J* = 6.8 Hz, 2H), 5.72 (t, *J* = 2.4 Hz, 1H), 4.01 (t, *J* = 5.6 Hz, 1H), 3.60-3.52 (m, 1H), 3.27 (dd, *J* = 11.6, 6.8 Hz, 1H), 2.95-2.93 (m, 1H), 2.58 (dd, *J* = 18.0, 10.0 Hz, 1H), 1.97 (dd, *J* = 18.0, 10.0 Hz, 1H), 1.63 (d, *J* = 7.6 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 214.9, 146.3, 143.9, 141.0, 140.4, 130.5, 129.9, 128.1, 127.6, 126.9, 126.7, 125.8, 123.1, 88.0, 50.0, 46.0, 41.3, 31.5, 29.7, 20.0 ppm; ESI-HRMS: calcd. for $C_{22}H_{20}O_2$+K: 355.1100, found 355.1102.

3. More screenings with two 2-bromoindanones



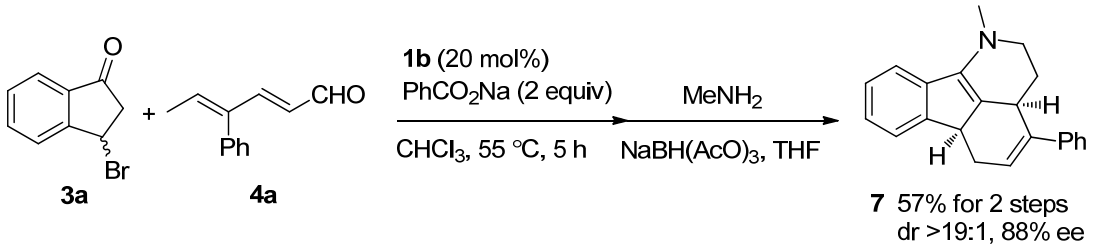
Except 3-bromoindanones **3**, two 2-bromoindanones **A** and **B** were tested under the optimized conditions, but there was no reaction between both substrates and 2,4-dienal **4a**. In this process, we found these two substrates couldn't even be eliminated with PhCOONa. So we tested other bases, including AcONa, triethylamine and pyridine, but no desired product was detected.

4. Sequential transformations with the Diels–Alder intermediate



The reaction was performed with 3-bromo-1-indanone **3a** (0.2 mmol), 2,4-dienal **4a** (0.1 mmol), amine catalyst **1b** (0.02 mmol) and PhCOONa (0.2 mmol) in $CHCl_3$ (1 mL) at 55 °C for 4 h. Then, to the mixture was added PCC (0.3 mmol) and silica at 55 °C. After the reaction completed, the reaction was concentrated and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate) to afford lactone **6.**

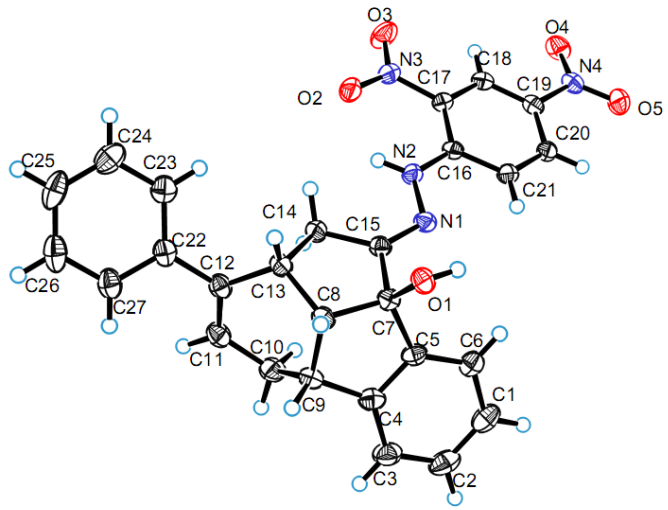
(3a*S*,6a*S*)-4-phenyl-3,3a,6,6a-tetrahydro-2*H*-indeno[1,2,3-*ij*]isochromen-2-one (6): $[\alpha]_D^{20}$ = -28.9 (*c* = 0.18 in $CHCl_3$); 89% ee, determined by HPLC analysis [Daicel Chiralcel OD, *n*hexane/*i*PrOH = 70/30, 1.0 mL/min, λ = 254 nm, t (major) = 10.63 min, t (minor) = 13.32 min]; dr = 4.6:1, determined by ^{1}H NMR analysis; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69 (t, *J* = 8.0 Hz, 2H), 7.56 (d, *J* = 7.6, 1H), 7.50-7.26 (m, 4H), 7.09 (dd, *J* = 8.0, 2.0 Hz, 2H), 5.68-5.64 (m, 1H), 3.86 (t, *J* = 8.4 Hz, 1H), 3.73-3.66 (m, 1H), 3.61-3.59 (m, 1H), 3.01-2.95 (m, 1H), 2.83 (dd, *J* = 16.0, 7.2 Hz, 1H), 2.61 (dd, *J* = 16.0, 7.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.9, 156.4, 140.9, 137.7, 136.7, 135.0, 134.7, 133.7, 128.6, 128.5, 127.8, 126.8, 125.8, 124.2, 124.0, 44.8, 41.0, 32.7, 31.6 ppm; ESI-HRMS: calcd. for $C_{21}H_{16}O_2$+K: 339.0787, found 339.0789.

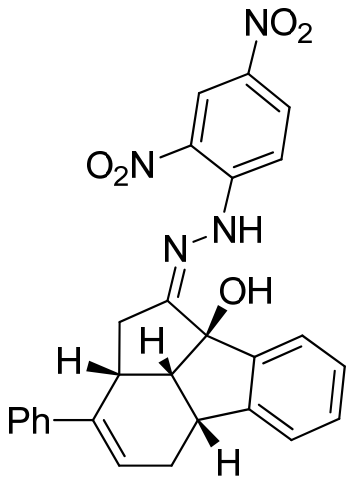


The reaction was performed with 2,4-dienal **4a** (0.1 mmol), **3a** (0.2 mmol), catalyst **1b** (0.02 mmol), and PhCOONa (0.2 mmol) in $CHCl_3$ (1 mL) at 55 °C for 4 h. Then, the mixture was concentrated and re-dissolved in 1 mL THF. To the solution of DA intermediate was added $MeNH_2$ (1 mmol) and $NaBH(AcO)_3$ (0.2 mmol) at 0 °C. After the reaction completed, the reaction was concentrated and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate) to afford enamine **7** in 57% yield**.**

(3a*S*,6a*S*)-1-methyl-4-phenyl-1,2,3,3a,6,6a-hexahydroindeno[1,2,3-*ij*]isoquinoline (7): $[\alpha]_D^{20}$ = -50.0 (*c* = 0.06 in $CHCl_3$); 88% ee, determined by HPLC analysis [Daicel Chiralpak AD, *n*hexane/*i*PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t (major) = 6.61 min, t (minor) = 6.04 min]; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.49 (d, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 7.6, 1H), 7.38-7.27 (m, 6H), 7.18 (t, *J* = 7.2 Hz, 1H), 5.96 (d, *J* = 5.6 Hz, 1H), 3.83-3.82 (m, 1H), 3.39 (t, *J* = 8.8 Hz, 1H), 3.22-3.14 (m, 2H), 3.05-2.97 (m, 1H), 2.95 (s, 3H), 1.95 (dt, *J* = 13.2, 2.4 Hz, 1H), 1.75-1.68 (m, 1H), 1.33-1.24 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.2, 148.6, 143.4, 141.3, 140.8, 140.7, 128.3, 126.8, 126.3, 126.2, 124.1, 123.8, 123.1, 119.5, 53.7, 41.4, 40.8, 34.7, 31.1, 27.2 ppm; ESI-HRMS: calcd. for $C_{22}H_{21}N$+H: 300.1752, found 300.1753.

5. Crystal data and structure refinement for 2,4-dinitrobenzenehydrazone of 5a





Identification code	**5a**

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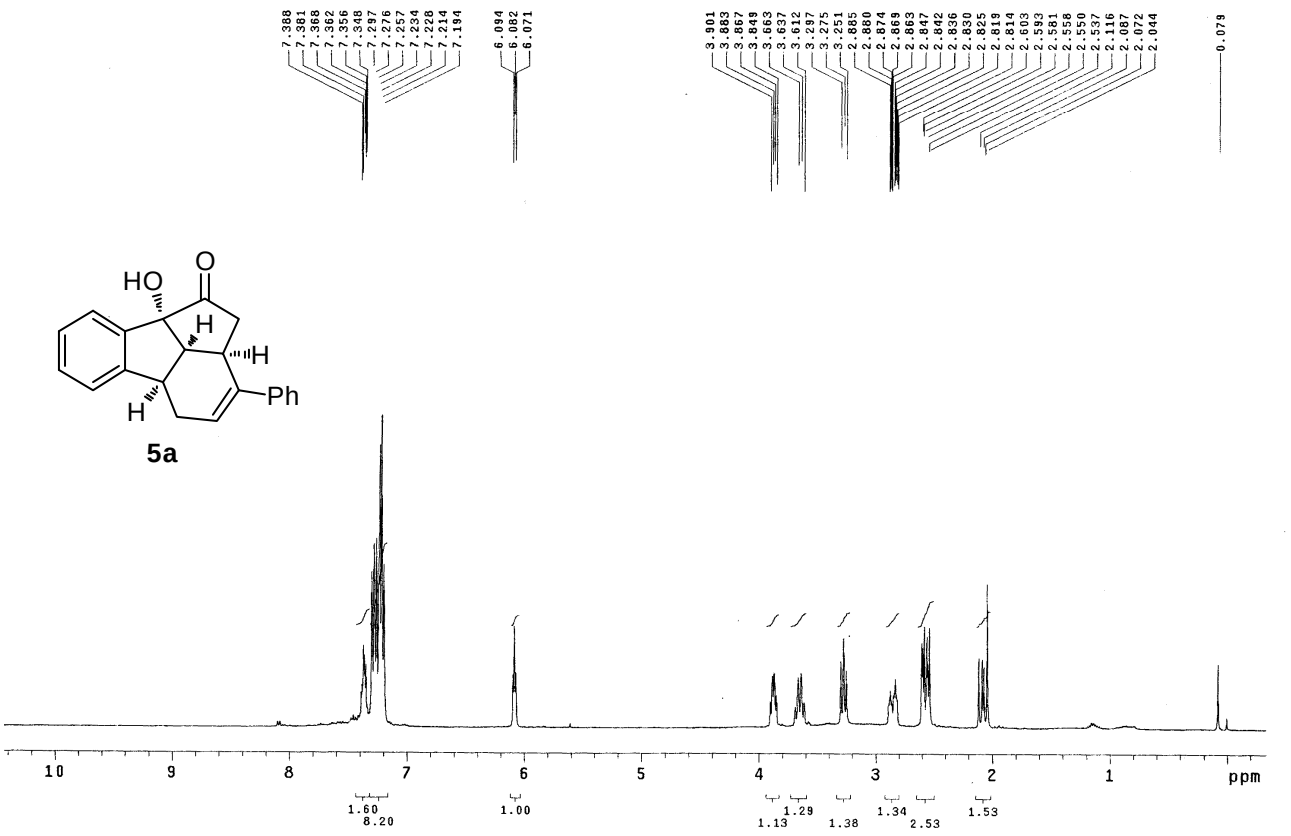
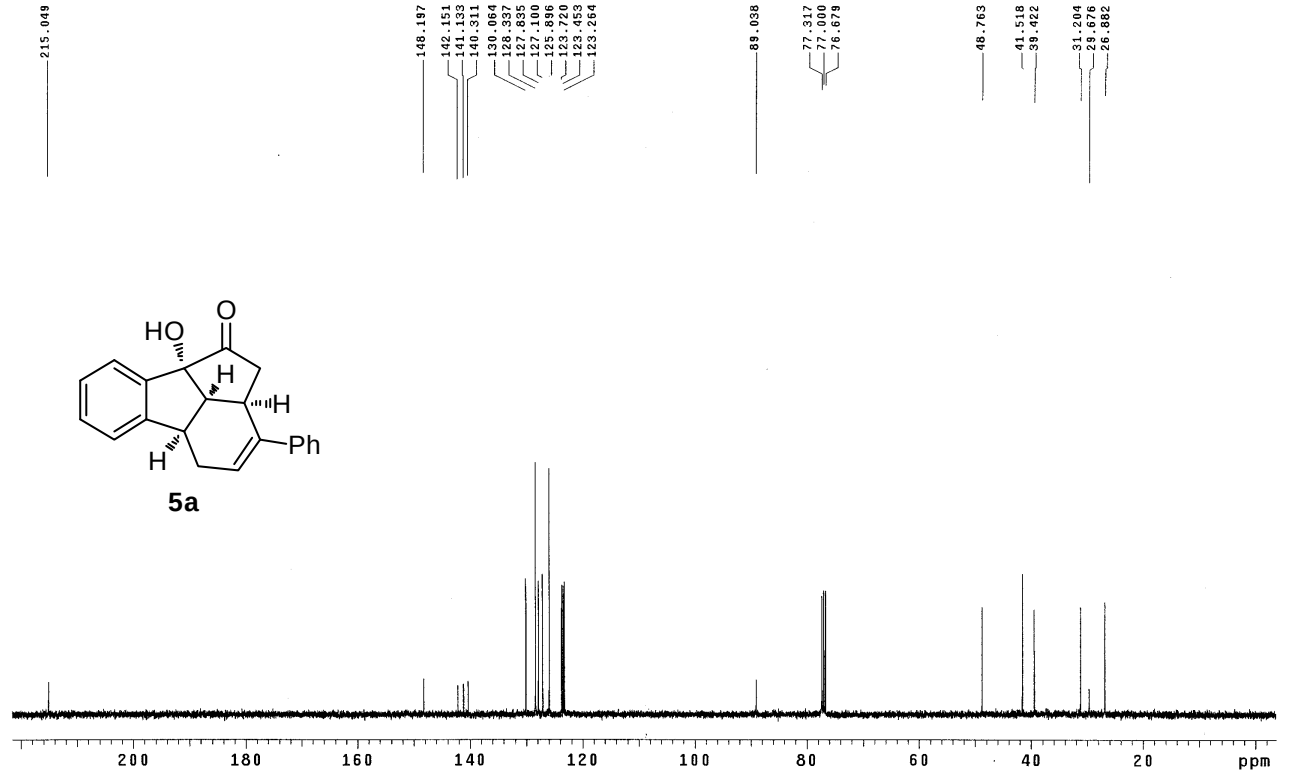
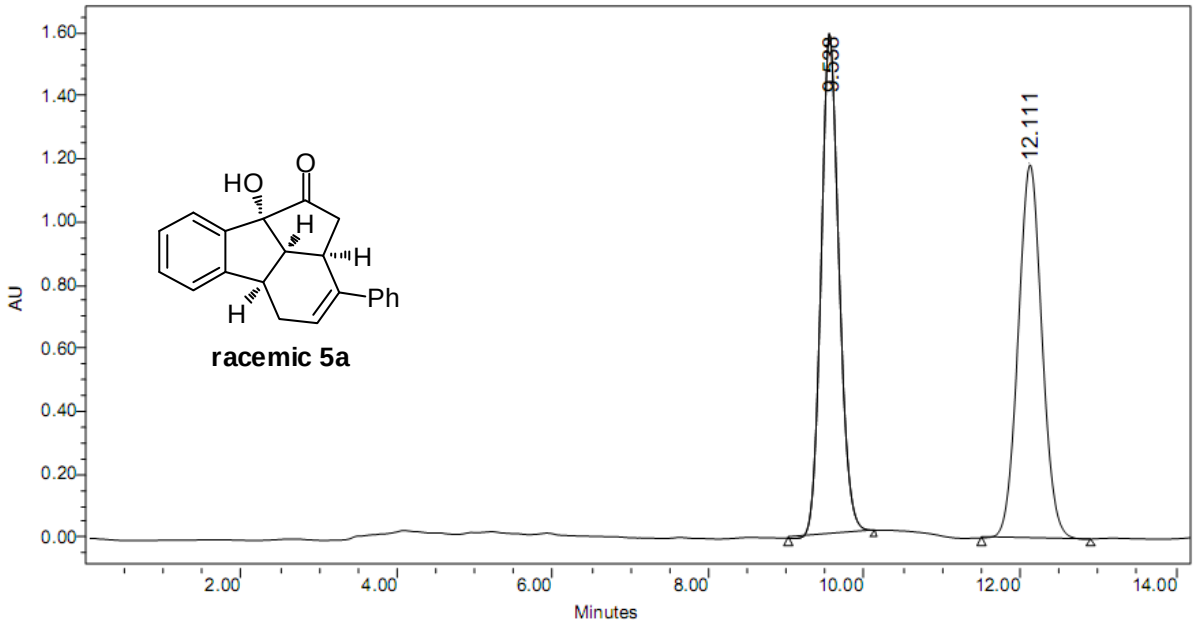
Empirical formula	$C_{27}H_{22}O_5N_4$
Formula weight	482.49
Temperature	150(2)
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a/Å, b/Å, c/Å	6.08870(10), 18.70010(10), 19.54790(10)
α/°, β/°, γ/°,	90.00, 90.00, 90.00
Volume/Å^3	2225.71(4)
Z	4
ρ_{calc}mg/mm^3	1.440
m/mm^{-1}	0.836
F(000)	1008
Crystal size	0.36 × 0.30 × 0.24
Theta range for data collection	3.27 to 69.54°
Index ranges	$-7 \le h \le 6$, $-22 \le k \le 22$, $-23 \le l \le 23$
Reflections collected	16749
Independent reflections	4134[R(int) = 0.0164]
Data/restraints/parameters	4134/0/326
Goodness-of-fit on F^2	1.069
Final R indexes [I>2σ (I)]	R_1 = 0.0256, wR_2 = 0.0669
Final R indexes [all data]	R_1 = 0.0258, wR_2 = 0.0672
Largest diff. peak/hole	0.129/-0.175

6. NMR spectra and HPLC chromatograms

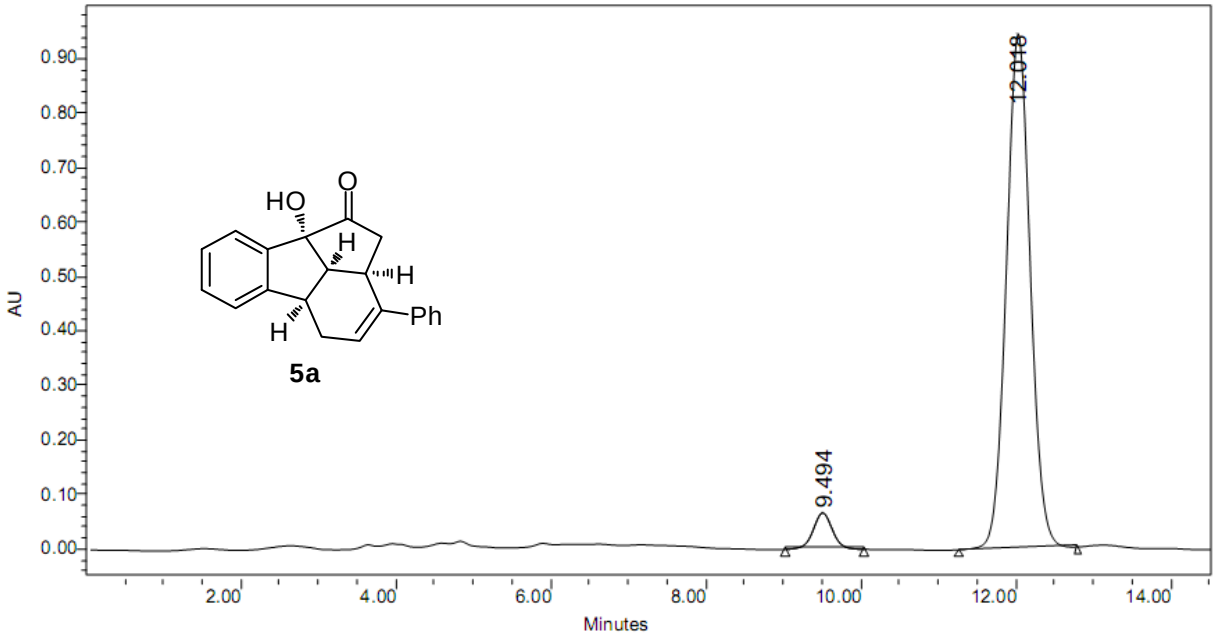






racemic 5a

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.538	25351442	50.18	1588580	57.28
2	12.111	25167069	49.82	1184540	42.72



5a

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
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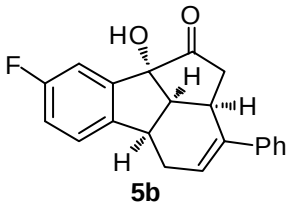
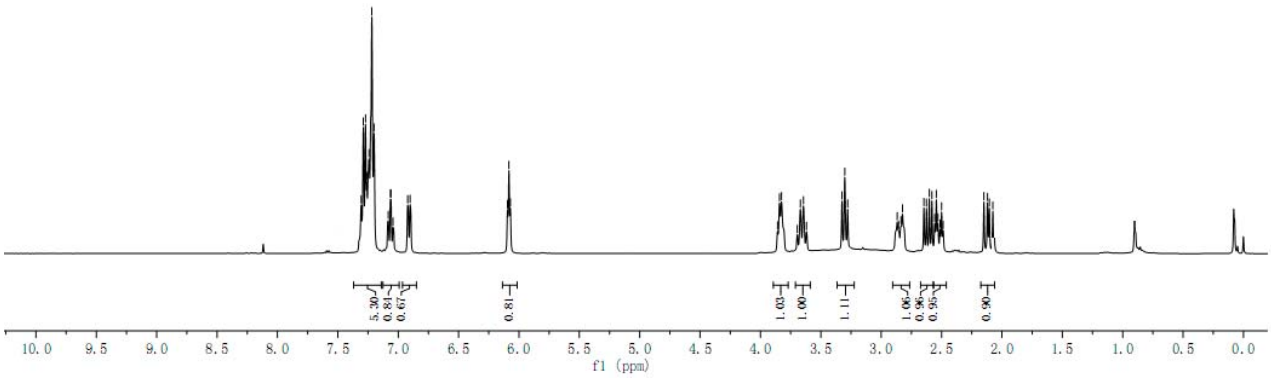
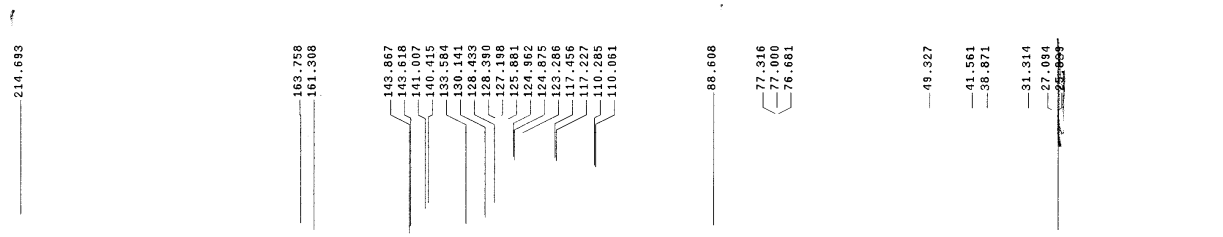
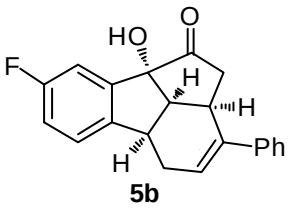
2	12.018	20248317	94.57	944784	93.40


HO
O
F
H
H
H
Ph

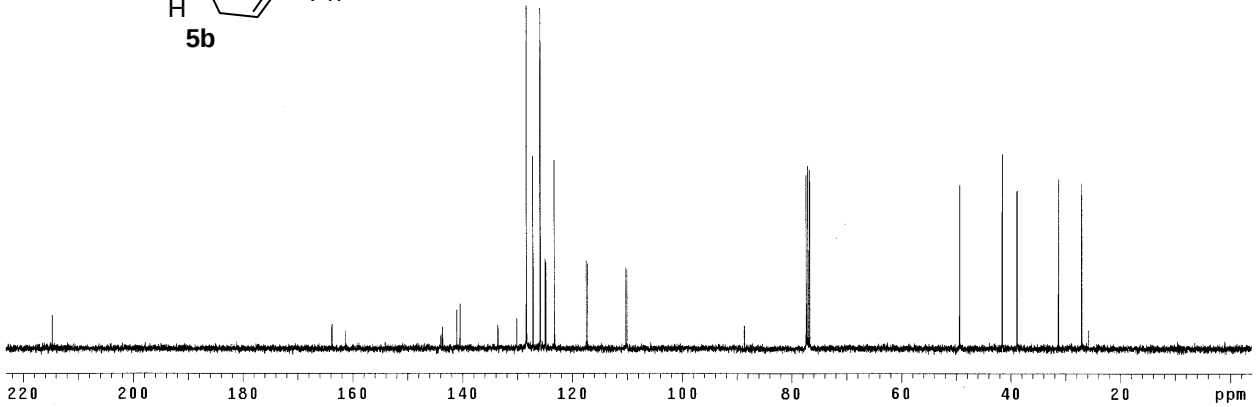
5b

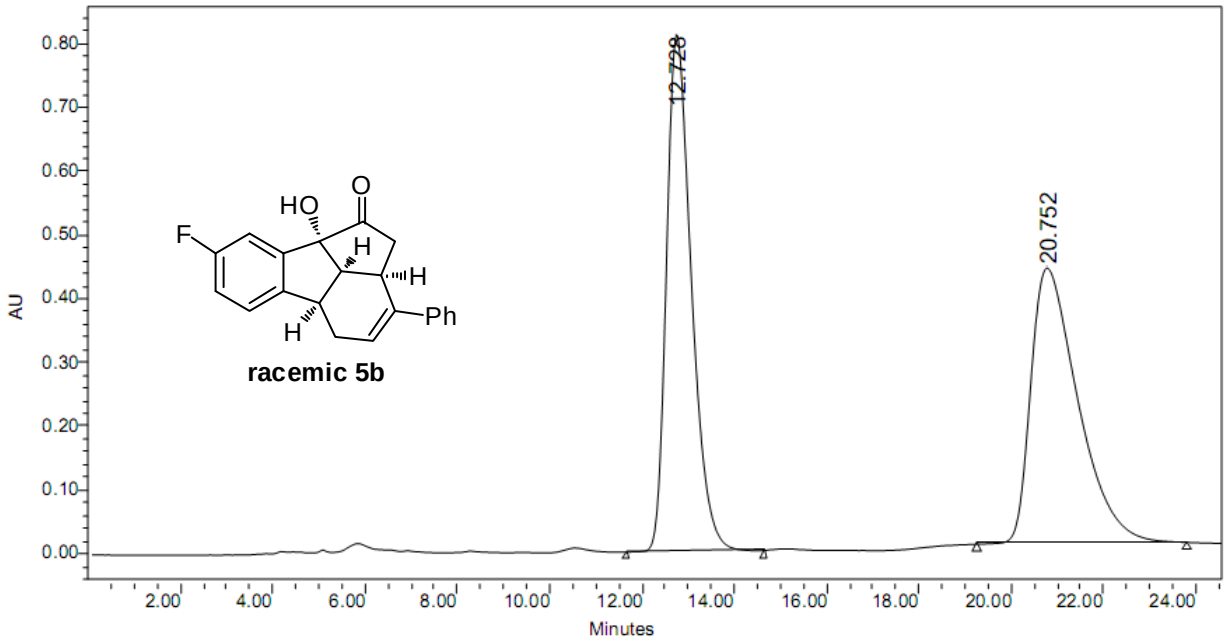

5.30
0.81
0.67
0.81
1.03
1.00
1.11
1.06
0.96
0.95
0.90
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



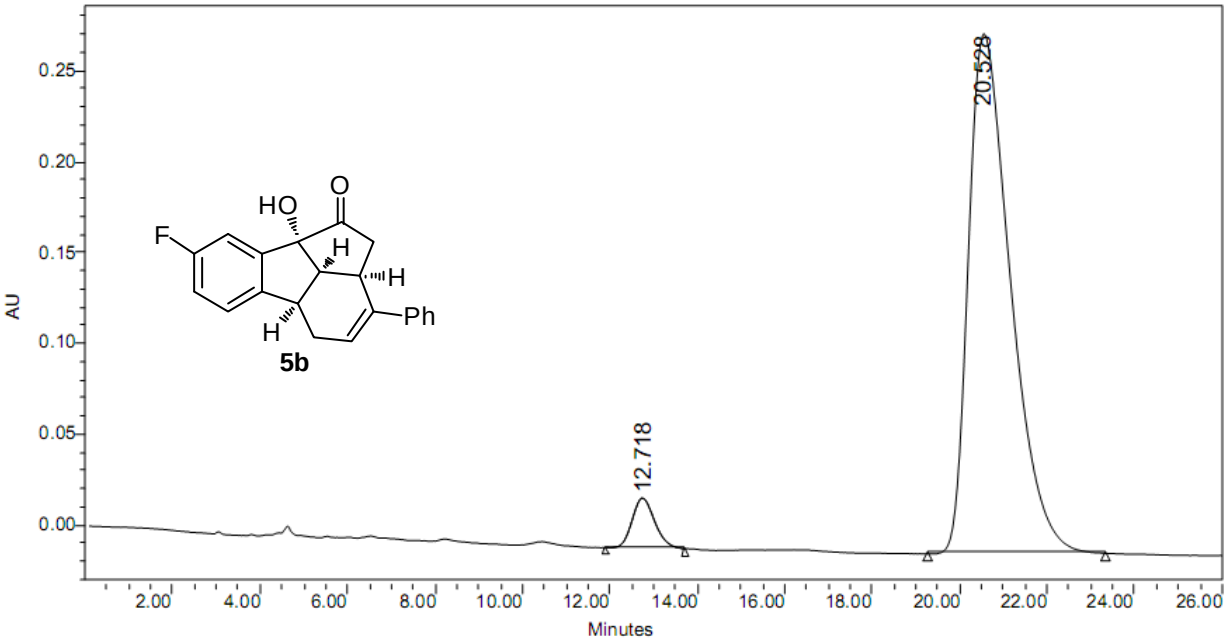

HO
O
F
H
H
H
Ph

5b


220 200 180 160 140 120 100 80 60 40 20 ppm



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.728	30378579	49.79	810352	65.22
2	20.752	30629162	50.21	432072	34.78



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
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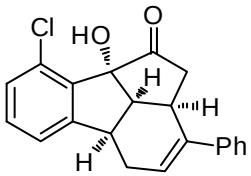
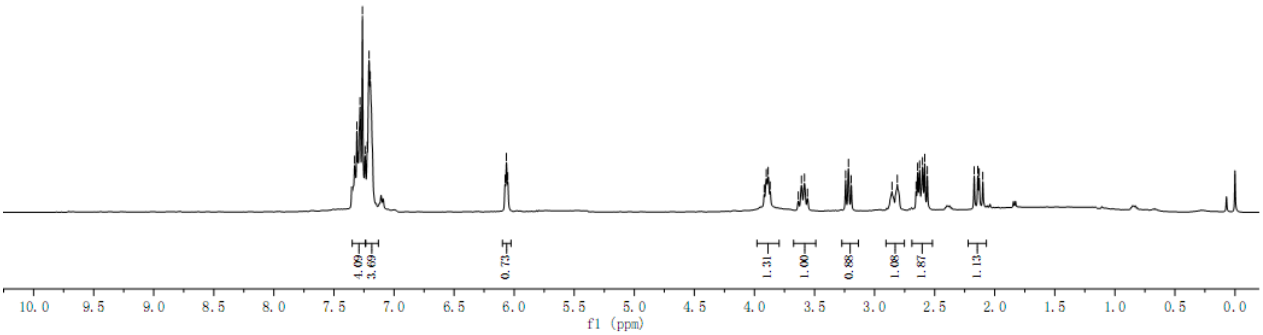
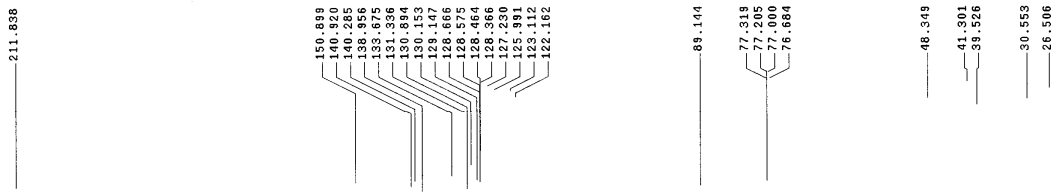
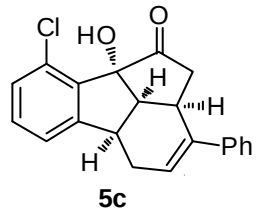
2	20.528	19145078	95.08	285991	91.24


Cl
HO
O
H
H
H
Ph

5c

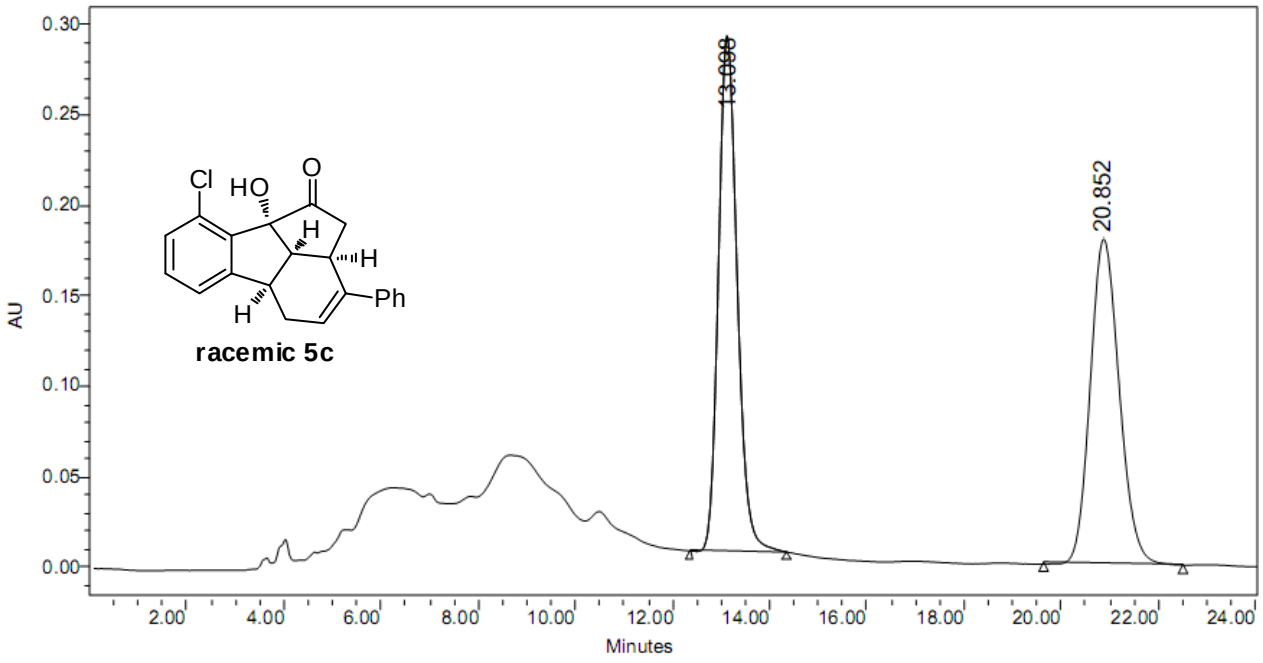

4.09
3.69
0.73
1.31
1.00
0.88
1.08
1.87
1.13
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)




Cl
HO
O
H
H
H
Ph

5c

200 180 160 140 120 100 80 60 40 20 ppm



racemic 5c

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
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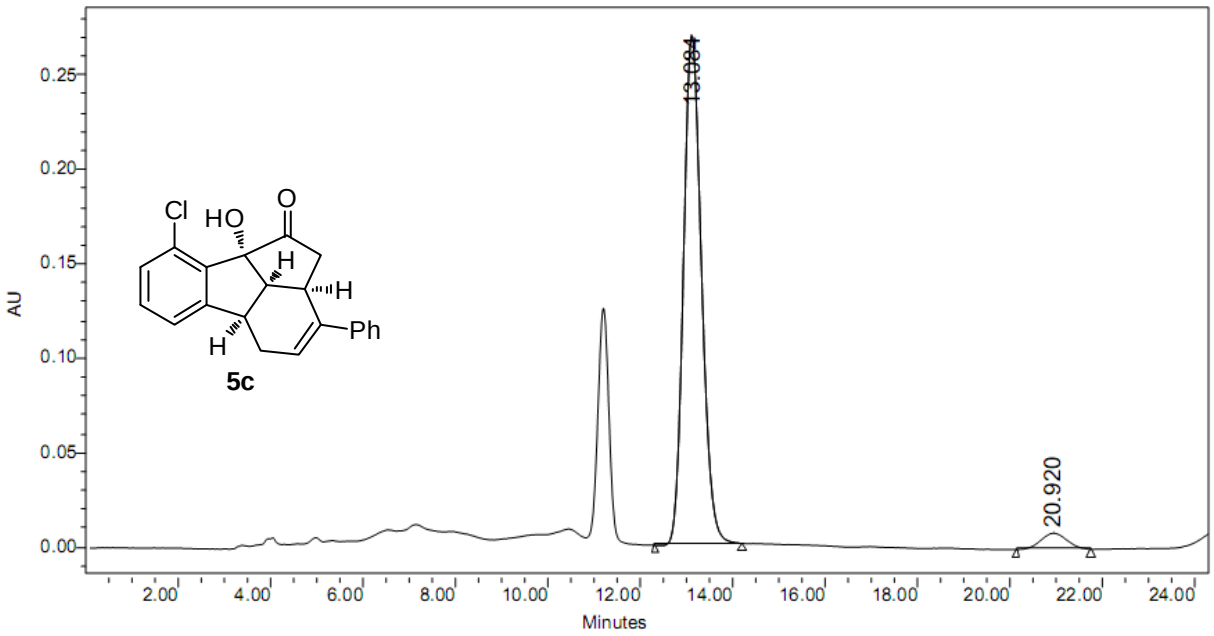
2	20.852	7497542	49.46	179936	38.70



5c

	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
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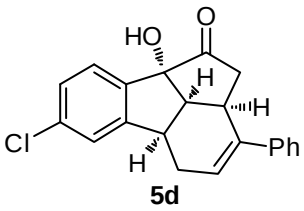
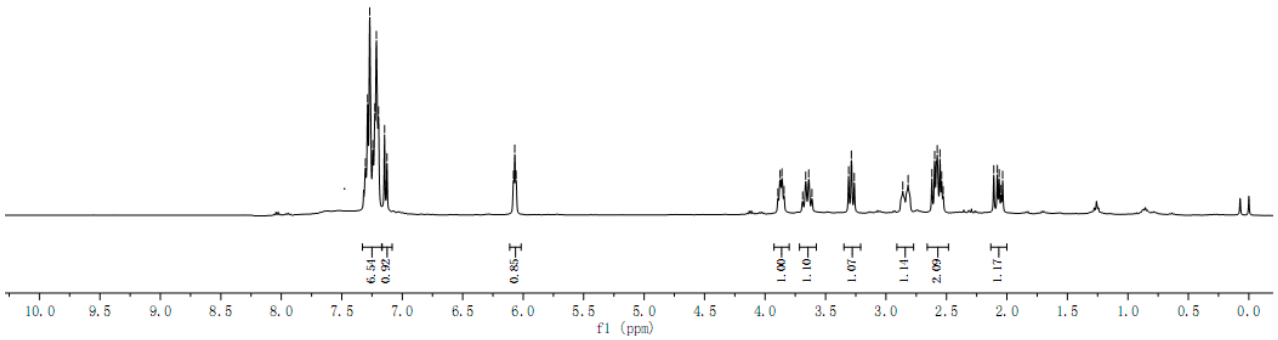
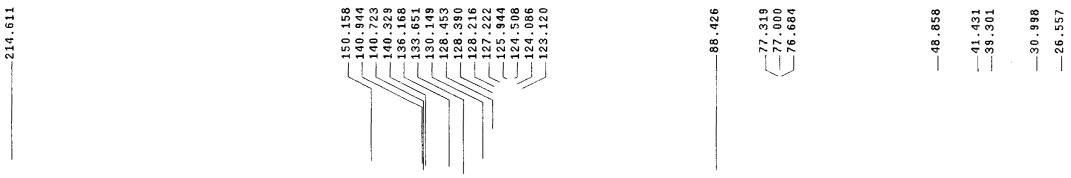
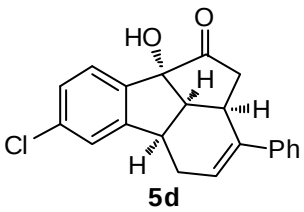

HO
O
H
H
Cl
Ph
H

5d

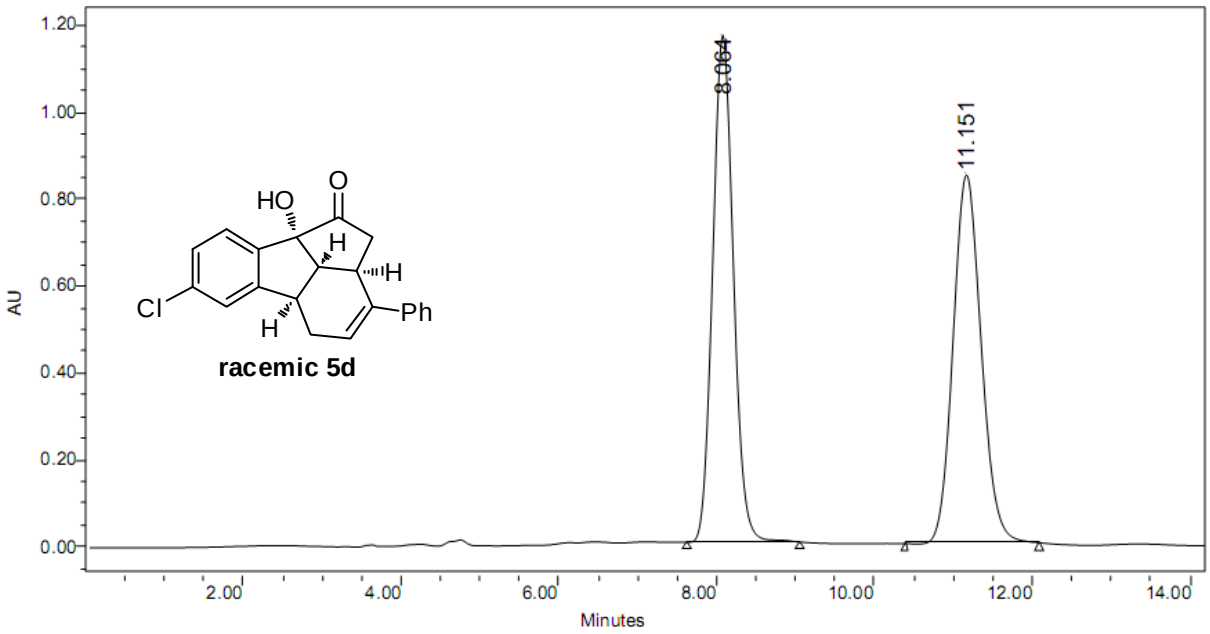

6.54
0.92
0.85
1.00
1.10
1.07
1.14
2.09
1.17
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



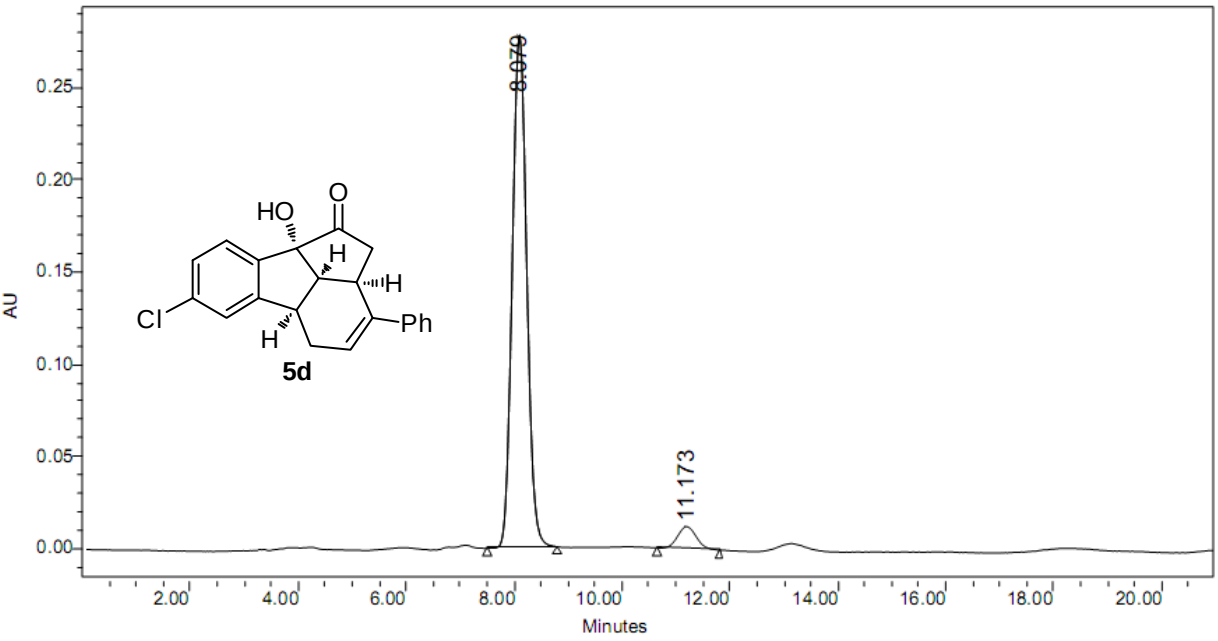

HO
O
H
H
Cl
Ph
H

5d

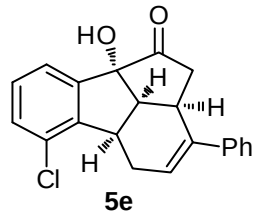
200 180 160 140 120 100 80 60 40 20 ppm



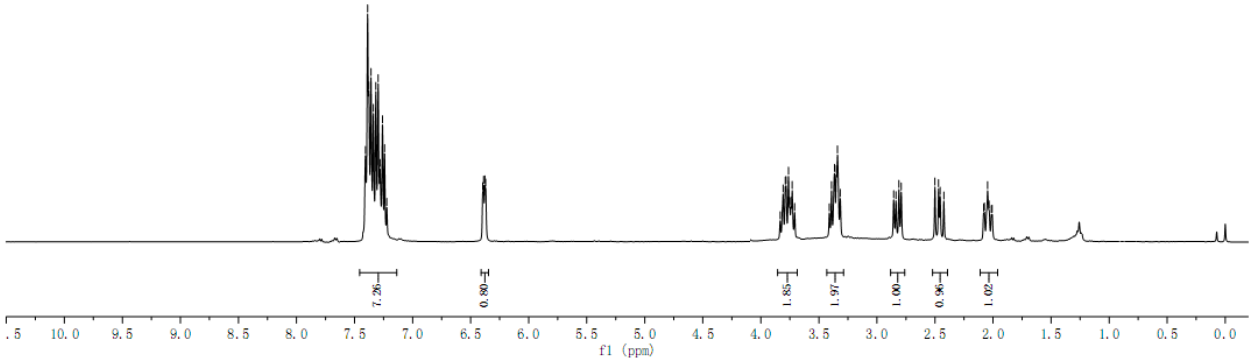
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	8.064	21302404	50.38	1168265	57.93
2	11.151	20983624	49.62	848275	42.07

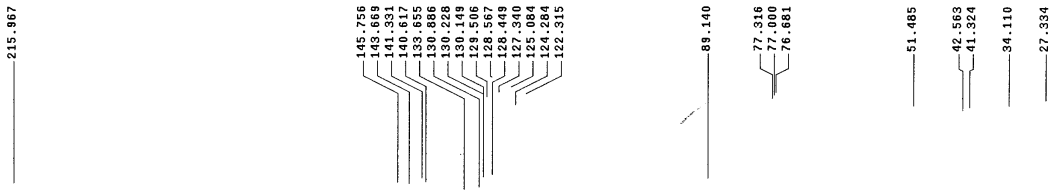


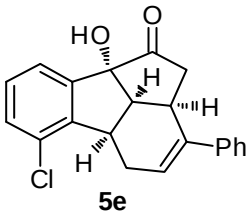
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	8.079	5181117	94.68	278513	95.84
2	11.173	291372	5.32	12103	4.16



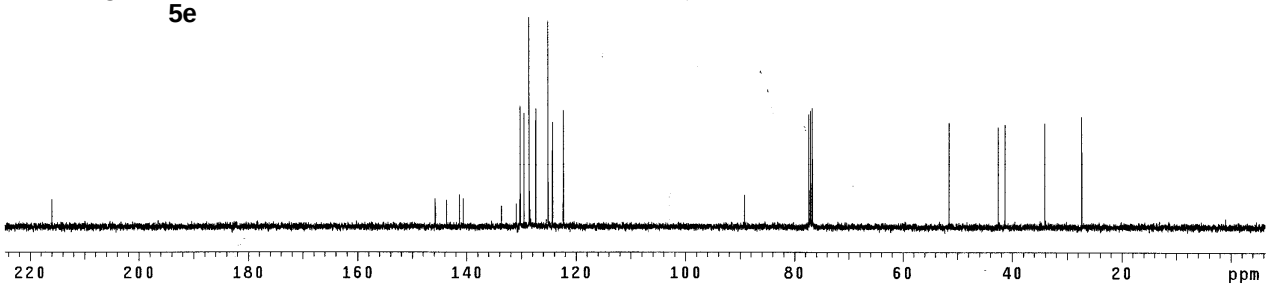
5e

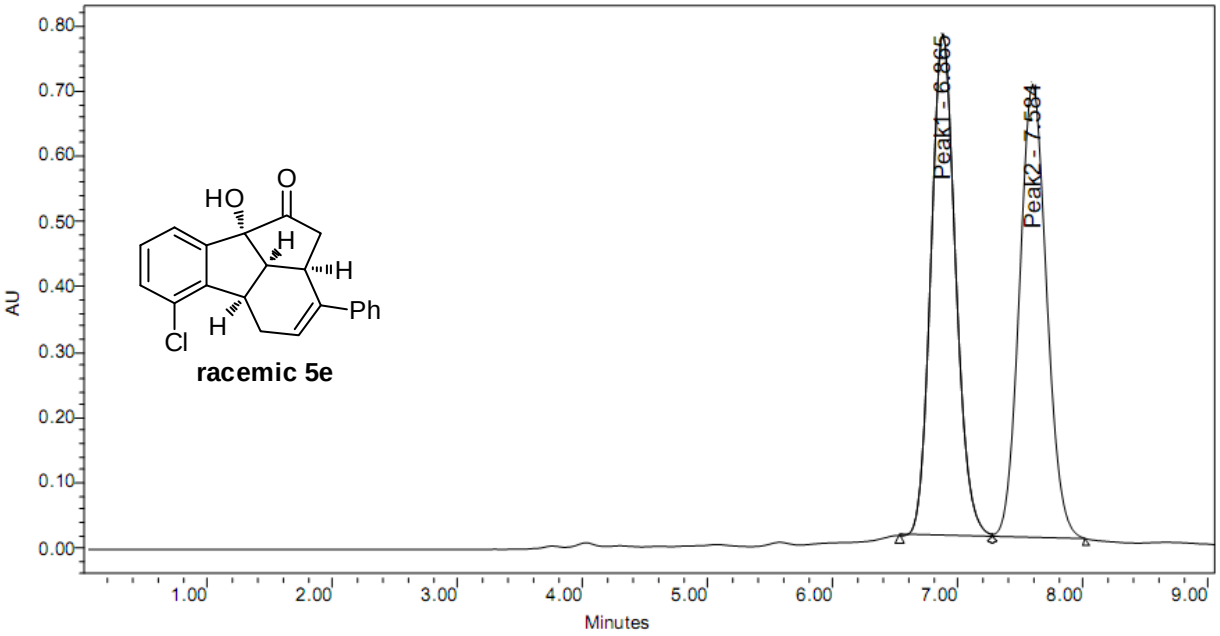




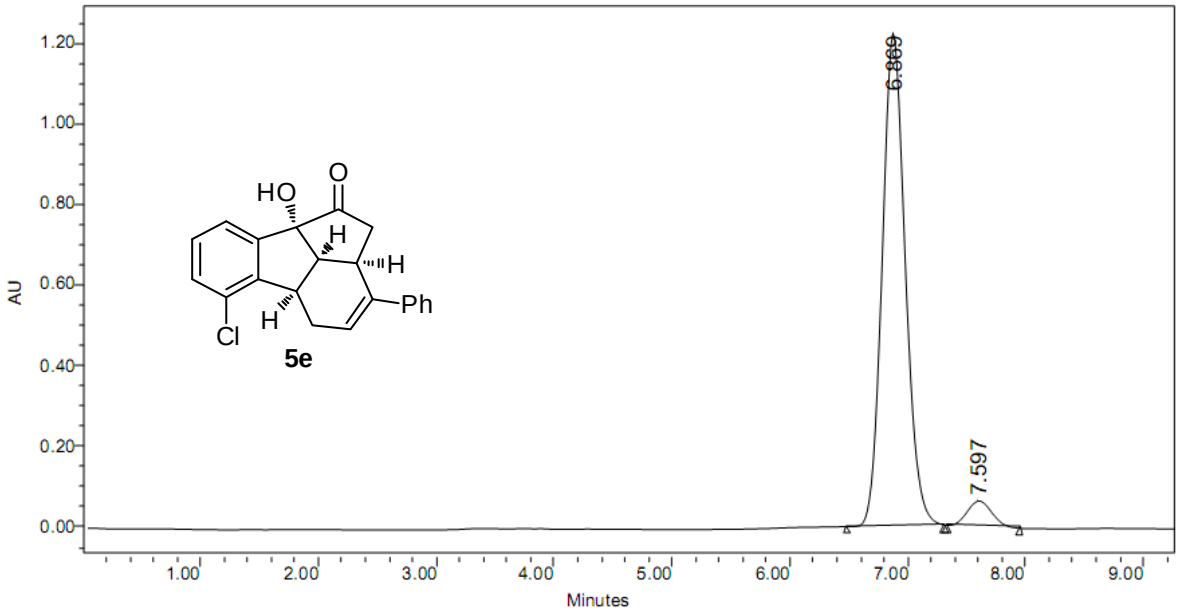


5e

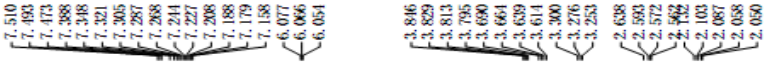




	Peak Name	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	Peak1	6.865	10566578	50.18	769411	52.54
2	Peak2	7.584	10492043	49.82	695046	47.46



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	6.869	16539009	94.70	1222212	95.06
2	7.597	926170	5.30	63572	4.94


7.510
7.493
7.473
7.388
7.348
7.321
7.305
7.287
7.268
7.244
7.227
7.208
7.188
7.179
7.158
6.077
6.066
6.054
3.846
3.829
3.813
3.795
3.690
3.661
3.639
3.614
3.300
3.276
3.253
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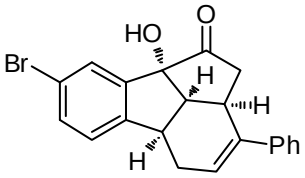
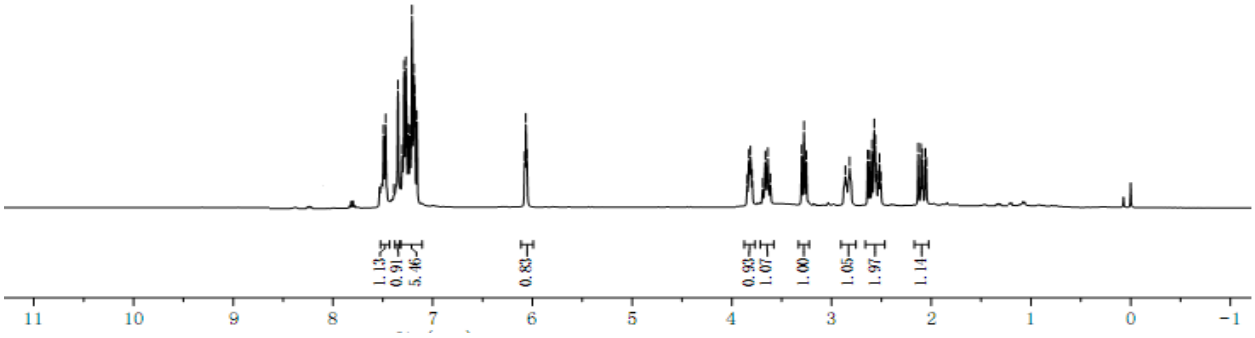
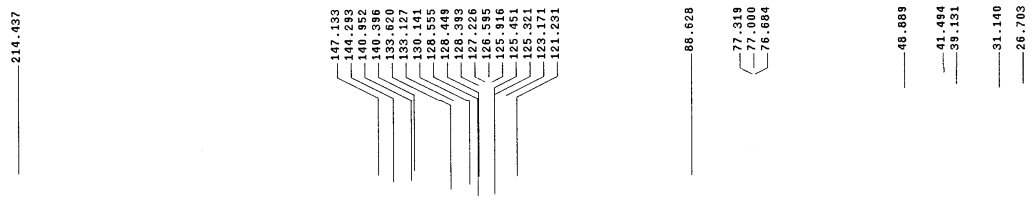
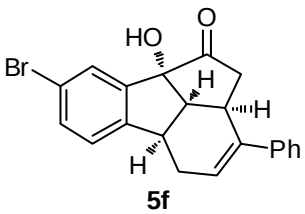
2.562
2.152
2.103
2.087
2.038
2.050


HO
O
Br
H
H
Ph
H

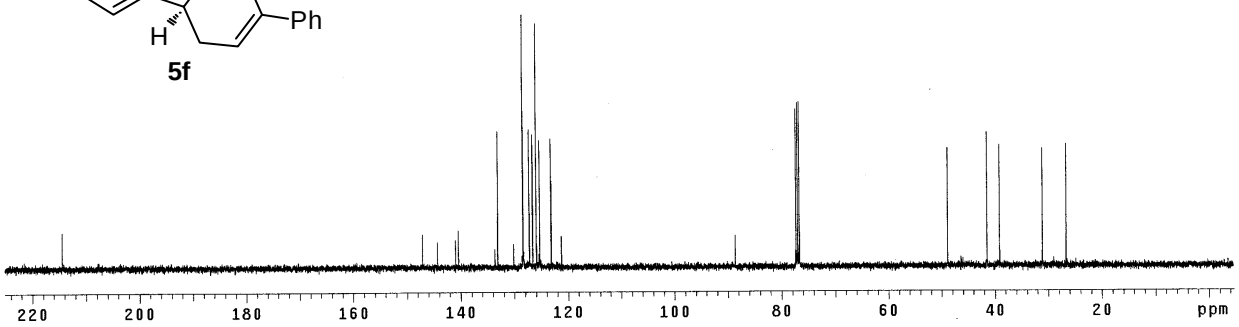
5f

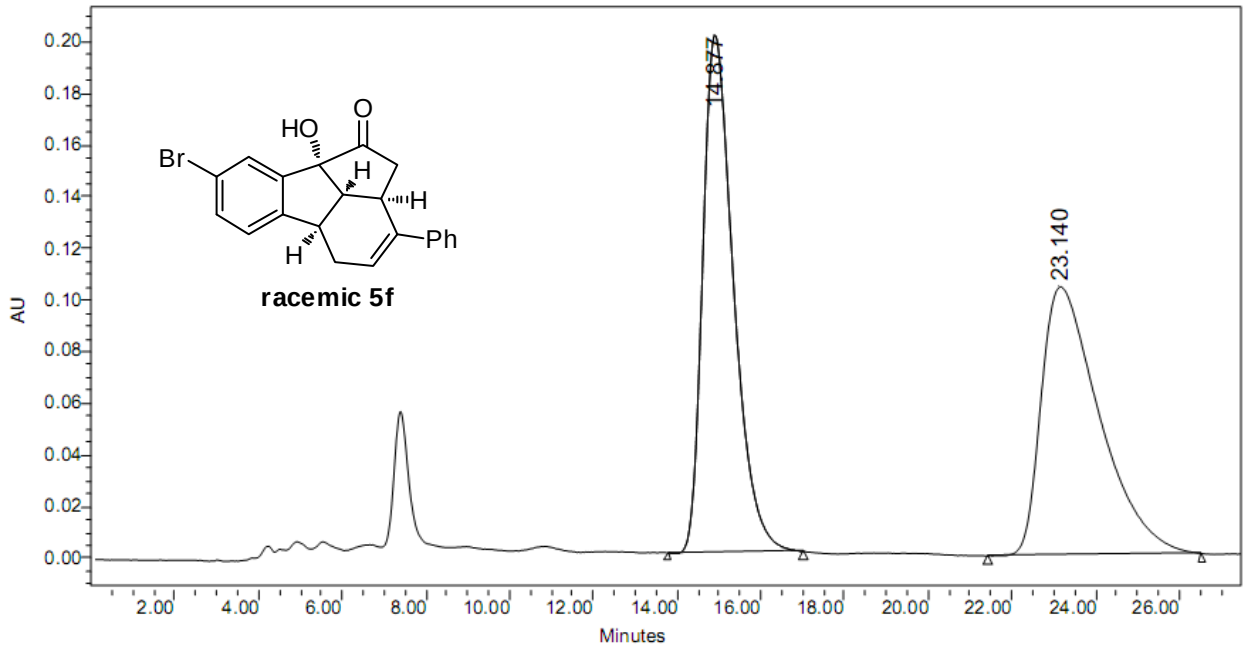

1.13
0.91
5.46
0.83
0.93
1.07
1.00
1.05
1.97
1.14
11
10
9
8
7
6
5
4
3
2
1
0
−1


214.437
147.133
144.293
140.952
140.396
133.620
133.127
130.141
128.555
128.449
128.393
127.226
126.595
125.416
125.321
123.171
121.231
88.628
77.319
77.000
76.684
48.889
41.494
39.131
31.140
26.703

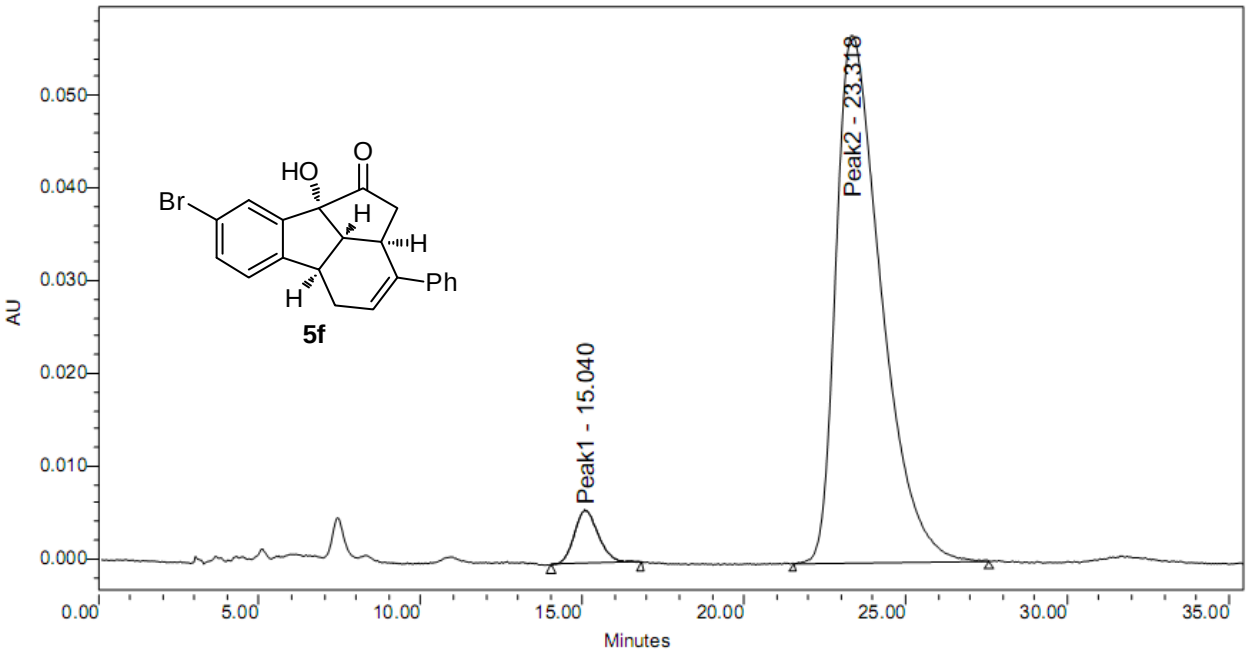

HO
O
Br
H
H
Ph
H

5f

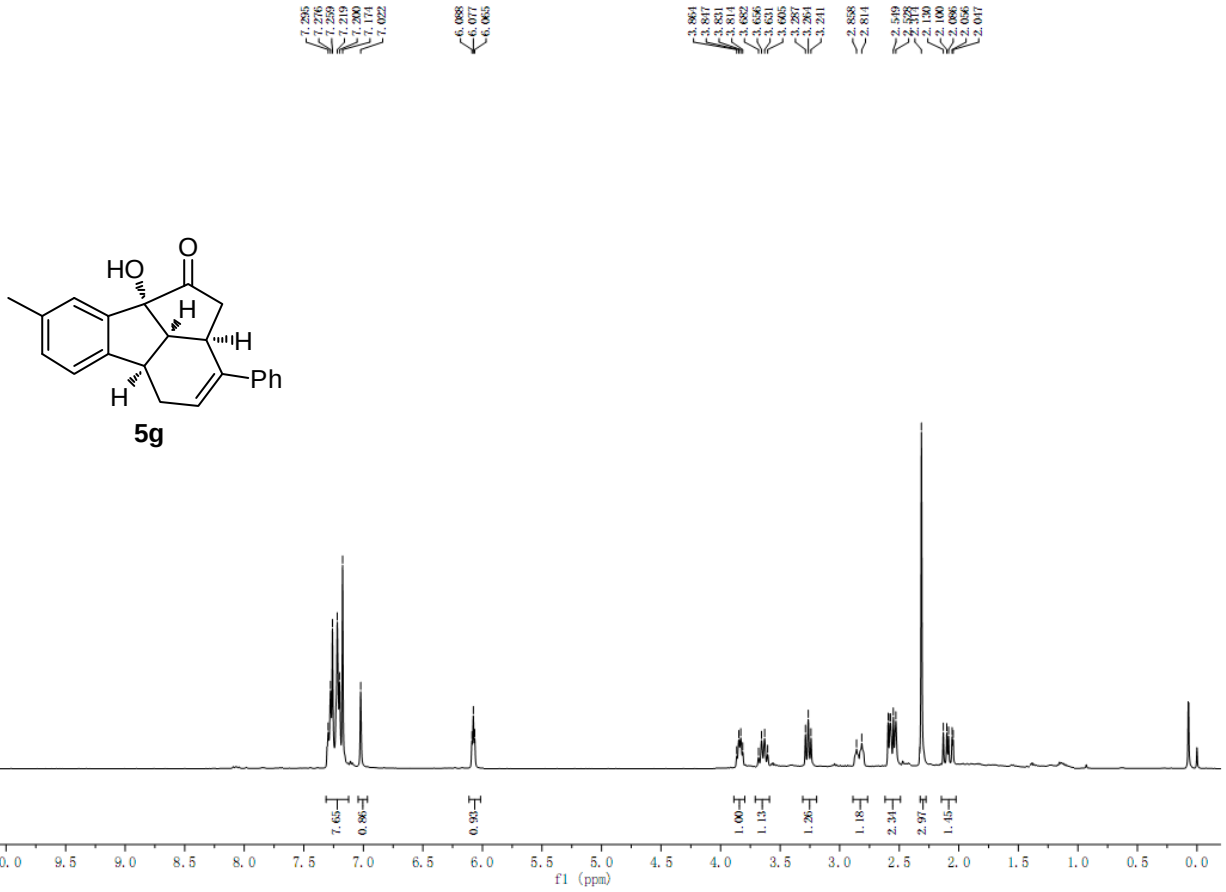

220
200
180
160
140
120
100
80
60
40
20
ppm

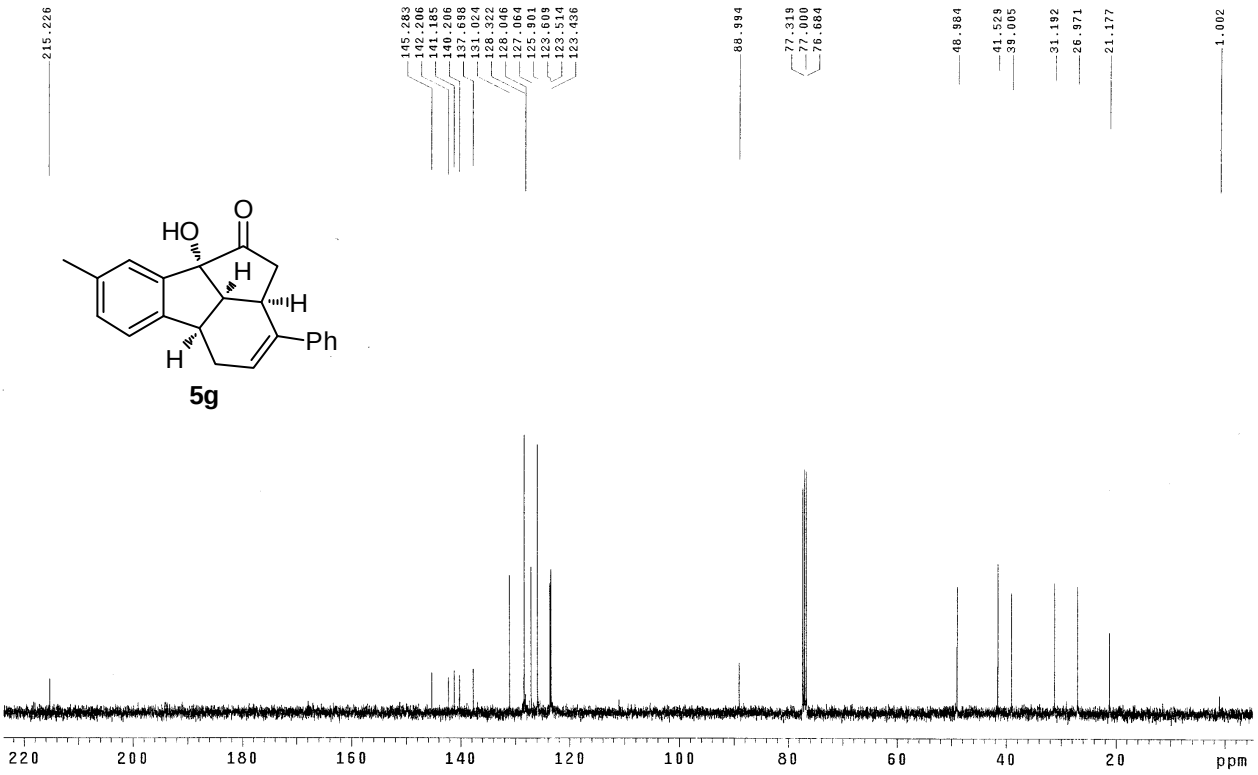


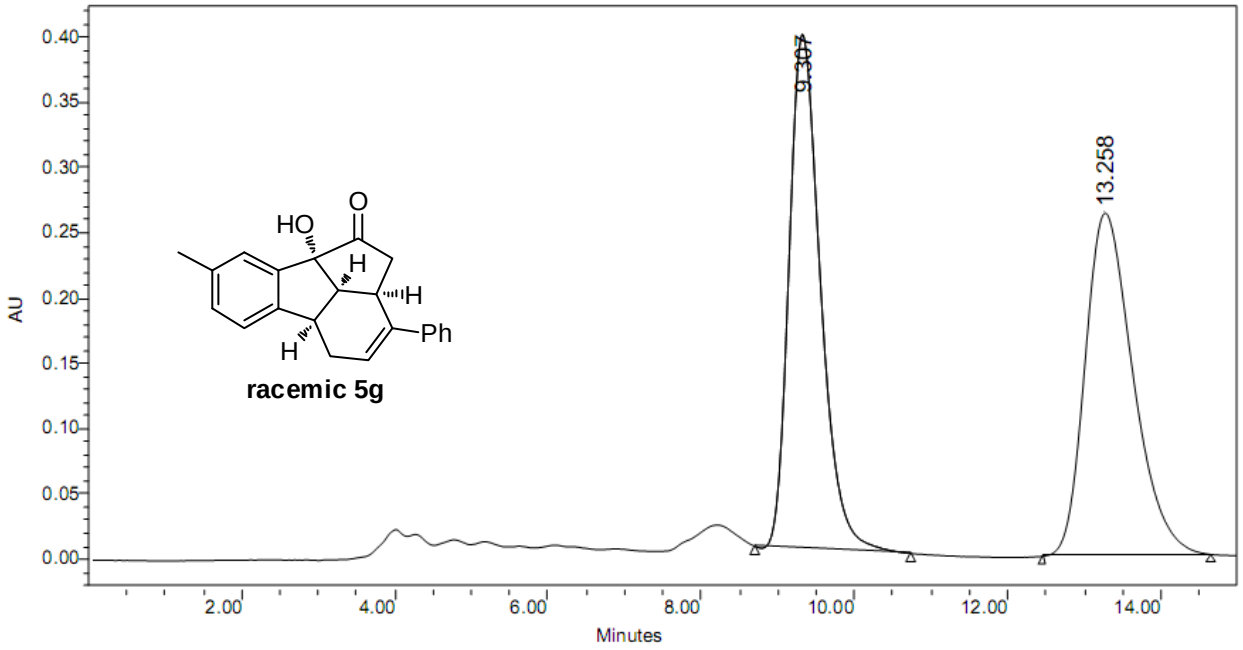
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	14.877	10255800	50.46	200310	65.83
2	23.140	10067201	49.54	103982	34.17



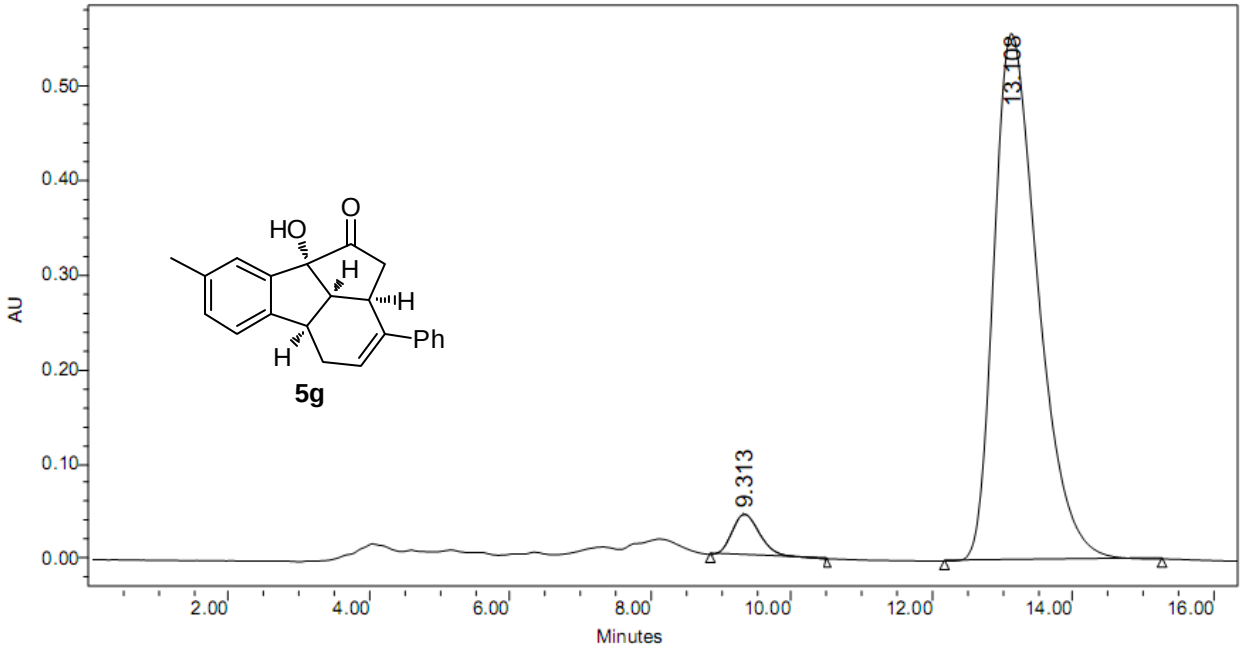
	Peak Name	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	Peak1	15.040	318683	5.42	5871	9.34
2	Peak2	23.318	5563516	94.58	56976	90.66


HO
O
H
H
H
Ph
5g
f1 (ppm)

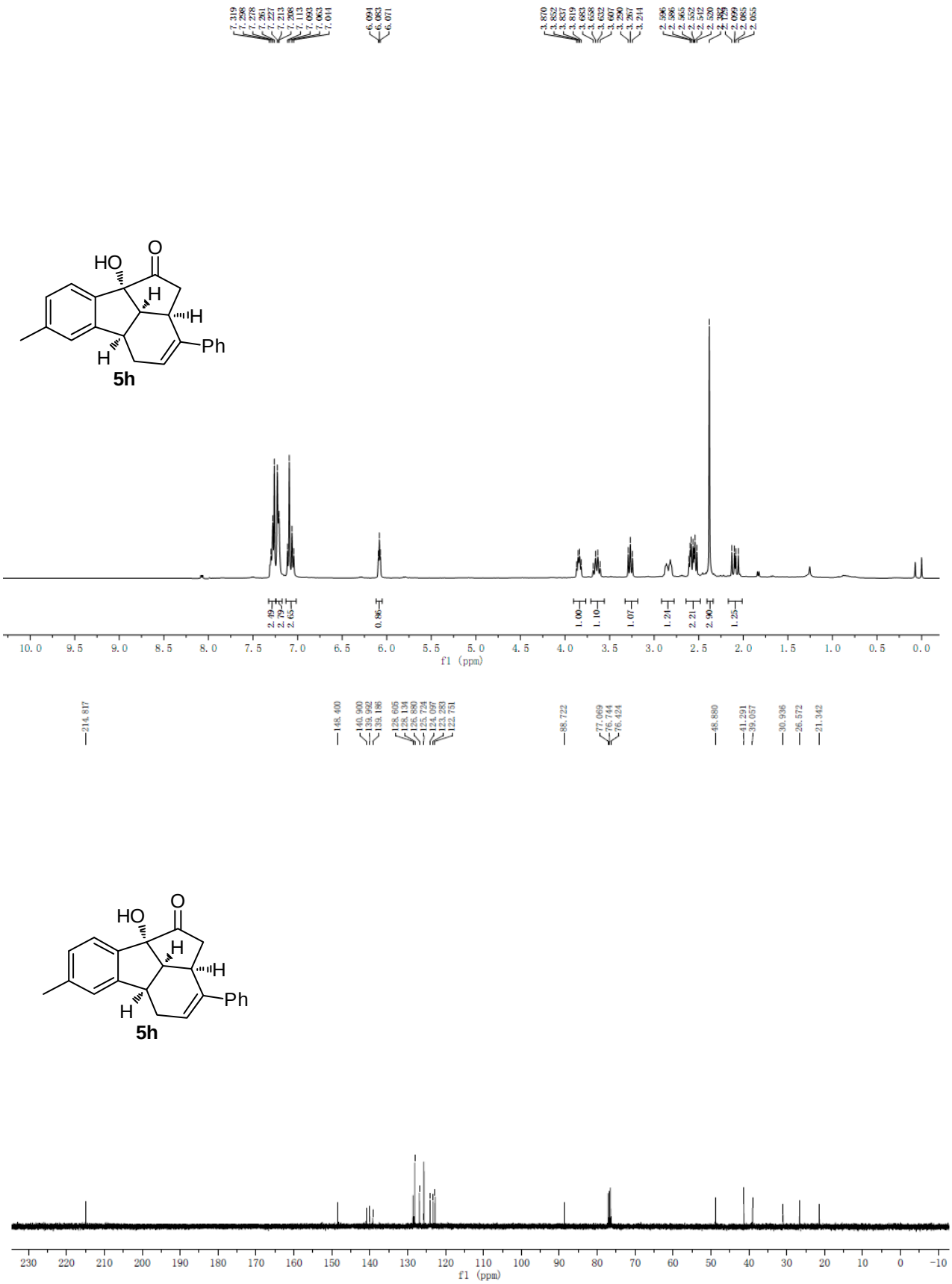

HO
O
H
H
H
Ph
5g
ppm

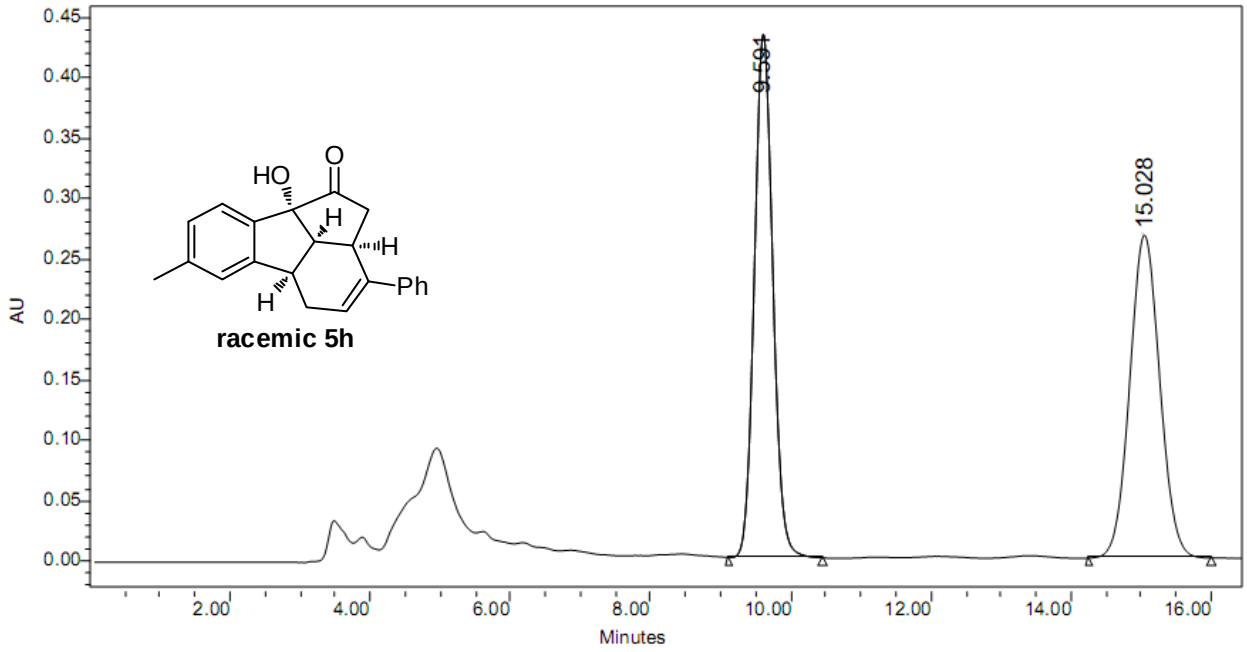


	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.307	11263295	49.72	394385	60.04
2	13.258	11390960	50.28	262476	39.96

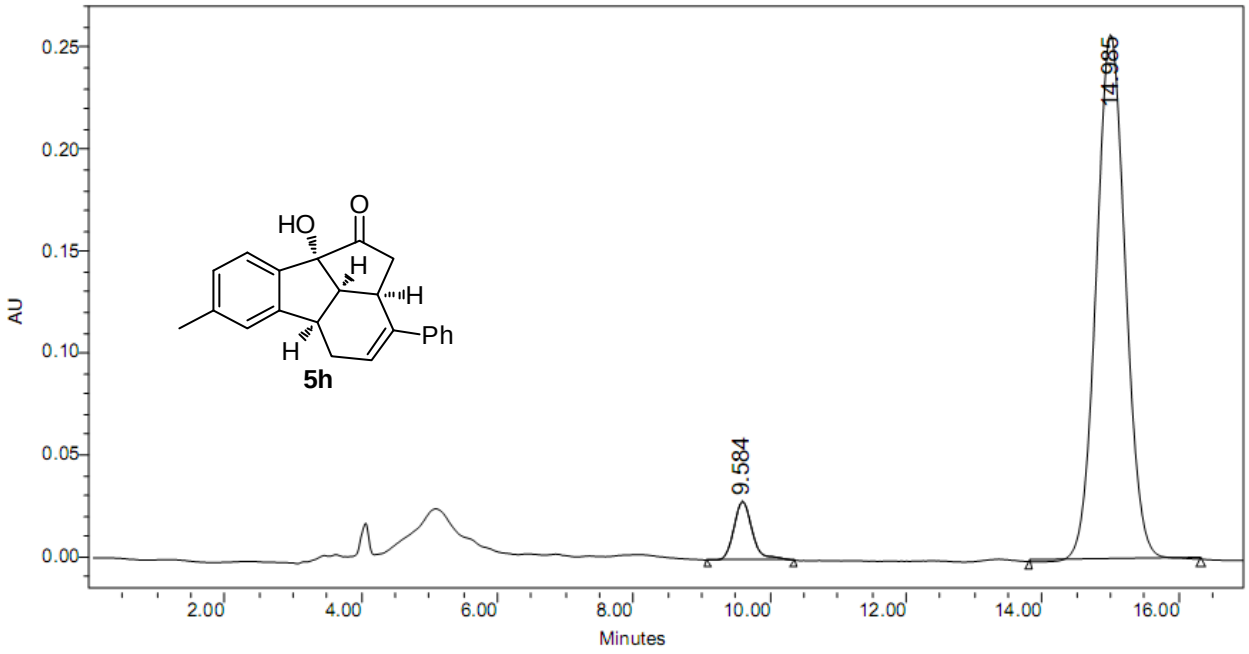


	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.313	1234383	4.83	43829	7.28
2	13.108	24313390	95.17	557986	92.72

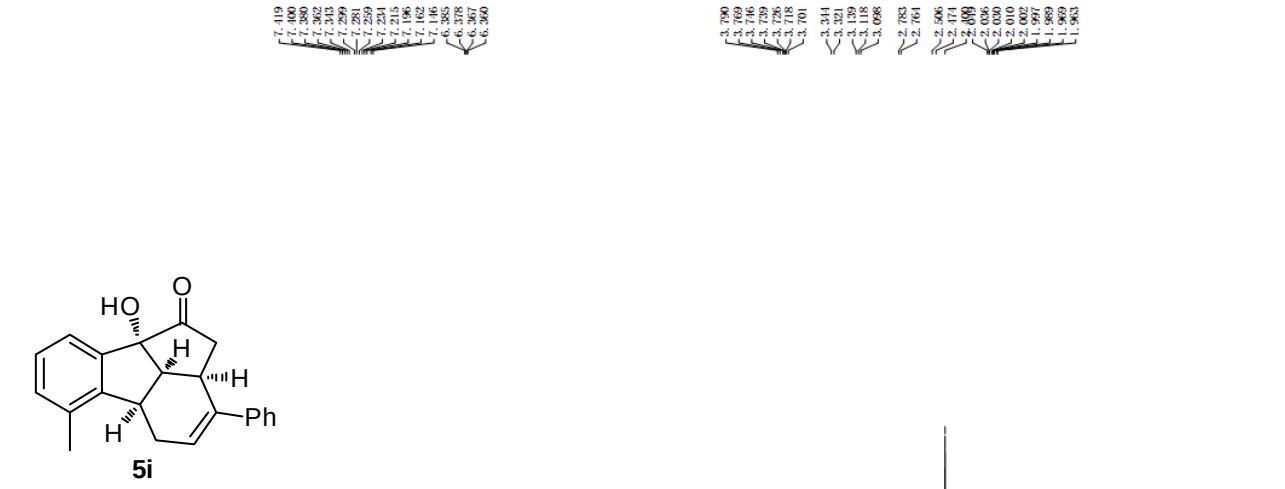

HO
O
H
H
H
Ph
5h
f1 (ppm)
HO
O
H
H
H
Ph
5h
f1 (ppm)

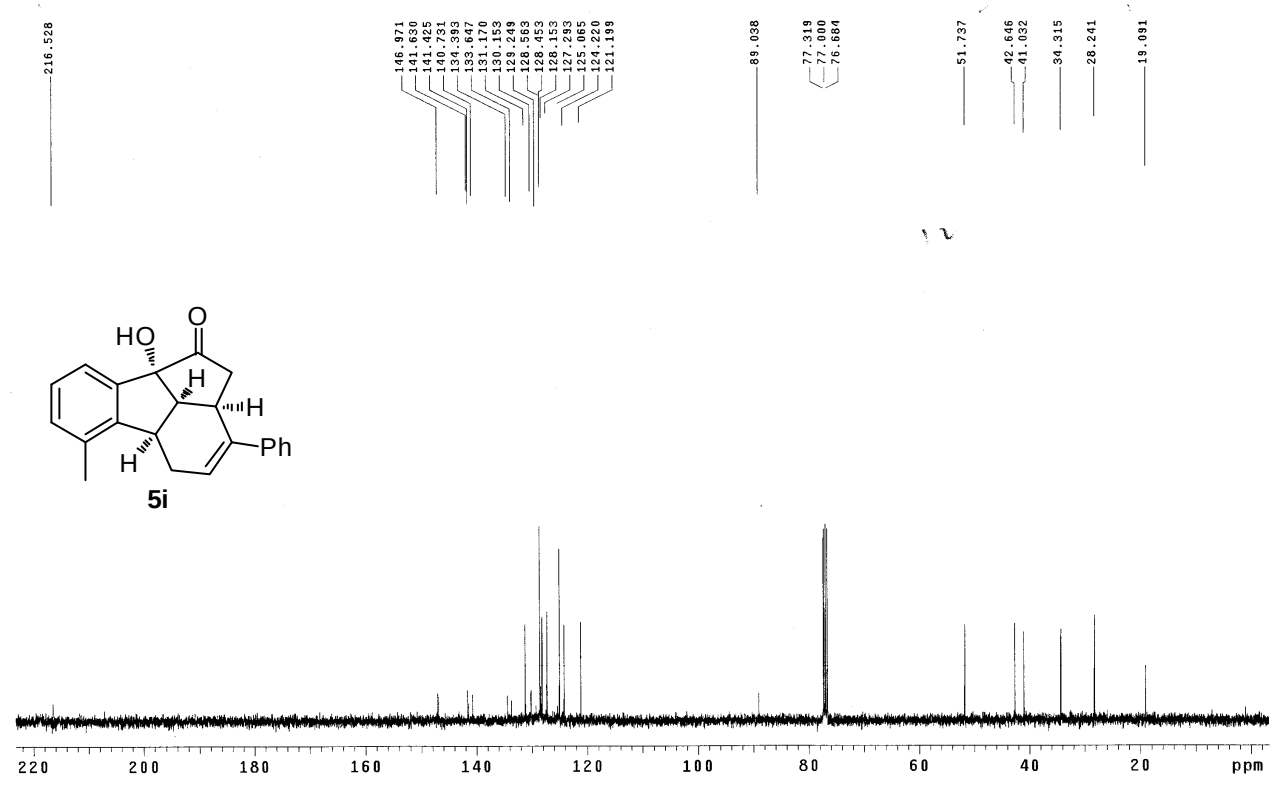


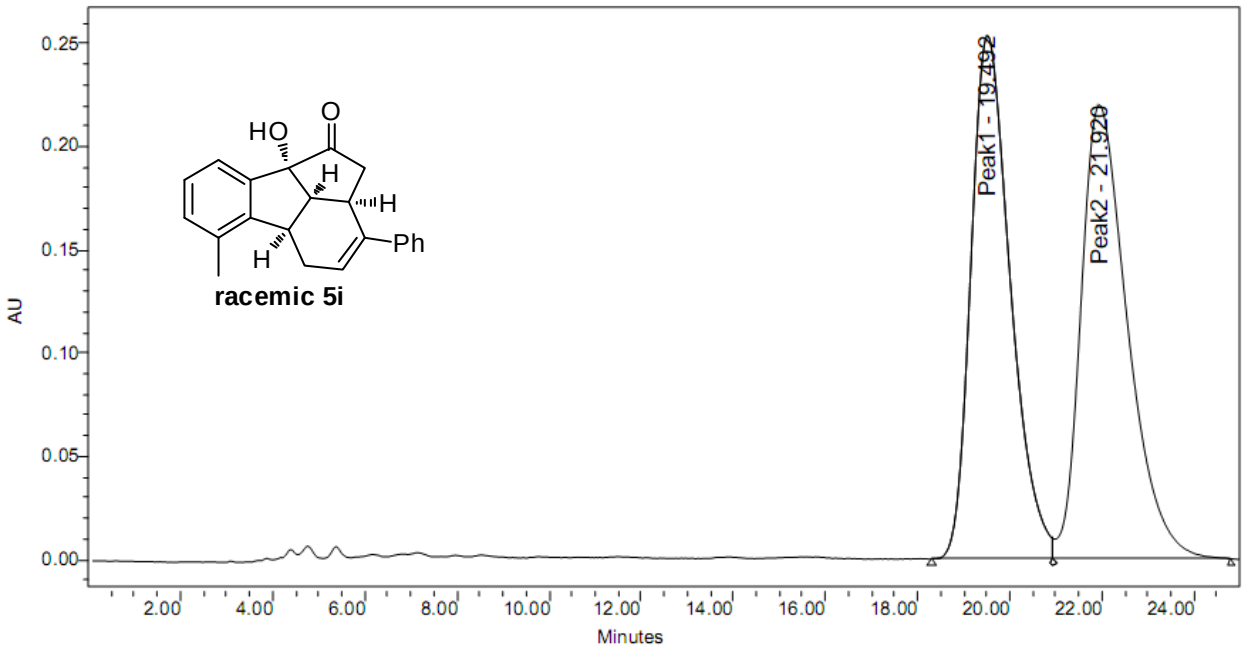
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.591	7935540	49.90	433316	61.85
2	15.028	7966432	50.10	267252	38.15



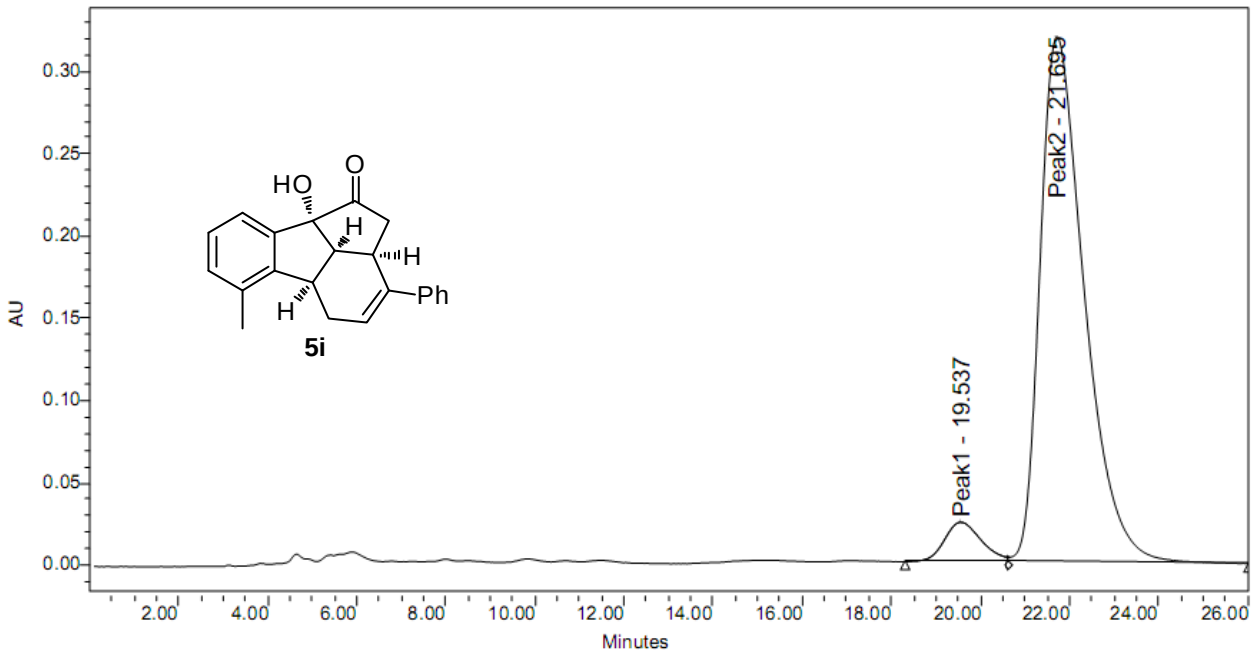
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	9.584	546451	6.61	28640	9.99
2	14.985	7726108	93.39	258073	90.01


HO
O
H
H
H
Ph
5i
f1 (ppm)

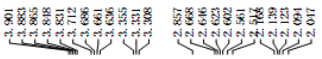

HO
O
H
H
H
Ph
5i
ppm

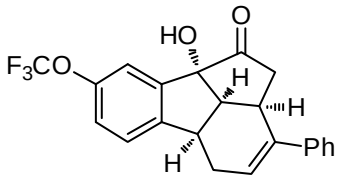


	Peak Name	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	Peak1	19.492	15105431	49.49	253462	53.54
2	Peak2	21.920	15415204	50.51	219933	46.46

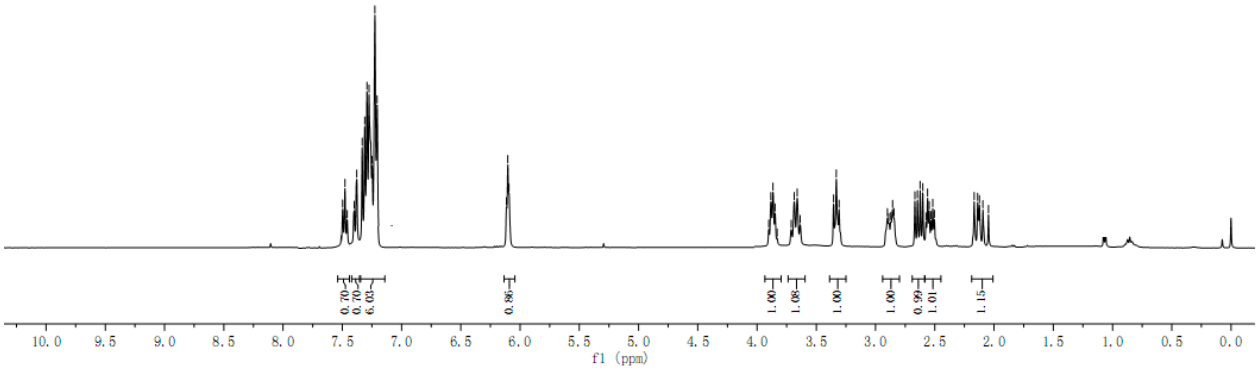


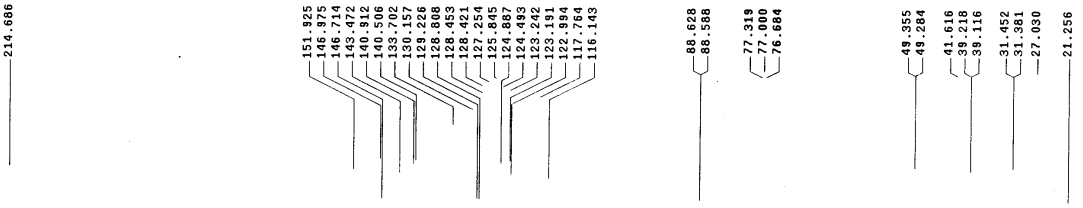
	Peak Name	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	Peak1	19.537	1422259	6.03	24229	7.05
2	Peak2	21.695	22144652	93.97	319628	92.95

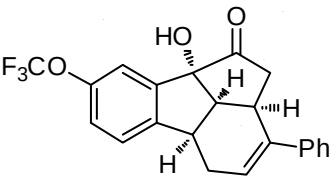



HO
O
F3CO
H
H
H
Ph

5j

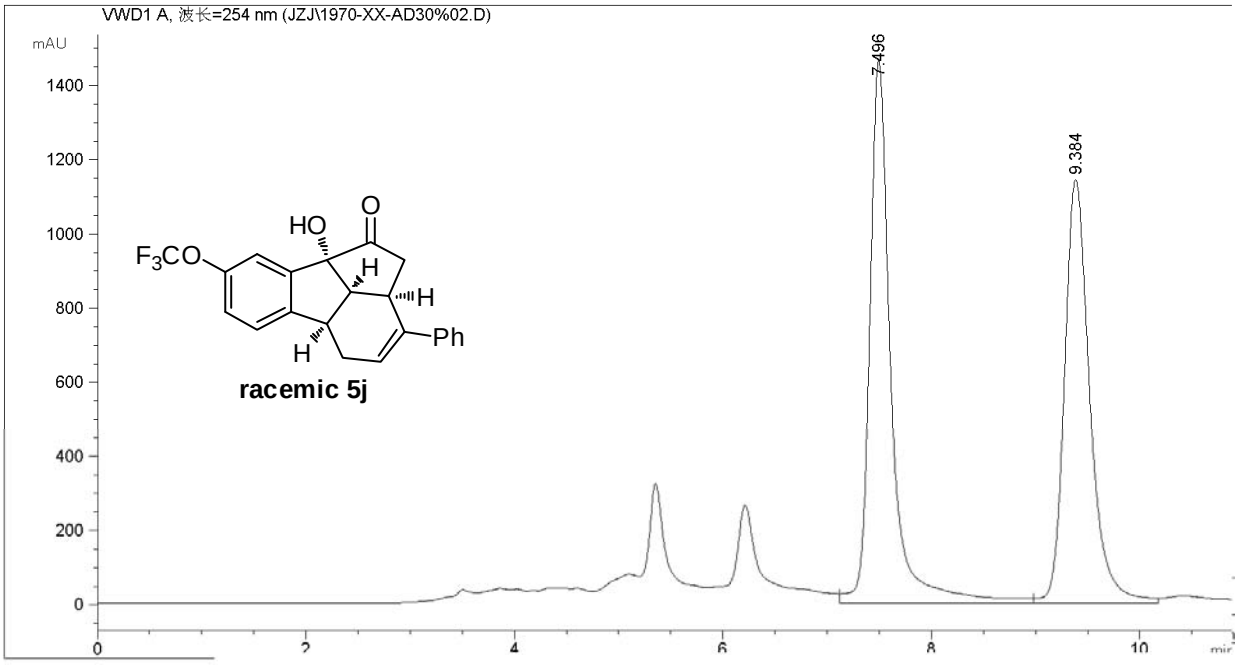

0.70
0.70
6.03
0.98
1.00
1.08
1.00
1.00
0.99
1.01
1.15
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



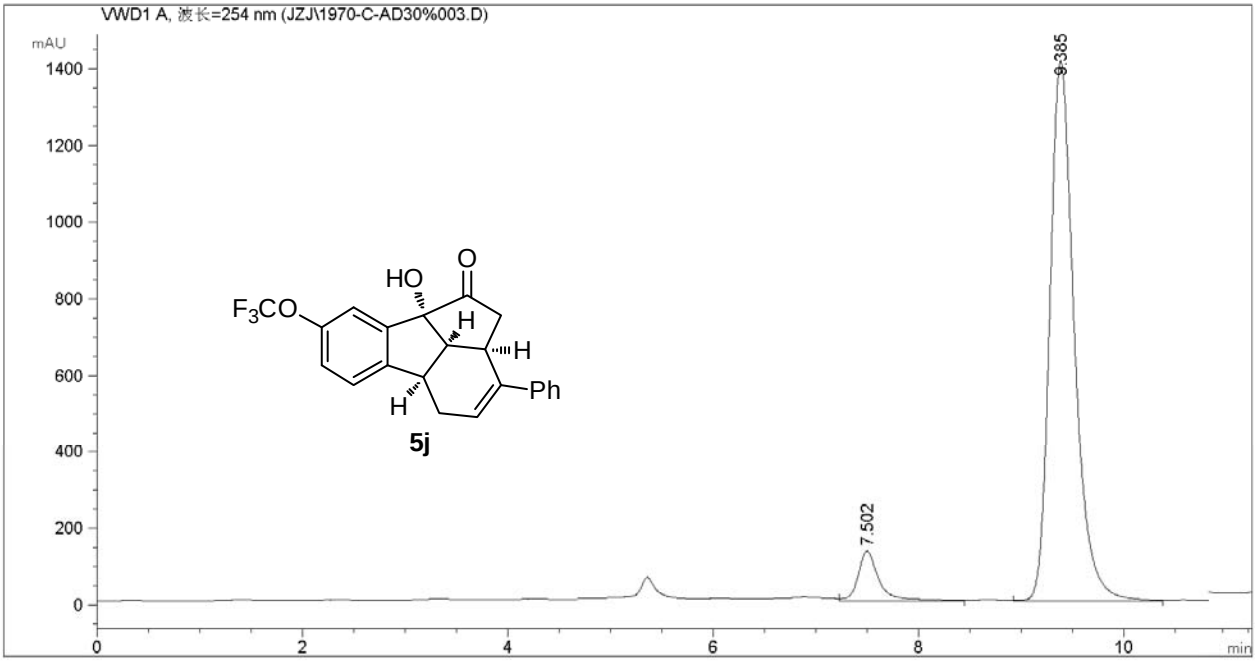

HO
O
F3CO
H
H
H
Ph

5j

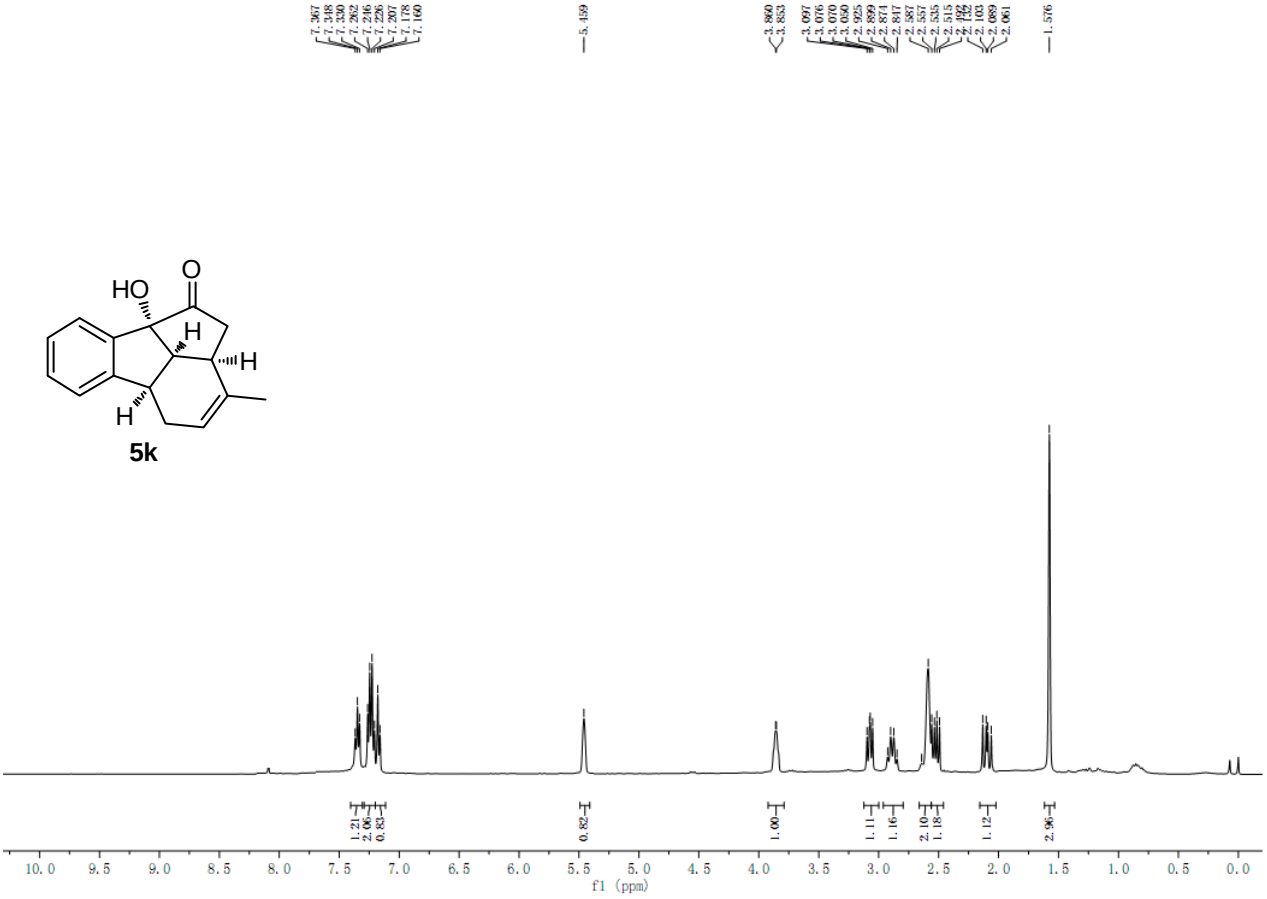
220 200 180 160 140 120 100 80 60 40 20 ppm

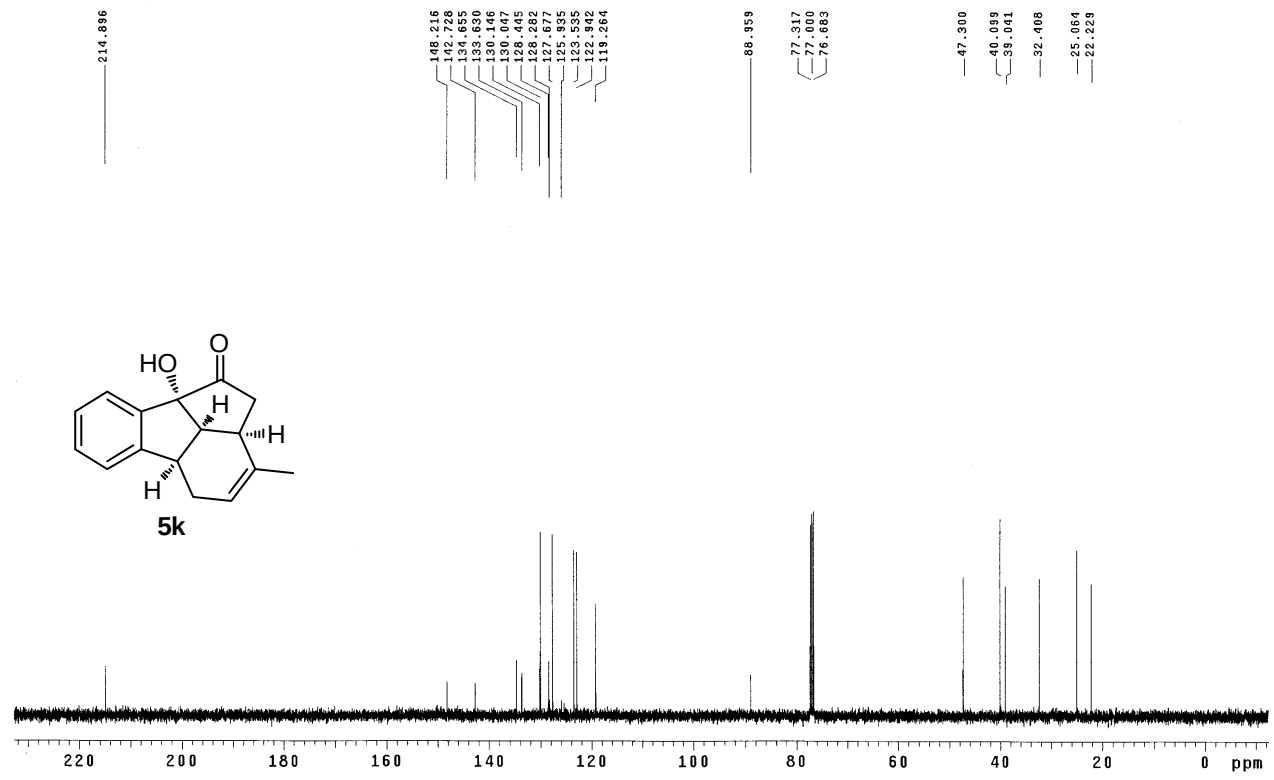


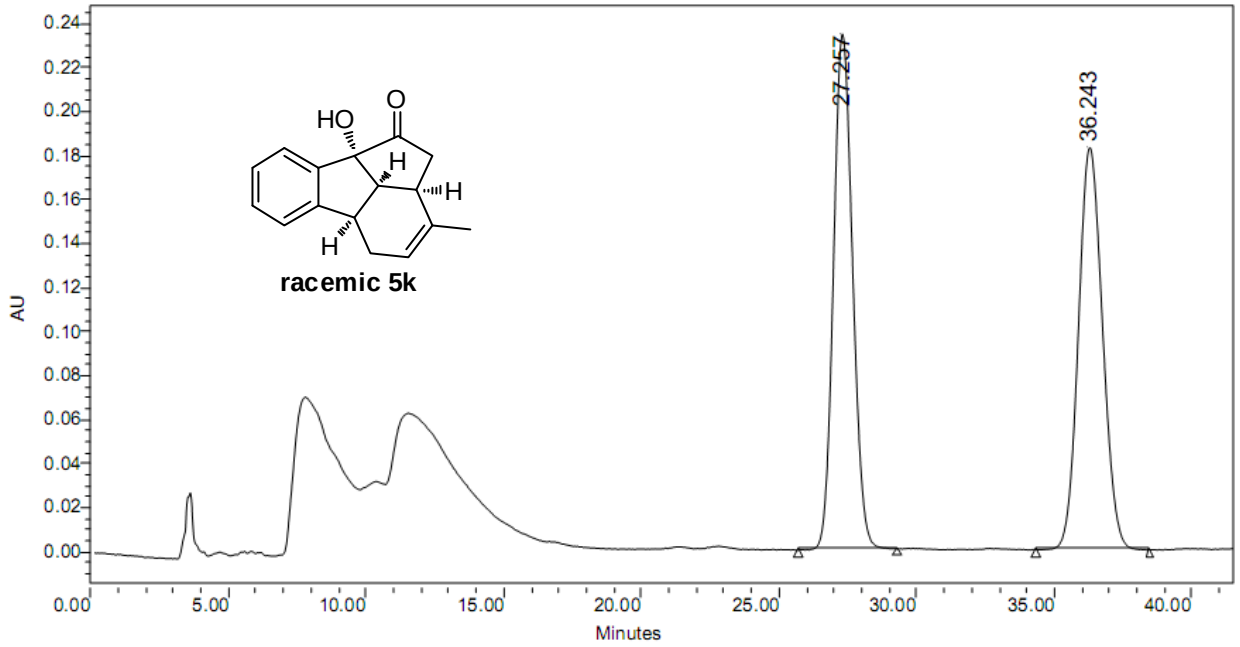
#	RT [min]	Width [min]	Area mAU *S	Height [mAU]	Area %
1	7.496 VV	0.2114	2.08179e4	1461.10498	51.9589
2	9.384 VV	0.2552	1.92482e4	1140.35535	48.0411



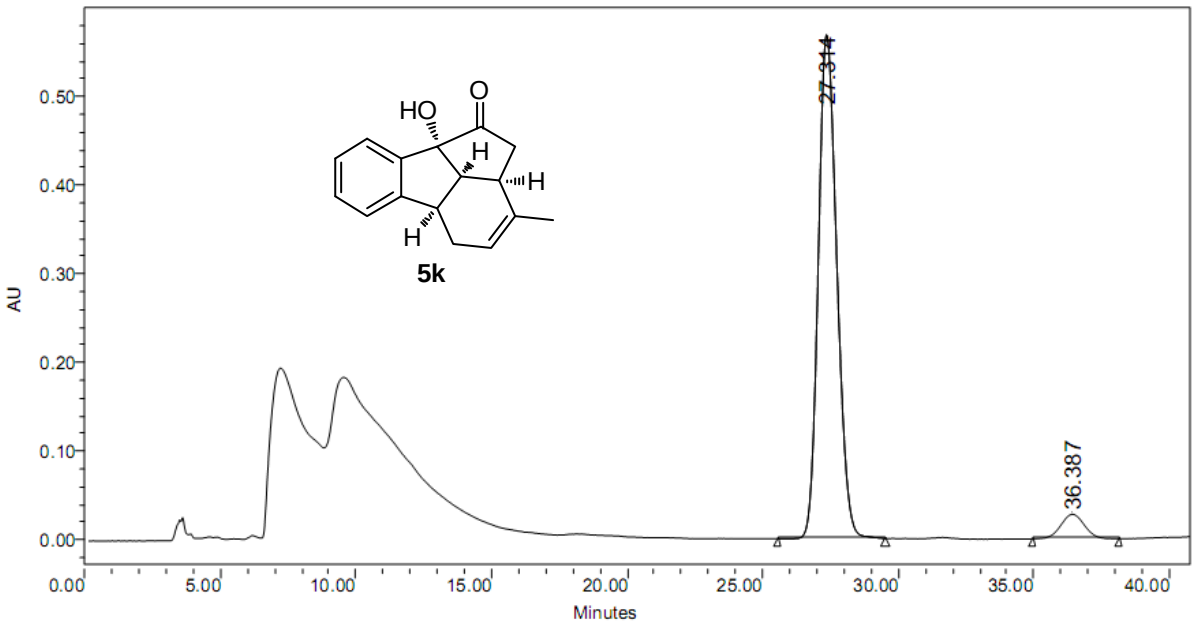
#	RT [min]	Width [min]	Area mAU *S	Height [mAU]	Area %
1	7.502 VV	0.2121	1879.75964	130.19792	7.4487
2	9.385 VV	0.2513	2.33562e4	1411.42517	92.5513


HO
O
H
H
H
5k
f1 (ppm)

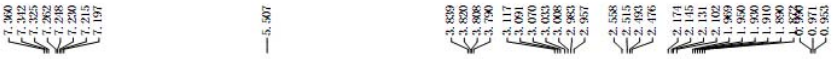

HO
O
H
H
H
5k
ppm

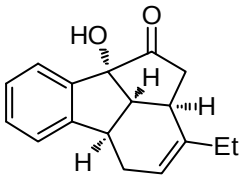


	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	27.257	11269354	50.32	234335	56.17
2	36.243	11124401	49.68	182822	43.83

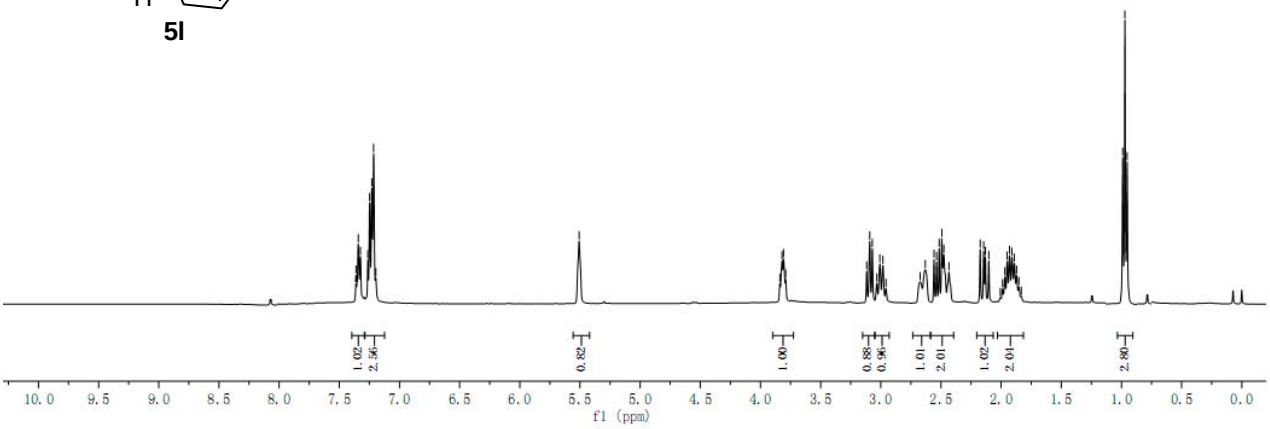


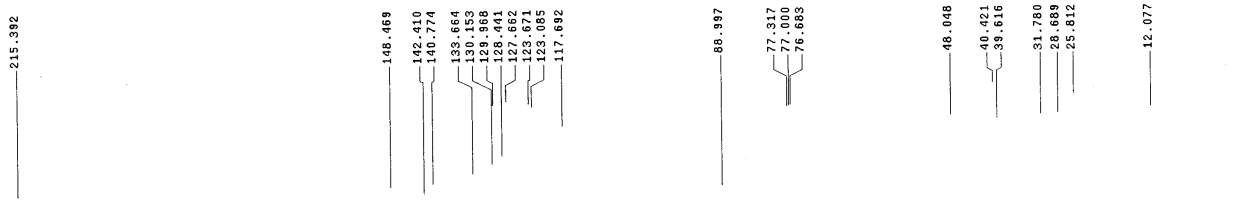
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	27.314	27013043	94.21	567977	95.36
2	36.387	1658723	5.79	27627	4.64

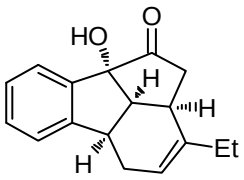




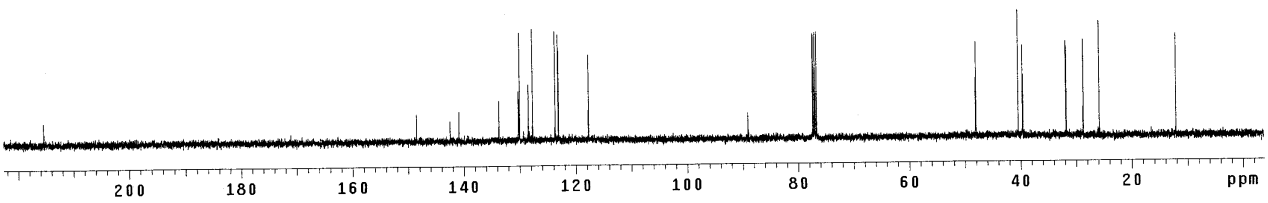
5l

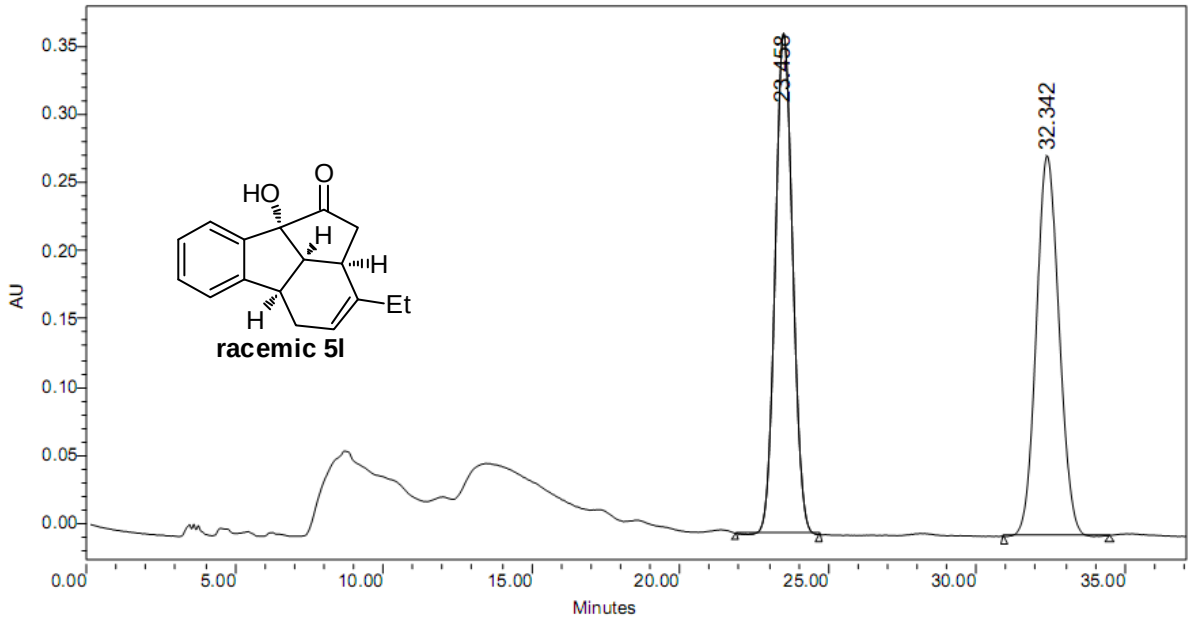




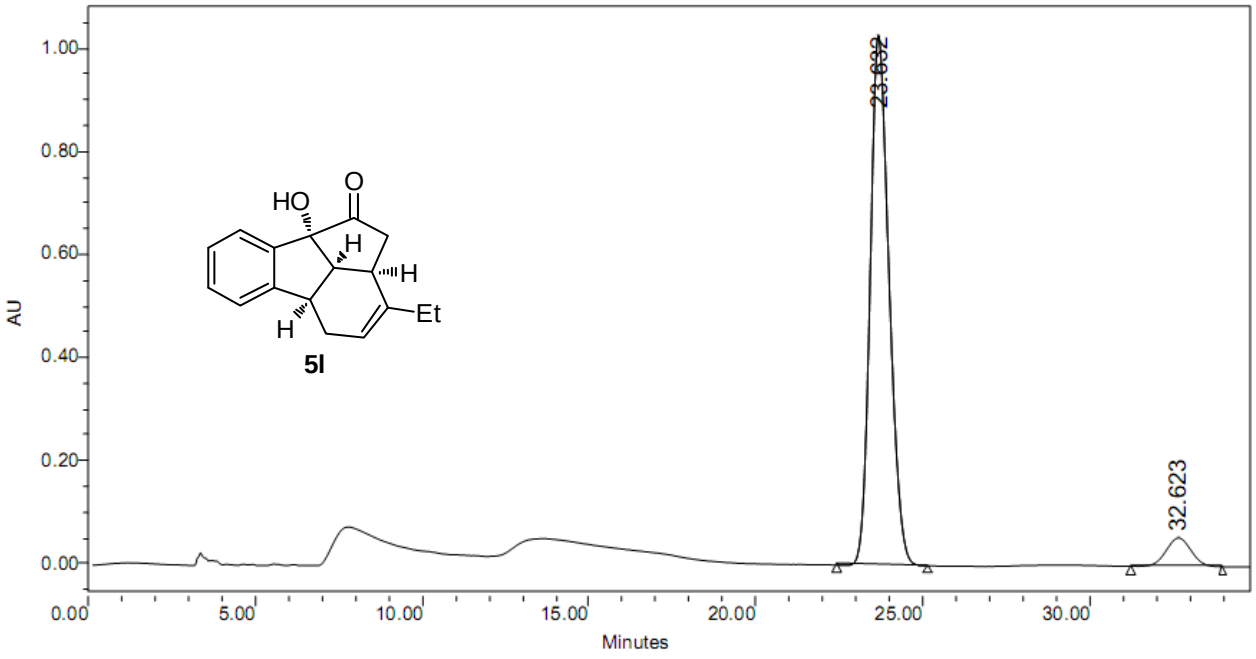


5l

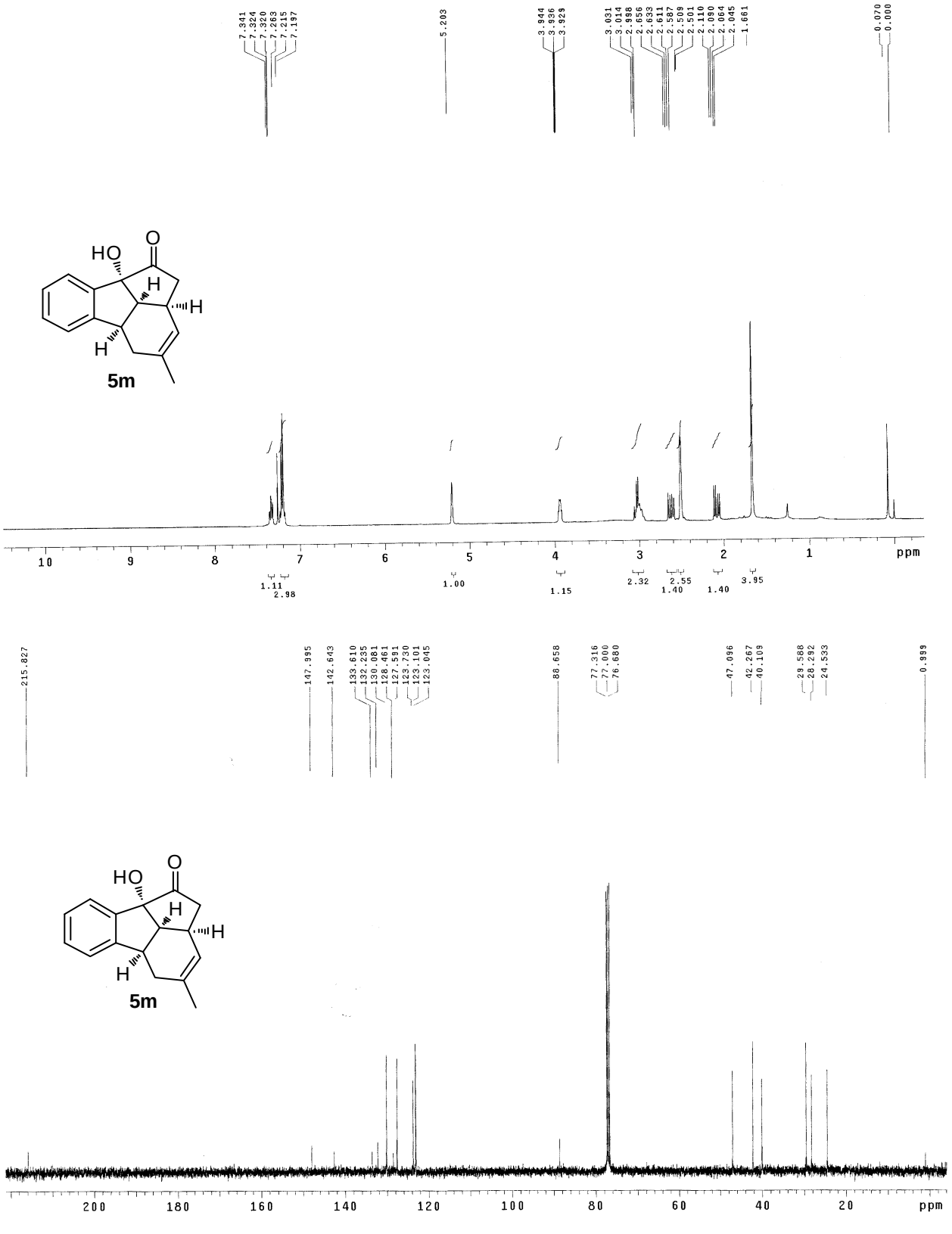


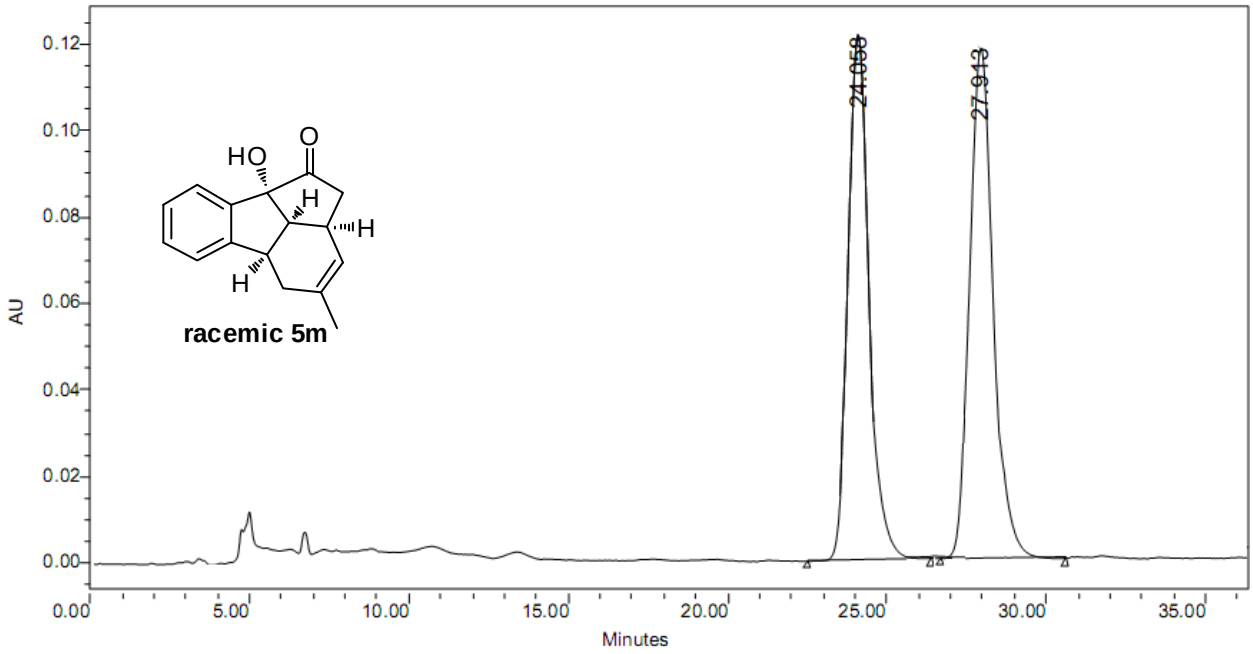


	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	23.458	15070231	50.24	367251	56.84
2	32.342	14926896	49.76	278870	43.16

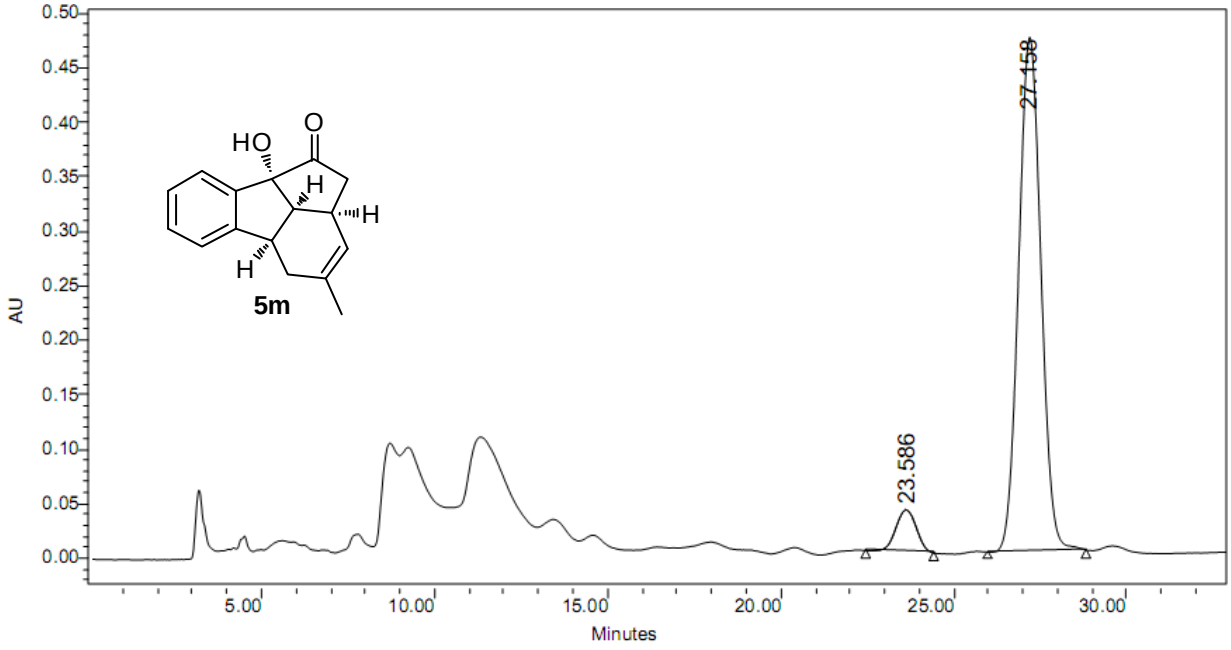


	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	23.632	40376449	93.30	1028975	94.88
2	32.623	2900159	6.70	55488	5.12

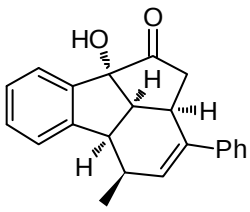

7.341
7.324
7.320
7.263
7.215
7.197
5.203
3.944
3.936
3.929
3.031
3.014
2.998
2.656
2.633
2.611
2.587
2.509
2.501
2.140
2.090
2.064
2.045
1.661
0.070
0.000
HO
O
H
H
H
5m
10
9
8
7
6
5
4
3
2
1
ppm
1.11
2.98
1.00
1.15
2.32
2.55
1.40
1.40
3.95
215.827
147.995
142.643
133.610
132.235
130.081
128.461
127.591
123.730
123.101
123.045
88.658
77.316
77.000
76.680
47.096
42.267
40.109
29.588
28.292
24.533
0.939
HO
O
H
H
H
5m
200
180
160
140
120
100
80
60
40
20
ppm



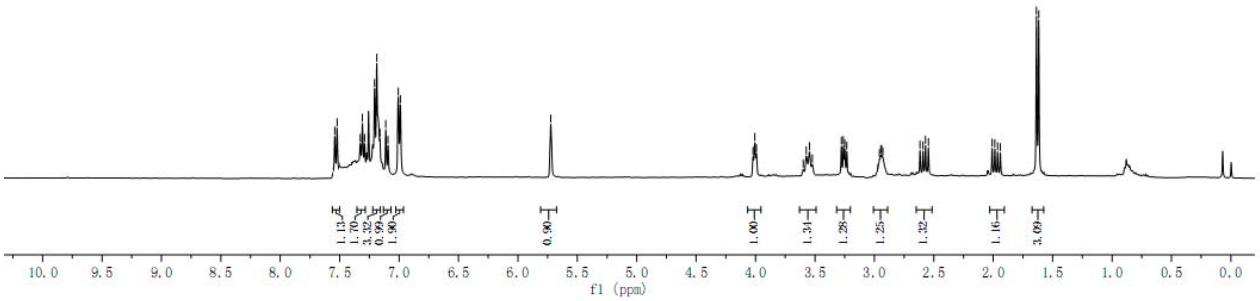
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	24.058	4836043	48.95	115284	51.39
2	27.913	5042859	51.05	109068	48.61

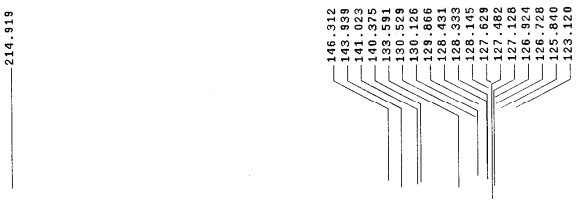


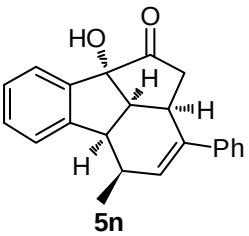
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	23.586	1623938	7.14	38935	7.62
2	27.158	21106862	92.86	471772	92.38


HO
O
H
H
H
Ph

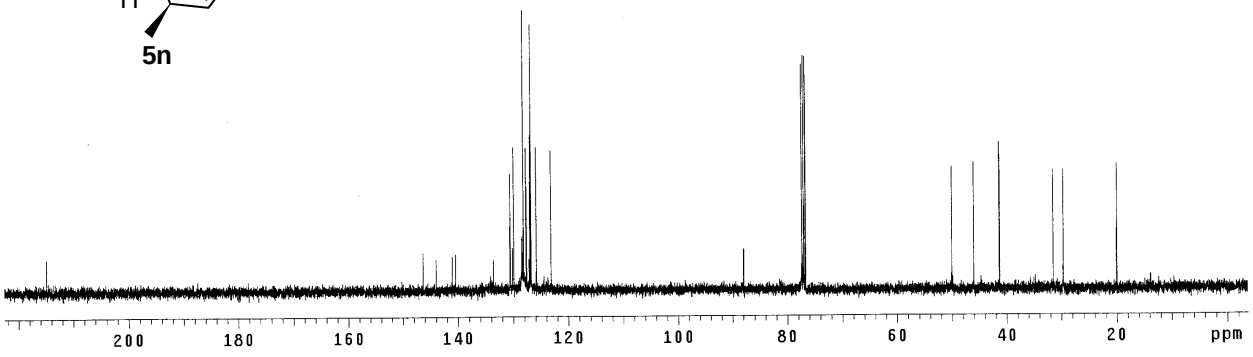
5n

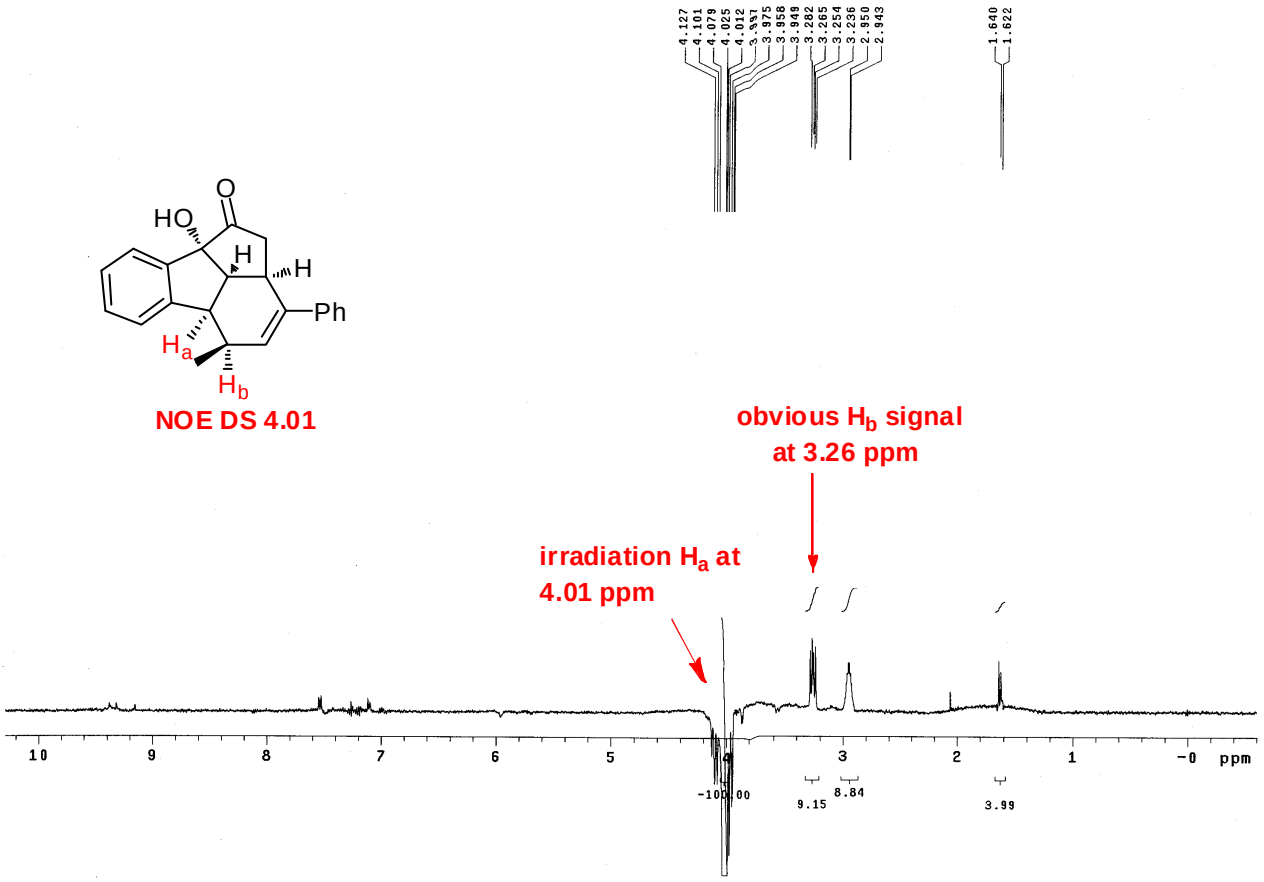

1.13
1.70
3.32
0.99
1.90
0.90
1.00
1.31
1.28
1.25
1.32
1.16
3.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

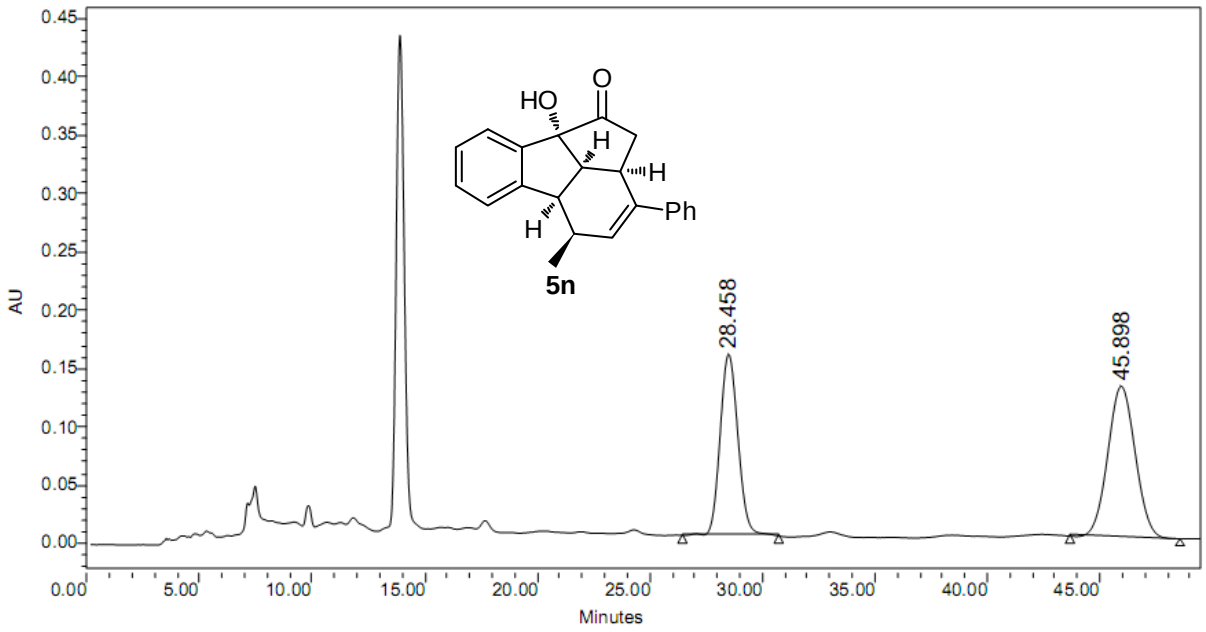



HO
O
H
H
H
Ph

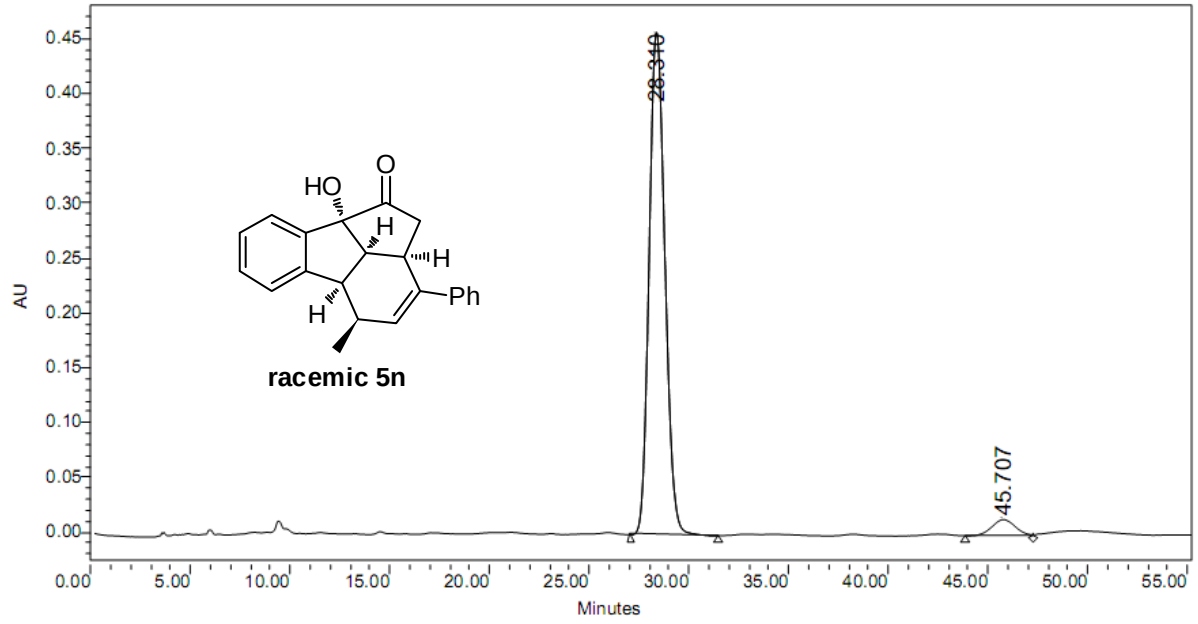
5n


200
180
160
140
120
100
80
60
40
20
ppm

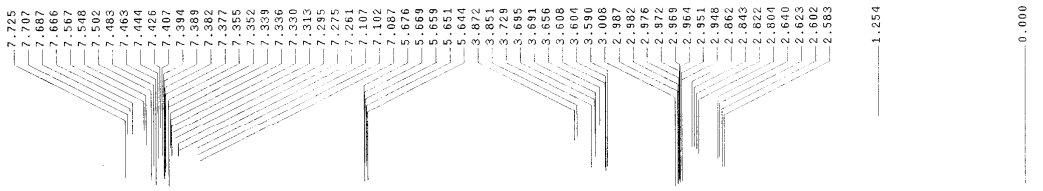

4.127
4.101
4.079
4.025
4.012
3.975
3.958
3.949
3.282
3.265
3.254
3.236
2.950
2.943
1.640
1.622
O
HO
H
H
Ph
Ha
Hb
NOE DS 4.01
obvious Hb signal
at 3.26 ppm
irradiation Ha at
4.01 ppm
10
9
8
7
6
5
4
3
2
1
-0
ppm
9.15
8.84
3.99

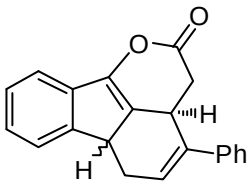


	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	28.458	8683467	44.01	155563	54.52
2	45.898	11046759	55.99	129780	45.48

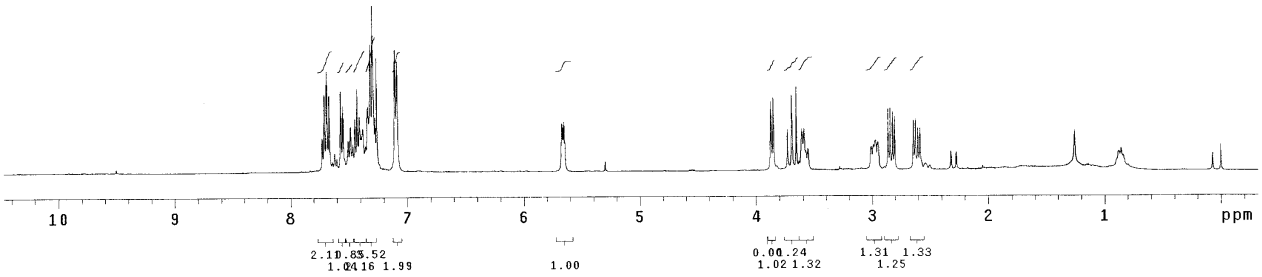


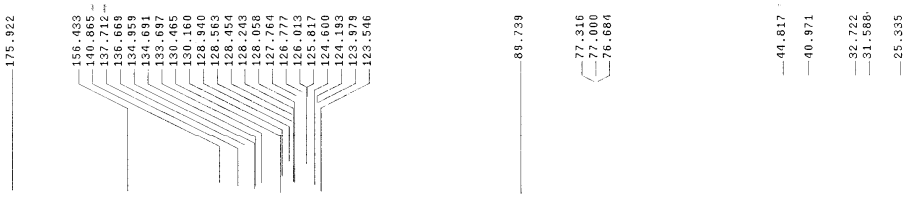
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	28.310	25249305	95.35	458275	96.86
2	45.707	1230675	4.65	14848	3.14

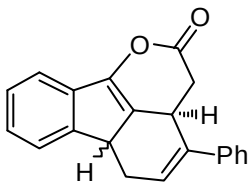

7.725
7.707
7.687
7.666
7.567
7.548
7.502
7.483
7.463
7.444
7.426
7.407
7.394
7.389
7.382
7.377
7.355
7.352
7.339
7.336
7.330
7.313
7.295
7.275
7.261
7.107
7.102
7.087
5.676
5.669
5.659
5.651
5.644
3.872
3.851
3.729
3.695
3.691
3.656
3.608
3.604
3.590
3.008
2.987
2.982
2.976
2.972
2.969
2.964
2.951
2.948
2.862
2.843
2.822
2.804
2.640
2.623
2.602
2.583
1.254
0.000


O
O
H
H
Ph

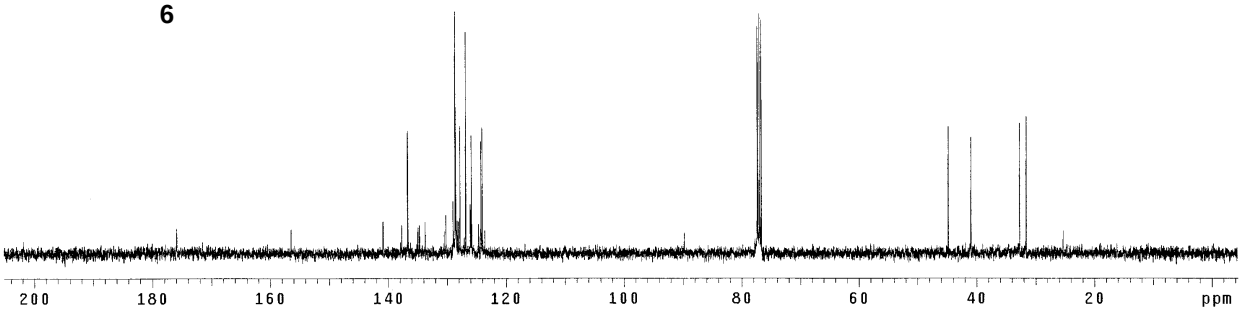
6

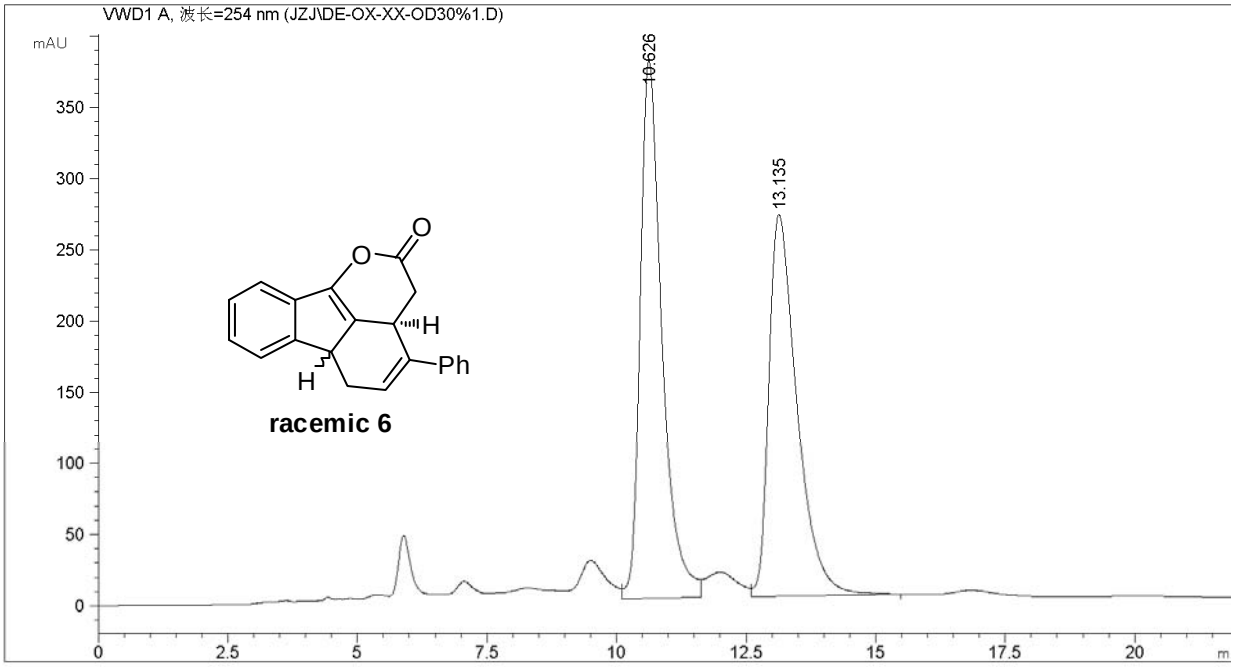

10
9
8
7
6
5
4
3
2
1
ppm
2.10 0.85 3.52
1.00
0.00 0.24
1.02 1.32
1.31 1.33
1.25


175.922
156.433
140.865
137.712
136.669
134.959
134.691
133.697
130.465
130.160
128.940
128.563
128.454
128.243
128.058
127.764
126.777
126.013
125.817
124.600
124.193
123.979
123.546
89.739
77.316
77.000
76.684
44.817
40.971
32.722
31.588
25.335

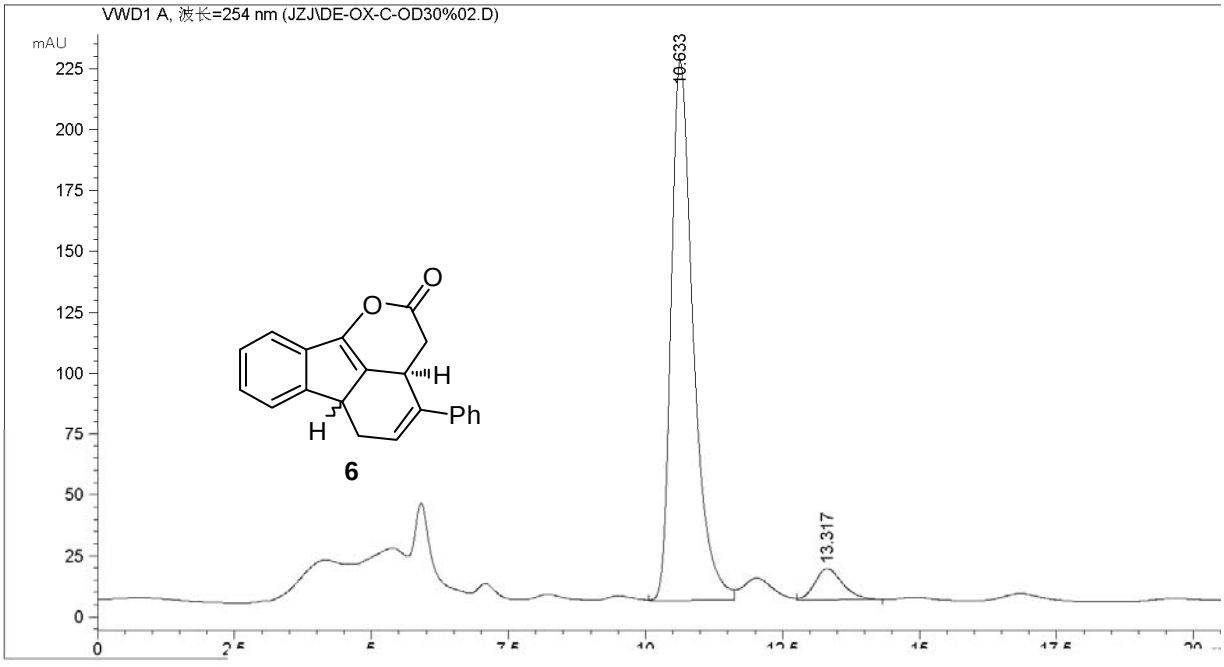

O
O
H
H
Ph

6

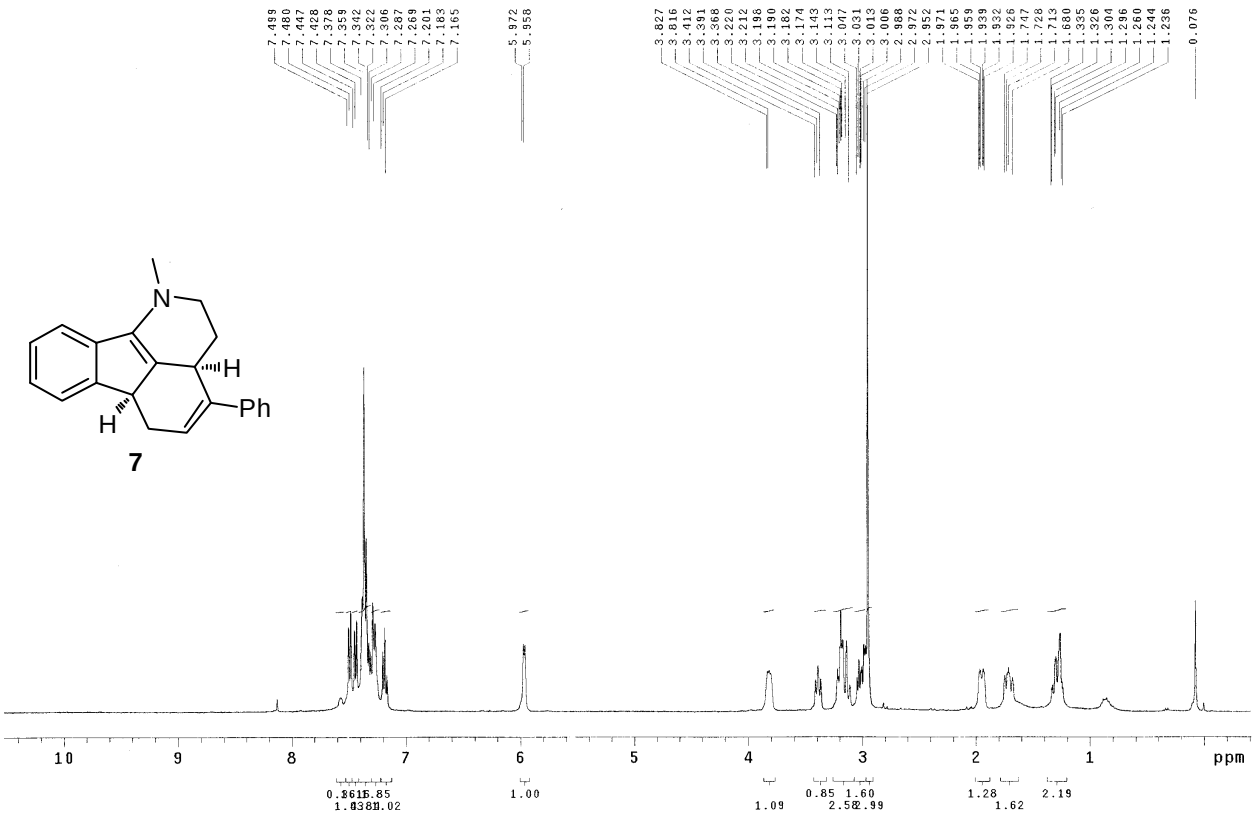

200
180
160
140
120
100
80
60
40
20
ppm

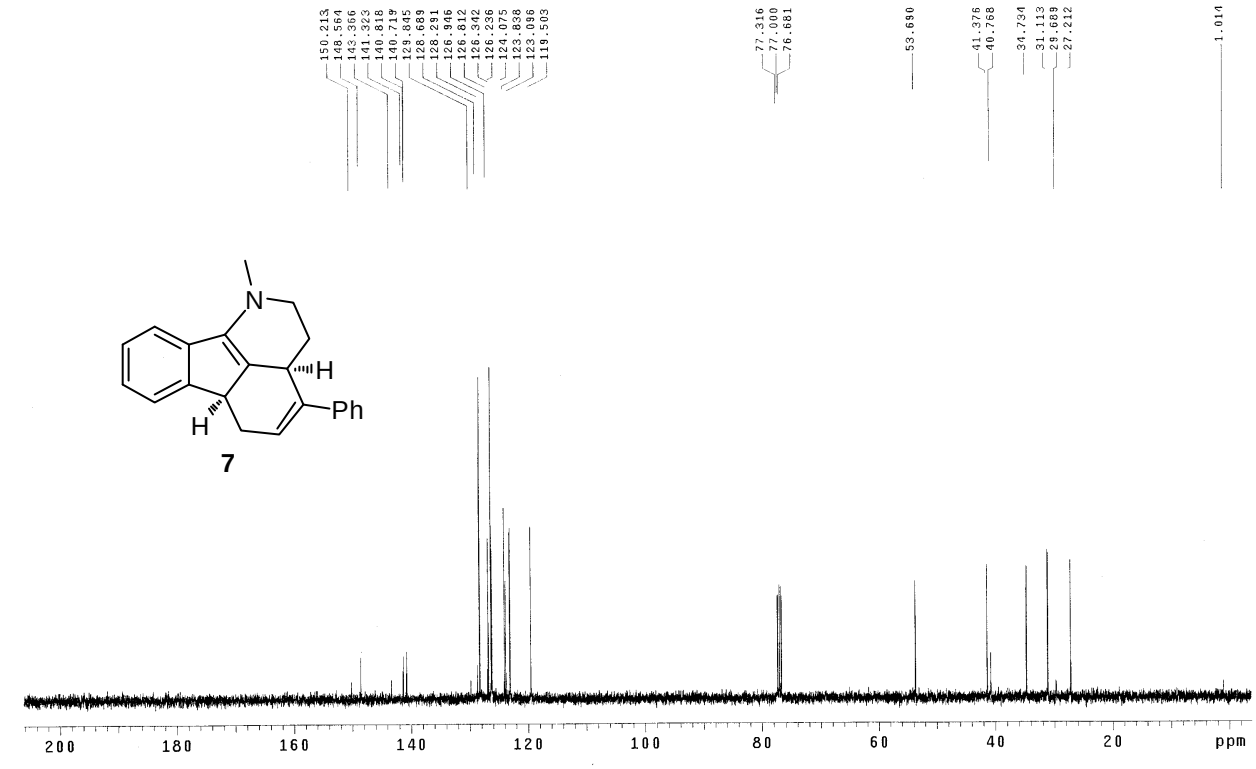


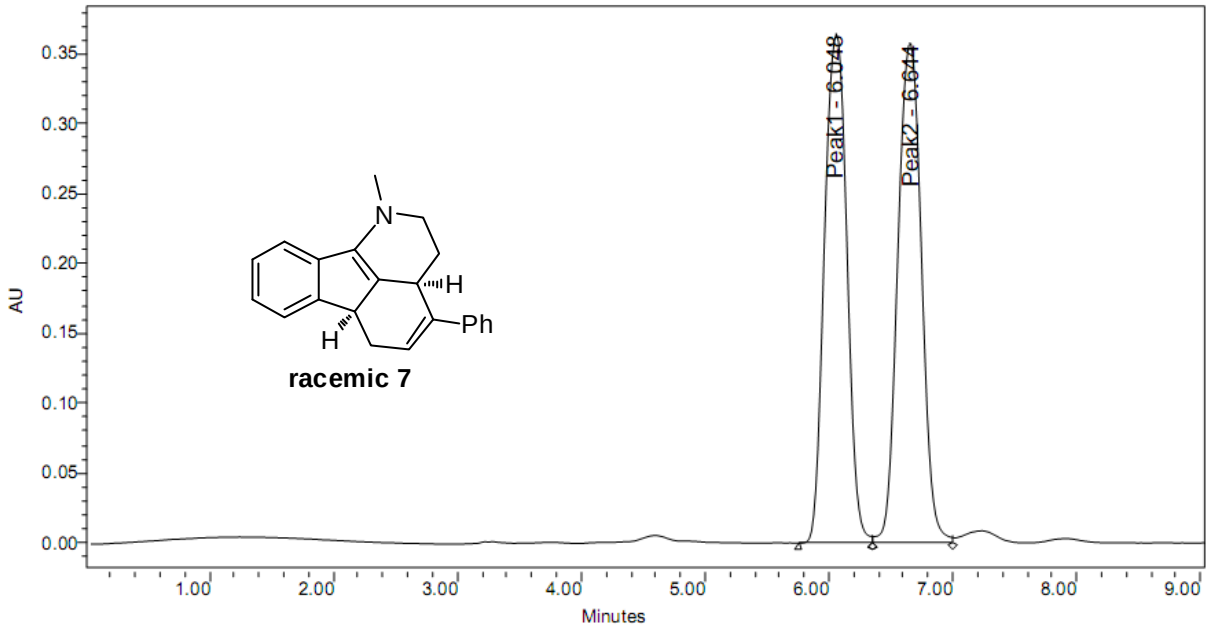
#	RT [min]		Width [min]	Area mAU *S	Height [mAU]	Area %
1	10.626	VV	0.4388	1.10340e4	377.28128	52.0812
2	13.135	VB	0.5688	1.01521e4	267.91684	47.9188



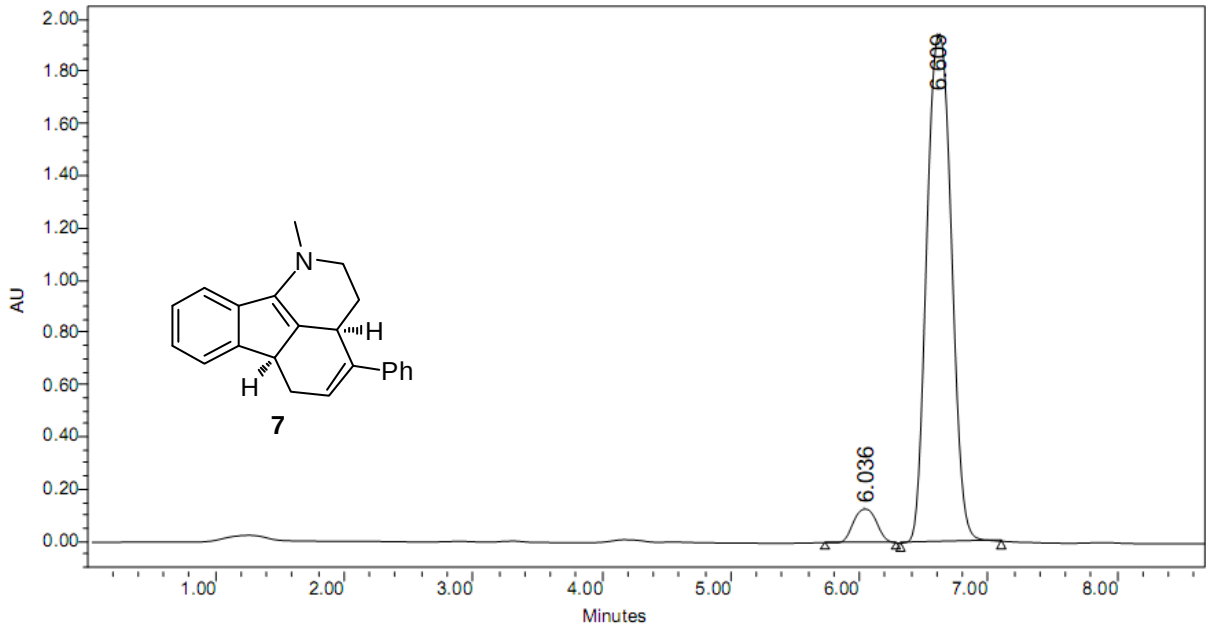
#	RT [min]		Width [min]	Area mAU *S	Height [mAU]	Area %
1	10.633	VV	0.4234	6213.39502	221.48660	93.2852
2	13.317	VB	0.5376	447.24606	12.69884	6.7148


7.499
7.480
7.447
7.428
7.378
7.359
7.342
7.322
7.306
7.287
7.269
7.201
7.183
7.165
5.972
5.958
3.827
3.816
3.412
3.391
3.368
3.220
3.212
3.196
3.190
3.182
3.174
3.143
3.113
3.047
3.031
3.013
3.006
2.988
2.972
2.952
1.971
1.965
1.959
1.939
1.932
1.926
1.747
1.728
1.713
1.680
1.335
1.326
1.304
1.296
1.260
1.244
1.236
0.076
N
H
H
Ph
7
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.09
0.85
1.60
1.28
1.62
2.19


150.213
148.564
143.366
141.323
140.818
140.719
129.845
128.689
128.291
126.946
126.812
126.342
126.236
124.075
123.838
123.096
119.503
77.316
77.000
76.681
53.690
41.376
40.768
34.734
31.113
29.689
27.212
1.014
N
H
H
Ph
7
200
180
160
140
120
100
80
60
40
20
ppm



	Peak Name	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	Peak1	6.048	4592355	49.11	364485	50.43
2	Peak2	6.644	4758572	50.89	358313	49.57



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	6.036	1636876	5.93	129901	6.24
2	6.609	25986140	94.07	1953187	93.76