

**N-Heterocyclic Carbene-Catalyzed Cyclocondensation of 2-Aryl
Carboxylic Acids and Enones: Highly Enantioselective Synthesis of
 δ -Lactones**

Jin-Tang Cheng, Xiang-Yu Chen and Song Ye*

Beijing National Laboratory for Molecular Sciences, CAS Key Laboratory of
Molecular Recognition and Function, Institute of Chemistry, Chinese Academy of
Sciences, Beijing 100190, China.

E-mail: songye@iccas.ac.cn

Part I General Information	2
Part II Experimental part.....	3
1. NHC-catalyzed [4 + 2] cyclocondensation of 2-aryl carboxylic acids and enones (Table 2).....	3
2. X-Ray structure of 3aa (Figure S1).....	17
3. References	17
Part III NMR Spectra	18
Part IV HPLC Spectra.....	35

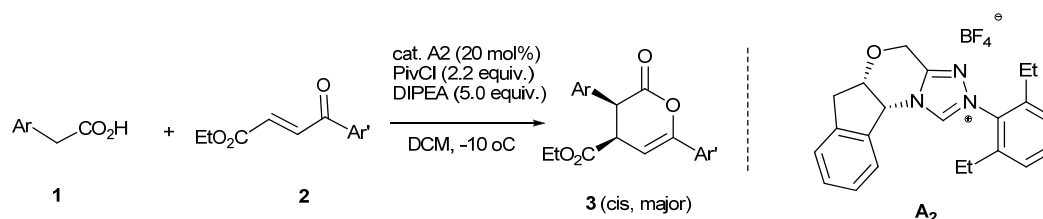
Part I General Information

Unless otherwise indicated, all reactions were carried out under N₂ at -10 °C with magnetic stirring. Anhydrous THF and toluene were distilled from sodium and benzophenone. Anhydrous CH₂Cl₂ was distilled from CaH₂. Chiral triazolium salts **A1**,¹ **A2**,² **B1**,³ **B2**,⁴ enones⁵ were synthesized according to literatures. Column chromatography was performed on silica gel 200~300 mesh. All ¹H NMR, ¹³C NMR spectra were recorded on a Bruker-DMX 300 spectrometer or Bruker-DMX 400 spectrometer in CDCl₃, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ¹H NMR Spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were recorded on a JASCO FT/IR-480 spectrophotometer and reported as wave number (cm⁻¹). Optical rotations were measured on Perkin Elmer/Model-343 digital polarimeter operating at the sodium D line with a 100 mm path cell, and are reported as follows: $[\alpha]_D^T$ (concentration (g/100 mL), solvent).

Part II Experimental part

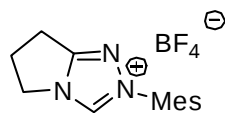
1. NHC-catalyzed [4+2] cyclocondensation of 2-aryl carboxylic acids and enones

(Table 2)

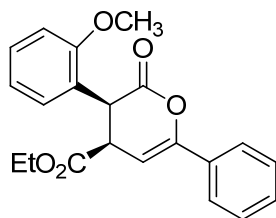


To a solution of 2-arylacetic acids **1** (0.44 mmol, 2.2 equiv) in CH₂Cl₂ (2.0 ml) was added DIPEA (0.6 mmol, 77.4 mg, 3.0 equiv.) and pivaloyl chloride (0.44 mmol, 53 mg, 2.2 equiv.) at -10 °C. After stirring at -10 °C for 30 minutes, to the reaction mixture was added NHC precursor **A2** (0.04 mmol, 17 mg, 0.2 equiv.), enones **2** (0.2 mmol) and another 2.0 equivalent of DIPEA (0.4 mmol, 51.6 mg). The reaction mixture was stirred at -10 °C until the full consumption of the enone (typically, 24-48 h). The reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent, typically 10:1-5:1) to furnish the corresponding cycloadduct **3**.

Racemic samples for the chiral phase HPLC analysis were prepared using NHC precursor **C** under the same conditions but at room temperature with longer reaction time (typically 2-3 d). It should be noted that sometimes the *trans*-isomers of cycloadduct **3** were isolated as majority (*cis/trans* = 20:1 to 1:8) for the reaction possibly due to the epimerization of *cis*-**3** to *trans*-**3** at room temperature with longer reaction time.



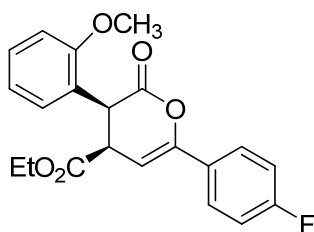
C



3aa

(3*S*,4*R*)-ethyl 3-(2-methoxyphenyl)-2-oxo-6-phenyl-3,4-dihydro-2H-pyran-4-carboxylate

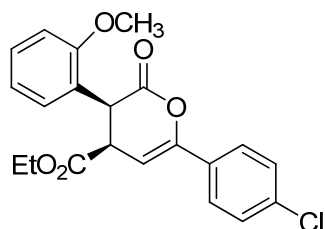
54.3 mg, 77% yield, *cis/trans* = 10:1, white solid, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -148.4 (*c* 0.2, CH₂Cl₂), HPLC analysis: >99% ee (*cis*), 79% ee (*trans*) [Daicel CHIRALPAK OD-3 column, 20 °C, 254 nm hexane/*i*-PrOH = 90:10, 1.0 mL/min, 254 nm, 13.4 min (*trans*-minor), 16.2 min (*trans*-major), 18.0 min(*cis*-major), 19.9 min (*cis*-minor)]; ¹H NMR (300 MHz, CDCl₃) δ 7.72-7.68 (m, 2H), 7.57 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.44 – 7.39 (m, 3H), 7.32 – 7.29 (m, 1H), 7.00 – 6.88 (m, 2H), 5.99 (d, *J* = 6.8 Hz, 1H), 4.69 (d, *J* = 6.2 Hz, 1H), 4.06 (q, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 3.67 (t, *J* = 6.5 Hz, 1H), 1.15 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 170.6, 167.6, 156.9, 152.6, 132.1, 130.2, 129.6, 129.2, 128.6, 125.0, 122.3, 120.7, 110.4, 99.0, 61.5, 55.6, 44.0, 39.3, 14.0. IR (KBr) 2919, 2849, 1775, 1732, 1646, 1600, 1495, 1180, 754; HRMS (ESI) calcd for C₂₁H₂₀O₅Na [M+Na]⁺ 375.1203, found 375.1197.



3ab

(3S,4R)-ethyl 6-(4-fluorophenyl)-3-(2-methoxyphenyl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

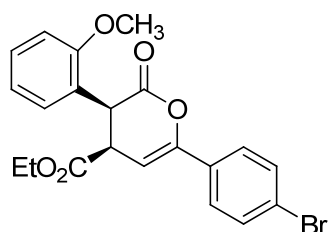
49.9 mg, 67% yield; *cis/trans* = 10:1, colorless oil, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -148.7 (*c* 0.2, CH_2Cl_2), HPLC analysis: 99% ee (*cis*), 62% ee (*trans*) [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH/MeOH = 80:10:10, 1.0 mL /min, 254 nm, 9.5 min (*trans*-major), 10.9 min (*trans*-minor), 12.0 min (*cis*-major)]; ^1H NMR (300 MHz, CDCl_3) δ 7.71-7.66 (m, 2H), 7.56 (dd, J = 7.7, 1.5 Hz, 1H), 7.32 – 7.26 (m, 1H), 7.11-7.06 (m, 2H), 6.99 – 6.88 (m, 2H), 5.92 (d, J = 6.8 Hz, 1H), 4.68 (d, J = 6.2 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 3.84 (s, 3H), 3.66 (t, J = 6.5 Hz, 1H), 1.14 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.5, 166.8, 156.9, 151.2, 130.1, 129.2, 127.0, 126.9, 122.1, 120.6, 115.7, 115.4, 110.3, 98.6, 61.4, 55.5, 43.9, 39.2, 13.9. IR (KBr) 2921, 2848, 1733, 1633, 1509, 1246, 1180; HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{19}\text{O}_5\text{FNa}$ $[\text{M}+\text{Na}]^+$ 393.1109, found 393.1104.



3ac

(3S,4R)-ethyl 6-(4-chlorophenyl)-3-(2-methoxyphenyl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

46.8 mg, 61% yield; *cis/trans* = 10:1, colorless oil, R_f = 0.3 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -184.1 (*c* 0.2, CH₂Cl₂), HPLC analysis: 99% ee (*cis*), 71% ee (*trans*) [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm, hexane/*i*-PrOH/MeOH = 80:10:10, 1.0 mL /min, 254 nm, 9.9 min (*trans*-major), 11.0 min (*trans*-minor), 13.6 min (*cis*-major), 32.6 min (*cis*-minor)]; ¹H NMR (300 MHz, CDCl₃) δ 7.65-7.60 (m, 2H), 7.55 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.39 – 7.36 (m, 2H), 7.32 – 7.27 (m, 1H), 6.99 – 6.88 (m, 2H), 5.97 (d, *J* = 6.8 Hz, 1H), 4.68 (d, *J* = 6.1 Hz, 1H), 4.06 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 3.67 (t, *J* = 6.5 Hz, 1H), 1.15 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 170.2, 166.8, 156.5, 151.2, 135.3, 130.5, 130.0, 129.2, 128.8, 126.2, 122.0, 120.6, 110.2, 99.3, 61.6, 55.5, 43.9, 39.1, 13.9. IR (KBr) 2922, 2850, 1779, 1732, 1647, 1602, 1494, 1180, 754; HRMS (ESI) calcd for C₂₁H₁₉O₅ClNa [M+Na]⁺ 409.0813, found 409.0807.

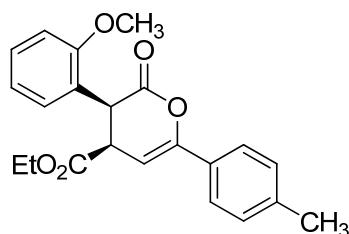


3ad

(3*S*,4*R*)-ethyl 6-(4-bromophenyl)-3-(2-methoxyphenyl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

47.7 mg, 55% yield; *cis/trans* = 7:1, colorless oil, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -17.4 (*c* 0.5, CH₂Cl₂), HPLC analysis: >99% ee (*cis*), 75% ee (*trans*) [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH/MeOH = 80:10:10, 1.0 mL /min, 254 nm, 10.8 min (*trans*-major), 11.8 min (*trans*-minor), 15.2

min (*cis*-major)]; ^1H NMR (300 MHz, CDCl_3) δ 7.59-7.51 (m, 5H), 7.32 – 7.26 (m, 1H), 6.99 – 6.88 (m, 2H), 5.99 (d, J = 6.8 Hz, 1H), 4.68 (d, J = 6.2 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 3.84 (s, 3H), 3.66 (t, J = 6.5 Hz, 1H), 1.14 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.2, 166.8, 156.9, 151.6, 131.7, 131.1, 130.1, 129.2, 126.4, 123.6, 121.9, 120.6, 110.3, 99.3, 61.5, 55.5, 43.9, 39.2, 13.8. IR (KBr) 2928, 1776, 1732, 1646, 1489, 1248, 1176, 754; HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{19}\text{O}_5\text{BrNa}$ $[\text{M}+\text{Na}]^+$ 453.0308, found 453.0300.

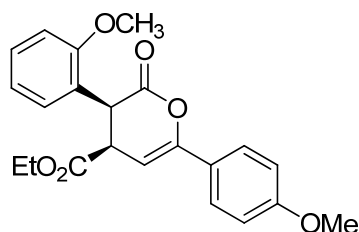


3ae

(3*S*,4*R*)-ethyl 3-(2-methoxyphenyl)-2-oxo-6-(*p*-tolyl)-3,4-dihydro-2H-pyran-4-carboxylate

40.5 mg, 55% yield; *cis/trans* = 6:1, colorless oil, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -8.0 (c 0.1, CH_2Cl_2), HPLC analysis: >99% ee (*cis*), 78% ee (*trans*) [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH /MeOH = 90:5:5, 1.0 mL /min, 254 nm, 13.2 min (*trans*-major), 14.7 min (*trans*-minor), 19.2 min (*cis*-major)]; ^1H NMR (300 MHz, CDCl_3) δ 7.60-7.55 (m, 3H), 7.31 – 7.19 (m, 3H), 6.99 – 6.88 (m, 2H), 5.93 (d, J = 6.8 Hz, 1H), 4.68 (d, J = 6.2 Hz, 1H), 4.05 (q, J = 7.1 Hz, 2H), 3.84 (s, 3H), 3.65 (t, J = 6.5 Hz, 1H), 2.38 (s, 3H), 1.15 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.5, 167.2, 156.5, 152.3, 139.3, 129.9, 129.0, 124.6, 122.0, 120.6, 110.4, 98.0, 61.5, 55.5, 43.9, 39.3, 21.3, 14.0. IR (KBr) 2920, 2849, 1733, 1651, 1606, 1495, 1180, 755; HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{22}\text{O}_5\text{Na}$

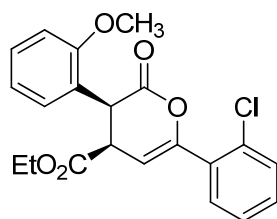
$[M+Na]^+$ 389.1359, found 389.1355.



3af

(3S,4R)-ethyl 3-(2-methoxyphenyl)-6-(4-methoxyphenyl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

61.1 mg, 80% yield; *cis/trans* = 10:1, colorless oil, R_f = 0.2 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -1.2 (c 0.3, CH_2Cl_2), HPLC analysis: >99% ee (*cis*), 30% ee (*trans*) [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/ *i*-PrOH/MeOH = 80:10:10, 1.0 mL /min, 254 nm, 13.0 min (*trans*-major), 15.0 min (*trans*-minor), 17.4 min (*cis*-major)]; 1H NMR (300 MHz, $CDCl_3$) δ 7.65-7.62 (m, 2H), 7.57 (dd, J = 7.7, 1.2 Hz, 1H), 7.31 – 7.26 (m, 1H), 6.99 – 6.87 (m, 4H), 5.85 (d, J = 6.8 Hz, 1H), 4.67 (d, J = 6.1 Hz, 1H), 4.05 (q, J = 7.1 Hz, 2H), 3.84 (s, 6H), 3.64 (t, J = 6.5 Hz, 1H), 1.15 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 167.2, 160.5, 156.5, 152.2, 129.9, 128.6, 126.3, 124.3, 122.3, 120.3, 113.7, 110.1, 96.7, 61.2, 55.5, 55.2, 43.6, 38.9, 13.6. IR (KBr) 2924, 2850, 1733, 1601, 1495, 1260, 1172, 757; HRMS (ESI) calcd for $C_{22}H_{22}O_6Na$ $[M+Na]^+$ 405.1309, found 405.1304.

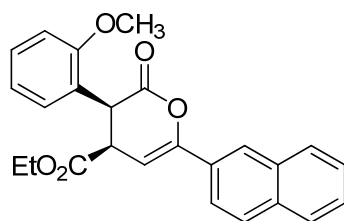


3ag

(3S,4R)-ethyl 6-(2-chlorophenyl)-3-(2-methoxyphenyl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

ran-4-carboxylate

48.9 mg, 63% yield; *cis/trans* = 6:1, colorless oil, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -65.4 (c 0.5, CH_2Cl_2), HPLC analysis: 98% ee (*cis*), 72% ee (*trans*) [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/ *i*-PrOH /MeOH = 90:5:5, 1.0 mL /min, 254 nm, 12.3 min (*trans*-major), 14.2 min (*cis*-major), 15.8 min (*trans*-minor), 18.0 min (*cis*-minor)]; ^1H NMR (300 MHz, CDCl_3) δ 7.60-7.53 (m, 2H), 7.45-7.42 (m, 1H), 7.35 – 7.26 (m, 3H), 7.00 – 6.89 (m, 2H), 5.88 (d, J = 6.7 Hz, 1H), 4.73 (d, J = 6.2 Hz, 1H), 4.09 (q, J = 7.1 Hz, 2H), 3.87 (s, 3H), 3.68 (t, J = 6.5 Hz, 1H), 1.18 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) 170.2, 167.2, 156.9, 150.5, 132.6, 131.9, 130.5, 130.4, 130.3, 130.1, 129.1, 126.8, 122.1, 120.6, 110.2, 104.9, 61.2, 55.2, 43.9, 38.9, 13.6. IR (KBr) 2923, 2849, 1732, 1673, 1600, 1495, 1260, 1173, 755; HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{19}\text{O}_5\text{ClNa}$ $[\text{M}+\text{Na}]^+$ 409.0813, found 409.0807.

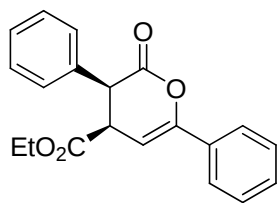
**3ah**

(3*S*,4*R*)-ethyl 3-(2-methoxyphenyl)-6-(naphthalen-2-yl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

ran-4-carboxylate

26.1 mg, 33% yield; *cis/trans* = 3:1, white solid, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -170.9 (c 0.1, CH_2Cl_2), HPLC analysis: >99% ee (*cis*), 88% ee (*trans*) [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL /min, 254 nm, 11.3 min (*trans*-major), 14.0 min (*trans*-minor), 17.9 min

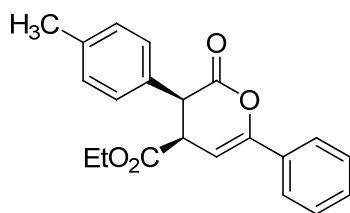
(*cis*-major)]; ^1H NMR (300 MHz, CDCl_3) δ 8.21-8.18 (m, 1H), 7.84-7.77 (m, 4H), 7.65-7.62 (m, 1H), 7.53 (d, $J = 7.8$ Hz, 1H), 7.46-7.43 (m, 2H), 7.25-7.19 (m, 1H), 6.93-6.83 (m, 2H), 6.07 (d, $J = 6.8$ Hz, 1H), 4.67 (d, $J = 6.2$ Hz, 1H), 4.08 (q, $J = 7.1$ Hz, 2H), 3.85 (s, 3H), 3.73 (t, $J = 6.4$ Hz, 1H), 1.17 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.2, 167.2, 156.5, 152.3, 133.6, 133.1, 130.1, 129.2, 129.0, 128.8, 128.3, 127.6, 126.9, 126.7, 124.7, 122.0, 120.6, 109.6, 99.4, 61.1, 55.2, 43.9, 38.9, 13.6. IR (KBr) 2920, 2849, 1732, 1722, 1180, 1129, 753; HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{22}\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 425.1359, found 425.1354.



3ba

(3*S*,4*R*)-ethyl 2-oxo-3,6-diphenyl-3,4-dihydro-2H-pyran-4-carboxylate

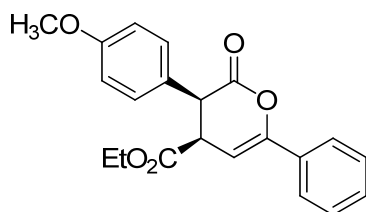
56.6 mg, 88% yield; *cis/trans* = 1:1, white solid, $R_f = 0.4$ (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25} -106.7$ (c 0.1, CH_2Cl_2), ^1H NMR (300 MHz, CDCl_3) δ 7.65-7.62 (m, 2H), 7.37-7.33 (m, 3H), 7.30-7.22 (m, 5H), 6.02 (d, $J = 5.3$ Hz, 1H), 4.17 (d, $J = 6.3$ Hz, 1H), 4.02 (q, $J = 7.1$ Hz, 2H), 3.77-3.74 (m, 1H), 1.07 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 171.0, 167.6, 151.5, 135.6, 131.8, 129.8, 129.1, 128.8, 128.7, 128.2, 125.0, 97.5, 62.0, 46.9, 45.4, 14.1. IR (KBr) 2920, 2849, 1774, 1732, 1181, 1130, 754; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{18}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 345.1079, found 345.1093.



3ca

(3S,4R)-ethyl 2-oxo-6-phenyl-3-(p-tolyl)-3,4-dihydro-2H-pyran-4-carboxylate

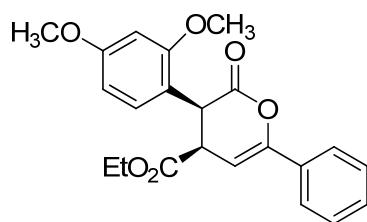
48.6 mg, 72% yield; *cis/trans* = 6:1, white solid, R_f = 0.3 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -145.9 (*c* 0.1, CH_2Cl_2), HPLC analysis: 98% ee (*cis*), 48% ee (*trans*) [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 17.3 min (*cis*-minor), 18.6 min (*trans*-major), 22.1 min (*cis*-major), 24.3 min (*trans*-minor)]; ^1H NMR (300 MHz, CDCl_3) δ 7.64-7.59 (m, 2H), 7.35-7.31 (m, 3H), 7.18-7.14 (m, 2H), 7.06 (t, J = 7.3 Hz, 2H), 6.00 (d, J = 5.3 Hz, 1H), 4.12 (d, J = 6.3 Hz, 1H), 4.02 (q, J = 7.1 Hz, 2H), 3.73 (t, J = 5.8 Hz, 1H), 2.24 (s, 3H), 1.08 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 169.9, 167.3, 151.9, 138.0, 131.8, 130.9, 129.6, 129.5, 129.4, 128.8, 128.6, 127.9, 124.9, 98.5, 61.4, 46.3, 44.3, 21.0, 13.8. IR (KBr) 2919, 2849, 1769, 1732, 1719, 1632, 1180, 1131, 755; HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{20}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 359.1254, found 359.1249.



3da

(3S,4R)-ethyl 3-(4-methoxyphenyl)-2-oxo-6-phenyl-3,4-dihydro-2H-pyran-4-carboxylate

45.5 mg, 58% yield; *cis/trans* = 3:1, white solid, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -161.0 (*c* 0.1, CH₂Cl₂), HPLC analysis: >99% ee (*cis*), 48% ee (*trans*) [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL /min, 254 nm, 14.5 min (*trans*-major), 18.8 min (*trans*-minor), 20.8 min (*cis*-major)]; ¹H NMR (400 MHz, CDCl₃) δ 7.64-7.58 (m, 2H), 7.36-7.33 (m, 4H), 7.21-7.19 (m, 1H), 6.81-6.77 (m, 2H), 6.00 (d, *J* = 5.3 Hz, 1H), 4.12 (d, *J* = 6.3 Hz, 1H), 4.02 (q, *J* = 7.2 Hz, 2H), 3.74-3.71 (m, 4H), 1.09 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 170.0, 167.0, 159.4, 151.7, 131.8, 130.1, 129.6, 129.3, 128.6, 125.9, 124.9, 114.4, 114.1, 98.4, 61.4, 54.7, 46.1, 44.4, 13.8. IR (KBr) 2918, 2849, 1732, 1646, 1180, 1132; HRMS (ESI) calcd for C₂₁H₂₀O₅Na [M+Na]⁺ 375.1203, found 375.1198.

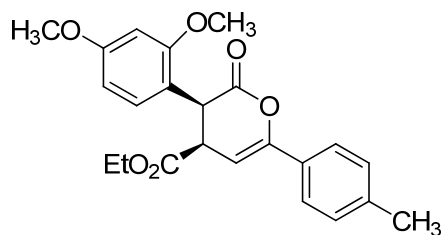


3ea

(3*S*,4*R*)-ethyl 3-(2,4-dimethoxyphenyl)-2-oxo-6-phenyl-3,4-dihydro-2H-pyran-4-carboxylate

41.3 mg, 54% yield; *cis/trans* = 6:1, colorless oil, R_f = 0.3 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -177.0 (*c* 0.1, CH₂Cl₂), HPLC analysis: >99% ee (*cis*), 80% ee (*trans*) [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm, hexane/ *i*-PrOH = 60:40, 1.0 mL /min, 254 nm, 10.9 min (*trans*-major), 12.6 min (*trans*-minor), 18.2 min (*cis*-major), 41.0 min (*cis*-minor)]; ¹H NMR (300 MHz, CDCl₃) δ 7.71-7.66 (m, 2H), 7.48 (d, *J* = 8.5 Hz, 1H), 7.42 – 7.38 (m, 3H), 6.52 – 6.44 (m, 2H), 5.98 (d, *J* = 6.7 Hz,

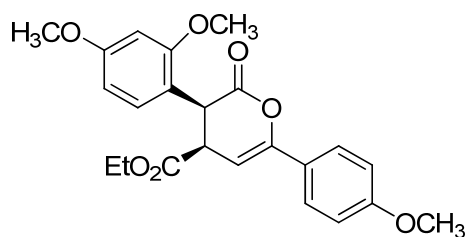
1H), 4.60 (d, $J = 6.2$ Hz, 1H), 4.06 (q, $J = 7.1$ Hz, 2H), 3.81 (s, 3H), 3.80 (s, 3H), 3.64 (t, $J = 6.5$ Hz, 1H), 1.17 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.5, 167.5, 160.5, 157.5, 152.2, 132.0, 130.7, 129.5, 128.5, 124.9, 114.7, 104.2, 98.9, 98.2, 61.3, 55.5, 55.4, 43.9, 38.6, 13.6. IR (KBr) 2920, 2849, 1772, 1732, 1646, 1209, 752; HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{22}\text{O}_6\text{Na}$ $[\text{M}+\text{Na}]^+$ 405.1309, found 405.1303.



3ee

(3*S*,4*R*)-ethyl 3-(2,4-dimethoxyphenyl)-2-oxo-6-(*p*-tolyl)-3,4-dihydro-2H-pyran-4-carboxylate

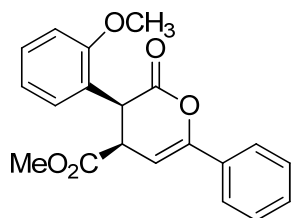
52.7 mg, 66% yield; *cis/trans* = 20:1, colorless oil, $R_f = 0.3$ (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25} -119.8$ (c 0.1, CH_2Cl_2), HPLC analysis: >99% ee for major *cis*-diastereoisomer [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm, hexane/*i*-PrOH/MeOH = 60:20:20, 1.0 mL /min, 254 nm, 11.1 min (*cis*-major)], ^1H NMR (300 MHz, CDCl_3) δ 7.50 (d, $J = 8.2$ Hz, 2H), 7.40 (d, $J = 8.5$ Hz, 1H), 7.11 (d, $J = 8.1$ Hz, 2H), 6.43-6.35 (m, 2H), 5.84 (d, $J = 6.7$ Hz, 1H), 4.51 (d, $J = 6.2$ Hz, 1H), 3.98 (q, $J = 7.2$ Hz, 2H), 3.72 (s, 3H), 3.71 (s, 3H), 3.54 (t, $J = 6.4$ Hz, 1H), 2.29 (s, 3H), 1.08 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.8, 167.9, 160.2, 157.9, 152.2, 139.3, 130.7, 129.3, 129.2, 124.8, 114.7, 104.4, 98.3, 98.0, 61.3, 55.5, 55.4, 44.1, 38.9, 21.3, 14.0. IR (KBr) 2923, 2849, 1772, 1732, 1614, 1508, 1464, 1209; HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{24}\text{O}_6\text{Na}$ $[\text{M}+\text{Na}]^+$ 419.1465, found 419.1459.



3ef

(3S,4R)-ethyl 3-(2,4-dimethoxyphenyl)-6-(4-methoxyphenyl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

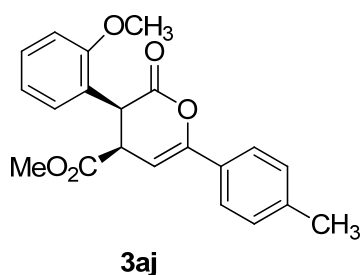
41.0 mg, 50% yield; *cis/trans* > 20:1, colorless oil, R_f = 0.2 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -194.0 (c 0.1, CH_2Cl_2), HPLC analysis: 99% ee for major *cis*-diastereoisomer [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH/MeOH = 60:20:20, 1.0 mL /min, 254 nm, 12.4 min (*cis*-major)], ^1H NMR (300 MHz, CDCl_3) δ 7.63 (d, J = 8.8 Hz, 2H), 7.47 (d, J = 8.5 Hz, 1H), 6.91 (d, J = 8.8 Hz, 2H), 6.52-6.44 (m, 2H), 5.84 (d, J = 6.7 Hz, 1H), 4.58 (d, J = 6.2 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 3.83 (s, 3H), 3.81 (s, 3H), 3.80 (s, 3H), 3.63-3.59 (m, 1H), 1.17 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 170.8, 168.0, 160.6, 160.5, 157.9, 152.3, 130.7, 126.4, 124.7, 114.8, 113.9, 104.4, 98.3, 97.0, 61.3, 55.5, 55.4, 44.0, 38.9, 14.0. IR (KBr) 2921, 2849, 1771, 1732, 1611, 1457, 1178, 1034, 836; HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{24}\text{O}_7\text{Na}$ $[\text{M}+\text{Na}]^+$ 435.1414, found 435.1407.



3ai

(3S,4R)-methyl 3-(2-methoxyphenyl)-2-oxo-6-phenyl-3,4-dihydro-2H-pyran-4-carboxylate

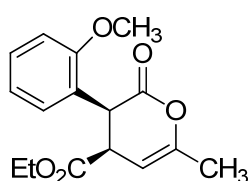
44.3 mg, 66% yield, *cis/trans* = 10:1, white solid, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -269.0 (*c* 0.1, CH₂Cl₂), HPLC analysis: >99% ee (*cis*), 88% ee (*trans*) [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm hexane/*i*-PrOH/MeOH = 80:10:10, 1.0 mL /min, 254 nm, 10.4 min (*trans*-major), 11.4 min (*trans*-minor), 12.7 min(*cis*-major)]; ¹H NMR (400 MHz, CDCl₃) δ 7.72-7.69 (m, 2H), 7.53 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.44 – 7.39 (m, 3H), 7.31 – 7.27 (m, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 8.3 Hz, 1H), 5.98 (d, *J* = 6.8 Hz, 1H), 4.69 (d, *J* = 6.2 Hz, 1H), 3.84 (s, 3H), 3.70 (t, *J* = 6.5 Hz, 1H), 3.61 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 167.3, 156.8, 152.6, 132.0, 130.0, 129.6, 129.2, 128.6, 124.9, 122.2, 120.7, 110.3, 98.7, 55.5, 52.2, 43.9, 39.3. IR (KBr) 2919, 2848, 1773, 1697, 1651, 1495, 1244, 752; HRMS (ESI) calcd for C₂₀H₁₈O₅Na [M+Na]⁺ 361.1043, found 361.1046.



(3*S*,4*R*)-methyl 3-(2-methoxyphenyl)-2-oxo-6-(*p*-tolyl)-3,4-dihydro-2H-pyran-4-carboxylate

45.7 mg, 65% yield, *cis/trans* = 10:1, white solid, R_f = 0.4 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -315.4 (*c* 0.1, CH₂Cl₂), HPLC analysis: >99% ee (*cis*), 70% ee (*trans*) [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm hexane/*i*-PrOH/MeOH = 80:10:10, 1.0 mL /min, 254 nm, 10.8 min (*trans*-major), 11.3 min (*trans*-minor), 14.7 min(*cis*-major)]; ¹H NMR (400 MHz, CDCl₃) δ 7.59 (d, *J* = 8.1 Hz, 2H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.31 – 7.26 (m, 1H), 7.20 (d, *J* = 8.1 Hz, 2H), 6.98 (t, *J* = 7.6 Hz, 1H),

6.89 (d, $J = 8.3$ Hz, 1H), 5.92 (d, $J = 6.8$ Hz, 1H), 4.68 (d, $J = 6.2$ Hz, 1H), 3.84 (s, 3H), 3.68 (t, $J = 6.5$ Hz, 1H), 3.60 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 171.0, 167.5, 156.8, 152.7, 139.7, 130.0, 129.3, 129.2, 129.1, 124.9, 122.3, 120.7, 110.3, 97.8, 55.5, 52.2, 43.9, 39.3, 21.3. IR (KBr) 2920, 2845, 1775, 1738, 1646, 1602, 1495, 1170, 754; HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{20}\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 375.1198, found 375.1203.



3ak

(3S,4R)-ethyl 3-(2-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate

23.6 mg, 27% yield, *cis/trans* > 20:1, white solid, $R_f = 0.3$ (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ -433.2 (c 0.1, CH_2Cl_2), HPLC analysis: 97% ee (*cis*) [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm hexane/*i*-PrOH/MeOH = 90:5:5, 1.0 mL/min, 254 nm, 9.2 min(*cis*-major), 10.6 min (*cis*-minor)]; ^1H NMR (400 MHz, CDCl_3) δ 7.51 (dd, $J = 7.7, 1.4$ Hz, 1H), 7.29-7.24 (m, 1H), 6.94 (t, $J = 7.6$ Hz, 1H), 6.87 (d, $J = 8.3$ Hz, 1H), 5.21 (dd, $J = 6.5, 1.0$ Hz, 1H), 4.55 (d, $J = 6.2$ Hz, 1H), 4.03 (q, $J = 7.1$ Hz, 2H), 3.83 (s, 3H), 3.42 (t, $J = 6.4$ Hz, 1H), 1.99 (s, 3H), 1.14 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) 171.0, 167.9, 156.8, 152.4, 130.1, 129.0, 122.4, 120.5, 110.2, 99.4, 61.2, 55.5, 43.6, 39.2, 19.0, 13.9. IR (KBr) 2919, 2849, 1775, 1732, 1602, 1495, 1245, 1180, 755; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{18}\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 313.1044, found 313.1046.

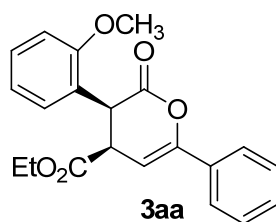
2. X-Ray structure of **3aa** (Figure S1)

The crystal suitable for X-ray analysis was prepared by slow evaporation of the solvent of the solution of **3aa** in petroleum ether/ethyl acetate at room temperature (Figure 1). The 3*S*, 4*R*-configuration of the cycloadduct **3aa** was determined by the X-ray analysis of its crystal with the Flack parameter of 0.05(7).

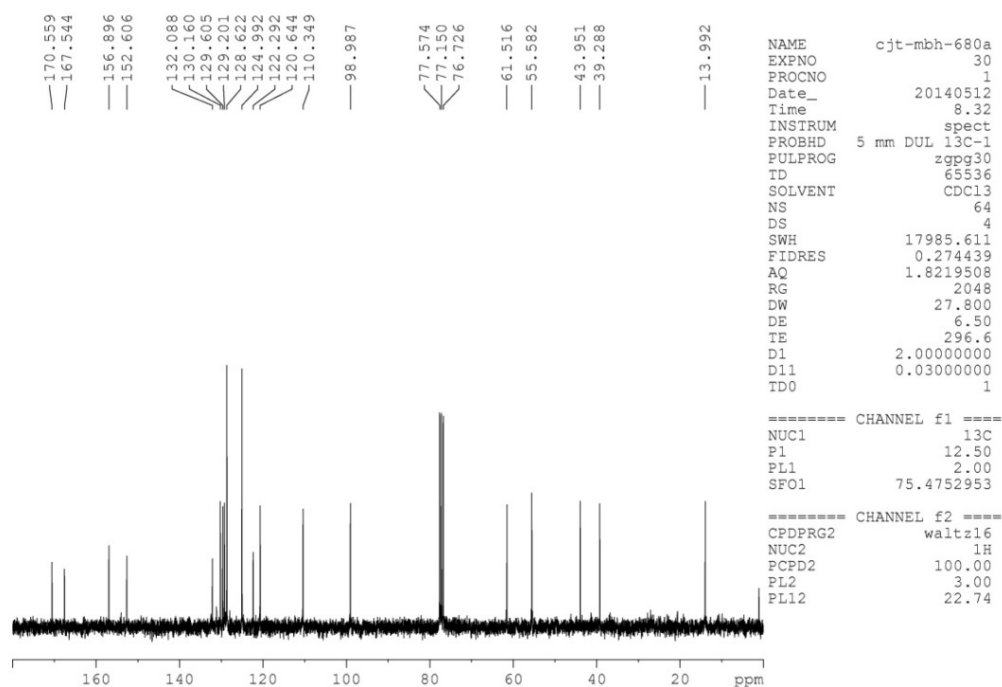
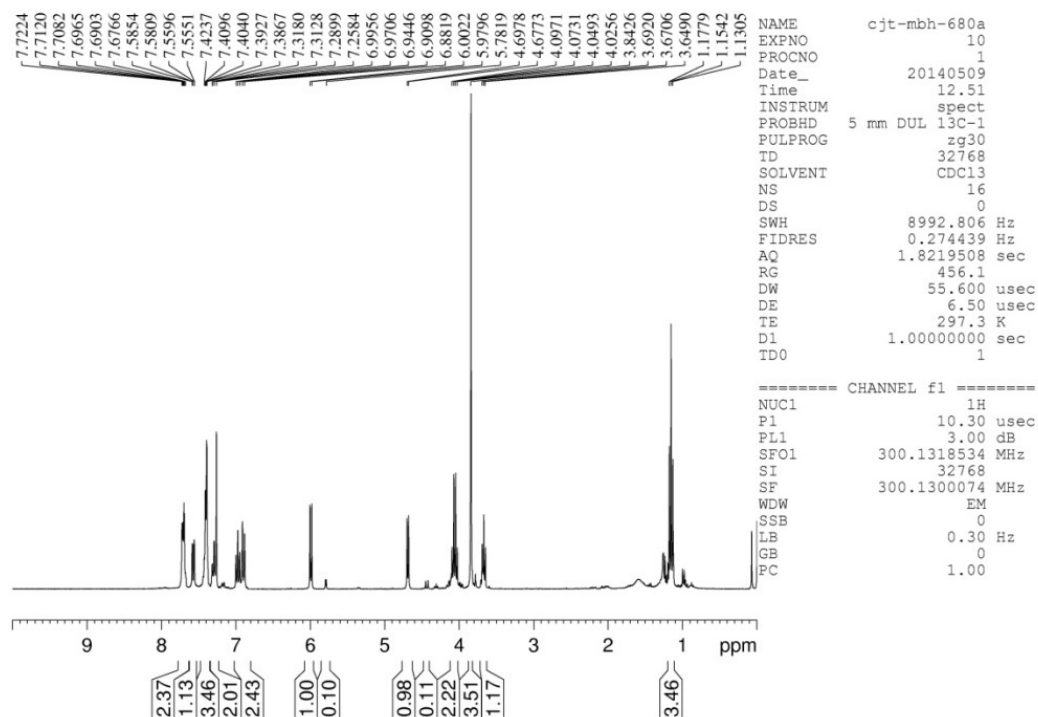
3. References

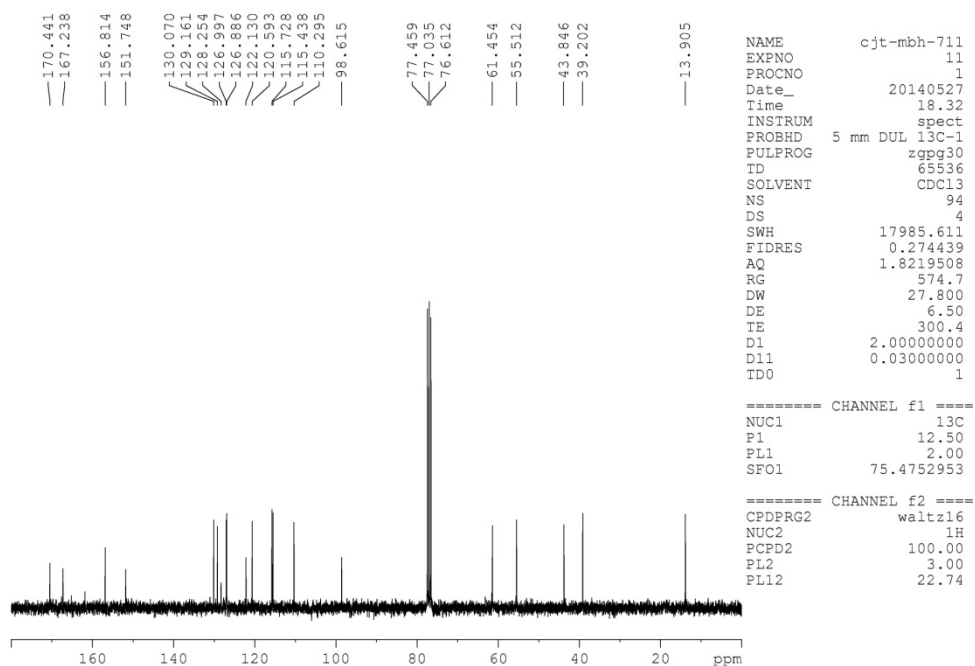
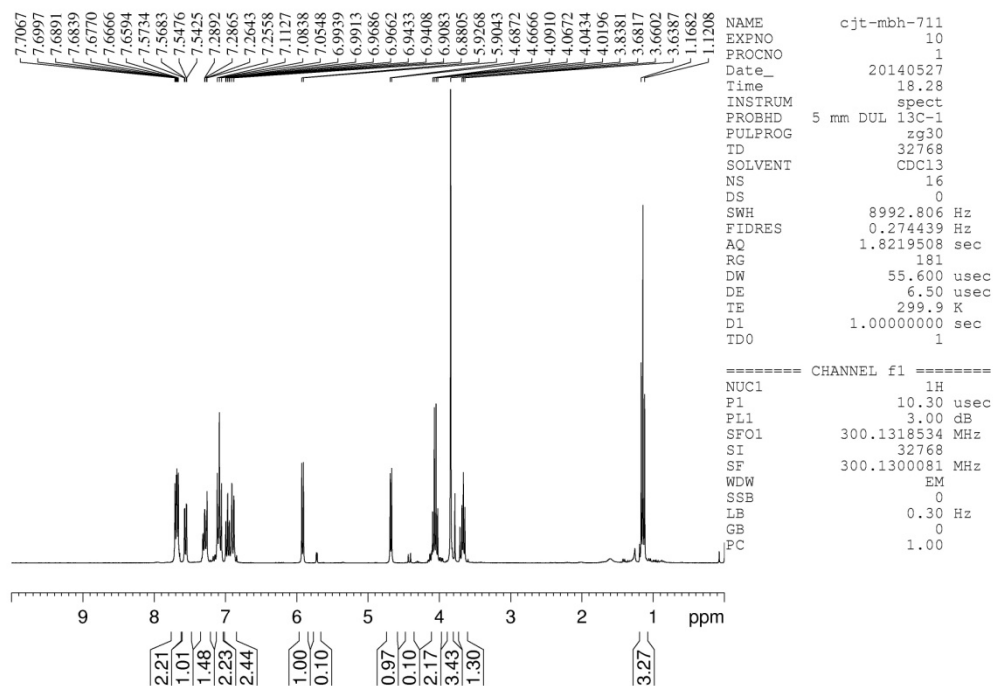
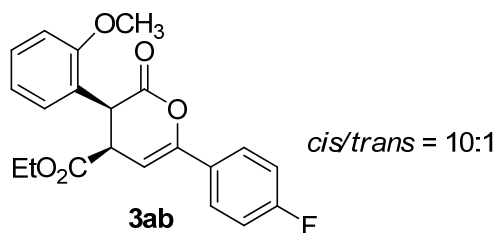
1. (a) A. K. Ghosh, S. P. McKee and W. M. Sanders, *Tetrahedron Lett.* 1991, **32**, 711; (b) M. S. Kerr, J. Read de Alaniz and T. Rovis, *J. Org. Chem.* 2005, **70**, 5725; (c) M. He, J. R. Struble, and J. W. Bode, *J. Am. Chem. Soc.* 2006, **128**, 8418.
2. B. Cardinal-David, D. E. A. Raup and K. A. Scheidt, *J. Am. Chem. Soc.*, 2010, **132**, 5345.
3. (a) Y. R. Zhang, L. He, X. Wu, P. L. Shao and S. Ye, *Org. Lett.*, 2007, **10**, 277; (b) D. Enders and J. Han, *Tetrahedron: Asymmetry*, 2008, **19**, 1367.
4. (a) L. He, Y. R. Zhang, X. L. Huang and S. Ye, *Synthesis*, 2008, 2825; (b) X. L. Huang, L. He, P. L. Shao and S. Ye, *Angew. Chem., Int. Ed.*, 2009, **48**, 192; (c) H. M. Zhang, Z. H. Gao and S. Ye, *Org. Lett.*, 2014, **16**, 3079.
5. Y. R. Zhang, H. Lv, D. Zhou and S. Ye, *Chem. Eur. J.*, 2008, **14**, 8473.

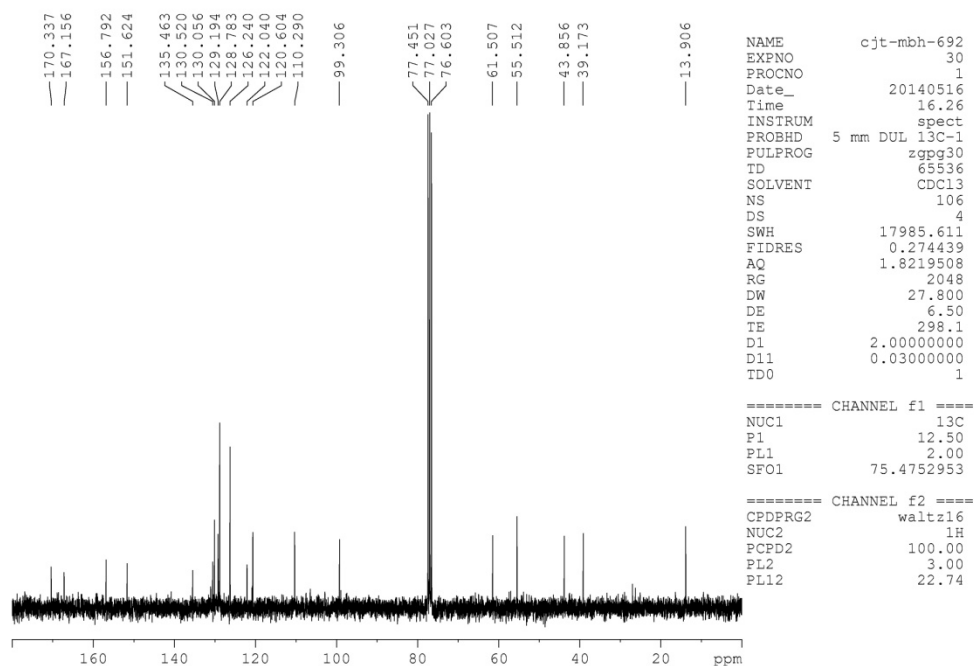
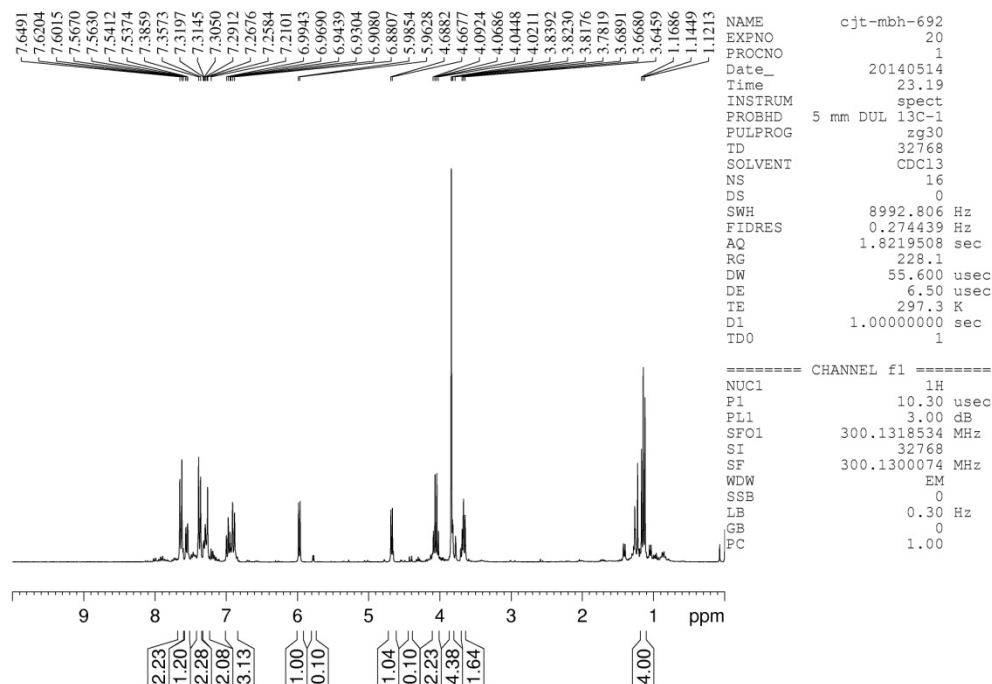
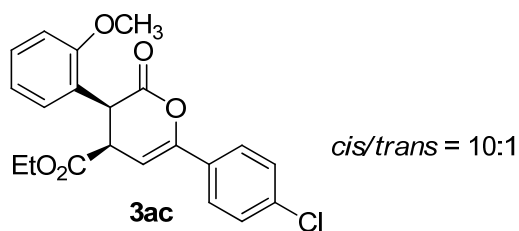
Part III NMR Spectra

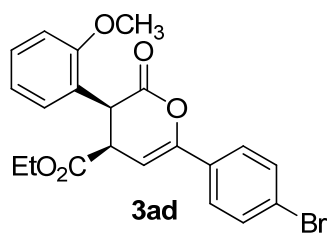


cis/trans = 10:1

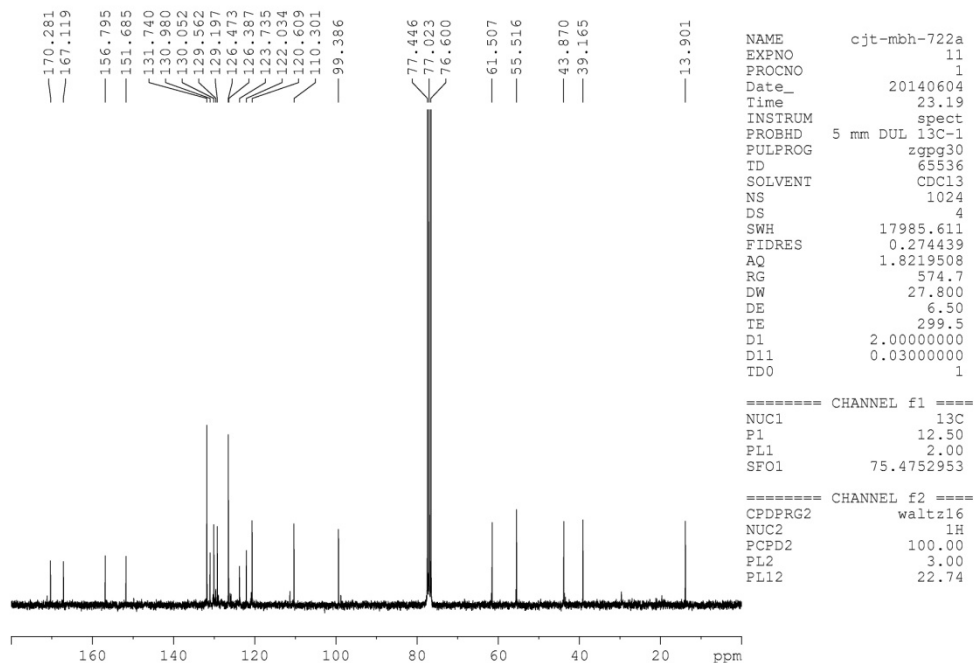
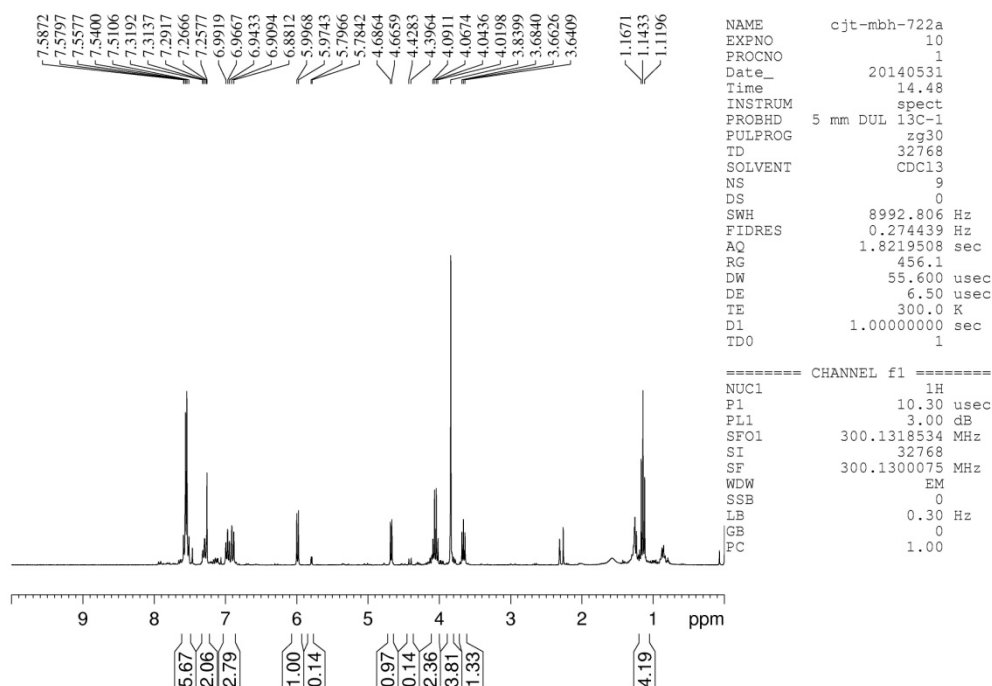


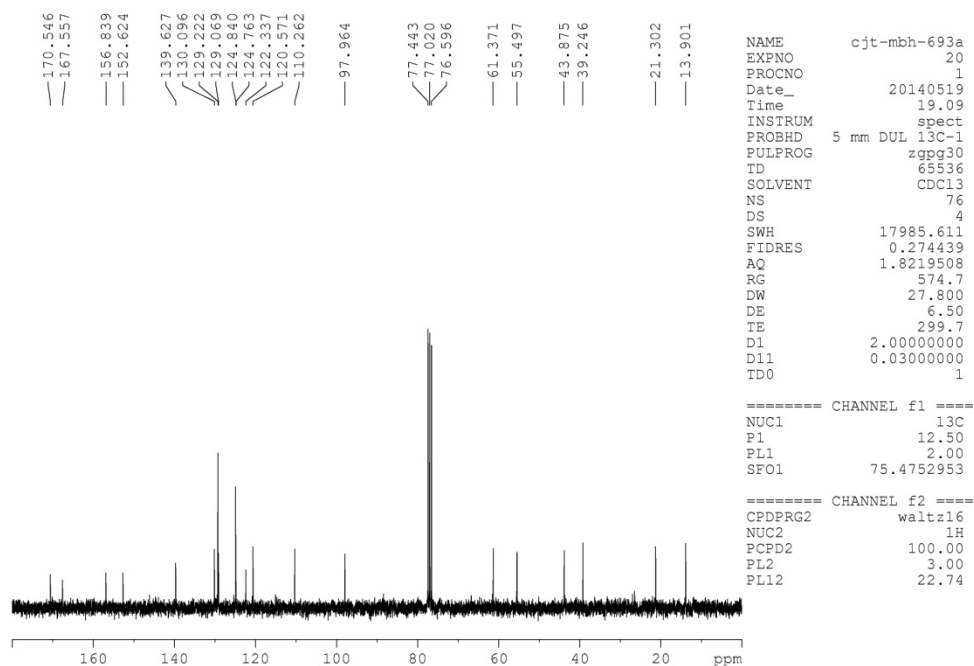
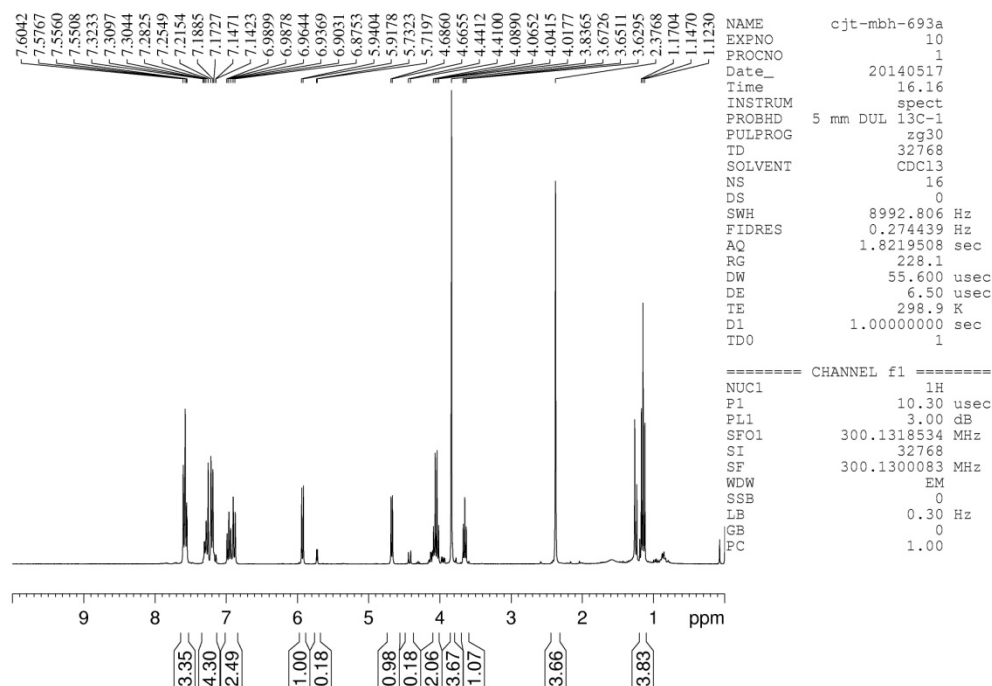
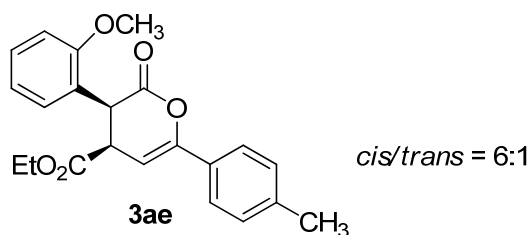


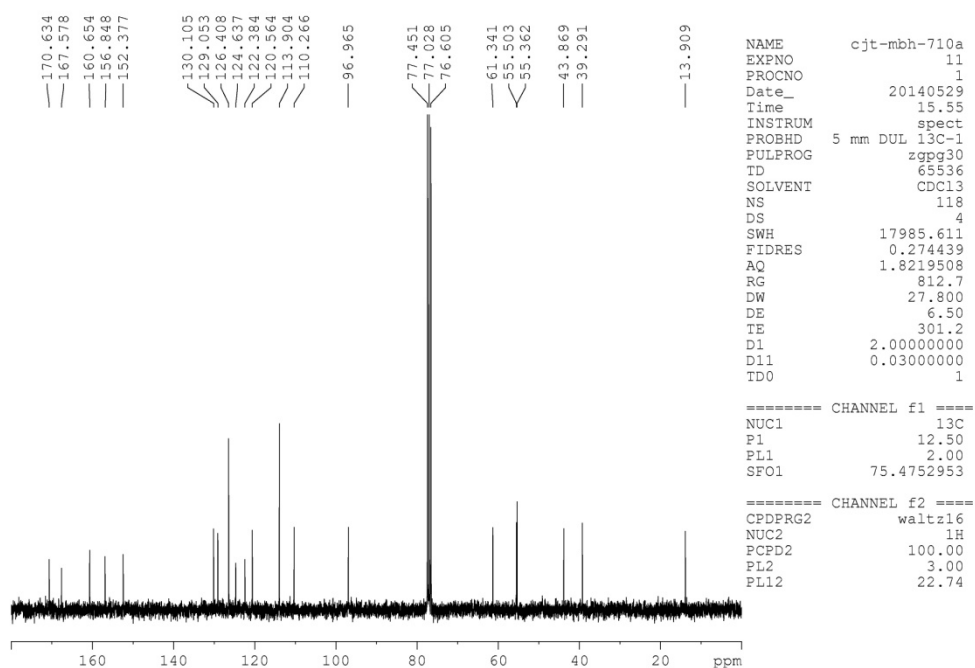
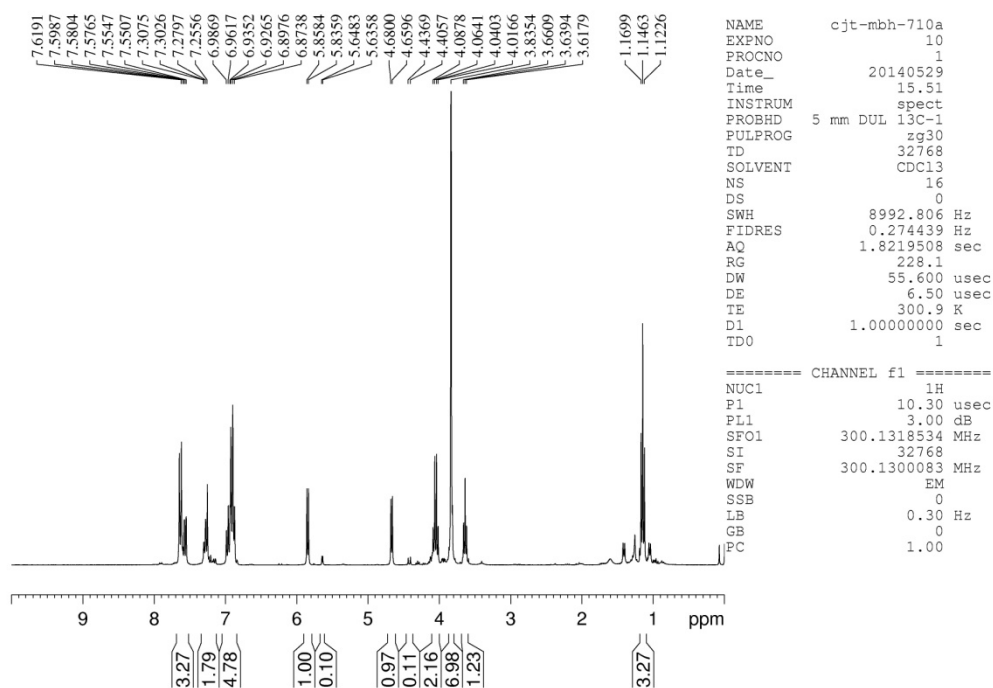
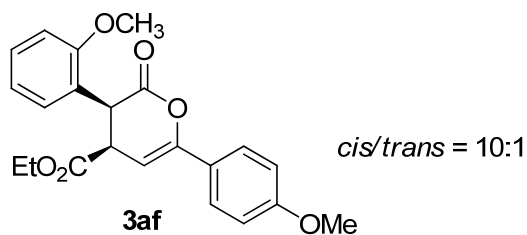


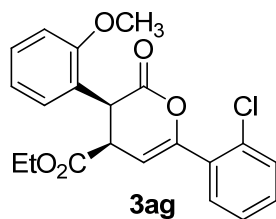


cis/trans = 7:1

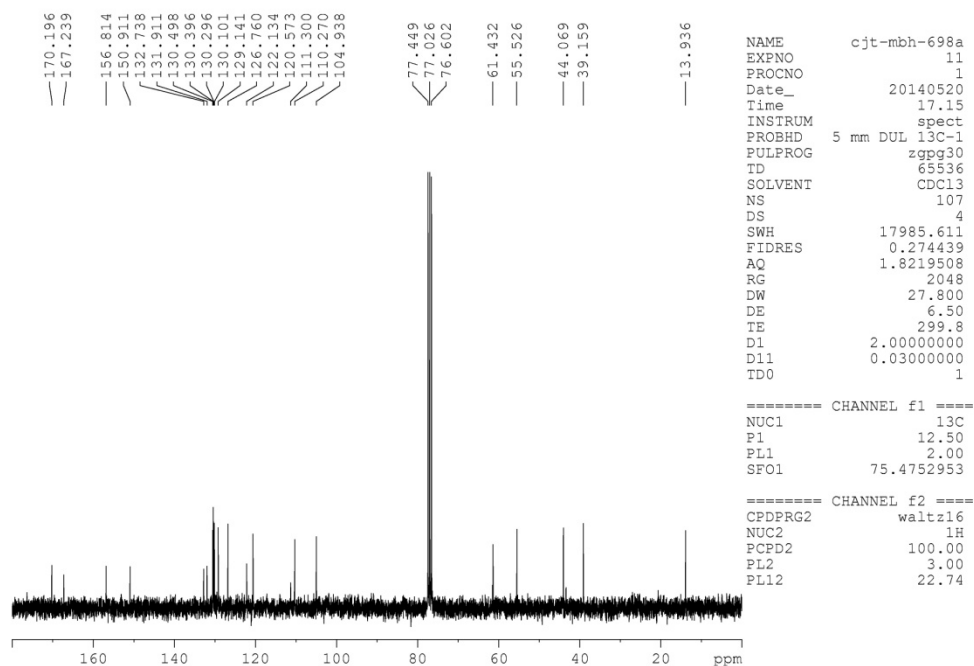
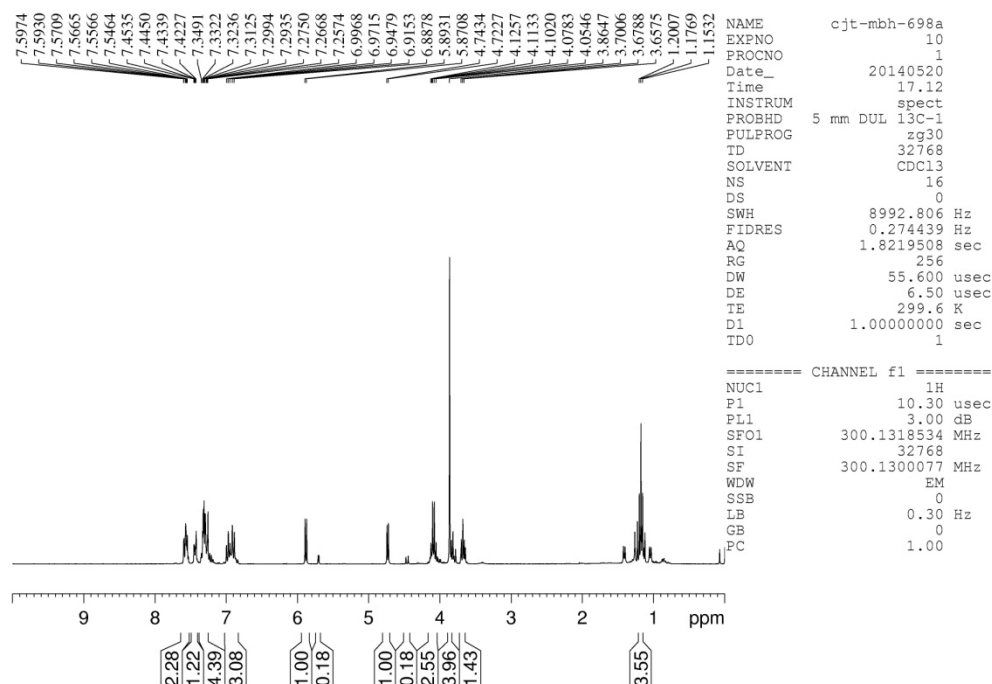


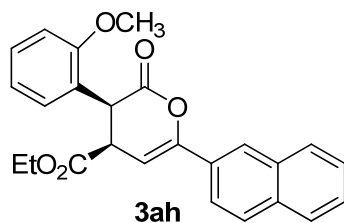




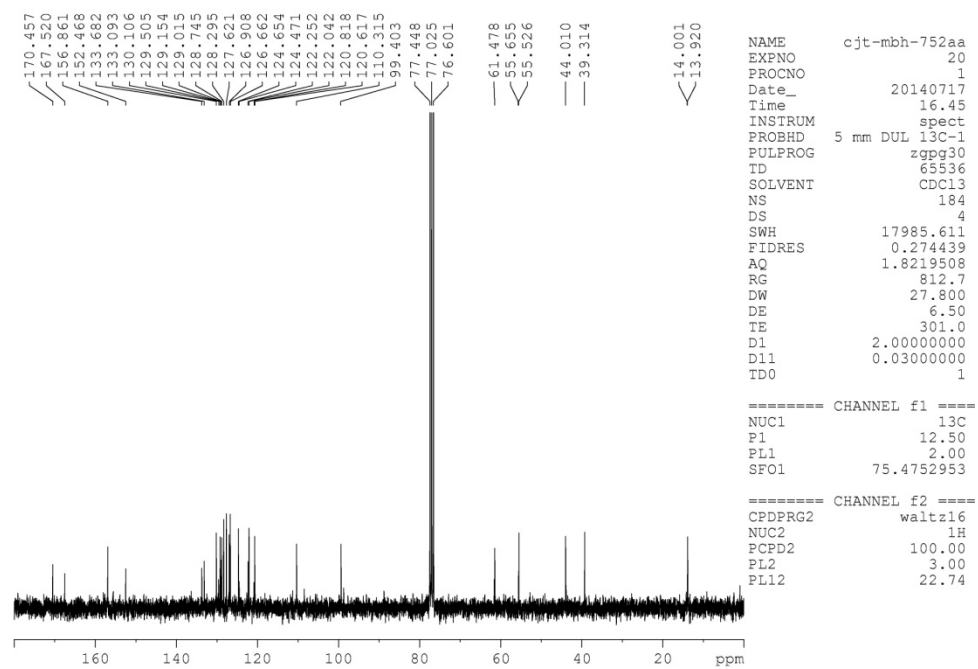
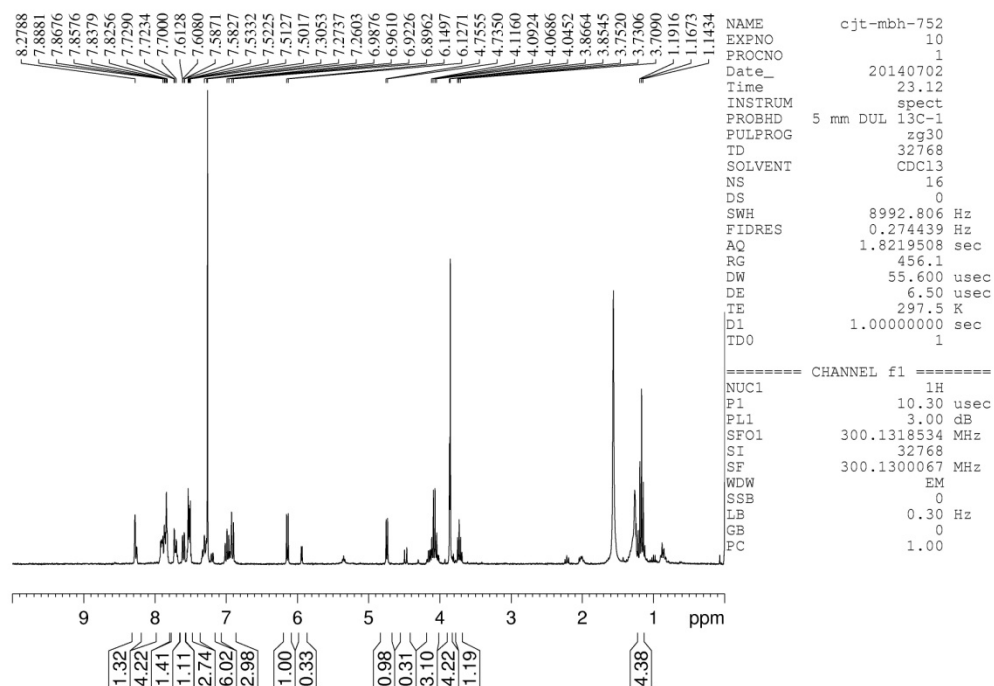


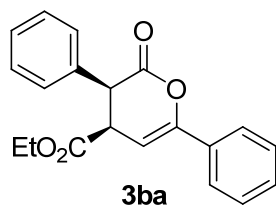
cis/trans = 6:1



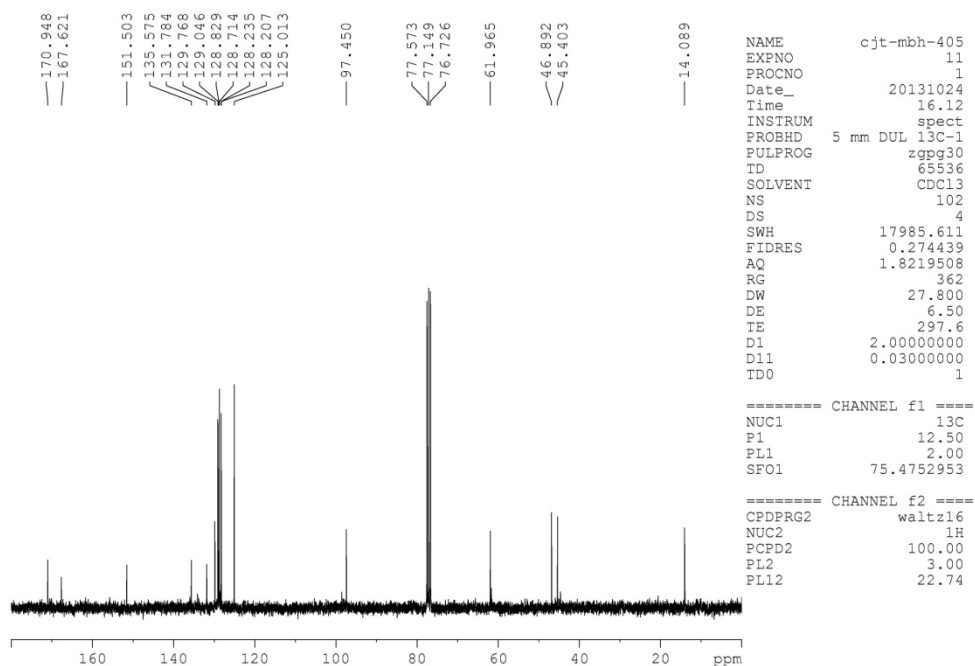
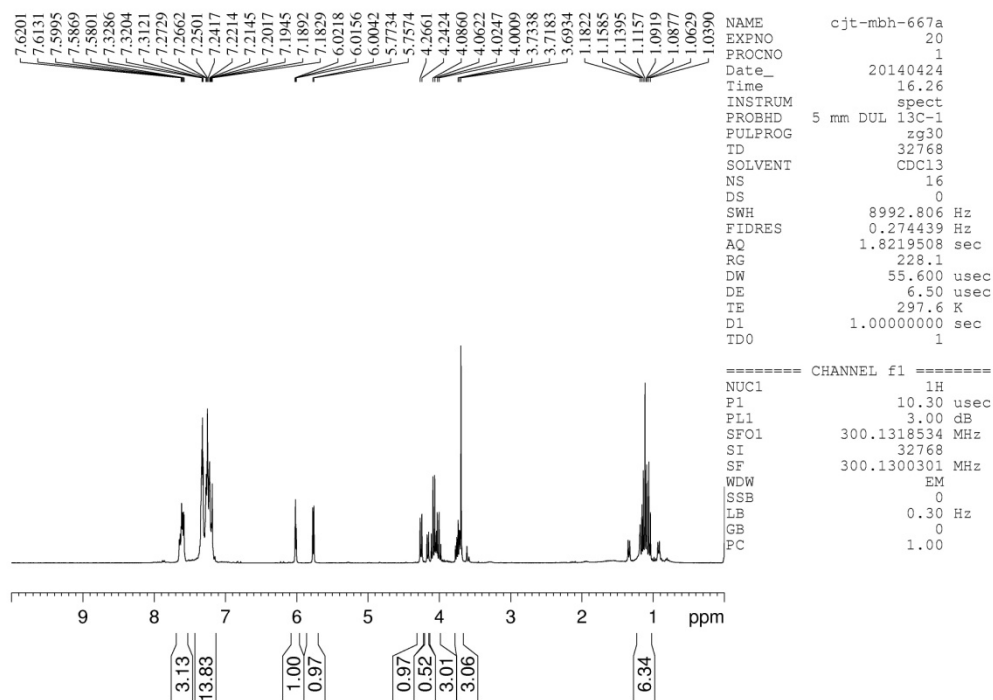


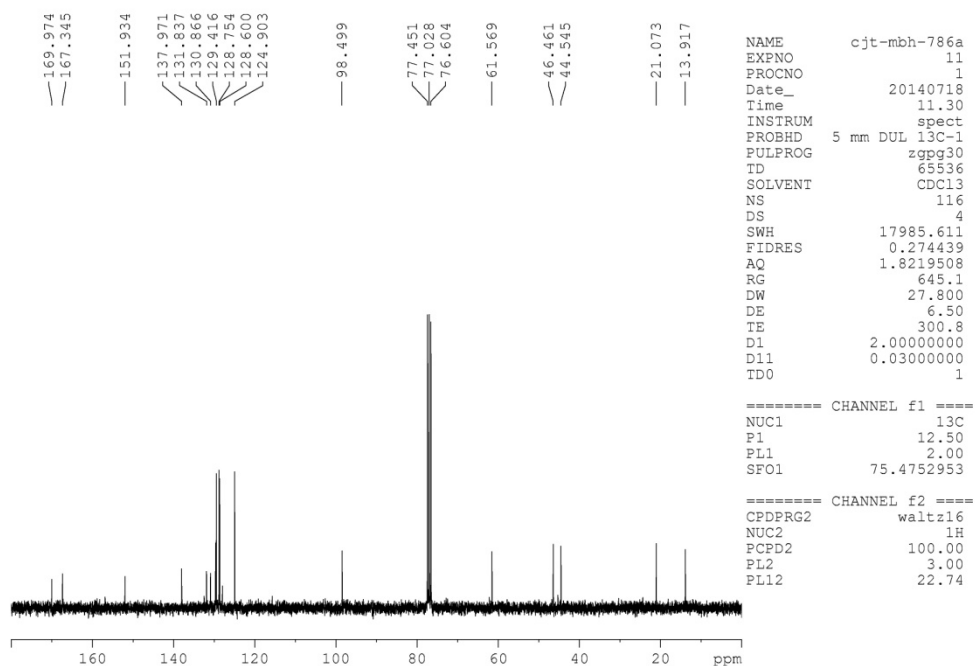
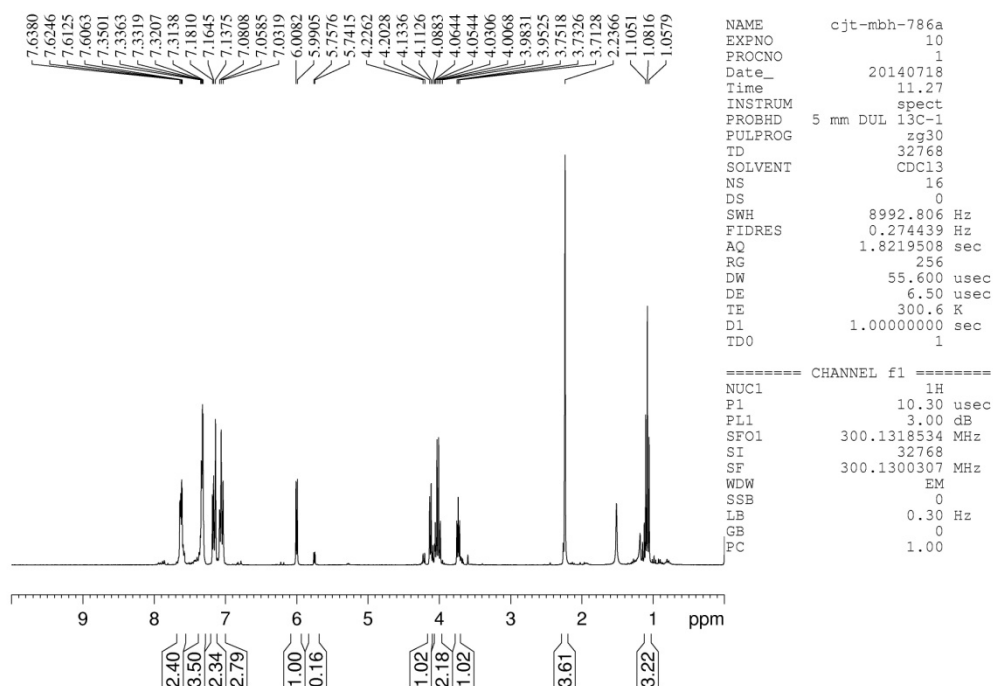
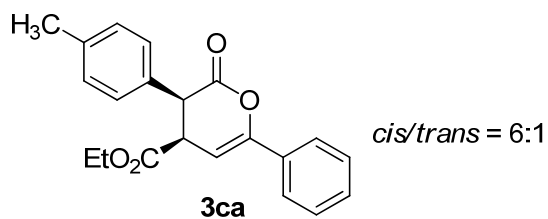
cis/trans = 3:1

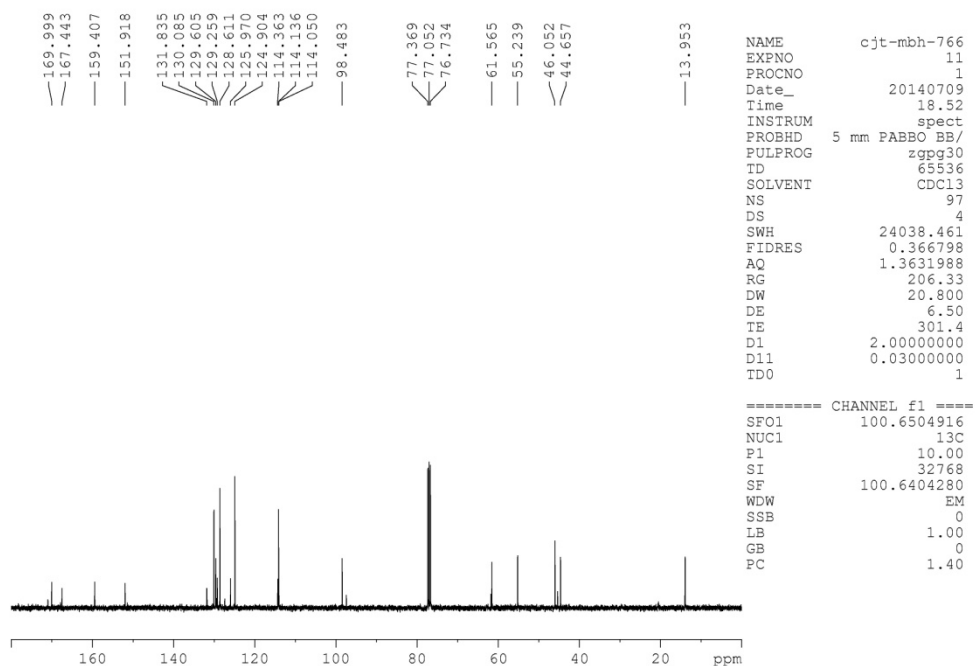
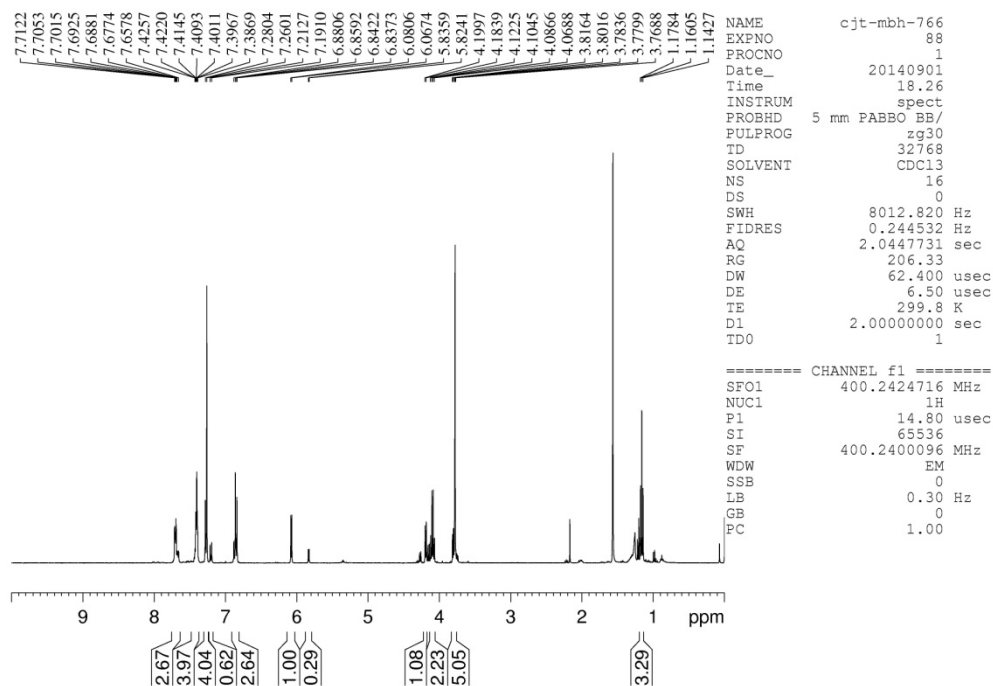
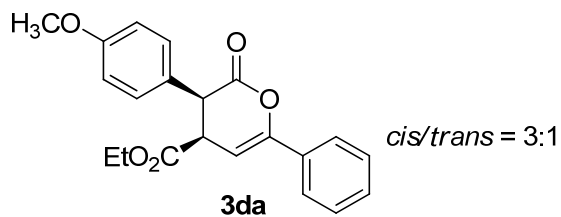


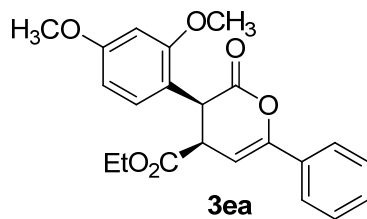


cis/trans = 1:1

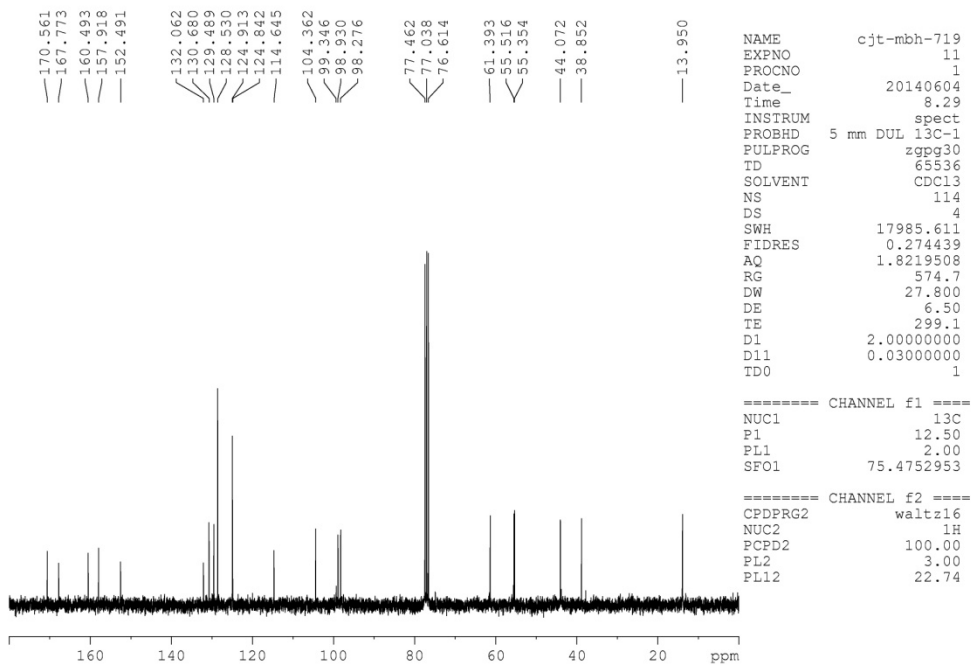
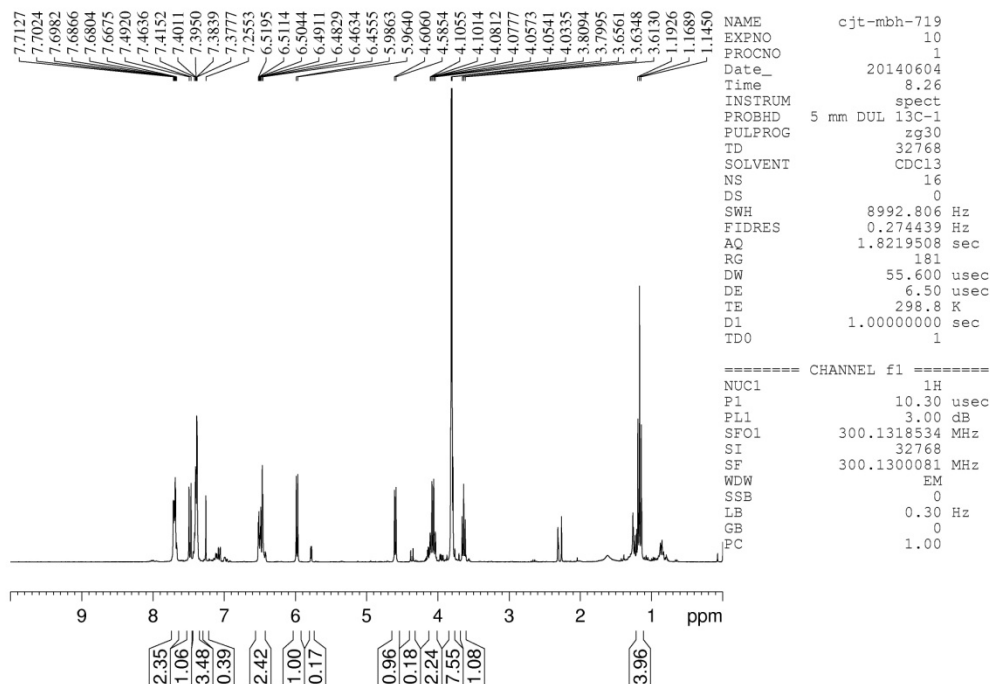


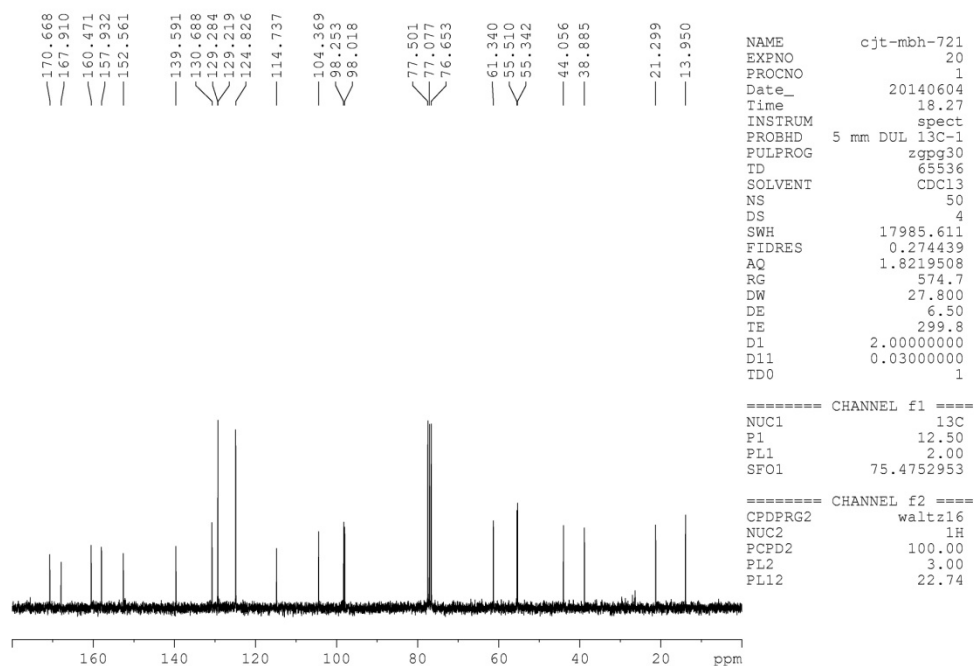
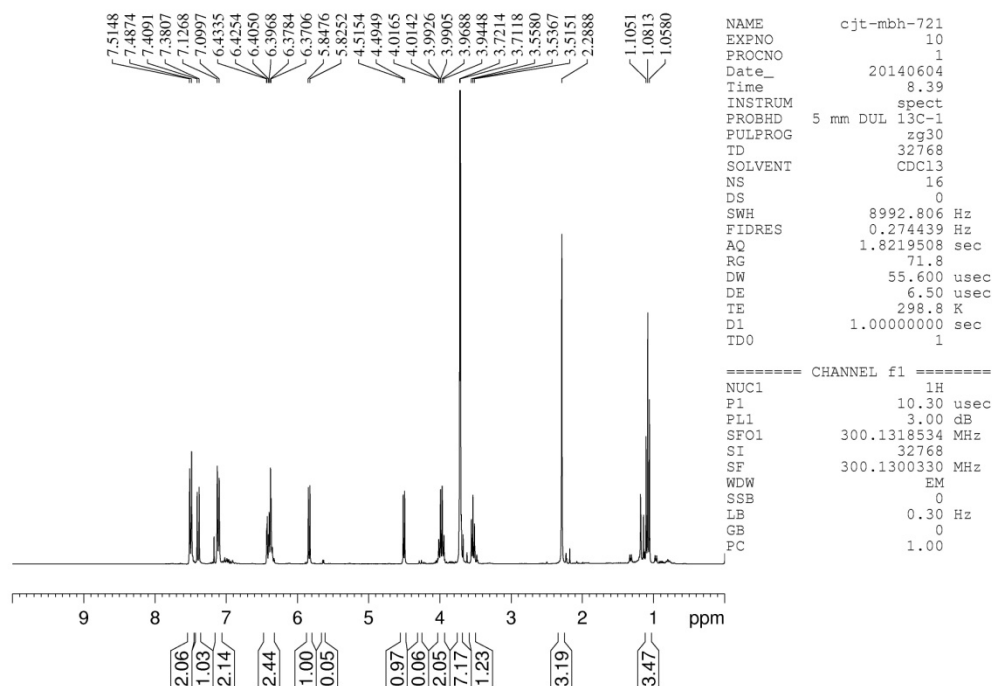
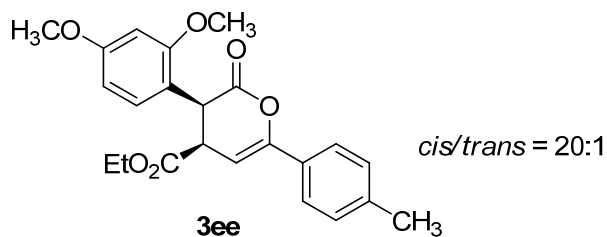


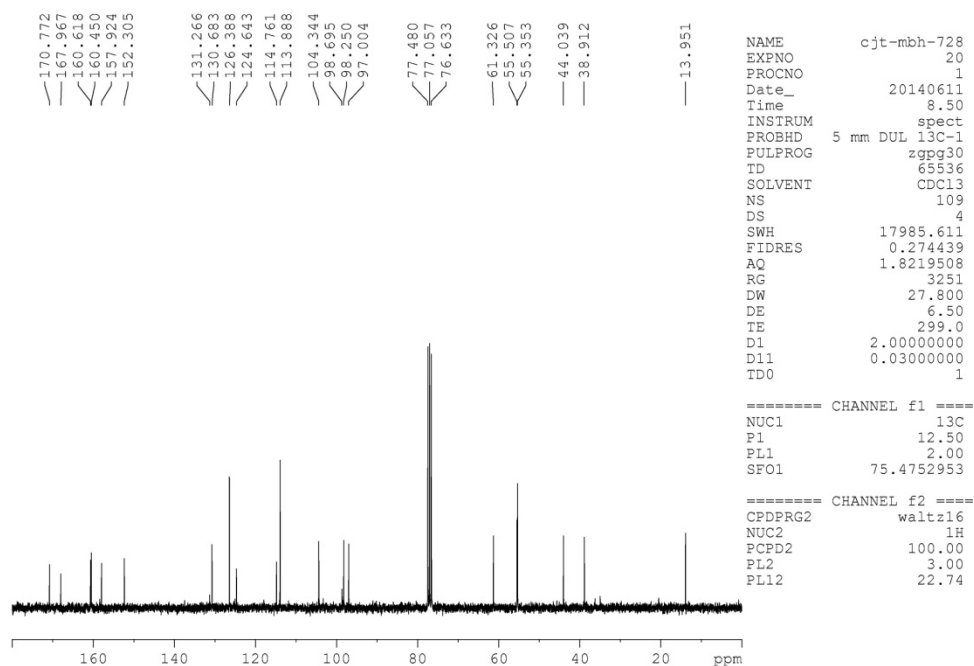
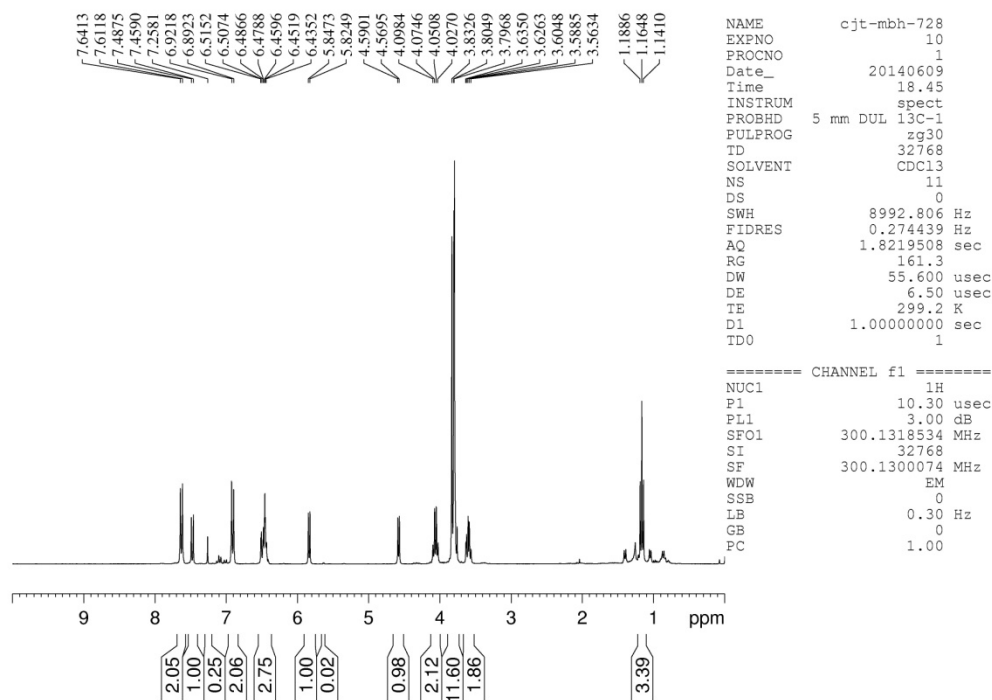
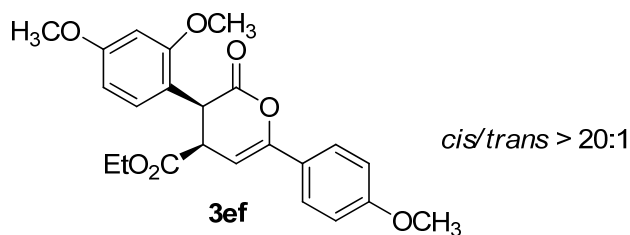


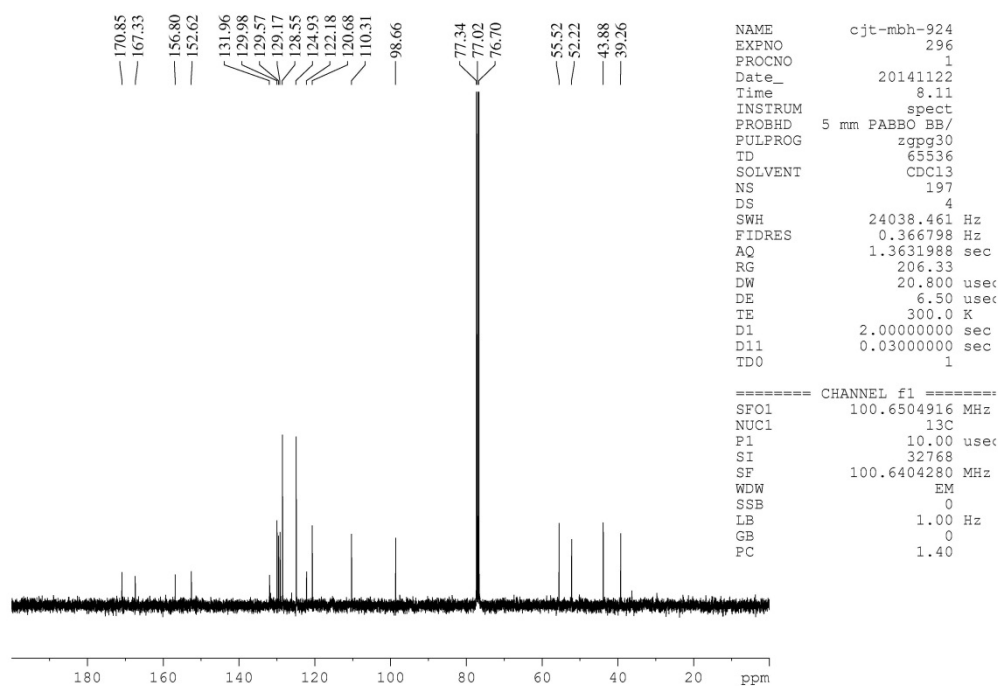
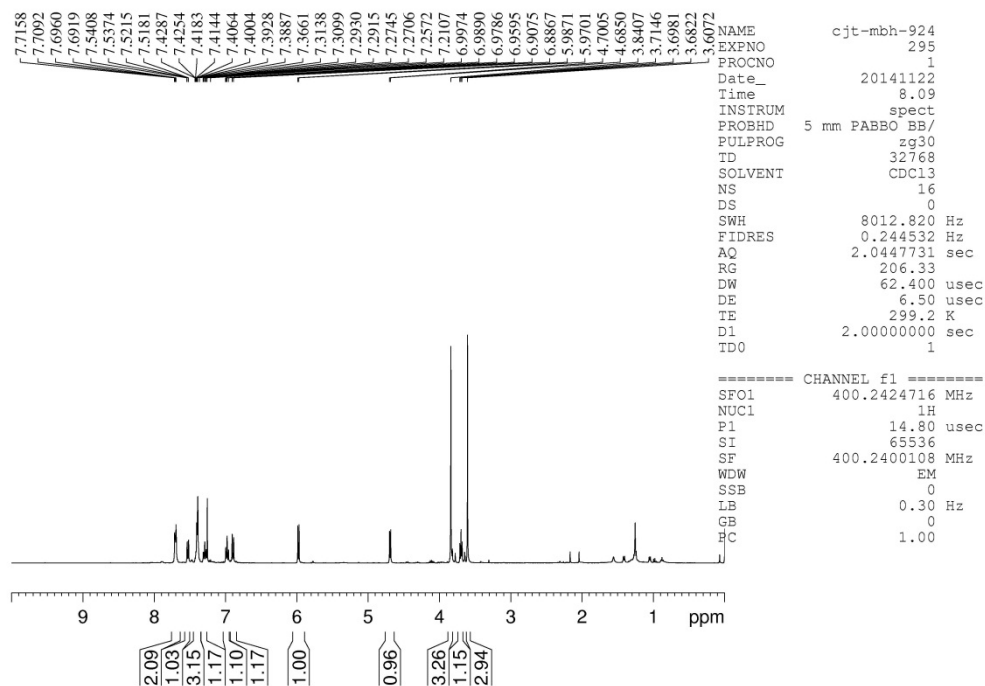
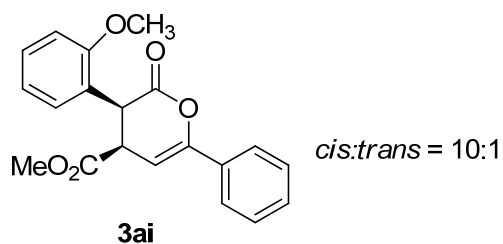


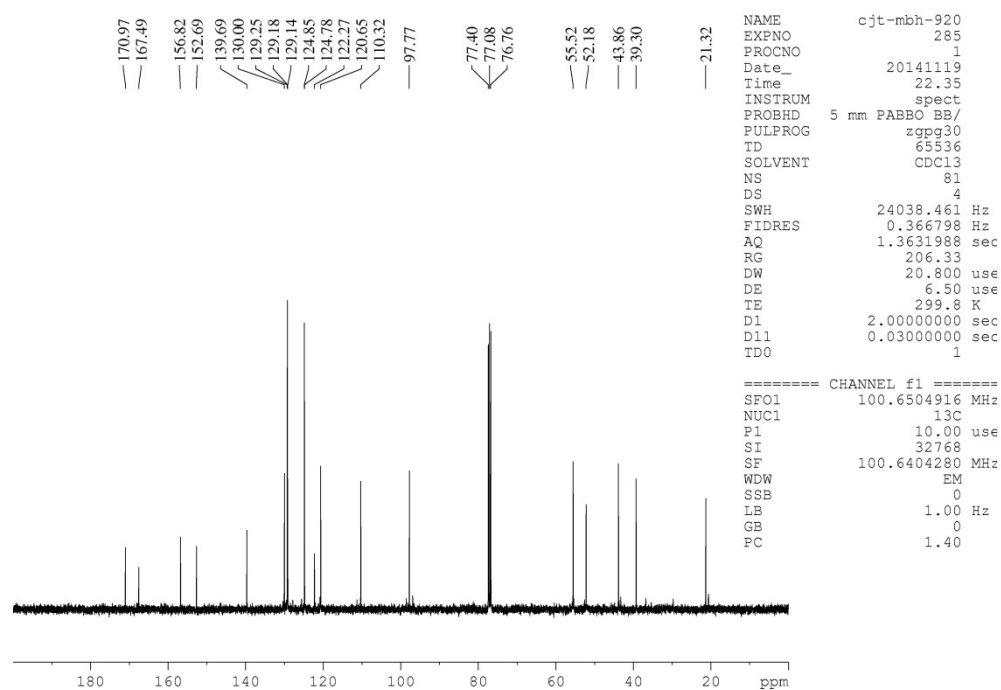
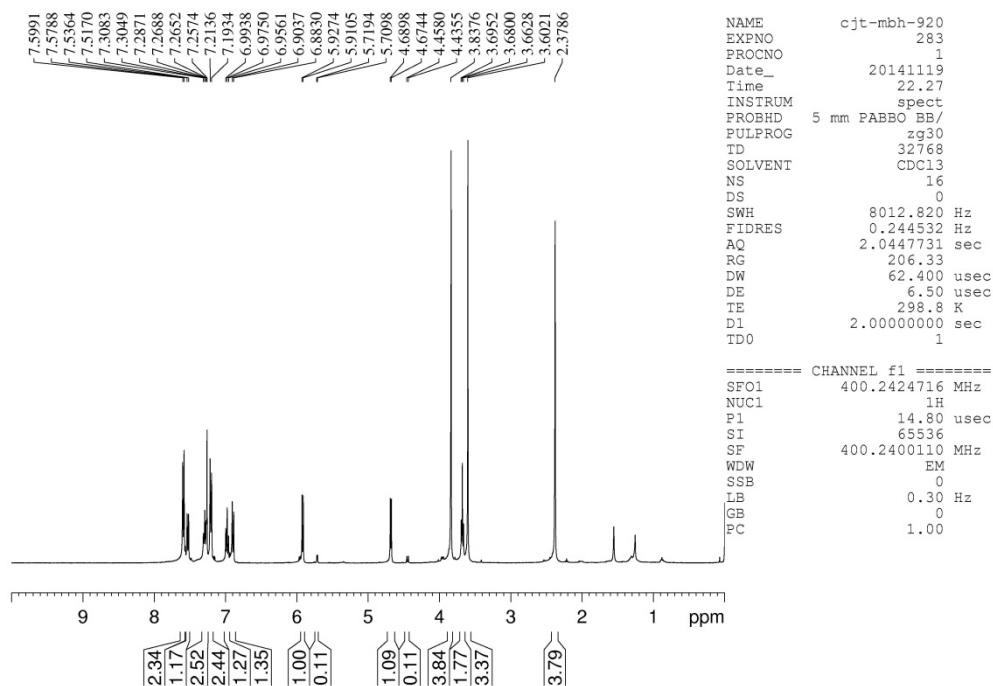
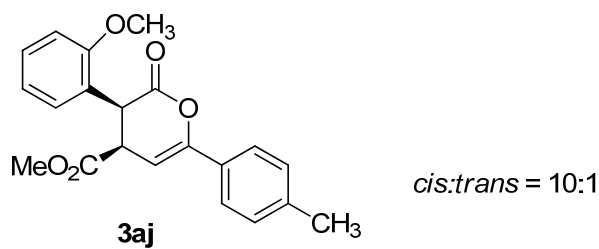
cis/trans = 6:1

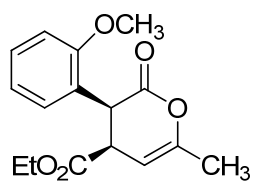






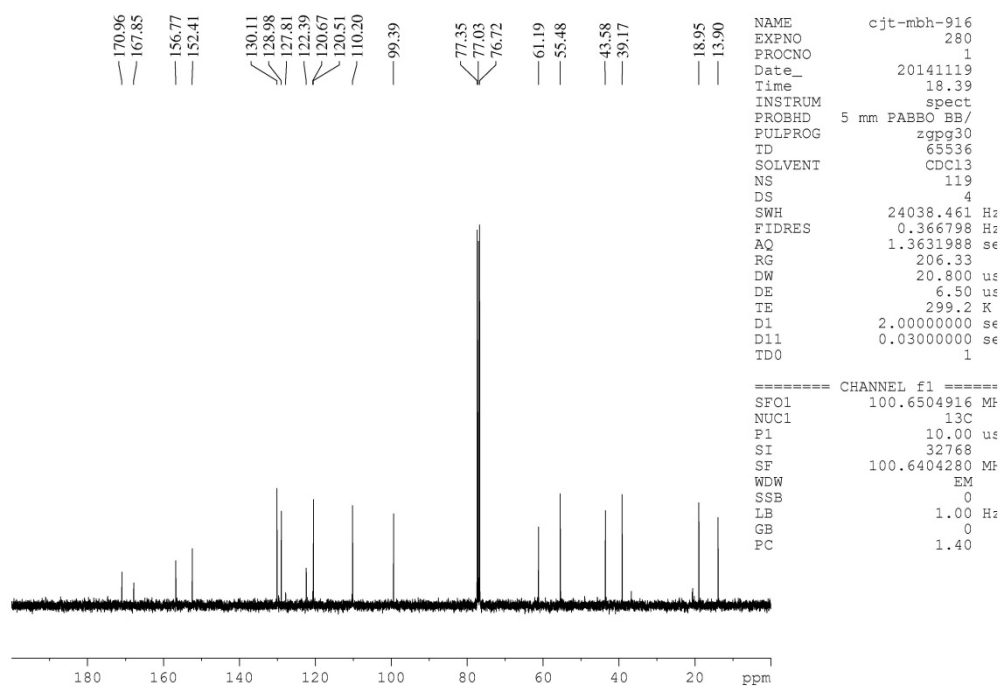
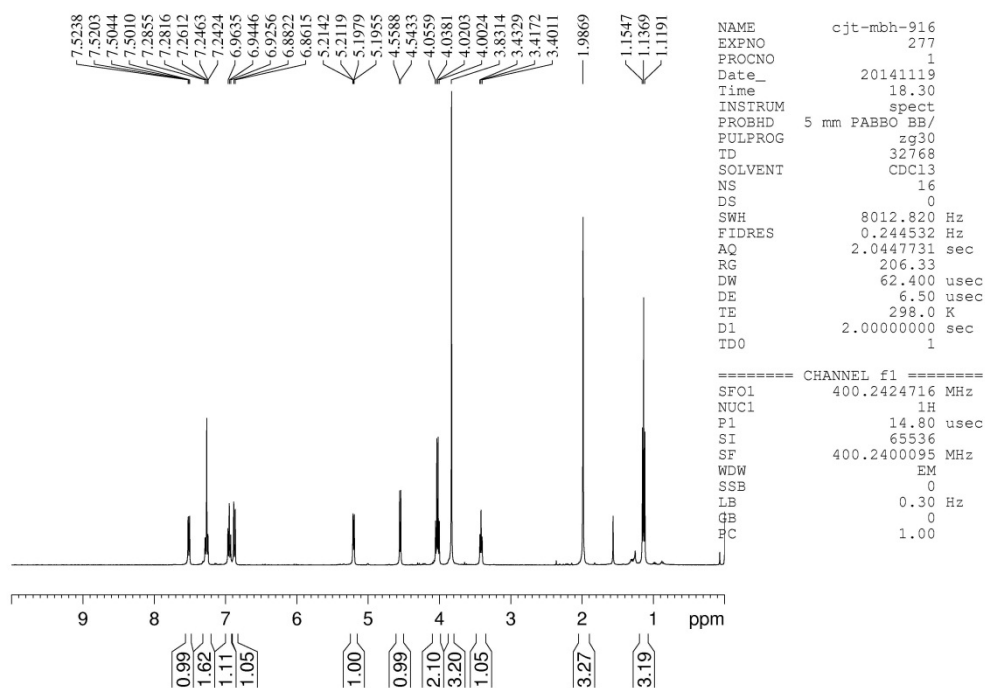




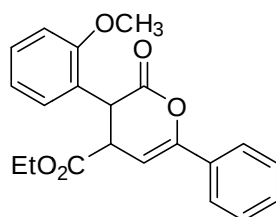


cis:trans > 20:1

3ak



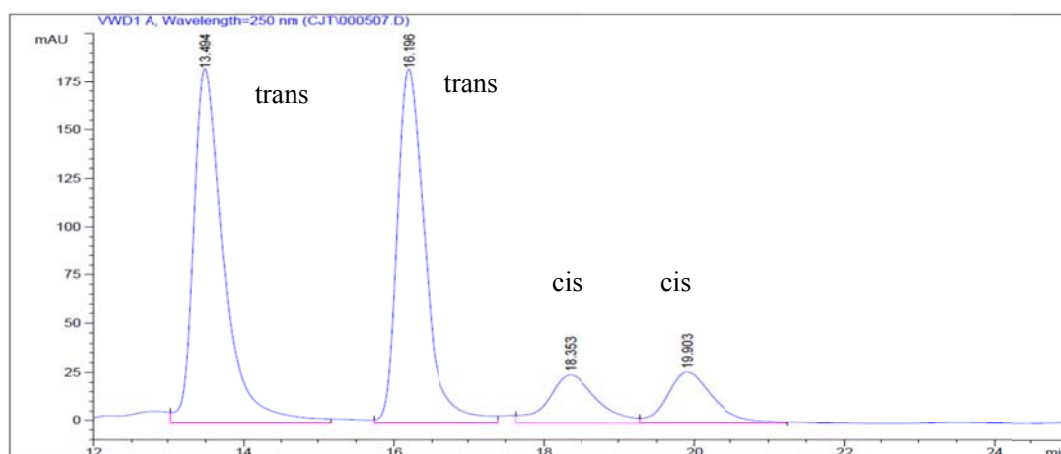
Part IV HPLC Spectra



rac-3aa cis/trans = 1:4

Data File C:\CHEM32\1\DATA\CJT\000507.D
Sample Name: cjt-mbh-669

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-5-5 10:10:00
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-5-5 10:07:34 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-8-28 16:15:49 by GZH
                  (modified after loading)
Sample Info     : OD-3 H:1=90:10 1.0ml/min 250nm
=====
```



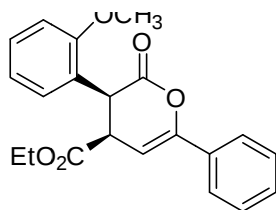
```
=====
Area Percent Report
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with :STDs

Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [nAU] %
-----|-----|-----|-----|-----|
1 13.494 VB 0.4182 5097.16602 182.95428 42.0005
2 16.196 BB 0.4082 4757.41699 182.92815 39.2010
3 18.353 BV 0.6703 1145.24414 25.18393 9.4368
4 19.903 VB 0.6437 1136.13782 26.61035 9.3617
Totals : 1.21360e4 417.67671
=====
*** End of Report ***
```

Instrument 1 2014-8-28 16:16:01 GZH

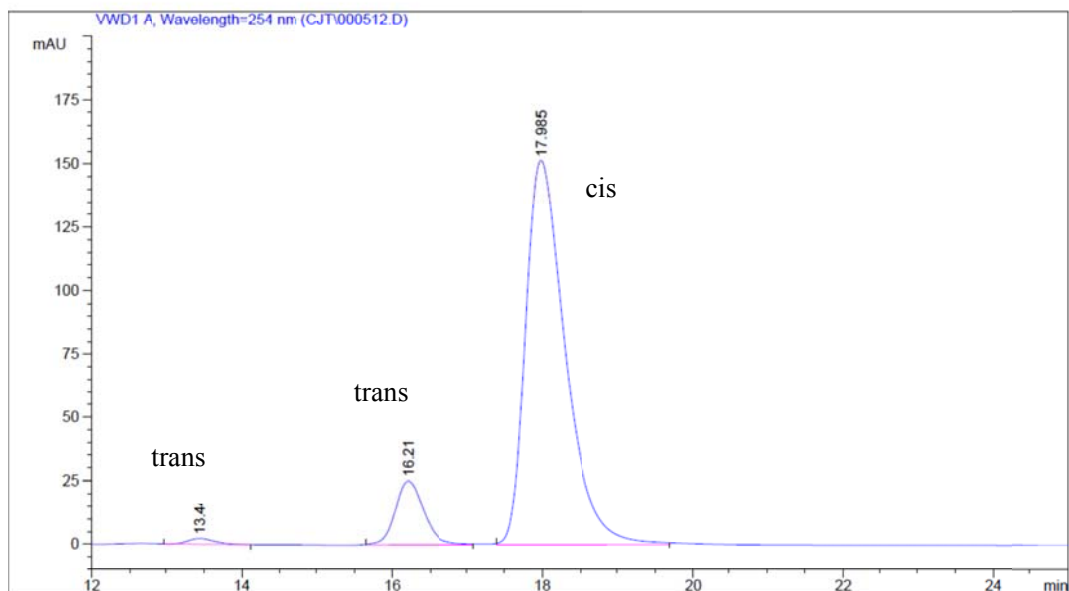
Page 1 of 1



3aa *cis/trans* = 10:1

Data File C:\CHEM32\1\DATA\CJT\000512.D
Sample Name: cjt-mbh-680a

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-5-9 10:41:43
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-5-9 10:02:56 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-8-28 16:18:37 by GZH
                  (modified after loading)
Sample Info     :
                  OD-3 H:I=90:10 1.0ml/min 250nm
=====
```



Area Percent Report

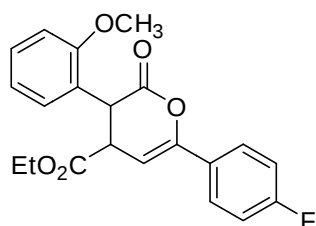
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [nAU]	Area %
1	13.444	BB	0.3877	56.44033	2.28126	0.9055
2	16.211	BB	0.4080	664.00891	25.07294	10.6531
3	17.985	BB	0.5524	5512.56543	151.60126	88.4414

Totals : 6233.01468 178.95546

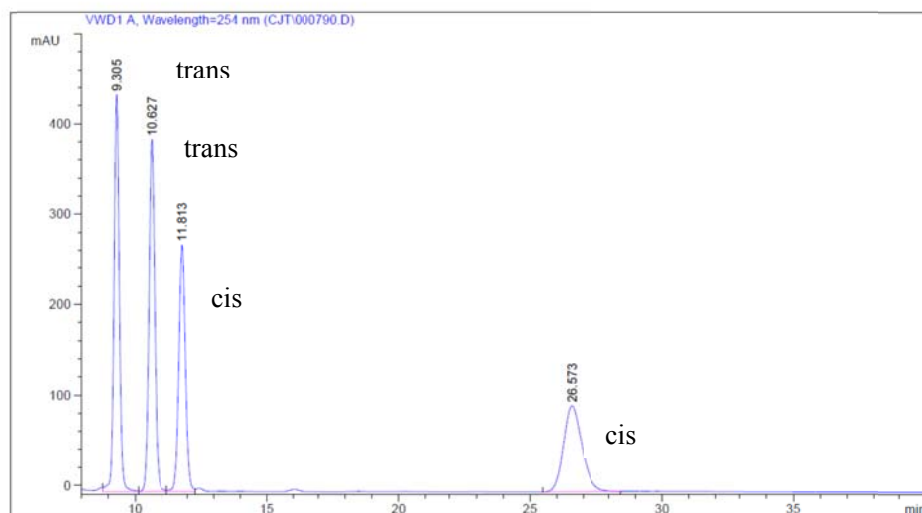
*** End of Report ***



rac-3ab *cis/trans* = 1:1

Data File C:\CHEM32\1\DATA\CJT\000790.D
Sample Name: cjt-mbh-711XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-10-28 14:47:03
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-28 12:57:23 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 13:09:06 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I/MeOH=80:10:10 mL/min 254 nm
=====
```



Area Percent Report

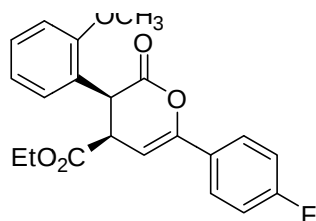
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.305	VV	0.2102	6050.67773	439.35370	28.3345
2	10.627	VV	0.2334	5943.51611	389.40335	27.8327
3	11.813	VV	0.2654	4706.00391	272.69302	22.0376
4	26.573	BB	0.7438	4654.24023	95.40834	21.7952

Totals : 2.13544e4 1196.85841

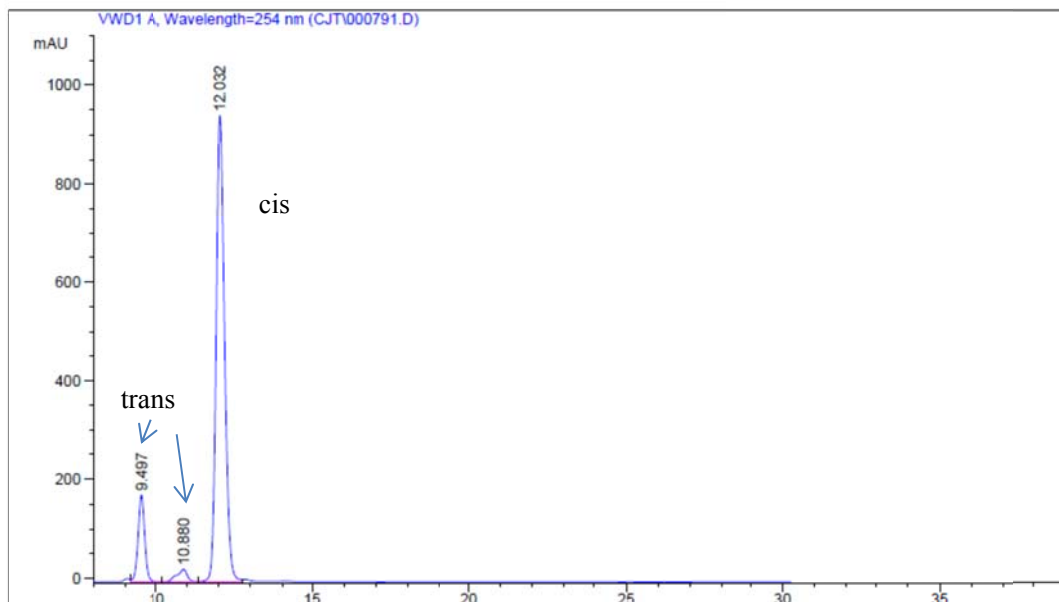
*** End of Report ***



3ab *cis/trans* = 10:1

Data File C:\CHEM32\1\DATA\CJT\000791.D
Sample Name: cjt-mbh-711

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                      Location : Vial 1
Injection Date  : 2014-10-28 15:34:30
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-28 12:57:23 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 13:12:25 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I/MeOH=80:10:10 1mL/min 254 nm
=====
```



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

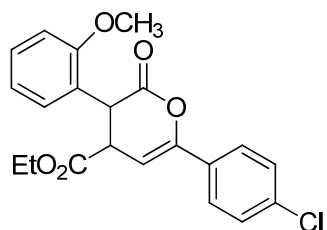
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU] %
-----|-----|-----|-----|-----|
1 9.497 VV 0.2215 2537.76318 175.07019 12.4061
2 10.880 VV 0.3268 601.44958 25.56925 2.9402
3 12.032 VV 0.2779 1.73166e4 944.73999 84.6537

Totals : 2.04559e4 1145.37943

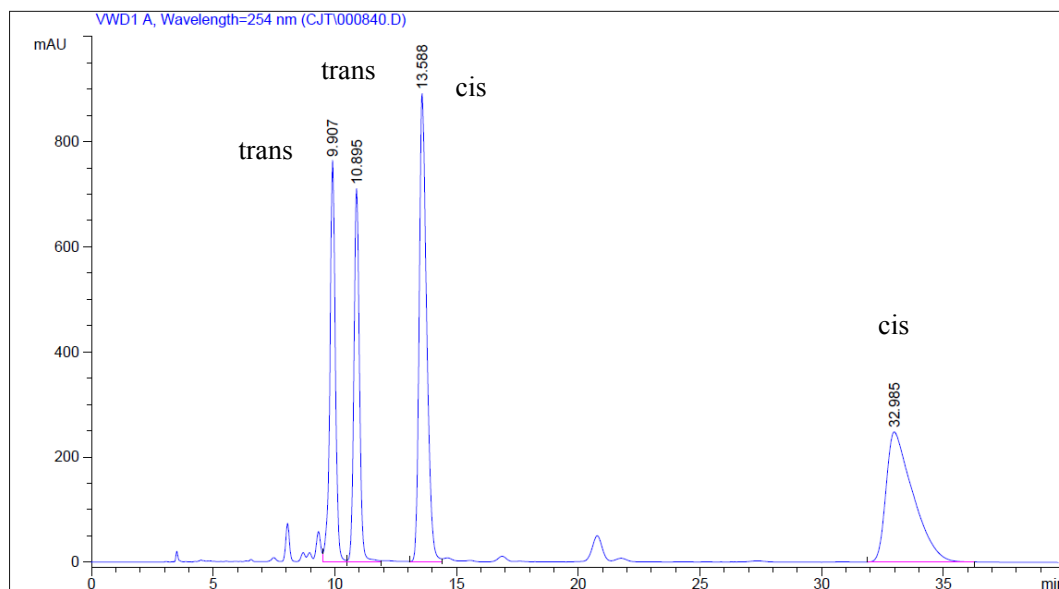
=====
*** End of Report ***
=====
```



rac-3ac *cis/trans* = 2:1

Data File C:\CHEM32\1\DATA\CJT\000840.D
Sample Name: CJT-MBH-692XIAOXUAN

```
=====
Acq. Operator   : CJT
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-11-15 11:56:54
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-15 10:56:39 by CJT
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 18:30:56 by ckq
                  (modified after loading)
Sample Info     : IA-H H:I/MeOH=80:10:10 1 mL/min 254 nm
=====
```



Area Percent Report

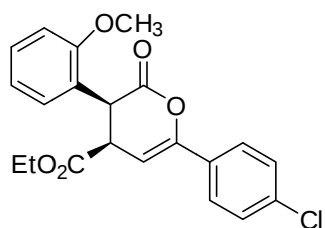
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	9.907	VV	0.2327	1.18031e4	763.89105	19.3574
2	10.895	VV	0.2388	1.11697e4	710.09094	18.3185
3	13.588	VV	0.3185	1.89833e4	890.84790	31.1330
4	32.985	BB	1.1166	1.90187e4	247.82388	31.1911

Totals : 6.09749e4 2612.65378

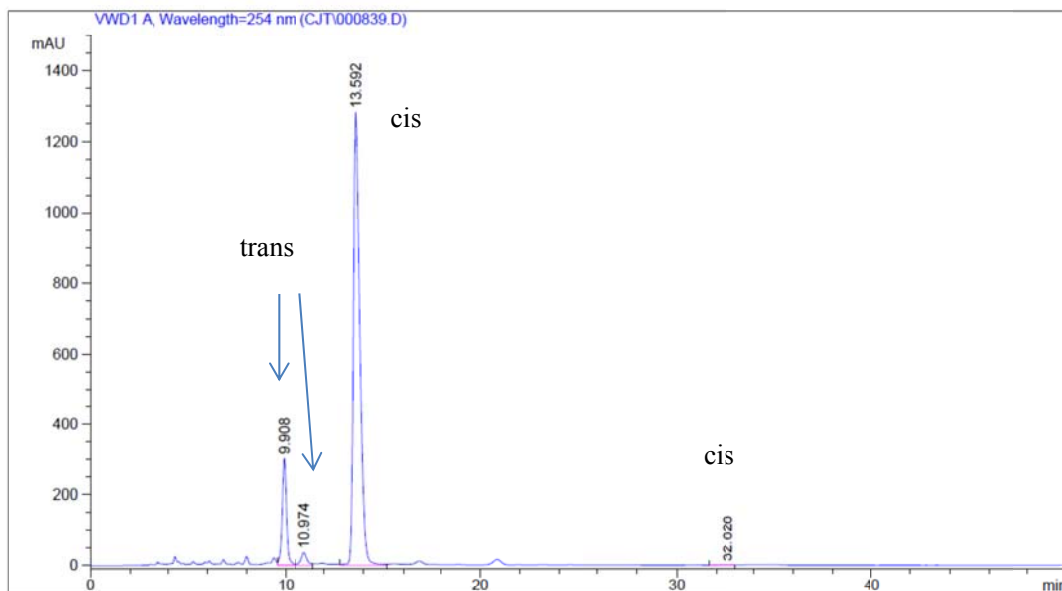
*** End of Report ***



3ac *cis/trans* = 10:1

Data File C:\CHEM32\1\DATA\CJT\000839.D
Sample Name: CJT-MBH-692

```
=====
Acq. Operator   : CJT
Acq. Instrument : Instrument 1
Injection Date  : 2014-11-15 10:59:30
Acq. Method    : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed   : 2014-11-15 10:56:39 by CJT
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed   : 2014-11-22 18:45:30 by ckq
                  (modified after loading)
Sample Info    : IA-H H:I/MeOH=80:10:10 1 mL/min 254 nm
=====
```



Area Percent Report

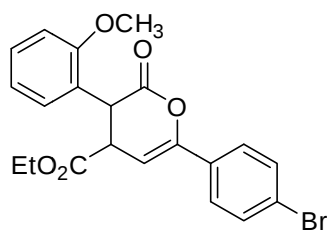
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	9.908	VV	0.2323	4675.60742	303.17447	13.6584
2	10.974	VV	0.3141	807.86011	37.25853	2.3599
3	13.592	VV	0.3343	2.87251e4	1281.78479	83.9120
4	32.626	VV	0.5592	23.84316	5.24752e-1	0.0697

Totals : 3.42324e4 1622.74254

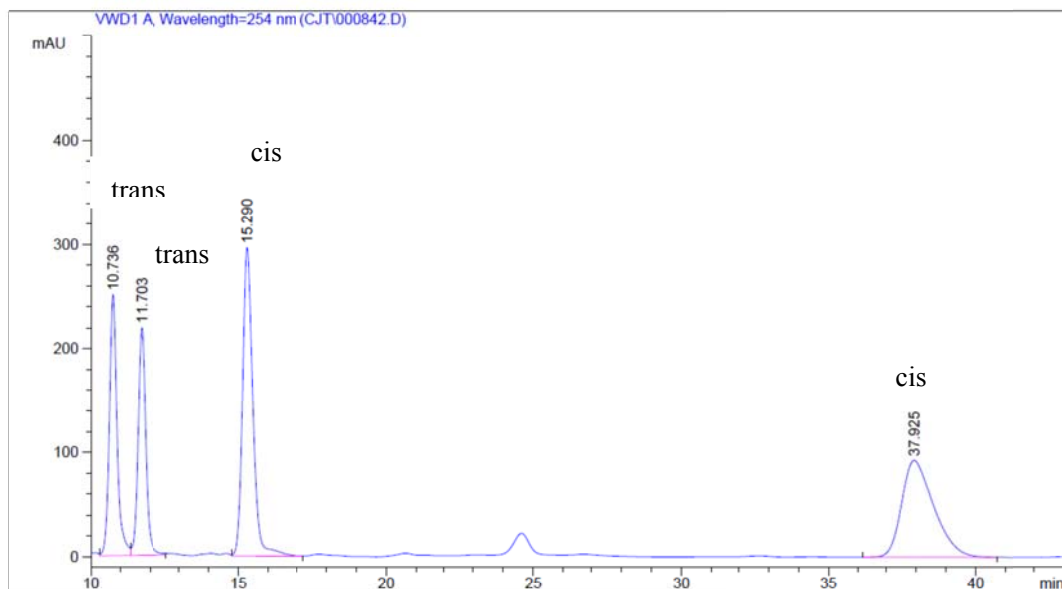
*** End of Report ***



rac-3ad *cis/trans* = 2:1

Data File C:\CHEM32\1\DATA\CJT\000842.D
Sample Name: CJT-MBH-722XIAOXUAN

```
=====
Acq. Operator   : CJT
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-11-15 13:33:35
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-15 10:56:39 by CJT
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 18:47:41 by ckq
                  (modified after loading)
Sample Info     : IA-H  H:I/MeOH=80:10:10 1 mL/min 254 nm
=====
```



Area Percent Report

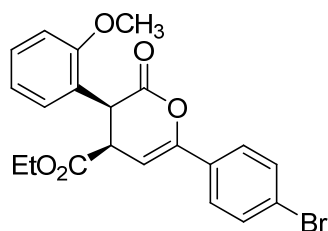
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	10.736	VV	0.2670	4483.65576	250.52625	19.9171
2	11.703	VB	0.2738	3935.37866	218.90436	17.4816
3	15.290	VB	0.3631	7148.25732	296.16669	31.7537
4	37.925	BB	1.1197	6944.30908	92.57652	30.8477

Totals : 2.25116e4 858.17381

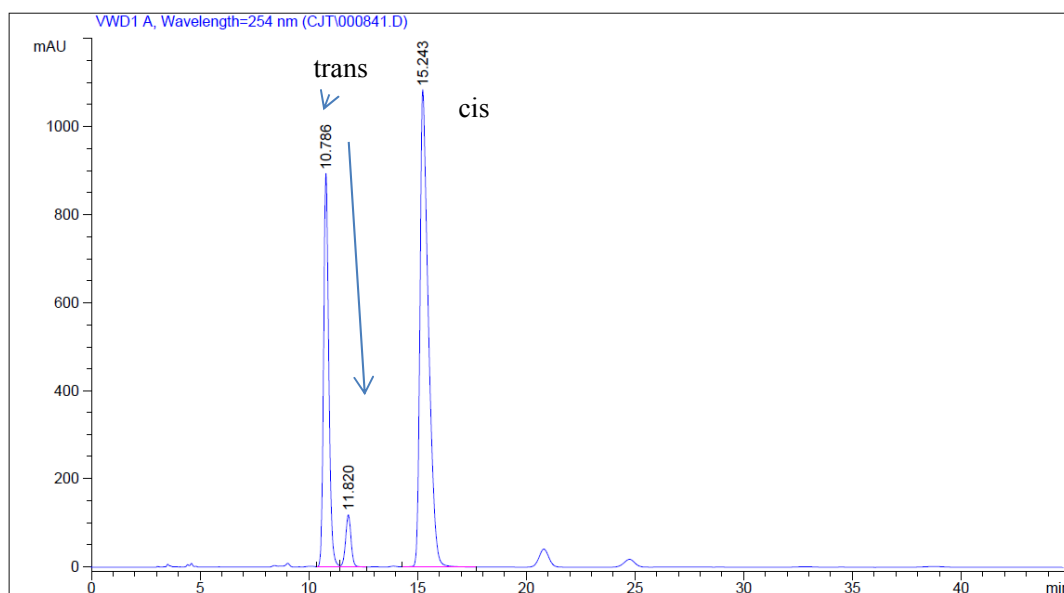
*** End of Report ***



3ad
cis/trans = 7:1

Data File C:\CHEM32\1\DATA\CJT\000841.D
 Sample Name: CJT-MBH-722

```
=====
Acq. Operator   : CJT
Acq. Instrument : Instrument 1                      Location : Vial 1
Injection Date  : 2014-11-15 12:45:47
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-15 10:56:39 by CJT
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 18:51:36 by ckq
                  (modified after loading)
Sample Info     : IA-H H:I/MeOH=80:10:10 1 mL/min 254 nm
=====
```



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

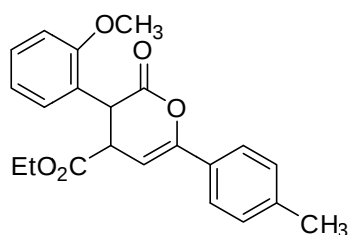
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	10.786	VV	0.2517	1.48247e4	893.87036	32.0991
2	11.820	VB	0.2697	2093.06323	118.72594	4.5320
3	15.243	VB	0.3967	2.92665e4	1083.29578	63.3689

Totals : 4.61843e4 2095.89207

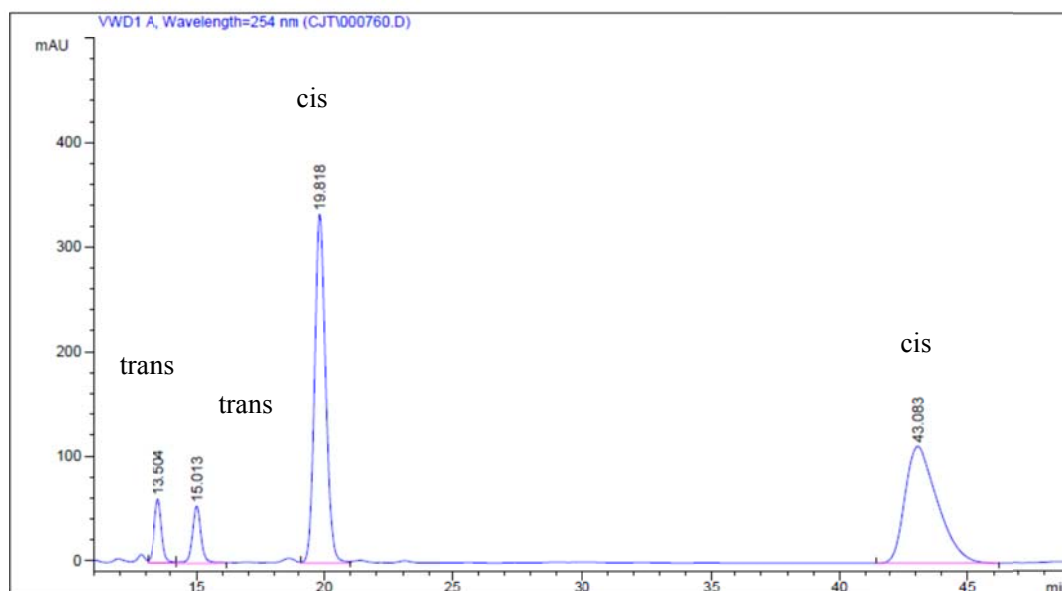
=====
 *** End of Report ***



rac-3ae *cis/trans* = 7:1

Data File C:\CHEM32\1\DATA\CJT\000760.D
Sample Name: cjt-mbh-693XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-10-22 15:24:43
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-22 15:21:04 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 14:01:49 by cjt
                  (modified after loading)
Sample Info     : IA-H H/I/MeOH=90:5:5 1.0 mL/min 254 nm
=====
```



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

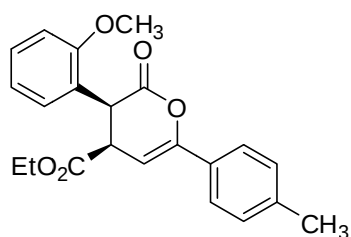
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [nAU]	Area %
1	13.504	VV	0.3256	1307.58142	61.03834	5.9275
2	15.013	VB	0.3572	1290.62793	54.61399	5.8507
3	19.818	VV	0.4470	9788.32227	333.78400	44.3722
4	43.083	BB	1.2863	9673.03027	111.45249	43.8496

Totals : 2.20596e4 560.88882

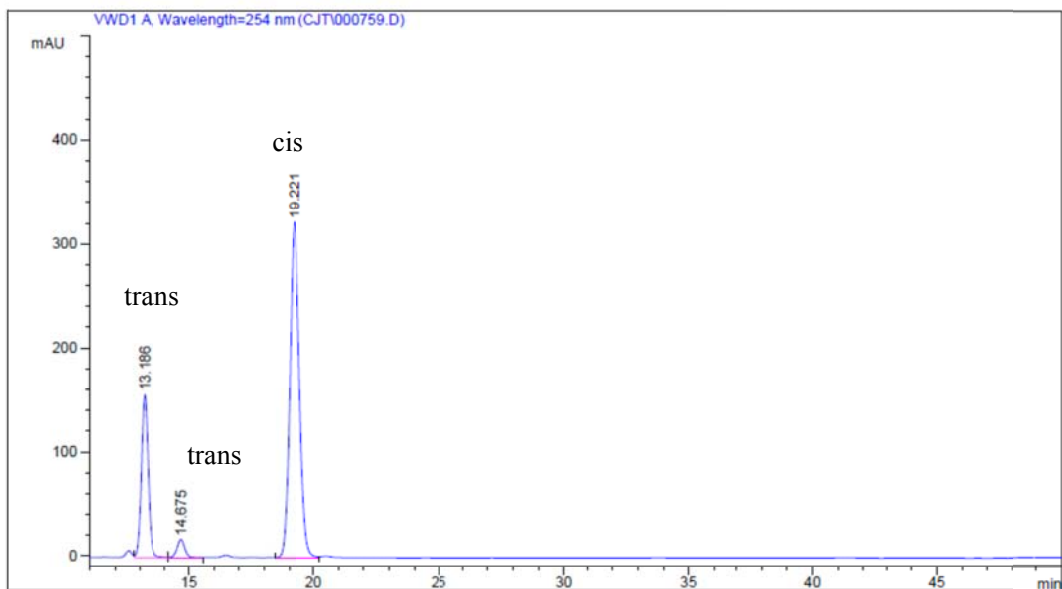
```
=====
*** End of Report ***
=====
```



3ae *cis/trans* = 6:1

Data File C:\CHEM32\1\DATA\CJT\000759.D
Sample Name: cjt-mbh-693

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-10-22 14:19:38
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-22 14:16:56 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 14:06:06 by cjt
                  (modified after loading)
Sample Info     : IA-H H/I/MeOH=90:5:5 1.0 mL/min 254 nm
=====
```



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

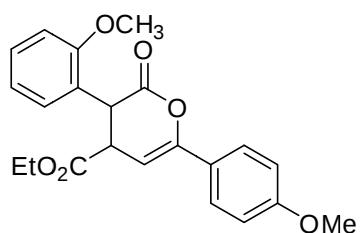
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU] %
----|-----|-----|-----|-----|-----|
1 13.186 VV 0.2978 3013.91504 156.30545 24.2877
2 14.675 VB 0.3301 378.42358 17.55454 3.0495
3 19.221 BV 0.4249 9016.87695 322.76044 72.6627

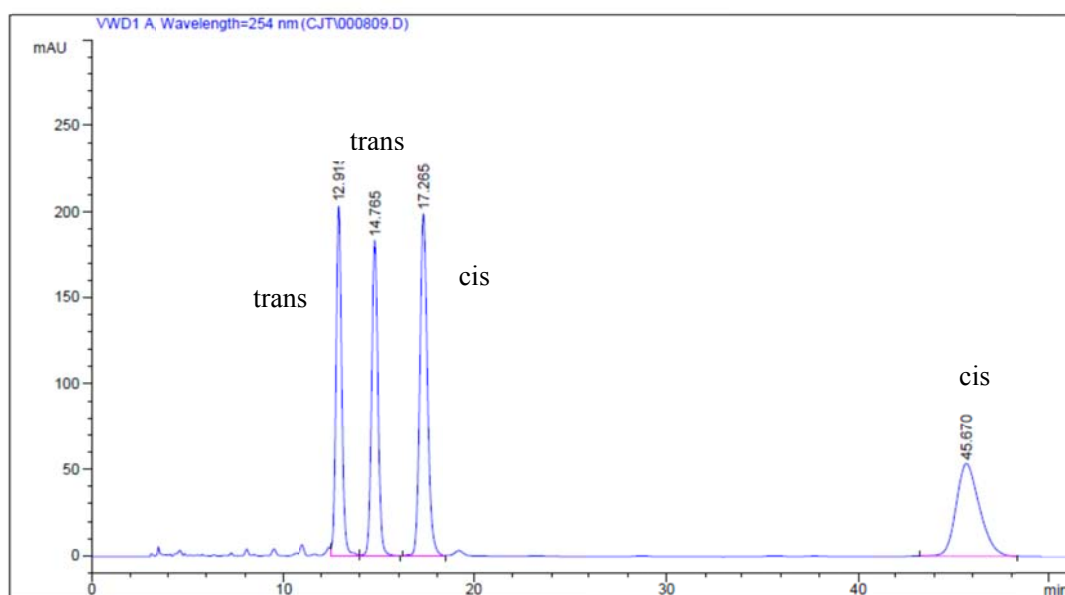
Totals :                      1.24092e4 496.62043

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



rac-3af cis/trans = 1:1

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                      Location : Vial 1
Injection Date  : 2014-10-29 21:02:35
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 19:50:49 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 22:20:29 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I/MeOH=80:10:10 1mL/min 254 nm
=====
```



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

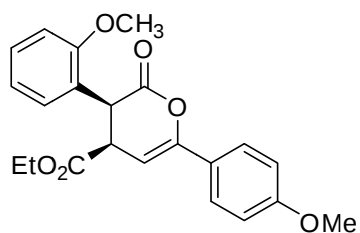
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU] %
----|-----|-----|-----|-----|-----|
1 12.915 VV 0.3114 4167.15479 203.75949 22.8077
2 14.765 VB 0.3466 4175.19580 183.75983 22.8517
3 17.265 BB 0.4138 5422.25146 199.11243 29.6771
4 45.670 BB 1.2684 4506.24268 53.63871 24.6636

Totals :                      1.82708e4 640.27046

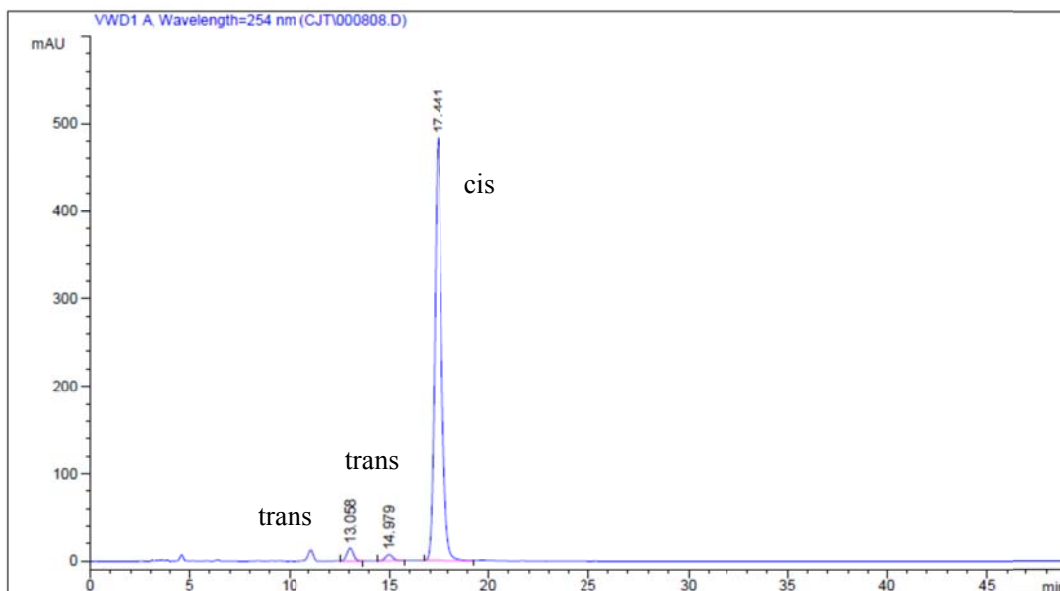
=====
*** End of Report ***
=====
```



3af *cis/trans* = 10:1

Data File C:\CHEM32\1\DATA\CJT\000808.D
Sample Name: cjt-mbh-710

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-10-29 20:11:28
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 19:50:49 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 22:23:03 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I/MeOH=80:10:10 1mL/min 254 nm
=====
```



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

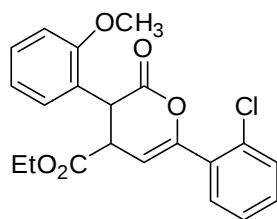
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

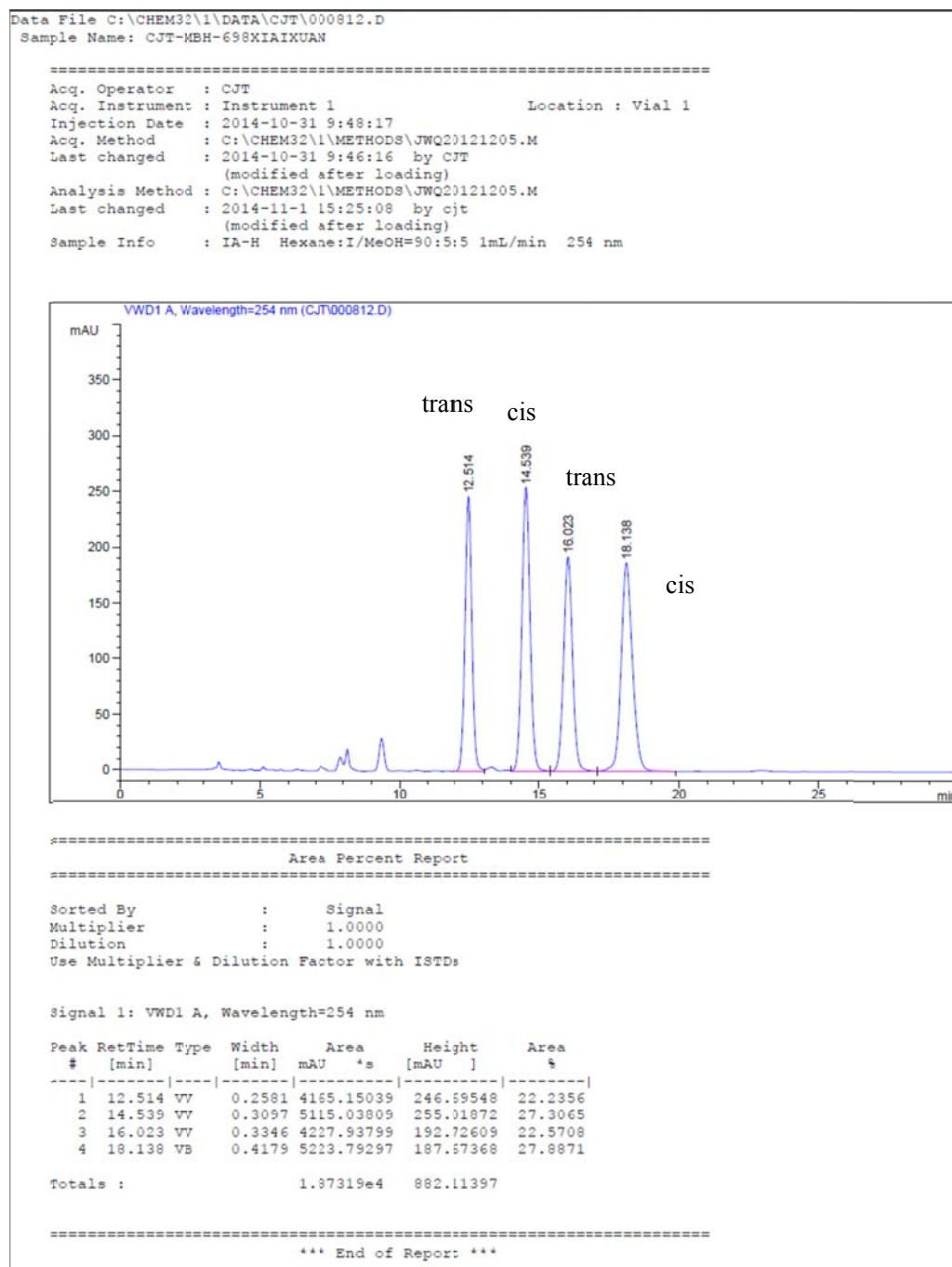
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	13.058	VB	0.3440	327.10480	14.70362	2.2691	
2	14.979	BB	0.3824	175.86322	7.09499	1.2199	
3	17.441	BB	0.4378	1.39128e4	483.22696	96.5110	

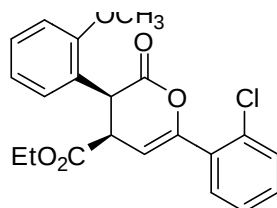
Totals : 1.44158e4 505.02557

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



rac-3ag *cis/trans* = 1:1

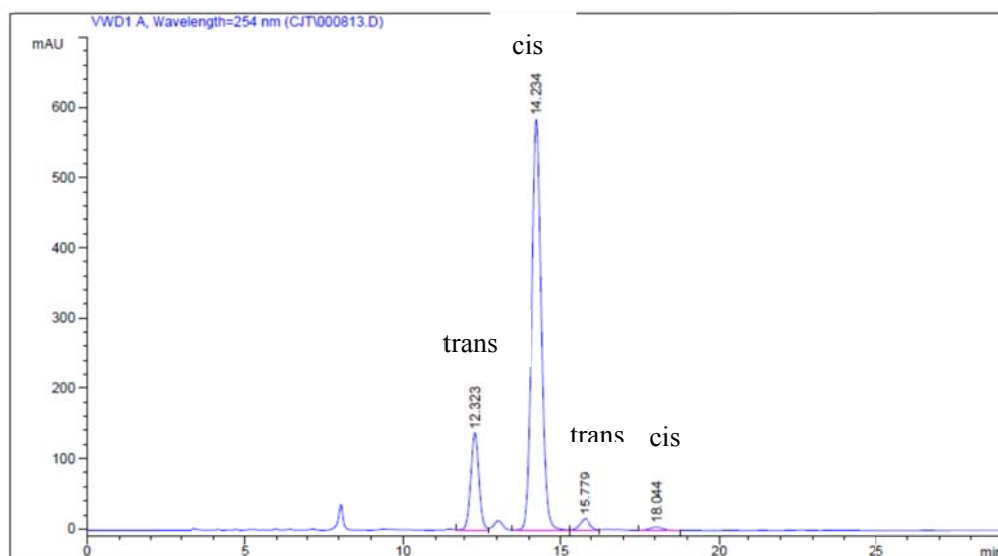




3ag *cis/trans* = 6:1

Data File C:\CHEM32\1\DATA\CJT\000813.D
Sample Name: CJT-MBH-698

```
=====
Acq. Operator   : CJT
Acq. Instrument : Instrument 1
Injection Date  : 2014-10-31 10:22:44
Acq. Method     : C:\CHEM32\1\METHODS\JWQ\0121205.M
Last changed    : 2014-10-31 9:46:16 by CJT
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ\0121205.M
Last changed    : 2014-11-1 15:28:58 by cjt
                  (modified after loading)
Sample Info     : IA-H Hexane:I/MeOH=90:5 1mL/min 254 nm
=====
```



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

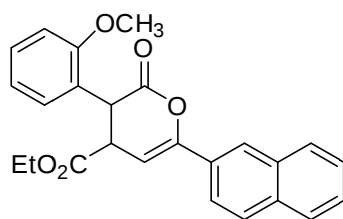
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

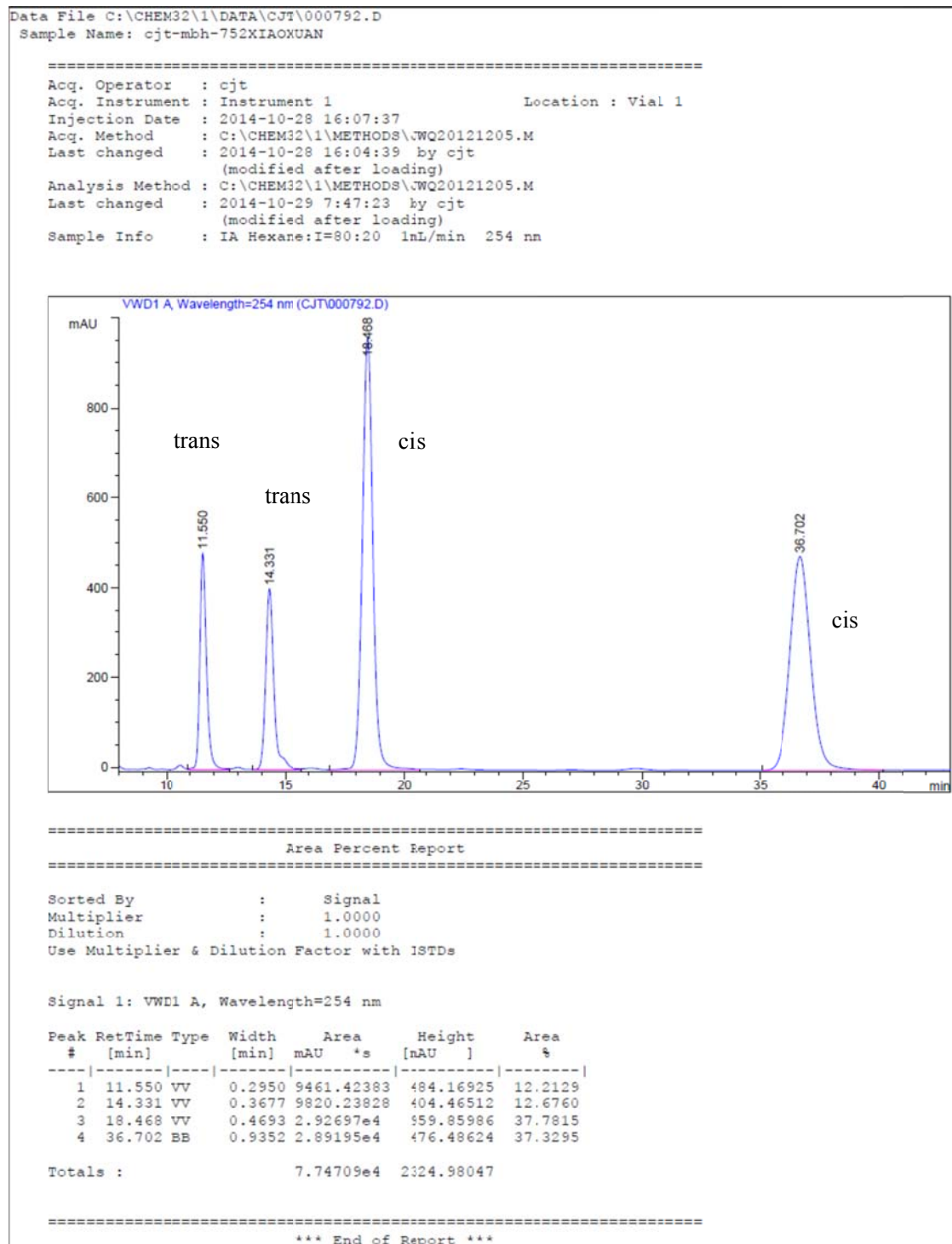
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	12.323	VV	0.2750	2470.68408	138.54913	16.2442	
2	14.234	VV	0.3191	1.21970e4	584.64868	80.1924	
3	15.779	VV	0.3697	404.04681	16.69590	2.6565	
4	18.044	BB	0.4508	137.93817	4.73090	0.9069	

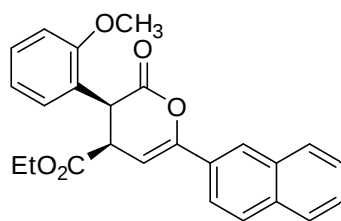
Totals : 1.52097e4 744.62462

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



rac-3ah *cis/trans*= 3:1

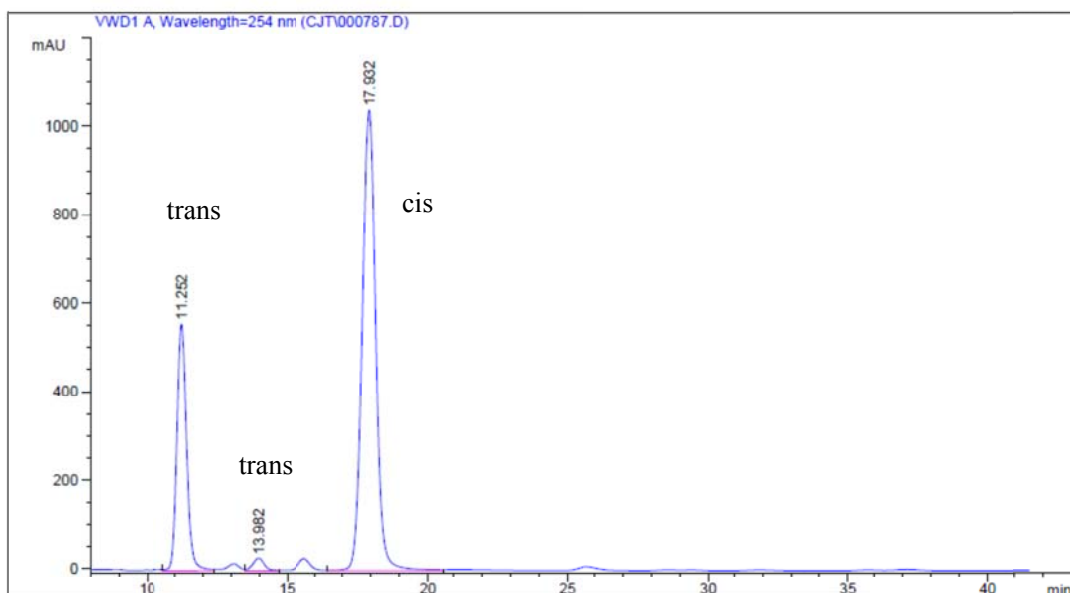




3ah *cis/trans* = 3:1

Data File C:\CHEM32\1\DATA\CJT\000787.D
Sample Name: cjt-mbh-752

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                      Location : Vial 1
Injection Date  : 2014-10-28 12:15:17
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-28 11:18:08 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 8:01:18 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I=80:20 1nL/min 254 nm
=====
```



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

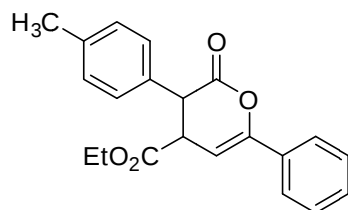
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	11.252	VB	0.3682	1.32851e4	557.61127	27.3758	
2	13.982	VB	0.4331	799.08057	28.40035	1.6466	
3	17.932	VB	0.5117	3.44444e4	1039.71582	70.9776	

Totals : 4.85286e4 1625.72744

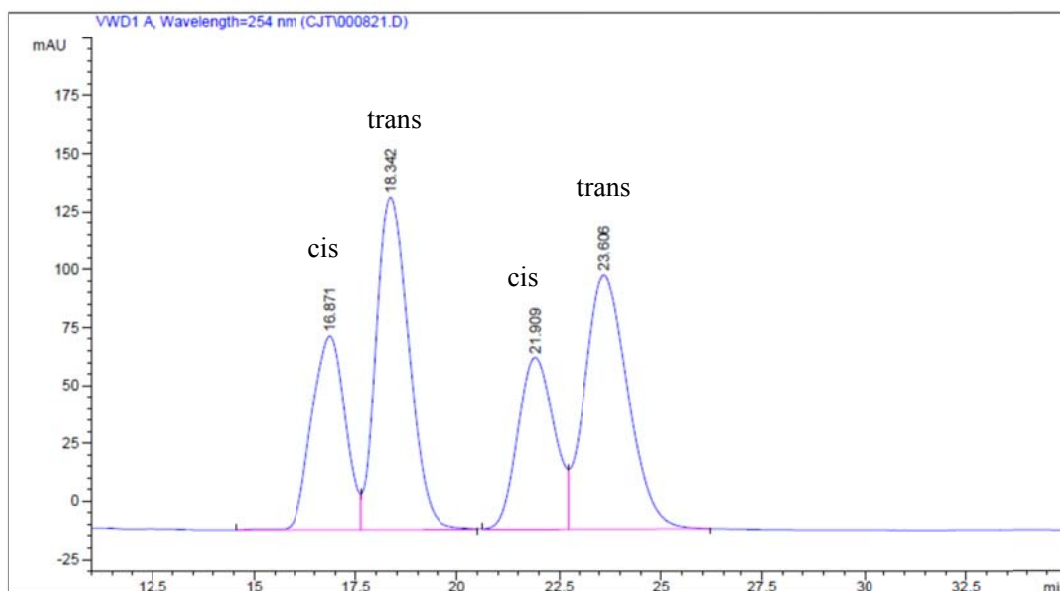
```
=====
*** End of Report ***
=====
```



rac-3ca
cis/trans = 1:2

Data File C:\CHEM32\1\DATA\CJT\000821.D
Sample Name: cjt-mbh-786xiaox

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-11-1 10:28:54
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-1 10:27:29  by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-1 15:30:48  by cjt
                  (modified after loading)
Sample Info     : OD H:I=90:10 1mL/min 254 nm
=====
```



Area Percent Report

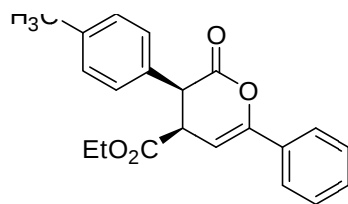
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [nAU]	Area %
1	16.871	BV	0.9318	4757.10449		83.60139	18.7354
2	18.342	VB	0.8825	7986.72607		143.44395	31.4550
3	21.909	BV	0.9663	4571.52148		74.19855	18.0045
4	23.606	VB	1.1336	8075.63037		109.56689	31.8051

Totals : 2.53910e4 410.81079

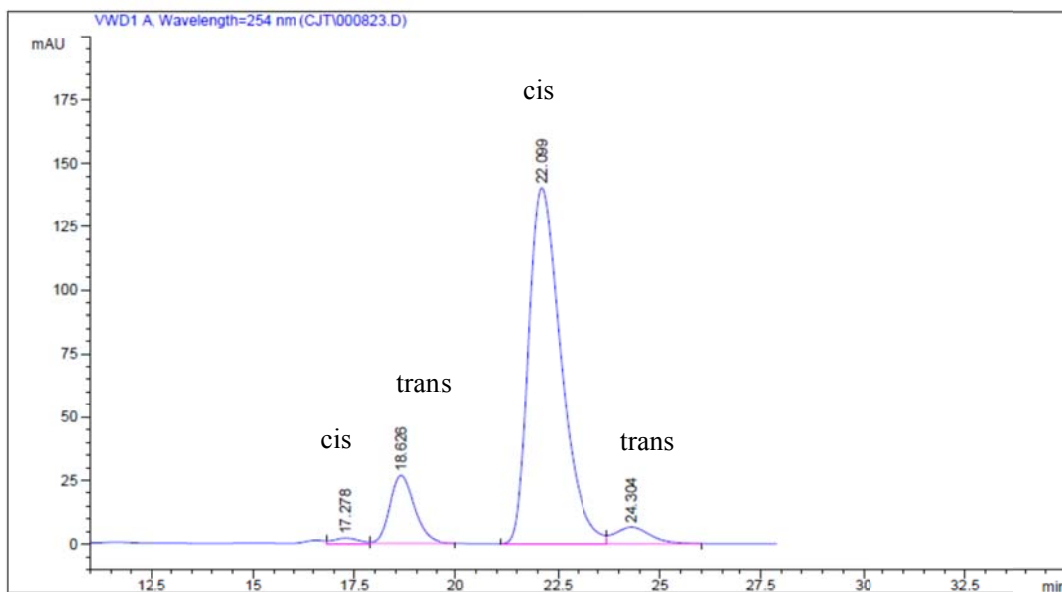
*** End of Report ***



3ca
cis/trans = 6:1

Data File C:\CHEM32\1\DATA\CJT\000823.D
Sample Name: cjt-mbh-786

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                      Location : Vial 1
Injection Date  : 2014-11-1 11:50:43
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-1 11:49:38  by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-1 15:35:20  by cjt
                  (modified after loading)
Sample Info     : OD H:I=95:5 1mL/min 254 nm
=====
```



Area Percent Report

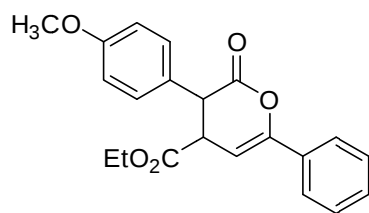
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	17.278	VV	0.6477	87.95447	2.06788	0.9447
2	18.626	VB	0.6551	1122.71045	26.61966	12.0585
3	22.099	BV	0.8453	7710.74951	140.23221	82.8173
4	24.304	VB	0.8785	389.14066	6.55918	4.1796

Totals : 9310.55508 175.47892

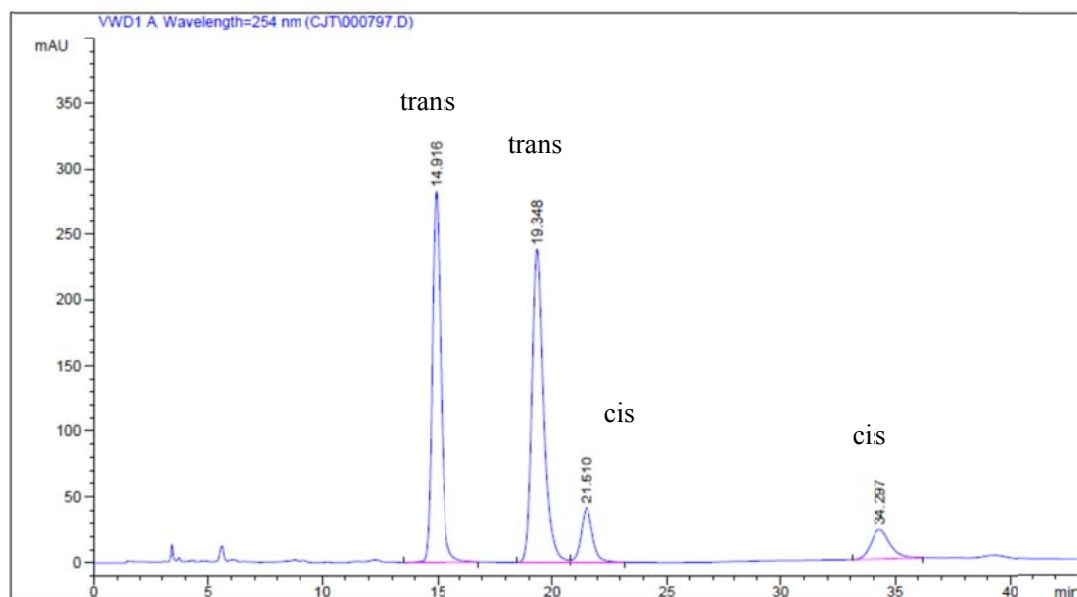
*** End of Report ***



rac-3da *cis/trans*=1:5

Data File C:\CHEM32\1\DATA\CJT\000797.D
Sample Name: cjt-mbh-766XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-10-29 10:56:29
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 10:52:30 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 13:01:26 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I=80:20 1ml/min 254 nm
=====
```



Area Percent Report

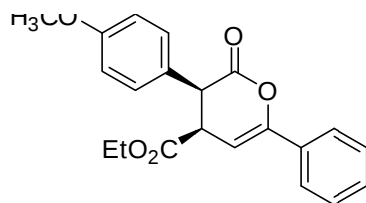
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [nAU]	Area %
1	14.916	VB	0.4111	7635.03564	282.73453	40.4779
2	19.348	BV	0.5235	8309.60547	238.16867	44.0542
3	21.510	VB	0.5664	1556.52783	41.70806	8.2521
4	34.297	BB	0.9130	1361.05676	22.48203	7.2158

Totals : 1.88622e4 585.09329

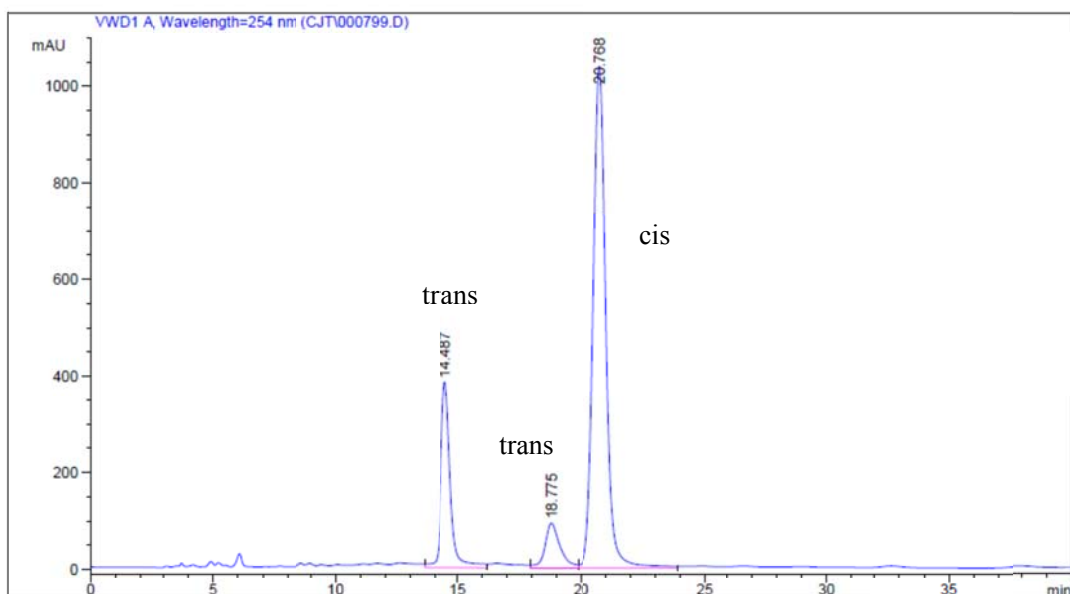
*** End of Report ***



3da
cis/trans = 3:1

Data File C:\CHEM32\1\DATA\CJT\000799.D
Sample Name: cjt-mbh-766

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-10-29 12:19:28
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 10:52:30 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-29 13:03:52 by cjt
                  (modified after loading)
Sample Info     : IA Hexane:I=80:20 1ml/min 254 nm
=====
```



Area Percent Report

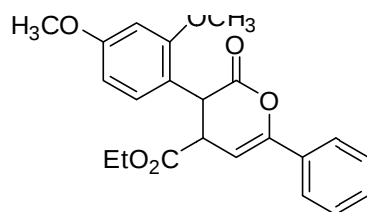
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	14.487	VV	0.4193	1.08515e4	384.71631	21.0244	
2	18.775	VV	0.6052	3836.57202	92.13644	7.4332	
3	20.768	VB	0.5492	3.69258e4	1037.41882	71.5424	

Totals : 5.16138e4 1514.27158

*** End of Report ***

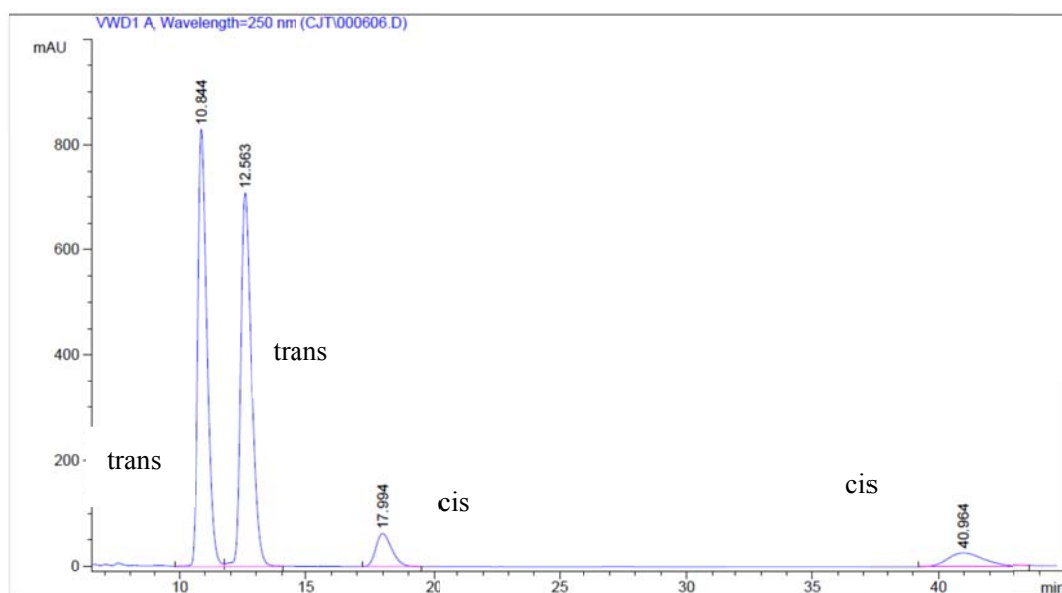


rac-3ea
cis/trans = 1:8

Data File C:\CHEM32\1\DATA\CJT\000606.D
Sample Name: cjt-mbh-724

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1           Location : Vial 1
Injection Date  : 2014-6-5 15:13:16
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-6-5 15:11:16 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-8-28 16:59:45 by GZH
                  (modified after loading)
Sample Info     : AD-H H:I=60:40

                  1.0ml/min 250nm
=====
```



Area Percent Report

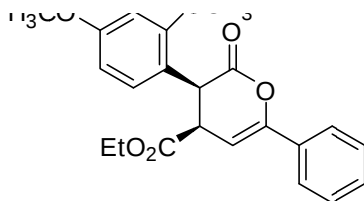
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [nAU]	Area %
1	10.844	VV	0.3857	2.08391e4	831.19727	44.2164
2	12.563	VB	0.4488	2.09081e4	709.23578	44.3628
3	17.994	BB	0.6640	2695.92896	62.78889	5.7202
4	40.964	BB	1.5215	2686.66260	26.47893	5.7006

Totals : 4.71297e4 1629.70086

*** End of Report ***

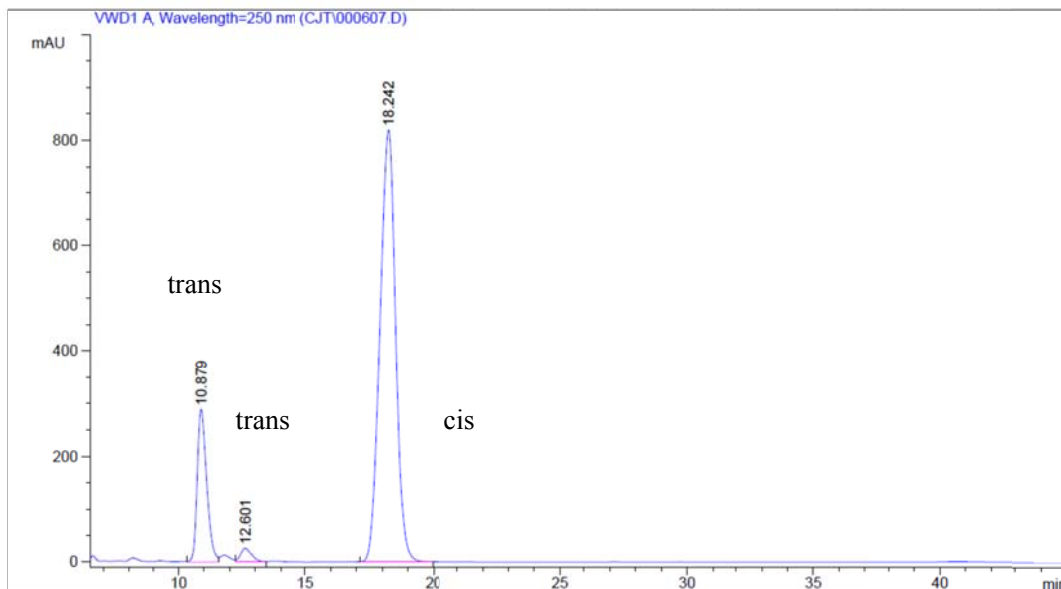


3ea *cis/trans* = 6:1

Data File C:\CHEM32\1\DATA\CJT\000607.D
Sample Name: cjt-mbh-719

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-6-5 15:59:02
Acq. Method     : C:\CHEM32\1\METHODS\WQ20121205.M
Last changed    : 2014-6-5 15:11:16 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WQ20121205.M
Last changed    : 2014-8-28 17:08:59 by GZH
                  (modified after loading)
Sample Info     : AD-H H:I=60:40

                  1.0ml/min 250nm
=====
```



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

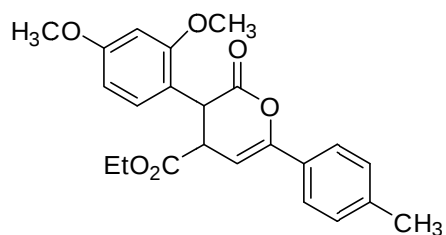
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [nAU] %
-----|-----|-----|-----|-----|-----|
1 10.879 BV 0.3946 7499.01660 190.27118 17.4582
2 12.601 VV 0.4663 823.27356 27.00923 1.9166
3 18.242 BB 0.6557 3.46319e4 820.19635 80.6252

Totals : 4.29542e4 1137.47676

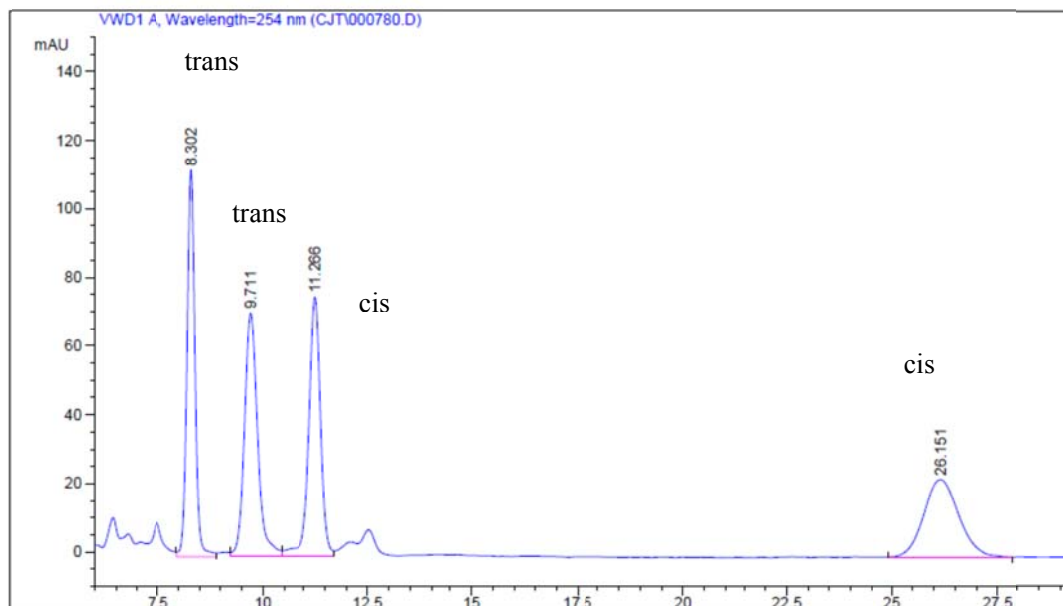
=====
*** End of Report ***
=====
```



rac-3ee *cis:trans* = 1:1

Data File C:\CHEM32\1\DATA\CJT\000780.D
Sample Name: cjt-mbh-721XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-10-25 15:58:28
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 15:42:14 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 19:07:46 by cjt
                  (modified after loading)
Sample Info     : IA-H H/I/MeOH=60:20:20 1.0 mL/min 254 nm
=====
```



Area Percent Report

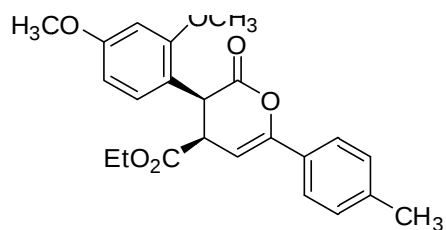
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.302	VV	0.1993	1475.63599	112.70657	25.3829
2	9.711	VV	0.3325	1544.56177	70.97509	26.5685
3	11.266	VV	0.2942	1474.04443	75.68402	25.3555
4	26.151	BB	0.8883	1319.25745	22.77850	22.6930

Totals : 5813.49963 282.14418

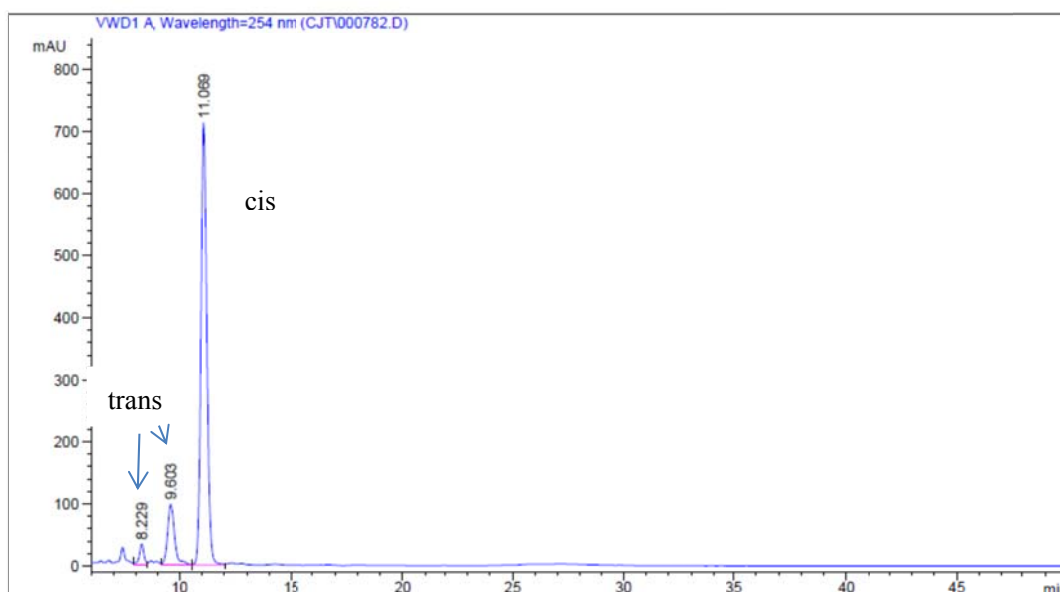
*** End of Report ***



3ee *cis:trans* = 20:1

Data File C:\CHEM32\1\DATA\CJT\000782.D
Sample Name: cjt-mbh-721

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-10-25 17:53:47
Acq. Method     : C:\CHEM32\1\METHODS\WQ20121205.M
Last changed    : 2014-10-25 17:31:52 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WQ20121205.M
Last changed    : 2014-10-25 19:09:23 by cjt
                  (modified after loading)
Sample Info     : IA-H H/1/MeOH=60:20:20 1.0 mL/min 254 nm
=====
```



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

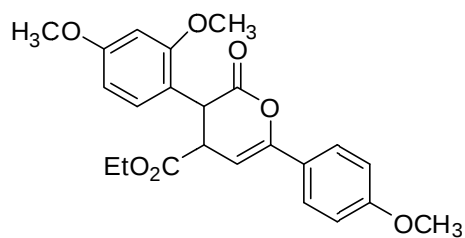
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	8.229	VV	0.2197	490.84866	33.65329	3.1427
2	9.603	VV	0.3314	2153.08643	98.23335	13.7853
3	11.069	VV	0.2793	1.29747e4	712.97015	83.0719

Totals : 1.56187e4 844.85680

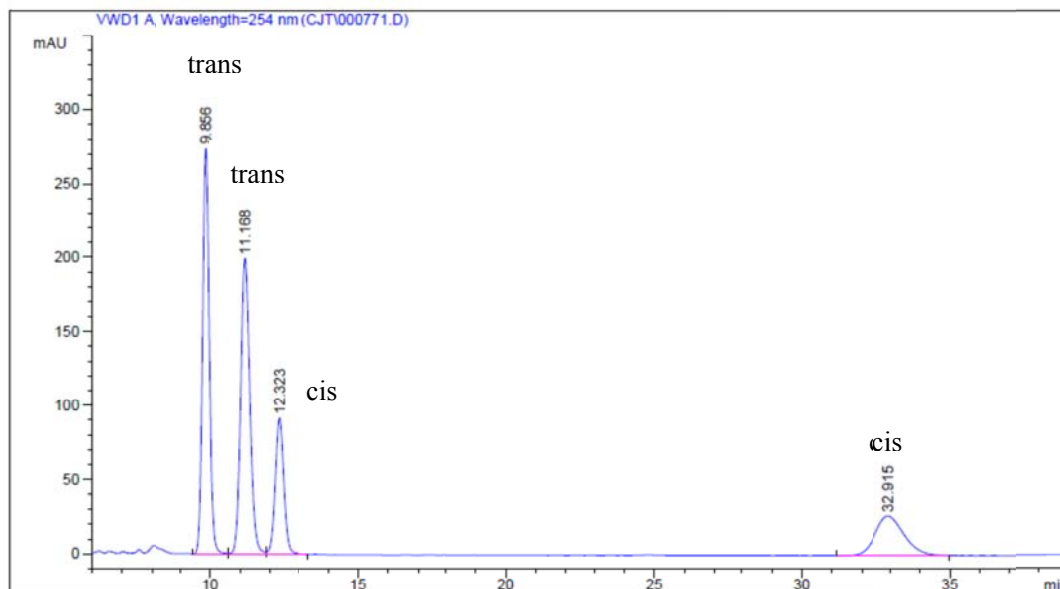
=====
*** End of Report ***



rac-3ef *cis:trans* = 1:3

Data File C:\CHEM32\1\DATA\CJT\000771.D
Sample Name: cjt-mbh-728xiaoxuan

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-10-25 9:06:50
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 8:53:57 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 14:08:35 by cjt
                  (modified after loading)
Sample Info     : IA-H H/I/MeOH=60:20:20 1.0 mL/min 254 nm
=====
```



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

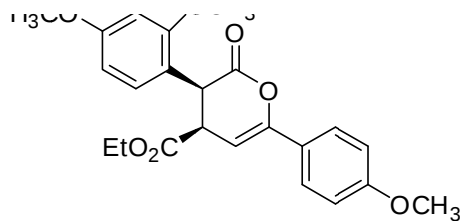
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU] %
-----|-----|-----|-----|-----|-----|
1 9.856 VV 0.2406 4286.94434 274.27359 35.0512
2 11.168 VV 0.3284 4271.32959 199.49014 34.9236
3 12.323 VB 0.3108 1842.64734 91.43143 15.0660
4 32.915 VB 1.0594 1829.58423 26.67376 14.9592

Totals : 1.22305e4 591.86892

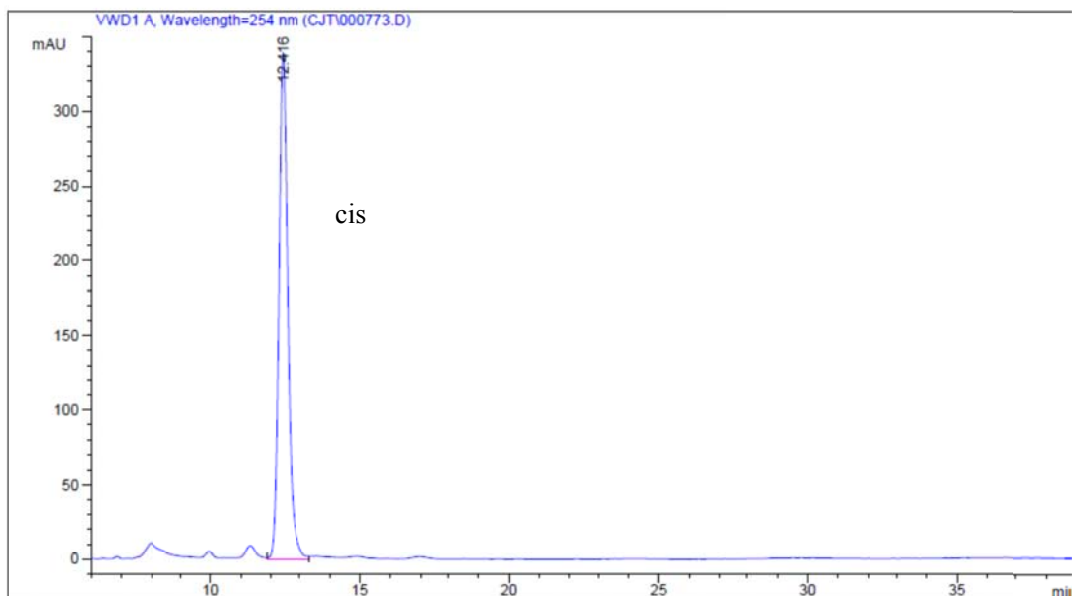
=====
*** End of Report ***
=====
```



3ef *cis:trans* >20:1

Data File C:\CHEM32\1\DATA\CJT\000773.D
Sample Name: cjt-mbh-728

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-10-25 10:31:53
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 8:53:57 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-10-25 14:08:35 by cjt
                  (modified after loading)
Sample Info     : IA-H H/I/MeOH=60:20:20 1.0 mL/min 254 nm
=====
```



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

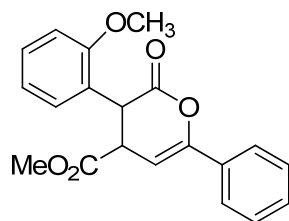
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU] *s [mAU] %
-----|-----|-----|-----|-----|
1 12.416 VV 0.3199 7181.74414 339.11667 100.0000

Totals : 7181.74414 339.11667

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

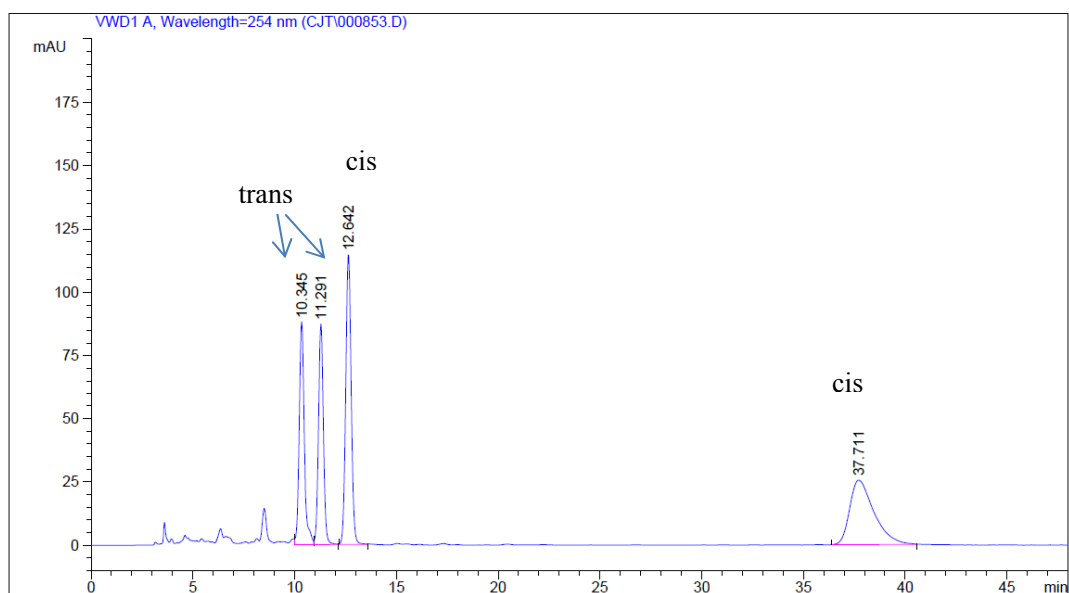


rac-3ai *cis:trans* = 1.5:1

Data File C:\CHEM32\1\DATA\CJT\000853.D

Sample Name: cjt-mbh-924XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-11-21 18:51:14
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-21 18:41:42 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 19:08:07 by ckq
                  (modified after loading)
Sample Info     : IA H:I:MeOH=80:10:10 1.0mL/min 254 nm
=====
```



Area Percent Report

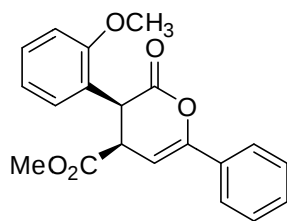
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	10.345	VV	0.2667	1531.76111	88.17112	21.1899	
2	11.291	VB	0.2598	1464.83020	87.31047	20.2640	
3	12.642	BB	0.2869	2129.82373	114.48811	29.4633	
4	37.711	BB	1.1991	2102.31470	25.38579	29.0828	

Totals : 7228.72974 315.35549

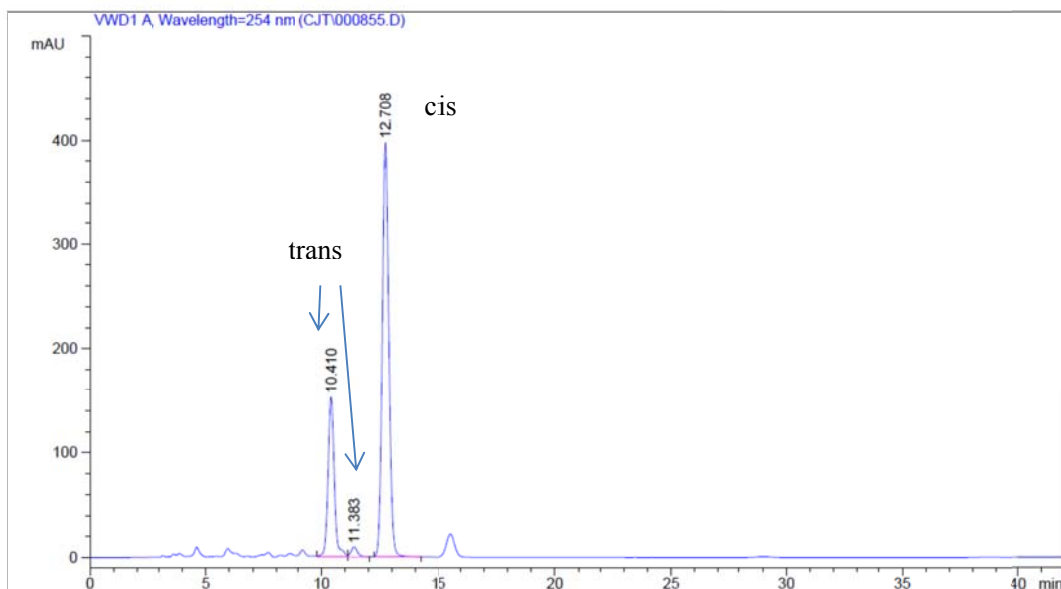
*** End of Report ***



3ai *cis:trans* = 10:1

Data File C:\CHEM32\1\DATA\CJT\000855.D
Sample Name: cjt-mbh-924

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-11-21 20:45:55
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-21 18:41:42 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 19:11:09 by ckq
                  (modified after loading)
Sample Info     : IA H:I:MeOH=80:10:10 1.0mL/min 254 nm
=====
```



Area Percent Report

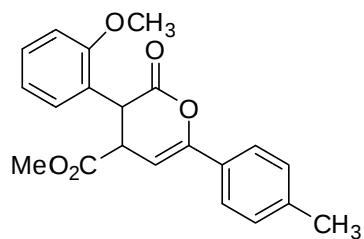
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	10.410	VV	0.2857	2832.38599	153.12532	26.4218	
2	11.383	VB	0.2949	189.62984	9.70689	1.7690	
3	12.708	BB	0.2987	7697.84668	397.57883	71.8092	

Totals : 1.07199e4 560.41104

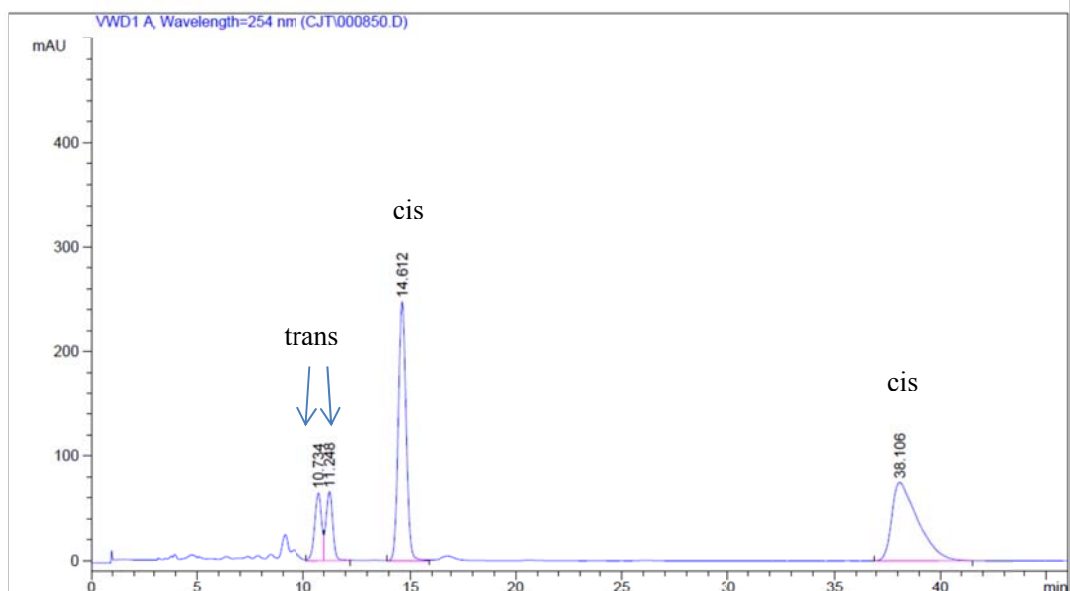
*** End of Report ***



rac-3aj cis:trans = 4:1

Data File C:\CHEM32\1\DATA\CJT\000850.D
Sample Name: cjt-mbh-920XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-11-21 16:14:38
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-21 16:11:02 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 18:59:43 by ckq
                  (modified after loading)
Sample Info     : IA H:I:MeOH=80:10:10 1.0mL/min 254 nm
=====
```



Area Percent Report

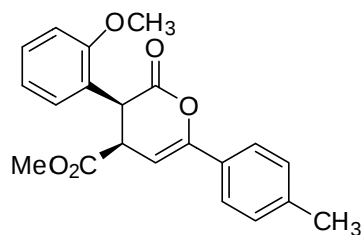
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [nAU]	Area %
1	10.734	VV	0.3439	1434.27979		64.50635	9.3204
2	11.248	VB	0.3401	1442.33716		65.82011	9.3728
3	14.612	VB	0.3893	6216.86572		247.39583	40.3992
4	38.106	BB	1.1689	6295.11182		74.74553	40.9076

Totals : 1.53886e4 452.46781

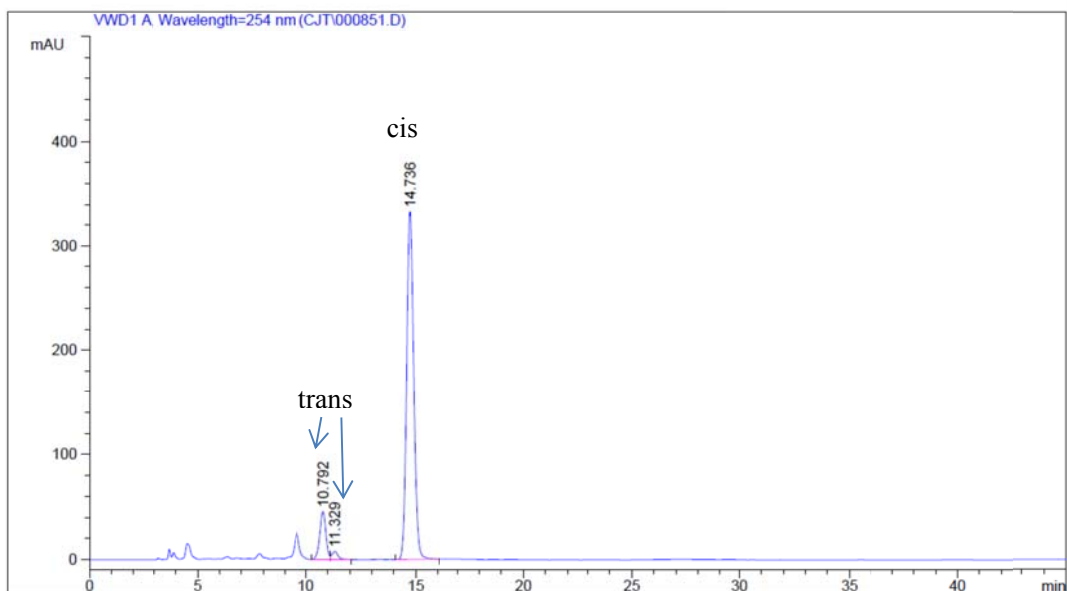
*** End of Report ***



3aj *cis:trans* = 10:1

Data File C:\CHEM32\1\DATA\CJT\000851.D
Sample Name: cjt-mbh-920

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-11-21 17:03:10
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-21 16:11:02 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 19:02:08 by ckq
                  (modified after loading)
Sample Info     : IA H:I:MeOH=80:10:10 1.0mL/min 254 nm
=====
```



Area Percent Report

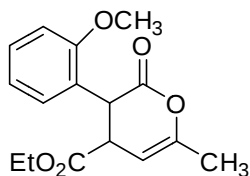
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	10.792	VV	0.2996	896.06219	46.09142	10.6277	
2	11.329	VB	0.2992	153.64058	7.71815	1.8222	
3	14.736	VB	0.3427	7381.70752	333.52679	87.5501	

Totals : 8431.41029 387.33636

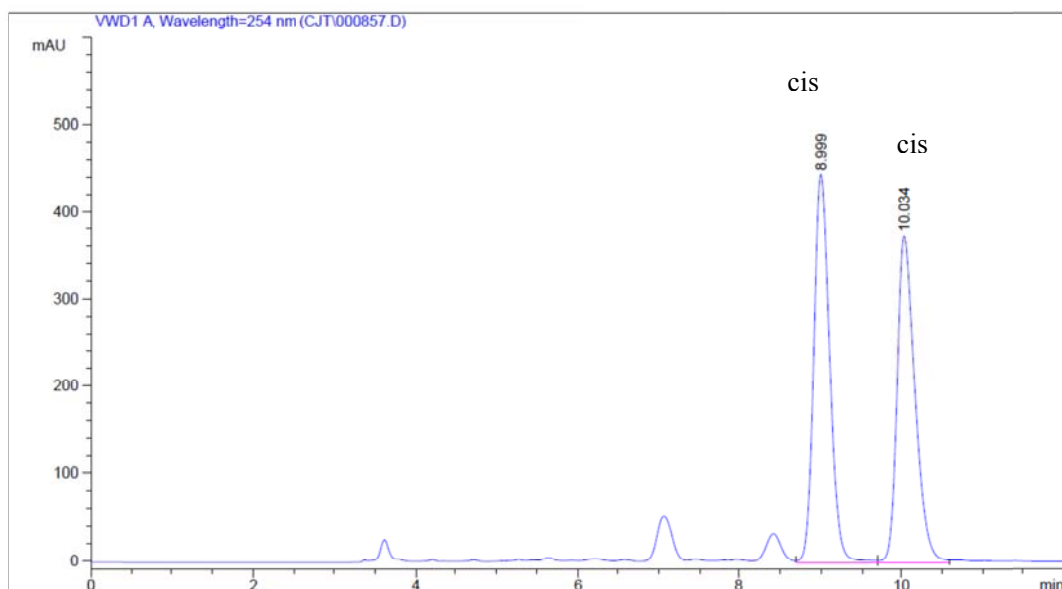
*** End of Report ***



rac-3ak cis:trans > 20:1

Data File C:\CHEM32\1\DATA\CJT\000857.D
Sample Name: cjt-mbh-916XIAOXUAN

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 2014-11-21 21:59:23
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-21 21:28:16 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 18:56:34 by ckq
                  (modified after loading)
Sample Info     : IA H:I:MeOH=90:5:5 1.0mL/min 254 nm
=====
```



Area Percent Report

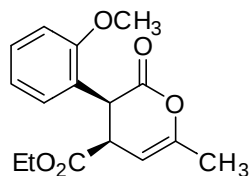
```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	8.999	VV	0.2055	5959.46094		445.56857	51.3304
2	10.034	VV	0.2287	5650.55127		374.04083	48.6696

Totals : 1.16100e4 819.60941

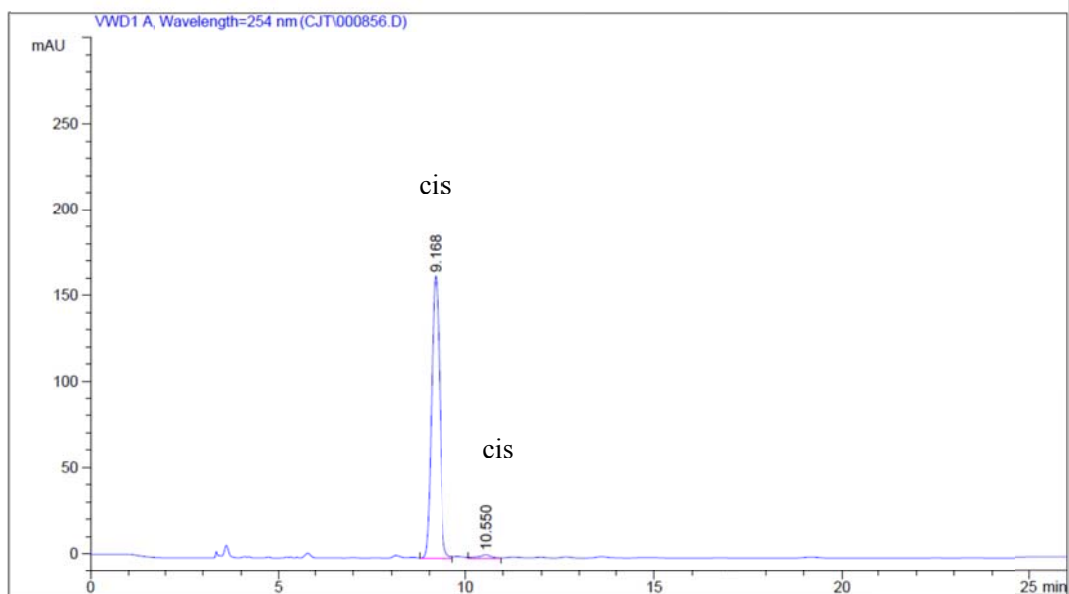
*** End of Report ***



3ak *cis:trans* > 20:1

Data File C:\CHEM32\1\DATA\CJT\000856.D
Sample Name: cjt-mbh-916

```
=====
Acq. Operator   : cjt
Acq. Instrument : Instrument 1                Location : Vial 1
Injection Date  : 2014-11-21 21:31:20
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-21 21:28:16 by cjt
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2014-11-22 18:58:11 by ckq
                  (modified after loading)
Sample Info     : IA H:I:MeOH=90:5:5 1.0mL/min 254 nm
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.168	VV	0.2467	2560.23730	163.57327	98.2915
2	10.550	VV	0.3333	44.50246	1.92857	1.7085

Totals : 2604.73976 165.50185

*** End of Report ***