Supporting Information

A New Chiral N-Heterocyclic Carbene for a New Catalytic Asymmetric Process. All-Carbon Quaternary Stereogenic Centers by Enantioselective Cu-Catalyzed Conjugate Addition of Organozinc Reagents to Unsaturated Cyclic γ-Ketoesters

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SUPPORTING INFORMATION (PART I)

General. Infrared (IR) spectra were recorded on a Nicolet 210 spectrophotometer, ν_{max} in cm^{-1}. Bands are characterized as broad (br), strong (s), medium (m), and weak (w). ^{1}H NMR spectra were recorded on a Varian Unity INOVA 400 (400 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: δ 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), and coupling constants (Hz). ^{13}C NMR spectra were recorded on a Varian Unity INOVA 400 (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: δ 77.16 ppm). High-resolution mass spectrometry were performed on a Micromass LCT ESI-MS (positive mode) at the Mass Spectrometry Facility, Boston College and at the University of Illinois Mass Spectrometry Laboratories (Urbana, Illinois). Elemental microanalyses were performed at Robertson Microlit Laboratories (Madison, NJ). Enantiomer ratios were determined by chiral GLC analysis (Alltech Associated Chiraldex GTA column (30 m x 0.25 mm) and Betadex 120 column (30 m x 0.25 mm) in comparison with authentic racemic materials. Optical rotations were measured on a Rudolph Research Analytical Autopol IV Polarimeter.

Unless otherwise noted, all reactions were carried out with distilled and degassed solvents under an atmosphere of dry N_2 in oven- (135 °C) and flame-dried glassware with standard dry box or vacuum-line techniques. Solvents were purified under a positive pressure of dry argon by a modified Innovative Technologies purification system: toluene and benzene were purified through a copper oxide and alumina column; CH_2Cl_2 and Et_2O were purged with argon and purified by passage through two alumnia columns. Tetrahydrofuran (Aldrich) and *t*-BuOMe (Acros, 99%) were purified by distillation from sodium benzophenone ketal immediately prior to use unless otherwise specified. All work-up and purification procedures were carried out with reagent grade solvents (purchased from Doe & Ingalls) in air.

Reagents and Catalysts:

Ag-complexes 1[1] **and 2**[2] were prepared by previously reported methods.

Acetic acid was purchased from Fisher and used as received.

Acetic anhydride was purchased from Aldrich and used as received.

2,2'-Azobisisobutyronitrile was purchased from Aldrich and used as received

Racemic-2,2'-bis(diphenylphosphino)-1,1'-binapthyl (*rac*-binap) was purchased from Aldrich and used as received.

2-Bromomesitylene was purchased from Aldrich and used as received.

2-Bromobenzenesulfonyl chloride was purchased from Lancaster and purified by washing a benzene solution of the sulfonyl chloride with a 1.0 M aq solution of KOH (see below for details).

***t*-BuOH** was purchased from Aldrich and used as received.

Carbon disulfide was purchased from Aldrich and used as received.

Chloroform was purchased from Fisher and purified by distillation over $CaCl_2$ before use.

Chromium trioxide was purchased from Strem Inc. and used as received.

Copper (I) triflate benzene complex (2:1) (white solid) was prepared by previously reported methods.[3]

Copper (I) triflate toluene complex (2:1) (brown solid) was purchased from Aldrich (99.99%) and used as received.

Copper (I) oxide (99.9%) was purchased from Strem Inc. and used as received.

Dicyclohexylcarbodiimide was purchased from Advanced Chem Tech and used as received.

Diethylzinc (neat) was purchased from Aldrich and used as received.

Diisopropylzinc (1M in toluene) was purchase from Aldrich and used as received.

Dimethylzinc (neat, 95%) was purchased from Strem Inc. and used as received.

Diphenylzinc (99%) was purchased from Strem Inc. (white solid) and used as received. Additionally, diphenylzinc can be prepared and purified (white solid) analogous to previously reported methods for the preparation of di-4-methoxyphenylzinc[4] and used with similar levels of efficiency and selectivity. Diphenylzinc purchased from Aldrich (brown solid) was ineffective in the present Cu-catalyzed ACA.

(-)-(*S,S*)-Diphenylethylenediamine (99% purity), was purchased from Astatech Inc. and used as received.

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[1] a) J. J. Van Veldhuizen, S. B. Garber, J. S. Kingsbury, A. H. Hoveyda, *J. Am. Chem. Soc.* **2002**, *124*, 4954-4955. b) A. O. Larsen, W. Leu, C. H. Oberhuber, J. E. Campbell, A. H. Hoveyda, *J. Am. Chem. Soc.* **2004**, *126*, 11130-11131.

[2] J. J. Van Veldhuizen, J. E. Campbell, R. E. Giudici, A. H. Hoveyda, *J. Am. Chem. Soc.* **2005**, *127*, 6877-6882.

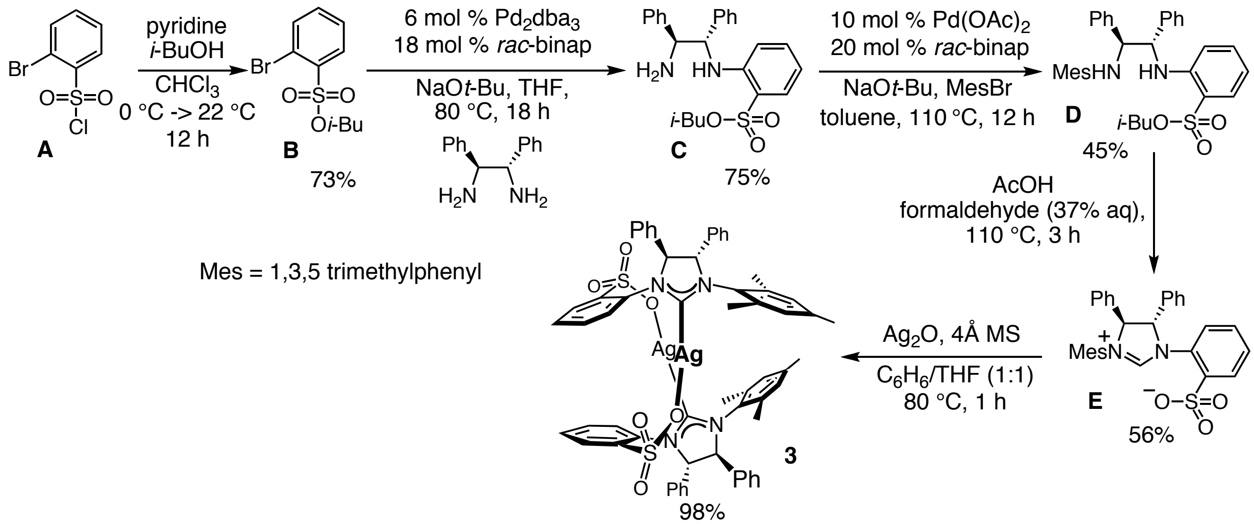
[3] R. G. Salomon, J. K. Kochi, *J. Am. Chem. Soc.* **1973**, *95*, 1889-1897.

[4] K-s. Lee, M. K. Brown, A. W. Hird, A. H. Hoveyda, *J. Am. Chem. Soc.* **2006**, *128*, 7182-7184.

4-Dimethylaminopyridine (DMAP) was purchased from Advanced Chem Tech and used as received.
Formaldehyde (37% aqueous solution) was purchased from Aldrich and used as received.
Iodomethane was purchased from Acros and used as received.
Lithium aluminum hydride (95%) was purchased from Strem Inc. and used as received.
$[(Me)_2CH(CH_2)_3]_2Zn$ was prepared by previously reported methods.[5]
Methyl 1-cyclopentene-1-carboxylate was purchased from Aldrich and used as received.
Methyl-1-cyclohexene-1-carboxylate (**F**) was purchased from Aldrich and used as received.
Tris(dibenzylideneacetone)dipalladium (0) ($Pd_2(dba)_3$) was purchased from Strem Inc. and used as received.
Palladium (II) acetate (99.9+% purity) was purchased from Aldrich and used as received.
Phenyllithium (2.0 M in *n*-Bu_2O) was purchased from Acros and used as received.
Pyridine was purchased from Aldrich and purified by distillation over KOH before use.
Silver (I) oxide was prepared by previously reported methods.[2]
Sodium *tert*-butoxide (98%) was purchased from Strem Inc. and used as received.
Sodium hydride was purchased from Strem Inc. and used as received.
Tri-*n*-butyltin hydride was purchased from Aldrich and used as received.
Triflic anhydride was prepared by distillation of triflic acid (Aldrich) over P_2O_5 (Aldrich).
Zinc (II) chloride (99.99%, ultradry), was purchased from Strem Inc. and used as received.

[5] P. Knochel, R. Singer, *Chem. Rev.* **1993**, *93*, 2117-2188 and references cited therein.

■ Experimental Procedures for the Preparation of Ag Complex 3



Isobutyl-2-bromobenzenesulfonate (B). (Prior to use in this reaction, commercially available 2-bromobenzenesulfonyl chloride (**A**) was dissolved in benzene and washed with a 1.0 M aq solution of KOH. The organic layer was dried with $MgSO_4$, filtered and concentrated in vacuo to afford **A** as a clear oil). In two separate syringes, pyridine (4.56 mL, 55.9 mmol) and a solution of 2-bromobenzenesulfonyl chloride (**A**) (6.49 g, 25.4 mmol) in $CHCl_3$ (13 mL) were added dropwise at the same time over 20 min to a solution of 2-methylpropanol (2.57 mL, 27.9 mmol) dissolved in $CHCl_3$ (13 mL) at 0 ºC under a N_2 atmosphere. The solution was allowed to stir at 22 ºC. After 20h, the reaction was quenched upon addition of a 0.1 M aq solution of HCl (20 mL) and allowed to stir for five minutes. The $CHCl_3$ layer was separated and washed with a 0.1 M aq solution of HCl (20 mL), water (2 × 15 mL) and brine (15 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo to afford a clear oil, which was purified by silica gel chromatography (10% Et_2O/petroleum ether) to yield 5.42 g (18.5 mmol, 72.8%) of sulfonate ester **B** as a clear oil. **IR (neat):** 2964 (m), 2926 (w), 2870 (w), 1573 (w), 1455 (w), 1362 (s), 1189 (s), 965 (m), 934 (m) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 8.10–8.09 (1H, m, *i*-BuOSO$_2$*o*-Ar**H**), 7.78–7.56 (1H, m, Br*o*-Ar**H**), 7.50–7.44 (2H, m, *i*-BuOSO$_2$*p*-Ar**H** and *i*-BuOSO$_2$*m*-Ar**H**), 3.85 (2H, d, J = 6.6 Hz, C**H**$_2$OSO$_2$), 2.00 (1H, qt, J = 6.8, 6.6 Hz, [CH$_3$]$_2$C**H**CH$_2$), 0.93 (6H, d, J = 6.8 Hz, CH[C**H**$_3$]$_2$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 135.9, 135.6, 134.6, 132.1, 127.6, 120.8, 77.2, 28.1, 18.7; **HRMS (EI+):** Calcd for $C_{10}H_{13}BrO_3S$: 291.9769, Found: 291.9773.

Isobutyl-2-((1*S*,2*S*)-2-amino-1,2-diphenylethylamino)benzenesulfonate (C). (−)-(*S*,*S*)-1,2-diphenylethylenediamine (1.00 g, 4.71 mmol), $Pd_2(dba)_3$ (258 mg, 0.283 mmol), *rac*-binap (528 mg, 0.848 mmol) and NaO*t*-Bu (815 mg, 8.48 mmol) were weighed out into an oven-dried 250 mL round bottom flask under a N_2 atmosphere in a glove box. The flask was removed from the glove box and fitted with a reflux condenser. A solution of **B** (1.38 g, 4.71 mmol) dissolved in

THF (47 mL) was added through a syringe and the resulting mixture was allowed to stir at reflux (~80 ºC) (the reaction mixture becomes deep red upon heating and remains this color for the length of the reaction). After 15 h, the mixture was allowed to cool to 22 ºC and the volatiles were removed in vacuo affording a deep red oil. The deep red oil was then dissolved in toluene, loaded on top of a column containing silica gel, and purified by silica gel chromatography (100% petroleum ether (to elute toluene) -> 50% Et_2O/petroleum ether) to afford 1.50 g (3.53 mmol, 74.9%) of **C** as a yellow solid. **mp:** 157-159 ºC; **IR (neat):** 3364 (br), 2966 (w), 2874 (w), 1601 (m), 1504 (m), 1461 (m), 1167 (m), 978 (m) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 7.67 (1H, dd, *J* = 8.1, 1.7 Hz, *i*-BuOSO$_2$*o*-Ar**H**), 7.49 (2H, d, *J* = 8.1 Hz, Ar**H**), 7.44 (1H, d, *J* = 7.0 Hz, N**H**) 7.35–7.22 (8H, m, Ar**H**), 7.13 (1H, dd, *J* = 8.1, 7.2 Hz, *i*-BuOSO$_2$*p*-Ar**H**), 6.57 (1H, dd, *J* = 8.1, 7.2 Hz, *i*-BuOSO$_2$*m*-Ar**H**), 6.31 (1H, d, *J* = 8.1 Hz, *i*-BuOSO$_2$*m*-Ar**H**), 4.59 (1H, dd, *J* = 7.0, 3.5 Hz, NHC**H**), 4.40 (1H, d, *J* = 3.5 Hz, NHC**H**), 3.74 (1H, dd, *J* = 9.3, 6.8 Hz, C**H**HOSO$_2$), 3.70 (1H, dd, *J* = 9.3, 6.6 Hz, CH**H**OSO$_2$), 1.95 (1H, ddqq, *J* = 6.8, 6.8, 6.8, 6.6 Hz, [CH$_3$]$_2$C**H**CH$_2$), 1.51 (2H, br, N**H**), 0.92 (3H, d, *J* = 6.8 Hz, CH[C**H**$_3$]$_2$), 0.85 (3H, d, *J* = 6.8 Hz, CH[C**H**$_3$]$_2$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 145.8, 142.6, 140.7, 135.1, 130.8, 128.8, 128.4, 127.6, 127.6, 127.1, 126.8, 116.5, 115.1, 113.6, 76.3, 63.0, 61.2, 28.1, 18.8, 18.7; **HRMS (EI+):** Calcd for $C_{24}H_{29}N_2O_3S$: 425.1899 (M^++1), Found 425.1895; **Optical Rotation:** $[\alpha]_D^{25}$ –98.9 (*c* 1.00, $CHCl_3$).

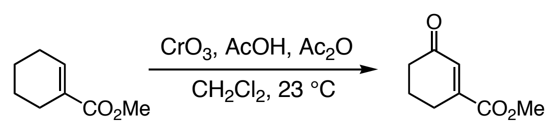
Isobutyl-2-((1*S*,2*S*)-2-(mesitylamino)-1,2-diphenylethylamino)benzenesulfonate (D). Diamine **C** (0.800 g, 1.88 mmol), $Pd(OAc)_2$ (42.0 mg, 0.188 mmol), *rac*-binap (234 mg, 0.376 mmol) and NaO*t*-Bu (272 mg, 2.83 mmol) were weighed out into an oven-dried 50 mL round bottom flask under a N_2 atmosphere in a glove box. The flask was removed from the glove box and fitted with a reflux condenser. A solution of 2-bromomesitylene (577 µL, 3.77 mmol) dissolved in toluene (19 mL) was added through a syringe and the resulting mixture was allowed to stir at 110 ºC (the mixture becomes deep red upon heating and remains this color for the length of the reaction). After 18 h, the mixture was allowed to cool to 22 ºC, loaded directly on top of a column containing silica gel and purified by silica gel column chromatography (100% petroleum ether (to elute toluene) -> 20% Et_2O/petroleum ether) to afford a pale yellow solid, which was rinsed with petroleum ether to yield 441 mg (0.811 mmol, 43.1%) of diamine **D** as a white solid. **mp:** 164-166 ºC; **IR (neat):** 3352 (m), 2962 (m), 2917 (w), 2861 (w), 1596 (s), 1350 (s) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 7.82 (1H, dd, *J* = 8.1, 1.7 Hz, *i*-BuOSO$_2$*o*-Ar**H**), 7.42 (1H, d, *J* = 4.5 Hz, N**H**), 7.30–7.12 (9H, m, *i*-BuOSO$_2$*p*-Ar**H** and Ar**H**), 7.07–7.04 (2H, m, Ar**H**), 6.75–6.72 (3H, m, *i*-BuOSO$_2$*m*-Ar**H** and Mes**H**), 6.57 (1H, d, *J* = 8.4 Hz, *i*-BuOSO$_2$*m*-Ar**H**), 4.97 (1H, dd, *J* = 7.0, 4.5 Hz, NHC**H**), 4.57 (1H, d, *J* = 7.0 Hz, NHC**H**), 3.88 (1H, dd, *J* = 9.3, 6.6 Hz, C**H**HOSO$_2$), 3.78 (1H, dd, *J* = 9.3, 6.4 Hz, CH**H**OSO$_2$), 3.65 (1H, br s, N**H**), 2.20 (3H, s, C**H**$_3$Ar), 2.15 (6H, s, C**H**$_3$Ar), 2.01 (1H, ddqq, *J* = 6.6, 6.4, 4.2, 4.0 Hz, [CH$_3$]$_2$C**H**CH$_2$), 0.99 (3H, d, *J* = 4.0 Hz, CH[C**H**$_3$]$_2$), 0.97 (3H, d, *J* = 4.2 Hz, CH[C**H**$_3$]$_2$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 145.7, 140.9, 140.2, 140.1, 135.1, 131.1, 130.9, 129.8, 129.2, 128.4, 128.2, 128.1, 127.8, 127.6, 117.6, 115.8, 113.9, 76.2, 66.7, 62.0, 28.2, 20.5, 19.2, 18.8; **HRMS (EI+):** Calcd for $C_{33}H_{39}N_2O_3S$: 543.2681 (M^++1), Found 543.2680; **Optical Rotation:** $[\alpha]_D^{25}$ –94.4 (*c* 0.100, $CHCl_3$).

Imidazolium Salt E. Diamine **D** (0.560 g, 1.03 mmol) was weighed out into a screw cap vial (2 x 8 cm), which was sealed with a septum and purged with N_2. Acetic acid (891 μL, 15.4 mmol) followed by formaldehyde (37% (aq), 383 μL, 5.15 mmol) were added through a syringe. The vial was sealed with a screw cap and allowed to stir at 110 ºC (the white heterogeneous mixture becomes yellow and homogeneous upon heating). After 3 h, the mixture was allowed to cool to 22 ºC and diluted with Et_2O (5 mL) and water (5 mL). The reaction was neutralized by the *slow* addition of solid K_2CO_3 until gas evolution ceased. Dichloromethane (10 mL) was added and the aqueous layer separated. The aqueous layer was then washed further with CH_2Cl_2 (2 × 10 mL) and the combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford a yellow solid. The yellow solid was purified by silica gel column chromatography (100% CH_2Cl_2 -> 0.5% MeOH/CH_2Cl_2 -> 1.0% MeOH/CH_2Cl_2) to afford 284 mg (0.572 mmol, 55.5%) of imidazolium salt **E** as a white solid. (Note: Separation of a yellow impurity by silica gel column chromatography can often be tedious. Precipitation of **E** from a CH_2Cl_2 solution with petroleum ether can remove trace amounts of this yellow impurity after careful silica gel column chromatography. This material was obtained in crystalline form by recrystallization from CH_2Cl_2:Et_2O, but recrystallization is not required for effective formation of Ag complex **3**.) **mp:** 223-225 ºC; **IR (neat):** 3058 (m), 2914 (m), 2861 (w), 1623 (s), 1579 (m), 1230 (s) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 8.72 (1H, s, NC**H**N), 8.23 (1H, dd, *J* = 7.9, 1.5 Hz, SO_2*o*-Ar**H**), 7.67–7.64 (2H, m, Ar**H**), 7.50–7.47 (2H, m, Ar**H**), 7.43–7.41 (3H, m, Ar**H**), 7.36–7.30 (4H, m, SO_2*m*-Ar**H** and Ar**H**), 7.10 (1H, td, *J* = 7.9, 1.5 Hz, SO_2*p*-Ar**H**), 6.93 (1H, s, Mes**H**), 6.73 (1H, s, Mes**H**), 6.72 (1H, dd, *J* = 7.9, 1.5 Hz, SO_2*m*-Ar**H**), 6.56 (1H, d, *J* = 11.8 Hz, NC**H**), 5.53 (1H, d, *J* = 11.8 Hz, NC**H**), 2.60 (3H, s, C**H**$_3$Ar), 2.23 (3H, s, C**H**$_3$Ar), 2.00 (3H, s, C**H**$_3$Ar); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 158.7, 144.0, 140.4, 138.6, 135.0, 134.3, 131.5, 130.7, 130.5, 130.5, 130.3, 130.0, 129.9, 129.7, 129.6, 129.1, 129.1, 127.5, 76.2, 74.5, 21.0, 18.8, 18.5; **HRMS (EI+):** Calcd for $C_{30}H_{29}N_2O_3S$: 497.1899 (M^++1), Found 497.1886; **Elemental Analysis:** Anal Calcd for $C_{30}H_{28}N_2O_3S$: C 72.55; H 5.68; N 5.64; Found C 72.27; H 5.41; N 5.45; **Optical Rotation:** $[\alpha]_D^{25}$ –14.9 (*c* 0.500, $CHCl_3$).

Ag Complex 3. Imidazolium salt **E** (100 mg, 0.201 mmol), Ag_2O (93.0 mg, 0.400 mmol) and oven-dried powdered <5 micron 4Å MS (ca. 50 mg) were weighed out into an oven-dried 10 mL round bottom flask under a N_2 atmosphere in a glove box. The flask was removed from the glove box, fitted with a reflux condenser, and wrapped with aluminum foil to exclude light. Tetrahydrofuran (1.0 mL) followed immediately by benzene (1.0 mL) were added through a syringe resulting in a black heterogeneous mixture. The mixture was allowed to stir at 80 ºC. After 1 h, the mixture was allowed to cool to 22 ºC and filtered through a short plug of Celite 545 (4 x 1 cm) eluted with THF (ca. 20 mL). The solution was then concentrated in vacuo to afford 119 mg (0.197 mmol, 98.0%) of Ag complex **3** as a white solid, which was stored under low light conditions. (Note: This material was obtained in crystalline form by recystallization from CH_2Cl_2:Et_2O bilayer, but recrystallization is not required for effective Cu-catalyzed ACA). **mp:** 247-249 ºC (decomp); **IR (neat):** 3062 (w), 3026 (w), 2908 (w), 1608 (w), 1480 (s), 1455 (s), 1226 (s), 1201 (s), 1027 (m), 754 (s) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 8.27 (1H, br d, *J* = 7.3 Hz, Ar**H**), 7.50–7.44 (2H, m, Ar**H**), 7.32–6.95 (10H, m, Ar**H**), 6.80 (2H, br s, Ar**H**), 6.55 (1H, br d, *J* = 10.4 Hz, NC**H**), 6.33 (2H, br s, Ar**H**), 5.18 (1H, br d, *J* = 10.4 Hz, NC**H**), 2.46 (3H, s, C**H**$_3$Ar), 2.29 (3H, s, C**H**$_3$Ar), 1.42 (3H, s, C**H**$_3$Ar); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 205.6 ($J_{C^{109}Ag}$ = 186.8 Hz, $J_{C^{107}Ag}$ = 182.5 Hz), 143.4, 138.6, 138.5, 136.5, 135.7, 135.2, 134.1, 131.1, 130.6, 130.0, 129.9, 129.6, 128.9, 128.8, 128.6, 128.5, 128.4, 128.2, 76.5, 74.0, 68.6, 21.1, 19.0, 17.9; **Optical Rotation:** $[\alpha]_D^{25}$ –104 (*c* 0.500, $CHCl_3$).

Important Note: The carbene carbon chemical shift for Ag(I) complexes **1** and **2** were incorrectly reported in the respective supporting informations. Correct values are as follows: **1** ^{13}C NMR ($CDCl_3$, 100 MHz): δ 206.3 ($J_{C^{109}Ag}$ = 268.4 Hz, $J_{C^{107}Ag}$ = 231.9 Hz). **2** ^{13}C NMR ($CDCl_3$, 100 MHz): δ 207.6 ($J_{C^{109}Ag}$ = 267.1 Hz, $J_{C^{107}Ag}$ = 231.5 Hz).

■ Representative experimental procedures for the preparation of methyl ester substrates 4-5b:[6]



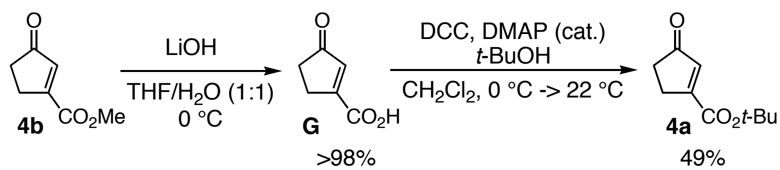
F **5b**

Methyl-3-oxocyclohex-1-enecarboxylate (5b). To a 100 mL round bottom flask charged with CrO_3 (9.22 g, 60.7 mmol) was added AcOH (28.6 mL, 500 mmol) and Ac_2O (14.5 mL, 142 mmol). The dark red mixture was allowed to stir for 1 h. In a 250 mL round bottom flask equipped with an addition funnel was added methyl-1-cyclohexene-1-carboxylate (**F**) (5.00 g, 35.7 mmol) and CH_2Cl_2 (71 mL). The solution of CrO_3 in AcOH/Ac_2O was transferred to the addition funnel and slowly added to the CH_2Cl_2 solution of methyl-1-cyclohexene-1-carboxylate over 1 h. (During the addition of CrO_3 to the substrate, the solution becomes black.) The mixture was allowed to stir for an additional 1 h, at which time the solution was allowed to cool to 0 ºC and the reaction quenched upon addition of a 10 M aq solution of KOH (~80 mL) until pH ~ 8. The mixture was then diluted with Et_2O (200 mL) and H_2O (200 mL). The organic layer was separated and the aqueous layer was washed with Et_2O (3 x 200 mL). The combined organic layers were then washed with a saturated aqueous solution of $NaHCO_3$ (3 x 200 mL) and

[6] a) A similar procedure for the preparation of these compounds (**4b** and **5b**) has been reported, see: G. L. Lang, C. D. Decicco, J. Willson, L. A. Strickland, *J. Org. Chem.* **1989**, *54*, 1805-1810. Though other catalytic allylic oxidation methods have been reported (see b) and c)) to afford **4-5b**, we found that product of the highest purity could be obtained with Cr-mediated allylic oxidation. b) For Pd-catalyzed allylic oxidation, see: J-Q. Yu, E. J. Corey, *J. Am. Chem. Soc.* **2003**, *125*, 3232-3233. c) For Rh-catalyzed allylic oxidation, see: A. J. Catino, R. E. Forslund, M. P. Doyle, *J. Am. Chem. Soc.* **2004**, *126*, 13622-13623.

brine (1 x 200 mL). The organic layer was dried over $MgSO_4$, filtered and concentrated to afford a yellow oil which was purified by silica gel column chromatography (5% Et_2O/petroleum ether -> 20% Et_2O/petroleum ether) then distilled under reduced pressure to yield 3.00 g (19.4 mmol, 54.3%) of **5b** as a pale yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 6.72 (1H, t, *J* = 2.0 Hz, C**H**), 3.82 (3H, s, OC**H**$_3$) 2.57 (2H, td, *J* = 6.0, 2.0 Hz), 2.45-2.42 (2H, m), 2.07-2.01 (2H, m); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 200.2, 167.1, 149.0, 133.2, 52.7, 37.8, 25.0, 22.3.

■ **Representative experimental procedures for the preparation of *tert*-butyl ester substrates 4-5a:**



3-Oxocyclopent-1-enecarboxylic acid (G). LiOH (1.50 g, 64.0 mmol) was added to **4b** (1.80 g, 12.8 mmol) dissolved in THF (180 mL) and H_2O (180 mL) at 0 ºC. The mixture was allowed to stir for two minutes, at which time the reaction was quenched upon addition of a 0.5 M solution of aqueous HCl (100 mL) at 0 ºC until pH < 4. The mixture was washed with EtOAc (3 x 200 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated to yield 1.50 g (12.8 mmol, >98.0% yield) of **G** as a white solid, which was used directly in the subsequent reaction.

***tert*-Butyl-3-oxocyclopent-1-enecarboxylate (4a).** To a solution of carboxylic acid **G** (206 mg, 1.64 mmol), DMAP (39.0 mg, 0.320 mmol) and *t*-BuOH (235 μL, 2.46 mmol) dissolved in CH_2Cl_2 (15 mL) at 0 ºC was added dropwise a solution of DCC (474 mg, 2.30 mmol) dissolved in CH_2Cl_2 (6.0 mL) through a syringe. The mixture was then allowed to warm to 22 ºC (during which time the mixture becomes brown) and stir for 15 h. At this time, the mixture was diluted with Et_2O (20 mL) and passed through a short column of Celite 545 (2 x 5 cm) layered on top of silica gel (2 x 10 cm) eluted with Et_2O. The filtrate was concentrated in vacuo to afford a pale yellow oil, which was purified by silica gel column chromatography (5% Et_2O/petroleum ether -> 20% Et_2O/petroleum ether) then distilled under reduced pressure to yield 146 mg (0.784 mmol, 49.0%) of **4a** as a clear oil. **IR (neat):** 2993 (m), 2945 (m), 1722 (s), 1721 (s), 1619 (m), 1443 (m), 1401 (m), 1377 (m), 1346 (m), 1249 (s), 1231 (s), 1153 (s), 1068 (m), 989 (w), 904 (w), 850 (w), 795 (w), 747 (m) cm^{-1}; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 6.55 (1H, t, *J* = 2.0 Hz, C**H**), 2.72-2.69 (2H, m), 2.41-2.39 (2H, m), 1.43 (9H, s, C(C**H**$_3$)$_3$); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 209.3, 166.2, 163.4, 137.3, 82.4, 35.6, 27.9, 27.4; **HRMS (CI+):** Calcd for $C_{10}H_{15}O_3$: 183.102120 [M$^+$+1], Found: 183.101802.

***tert*-Butyl-3-oxocyclohex-1-enecarboxylate (5a). IR (neat):** 2984 (m), 2964 (m), 2875 (w), 1717 (s), 1690 (s), 1468 (w), 1387 (m), 1273 (s), 1165 (s), 1073 (m), 1029 (w), 975 (w), 905 (w), 851 (w), 742 (w) cm^{-1}; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 6.65 (1H, t, *J* = 2.0 Hz, C**H**), 2.52 (2H, dt, *J* = 6.0, 2.0 Hz), 2.41, (2H, dd, *J* = 8.0, 6.8 Hz), 2.02 (2H, tt, *J* = 6.0, 6.0 Hz), 1.49 (9H, s, $C(CH_3)_3$); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 200.8, 165.8, 151.1, 132.5, 82.4, 37.9, 28.1, 25.0, 22.4; **HRMS (CI+):** Calcd for $C_{11}H_{17}O_3$ [M^++1]: 197.117770, Found: 197.117307.

■ **Representative experimental procedure for Cu-catalyzed conjugate addition of *Me_2Zn* to unsaturated cyclic γ-ketoesters:** An oven-dried 13x100 mm test tube was charged with chiral Ag complex **3** (3.02 mg, 2.50 µmol) and $(CuOTf)_2$•C_6H_6 (1.30 mg, 2.50 µmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm before removal from the glove box. *tert*-Butylmethylether (1.0 mL) was added through a syringe and the resulting solution was allowed to stir for five minutes before cooling to –78 ºC (dry ice/acetone bath). Dimethylzinc (21.0 µL, 0.300 mmol) (PYROPHORIC, USE EXTREME CAUTION) was added and the resulting light yellow mixture was allowed to warm to –30 ºC (cryocool). (During this time the mixture became dark brown.) After 10 minutes at –30 ºC, methyl 3-oxocyclohex-1-enecarboxylate (**5b**) (13.4 µL, 15.4 mg, 0.100 mmol) was added to the mixture through a syringe.[7] After 15 h at –30 ºC, the reaction was quenched upon addition of a saturated aqueous solution of ammonium chloride (1 mL) and H_2O (1 mL). After allowing the mixture to warm to 22 ºC, it was washed with EtOAc (2 x 1 mL) and passed through a short plug of silica gel (4 cm x 1 cm) eluted with EtOAc. The volatiles were removed in vacuo, resulting in a yellow oil that was purified by silica gel column chromatography (5% Et_2O/petroleum ether -> 20% Et_2O/petroleum ether) to afford 15.1 mg of the desired product as a clear oil (0.0888 mmol, 88.8%).

■ **Representative experimental procedure for Cu-catalyzed conjugate addition of *Et_2Zn*, *$[(Me)_2CH(CH_2)_2]_2Zn$* and *i-Pr_2Zn*[8] to unsaturated cyclic γ-ketoesters:** An oven-dried 13x100 mm test tube was charged with chiral Ag complex **3** (3.02 mg, 2.50 µmol) and $(CuOTf)_2$•C_6H_6 (1.30 mg, 2.50 µmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm before removal from the glove box. *tert*-Butylmethylether (1.0 mL) was added through a syringe and the resulting solution was allowed to stir for five minutes before cooling to –30 ºC (dry ice/acetone bath). Diethylzinc (30.7 µL, 0.300 mmol) (PYROPHORIC, USE EXTREME CAUTION) followed by methyl 3-oxocyclopent-1-enecarboxylate (**4b**) (14.0 mg, 0.100 mmol) dissolved in *t*-BuOMe (250 µL) were added through a syringe.[9,10] (During the addition of Et_2Zn the mixture became dark brown.)

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[7] Methyl 3-oxocyclopent-1-enecarboxylate (**4b**) (white solid) was added as a solution in *t*-BuOMe (250 µL).
[8] *i*-Pr_2Zn was used as a 1.0 M solution in toluene (Aldrich).
[9] Cyclic enones **4a** and **5a-b** were added neat through a syringe.

After 1 h at –30 ºC, the reaction was quenched upon addition of a saturated aqueous solution of ammonium chloride (1 mL) and H_2O (1 mL). After allowing the mixture to warm to 22 ºC, it was washed with EtOAc (2 x 1 mL) and passed through a short plug of silica gel (4 cm x 1 cm) eluted with EtOAc. The volatiles were removed in vacuo, resulting in a yellow oil that was purified by silica gel column chromatography (5% Et_2O/petroleum ether -> 20% Et_2O/ petroleum ether) to afford 14.9 mg of the desired product as a clear oil (0.0876 mmol, 87.6%).

■ **Representative experimental procedure for Cu-catalyzed conjugate addition of *Me_2Zn* to unsaturated cyclic γ-ketoesters *set up on a bench top and carried out in undistilled t-BuOMe*:** A 13x100 mm test tube was charged with chiral Ag complex **3** (3.02 mg, 2.50 μmol) and $(CuOTf)_2$•toluene (1.30 mg, 2.50 μmol), weighed out on a bench top. The test tube was sealed with a septum and wrapped with parafilm before being purged with N_2 for five minutes. Undistilled *t*-BuOMe (1.0 mL) was added through a syringe and the resulting solution was allowed to stir for five minutes before cooling to –78 ºC (dry ice/acetone bath). Dimethylzinc (21.0 μL, 0.300 mmol) (PYROPHORIC, USE EXTREME CAUTION) was added and the resulting light yellow mixture was allowed to warm to –30 ºC (cryocool). (During this time the mixture became dark brown.) After 10 minutes at –30 ºC, methyl 3-oxocyclohex-1-enecarboxylate (**5b**) (13.4 μL, 15.4 mg, 0.100 mmol) was added to the mixture through a syringe.[7] After 15 h at –30 ºC, the reaction was quenched upon addition of a saturated aqueous solution of ammonium chloride (1 mL) and H_2O (1 mL). After allowing the mixture to warm to 22 ºC, it was washed with EtOAc (2 x 1 mL) and passed through a short plug of silica gel (4 cm x 1 cm) eluted with EtOAc. The volatiles were removed in vacuo, resulting in a yellow oil that was purified by silica gel column chromatography (5% Et_2O/petroleum ether -> 20% Et_2O/petroleum ether) to afford 12.3 mg of the desired product as a clear oil (0.0723 mmol, 72.3%).

■ **Representative experimental procedure for Cu-catalyzed conjugate addition of *Ph_2Zn* to unsaturated cyclic γ-ketoesters:** An oven-dried 13x100 mm test tube was charged with chiral Ag complex **3** (3.02 mg, 2.50 μmol), $(CuOTf)_2$•C_6H_6 (1.3 mg, 2.50 μmol), and Ph_2Zn (65.7 mg, 0.300 mmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm prior to removal from the glove box. The test tube containing the solids was allowed to cool to –30 ºC (dry ice/acetone bath) and Et_2O (1.0 mL) was added through a syringe. The resulting mixture was allowed to stir for 10 minutes. (During this time the mixture became dark brown.) At this time, a solution of methyl 3-oxocyclopent-1-enecarboxylate (**4b**) (15.4 mg, 0.100 mmol) in Et_2O (500 μL) was added to the mixture through a syringe.[9] After 42 h at –30 ºC, the reaction was quenched upon addition of a saturated aqueous solution of ammonium chloride (1 mL) and H_2O (1 mL). After allowing the mixture to warm to 22 ºC, it was washed with EtOAc (2 x 1 mL) and passed through a short plug of silica gel (4 cm

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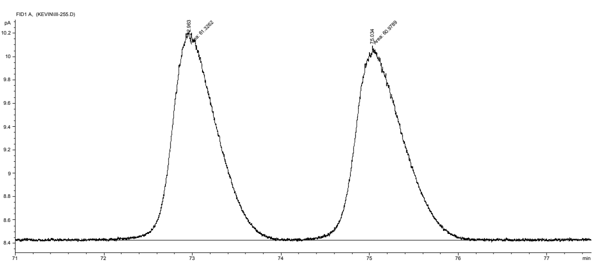
[10] For cases involving styrene as an additive; it was added neat through a syringe after the addition of dialkylzinc reagent but before cyclic enone.

x 1 cm) eluted with EtOAc. The volatiles were removed in vacuo, to afford a yellow oil that was purified by silica gel chromatography (10% Et_2O/petroleum ether) to yield 15.3 mg (0.0701 mmol, 70.1%) of the desired product as a clear oil.

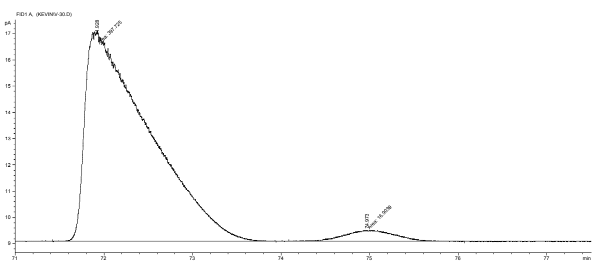
■ **Representative experimental procedure for Cu-catalyzed conjugate addition of *in-situ prepared Ph_2Zn* to unsaturated cyclic γ-ketoesters:** An oven-dried 13x100 mm test tube was charged with chiral Ag complex **2** (3.07 mg, 2.50 μmol) and $(CuOTf)_2$•C_6H_6 (1.3 mg, 2.50 μmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm prior to removal from the glove box. The test tube containing the solids was allowed to cool to –30 ºC (dry ice/acetone bath). Another oven-dried 13x100 mm test tube was charged with $ZnCl_2$ (0.124 g, 0.900 mmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm prior to removal from the glove box. Diethyl ether (2.2 mL) was added through a syringe to the test tube containing $ZnCl_2$, first allowing the $ZnCl_2$ to dissolve and then allowing the mixture to cool to 0 ºC. Phenyllithium (818 μL, 1.80 mmol, 2.20 M in *n*-Bu_2O) was added dropwise through a syringe to the test tube containing a solution of $ZnCl_2$ in Et_2O and allowed to stir at 22 ºC for 15 minutes. During this time the solution of PhLi + $ZnCl_2$ became cloudy. The test tube, now containing a solution of Ph_2Zn and solid LiCl, was centrifuged for 15 minutes to assist with settling of the LiCl to the bottom of the test tube. At this time, 1.0 mL of the Ph_2Zn solution was added through a syringe to the test tube containing $(CuOTf)_2$•C_6H_6 and chiral Ag complex **2**, cooled to –30 ºC (dry ice/acetone). The resulting mixture was allowed to stir for five minutes. A solution of methyl 3-oxocyclohex-1-enecarboxylate (**5b**) (15.4 mg, 0.100 mmol) in Et_2O (500 μL) was added through a syringe. After 24 h at –30 ºC (cryocool), the reaction was quenched upon addition of a saturated aqueous solution of ammonium chloride (1 mL) and H_2O (1 mL). After allowing the mixture to warm to 22 ºC, it was washed with EtOAc (2 x 1 mL) and passed through a short plug of silica gel (4 cm x 1 cm) eluted with EtOAc. The volatiles were removed in vacuo, to afford a yellow oil that was purified by silica gel chromatography (10% Et_2O/petroleum ether) to yield 19.0 mg (0.0820 mmol, 82.0%) of the desired product as a white solid.

(*R*)-Methyl-1-methyl-3-oxocyclopentanecarboxylate (Table 3, entry 1). IR (neat): 2963 (m), 2929 (m), 2886 (w), 2750 (s), 1721 (s), 1475 (m), 1442 (m), 1413 (m), 1336 (m), 1244 (m), 1220 (s), 1177 (m), 1110 (m), 994 (w), 879 (w), 787 (w) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 3.70 (3H, s, OC**H**$_3$), 2.74 (1H, d, J = 18.4 Hz, C(O)C**H**HC), 2.41-2.27 (3H, m, C(O)CH_2CH_2C), 2.10 (1H, d, J = 18.4 Hz, C(O)CH**H**C), 1.94-1.87 (1H, m, C(O)CH_2CH_2C), 1.36 (3H, s, CC**H**$_3$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 217.1, 177.0, 52.6, 49.4, 46.5, 36.9, 34.0, 24.1; **HRMS (EI+):** Calcd for $C_8H_{11}O_3$ [M^+-1]: 155.070819, Found: 155.071395; **Optical Rotation:** $[\alpha]_D^{20}$ –12.7 (c 1.50, $CHCl_3$) for an enantiomerically enriched sample of 92% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (92% ee shown; β-dex column, 15 psi, 90 ºC).



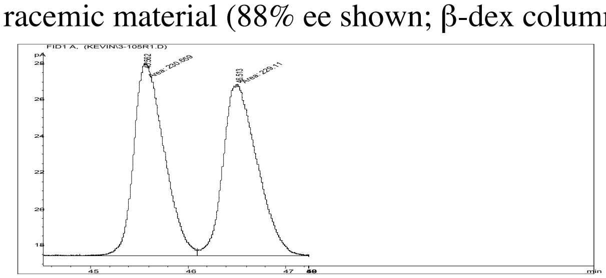
#	Time	Area	Height	Width	Area (%)
1	72.963	61.3	1.8	0.567	50.142
2	75.034	61	1.7	0.6099	49.858



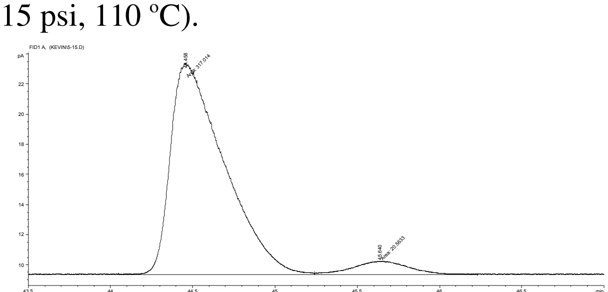
#	Time	Area	Height	Width	Area%
1	71.928	397.7	8	0.8242	95.923
2	74.973	16.9	4.2E-1	0.6733	4.077

(*R*)-Methyl-1-ethyl-3-oxocyclopentanecarboxylate (Table 3, entry 3). IR (neat): 2977 (m), 2885 (w), 1759 (s), 1729 (s), 1466 (w), 1350 (w), 1258 (m), 1203 (m), 1166 (m), 995 (w) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 3.68 (3H, s, OC**H**$_3$), 2.73 (1H, d, J = 18.4 Hz, C(O)C**H**HC), 2.36 (1H, ddd, J = 8.4, 7.2, 1.2 Hz, CH_2CH_2C(O)), 2.25 (2H, dd, J = 7.2, 7.2 Hz, CH_2CH_2C(O)), 2.09 (1H, d, J = 18.4 Hz, C(O)CH**H**C), 1.92-1.79 (2H, m, CH_2CH_2C(O) and C**H**HCH_3), 1.68-1.59 (1H, m, CH**H**CH_3), 0.85 (3H, t, J = 7.6 Hz, C**H**$_3$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 217.1, 176.3, 52.4, 51.8, 47.1, 36.9, 32.4, 31.3, 10.1; **HRMS (EI+):** Calcd for $C_9H_{14}O_3$: 170.0943, Found: 170.0941; **Optical Rotation:** $[\alpha]_D^{20}$ –45.1 (c 0.286, $CHCl_3$) for an enantiomerically enriched sample of 88% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (88% ee shown; β-dex column, 15 psi, 110 ºC).



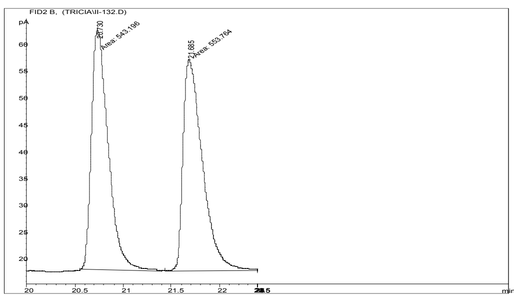
#	Time	Area	Height	Width	Area%
1	45.562	230.7	10.6	0.3611	50.168
2	46.513	229.1	9.4	0.4054	49.832



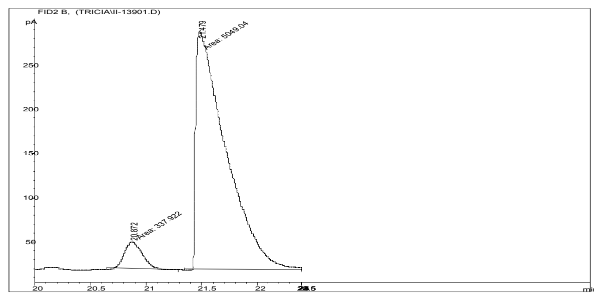
#	Time	Area	Height	Width	Area%
1	44.458	317	14	0.377	93.909
2	45.64	20.6	8.8E-1	0.3916	6.091

(*R*)-Methyl-1-(4-methylpentyl)-3-oxocyclopentanecarboxylate (Table 3, entry 4). IR (neat): 2955 (m), 2867 (w), 1747 (s), 1734 (s), 1457 (w), 1407 (w), 1388 (w), 1369 (w), 1338 (w), 1205 (m), 1155 (s), 985 (w), 872 (w) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 3.72 (3H, s, OC**H**$_3$), 2.79 (1H, dd, *J* = 18.4, 1.2 Hz, C(O)C**H**HC), 2.43-2.36 (1H, m), 2.28 (2H, t, *J* = 8.8 Hz, (CH_2CH_2C(O)), 2.12 (1H, d, *J* = 18.4 Hz, C(O)CH**H**C), 1.95-1.87 (1H, m), 1.83-1.76 (1H, m), 1.60-1.48 (2H, m), 1.26-1.12 (4H, m), 0.86 (3H, s, $CHCH_3C$**H**$_3$), 0.84 (3H, s, CHC**H**$_3CH_3$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 217.1, 176.5, 52.4, 51.3, 47.6, 39.2, 38.8, 36.9, 32.9, 27.9, 23.6, 22.7, 22.7; **HRMS (EI$^+$):** Calcd for $C_{13}H_{22}O_3$: 226.1569, Found: 226.1569; **Optical Rotation:** $[\alpha]_D^{20}$ –30.8 (*c* 0.647, $CHCl_3$) for an enantiomerically enriched sample of 87% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (87% ee shown; chiral dex GTA column, 15 psi, 140 ºC).



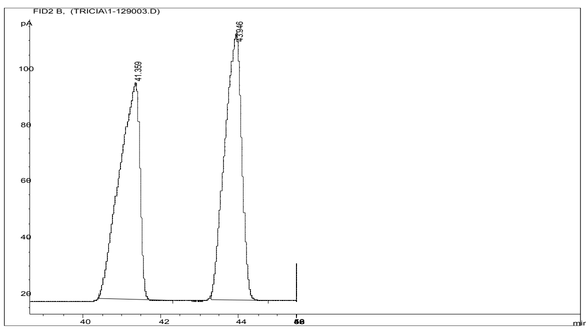
#	Time	Area	Height	Width	Area (%)
1	20.73	543.2	44.4	0.2039	49.518
2	21.685	553.8	39.3	0.2351	50.482



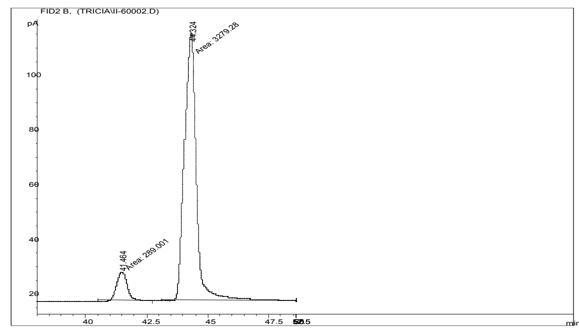
#	Time	Area	Height	Width	Area (%)
1	20.872	337.9	30.2	0.1864	6.273
2	21.479	5049	269.6	0.3122	93.727

(*R*)-Methyl-3-oxo-1-phenylcyclopentanecarboxylate (Table 3, entry 6). IR (neat): 2962 (w), 2924 (m), 2848 (w), 1753 (s), 1734 (s), 1445 (w), 1249 (m), 1212 (m), 1162 (m), 752 (m), 695 (m) cm^{-1}; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.36-7.24 (5H, m, Ar**H**), 3.64 (3H, s, OC**H**$_3$), 3.23 (1H, dd, *J* = 16.0, 2.0 Hz, C(O)CH**H**C), 2.98-2.95 (1H, m, C(O)CH_2C**H**$_2$), 2.62 (1H, d, *J* = 18.0 Hz, C(O)C**H**HC), 2.35-2.32 (3H, m, C(O)C**H**$_2CH_2$C and C(O)CH_2CH**H**C); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 215.6, 175.0, 141.3, 129.0, 127.8, 126.6, 55.2, 53.1, 48.4, 37.2, 33.0; **Elemental Analysis:** Anal Calcd for $C_{13}H_{14}O_3$: C, 71.54; H, 6.47; Found C, 71.53; H, 6.68.; **Optical Rotation:** $[\alpha]_D^{20}$ -7.05 (*c* 0.313, $CHCl_3$) for an enantiomerically enriched sample of 83% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (84% ee shown; chiral dex GTA column, 15 psi, 140 ºC).



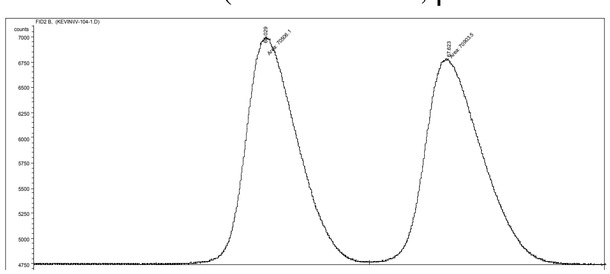
#	Time	Area	Height	Width	Area (%)
1	41.359	2848.3	77.1	0.4342	49.748
2	43.946	2877.2	94.9	0.3579	50.252



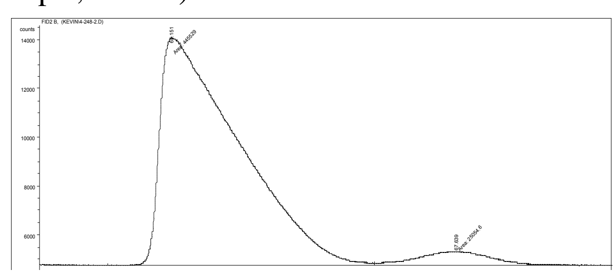
#	Time	Area	Height	Width	Area (%)
1	41.464	289	10.3	0.4693	8.099
2	44.324	3279.3	97.7	0.5593	91.901

(*R*)-*tert*-Butyl-1-methyl-3-oxocyclopentanecarboxylate (Table 3, entry 2). IR (neat): 2983 (m), 2946 (w), 2879 (w), 1759 (s), 1729 (s), 1613 (w), 1466 (w), 1368 (m), 1246 (m), 1160 (s), 1111 (m), 854 (m) cm^{-1}; **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 2.70 (1H, d, *J* = 18.0 Hz, C(O)C**H**HC), 2.41-2.24 (3H, m, $CH_2CH_2C(O)$), 2.06 (1H, d, *J* = 18.0 Hz, C(O)CH**H**C), 1.91-1.82 (1H, m, $CH_2CH_2C(O)$), 1.44 (9H, s, $C(C\mathbf{H}_3)_3$), 1.33 (3H, s, $C\mathbf{H}_3$); **^{13}C NMR ($CDCl_3$, 100 MHz):** 217.6, 175.7, 81.3, 49.5, 47.2, 37.1, 34.1, 28.1, 24.1; **HRMS (CI+):** Calcd for $C_{11}H_{19}O_3$ [M^++1]: 199.133420, Found: 199.133352; **Optical Rotation:** $[\alpha]_D^{20}$ –21.3 (*c* 1.05, $CHCl_3$) for an enantiomerically enriched sample of 90% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (89% ee shown; β-dex column, 15 psi, 100 °C).



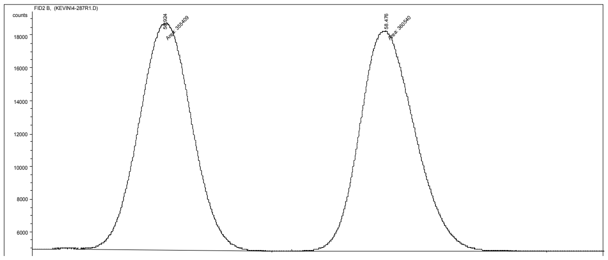
#	Time	Area	Height	Width	Area (%)
1	66.029	70506.1	2252.3	0.5217	49.859
2	67.623	70903.5	2040.2	0.5792	50.141



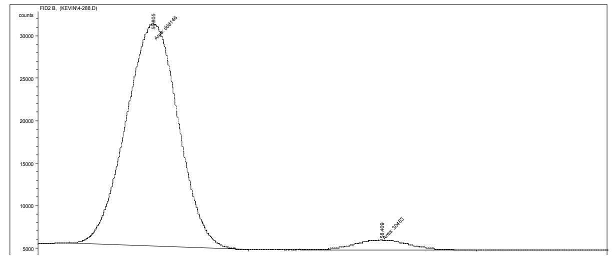
#	Time	Area	Height	Width	Area (%)
1	65.151	445508.2	9378.6	0.7917	94.675
2	67.639	25054.6	561.7	0.7434	5.325

(*R*)-*tert*-Butyl-1-isopropyl-3-oxocyclopentanecarboxylate (Table 3, entry 5). IR (neat): 2975 (m), 2929 (w), 2877 (w), 1752 (s), 1718 (s), 1463 (w), 1376 (m), 1266 (m), 1156 (s), 854 (w), 675 (w); **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 2.75 (1H, d, *J* = 18.4 Hz, C(O)C**H**HC), 2.42-2.36 (1H, m, $CH_2CH_2C(O)$), 2.31-2.14 (2H, m, $CH_2CH_2C(O)$), 2.06-1.95 (2H, m, C(O)CH**H**C and $C\mathbf{H}(CH_3)_2$), 1.86 (1H, ddd, *J* = 13.2, 10.4, 10.4 Hz, $CH_2CH_2C(O)$), 1.44 (9H, s, $C(C\mathbf{H}_3)_3$), 0.95 (6H, dd, *J* = 8.0 Hz, $CH(C\mathbf{H}_3)_2$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 217.7, 174.7, 81.4, 56.2, 45.4, 37.5, 35.2, 30.8, 28.1, 18.7, 18.5; **HRMS (CI+):** Calcd for $C_{13}H_{23}O_3$: 227.164720 [M^++1], Found: 227.164414; **Optical Rotation:** $[\alpha]_D^{20}$ –100 (*c* 0.893, $CHCl_3$) for an enantiomerically enriched sample of 91% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (91% ee shown; β-dex column, 15 psi, 120 ºC).



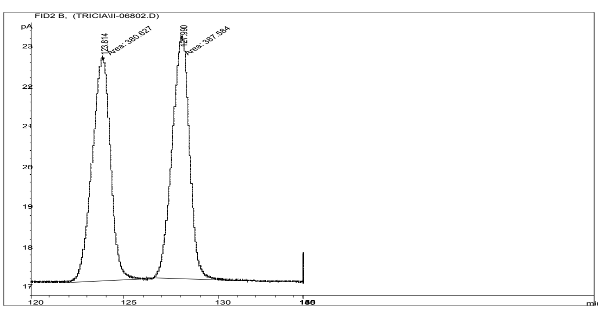
#	Time	Area	Height	Width	Area (%)
1	56.924	355409.4	13815.9	0.4287	49.599
2	58.476	360539.9	13413.4	0.448	50.400



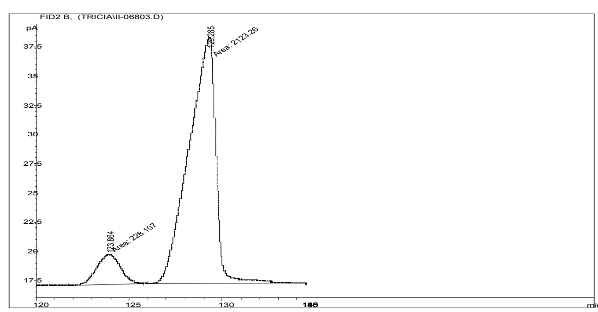
#	Time	Area	Height	Width	Area (%)
1	56.805	668145.7	26211	0.4248	95.618
2	58.409	30483	1143.6	0.4443	4.382

(*R*)-*tert*-Butyl-3-oxo-1-phenylcyclopentanecarboxylate (Table 3, entry 7). mp: 69-74 ºC; **IR (neat):** 2950 (m), 2930 (m), 1747 (s), 1722 (s), 1596 (w), 1501 (w), 1451 (w), 1394 (w), 1369 (m), 1148 (s), 847 (m), 702 (m) cm^{-1}; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.36-7.25 (5H, m, Ar**H**), 3.20 (1H, dd, *J* = 18.0, 2.0 Hz, C(O)C**H**HC), 2.96-2.90 (1H, m, C(O)CH_2CH**H**), 2.54 (1H, d, *J* = 18.0 Hz, C(O)CH**H**C), 2.35-2.25 (3H, m, C(O)C**H_2**CH_2 and C(O)CH_2CH**H**), 1.33 (9H, s, C(C**H_3**)$_3$); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 216.3, 173.5, 141.8, 128.8, 127.5, 126.6, 82.0, 55.9, 48.4, 37.3, 33.0, 27.9; **HRMS (CI+):** Calcd for $C_{16}H_{21}O_3$: 261.1492 [M^++1], Found 261.1491; **Optical Rotation:** $[\alpha]_D^{20}$ -7.11 (*c* 0.780, $CHCl_3$) for an enantiomerically enriched sample of 79% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (80% ee shown; chiral dex GTA column, 15 psi, 120 ºC).



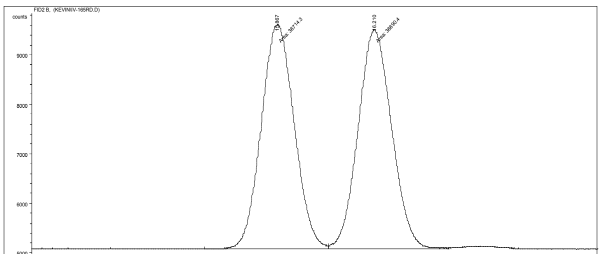
#	Time	Area	Height	Width	Area (%)
1	123.814	380.6	5.6	1.133	49.547
2	127.99	387.6	6.1	1.0667	50.453



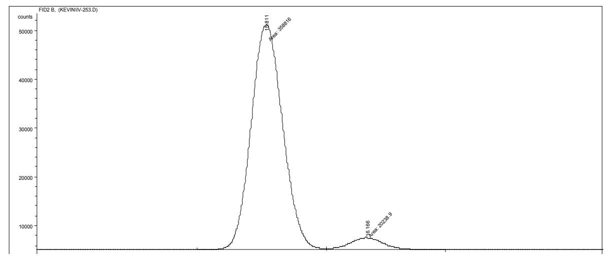
#	Time	Area	Height	Width	Area (%)
1	123.864	228.1	2.6	1.4718	9.701
2	129.285	2123.3	21.2	1.6727	90.299

(*R*)-Methyl-1-methyl-3-oxocyclohexanecarboxylate (Table 3, entry 8). IR (neat): 2952 (m), 2877 (w), 1734 (s), 1462 (m), 1318 (w), 1208 (m), 1168 (m), 1139 (m), 1110 (m); **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 3.61 (3H, s, OC**H_3**), 2.66 (1H, dt, *J* = 14.8, 1.6 Hz, CC**H**HC(O)), 2.31-2.24 (1H, m, $CCH_2CH_2CH_2C(O)$), 2.21-2.13 (1H, m, $CCH_2CH_2CH_2C(O)$, 2.11-2.02 (3H, m, $CCH_2CH_2CH_2C(O)$ and CCH**H**C(O)), 1.89-1.79 (1H, m, $CCH_2CH_2CH_2C(O)$), 1.73-1.60 (2H, m, $CCH_2CH_2CH_2C(O)$), 1.19 (3H, s, CC**H_3**); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 209.3, 176.6, 52.3, 50.0, 46.7, 40.3, 34.7, 24.8, 22.2; **HRMS (EI+):** Calcd for $C_9H_{14}O_3$: 170.094294, Found: 170.094668; **Optical Rotation:** $[\alpha]_D^{20}$ –17.7 (*c* 1.66, $CHCl_3$) for an enantiomerically enriched sample of 79% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (89% ee shown; β-dex column, 15 psi, 140 ºC).



#	Time	Area	Height	Width	Area (%)
1	15.867	36714.3	4530.7	0.1351	50.035
2	16.21	36690.4	4424.2	0.1382	49.964



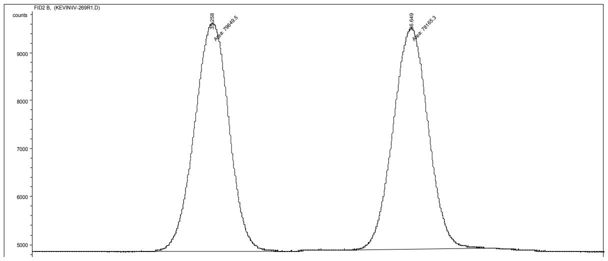
#	Time	Area	Height	Width	Area (%)
1	15.811	358815.7	46138.6	0.1296	94.734
2	16.166	20238.9	2447.1	0.1378	5.265

Proof of absolute stereochemistry: Basic hydrolysis (LiOH (5.0 equiv), THF/H_2O (0.8 M/0.8 M) 0 ºC -> 22 °C, 30 minutes, 68% yield, unoptimized) of (*R*)-methyl 1-methyl-3-oxocyclohexanecarboxylate yielded (*R*)-1-methyl-3-oxocyclohexanecarboxylic acid. $[\alpha]_D^{20}$ –13.1 (*c* 0.806, $CHCl_3$) for an enantiomerically enriched sample of 79% ee. Literature: $[\alpha]_D^{22}$ –14.0 (*c* 1.00, $CHCl_3$) for an enantiomerically enriched sample of 85% ee in the *R* enantiomer.[11]

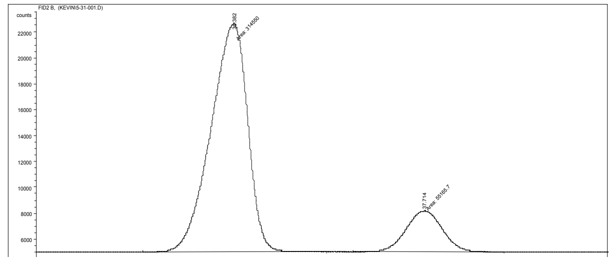
(*R*)-Methyl-1-isopropyl-3-oxocyclohexanecarboxylate (Table 3, entry 10). IR (neat): 2969 (m), 2877 (w), 1741 (s), 1567 (w), 1451 (m), 1382 (m), 1295 (m), 1242 (m), 1208 (m), 1167 (m), 1127 (m), 1092 (m), 988 (w), 889 (w), 750 (w); **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 3.66 (3H, s, OC**H**$_3$), 2.70 (1H, dt, *J* = 14.8, 2.4 Hz, CC**H**HC(O)), 2.40-2.33 (1H, m, $CH_2CH_2CH_2C$(O)), 2.21-1.91 (5H, m, $CH_2CH_2CH_2C$(O), C**H**$(CH_3)_2$ and CCH**H**C(O), 1.75 (1H, dt, *J* = 12.8, 3.6 Hz, $CH_2CH_2CH_2C$(O)), 1.58-1.46 (1H, m, $CH_2CH_2CH_2C$(O)), 0.91 (3H, d, *J* = 6.8 Hz, CHC**H**$_3$), 0.84 (3H, d, *J* = 6.8 Hz, CHC**H**$_3$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 209.8, 176.1, 65.0, 54.7, 52.1, 43.8, 40.6, 34.9, 31.9, 22.4, 18.2, 17.2; **HRMS (EI+):** Calcd for $C_{11}H_{18}O_3$: 198.125595, Found: 198.126392; **Optical Rotation:** $[\alpha]_D^{20}$ –23.8 (*c* 0.673, $CHCl_3$) for an enantiomerically enriched sample of 70% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (70% ee shown; β-dex column, 15 psi, 140 ºC).

[11] E. Díez, R. Fernández, C. Gasch, J. M. Lassaletta, J. M. Llera, E. Martín-Zamora, J. Vázquez, *J. Org. Chem.* **1997**, *62*, 5144-5155.



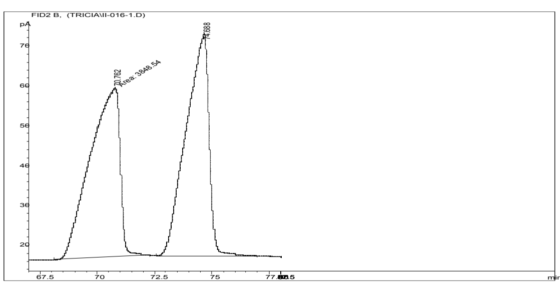
#	Time	Area	Height	Width	Area (%)
1	35.258	79649.5	4750.4	0.2794	50.546
2	36.649	78165.3	4595.4	0.2835	49.454



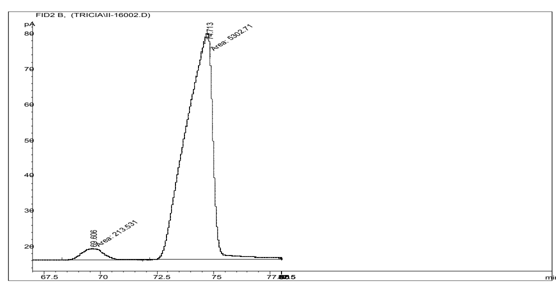
#	Time	Area	Height	Width	Area (%)
1	36.382	314549.9	17661.7	0.2968	84.851
2	37.714	55165.7	3147.9	0.2921	15.149

(*R*)-Methyl-3-oxo-1-phenylcyclohexanecarboxylate (Table 3, entry 11). mp: 96-98 °C; **IR (neat):** 2974 (m), 2961 (m), 2955 (m), 2879 (w), 1727 (s), 1495 (w), 1451 (m), 1325 (m), 1300 (m), 1249 (s), 1211 (s), 1155 (m), 1111 (m), 985 (w), 885 (w), 783 (w), 746 (m), 714 (m) cm^{-1}; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.36-7.28 (4H, m, Ar**H**), 7.28-7.24 (1H, m, Ar**H**), 3.64 (3H, s, $OC\mathbf{H}_3$), 3.04 (1H, dt, *J* = 14.8, 1.6 Hz, C(O)C**H**HC), 2.74 (1H, d, *J* = 14.8 Hz, C(O)CH**H**), 2.57-2.51 (1H, m, $CC\mathbf{H}_2CH_2$), 2.42-2.24 (3H, m, $CC\mathbf{H}_2CH_2$ and C(O)C**H**H), 1.88-1.66 (2H, m, $C(O)CH_2C\mathbf{H}_2$); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 208.8, 174.9, 141.3, 129.1, 127.7, 126.2, 54.1, 52.8, 49.5, 40.5, 33.3, 21.7; **HRMS (EI+):** Calcd for $C_{14}H_{16}O_3$: 232.2750, Found 232.1096; **Optical Rotation:** $[\alpha]_D^{20}$ –4.61 (*c* 0.700, $CHCl_3$) for an enantiomerically enriched sample of 92% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (92% ee shown; chiral dex GTA column, 15 psi, 140 °C).

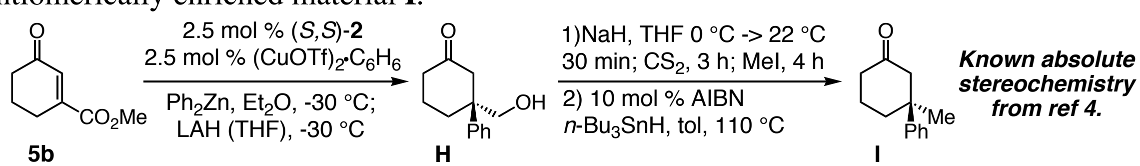


#	Time	Area	Height	Width	Area%
1	70.762	3848.5	42.3	1.5154	49.297
2	74.688	3958.2	56	1.1783	50.703



#	Time	Area	Height	Width	Area%
1	69.606	213.5	3.2	1.1166	3.871
2	74.713	5302.7	64	1.3818	96.129

Proof of absolute stereochemistry: The following sequence was carried out in order to obtain enantiomerically enriched material **I**.

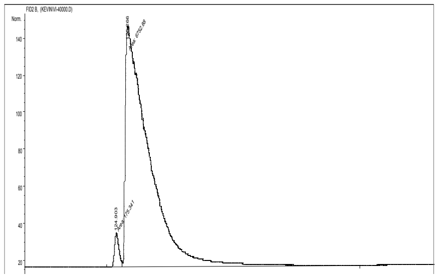


An oven-dried 13x100 mm test tube was charged with chiral Ag complex **3** (3.02 mg, 2.50 μmol), $(CuOTf)_2 \bullet C_6H_6$ (1.3 mg, 2.50 μmol), and Ph_2Zn (65.7 mg, 0.300 mmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm prior to removal from the glove box. The test tube containing the solids was allowed to cool to –30 ºC (dry ice/acetone bath) and Et_2O (1.0 mL) was added through a syringe. The resulting mixture was allowed to stir for 10 minutes. (During this time the mixture became dark brown.) At this time, methyl 3-oxocyclohex-1-enecarboxylate (**5b**) (13.4 μL, 15.4 mg, 0.100 mmol) was added to the mixture through a syringe. After 42 h at –30 ºC, $LiAlH_4$ (18.9 mg, 0.500 mmol) was added as a solution in THF (500 μL) via cannula. After 10 minutes at –30 ºC, the reaction was quenched upon addition of a saturated aqueous solution of sodium potassium tartrate (5 mL) and diluted with CH_2Cl_2 (5 mL). The mixture was allowed to warm to 22 ºC and stir for 2 h before it was extracted with CH_2Cl_2 (2 x 5 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated in vacuo to afford a clear oil. The clear oil was purified by silica gel chromatography (10% Et_2O/hexanes) to yield 9.6 mg of an inseparable mixture of desired product (**H**) and an unidentified compound (~25%). The mixture was carried on in the subsequent transformation.

Note: The following procedures are unoptimized. To a suspension of NaH (2.2 mg, 0.037 mmol, 60% dispersion in mineral oil) in THF (250 μL) at 0 ºC was added **H** (7.0 mg, 0.034 mmol) dissolved in THF (250 μL) via cannula. The mixture was allowed to warm to 22 ºC and stir for 1 h. At this time, CS_2 (8.2 μL, 0.14 mmol) was added to the mixture through a syringe resulting in a yellow solution. The mixture was allowed to stir for 3 h until MeI (6.7 mL, 0.11 mmol) was added through a syringe. After 30 min, the reaction was quenched upon addition of H_2O (1 mL) and washed with $CHCl_3$ (2 x 2 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated, resulting in a yellow solid that was purified by silica gel chromatography (10% Et_2O/petroleum ether -> 30% Et_2O/petroleum ether) to afford 2.6 mg of the desired xanthate contaminated with minor impurities. This material was carried on in the subsequent transformation.

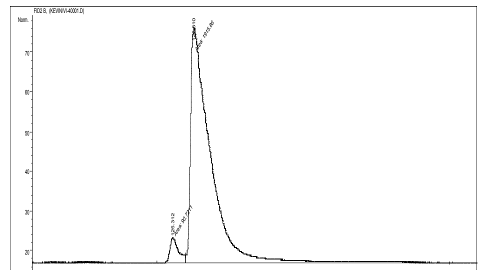
2,2'-Azobisisobutyronitrile (0.10 mg, 0.84 μmol) was added to the xanthate (2.6 mg, .0084 mmol) and Bu_3SnH (5.6 mL, 0.21 mmol) dissolved in toluene (250 μL) in a screw cap vial. The vial was sealed with a cap and allowed to stir at 110 ºC. After 1 h, the mixture was allowed to cool to 22 ºC and was passed through a short plug of silica gel (4 cm x 1 cm) eluted with 50% Et_2O/petroleum ether. The volatiles were removed in vacuo, resulting in a clear oil that was purified by silica gel chromatography (25% Et_2O/petroleum ether) to afford **I** as a colorless oil. The ^1H NMR spectrum of **I** was identical to previously reported data.[4] Based on the retention times from GLC chromatograms (illustrated below) of **I** derived from reactions outlined in ref 4 (which was assigned *S* absolute stereochemistry) and **H**, we assigned *R* absolute stereochemistry to the product in Table 3, entry 12.

***I** From Ref (4)*



#	Time	Area	Height	Width	Area (%)
1	124.903	175.3	18.8	0.1556	2.528
2	125.266	6752.9	129.8	0.8671	97.47

I** From **H



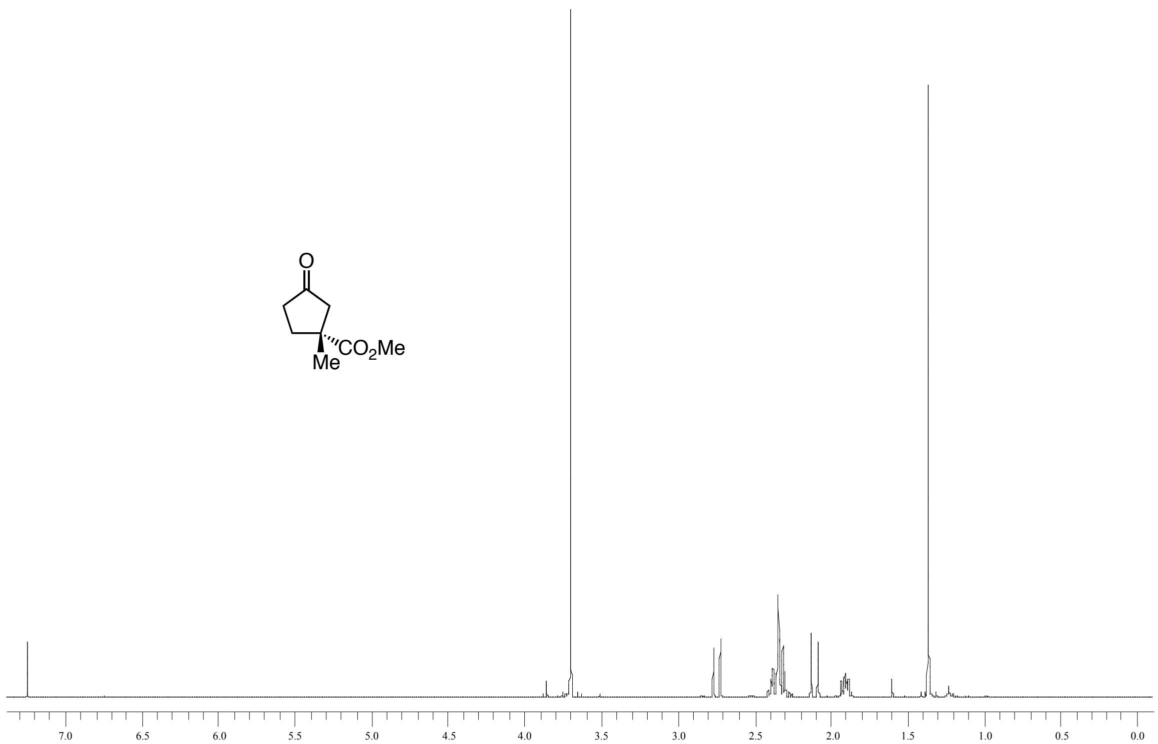
#	Time	Area	Height	Width	Area (%)
1	125.312	90.7	6.5	0.2326	4.520
2	125.81	1915.9	59.2	0.5391	95.48

(*R*)-*tert*-Butyl-1-methyl-3-oxocyclohexanecarboxylate (Table, 3, entry 9). IR (neat): 2978 (m), 2941 (m), 2884 (w), 1737 (s), 1469 (m), 1379 (m), 1327 (m), 1264 (m), 1232 (m), 1158 (s), 1132 (s), 848 (w); **^{1}H NMR ($CDCl_3$, 400 MHz):** δ 2.67 (1H, dt, *J* = 14.4, 2.0 Hz, C(O)C**H**HC), 2.39-2.31 (1H, m, $CH_2CH_2CH_2C(O)$), 2.20 (1H, dddd, *J* = 16.0, 9.6, 6.4, 1.2 Hz), 2.11-2.05 (2H, m, C(O)CH**H**C and $CH_2CH_2CH_2C(O)$), 1.95-1.86 (1H, m, $CH_2CH_2CH_2C(O)$), 1.82-1.68 (1H, m, $CH_2CH_2CH_2C(O)$), 1.63 (1H, ddd, *J* = 14.0, 10.4, 4.0 Hz, $CH_2CH_2CH_2C(O)$), 1.42 (9H, s, $C(C\mathbf{H}_3)_3$), 1.21 (3H, s, $C\mathbf{H}_3$); **^{13}C NMR ($CDCl_3$, 100 MHz):** δ 209.6, 175.3, 81.3, 50.3, 47.1, 40.3, 34.9, 28.1, 25.2, 22.3; **HRMS (CI+):** Calcd for $C_{12}H_{21}O_3$: 213.149070 [M^++1], Found: 213.149254; **Optical Rotation:** $[\alpha]_D^{20}$ –19.6 (*c* 1.25, $CHCl_3$) for an enantiomerically enriched sample of 85% ee.

Enantiomeric purity was determined by chiral GLC analysis in comparison with authentic racemic material (β-dex column, 15 psi, 140 °C, t_{major} = 19.98 min, t_{minor} = 20.25 min).

■ **Experimental procedure for Cu-catalyzed conjugate addition of *Me_2Zn* to unsaturated cyclic γ-ketoester 5b and in situ reduction with $LiAlH_4$**

(*R*)-3-(hydroxymethyl)-3-methylcyclohexanone (11). An oven-dried 13x100 mm test tube was charged with chiral Ag complex **3** (3.02 mg, 2.50 μmol) and $(CuOTf)_2$•C_6H_6 (1.30 mg, 2.50 μmol), weighed out under a N_2 atmosphere in a glove box. The test tube was sealed with a septum and wrapped with parafilm before removal from the glove box. *tert*-Butylmethylether (1.0 mL) was added through a syringe and the resulting solution was allowed to stir for five minutes before cooling to –78 °C (dry ice/acetone bath). Dimethylzinc (21.0 μL, 0.300 mmol) (PYROPHORIC, USE EXTREME CAUTION) was added and the resulting light yellow mixture was allowed to warm to –30 °C (cryocool). (During this time the mixture became dark brown.) After 10 minutes at –30 °C, methyl 3-oxocyclohex-1-enecarboxylate (**5b**) (13.4 μL, 15.4 mg, 0.100 mmol) was added to the mixture through a syringe. After 15 h at –30 °C, $LiAlH_4$ (4.00 mg, 0.100 mmol) dissolved in THF (1.0 mL) was added to the mixture via cannula. The solution was allowed to warm to 0 °C and stir for 6 h at which time the reaction was quenched upon addition of a saturated aqueous solution of sodium potassium tartrate (2.0 mL) and diluted with CH_2Cl_2 (2.0 mL). The resulting mixture was allowed to warm to 22 ºC and stir for 2 h, after which time the reaction was washed with CH_2Cl_2 (4 x 2 mL). The combined organic layers were dried over $MgSO_4$ and filtered through filter paper. The volatiles were removed in vacuo resulting in a yellow oil, which was purified by silica gel column chromatography (50% Et_2O/petroleum ether) to afford 10.8 mg (0.0760 mmol, 76.0%) of the desired product as a clear oil. **IR (neat):** 3390 (br), 2955 (m), 2867 (m), 1703 (s), 1457 (w), 1420 (w), 1055 (m), 671 (w) cm^{-1}; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 3.39 (1H, d, *J* = 11.6 Hz, CH**H**OH), 3.36 (1H, d, *J* = 12.4 Hz, C**H**HOH), 2.36 (1H, d, *J* = 13.6 Hz, C(O)CH**H**C), 2.32-2.23 (2H, m, C(O)C**H**$_2$CH$_2$), 2.06 (1H, dt, *J* = 13.6, 1.6 Hz, C(O)CH**H**C$_2$), 2.00-1.76 (3H, m, C(O)CH$_2$C**H**$_2$ and C(O)CH$_2$CH$_2$C**H**H), 1.52-1.47 (2H, m, CH$_2$O**H** and C(O)CH$_2$CH$_2$CH**H**), 0.92 (3H, s, C**H**$_3$); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 201.2, 71.3, 50.1, 41.1, 32.8, 22.6, 22.2, 10.6; **HRMS (EI+):** Calcd for $C_8H_{14}O_2$: 142.0994, Found 142.0944.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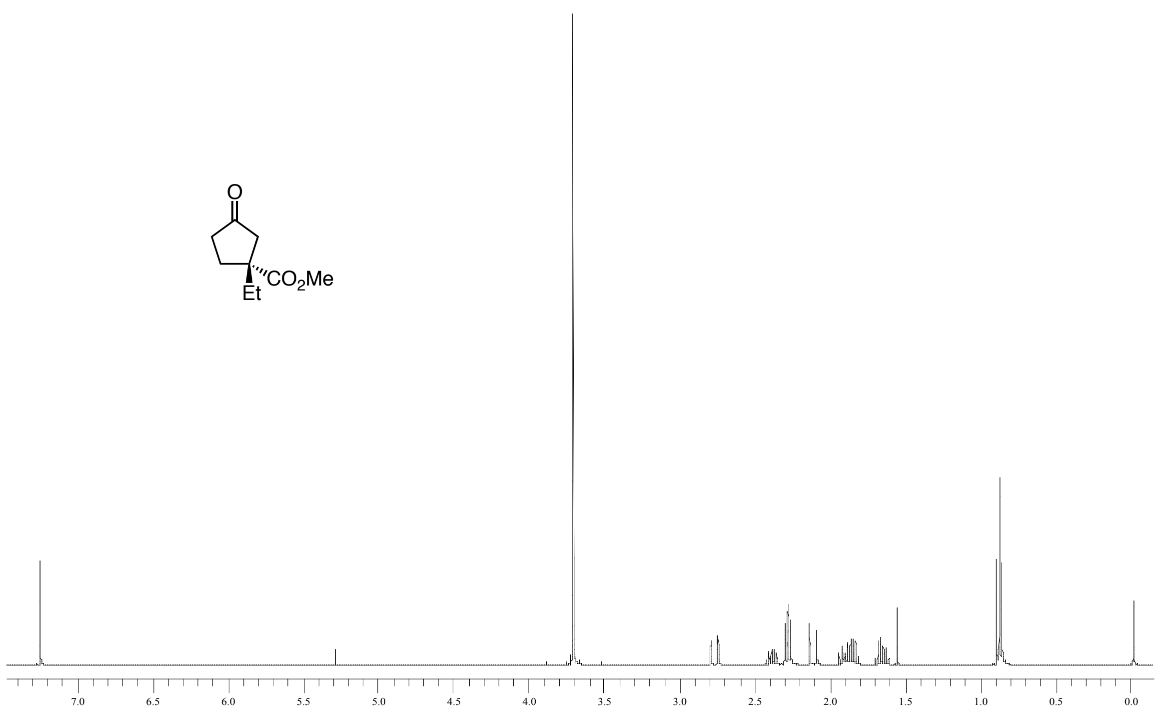
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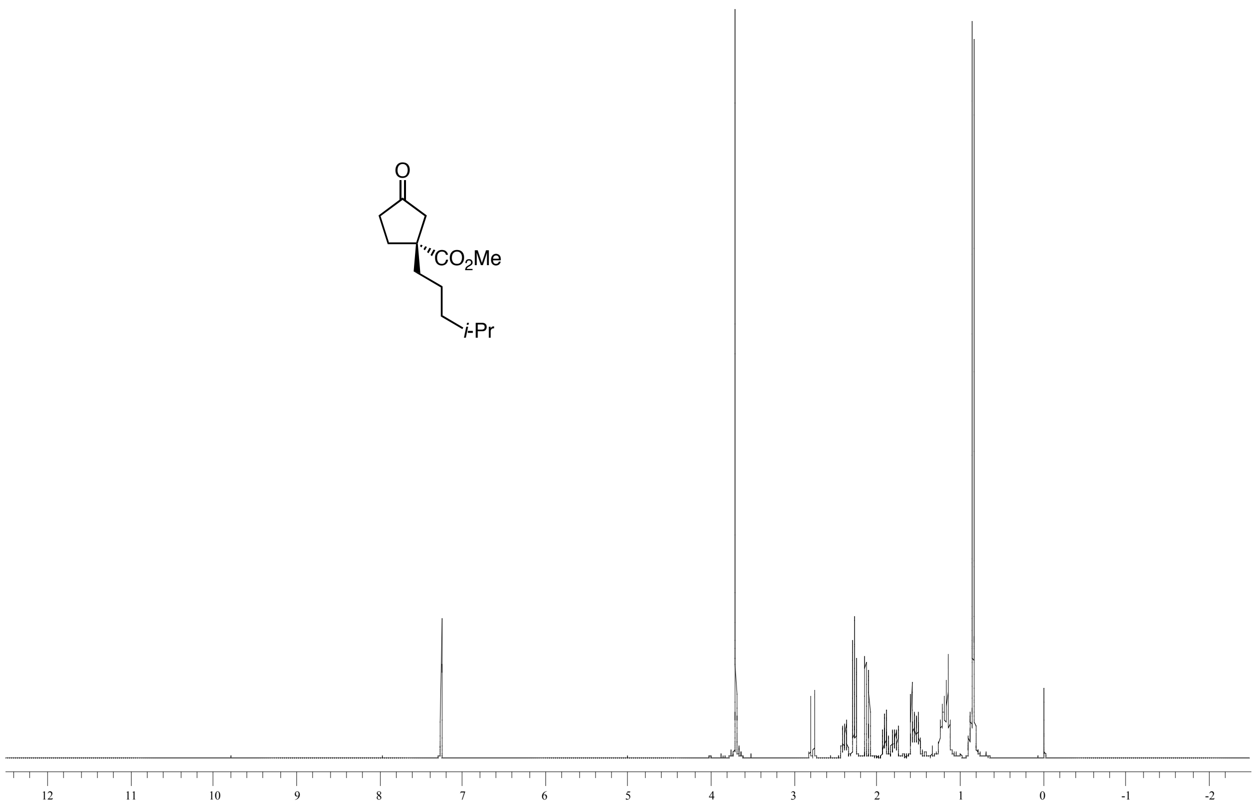
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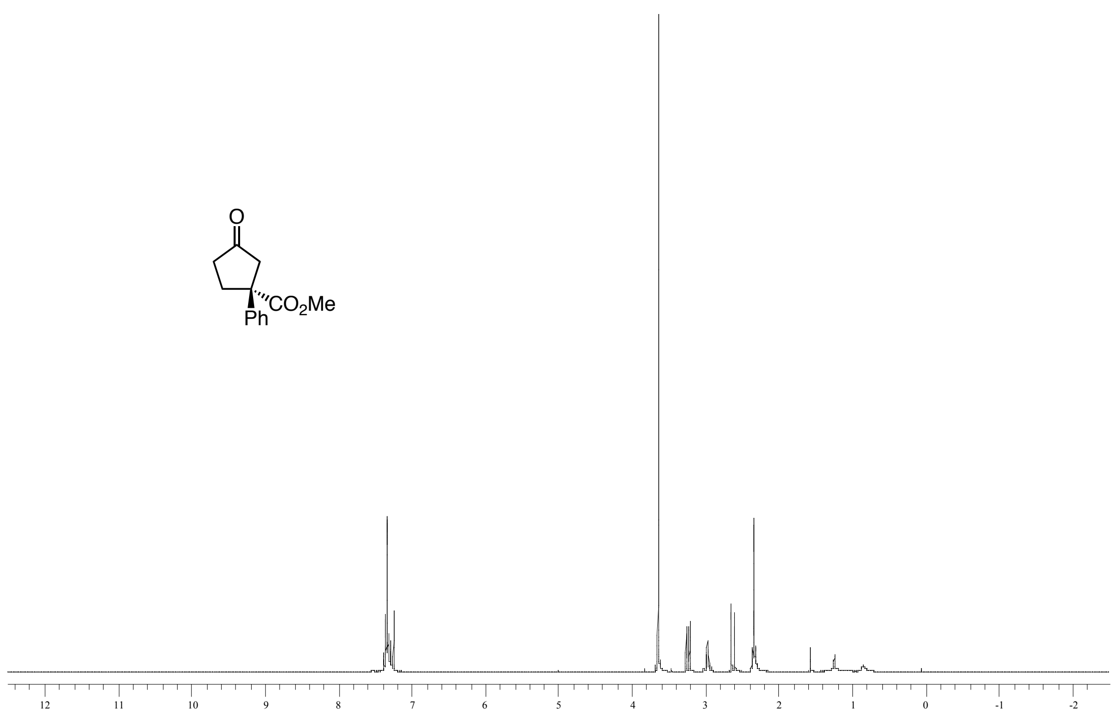
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CO2Me
Ph
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11
10
9
8
7
6
5
4
3
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1
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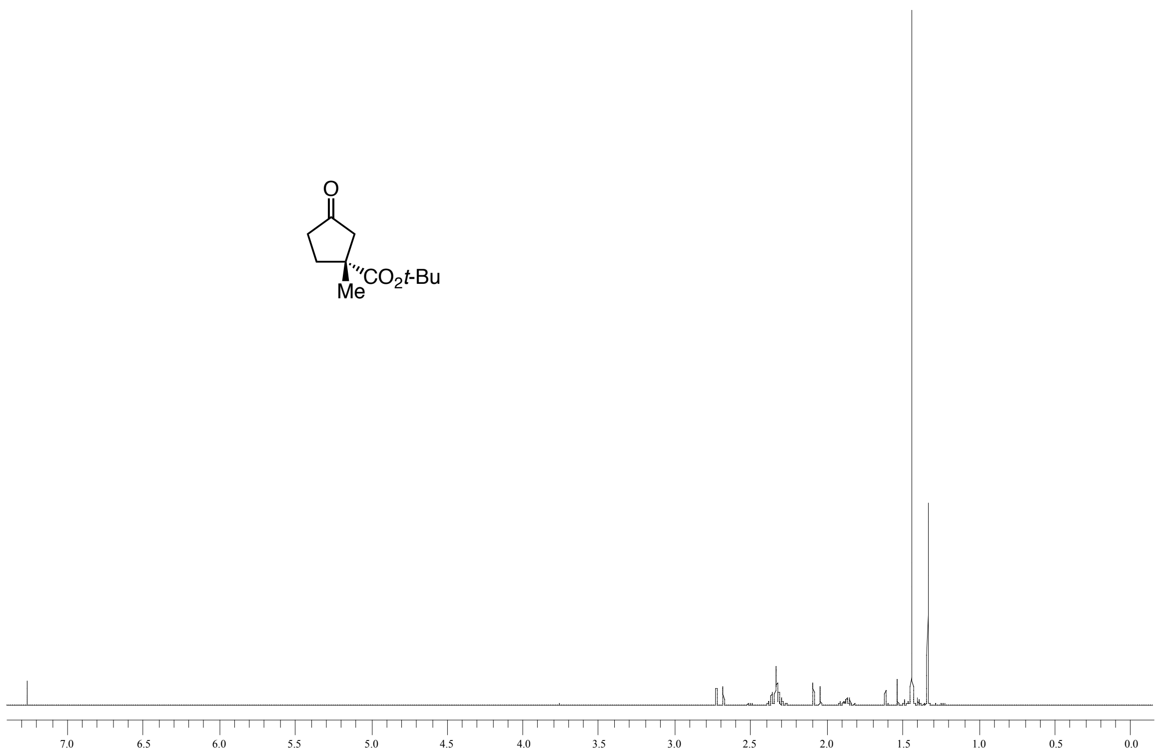
-1
-2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6.5
6.0
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3.5
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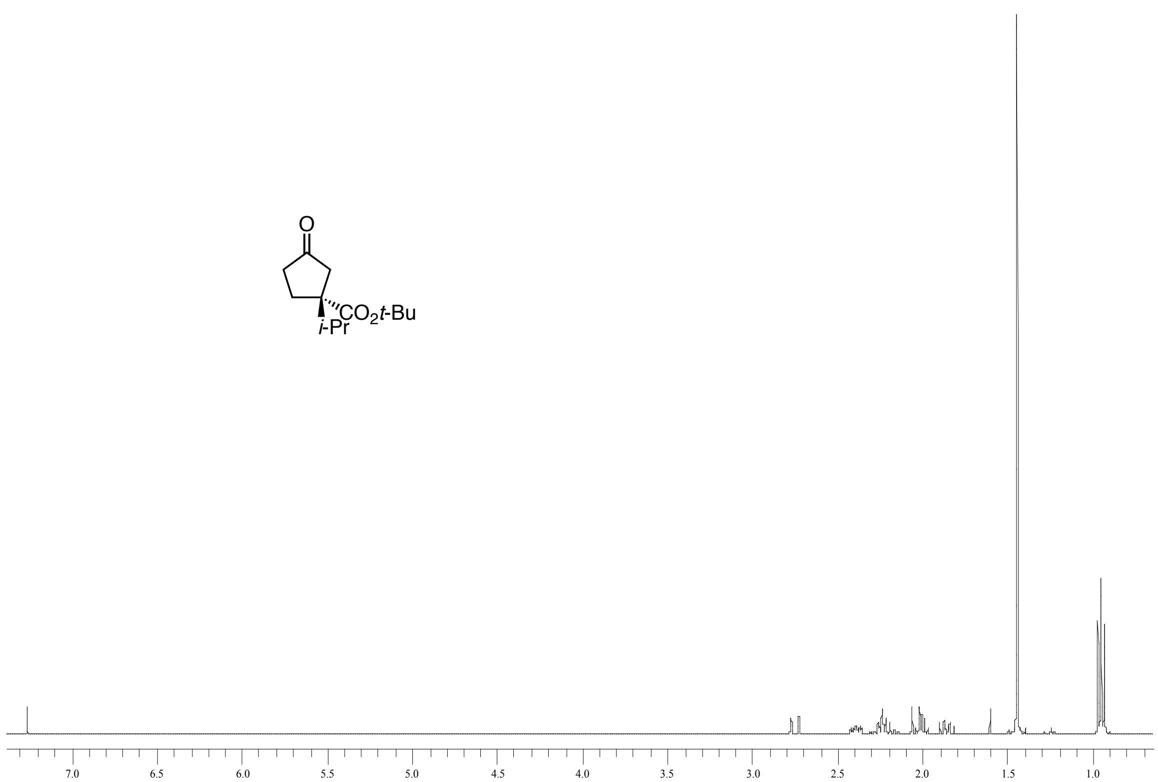
2.0
1.5
1.0
0.5
0.0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3.0
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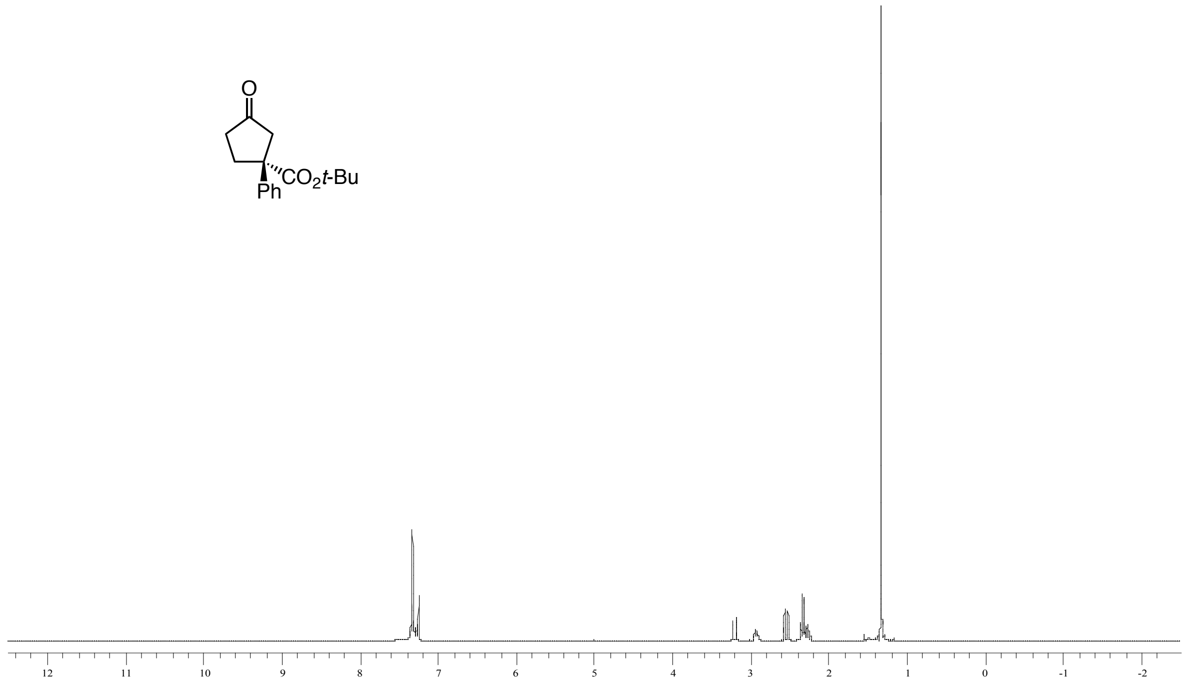
2.0
1.5
1.0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Ph
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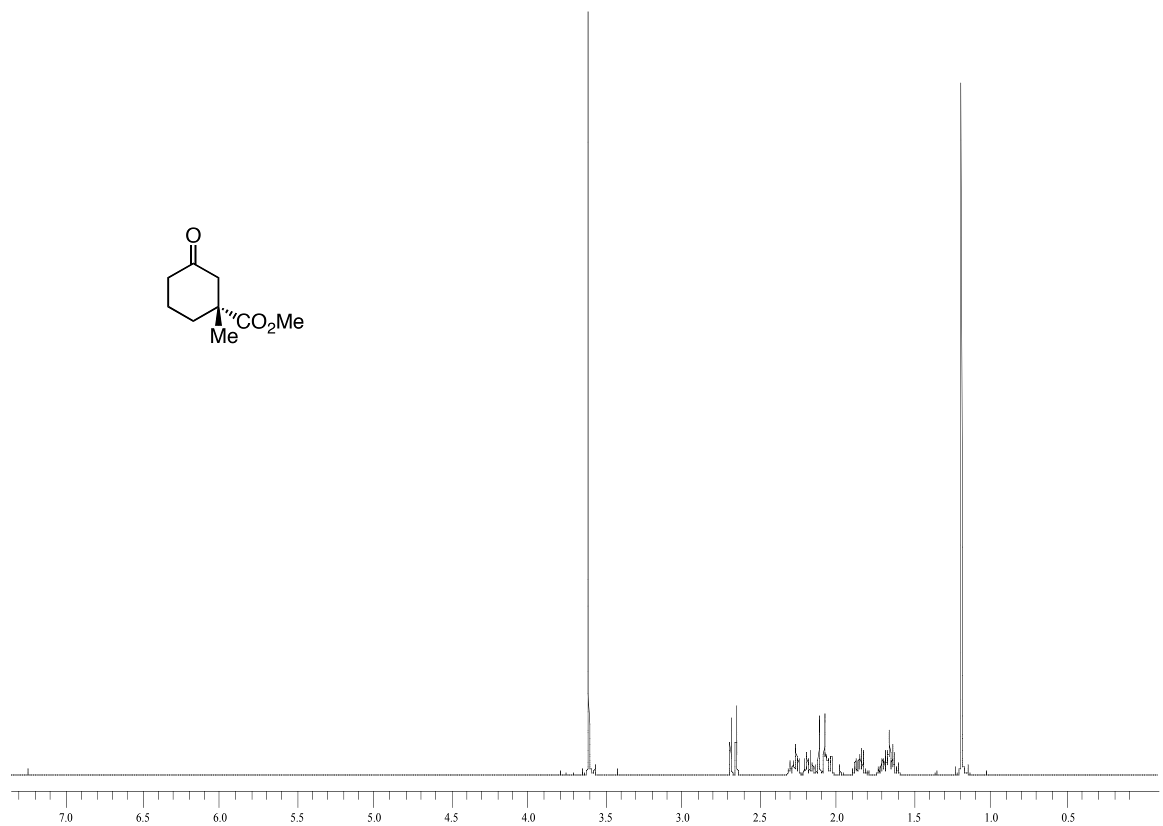
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Me
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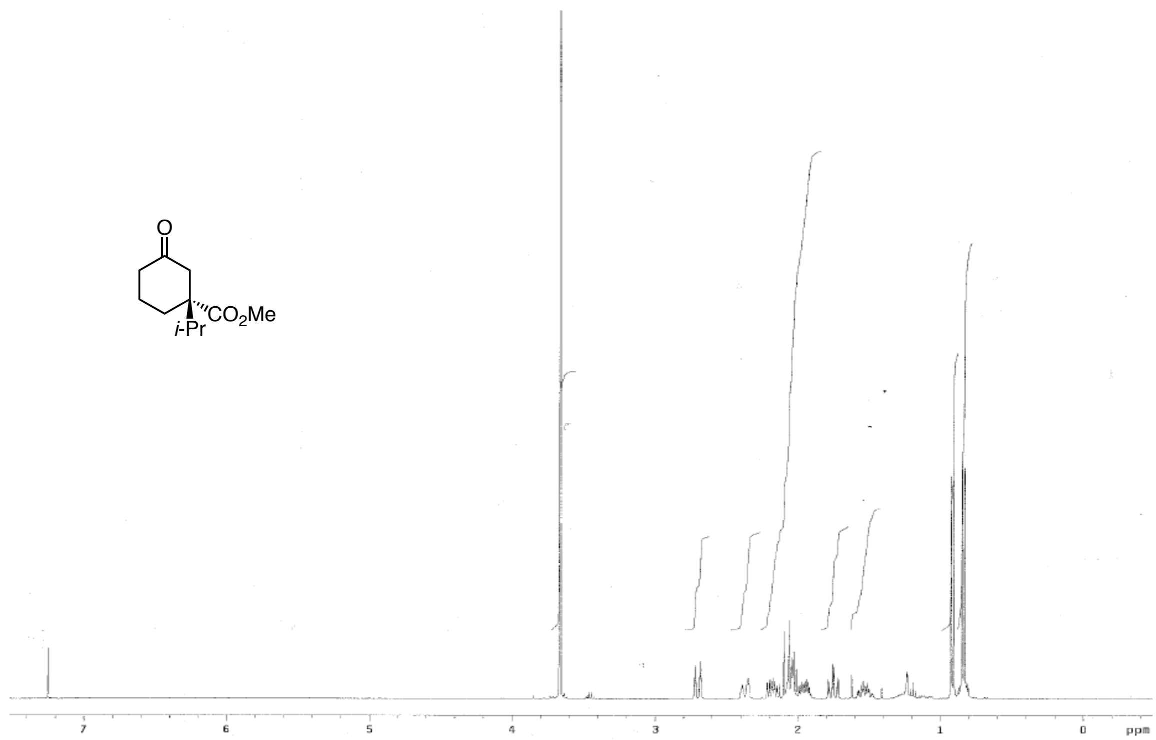
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CO2Me
i-Pr
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4
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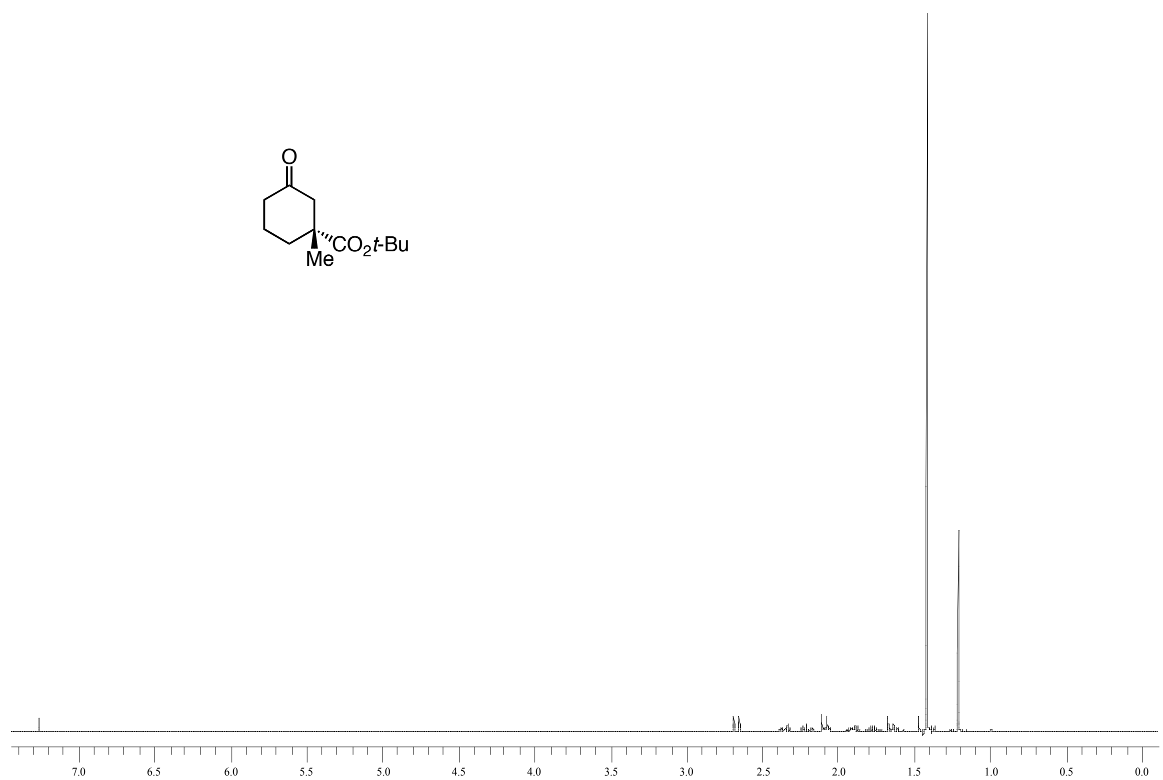
2
1
0
ppm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6.0
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2.5
2.0
1.5
1.0
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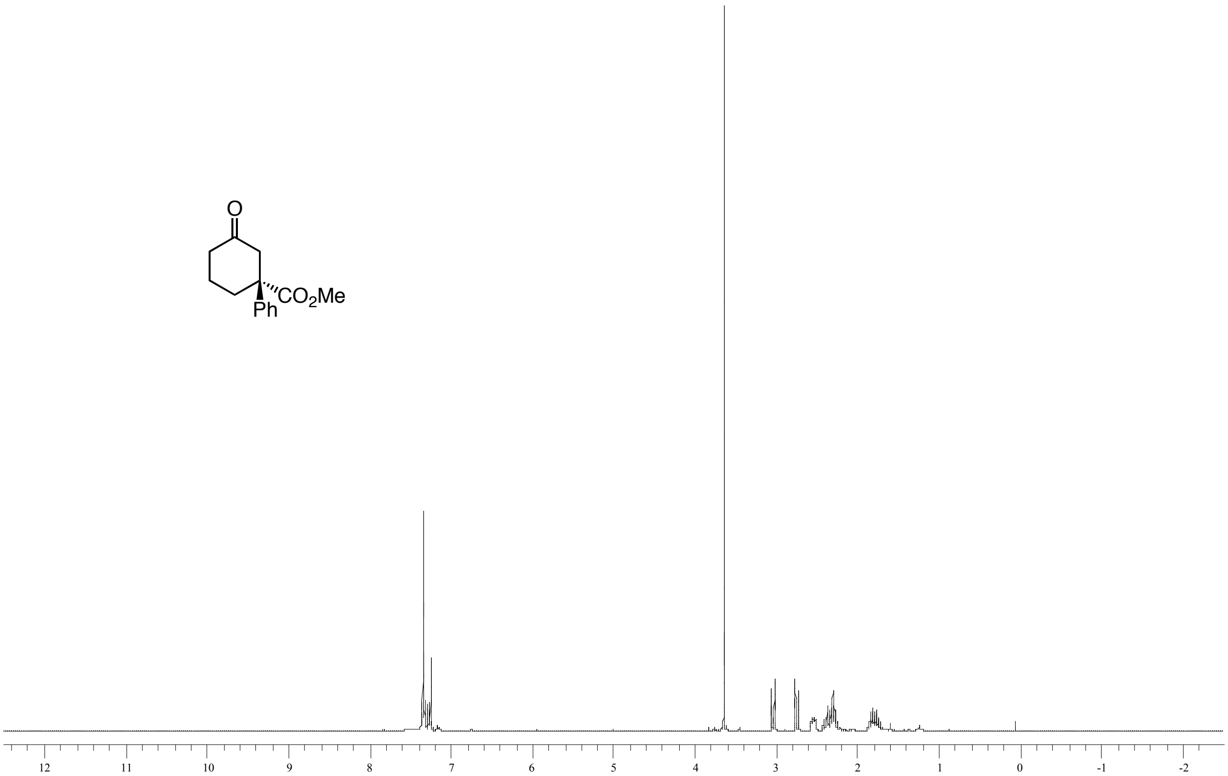
0.0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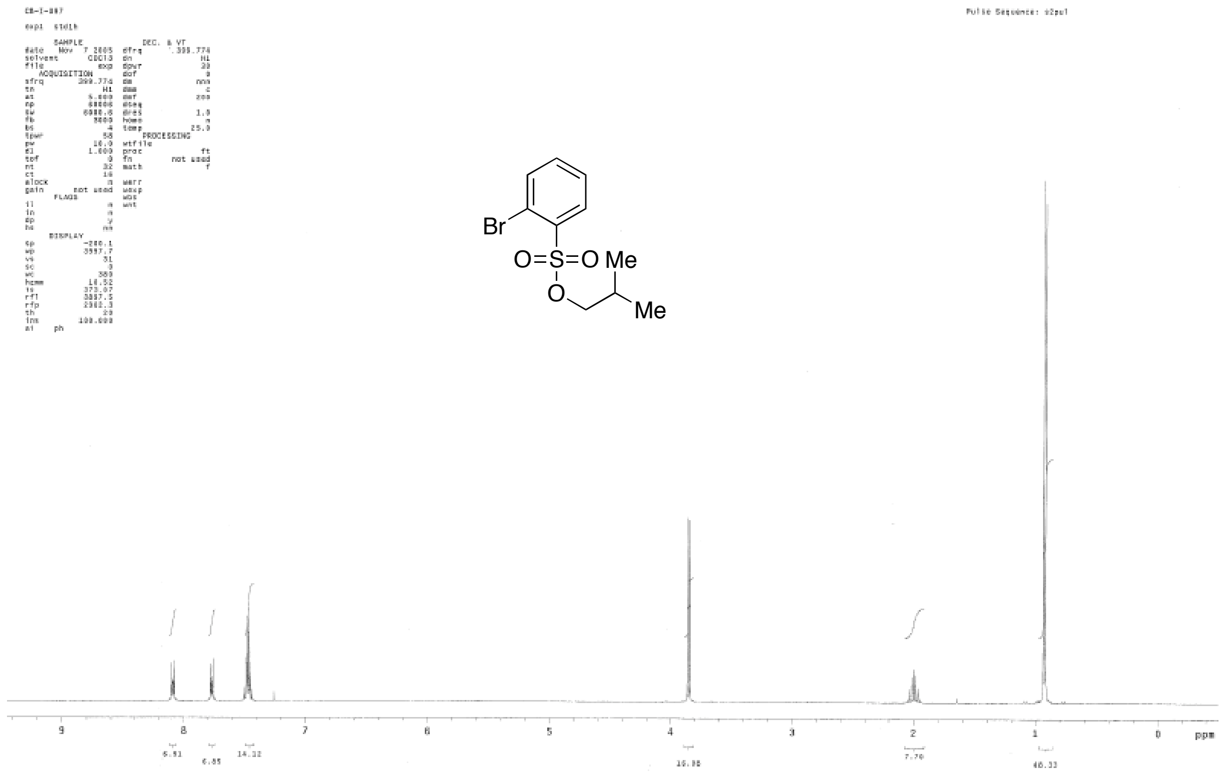
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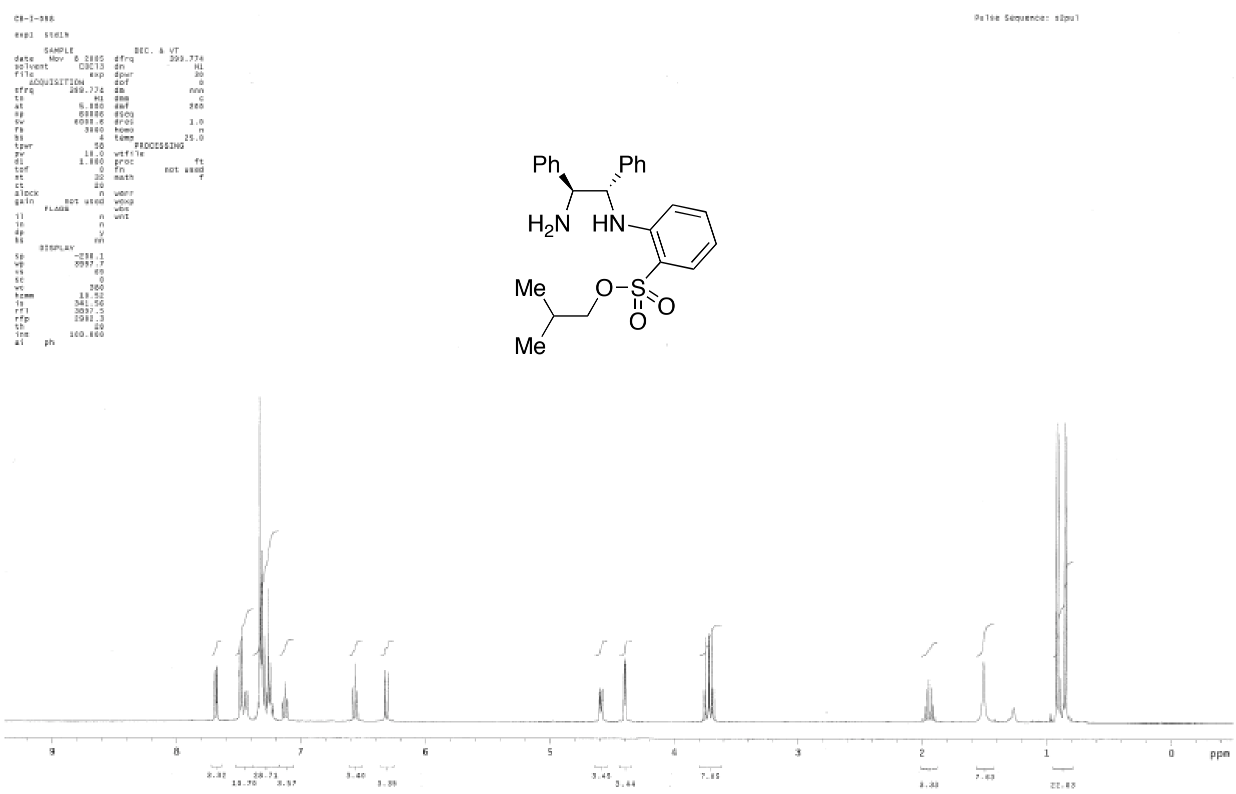
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Ph
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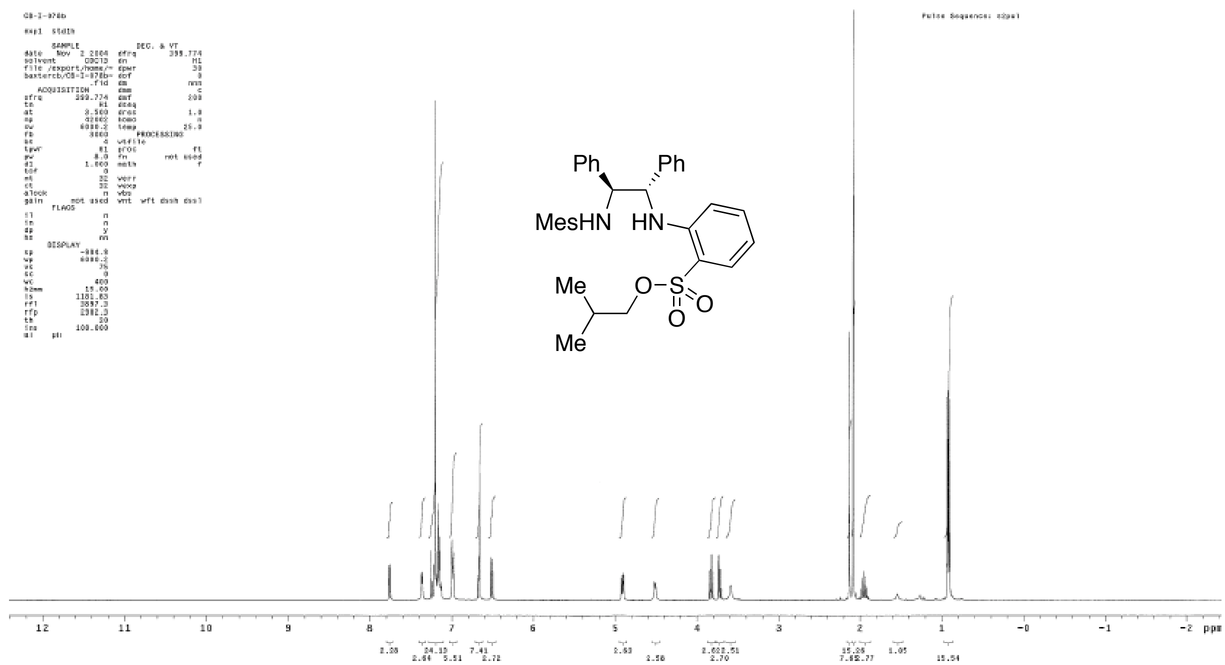
HN
Me
O–S
O
O
Me
ppm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Ph
MesHN
HN
Me
Me
O–S
O
O
12
11
10
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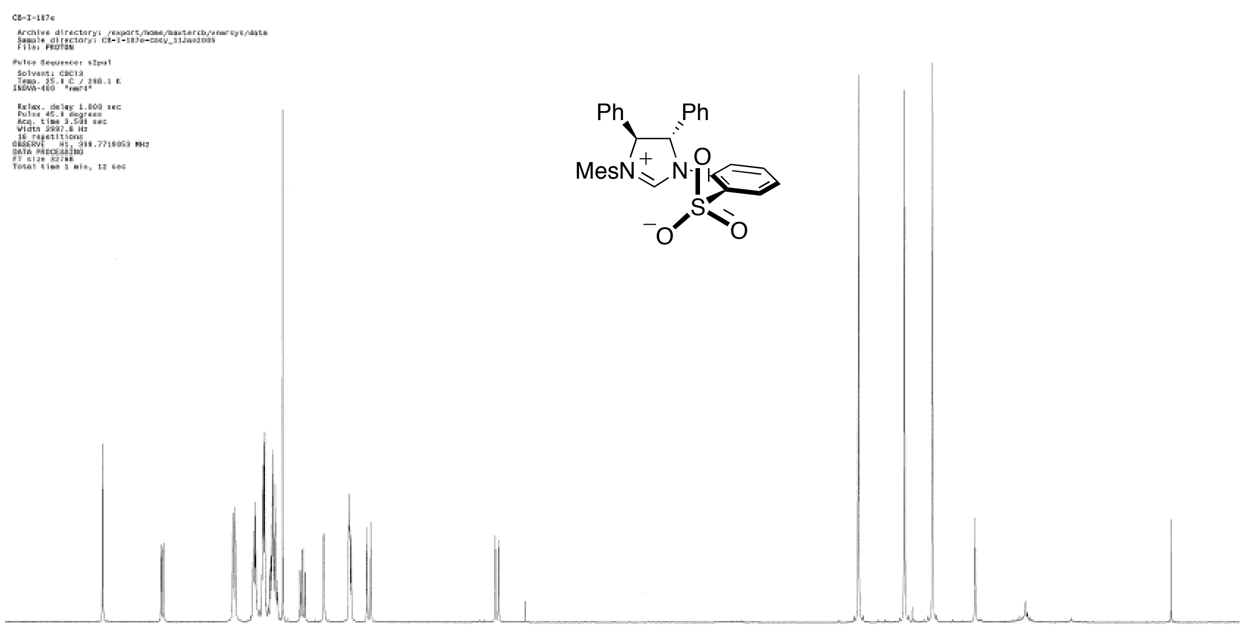
8
7
6
5
4
3
2
1
-0
-1
-2
ppm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Ph
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O
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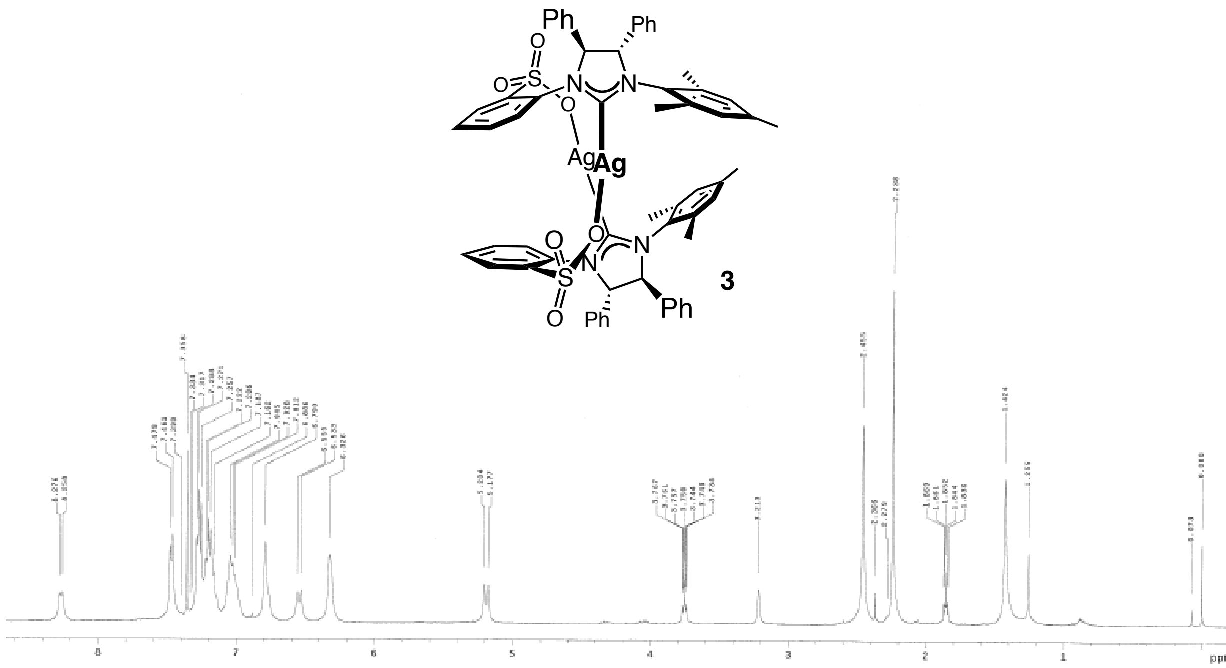
5
4
3
2
1
0
ppm



3

A New Chiral N-Heterocyclic Carbene for a New Catalytic Asymmetric Process. All-Carbon Quaternary Stereogenic Centers by Enantioselective Cu-Catalyzed Conjugate Addition of Organozinc Reagents to Unsaturated Cyclic γ-Ketoesters

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Boston College, Chestnut Hill, Massachusetts 02467

SUPPORTING INFORMATION (PART II)
X-RAY CRYSTAL INFORMATION

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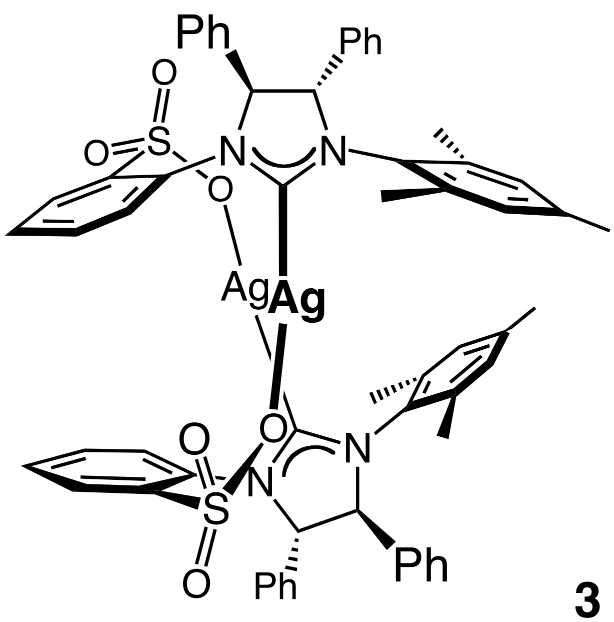
Ag Complex **3**



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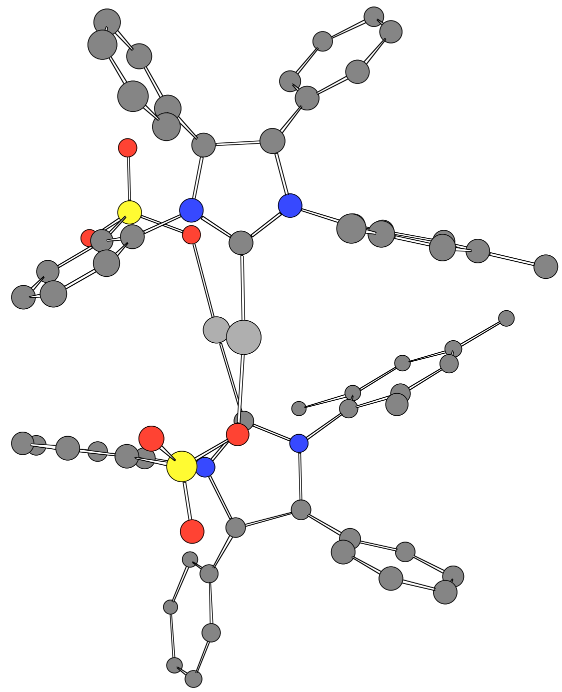


(Solvent molecules omitted for clarity)

Table 1. Crystal data and structure refinement for cb2t.

Identification code	cb2t	
Empirical formula	C37.50 H35 Ag Cl N2 O3 S	
Formula weight	737.05	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 8.520(2) Å	α= 90°.
	b = 24.792(7) Å	β= 97.595(6)°.
	c = 14.551(4) Å	γ = 90°.
Volume	3046.6(15) Å^3	
Z	4	
Density (calculated)	1.607 Mg/m^3	
Absorption coefficient	0.861 mm^{-1}	
F(000)	1512	
Crystal size	0.10 x 0.04 x 0.04 mm^3	

Theta range for data collection	1.41 to 22.50°.
Index ranges	-9<=h<=9, -26<=k<=26, -13<=l<=15
Reflections collected	13774
Independent reflections	7317 [R(int) = 0.0923]
Completeness to theta = 22.50°	99.8 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7317 / 433 / 736
Goodness-of-fit on F^2	0.737
Final R indices [I>2sigma(I)]	R1 = 0.0574, wR2 = 0.0885
R indices (all data)	R1 = 0.1209, wR2 = 0.1035
Absolute structure parameter	-0.02(4)
Largest diff. peak and hole	0.538 and -0.585 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for cb2t. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ag(1)	10711(1)	8097(1)	6896(1)	37(1)
Ag(2)	7153(1)	8147(1)	6852(1)	41(1)
S(1)	7045(4)	8326(2)	9032(3)	34(1)
S(2)	10844(4)	6889(2)	6174(3)	38(1)
O(1)	5689(10)	7978(4)	9019(5)	45(3)
O(4)	10866(10)	6550(4)	5365(6)	38(3)
O(5)	12169(13)	6801(4)	6955(8)	66(4)
O(7)	7233(10)	8707(4)	9769(5)	40(3)
O(8)	10633(9)	7459(3)	5906(6)	28(2)
O(9)	7127(9)	8579(3)	8140(5)	31(2)
N(3)	7309(14)	7964(4)	4774(8)	29(3)
N(4)	7258(12)	7217(5)	5504(7)	29(3)
N(5)	10608(12)	8556(4)	8805(7)	17(3)
N(6)	10873(12)	9205(5)	7842(6)	26(3)
C(1)	8764(18)	7881(5)	9216(8)	31(4)
C(2)	10244(14)	8028(6)	9102(8)	21(4)
C(3)	11369(16)	7644(5)	9218(9)	28(4)
C(4)	11090(20)	7127(8)	9428(11)	55(6)
C(5)	9640(20)	6982(7)	9541(10)	48(5)
C(6)	8470(20)	7360(7)	9419(9)	46(4)
C(007)	7550(19)	6865(6)	6279(10)	38(4)
C(8)	9976(16)	10016(5)	5721(9)	34(4)
C(9)	8281(17)	7646(5)	3308(9)	37(4)
C(10)	6919(19)	8517(6)	4578(10)	35(4)
C(11)	8122(17)	8884(6)	4330(9)	33(4)
C(12)	9846(14)	8708(6)	4443(9)	46(5)
C(13)	7746(19)	9403(6)	4092(10)	40(4)
C(014)	9135(19)	6684(6)	6670(9)	31(4)

C(14)	5410(16)	8718(6)	4520(9)	34(4)
C(15)	4067(15)	8341(6)	4777(9)	60(6)
C(16)	12784(17)	9833(6)	5994(11)	49(4)
C(17)	11450(20)	10101(5)	5427(10)	42(4)
C(18)	6230(20)	9597(8)	4026(12)	54(5)
C(19)	12560(19)	9534(7)	6760(10)	31(4)
C(20)	14021(14)	9300(6)	7307(10)	50(5)
C(021)	11070(18)	9481(6)	6974(10)	32(4)
C(21)	8078(16)	9590(6)	6660(10)	42(5)
C(23)	8826(19)	10083(7)	8949(9)	55(5)
C(024)	7148(16)	7567(5)	4016(9)	27(4)
C(24)	10353(16)	10010(6)	8759(9)	32(4)
C(25)	11135(14)	9483(5)	8753(8)	28(4)
C(26)	7312(16)	7030(5)	4538(9)	29(4)
C(27)	6073(19)	6639(6)	4177(10)	31(4)
C(28)	6418(17)	6266(6)	3556(11)	44(4)
C(29)	9822(16)	7447(5)	3418(9)	34(4)
C(30)	7800(16)	7948(5)	2532(8)	45(5)
C(31)	8771(18)	8056(9)	1866(10)	63(4)
C(032)	9774(18)	9701(6)	6456(9)	31(4)
C(32)	9310(20)	6345(6)	7403(10)	56(5)
C(33)	8020(20)	6149(6)	7786(11)	56(5)
C(34)	6528(18)	6300(5)	7448(9)	42(4)
C(35)	6379(19)	6665(6)	6711(10)	43(4)
C(36)	11767(17)	8981(5)	10285(10)	35(4)
C(37)	11090(20)	10463(7)	8595(9)	57(5)
C(38)	10360(20)	10968(7)	8589(12)	73(6)
C(39)	8850(30)	11025(8)	8758(13)	88(7)
C(40)	8100(20)	10579(7)	8945(11)	70(6)
C(41)	10717(17)	9039(6)	9393(11)	30(4)
C(42)	5120(18)	9237(6)	4286(9)	41(4)
C(43)	11694(18)	10432(6)	4561(9)	65(5)
C(44)	4586(17)	6674(6)	4360(10)	43(4)
C(45)	3384(19)	6313(6)	3923(10)	57(5)
C(46)	3770(20)	5957(6)	3296(10)	57(5)
C(47)	5307(19)	5920(6)	3119(11)	49(4)

C(48)	10317(18)	7829(6)	1969(10)	53(5)
C(49)	10830(15)	7537(6)	2753(9)	40(4)
C(50)	5793(19)	10154(6)	3749(11)	65(6)
C(51)	10631(14)	8673(6)	7925(11)	36(4)
C(52)	7244(16)	7753(6)	5593(10)	39(4)
C(56)	13304(17)	8781(6)	10287(9)	47(4)
C(57)	11318(19)	9146(6)	11105(10)	54(4)
C(58)	12346(19)	9103(7)	11930(10)	60(5)
C(60)	14301(19)	8706(6)	11081(9)	56(5)
C(61)	13840(30)	8856(8)	11914(13)	83(7)
Cl(1)	5353(6)	6492(2)	355(4)	107(2)
Cl(2)	4231(7)	7219(3)	1688(4)	109(2)
C(53)	5580(20)	7111(8)	931(14)	117(8)
Cl(3)	2009(9)	5263(3)	8482(5)	180(3)
Cl(4)	5247(9)	5215(3)	9392(6)	170(3)
C(55)	3540(20)	4821(8)	8826(17)	146(10)
O(1S)	4023(10)	7769(3)	7262(6)	49(3)

Table 3. Bond lengths [Å] and angles [°] for cb2t.

Ag(1)-C(51)	2.076(14)
Ag(1)-O(8)	2.133(8)
Ag(1)-Ag(2)	3.0268(17)
Ag(2)-C(52)	2.088(15)
Ag(2)-O(9)	2.160(8)
S(1)-O(7)	1.423(8)
S(1)-O(9)	1.453(8)
S(1)-O(1)	1.439(9)
S(1)-C(1)	1.824(13)
S(2)-O(4)	1.449(8)
S(2)-O(8)	1.472(8)
S(2)-O(5)	1.508(10)
S(2)-C(014)	1.782(15)
N(3)-C(52)	1.308(17)
N(3)-C(10)	1.431(17)
N(3)-C(024)	1.472(16)
N(4)-C(52)	1.336(16)
N(4)-C(007)	1.421(16)
N(4)-C(26)	1.487(16)
N(5)-C(51)	1.316(17)
N(5)-C(2)	1.426(16)
N(5)-C(41)	1.467(17)
N(6)-C(51)	1.344(15)
N(6)-C(021)	1.465(17)
N(6)-C(25)	1.485(14)
C(1)-C(2)	1.343(17)
C(1)-C(6)	1.356(17)
C(2)-C(3)	1.347(16)
C(3)-C(4)	1.347(19)
C(3)-H(3)	0.9300
C(4)-C(5)	1.32(2)
C(4)-H(4)	0.9300
C(5)-C(6)	1.360(19)
C(5)-H(5)	0.9300

C(6)-H(6)	0.9300
C(007)-C(35)	1.342(19)
C(007)-C(014)	1.465(19)
C(8)-C(032)	1.354(17)
C(8)-C(17)	1.393(18)
C(8)-H(8)	0.9300
C(9)-C(30)	1.371(16)
C(9)-C(29)	1.391(16)
C(9)-C(024)	1.515(18)
C(10)-C(14)	1.370(18)
C(10)-C(11)	1.45(2)
C(11)-C(13)	1.360(17)
C(11)-C(12)	1.520(17)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-C(18)	1.37(2)
C(13)-H(13)	0.9300
C(014)-C(32)	1.351(18)
C(14)-C(42)	1.346(17)
C(14)-C(15)	1.560(17)
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-C(19)	1.374(19)
C(16)-C(17)	1.474(18)
C(16)-H(16)	0.9300
C(17)-C(43)	1.541(17)
C(18)-C(42)	1.39(2)
C(18)-C(50)	1.47(2)
C(19)-C(021)	1.35(2)
C(19)-C(20)	1.502(18)
C(20)-H(20A)	0.9600
C(20)-H(20B)	0.9600
C(20)-H(20C)	0.9600
C(021)-C(032)	1.365(18)

C(21)-C(032)	1.538(18)
C(21)-H(21A)	0.9600
C(21)-H(21B)	0.9600
C(21)-H(21C)	0.9600
C(23)-C(24)	1.378(18)
C(23)-C(40)	1.378(19)
C(23)-H(23)	0.9300
C(024)-C(26)	1.530(17)
C(024)-H(024)	0.9800
C(24)-C(37)	1.324(18)
C(24)-C(25)	1.468(17)
C(25)-C(41)	1.514(18)
C(25)-H(25)	0.9800
C(26)-C(27)	1.477(18)
C(26)-H(26)	0.9800
C(27)-C(28)	1.351(19)
C(27)-C(44)	1.331(18)
C(28)-C(47)	1.371(18)
C(28)-H(28)	0.9300
C(29)-C(49)	1.395(16)
C(29)-H(29)	0.9300
C(30)-C(31)	1.381(18)
C(30)-H(30)	0.9300
C(31)-C(48)	1.42(2)
C(31)-H(31)	0.9300
C(32)-C(33)	1.38(2)
C(32)-H(32)	0.9300
C(33)-C(34)	1.355(18)
C(33)-H(33)	0.9300
C(34)-C(35)	1.396(18)
C(34)-H(34)	0.9300
C(35)-H(35)	0.9300
C(36)-C(57)	1.362(17)
C(36)-C(56)	1.400(17)
C(36)-C(41)	1.483(19)
C(37)-C(38)	1.40(2)

C(37)-H(37)	0.9300
C(38)-C(39)	1.35(2)
C(38)-H(38)	0.9300
C(39)-C(40)	1.33(2)
C(39)-H(39)	0.9300
C(40)-H(40)	0.9300
C(41)-H(41)	0.9800
C(42)-H(42)	0.9300
C(43)-H(43A)	0.9600
C(43)-H(43B)	0.9600
C(43)-H(43C)	0.9600
C(44)-C(45)	1.444(18)
C(44)-H(44)	0.9300
C(45)-C(46)	1.342(18)
C(45)-H(45)	0.9300
C(46)-C(47)	1.370(19)
C(46)-H(46)	0.9300
C(47)-H(47)	0.9300
C(48)-C(49)	1.373(17)
C(48)-H(48)	0.9300
C(49)-H(49)	0.9300
C(50)-H(50A)	0.9600
C(50)-H(50B)	0.9600
C(50)-H(50C)	0.9600
C(56)-C(60)	1.353(16)
C(56)-H(56)	0.9300
C(57)-C(58)	1.394(18)
C(57)-H(57)	0.9300
C(58)-C(61)	1.42(2)
C(58)-H(58)	0.9300
C(60)-C(61)	1.37(2)
C(60)-H(60)	0.9300
C(61)-H(61)	0.9300
Cl(1)-C(53)	1.746(17)
Cl(2)-C(53)	1.717(18)
C(53)-H(53A)	0.9700

C(53)-H(53B)	0.9700
Cl(3)-C(55)	1.729(16)
Cl(4)-C(55)	1.85(2)
C(55)-H(55A)	0.9700
C(55)-H(55B)	0.9700
O(1S)-H(1S)	0.8499
O(1S)-H(2S)	0.8499

C(51)-Ag(1)-O(8)	174.3(5)
C(51)-Ag(1)-Ag(2)	81.8(3)
O(8)-Ag(1)-Ag(2)	94.3(2)
C(52)-Ag(2)-O(9)	177.7(5)
C(52)-Ag(2)-Ag(1)	81.1(4)
O(9)-Ag(2)-Ag(1)	97.3(2)
O(7)-S(1)-O(9)	111.8(5)
O(7)-S(1)-O(1)	114.8(5)
O(9)-S(1)-O(1)	112.4(5)
O(7)-S(1)-C(1)	106.4(6)
O(9)-S(1)-C(1)	105.0(6)
O(1)-S(1)-C(1)	105.5(6)
O(4)-S(2)-O(8)	110.9(6)
O(4)-S(2)-O(5)	116.0(7)
O(8)-S(2)-O(5)	113.1(6)
O(4)-S(2)-C(014)	105.2(6)
O(8)-S(2)-C(014)	107.7(6)
O(5)-S(2)-C(014)	102.9(7)
S(2)-O(8)-Ag(1)	122.8(5)
S(1)-O(9)-Ag(2)	124.6(5)
C(52)-N(3)-C(10)	121.9(13)
C(52)-N(3)-C(024)	113.8(12)
C(10)-N(3)-C(024)	119.7(12)
C(52)-N(4)-C(007)	122.5(12)
C(52)-N(4)-C(26)	113.7(11)
C(007)-N(4)-C(26)	122.4(12)
C(51)-N(5)-C(2)	121.8(12)
C(51)-N(5)-C(41)	112.4(11)

C(2)-N(5)-C(41)	124.9(11)
C(51)-N(6)-C(021)	124.9(12)
C(51)-N(6)-C(25)	112.5(11)
C(021)-N(6)-C(25)	122.1(11)
C(2)-C(1)-C(6)	119.1(14)
C(2)-C(1)-S(1)	124.5(11)
C(6)-C(1)-S(1)	116.2(13)
C(3)-C(2)-C(1)	117.1(14)
C(3)-C(2)-N(5)	120.6(12)
C(1)-C(2)-N(5)	122.1(12)
C(2)-C(3)-C(4)	124.0(16)
C(2)-C(3)-H(3)	118.0
C(4)-C(3)-H(3)	118.0
C(5)-C(4)-C(3)	118.9(18)
C(5)-C(4)-H(4)	120.6
C(3)-C(4)-H(4)	120.6
C(4)-C(5)-C(6)	118.6(17)
C(4)-C(5)-H(5)	120.7
C(6)-C(5)-H(5)	120.7
C(5)-C(6)-C(1)	122.3(16)
C(5)-C(6)-H(6)	118.9
C(1)-C(6)-H(6)	118.9
C(35)-C(007)-N(4)	122.4(15)
C(35)-C(007)-C(014)	114.1(14)
N(4)-C(007)-C(014)	123.5(13)
C(032)-C(8)-C(17)	122.9(14)
C(032)-C(8)-H(8)	118.5
C(17)-C(8)-H(8)	118.5
C(30)-C(9)-C(29)	117.6(14)
C(30)-C(9)-C(024)	118.8(13)
C(29)-C(9)-C(024)	123.5(13)
C(14)-C(10)-N(3)	123.4(17)
C(14)-C(10)-C(11)	116.5(14)
N(3)-C(10)-C(11)	119.8(14)
C(13)-C(11)-C(10)	120.4(14)
C(13)-C(11)-C(12)	119.4(15)

C(10)-C(11)-C(12)	119.9(13)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-C(18)	122.6(16)
C(11)-C(13)-H(13)	118.7
C(18)-C(13)-H(13)	118.7
C(32)-C(014)-C(007)	119.8(14)
C(32)-C(014)-S(2)	119.1(14)
C(007)-C(014)-S(2)	121.1(11)
C(42)-C(14)-C(10)	120.1(15)
C(42)-C(14)-C(15)	121.1(13)
C(10)-C(14)-C(15)	118.7(14)
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(19)-C(16)-C(17)	121.2(14)
C(19)-C(16)-H(16)	119.4
C(17)-C(16)-H(16)	119.4
C(8)-C(17)-C(16)	114.9(13)
C(8)-C(17)-C(43)	123.9(14)
C(16)-C(17)-C(43)	121.2(14)
C(42)-C(18)-C(13)	115.2(17)
C(42)-C(18)-C(50)	121.3(18)
C(13)-C(18)-C(50)	123.4(18)
C(16)-C(19)-C(021)	118.3(15)
C(16)-C(19)-C(20)	116.4(14)
C(021)-C(19)-C(20)	125.3(14)
C(19)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20B)	109.5

H(20A)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(19)-C(021)-C(032)	123.3(15)
C(19)-C(021)-N(6)	117.5(12)
C(032)-C(021)-N(6)	119.1(14)
C(032)-C(21)-H(21A)	109.5
C(032)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5
C(032)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5
C(24)-C(23)-C(40)	123.7(17)
C(24)-C(23)-H(23)	118.2
C(40)-C(23)-H(23)	118.2
N(3)-C(024)-C(26)	102.5(10)
N(3)-C(024)-C(9)	114.7(11)
C(26)-C(024)-C(9)	115.2(12)
N(3)-C(024)-H(024)	108.0
C(26)-C(024)-H(024)	108.0
C(9)-C(024)-H(024)	108.0
C(37)-C(24)-C(23)	114.2(15)
C(37)-C(24)-C(25)	121.8(14)
C(23)-C(24)-C(25)	124.1(14)
C(24)-C(25)-N(6)	113.8(10)
C(24)-C(25)-C(41)	120.0(12)
N(6)-C(25)-C(41)	101.1(11)
C(24)-C(25)-H(25)	107.1
N(6)-C(25)-H(25)	107.1
C(41)-C(25)-H(25)	107.1
N(4)-C(26)-C(27)	115.5(12)
N(4)-C(26)-C(024)	100.7(11)
C(27)-C(26)-C(024)	112.3(12)
N(4)-C(26)-H(26)	109.3
C(27)-C(26)-H(26)	109.3

C(024)-C(26)-H(26)	109.3
C(28)-C(27)-C(44)	118.4(15)
C(28)-C(27)-C(26)	118.5(15)
C(44)-C(27)-C(26)	122.7(14)
C(27)-C(28)-C(47)	122.7(15)
C(27)-C(28)-H(28)	118.6
C(47)-C(28)-H(28)	118.6
C(9)-C(29)-C(49)	121.4(13)
C(9)-C(29)-H(29)	119.3
C(49)-C(29)-H(29)	119.3
C(9)-C(30)-C(31)	122.8(15)
C(9)-C(30)-H(30)	118.6
C(31)-C(30)-H(30)	118.6
C(30)-C(31)-C(48)	118.8(16)
C(30)-C(31)-H(31)	120.6
C(48)-C(31)-H(31)	120.6
C(8)-C(032)-C(021)	119.3(15)
C(8)-C(032)-C(21)	118.4(13)
C(021)-C(032)-C(21)	122.3(13)
C(33)-C(32)-C(014)	121.7(16)
C(33)-C(32)-H(32)	119.1
C(014)-C(32)-H(32)	119.1
C(34)-C(33)-C(32)	120.9(15)
C(34)-C(33)-H(33)	119.5
C(32)-C(33)-H(33)	119.5
C(33)-C(34)-C(35)	116.3(15)
C(33)-C(34)-H(34)	121.8
C(35)-C(34)-H(34)	121.8
C(007)-C(35)-C(34)	127.0(16)
C(007)-C(35)-H(35)	116.5
C(34)-C(35)-H(35)	116.5
C(57)-C(36)-C(56)	118.4(15)
C(57)-C(36)-C(41)	122.0(15)
C(56)-C(36)-C(41)	119.5(13)
C(24)-C(37)-C(38)	122.5(18)
C(24)-C(37)-H(37)	118.7

C(38)-C(37)-H(37)	118.7
C(39)-C(38)-C(37)	122(2)
C(39)-C(38)-H(38)	119.1
C(37)-C(38)-H(38)	119.1
C(40)-C(39)-C(38)	116.9(19)
C(40)-C(39)-H(39)	121.5
C(38)-C(39)-H(39)	121.5
C(39)-C(40)-C(23)	121(2)
C(39)-C(40)-H(40)	119.6
C(23)-C(40)-H(40)	119.6
N(5)-C(41)-C(36)	114.5(13)
N(5)-C(41)-C(25)	103.7(12)
C(36)-C(41)-C(25)	116.4(12)
N(5)-C(41)-H(41)	107.2
C(36)-C(41)-H(41)	107.2
C(25)-C(41)-H(41)	107.2
C(18)-C(42)-C(14)	125.1(15)
C(18)-C(42)-H(42)	117.5
C(14)-C(42)-H(42)	117.5
C(17)-C(43)-H(43A)	109.5
C(17)-C(43)-H(43B)	109.5
H(43A)-C(43)-H(43B)	109.5
C(17)-C(43)-H(43C)	109.5
H(43A)-C(43)-H(43C)	109.5
H(43B)-C(43)-H(43C)	109.5
C(27)-C(44)-C(45)	120.6(14)
C(27)-C(44)-H(44)	119.7
C(45)-C(44)-H(44)	119.7
C(46)-C(45)-C(44)	119.0(16)
C(46)-C(45)-H(45)	120.5
C(44)-C(45)-H(45)	120.5
C(47)-C(46)-C(45)	119.9(16)
C(47)-C(46)-H(46)	120.1
C(45)-C(46)-H(46)	120.1
C(46)-C(47)-C(28)	119.3(15)
C(46)-C(47)-H(47)	120.3

C(28)-C(47)-H(47)	120.3
C(49)-C(48)-C(31)	119.0(15)
C(49)-C(48)-H(48)	120.5
C(31)-C(48)-H(48)	120.5
C(48)-C(49)-C(29)	120.2(13)
C(48)-C(49)-H(49)	119.9
C(29)-C(49)-H(49)	119.9
C(18)-C(50)-H(50A)	109.5
C(18)-C(50)-H(50B)	109.5
H(50A)-C(50)-H(50B)	109.5
C(18)-C(50)-H(50C)	109.5
H(50A)-C(50)-H(50C)	109.5
H(50B)-C(50)-H(50C)	109.5
N(6)-C(51)-N(5)	108.9(12)
N(6)-C(51)-Ag(1)	126.2(11)
N(5)-C(51)-Ag(1)	123.9(11)
N(3)-C(52)-N(4)	108.0(13)
N(3)-C(52)-Ag(2)	128.5(12)
N(4)-C(52)-Ag(2)	123.5(11)
C(60)-C(56)-C(36)	122.1(14)
C(60)-C(56)-H(56)	118.9
C(36)-C(56)-H(56)	118.9
C(36)-C(57)-C(58)	120.9(16)
C(36)-C(57)-H(57)	119.5
C(58)-C(57)-H(57)	119.5
C(61)-C(58)-C(57)	118.9(15)
C(61)-C(58)-H(58)	120.6
C(57)-C(58)-H(58)	120.6
C(56)-C(60)-C(61)	119.8(17)
C(56)-C(60)-H(60)	120.1
C(61)-C(60)-H(60)	120.1
C(58)-C(61)-C(60)	119.5(18)
C(58)-C(61)-H(61)	120.3
C(60)-C(61)-H(61)	120.3
Cl(2)-C(53)-Cl(1)	113.7(10)
Cl(2)-C(53)-H(53A)	108.8

Cl(1)-C(53)-H(53A)	108.8
Cl(2)-C(53)-H(53B)	108.8
Cl(1)-C(53)-H(53B)	108.8
H(53A)-C(53)-H(53B)	107.7
Cl(3)-C(55)-Cl(4)	108.3(12)
Cl(3)-C(55)-H(55A)	110.0
Cl(4)-C(55)-H(55A)	110.0
Cl(3)-C(55)-H(55B)	110.0
Cl(4)-C(55)-H(55B)	110.0
H(55A)-C(55)-H(55B)	108.4
H(1S)-O(1S)-H(2S)	96.2

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for cb2t. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ag(1)	44(1)	38(1)	28(1)	-2(1)	3(1)	0(1)
Ag(2)	53(1)	44(1)	25(1)	-2(1)	3(1)	15(1)
S(1)	20(2)	51(3)	31(2)	3(2)	5(2)	-9(2)
S(2)	29(2)	44(3)	40(3)	-3(2)	-2(2)	-5(2)
O(1)	49(6)	57(9)	34(6)	-3(5)	22(5)	-24(6)
O(4)	27(6)	59(7)	29(6)	-16(5)	11(5)	3(5)
O(5)	40(8)	64(9)	81(10)	18(7)	-39(8)	-8(7)
O(7)	20(6)	89(8)	14(5)	-30(5)	14(5)	-16(5)
O(8)	25(6)	22(6)	35(6)	-5(4)	-7(5)	-6(5)
O(9)	24(5)	43(6)	25(6)	2(4)	2(5)	7(5)
N(3)	30(7)	35(10)	22(7)	5(6)	7(6)	-2(6)
N(4)	19(7)	48(8)	22(7)	7(6)	5(6)	0(6)
N(5)	1(6)	43(8)	4(6)	7(6)	-7(5)	2(5)
N(6)	23(7)	50(8)	3(6)	-5(5)	-9(6)	9(6)
C(1)	55(11)	24(9)	12(8)	-7(6)	-4(7)	9(8)
C(2)	0(7)	33(11)	29(7)	-3(8)	-9(6)	3(8)
C(3)	29(9)	20(9)	33(10)	0(8)	-5(8)	15(8)
C(4)	70(13)	66(13)	18(10)	1(9)	-28(10)	9(11)
C(5)	63(12)	37(11)	44(10)	9(8)	9(10)	-17(10)
C(6)	59(12)	55(12)	20(9)	2(8)	-7(9)	-4(10)
C(007)	48(11)	40(10)	23(10)	-6(8)	1(9)	-7(9)
C(8)	25(9)	42(10)	35(10)	0(8)	0(8)	-2(8)
C(9)	33(9)	46(9)	33(9)	-9(8)	11(8)	3(8)
C(10)	37(10)	58(12)	8(8)	-4(8)	-7(8)	35(9)
C(11)	26(9)	57(11)	15(8)	10(8)	-2(7)	25(9)
C(12)	4(8)	84(13)	52(11)	5(9)	10(8)	29(8)
C(13)	45(11)	33(10)	43(10)	15(8)	11(9)	10(9)
C(014)	52(11)	33(10)	13(9)	-4(7)	20(9)	-16(9)
C(14)	28(9)	51(11)	26(8)	0(7)	12(7)	11(8)
C(15)	45(11)	82(16)	58(11)	4(9)	18(9)	2(10)

C(16)	35(10)	49(10)	61(11)	-12(9)	1(9)	-9(8)
C(17)	72(12)	17(9)	34(10)	-3(7)	-3(9)	-25(9)
C(18)	78(14)	55(13)	27(11)	-7(9)	4(10)	9(11)
C(19)	34(10)	42(11)	19(9)	13(7)	15(8)	-6(8)
C(20)	16(8)	65(12)	71(12)	26(9)	12(9)	10(8)
C(021)	35(10)	30(9)	24(9)	0(7)	-27(9)	3(8)
C(21)	19(10)	70(13)	30(10)	7(9)	-22(9)	12(9)
C(23)	64(12)	61(12)	40(10)	5(8)	10(9)	22(10)
C(024)	24(8)	39(9)	17(9)	13(7)	1(7)	7(7)
C(24)	22(9)	37(9)	35(9)	-4(7)	1(7)	-9(8)
C(25)	8(7)	59(10)	14(8)	-8(7)	-8(6)	13(7)
C(26)	16(8)	38(10)	34(9)	-1(7)	3(7)	11(7)
C(27)	28(9)	45(10)	20(9)	-18(7)	5(8)	-3(8)
C(28)	22(9)	55(12)	53(11)	-3(9)	-5(8)	9(8)
C(29)	37(9)	44(9)	22(8)	-2(7)	8(8)	7(8)
C(30)	35(9)	79(14)	19(8)	10(8)	-7(7)	7(8)
C(31)	64(10)	94(12)	30(7)	-3(11)	-1(7)	1(13)
C(032)	39(10)	43(10)	12(9)	6(7)	6(8)	5(8)
C(32)	80(13)	45(11)	40(10)	20(8)	-3(10)	-3(10)
C(33)	70(12)	57(12)	40(11)	5(8)	0(10)	2(11)
C(34)	58(11)	36(10)	35(10)	4(7)	15(9)	-27(9)
C(35)	47(11)	45(11)	39(10)	-10(8)	10(9)	-17(9)
C(36)	30(9)	38(9)	39(10)	-10(8)	10(8)	7(8)
C(37)	77(12)	47(11)	44(10)	-8(8)	-6(9)	1(10)
C(38)	93(15)	36(11)	84(13)	-3(10)	-16(13)	-3(11)
C(39)	111(17)	65(14)	78(13)	-28(11)	-21(13)	71(14)
C(40)	98(15)	59(13)	53(11)	9(10)	13(10)	20(12)
C(41)	16(8)	38(10)	40(10)	-6(8)	16(8)	-12(8)
C(42)	38(10)	51(11)	28(9)	7(8)	-13(8)	30(9)
C(43)	80(13)	65(13)	49(11)	27(9)	3(10)	-27(10)
C(44)	40(10)	50(11)	35(9)	-26(8)	-4(8)	-11(9)
C(45)	59(11)	78(13)	32(10)	18(9)	-9(9)	10(10)
C(46)	79(13)	63(12)	23(9)	-20(8)	-14(10)	-8(10)
C(47)	57(11)	32(10)	54(11)	-7(8)	-1(10)	17(9)
C(48)	49(11)	79(12)	31(9)	-4(8)	4(9)	4(9)
C(49)	7(8)	75(11)	37(10)	6(8)	-2(8)	8(8)

C(50)	89(15)	40(12)	66(13)	13(10)	10(12)	13(11)
C(51)	0(8)	36(10)	71(12)	-18(9)	3(8)	8(7)
C(52)	18(8)	56(11)	41(10)	-9(9)	-4(8)	-9(8)
C(56)	45(10)	81(11)	13(8)	-10(8)	-10(8)	4(9)
C(57)	69(12)	66(11)	30(10)	-8(8)	20(9)	-20(9)
C(58)	57(12)	103(13)	20(9)	-19(9)	4(9)	6(11)
C(60)	59(11)	96(13)	9(9)	-34(9)	-10(8)	17(10)
C(61)	89(15)	103(16)	54(13)	-15(12)	-2(12)	-26(14)
Cl(1)	95(4)	93(5)	142(5)	-31(4)	49(4)	-26(4)
Cl(2)	70(4)	139(6)	123(6)	-29(5)	32(4)	-15(4)
C(53)	99(17)	112(18)	150(20)	-51(16)	73(16)	-11(15)
Cl(3)	200(8)	197(8)	156(6)	57(6)	74(6)	111(7)
Cl(4)	146(7)	145(7)	222(8)	1(6)	38(6)	8(5)
C(55)	69(16)	150(20)	200(30)	20(18)	-44(17)	53(15)
O(1S)	30(6)	65(7)	51(7)	21(5)	-1(6)	13(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for cb2t.

	x	y	z	U(eq)
H(3)	12401	7742	9150	34
H(4)	11908	6875	9491	66
H(5)	9420	6630	9701	57
H(6)	7443	7257	9477	55
H(8)	9092	10183	5398	41
H(12A)	10173	8655	3843	69
H(12B)	9962	8377	4787	69
H(12C)	10492	8982	4771	69
H(13)	8549	9635	3968	48
H(15A)	4105	8328	5439	91
H(15B)	4212	7985	4544	91
H(15C)	3058	8479	4507	91
H(16)	13798	9867	5829	59
H(20A)	14253	9491	7883	76
H(20B)	14896	9334	6958	76
H(20C)	13847	8926	7430	76
H(21A)	7764	9234	6453	63
H(21B)	7370	9850	6338	63
H(21C)	8040	9619	7314	63
H(23)	8255	9781	9088	66
H(024)	6066	7591	3692	32
H(25)	12277	9547	8897	33
H(26)	8356	6875	4490	35
H(28)	7452	6244	3420	53
H(29)	10186	7250	3947	41
H(30)	6777	8087	2452	54
H(31)	8419	8273	1359	76
H(32)	10323	6240	7656	67

H(33)	8182	5911	8283	67
H(34)	5650	6167	7693	50
H(35)	5359	6783	6497	52
H(37)	12140	10444	8481	69
H(38)	10932	11274	8464	88
H(39)	8366	11361	8743	106
H(40)	7058	10602	9076	84
H(41)	9650	9116	9541	37
H(42)	4097	9365	4299	49
H(43A)	11459	10211	4018	98
H(43B)	12774	10551	4612	98
H(43C)	11004	10739	4513	98
H(44)	4320	6934	4774	51
H(45)	2357	6327	4073	69
H(46)	2997	5735	2982	68
H(47)	5593	5662	2707	58
H(48)	10971	7878	1512	64
H(49)	11854	7399	2841	48
H(50A)	5108	10150	3170	98
H(50B)	6732	10357	3681	98
H(50C)	5254	10319	4216	98
H(56)	13652	8698	9725	57
H(57)	10310	9288	11114	64
H(58)	12055	9234	12482	72
H(60)	15293	8553	11063	68
H(61)	14511	8796	12464	100
H(53A)	6641	7129	1272	140
H(53B)	5489	7398	475	140
H(55A)	3839	4630	8292	175
H(55B)	3218	4557	9256	175
H(1S)	4061	8056	7582	74
H(2S)	4406	7904	6803	74

Table 6. Torsion angles [°] for cb2t.

C(51)-Ag(1)-Ag(2)-C(52)	164.3(6)
O(8)-Ag(1)-Ag(2)-C(52)	-19.9(5)
C(51)-Ag(1)-Ag(2)-O(9)	-13.9(5)
O(8)-Ag(1)-Ag(2)-O(9)	161.9(3)
O(4)-S(2)-O(8)-Ag(1)	-175.2(5)
O(5)-S(2)-O(8)-Ag(1)	-42.9(9)
C(014)-S(2)-O(8)-Ag(1)	70.1(7)
C(51)-Ag(1)-O(8)-S(2)	-49(5)
Ag(2)-Ag(1)-O(8)-S(2)	-96.3(6)
O(7)-S(1)-O(9)-Ag(2)	173.2(5)
O(1)-S(1)-O(9)-Ag(2)	-56.1(7)
C(1)-S(1)-O(9)-Ag(2)	58.2(7)
C(52)-Ag(2)-O(9)-S(1)	-142(12)
Ag(1)-Ag(2)-O(9)-S(1)	-94.1(6)
O(7)-S(1)-C(1)-C(2)	-70.6(12)
O(9)-S(1)-C(1)-C(2)	48.1(12)
O(1)-S(1)-C(1)-C(2)	167.0(11)
O(7)-S(1)-C(1)-C(6)	114.0(10)
O(9)-S(1)-C(1)-C(6)	-127.3(10)
O(1)-S(1)-C(1)-C(6)	-8.3(11)
C(6)-C(1)-C(2)-C(3)	-1.4(19)
S(1)-C(1)-C(2)-C(3)	-176.6(10)
C(6)-C(1)-C(2)-N(5)	174.2(11)
S(1)-C(1)-C(2)-N(5)	-1.0(18)
C(51)-N(5)-C(2)-C(3)	82.7(17)
C(41)-N(5)-C(2)-C(3)	-108.5(15)
C(51)-N(5)-C(2)-C(1)	-92.7(16)
C(41)-N(5)-C(2)-C(1)	76.1(17)
C(1)-C(2)-C(3)-C(4)	1(2)
N(5)-C(2)-C(3)-C(4)	-174.7(13)
C(2)-C(3)-C(4)-C(5)	-1(3)
C(3)-C(4)-C(5)-C(6)	2(3)
C(4)-C(5)-C(6)-C(1)	-2(2)
C(2)-C(1)-C(6)-C(5)	2(2)

S(1)-C(1)-C(6)-C(5)	177.7(12)
C(52)-N(4)-C(007)-C(35)	91.4(18)
C(26)-N(4)-C(007)-C(35)	-102.9(17)
C(52)-N(4)-C(007)-C(014)	-89.2(18)
C(26)-N(4)-C(007)-C(014)	76.4(18)
C(52)-N(3)-C(10)-C(14)	-70(2)
C(024)-N(3)-C(10)-C(14)	84(2)
C(52)-N(3)-C(10)-C(11)	116.7(17)
C(024)-N(3)-C(10)-C(11)	-88.9(16)
C(14)-C(10)-C(11)-C(13)	3(2)
N(3)-C(10)-C(11)-C(13)	176.3(13)
C(14)-C(10)-C(11)-C(12)	175.5(13)
N(3)-C(10)-C(11)-C(12)	-11(2)
C(10)-C(11)-C(13)-C(18)	-3(2)
C(12)-C(11)-C(13)-C(18)	-176.4(14)
C(35)-C(007)-C(014)-C(32)	0(2)
N(4)-C(007)-C(014)-C(32)	-179.4(14)
C(35)-C(007)-C(014)-S(2)	178.2(11)
N(4)-C(007)-C(014)-S(2)	-1(2)
O(4)-S(2)-C(014)-C(32)	99.6(12)
O(8)-S(2)-C(014)-C(32)	-142.1(12)
O(5)-S(2)-C(014)-C(32)	-22.4(14)
O(4)-S(2)-C(014)-C(007)	-78.6(12)
O(8)-S(2)-C(014)-C(007)	39.7(13)
O(5)-S(2)-C(014)-C(007)	159.4(12)
N(3)-C(10)-C(14)-C(42)	-176.6(13)
C(11)-C(10)-C(14)-C(42)	-3(2)
N(3)-C(10)-C(14)-C(15)	6(2)
C(11)-C(10)-C(14)-C(15)	179.9(12)
C(032)-C(8)-C(17)-C(16)	-2(2)
C(032)-C(8)-C(17)-C(43)	176.5(14)
C(19)-C(16)-C(17)-C(8)	-1(2)
C(19)-C(16)-C(17)-C(43)	-179.3(14)
C(11)-C(13)-C(18)-C(42)	4(2)
C(11)-C(13)-C(18)-C(50)	-178.6(15)
C(17)-C(16)-C(19)-C(021)	1(2)

C(17)-C(16)-C(19)-C(20)	-177.3(13)
C(16)-C(19)-C(021)-C(032)	1(3)
C(20)-C(19)-C(021)-C(032)	179.9(15)
C(16)-C(19)-C(021)-N(6)	-174.2(13)
C(20)-C(19)-C(021)-N(6)	4(2)
C(51)-N(6)-C(021)-C(19)	-90.9(17)
C(25)-N(6)-C(021)-C(19)	80.8(18)
C(51)-N(6)-C(021)-C(032)	93.3(17)
C(25)-N(6)-C(021)-C(032)	-95.0(16)
C(52)-N(3)-C(024)-C(26)	-11.5(17)
C(10)-N(3)-C(024)-C(26)	-167.9(13)
C(52)-N(3)-C(024)-C(9)	-137.1(13)
C(10)-N(3)-C(024)-C(9)	66.4(18)
C(30)-C(9)-C(024)-N(3)	-92.7(15)
C(29)-C(9)-C(024)-N(3)	84.5(17)
C(30)-C(9)-C(024)-C(26)	148.6(13)
C(29)-C(9)-C(024)-C(26)	-34.3(19)
C(40)-C(23)-C(24)-C(37)	2(2)
C(40)-C(23)-C(24)-C(25)	-179.4(13)
C(37)-C(24)-C(25)-N(6)	-92.9(15)
C(23)-C(24)-C(25)-N(6)	88.5(16)
C(37)-C(24)-C(25)-C(41)	147.3(14)
C(23)-C(24)-C(25)-C(41)	-31.3(19)
C(51)-N(6)-C(25)-C(24)	-139.7(12)
C(021)-N(6)-C(25)-C(24)	47.6(17)
C(51)-N(6)-C(25)-C(41)	-9.7(14)
C(021)-N(6)-C(25)-C(41)	177.6(12)
C(52)-N(4)-C(26)-C(27)	-127.1(13)
C(007)-N(4)-C(26)-C(27)	66.1(17)
C(52)-N(4)-C(26)-C(024)	-5.8(14)
C(007)-N(4)-C(26)-C(024)	-172.6(12)
N(3)-C(024)-C(26)-N(4)	9.4(13)
C(9)-C(024)-C(26)-N(4)	134.7(12)
N(3)-C(024)-C(26)-C(27)	132.9(13)
C(9)-C(024)-C(26)-C(27)	-101.8(14)
N(4)-C(26)-C(27)-C(28)	-148.5(14)

C(024)-C(26)-C(27)-C(28)	96.8(17)
N(4)-C(26)-C(27)-C(44)	39(2)
C(024)-C(26)-C(27)-C(44)	-75.9(19)
C(44)-C(27)-C(28)-C(47)	-1(3)
C(26)-C(27)-C(28)-C(47)	-173.9(14)
C(30)-C(9)-C(29)-C(49)	-2(2)
C(024)-C(9)-C(29)-C(49)	-179.0(13)
C(29)-C(9)-C(30)-C(31)	1(2)
C(024)-C(9)-C(30)-C(31)	177.9(15)
C(9)-C(30)-C(31)-C(48)	2(3)
C(17)-C(8)-C(032)-C(021)	4(2)
C(17)-C(8)-C(032)-C(21)	-174.1(13)
C(19)-C(021)-C(032)-C(8)	-4(2)
N(6)-C(021)-C(032)-C(8)	171.4(13)
C(19)-C(021)-C(032)-C(21)	174.0(15)
N(6)-C(021)-C(032)-C(21)	-10(2)
C(007)-C(014)-C(32)-C(33)	2(2)
S(2)-C(014)-C(32)-C(33)	-176.5(12)
C(014)-C(32)-C(33)-C(34)	-1(2)
C(32)-C(33)-C(34)-C(35)	-1(2)
N(4)-C(007)-C(35)-C(34)	176.9(13)
C(014)-C(007)-C(35)-C(34)	-2(2)
C(33)-C(34)-C(35)-C(007)	3(2)
C(23)-C(24)-C(37)-C(38)	-2(2)
C(25)-C(24)-C(37)-C(38)	179.3(13)
C(24)-C(37)-C(38)-C(39)	1(3)
C(37)-C(38)-C(39)-C(40)	1(3)
C(38)-C(39)-C(40)-C(23)	-1(3)
C(24)-C(23)-C(40)-C(39)	0(3)
C(51)-N(5)-C(41)-C(36)	-138.6(13)
C(2)-N(5)-C(41)-C(36)	51.8(18)
C(51)-N(5)-C(41)-C(25)	-10.6(15)
C(2)-N(5)-C(41)-C(25)	179.7(11)
C(57)-C(36)-C(41)-N(5)	-133.5(14)
C(56)-C(36)-C(41)-N(5)	50.4(19)
C(57)-C(36)-C(41)-C(25)	105.4(17)

C(56)-C(36)-C(41)-C(25)	-70.8(18)
C(24)-C(25)-C(41)-N(5)	137.3(11)
N(6)-C(25)-C(41)-N(5)	11.3(13)
C(24)-C(25)-C(41)-C(36)	-96.0(16)
N(6)-C(25)-C(41)-C(36)	138.0(12)
C(13)-C(18)-C(42)-C(14)	-5(2)
C(50)-C(18)-C(42)-C(14)	177.8(14)
C(10)-C(14)-C(42)-C(18)	5(2)
C(15)-C(14)-C(42)-C(18)	-178.4(14)
C(28)-C(27)-C(44)-C(45)	2(2)
C(26)-C(27)-C(44)-C(45)	174.2(14)
C(27)-C(44)-C(45)-C(46)	-3(2)
C(44)-C(45)-C(46)-C(47)	4(2)
C(45)-C(46)-C(47)-C(28)	-3(2)
C(27)-C(28)-C(47)-C(46)	2(2)
C(30)-C(31)-C(48)-C(49)	-4(3)
C(31)-C(48)-C(49)-C(29)	3(2)
C(9)-C(29)-C(49)-C(48)	0(2)
C(021)-N(6)-C(51)-N(5)	176.0(11)
C(25)-N(6)-C(51)-N(5)	3.6(15)
C(021)-N(6)-C(51)-Ag(1)	7.0(18)
C(25)-N(6)-C(51)-Ag(1)	-165.4(8)
C(2)-N(5)-C(51)-N(6)	174.8(10)
C(41)-N(5)-C(51)-N(6)	4.8(15)
C(2)-N(5)-C(51)-Ag(1)	-15.9(17)
C(41)-N(5)-C(51)-Ag(1)	174.1(9)
O(8)-Ag(1)-C(51)-N(6)	-152(4)
Ag(2)-Ag(1)-C(51)-N(6)	-105.0(11)
O(8)-Ag(1)-C(51)-N(5)	40(5)
Ag(2)-Ag(1)-C(51)-N(5)	87.6(11)
C(10)-N(3)-C(52)-N(4)	164.0(13)
C(024)-N(3)-C(52)-N(4)	8.2(17)
C(10)-N(3)-C(52)-Ag(2)	-17(2)
C(024)-N(3)-C(52)-Ag(2)	-172.3(9)
C(007)-N(4)-C(52)-N(3)	165.8(13)
C(26)-N(4)-C(52)-N(3)	-1.0(16)

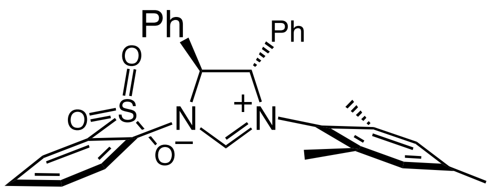
C(007)-N(4)-C(52)-Ag(2)	-13.7(18)
C(26)-N(4)-C(52)-Ag(2)	179.5(9)
O(9)-Ag(2)-C(52)-N(3)	-40(12)
Ag(1)-Ag(2)-C(52)-N(3)	-87.7(13)
O(9)-Ag(2)-C(52)-N(4)	140(11)
Ag(1)-Ag(2)-C(52)-N(4)	91.7(11)
C(57)-C(36)-C(56)-C(60)	5(2)
C(41)-C(36)-C(56)-C(60)	-178.2(14)
C(56)-C(36)-C(57)-C(58)	-2(2)
C(41)-C(36)-C(57)-C(58)	-177.9(14)
C(36)-C(57)-C(58)-C(61)	-4(2)
C(36)-C(56)-C(60)-C(61)	-4(3)
C(57)-C(58)-C(61)-C(60)	5(3)
C(56)-C(60)-C(61)-C(58)	-2(3)

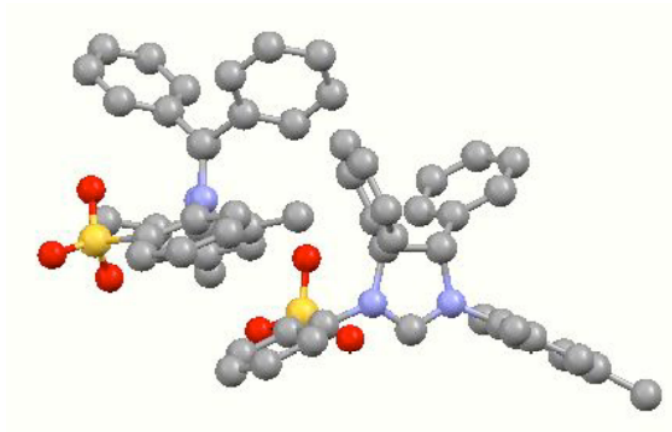
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cb2t [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

Imidazolium Salt **E** (supporting information)





(Solvent molecules omitted for clarity)

Table 1. Crystal data and structure refinement for cb3t.

Identification code	cb3t	
Empirical formula	C30.75 H29.50 Cl1.50 N2 O3 S	
Formula weight	560.30	
Temperature	193(2) K	
Wavelength	0.71073 ≈	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 14.907(3) ≈	α= 90∞.
	b = 17.454(3) ≈	β= 90∞.
	c = 25.055(4) ≈	γ = 90∞.
Volume	6519(2) $≈^3$	
Z	8	
Density (calculated)	1.142 Mg/m^3	
Absorption coefficient	0.253 mm^{-1}	
F(000)	2348	
Crystal size	0.20 x 0.16 x 0.02 mm^3	
Theta range for data collection	1.42 to 25.00∞.	
Index ranges	-17<=h<=17, -20<=k<=20, -29<=l<=27	
Reflections collected	49017	
Independent reflections	11480 [R(int) = 0.0942]	

Completeness to theta = 25.00∞	100.0 %
Absorption correction	None
Max. and min. transmission	0.9950 and 0.9512
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11480 / 0 / 736
Goodness-of-fit on F^2	1.080
Final R indices [I>2sigma(I)]	R1 = 0.0859, wR2 = 0.2232
R indices (all data)	R1 = 0.1723, wR2 = 0.2822
Absolute structure parameter	0.01(14)
Largest diff. peak and hole	0.934 and -0.387 e.≈$^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3)
for cb3t. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1A)	1355(1)	4129(1)	11246(1)	48(1)
O(1A)	2070(3)	4677(3)	11317(2)	61(2)
O(2A)	885(4)	3933(4)	11727(2)	71(2)
O(3A)	1617(4)	3468(3)	10935(2)	60(2)
N(1A)	1561(3)	4609(3)	10060(2)	35(1)
N(2A)	3036(3)	4506(3)	10012(2)	37(1)
C(1A)	2354(4)	4877(4)	10215(3)	35(2)
C(2A)	2698(4)	3907(4)	9620(3)	38(2)
C(3A)	1668(4)	3927(4)	9715(3)	35(2)
C(4A)	720(4)	4811(4)	10307(3)	35(2)
C(5A)	564(4)	4620(4)	10838(3)	39(2)
C(6A)	-274(4)	4811(4)	11057(3)	48(2)
C(7A)	-927(5)	5157(4)	10744(3)	50(2)
C(8A)	-757(4)	5339(4)	10216(3)	45(2)
C(9A)	80(4)	5163(4)	9992(3)	37(2)

C(10A)	3964(4)	4715(4)	10042(3)	40(2)
C(11A)	4487(5)	4402(4)	10456(3)	44(2)
C(12A)	5381(5)	4598(5)	10482(3)	47(2)
C(13A)	5771(5)	5097(5)	10115(3)	51(2)
C(14A)	5212(5)	5403(4)	9725(3)	47(2)
C(15A)	4326(4)	5232(4)	9679(3)	39(2)
C(16A)	4098(5)	3859(5)	10859(4)	58(2)
C(17A)	6749(5)	5293(5)	10138(4)	68(3)
C(18A)	3761(5)	5568(4)	9239(3)	50(2)
C(19A)	3146(5)	3149(4)	9689(3)	45(2)
C(20A)	2877(5)	2599(4)	10050(4)	53(2)
C(21A)	3334(6)	1923(5)	10081(4)	76(3)
C(22A)	4057(7)	1780(5)	9777(5)	83(3)
C(23A)	4348(6)	2291(6)	9428(5)	86(3)
C(24A)	3897(6)	3006(5)	9367(4)	76(3)
C(25A)	1135(4)	3967(4)	9204(3)	41(2)
C(26A)	506(6)	3409(5)	9112(3)	65(2)
C(27A)	-12(7)	3440(6)	8639(4)	80(3)
C(28A)	171(7)	3997(6)	8255(4)	71(3)
C(29A)	793(6)	4540(5)	8361(4)	58(2)
C(30A)	1269(5)	4538(4)	8836(3)	46(2)
S(1B)	1730(1)	6873(1)	9957(1)	39(1)
O(1B)	1933(3)	7603(3)	9703(2)	53(1)
O(2B)	1604(3)	6254(3)	9571(2)	45(1)
O(3B)	2326(3)	6662(3)	10385(2)	48(1)
N(1B)	-3(4)	7465(3)	9408(2)	38(1)
N(2B)	439(4)	8157(3)	8721(2)	42(1)
C(1B)	309(4)	8120(4)	9240(3)	40(2)
C(2B)	185(5)	7412(4)	8471(3)	47(2)
C(3B)	-86(4)	6904(4)	8959(3)	40(2)
C(4B)	-79(4)	7290(4)	9967(3)	39(2)
C(5B)	657(4)	7018(4)	10256(3)	37(2)
C(6B)	558(5)	6876(4)	10798(3)	44(2)
C(7B)	-274(5)	7016(4)	11046(3)	51(2)
C(8B)	-993(5)	7281(4)	10759(3)	51(2)
C(9B)	-889(5)	7413(4)	10218(3)	51(2)

C(10B)	578(5)	8860(4)	8427(3)	48(2)
C(11B)	1464(5)	9064(4)	8302(3)	47(2)
C(12B)	1601(7)	9749(5)	8033(4)	70(3)
C(13B)	877(9)	10235(5)	7904(4)	76(3)
C(14B)	20(7)	10015(5)	8044(4)	69(3)
C(15B)	-157(6)	9327(5)	8307(3)	59(2)
C(16B)	2246(5)	8568(5)	8435(4)	64(2)
C(17B)	1013(10)	11004(7)	7640(5)	117(5)
C(18B)	-1089(6)	9133(6)	8465(4)	79(3)
C(19B)	890(5)	7098(4)	8116(3)	45(2)
C(20B)	847(6)	7228(5)	7561(3)	58(2)
C(21B)	1484(7)	6971(7)	7230(4)	82(3)
C(22B)	2195(7)	6537(6)	7450(5)	88(3)
C(23B)	2273(7)	6414(5)	7982(4)	71(3)
C(24B)	1617(5)	6703(5)	8319(4)	55(2)
C(25B)	-983(4)	6530(4)	8915(3)	39(2)
C(26B)	-1030(5)	5763(5)	8817(3)	53(2)
C(27B)	-1841(6)	5393(5)	8743(4)	68(2)
C(28B)	-2607(5)	5818(6)	8762(4)	68(3)
C(29B)	-2599(5)	6588(5)	8868(4)	61(2)
C(30B)	-1793(5)	6948(5)	8938(3)	58(2)
C(1S)	1402(13)	1838(9)	1550(6)	59(4)
Cl(1S)	2168(5)	1387(3)	1970(2)	99(2)
Cl(2S)	1168(3)	1244(3)	1001(2)	73(1)
C(2S)	1410(30)	5631(13)	2421(12)	158(16)
Cl(3S)	1919(7)	5715(4)	2920(3)	157(4)
Cl(4S)	480(20)	6110(14)	2325(7)	620(30)
C(3S)	810(30)	8650(30)	2485(18)	300(40)
Cl(5S)	95(5)	8569(6)	1901(3)	140(3)
Cl(6S)	1135(10)	9452(18)	2509(7)	500(20)

Table 3. Bond lengths [≈] and angles [∞] for cb3t.

S(1A)-O(2A)	1.436(6)
S(1A)-O(1A)	1.443(6)
S(1A)-O(3A)	1.446(6)
S(1A)-C(5A)	1.780(7)
N(1A)-C(1A)	1.329(8)
N(1A)-C(4A)	1.442(8)
N(1A)-C(3A)	1.480(8)
N(2A)-C(1A)	1.308(8)
N(2A)-C(10A)	1.434(8)
N(2A)-C(2A)	1.520(8)
C(1A)-H(1A)	0.9500
C(2A)-C(19A)	1.492(10)
C(2A)-C(3A)	1.554(9)
C(2A)-H(2A)	1.0000
C(3A)-C(25A)	1.509(10)
C(3A)-H(3A)	1.0000
C(4A)-C(9A)	1.382(9)
C(4A)-C(5A)	1.392(10)
C(5A)-C(6A)	1.404(10)
C(6A)-C(7A)	1.388(10)
C(6A)-H(6A)	0.9500
C(7A)-C(8A)	1.383(11)
C(7A)-H(7A)	0.9500
C(8A)-C(9A)	1.403(9)
C(8A)-H(8A)	0.9500
C(9A)-H(9A)	0.9500
C(10A)-C(15A)	1.389(10)
C(10A)-C(11A)	1.408(10)
C(11A)-C(12A)	1.378(10)
C(11A)-C(16A)	1.500(11)
C(12A)-C(13A)	1.393(11)
C(12A)-H(12A)	0.9500
C(13A)-C(14A)	1.391(11)
C(13A)-C(17A)	1.498(10)

C(14A)-C(15A)	1.360(10)
C(14A)-H(14A)	0.9500
C(15A)-C(18A)	1.506(10)
C(16A)-H(16A)	0.9800
C(16A)-H(16B)	0.9800
C(16A)-H(16C)	0.9800
C(17A)-H(17A)	0.9800
C(17A)-H(17B)	0.9800
C(17A)-H(17C)	0.9800
C(18A)-H(18A)	0.9800
C(18A)-H(18B)	0.9800
C(18A)-H(18C)	0.9800
C(19A)-C(20A)	1.379(11)
C(19A)-C(24A)	1.403(11)
C(20A)-C(21A)	1.364(11)
C(20A)-H(20A)	0.9500
C(21A)-C(22A)	1.343(14)
C(21A)-H(21A)	0.9500
C(22A)-C(23A)	1.323(14)
C(22A)-H(22A)	0.9500
C(23A)-C(24A)	1.425(12)
C(23A)-H(23A)	0.9500
C(24A)-H(24A)	0.9500
C(25A)-C(26A)	1.370(11)
C(25A)-C(30A)	1.373(11)
C(26A)-C(27A)	1.415(12)
C(26A)-H(26A)	0.9500
C(27A)-C(28A)	1.394(13)
C(27A)-H(27A)	0.9500
C(28A)-C(29A)	1.351(13)
C(28A)-H(28A)	0.9500
C(29A)-C(30A)	1.386(11)
C(29A)-H(29A)	0.9500
C(30A)-H(30A)	0.9500
S(1B)-O(3B)	1.441(5)
S(1B)-O(1B)	1.456(5)

S(1B)-O(2B)	1.462(5)
S(1B)-C(5B)	1.785(7)
N(1B)-C(1B)	1.303(9)
N(1B)-C(4B)	1.438(10)
N(1B)-C(3B)	1.498(9)
N(2B)-C(1B)	1.318(9)
N(2B)-C(10B)	1.446(10)
N(2B)-C(2B)	1.492(10)
C(1B)-H(1B)	0.9500
C(2B)-C(19B)	1.481(10)
C(2B)-C(3B)	1.564(10)
C(2B)-H(2B)	1.0000
C(3B)-C(25B)	1.493(9)
C(3B)-H(3B)	1.0000
C(4B)-C(9B)	1.377(10)
C(4B)-C(5B)	1.397(10)
C(5B)-C(6B)	1.388(10)
C(6B)-C(7B)	1.409(11)
C(6B)-H(6B)	0.9500
C(7B)-C(8B)	1.373(11)
C(7B)-H(7B)	0.9500
C(8B)-C(9B)	1.383(11)
C(8B)-H(8B)	0.9500
C(9B)-H(9B)	0.9500
C(10B)-C(15B)	1.399(11)
C(10B)-C(11B)	1.403(11)
C(11B)-C(12B)	1.387(11)
C(11B)-C(16B)	1.491(11)
C(12B)-C(13B)	1.409(14)
C(12B)-H(12B)	0.9500
C(13B)-C(14B)	1.379(14)
C(13B)-C(17B)	1.511(14)
C(14B)-C(15B)	1.395(12)
C(14B)-H(14B)	0.9500
C(15B)-C(18B)	1.484(13)
C(16B)-H(16D)	0.9800

C(16B)-H(16E)	0.9800
C(16B)-H(16F)	0.9800
C(17B)-H(17D)	0.9800
C(17B)-H(17E)	0.9800
C(17B)-H(17F)	0.9800
C(18B)-H(18D)	0.9800
C(18B)-H(18E)	0.9800
C(18B)-H(18F)	0.9800
C(19B)-C(24B)	1.382(11)
C(19B)-C(20B)	1.409(11)
C(20B)-C(21B)	1.339(13)
C(20B)-H(20B)	0.9500
C(21B)-C(22B)	1.416(15)
C(21B)-H(21B)	0.9500
C(22B)-C(23B)	1.355(14)
C(22B)-H(22B)	0.9500
C(23B)-C(24B)	1.387(11)
C(23B)-H(23B)	0.9500
C(24B)-H(24B)	0.9500
C(25B)-C(26B)	1.363(10)
C(25B)-C(30B)	1.411(10)
C(26B)-C(27B)	1.384(11)
C(26B)-H(26B)	0.9500
C(27B)-C(28B)	1.362(13)
C(27B)-H(27B)	0.9500
C(28B)-C(29B)	1.371(12)
C(28B)-H(28B)	0.9500
C(29B)-C(30B)	1.368(11)
C(29B)-H(29B)	0.9500
C(30B)-H(30B)	0.9500
C(1S)-Cl(1S)	1.742(16)
C(1S)-Cl(2S)	1.758(16)
C(1S)-H(1S1)	0.9900
C(1S)-H(1S2)	0.9900
C(2S)-Cl(3S)	1.47(4)
C(2S)-Cl(4S)	1.63(4)

C(2S)-H(2S1)	0.9900
C(2S)-H(2S2)	0.9900
C(3S)-Cl(6S)	1.49(4)
C(3S)-Cl(5S)	1.81(5)
C(3S)-H(3S1)	0.9900
C(3S)-H(3S2)	0.9900

O(2A)-S(1A)-O(1A)	114.4(4)
O(2A)-S(1A)-O(3A)	113.2(4)
O(1A)-S(1A)-O(3A)	113.4(4)
O(2A)-S(1A)-C(5A)	105.8(3)
O(1A)-S(1A)-C(5A)	104.0(3)
O(3A)-S(1A)-C(5A)	104.7(3)
C(1A)-N(1A)-C(4A)	124.3(6)
C(1A)-N(1A)-C(3A)	110.9(5)
C(4A)-N(1A)-C(3A)	122.8(5)
C(1A)-N(2A)-C(10A)	127.1(6)
C(1A)-N(2A)-C(2A)	109.6(5)
C(10A)-N(2A)-C(2A)	121.9(5)
N(2A)-C(1A)-N(1A)	113.8(6)
N(2A)-C(1A)-H(1A)	123.1
N(1A)-C(1A)-H(1A)	123.1
C(19A)-C(2A)-N(2A)	112.7(6)
C(19A)-C(2A)-C(3A)	116.4(6)
N(2A)-C(2A)-C(3A)	102.2(5)
C(19A)-C(2A)-H(2A)	108.4
N(2A)-C(2A)-H(2A)	108.4
C(3A)-C(2A)-H(2A)	108.4
N(1A)-C(3A)-C(25A)	113.7(5)
N(1A)-C(3A)-C(2A)	102.4(5)
C(25A)-C(3A)-C(2A)	113.0(6)
N(1A)-C(3A)-H(3A)	109.2
C(25A)-C(3A)-H(3A)	109.2
C(2A)-C(3A)-H(3A)	109.2
C(9A)-C(4A)-C(5A)	122.5(6)
C(9A)-C(4A)-N(1A)	117.7(6)

C(5A)-C(4A)-N(1A)	119.7(6)
C(4A)-C(5A)-C(6A)	117.7(6)
C(4A)-C(5A)-S(1A)	123.6(5)
C(6A)-C(5A)-S(1A)	118.7(6)
C(7A)-C(6A)-C(5A)	120.5(7)
C(7A)-C(6A)-H(6A)	119.8
C(5A)-C(6A)-H(6A)	119.8
C(8A)-C(7A)-C(6A)	120.8(7)
C(8A)-C(7A)-H(7A)	119.6
C(6A)-C(7A)-H(7A)	119.6
C(7A)-C(8A)-C(9A)	119.7(7)
C(7A)-C(8A)-H(8A)	120.1
C(9A)-C(8A)-H(8A)	120.1
C(4A)-C(9A)-C(8A)	118.8(7)
C(4A)-C(9A)-H(9A)	120.6
C(8A)-C(9A)-H(9A)	120.6
C(15A)-C(10A)-C(11A)	121.3(6)
C(15A)-C(10A)-N(2A)	120.4(6)
C(11A)-C(10A)-N(2A)	118.3(7)
C(12A)-C(11A)-C(10A)	118.2(7)
C(12A)-C(11A)-C(16A)	120.0(7)
C(10A)-C(11A)-C(16A)	121.8(6)
C(11A)-C(12A)-C(13A)	121.9(7)
C(11A)-C(12A)-H(12A)	119.0
C(13A)-C(12A)-H(12A)	119.0
C(14A)-C(13A)-C(12A)	116.9(6)
C(14A)-C(13A)-C(17A)	121.5(8)
C(12A)-C(13A)-C(17A)	121.6(8)
C(15A)-C(14A)-C(13A)	123.9(7)
C(15A)-C(14A)-H(14A)	118.1
C(13A)-C(14A)-H(14A)	118.1
C(14A)-C(15A)-C(10A)	117.7(7)
C(14A)-C(15A)-C(18A)	121.3(7)
C(10A)-C(15A)-C(18A)	121.0(6)
C(11A)-C(16A)-H(16A)	109.5
C(11A)-C(16A)-H(16B)	109.5

H(16A)-C(16A)-H(16B)	109.5
C(11A)-C(16A)-H(16C)	109.5
H(16A)-C(16A)-H(16C)	109.5
H(16B)-C(16A)-H(16C)	109.5
C(13A)-C(17A)-H(17A)	109.5
C(13A)-C(17A)-H(17B)	109.5
H(17A)-C(17A)-H(17B)	109.5
C(13A)-C(17A)-H(17C)	109.5
H(17A)-C(17A)-H(17C)	109.5
H(17B)-C(17A)-H(17C)	109.5
C(15A)-C(18A)-H(18A)	109.5
C(15A)-C(18A)-H(18B)	109.5
H(18A)-C(18A)-H(18B)	109.5
C(15A)-C(18A)-H(18C)	109.5
H(18A)-C(18A)-H(18C)	109.5
H(18B)-C(18A)-H(18C)	109.5
C(20A)-C(19A)-C(24A)	119.1(7)
C(20A)-C(19A)-C(2A)	124.3(6)
C(24A)-C(19A)-C(2A)	116.6(7)
C(21A)-C(20A)-C(19A)	119.6(8)
C(21A)-C(20A)-H(20A)	120.2
C(19A)-C(20A)-H(20A)	120.2
C(22A)-C(21A)-C(20A)	121.9(10)
C(22A)-C(21A)-H(21A)	119.0
C(20A)-C(21A)-H(21A)	119.0
C(23A)-C(22A)-C(21A)	120.9(9)
C(23A)-C(22A)-H(22A)	119.6
C(21A)-C(22A)-H(22A)	119.6
C(22A)-C(23A)-C(24A)	120.4(9)
C(22A)-C(23A)-H(23A)	119.8
C(24A)-C(23A)-H(23A)	119.8
C(19A)-C(24A)-C(23A)	118.0(9)
C(19A)-C(24A)-H(24A)	121.0
C(23A)-C(24A)-H(24A)	121.0
C(26A)-C(25A)-C(30A)	120.1(7)
C(26A)-C(25A)-C(3A)	118.1(7)

C(30A)-C(25A)-C(3A)	121.8(6)
C(25A)-C(26A)-C(27A)	119.2(8)
C(25A)-C(26A)-H(26A)	120.4
C(27A)-C(26A)-H(26A)	120.4
C(28A)-C(27A)-C(26A)	119.8(9)
C(28A)-C(27A)-H(27A)	120.1
C(26A)-C(27A)-H(27A)	120.1
C(29A)-C(28A)-C(27A)	119.2(9)
C(29A)-C(28A)-H(28A)	120.4
C(27A)-C(28A)-H(28A)	120.4
C(28A)-C(29A)-C(30A)	121.2(9)
C(28A)-C(29A)-H(29A)	119.4
C(30A)-C(29A)-H(29A)	119.4
C(25A)-C(30A)-C(29A)	120.2(7)
C(25A)-C(30A)-H(30A)	119.9
C(29A)-C(30A)-H(30A)	119.9
O(3B)-S(1B)-O(1B)	114.9(3)
O(3B)-S(1B)-O(2B)	112.5(3)
O(1B)-S(1B)-O(2B)	112.7(3)
O(3B)-S(1B)-C(5B)	106.0(3)
O(1B)-S(1B)-C(5B)	104.3(3)
O(2B)-S(1B)-C(5B)	105.4(3)
C(1B)-N(1B)-C(4B)	122.0(6)
C(1B)-N(1B)-C(3B)	111.1(6)
C(4B)-N(1B)-C(3B)	125.9(6)
C(1B)-N(2B)-C(10B)	124.4(6)
C(1B)-N(2B)-C(2B)	109.6(6)
C(10B)-N(2B)-C(2B)	124.2(6)
N(1B)-C(1B)-N(2B)	114.5(7)
N(1B)-C(1B)-H(1B)	122.7
N(2B)-C(1B)-H(1B)	122.7
C(19B)-C(2B)-N(2B)	113.2(6)
C(19B)-C(2B)-C(3B)	116.3(6)
N(2B)-C(2B)-C(3B)	103.3(6)
C(19B)-C(2B)-H(2B)	107.9
N(2B)-C(2B)-H(2B)	107.9

C(3B)-C(2B)-H(2B)	107.9
C(25B)-C(3B)-N(1B)	114.5(6)
C(25B)-C(3B)-C(2B)	114.9(6)
N(1B)-C(3B)-C(2B)	101.3(5)
C(25B)-C(3B)-H(3B)	108.6
N(1B)-C(3B)-H(3B)	108.6
C(2B)-C(3B)-H(3B)	108.6
C(9B)-C(4B)-C(5B)	120.4(7)
C(9B)-C(4B)-N(1B)	118.7(6)
C(5B)-C(4B)-N(1B)	120.9(6)
C(6B)-C(5B)-C(4B)	118.9(6)
C(6B)-C(5B)-S(1B)	118.8(5)
C(4B)-C(5B)-S(1B)	122.3(5)
C(5B)-C(6B)-C(7B)	119.7(7)
C(5B)-C(6B)-H(6B)	120.2
C(7B)-C(6B)-H(6B)	120.2
C(8B)-C(7B)-C(6B)	121.0(7)
C(8B)-C(7B)-H(7B)	119.5
C(6B)-C(7B)-H(7B)	119.5
C(7B)-C(8B)-C(9B)	118.8(7)
C(7B)-C(8B)-H(8B)	120.6
C(9B)-C(8B)-H(8B)	120.6
C(4B)-C(9B)-C(8B)	121.3(7)
C(4B)-C(9B)-H(9B)	119.4
C(8B)-C(9B)-H(9B)	119.4
C(15B)-C(10B)-C(11B)	122.8(7)
C(15B)-C(10B)-N(2B)	119.5(7)
C(11B)-C(10B)-N(2B)	117.6(6)
C(12B)-C(11B)-C(10B)	117.8(7)
C(12B)-C(11B)-C(16B)	119.6(8)
C(10B)-C(11B)-C(16B)	122.6(7)
C(11B)-C(12B)-C(13B)	121.1(9)
C(11B)-C(12B)-H(12B)	119.4
C(13B)-C(12B)-H(12B)	119.4
C(14B)-C(13B)-C(12B)	118.9(9)
C(14B)-C(13B)-C(17B)	118.8(10)

C(12B)-C(13B)-C(17B)	122.2(11)
C(13B)-C(14B)-C(15B)	122.3(9)
C(13B)-C(14B)-H(14B)	118.8
C(15B)-C(14B)-H(14B)	118.8
C(14B)-C(15B)-C(10B)	117.1(9)
C(14B)-C(15B)-C(18B)	120.0(8)
C(10B)-C(15B)-C(18B)	122.9(8)
C(11B)-C(16B)-H(16D)	109.5
C(11B)-C(16B)-H(16E)	109.5
H(16D)-C(16B)-H(16E)	109.5
C(11B)-C(16B)-H(16F)	109.5
H(16D)-C(16B)-H(16F)	109.5
H(16E)-C(16B)-H(16F)	109.5
C(13B)-C(17B)-H(17D)	109.5
C(13B)-C(17B)-H(17E)	109.5
H(17D)-C(17B)-H(17E)	109.5
C(13B)-C(17B)-H(17F)	109.5
H(17D)-C(17B)-H(17F)	109.5
H(17E)-C(17B)-H(17F)	109.5
C(15B)-C(18B)-H(18D)	109.5
C(15B)-C(18B)-H(18E)	109.5
H(18D)-C(18B)-H(18E)	109.5
C(15B)-C(18B)-H(18F)	109.5
H(18D)-C(18B)-H(18F)	109.5
H(18E)-C(18B)-H(18F)	109.5
C(24B)-C(19B)-C(20B)	118.5(8)
C(24B)-C(19B)-C(2B)	121.4(7)
C(20B)-C(19B)-C(2B)	120.0(7)
C(21B)-C(20B)-C(19B)	121.8(9)
C(21B)-C(20B)-H(20B)	119.1
C(19B)-C(20B)-H(20B)	119.1
C(20B)-C(21B)-C(22B)	117.9(9)
C(20B)-C(21B)-H(21B)	121.1
C(22B)-C(21B)-H(21B)	121.1
C(23B)-C(22B)-C(21B)	122.2(9)
C(23B)-C(22B)-H(22B)	118.9

C(21B)-C(22B)-H(22B)	118.9
C(22B)-C(23B)-C(24B)	118.7(9)
C(22B)-C(23B)-H(23B)	120.7
C(24B)-C(23B)-H(23B)	120.7
C(19B)-C(24B)-C(23B)	120.8(9)
C(19B)-C(24B)-H(24B)	119.6
C(23B)-C(24B)-H(24B)	119.6
C(26B)-C(25B)-C(30B)	118.2(7)
C(26B)-C(25B)-C(3B)	119.2(6)
C(30B)-C(25B)-C(3B)	122.5(6)
C(25B)-C(26B)-C(27B)	121.8(8)
C(25B)-C(26B)-H(26B)	119.1
C(27B)-C(26B)-H(26B)	119.1
C(28B)-C(27B)-C(26B)	118.2(8)
C(28B)-C(27B)-H(27B)	120.9
C(26B)-C(27B)-H(27B)	120.9
C(27B)-C(28B)-C(29B)	122.3(8)
C(27B)-C(28B)-H(28B)	118.8
C(29B)-C(28B)-H(28B)	118.8
C(30B)-C(29B)-C(28B)	118.9(7)
C(30B)-C(29B)-H(29B)	120.6
C(28B)-C(29B)-H(29B)	120.6
C(29B)-C(30B)-C(25B)	120.6(7)
C(29B)-C(30B)-H(30B)	119.7
C(25B)-C(30B)-H(30B)	119.7
Cl(1S)-C(1S)-Cl(2S)	109.7(8)
Cl(1S)-C(1S)-H(1S1)	109.7
Cl(2S)-C(1S)-H(1S1)	109.7
Cl(1S)-C(1S)-H(1S2)	109.7
Cl(2S)-C(1S)-H(1S2)	109.7
H(1S1)-C(1S)-H(1S2)	108.2
Cl(3S)-C(2S)-Cl(4S)	121.0(16)
Cl(3S)-C(2S)-H(2S1)	107.1
Cl(4S)-C(2S)-H(2S1)	107.1
Cl(3S)-C(2S)-H(2S2)	107.1
Cl(4S)-C(2S)-H(2S2)	107.1

H(2S1)-C(2S)-H(2S2)	106.8
Cl(6S)-C(3S)-Cl(5S)	107(4)
Cl(6S)-C(3S)-H(3S1)	110.3
Cl(5S)-C(3S)-H(3S1)	110.3
Cl(6S)-C(3S)-H(3S2)	110.3
Cl(5S)-C(3S)-H(3S2)	110.3
H(3S1)-C(3S)-H(3S2)	108.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for cb3t. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[h^2 $a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1A)	34(1)	66(1)	45(1)	7(1)	-5(1)	2(1)
O(1A)	44(3)	85(4)	55(4)	1(3)	-14(3)	-9(3)
O(2A)	47(3)	122(5)	45(3)	22(3)	0(3)	3(3)
O(3A)	68(4)	53(3)	59(4)	0(3)	-4(3)	21(3)
N(1A)	29(3)	29(3)	46(4)	-1(3)	0(3)	7(2)
N(2A)	25(3)	34(3)	53(4)	-9(3)	-9(3)	1(2)
C(1A)	31(3)	33(4)	40(4)	-7(3)	-8(3)	0(3)
C(2A)	32(3)	36(4)	46(5)	-8(3)	-1(3)	5(3)
C(3A)	30(3)	34(4)	42(4)	-6(3)	-5(3)	2(3)
C(4A)	22(3)	33(4)	51(5)	-6(3)	0(3)	-2(3)
C(5A)	29(3)	47(4)	41(5)	-6(4)	-6(3)	1(3)
C(6A)	33(4)	61(5)	52(5)	-6(4)	-3(4)	2(3)
C(7A)	30(4)	58(5)	62(6)	-10(4)	0(4)	4(4)
C(8A)	27(3)	50(5)	58(5)	-2(4)	-7(3)	4(3)
C(9A)	33(3)	33(4)	45(4)	-5(3)	-5(3)	2(3)
C(10A)	26(3)	40(4)	54(5)	-9(4)	-1(3)	7(3)
C(11A)	34(4)	46(5)	52(5)	1(4)	1(4)	3(3)
C(12A)	31(4)	61(5)	50(5)	-2(4)	1(3)	5(4)
C(13A)	31(4)	55(5)	67(6)	-17(4)	3(4)	0(3)
C(14A)	37(4)	45(4)	58(5)	0(4)	3(4)	2(3)
C(15A)	30(3)	37(4)	51(5)	-1(4)	8(3)	1(3)
C(16A)	39(4)	70(6)	66(6)	6(5)	-13(4)	-4(4)
C(17A)	37(4)	70(6)	97(8)	-10(5)	-4(5)	-9(4)
C(18A)	45(4)	42(5)	63(5)	11(4)	-1(4)	0(3)
C(19A)	39(4)	40(4)	57(5)	-3(4)	-3(4)	15(3)
C(20A)	52(4)	44(4)	64(6)	1(4)	4(4)	6(4)
C(21A)	71(6)	42(5)	115(8)	0(5)	4(6)	25(4)
C(22A)	78(7)	42(5)	129(9)	-5(6)	12(7)	25(5)
C(23A)	55(6)	61(6)	142(10)	-12(7)	23(6)	28(5)
C(24A)	66(6)	68(6)	93(7)	11(6)	24(5)	24(5)

C(25A)	36(4)	37(4)	50(5)	-14(4)	-5(3)	-1(3)
C(26A)	75(6)	76(6)	43(5)	5(4)	-13(4)	-20(5)
C(27A)	93(7)	76(7)	72(7)	0(6)	-25(6)	-25(6)
C(28A)	78(6)	80(7)	55(6)	-9(5)	-19(5)	-2(5)
C(29A)	61(5)	57(5)	58(6)	2(4)	-8(5)	14(5)
C(30A)	45(4)	49(5)	44(5)	2(4)	-4(4)	6(4)
S(1B)	25(1)	40(1)	53(1)	4(1)	-3(1)	-2(1)
O(1B)	31(2)	47(3)	81(4)	20(3)	0(3)	-4(2)
O(2B)	32(2)	49(3)	53(3)	-10(3)	-1(2)	5(2)
O(3B)	35(2)	46(3)	65(4)	2(3)	-14(2)	-2(2)
N(1B)	32(3)	43(4)	39(4)	-4(3)	-3(3)	2(3)
N(2B)	44(3)	41(4)	42(4)	-3(3)	0(3)	1(3)
C(1B)	27(3)	40(4)	52(5)	-8(4)	-4(3)	6(3)
C(2B)	46(4)	49(5)	46(5)	-8(4)	1(4)	7(4)
C(3B)	35(4)	39(4)	46(4)	-10(4)	-8(3)	6(3)
C(4B)	32(3)	41(4)	44(5)	-5(4)	-1(3)	-2(3)
C(5B)	32(3)	37(4)	42(5)	-9(3)	-2(3)	-3(3)
C(6B)	44(4)	44(4)	43(5)	0(4)	-6(4)	-7(3)
C(7B)	60(5)	54(5)	39(5)	0(4)	-2(4)	-16(4)
C(8B)	42(4)	54(5)	58(6)	4(4)	12(4)	-1(4)
C(9B)	32(4)	55(5)	66(6)	-13(4)	6(4)	3(3)
C(10B)	63(5)	33(4)	47(5)	1(4)	-6(4)	8(4)
C(11B)	54(5)	39(4)	47(5)	6(4)	2(4)	4(4)
C(12B)	85(7)	66(6)	57(6)	9(5)	17(5)	16(5)
C(13B)	126(9)	57(6)	44(6)	4(5)	1(6)	26(6)
C(14B)	87(7)	54(6)	65(6)	8(5)	-13(5)	24(5)
C(15B)	69(6)	59(6)	51(5)	-2(4)	-7(4)	19(4)
C(16B)	44(5)	65(6)	83(7)	18(5)	-5(4)	-6(4)
C(17B)	162(12)	87(8)	102(9)	50(7)	38(9)	46(8)
C(18B)	59(6)	70(6)	106(8)	-9(6)	-14(5)	24(5)
C(19B)	49(4)	34(4)	53(5)	-8(4)	4(4)	-2(3)
C(20B)	62(5)	63(5)	49(6)	-9(4)	-4(4)	1(4)
C(21B)	91(7)	105(8)	52(6)	2(6)	20(6)	-10(7)
C(22B)	88(8)	86(8)	89(9)	-12(7)	43(7)	19(6)
C(23B)	72(6)	67(6)	74(7)	8(5)	36(5)	22(5)
C(24B)	48(5)	60(5)	58(5)	1(4)	13(4)	10(4)

C(25B)	30(3)	46(4)	41(4)	-10(3)	-1(3)	-3(3)
C(26B)	50(4)	53(5)	57(5)	-13(4)	-6(4)	4(4)
C(27B)	68(6)	58(5)	78(7)	-5(5)	-12(5)	-18(5)
C(28B)	35(4)	97(8)	72(6)	-2(6)	2(4)	-12(5)
C(29B)	31(4)	64(6)	88(7)	-28(5)	-4(4)	-1(4)
C(30B)	44(4)	56(5)	74(6)	-17(5)	-1(4)	3(4)
C(1S)	93(13)	34(9)	49(10)	3(8)	-6(9)	22(9)
Cl(1S)	142(5)	56(3)	98(4)	-18(3)	-59(4)	15(3)
Cl(2S)	82(3)	53(3)	84(3)	-20(2)	-31(3)	21(2)
C(2S)	290(40)	44(13)	140(30)	5(15)	110(30)	43(19)
Cl(3S)	255(11)	105(5)	109(6)	-4(4)	-102(7)	36(6)
Cl(4S)	1120(70)	520(30)	208(15)	-170(19)	-340(30)	620(50)
C(3S)	280(50)	340(60)	300(50)	-270(50)	240(50)	-250(50)
Cl(5S)	91(4)	238(9)	93(5)	-60(6)	-7(4)	-14(5)
Cl(6S)	234(14)	980(60)	283(17)	-430(30)	196(14)	-360(20)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for cb3t.

	x	y	z	U(eq)
H(1A)	2420	5300	10450	41
H(2A)	2821	4093	9250	45
H(3A)	1486	3460	9919	42
H(6A)	-396	4702	11421	58
H(7A)	-1496	5271	10894	60
H(8A)	-1206	5582	10007	54
H(9A)	204	5282	9629	44
H(12A)	5742	4387	10757	57
H(14A)	5465	5752	9476	56
H(16A)	3871	3402	10676	88
H(16B)	3605	4111	11049	88
H(16C)	4564	3710	11114	88
H(17A)	7078	4979	9879	102
H(17B)	6978	5191	10498	102
H(17C)	6831	5836	10053	102
H(18A)	4112	5950	9043	75
H(18B)	3229	5812	9393	75
H(18C)	3574	5159	8995	75
H(20A)	2376	2689	10276	64
H(21A)	3134	1542	10325	91
H(22A)	4364	1306	9814	100
H(23A)	4861	2181	9216	103
H(24A)	4098	3374	9115	91
H(26A)	420	3007	9362	78
H(27A)	-483	3083	8583	96
H(28A)	-137	3995	7923	85
H(29A)	905	4930	8105	70
H(30A)	1690	4933	8907	55

H(1B)	432	8536	9473	48
H(2B)	-365	7499	8250	57
H(3B)	379	6496	9007	47
H(6B)	1048	6685	11000	52
H(7B)	-338	6925	11418	61
H(8B)	-1553	7373	10928	62
H(9B)	-1385	7592	10015	61
H(12B)	2192	9893	7933	84
H(14B)	-467	10344	7958	82
H(16D)	2759	8889	8533	96
H(16E)	2402	8254	8124	96
H(16F)	2091	8233	8735	96
H(17D)	572	11069	7354	176
H(17E)	1619	11030	7490	176
H(17F)	937	11412	7905	176
H(18D)	-1496	9539	8345	118
H(18E)	-1123	9086	8854	118
H(18F)	-1263	8646	8300	118
H(20B)	353	7504	7419	69
H(21B)	1459	7077	6858	99
H(22B)	2633	6325	7217	105
H(23B)	2768	6135	8121	85
H(24B)	1669	6629	8693	67
H(26B)	-490	5474	8799	64
H(27B)	-1864	4856	8680	82
H(28B)	-3165	5572	8699	82
H(29B)	-3145	6867	8892	73
H(30B)	-1777	7484	9004	69
H(1S1)	1654	2331	1424	70
H(1S2)	841	1947	1748	70
H(2S1)	1257	5080	2384	189
H(2S2)	1815	5761	2124	189
H(3S1)	460	8523	2811	364
H(3S2)	1317	8288	2457	364

Table 6. Torsion angles [∞] for cb3t.

C(10A)-N(2A)-C(1A)-N(1A)	170.9(7)
C(2A)-N(2A)-C(1A)-N(1A)	4.5(8)
C(4A)-N(1A)-C(1A)-N(2A)	166.8(6)
C(3A)-N(1A)-C(1A)-N(2A)	2.7(8)
C(1A)-N(2A)-C(2A)-C(19A)	-134.8(6)
C(10A)-N(2A)-C(2A)-C(19A)	57.9(9)
C(1A)-N(2A)-C(2A)-C(3A)	-9.1(7)
C(10A)-N(2A)-C(2A)-C(3A)	-176.4(6)
C(1A)-N(1A)-C(3A)-C(25A)	-130.5(6)
C(4A)-N(1A)-C(3A)-C(25A)	65.1(8)
C(1A)-N(1A)-C(3A)-C(2A)	-8.2(7)
C(4A)-N(1A)-C(3A)-C(2A)	-172.6(6)
C(19A)-C(2A)-C(3A)-N(1A)	133.1(6)
N(2A)-C(2A)-C(3A)-N(1A)	9.8(6)
C(19A)-C(2A)-C(3A)-C(25A)	-104.2(7)
N(2A)-C(2A)-C(3A)-C(25A)	132.5(6)
C(1A)-N(1A)-C(4A)-C(9A)	119.7(7)
C(3A)-N(1A)-C(4A)-C(9A)	-78.1(8)
C(1A)-N(1A)-C(4A)-C(5A)	-63.1(9)
C(3A)-N(1A)-C(4A)-C(5A)	99.1(8)
C(9A)-C(4A)-C(5A)-C(6A)	-1.3(10)
N(1A)-C(4A)-C(5A)-C(6A)	-178.3(6)
C(9A)-C(4A)-C(5A)-S(1A)	177.1(5)
N(1A)-C(4A)-C(5A)-S(1A)	0.0(9)
O(2A)-S(1A)-C(5A)-C(4A)	-170.6(6)
O(1A)-S(1A)-C(5A)-C(4A)	68.5(7)
O(3A)-S(1A)-C(5A)-C(4A)	-50.7(7)
O(2A)-S(1A)-C(5A)-C(6A)	7.7(7)
O(1A)-S(1A)-C(5A)-C(6A)	-113.2(6)
O(3A)-S(1A)-C(5A)-C(6A)	127.5(6)
C(4A)-C(5A)-C(6A)-C(7A)	1.8(11)
S(1A)-C(5A)-C(6A)-C(7A)	-176.6(6)
C(5A)-C(6A)-C(7A)-C(8A)	-1.6(12)
C(6A)-C(7A)-C(8A)-C(9A)	0.8(11)

C(5A)-C(4A)-C(9A)-C(8A)	0.5(10)
N(1A)-C(4A)-C(9A)-C(8A)	177.6(6)
C(7A)-C(8A)-C(9A)-C(4A)	-0.2(10)
C(1A)-N(2A)-C(10A)-C(15A)	-84.0(10)
C(2A)-N(2A)-C(10A)-C(15A)	80.9(8)
C(1A)-N(2A)-C(10A)-C(11A)	94.2(9)
C(2A)-N(2A)-C(10A)-C(11A)	-100.8(8)
C(15A)-C(10A)-C(11A)-C(12A)	-2.4(10)
N(2A)-C(10A)-C(11A)-C(12A)	179.4(7)
C(15A)-C(10A)-C(11A)-C(16A)	178.4(7)
N(2A)-C(10A)-C(11A)-C(16A)	0.2(10)
C(10A)-C(11A)-C(12A)-C(13A)	0.5(11)
C(16A)-C(11A)-C(12A)-C(13A)	179.8(7)
C(11A)-C(12A)-C(13A)-C(14A)	1.2(11)
C(11A)-C(12A)-C(13A)-C(17A)	-178.5(8)
C(12A)-C(13A)-C(14A)-C(15A)	-1.1(11)
C(17A)-C(13A)-C(14A)-C(15A)	178.6(8)
C(13A)-C(14A)-C(15A)-C(10A)	-0.7(11)
C(13A)-C(14A)-C(15A)-C(18A)	-178.5(7)
C(11A)-C(10A)-C(15A)-C(14A)	2.4(10)
N(2A)-C(10A)-C(15A)-C(14A)	-179.4(7)
C(11A)-C(10A)-C(15A)-C(18A)	-179.8(7)
N(2A)-C(10A)-C(15A)-C(18A)	-1.6(10)
N(2A)-C(2A)-C(19A)-C(20A)	85.0(9)
C(3A)-C(2A)-C(19A)-C(20A)	-32.6(11)
N(2A)-C(2A)-C(19A)-C(24A)	-94.0(9)
C(3A)-C(2A)-C(19A)-C(24A)	148.3(7)
C(24A)-C(19A)-C(20A)-C(21A)	-1.0(13)
C(2A)-C(19A)-C(20A)-C(21A)	180.0(8)
C(19A)-C(20A)-C(21A)-C(22A)	1.5(15)
C(20A)-C(21A)-C(22A)-C(23A)	-1.0(17)
C(21A)-C(22A)-C(23A)-C(24A)	0.0(18)
C(20A)-C(19A)-C(24A)-C(23A)	0.0(14)
C(2A)-C(19A)-C(24A)-C(23A)	179.2(8)
C(22A)-C(23A)-C(24A)-C(19A)	0.5(16)
N(1A)-C(3A)-C(25A)-C(26A)	-120.2(7)

C(2A)-C(3A)-C(25A)-C(26A)	123.6(7)
N(1A)-C(3A)-C(25A)-C(30A)	60.2(8)
C(2A)-C(3A)-C(25A)-C(30A)	-56.0(9)
C(30A)-C(25A)-C(26A)-C(27A)	-1.3(13)
C(3A)-C(25A)-C(26A)-C(27A)	179.1(8)
C(25A)-C(26A)-C(27A)-C(28A)	5.2(15)
C(26A)-C(27A)-C(28A)-C(29A)	-5.6(15)
C(27A)-C(28A)-C(29A)-C(30A)	2.0(14)
C(26A)-C(25A)-C(30A)-C(29A)	-2.2(11)
C(3A)-C(25A)-C(30A)-C(29A)	177.4(7)
C(28A)-C(29A)-C(30A)-C(25A)	1.9(12)
C(4B)-N(1B)-C(1B)-N(2B)	172.1(6)
C(3B)-N(1B)-C(1B)-N(2B)	3.1(8)
C(10B)-N(2B)-C(1B)-N(1B)	165.0(6)
C(2B)-N(2B)-C(1B)-N(1B)	-0.2(8)
C(1B)-N(2B)-C(2B)-C(19B)	-129.2(7)
C(10B)-N(2B)-C(2B)-C(19B)	65.6(9)
C(1B)-N(2B)-C(2B)-C(3B)	-2.5(7)
C(10B)-N(2B)-C(2B)-C(3B)	-167.8(6)
C(1B)-N(1B)-C(3B)-C(25B)	-128.6(6)
C(4B)-N(1B)-C(3B)-C(25B)	62.9(8)
C(1B)-N(1B)-C(3B)-C(2B)	-4.3(7)
C(4B)-N(1B)-C(3B)-C(2B)	-172.8(6)
C(19B)-C(2B)-C(3B)-C(25B)	-107.4(7)
N(2B)-C(2B)-C(3B)-C(25B)	127.9(6)
C(19B)-C(2B)-C(3B)-N(1B)	128.5(6)
N(2B)-C(2B)-C(3B)-N(1B)	3.9(6)
C(1B)-N(1B)-C(4B)-C(9B)	95.3(8)
C(3B)-N(1B)-C(4B)-C(9B)	-97.3(8)
C(1B)-N(1B)-C(4B)-C(5B)	-83.2(8)
C(3B)-N(1B)-C(4B)-C(5B)	84.2(8)
C(9B)-C(4B)-C(5B)-C(6B)	-0.2(10)
N(1B)-C(4B)-C(5B)-C(6B)	178.3(6)
C(9B)-C(4B)-C(5B)-S(1B)	-178.2(5)
N(1B)-C(4B)-C(5B)-S(1B)	0.3(9)
O(3B)-S(1B)-C(5B)-C(6B)	-3.3(6)

O(1B)-S(1B)-C(5B)-C(6B)	-125.0(6)
O(2B)-S(1B)-C(5B)-C(6B)	116.1(6)
O(3B)-S(1B)-C(5B)-C(4B)	174.7(6)
O(1B)-S(1B)-C(5B)-C(4B)	53.0(6)
O(2B)-S(1B)-C(5B)-C(4B)	-65.9(6)
C(4B)-C(5B)-C(6B)-C(7B)	-0.7(10)
S(1B)-C(5B)-C(6B)-C(7B)	177.4(5)
C(5B)-C(6B)-C(7B)-C(8B)	0.9(11)
C(6B)-C(7B)-C(8B)-C(9B)	-0.3(12)
C(5B)-C(4B)-C(9B)-C(8B)	0.8(11)
N(1B)-C(4B)-C(9B)-C(8B)	-177.7(7)
C(7B)-C(8B)-C(9B)-C(4B)	-0.6(12)
C(1B)-N(2B)-C(10B)-C(15B)	-77.8(9)
C(2B)-N(2B)-C(10B)-C(15B)	85.4(9)
C(1B)-N(2B)-C(10B)-C(11B)	98.5(8)
C(2B)-N(2B)-C(10B)-C(11B)	-98.3(8)
C(15B)-C(10B)-C(11B)-C(12B)	-2.1(12)
N(2B)-C(10B)-C(11B)-C(12B)	-178.3(7)
C(15B)-C(10B)-C(11B)-C(16B)	179.5(8)
N(2B)-C(10B)-C(11B)-C(16B)	3.3(12)
C(10B)-C(11B)-C(12B)-C(13B)	2.0(13)
C(16B)-C(11B)-C(12B)-C(13B)	-179.6(9)
C(11B)-C(12B)-C(13B)-C(14B)	-0.9(14)
C(11B)-C(12B)-C(13B)-C(17B)	176.3(9)
C(12B)-C(13B)-C(14B)-C(15B)	-0.2(14)
C(17B)-C(13B)-C(14B)-C(15B)	-177.6(9)
C(13B)-C(14B)-C(15B)-C(10B)	0.1(13)
C(13B)-C(14B)-C(15B)-C(18B)	177.8(9)
C(11B)-C(10B)-C(15B)-C(14B)	1.1(12)
N(2B)-C(10B)-C(15B)-C(14B)	177.2(7)
C(11B)-C(10B)-C(15B)-C(18B)	-176.5(9)
N(2B)-C(10B)-C(15B)-C(18B)	-0.4(12)
N(2B)-C(2B)-C(19B)-C(24B)	80.8(9)
C(3B)-C(2B)-C(19B)-C(24B)	-38.6(10)
N(2B)-C(2B)-C(19B)-C(20B)	-96.4(8)
C(3B)-C(2B)-C(19B)-C(20B)	144.2(7)

C(24B)-C(19B)-C(20B)-C(21B)	0.9(13)
C(2B)-C(19B)-C(20B)-C(21B)	178.1(8)
C(19B)-C(20B)-C(21B)-C(22B)	2.2(15)
C(20B)-C(21B)-C(22B)-C(23B)	-3.8(17)
C(21B)-C(22B)-C(23B)-C(24B)	2.1(16)
C(20B)-C(19B)-C(24B)-C(23B)	-2.6(12)
C(2B)-C(19B)-C(24B)-C(23B)	-179.8(8)
C(22B)-C(23B)-C(24B)-C(19B)	1.1(14)
N(1B)-C(3B)-C(25B)-C(26B)	-137.0(7)
C(2B)-C(3B)-C(25B)-C(26B)	106.3(8)
N(1B)-C(3B)-C(25B)-C(30B)	47.1(10)
C(2B)-C(3B)-C(25B)-C(30B)	-69.6(9)
C(30B)-C(25B)-C(26B)-C(27B)	-0.3(13)
C(3B)-C(25B)-C(26B)-C(27B)	-176.3(8)
C(25B)-C(26B)-C(27B)-C(28B)	1.1(14)
C(26B)-C(27B)-C(28B)-C(29B)	-2.2(15)
C(27B)-C(28B)-C(29B)-C(30B)	2.4(15)
C(28B)-C(29B)-C(30B)-C(25B)	-1.6(14)
C(26B)-C(25B)-C(30B)-C(29B)	0.5(13)
C(3B)-C(25B)-C(30B)-C(29B)	176.5(8)

Symmetry transformations used to generate equivalent atoms: