Asymmetric, Anti-Selective Scandium-Catalyzed Sakurai-Hosomi Reaction and its application to Stereoselective Syntheses of *N*-Boc- D-alloisoleucine and D-isoleucine

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Supporting Information

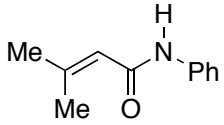
General Information. All reactions were carried out under an atmosphere of argon or nitrogen in oven-dried glassware with magnetic stirring. THF, toluene, ether, and CH_2Cl_2 were purified by passage through a bed of activated alumina.[1] Solvents and reagents were purified prior to use following the guidelines of Perrin and Armarego.[2] Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and ceric ammonium nitrate stain followed by heating. Optical rotations were measured on a Jasco DIP-0181 digital polarimiter with a sodium lamp and are reported as follows: $[\alpha]_D^{T\ ^\circ C}$ (*c* g/100 mL, solvent). Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. ^{1}H NMR spectra were recorded on a Varian Inova-500 (500 MHz) or Varian Mercury-400 (400 MHz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 7.26 ppm). Data reported as (s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet, brs=broad; integration; coupling constant(s) in Hz). Proton-decoupled ^{13}C NMR spectra were recorded on Varian Inova-500 (125 MHz) or Varian Mercury-400 (100 MHz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 77.0 ppm). High resolution mass spectra were obtained on Jeol AX-505 or SX-102 spectrometers at the Harvard University Mass

(1) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

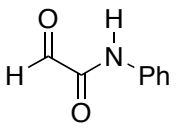
(2) Perrin, D. D. and Armarego, W. L. F. *Purification of Laboratory Chemicals*; 3rd ed., Pergamon Press, Oxford, 1988.

Spectrometry Laboratory. Analytical high performance liquid chromatography (HPLC) was performed on a Hewlett-Packard 1100 Series HPLC with a diode array detector using the indicated chiral column.

Preparation of *N*-Phenyl Glyoxamide



3-Methyl-*N*-phenylbut-2-enamide[3]. A previously dried round-bottom flask was charged with aniline (12.0 mL, 132 mmol), pyridine (19.4 mL, 239 mmol) and Et_2O (400 mL). The solution was cooled to 0 °C and 3,3-dimethylacryloyl chloride (14.4 mL, 129 mmol) was added dropwise (cannula transfer), forming a white paste. The ice bath was removed and the mixture was stirred at rt for 75 min. Then the mixture was diluted with Et_2O and washed 3 times with HCl 10% (v/v), saturated aqueous $NaHCO_3$, and brine. The organice phase was dried over $MgSO_4$, filtered and concentrated under reduced pressure. The unpurified solid (>95% pure as indicated by ^{1}H NMR spectroscopy) was used without any further purification. R_f 0.24 (20% EtOAc in hexanes); IR (neat) 3301, 3132, 3068, 2973, 1663, 1641, 1599, 1538, 1439, 1310, 1251, 1164, 753, 693 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, 2H, *J* = 7 Hz), 7.35 (brs, 1H), 7.29 (t, 2H, *J* = 7 Hz), 7.07 (t, 1H, *J* = 7 Hz), 5.72 (s, 1H), 2.21 (d, 3H, *J* = 1 Hz), 1.88 (d, 3H, *J* = 1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 153.2, 138.2, 128.9, 123.9, 119.7, 118.7, 27.3, 19.9; HRMS (ES): Exact mass calcd for $C_{11}H_{13}NO$ $[M+H]^+$, requires *m/z*: 176.1075, found *m/z*: 176.1073.



***N*-Phenylglyoxamide (2)**. A 500-mL round-bottom flask equipped with a stirbar, 3-methyl-*N*-phenylbut-2-enamide (14.0 g, 79.9 mmol) was solubilized using ACS reagent grade CH_2Cl_2 (100 mL) and MeOH (100 mL). The flask was fitted with a septum, flushed using a N_2 stream and then cooled to –78 °C (a white precipitate forms upon cooling). Once at this temperature (internal monitoring), ozone/oxygen was introduced into the reaction using a fritted glass inlet at an approx. rate of 5 L per minute. The reaction was monitored by TLC and was found at all times to be complete upon apperance of the blue coloration of the saturated ozone solution. Oxygen was bubbled through for 15 min, yielding a clear colorless solution. Methyl sulfide (29.3 mL, 399 mmol) was then added and the dry ice/acetone bath was

(3) We thank Dr. André Beauchemin for providing an efficient synthesis of this compound.

removed. Stirring was continued until all ozonide [R_f 0.18 (20% EtOAc in hexanes)] was consumed (2.5 – 3 h). The solution was then concentrated at 50 Torr and the resulting crude oil was transferred to a sepratory funnel using Et_2O and diluted to a total volume of approx. 3.5 L. The solution was washed 4 times with water (150 mL) and concentrated under reduced pressure. Then, MeOH was azeotroped by adding Et_2O (~100 mL) and H_2O (~15 mL) and concentrated in vacuo at ~10 Torr (repeated once). At this stage a white solid forms, determined to be the hydrate of the title compound. Recrystallization from 1,2-dichloroethane (310 mL, avoid heating above 50 °C, allowed to stand at room temperature overnight) affords, after filtration and wash with cold CH_2Cl_2, 8.1 g (61%) of the title compound as colorless needles. mp = 110 – 113 °C; ^{1}H NMR (400 MHz, DMSO–d_6) δ 9.73 (s, 1H), 7.69 – 7.65 (m, 2H), 7.31 – 7.26 (m, 2H), 7.06 – 7.03 (m, 1H), 6.59 (d, 2H, J = 7 Hz), 5.06 (t, 1H, J = 7 Hz). A portion of this solid was then dehydrated by vacuum sublimation as follows: *N*-phenylglyoxamide hydrate (4.0 g, 23.9 mmol) was charged in a vacuum sublimation apparatus. The vessel was immersed in an oil bath (65 °C), covered with aluminum foil, and after 5 min the flask was carefully opened to vacuum (55 mTorr). After 5 min, water was allowed to flow to the condenser. Then the temperature was slowly increased to 105 °C (~30 °C/hr). Sublimation occurred overnight, forming a slightly yellow solid and a dark orange tar at the bottom of the vessel. The next morning, the apparatus was cooled to room temperature, filled with argon and the solid was isolated under inert atmosphere to afford 2.62 g (73%) of the title compound. IR (neat) 3332, 3118, 3056, 2868, 1737, 1682, 1600, 1551, 1493, 1444, 761, 716, 693 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.38 (s, 1H), 8.37 (brs, 1H), 7.66 (dd, 2H, J = 1, 8 Hz), 7.21 (tt, 1H, J = 1, 7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.8, 157.6, 136.0, 129.6, 125.9, 120.1.

General Procedure for the Corey–Kim Chlorination.[4] To a round-bottom flask containing a magenetic stirring bar, was added recrystallized *N*-chlorosuccinimide (1.1 equiv) and CH_2Cl_2 (0.20 M with respect to allylic alcohol). To this solution, which was cooled to –20 °C, was added dimethylsulfide (1.2 equiv) dropwise *via* syringe, and the appropriate *E* allylic alcohol (1.0 equiv). The reaction mixture was warmed to 0 °C and

(4) Corey, E. J.; Kim, C. U.; Takeda, M. *Tetrahedron Lett*. **1972**, *42*, 4339.

aged for 1.5 h or until complete as judged by TLC analysis. The reaction was transferred to a separatory funnel, washed with 2 x brine, dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified *via* flash chromatography to afford the product as a colorless oil that was judged homogeneous by ^{1}H NMR spectroscopy.

Procedure for the Preparation of (*Z*)-Crotyltrichlorosilane.[5] In a sealed tube, equipped with a side-arm and magnetic stirring bar, was condensed 1,3-butadiene (1 equiv) at –78 °C, under a flow of N_2. To this tube was sequentially added $Pd(PPh_3)_4$ (0.05 equiv) and $HSiCl_3$ (1.0 equiv) at –78 °C, once again under a flow of N_2. The reaction mixture was aged overnight at rt. The entire reaction mixture was filtered through filter paper and rinsed with ether. The filtrate was concentrated *in vacuo* and the residue purified by distillation (142 °C). The title compound was isolated as a colorless oil that was judged homogenous as determined by ^{1}H NMR spectroscopy.

General Procedure for the Preparation of *E* Allylic Trichlorosilanes.[6] A round-bottom flask, equipped with a magnetic stirring bar, was charged with CuCl (0.03 equiv) in an inert atmosphere (N_2) glove box. The flask was fitted with a rubber septum, removed from the inert atmosphere box, and charged with Et_2O (2.0 M with respect to allylic chloride). To the resulting suspension, which was cooled to 0 °C, was added a solution of the appropriate *E* allylic chloride (1.0 equiv) and $HSiCl_3$ (1.4 equiv) *via* cannula over 10 min. A grey precipitate immediately formed and a slight exotherm was observed at this point. The reaction was warmed to rt and aged for 1.5 h or until complete as judged by TLC analysis. The reaction mixture was then filtered through a pad of celite, concentrated *in vacuo* and used as is without further purification. The title compounds were used within 2 – 3 hours of isolation. Upon standing, a small amount of yellow precipitate begins to form. These solids were filtered once again through a pad of celite before proceeding.

General Procedure A: Preparation of *E* and *Z* Allylic Trimethylsilanes.[7] To a 2-neck round-bottom flask, equipped with a magnetic stirring bar and condenser, was

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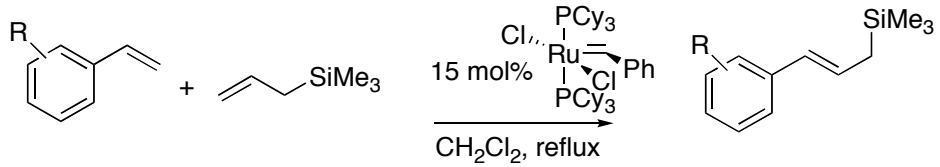
(5) Kira, M.; Hino, T.; Sakurai, H. *Tetrahedron Lett*. **1989**, *30*, 1099.

(6) D'Aniello, F.; Falorni, M.; Mann, A.; Taddei, M. *Tetrahedron: Asymm*. **1996**, *7*, 1217.

(7) D'Aniello, F.; Falorni, M.; Mann, A.; Taddei, M. *Tetrahedron: Asymmetry* **1996**, *7*, 1217.

charged with Et_2O (1.1 M with respect to *E* or *Z* allylic trichlorosilane as appropriate) and MeMgI (4.5 equiv, 2.4 M in Et_2O). To this solution, which was cooled to 0 °C, was added the appropriate *E* or *Z* allylic trichlorosilane. A small exotherm was observed at this point. The reaction mixture was refluxed overnight, cooled to 0 °C, and slowly quenched with cold, saturated aq. NH_4Cl. A significant amount of white solids precipitated from the solution. This mixture was diluted with water and extracted with 2 x Et_2O. The organics were washed once with brine, dried with $MgSO_4$, and concentrated *in vacuo*. The rotovap pressure was carefully monitored to not drop below 180 Torr since the title compounds were all slightly to moderately volatile. The title compounds were then isolated either by distillation or flash chromatography.

General Procedure B: Preparation of *E* Aryl Allylic Trimethylsilanes via Cross-Methathesis.[8]



To a 2-neck round-bottom flask, equipped with a magenetic stirring bar, was charged with Grubb's 1st generation metathesis catalyst (0.15 equiv) in an inert atmosphere (N_2) glove box. The flask was then equipped with a condenser, fitted with rubber septa, removed from the inert atmosphere box, and charged with CH_2Cl_2 (0.5 M with respect to styrene). To this deep purple solution was simultaneously added the appropriate styrene (1.0 equiv) and allyltrimethylsilane (1.5 equiv). The reaction mixture was refluxed overnight under a gentle stream of N_2, filtered through a plug of silica gel, and eluted with neat hexanes. The solution was concentrated *in vacuo* and the residue initially purified *via* flash chromatography (hexanes). As the title compounds were often contaminated with small amounts of styrene, which could not be separated by flash chromatography, this material was subsequently subjected to flash chromatography with $AgNO_3$ impregnated silica gel.[9]

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(8) Chatterjee, A. K.; Choi, T.-L.; Sanders, D. P.; Grubbs, R. H. *J. Am. Chem. Soc.* **2003**, *125*, 11360.

(9) (a) Li, T.-S.; Li, J.-T.; Li, H.-Z. *J. Chromatography A* **1995**, *715*, 372-375. (b) Williams, C. M.; Mander, L. N. *Tetrahedron*, **2001**, *57*, 425.

General Procedure C: Scandium-Catalyzed Sakurai–Hosomi Reaction. To an oven-dried, 4-mL vial containing a magnetic stirring bar, was added the appropriate (*S,S*)-bis(oxazolinyl)pyridine ligand[10] (0.055 – 0.165 equiv), $Sc(OTf)_3$ (0.05 – 0.15 equiv), and 4Å MS (100 mg/mmol) in an inert atmosphere (N_2) glove box. To a separate vial was added **2** (1 equiv). Both vials were fitted with serum caps and removed from the inert atmosphere box. The vial containing the metal/ligand was charged with CH_2Cl_2 (0.17 M with respect to **2**). The resulting suspension was stirred at room temperature for 1.5 h. To this solution was added the appropriate allylic silane (8.5 equiv) followed by **2** as a solution in CH_2Cl_2 (0.25 M) via syringe. The vial that had contained **2** was rinsed with CH_2Cl_2 (0.5 M with respect to **2**) and the solution was transferred to the reaction vial via syringe. The reaction mixture was stirred at the appropriate temperature overnight, at which point all reactions were deemed complete by TLC analysis.

The entire solution was filtered through a plug of silica gel and eluted with Et_2O. The filtrate was concentrated *in vacuo* and diluted with MeOH (0.07 M with respect to **6**) and a few drops of conc. HCl. This solution was stirred for 5 – 10 minutes, diluted with Et_2O and washed successively with saturated aq. $NaHCO_3$ and brine. The organic layer was concentrated *in vacuo* and the residue was purified *via* flash chromatography (10 – 15% EtOAc/hexanes) to afford the title compounds. Importantly, unreacted allylic silane could be isolated and reused in subsequent reactions without compromising yield or selectivities.

General Procedure D: Mesylation of α-Hydroxy *N*-Phenylamides.[11] An oven-dried round-bottom flask, equipped with a magnetic stirring bar was charged with 1.0 M solution of appropriate α-hydroxy amide in CH_2Cl_2. This solution was treated with Et_3N (2.0 equiv) and methanesulfonyl chloride (2.0 equiv) at room temperature for 36 h or until complete as judged by TLC analysis. The reaction mixture was quenched with pH 7 buffer solution, diluted with DCM and transferred to a separatory funnel. The organic layer was extracted and washed with saturated $NaHCO_3$, dried over $MgSO_4$ and

(10) During the course of this study, all pybox ligands were prepared as described in Davies, I. W.; Gerena, L.; Nu, L.; Larsen, R. D.; Reider, P. J. *J. Org. Chem.* **1996**, *61*, 9629. The ligands were stored and dispensed in a dry-box under a positive pressure of N_2.

(11) Evans, D. A.; Dow, R. L.; Shih, T. L.; Takacs, J. M.; Zahler, R. *J. Am. Chem. Soc.* **1990**, *112*, 5290.

concentrated *in vacuo* to afford α-methanesulfonyloxy *N*-phenylamides cleanly without the need for flash chromatography.

General Procedure E: Azidation of α-methanesulfonyloxy *N*-Phenylamides.[12] An oven-dried round-bottom flask, equipped with a magnetic stirring bar was charged with 0.5 M solution of appropriate α-sulfonyloxy amide in DMF. This solution was treated with NaN_3 (1.1 equiv) at 70 °C for 48 h or until complete as judged by TLC analysis. The reaction mixture was quenched with pH 7 buffer solution, diluted with DCM and transferred to a separatory funnel. The organic layer was extracted and washed with H_2O, dried over $MgSO_4$ and concentrated *in vacuo* to afford α-azido *N*-phenylamides cleanly without the need for flash chromatography.

General Procedure F: One-pot Reduction and bisBoc protection of α-azido *N*-phenylamides. A 0.07 M solution of appropriate α-azido *N*-phenylamide in EtOH was stirred with 30% by mass (based on substrate) of 5% Pd on C catalyst under 1 atm of H_2 at room temperature for 5 h or until complete as judged by TLC analysis.[13] The reaction mixture was filtered over Celite and the filtrate was concentrated in vacuo. The crude intermediate was subsequently treated with Boc_2O (5.0 equiv) and DMAP (2.0 equiv)[14] in 1:9 CH_2Cl_2:MeCN at room tempertature over 1 h or until complete as judged by TLC analysis. The reaction mixture was concentrated *in vacuo*. The residue was purified *via* flash chromatography (2% EtOAc/hexanes) to afford the corresponding bisBoc protected derivatives.

General Procedure G: *N*-(*tert*-butoxy)carbonyl-*N*-phenylamide Hydrolysis.[15]

A round-bottom flask equipped with a magnetic stirring bar was charged with 0.05 M solution of appropriate carbamate in 3:1 THF:H_2O. To this solution, which was cooled to 0 °C, was added 30% (by wt.) aqueous solution of H_2O_2 (5.0 equiv), followed by LiOH (2.0 equiv). The mixture was warmed slowly to room temperature and stirred for 3 h at rt

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(12) Danieli, B.; Lesma, G.; Passarella, D.; Sacchetti, A.; Silvani, A.; Virdis, A. *Org. Lett.* **2004**, *6*, 493.

(13) Corey, E. J.; Nicolaou, K. C.; Balanson, R. D.; Machida, Y. *Synthesis* **1975**, *9*, 590.

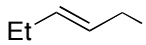
(14) Evans, D., A.; Fitch, D., M.; Smith, T., E.; Cee. V., J. *J. Am. Chem. Soc.* **2000**, *122*, 10033.

(15) Evans, D. A.; Britton, T. C.; Ellman, J. A. *Tetrahedron Lett.* **1987**, *28*, 6141.

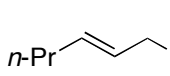
or until complete as judged by TLC analysis. The reaction was cooled to 0 °C, and treated with 1.5 N aqueous solution of Na_2SO_3 (1.1 equiv). The mixture was aged for 5 min at room temperature, and was diluted with H_2O and the organic layer was extracted with CH_2Cl_2 to remove HN(Boc)Ph. The remaining basic aqueous layer was acidified down to pH 4 and extracted with CH_2Cl_2. The organic layer was concentrated *in vacuo* to afford the corresponding α-(*tert*-butoxycarbonyl)-aminoacid in quantitative yield, which was judged homogeneous by ^{1}H NMR spectroscopy.

Substrate 3 [16] and **9**[7] are known compounds but were prepared according to General Procedure A from the corresponding trichloroallylsilanes.

Cinnamyl-trimethylallylsilane [17] is a known compound but was prepared according to General Procedure B and isolated by flash chromatography over $AgNO_3$ impregnated silica gel (1% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (85% yield).



(*E*)-trimethyl(pent-2-enyl)silane. The indicated compound was prepared according to General Procedure A and isolated by flash chromatography (1% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (59% yield): IR (film) 2958, 1458, 1404, 1248, 1156, 1048, 962, 846 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.42 – 5.38 (m, 1H), 5.35 – 5.31 (m, 1H), 2.04 – 2.01 (m, 2H), 1.44 (d, 2H, *J* = 7.6 Hz), 0.99 (t, 3H, *J* = 7.3 Hz) 0.02 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 130.9, 125.1, 26.1, 22.7, 14.7, –1.8.



(*E*)-hex-2-enyltrimethylsilane. The indicated compound was prepared according to General Procedure A and isolated by flash chromatography (1% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (57% yield): IR (film) 2957, 1648, 1462, 1405, 1248, 1157, 1060, 963, 843 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.42 – 5.35 (m, 1H), 5.29 – 5.20 (m, 1H), 1.95 (dt, 2H, J = 7.0 Hz), 1.41 – 1.31 (m, 4H), 0.88 (t, 3H, *J* = 7.3 Hz), 0.01 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 129.0, 126.3, 35.2, 23.3, 22.8, 13.8, –1.8.

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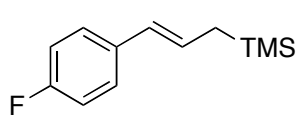
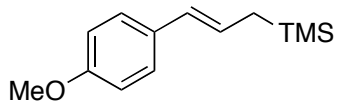
(16) Denmark, S. E.; Fu, J. *J. Am. Chem. Soc.* **2001**, *123*, 9488.

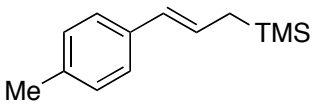
(17) Terao, J.; Jin, Y.; Torii, K.; Kambe, N. *Tetrahedron* **2004**, *60*, 1301.



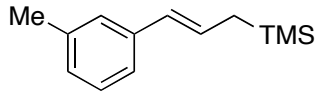
(*E*)-*p*-fluorophenyl-allyltrimethylsilane. The indicated compound was prepared according to General Procedure B and isolated by flash chromatography over $AgNO_3$ impregnated silica gel (1% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (55% yield): IR (film) 3017, 2955, 2897, 1882, 1645, 1601, 1508, 1402, 1230, 1156, 1093, 1026, 963, 863, 766, 695 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 2H), 6.99 – 6.93 (m, 2H), 6.22 – 6.11 (m, 2H), 1.65 (d, 2H, *J* = 7.0 Hz), 0.05 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 160.5, 134.9, 127.8, 127.2, 115.5, 24.1, –1.6.



(*E*)-*p*-methoxyphenyl-allyltrimethylsilane. The indicated compound was prepared according to General Procedure B and isolated by flash chromatography over $AgNO_3$ impregnated silica gel (5% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (61% yield): IR (film) 3014, 2953, 2901, 2835, 2358, 1644, 1608, 1510, 164, 142, 1286, 1247, 1174, 1150, 1038, 962, 863, 814 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.22 (m, 2H), 6.85 – 6.81 (m, 2H), 6.21 – 6.05 (m, 2H), 3.80 (s, 3H), 1.63 (d, 2H, *J* = 0.7 Hz), 0.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 152.3, 131.7, 127.8, 126.8, 126.7, 125.8, 114.2, 114.0, 55.6, 23.9, –1.5; HRMS (CI$^+$): Exact mass calcd for $C_{13}H_{20}OSi$ $[M+NH_4]^+$, requires m/z: 238.1621, found m/z: 238.1624.

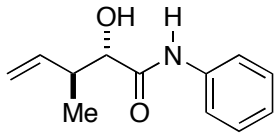


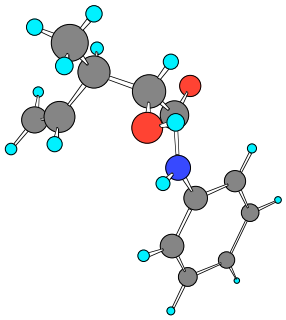
(*E*)-*p*-tolyl-allyltrimethylsilane. The indicated compound was prepared according to General Procedure B and isolated by flash chromatography over $AgNO_3$ impregnated silica gel (1% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (83% yield): IR (film) 3019, 2954, 1644, 1513, 1449, 1402, 1248, 1150, 1027, 962, 864, 838, 802, 752, 693, 660 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.22 (m, 2H), 7.11 – 7.09 (m, 2H), 6.22 – 6.21 (m, 2H), 2.33 (s, 3H), 1.67 (d, 2H, *J* = 7.0 Hz), 0.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.1, 136.0, 129.4, 128.3, 127.0, 125.6, 24.1, 21.3, –1.6; HRMS (CI$^+$): Exact mass calcd for $C_{13}H_{20}Si$ $[M+NH_4]^+$, requires m/z: 222.1678, found m/z: 222.1666.



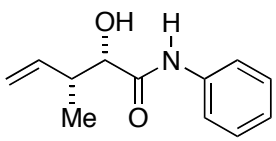
(*E*)-*m*-tolyl-allyltrimethylsilane. The indicated compound was prepared according to General Procedure B and isolated

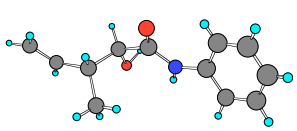
by flash chromatography over $AgNO_3$ impregnated silica gel (1% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (66% yield): IR (film) 3016, 2954, 2920, 2361, 1934, 1642, 1603, 1584, 1488, 1448, 1401, 1248, 1149, 1091, 1028, 961, 852, 777, 751, 695, 550, 515 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 1H), 7.20 – 7.11 (m, 2H), 6.99 (d, 1H, *J* = 7.5 Hz), 6.29 – 6.18 (m, 2H), 2.34 (s, 3H), 1.67 (d, 2H, *J* = 6.2 Hz), 0.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 138.2, 128.5, 127.8, 127.3, 127.2, 126.9, 126.6, 123.0, 122.8, 24.1, 21.6, –1.6; HRMS (CI$^+$): Exact mass calcd for $C_{13}H_{20}Si$ $[M+H]^+$, requires m/z: 205.1412, found m/z: 205.103.





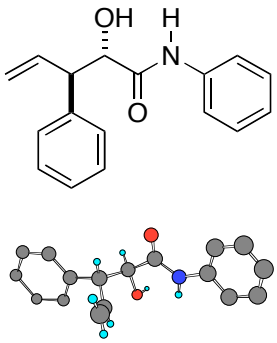
(2*S*,3*S*)-2-hydroxy-3-methyl-*N*-phenylpent-4-enamide (4). The indicated compound was prepared according to General Procedure C as a 26:1 anti/syn mixture of diastereomers. Flash chromatography (20% EtOAc/hexanes) yielded a single diastereomer (anti) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (89% yield); mp 104 – 105 °C; $[\alpha]_D$ –104.6° (*c* = 0.685, $CHCl_3$); IR (film) 3365, 2967, 1667, 1600, 1538, 1446, 1317, 1241, 1127, 1018, 920, 753, 692 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (brs, 1H), 7.54 (d, 2H, *J* = 7.8 Hz), 7.33 (t, 2H, *J* = 7.8 Hz), 7.13 (t, 1H, *J* = 7.3 Hz), 5.87 – 5.80 (m, 1H), 5.22 (s, 1H), 5.19 (d, 1H, *J* = 5.9 Hz), 4.12 (t, 1H, *J* = 4.4 Hz), 2.96 – 2.92 (m, 1H), 2.79 (d, 1H, *J* = 5.4 Hz), 1.21 (d, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 137.7, 137.3, 129.3, 124.8, 120.1, 118.0, 75.8, 41.4, 15.8; HRMS (ESI): Exact mass calcd for $C_{12}H_{15}NO_2$ $[M+H]^+$, requires *m/z*: 206.1181, found *m/z*: 206.1184; Enantiomeric excess determined by HPLC with Chiracel AD-H column, 10% *i*-PrOH/hexanes, 0.9 mL/min, 254 nm; t_r (anti major) = 15.8, t_r (anti minor) = 10.5, 95% ee, t_r (syn major) = 14.0, t_r (syn minor) 11.2, 67% ee.



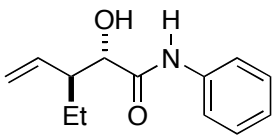


(2*S*,3*R*)-2-hydroxy-3-methyl-*N*-phenylpent-4-enamide (10). The indicated compound was prepared according to General Procedure C as a 4:1 syn/anti mixture of diastereomers. Flash chromatography (15% EtOAc/hexanes) yielded a single diastereomer (syn) as a colorless oil that was homogeneous as

judged by ^{1}H NMR spectroscopy (76% yield); mp 90 – 91°C; $[\alpha]_D$ –89.2° (*c* = 0.1, $CHCl_3$); IR (film) 3603, 2965, 1607, 1600, 1540, 1419, 1320, 1261, 1150, 1018, 960, 757 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (brs, 1H), 7.57 (d, 2H, *J* = 7.3 Hz), 7.34 (t, 2H, *J* = 7.9 Hz), 7.13 (t, 1H, *J* = 7.5 Hz), 6.01 – 5.92 (m, 1H), 5.26 – 5.19 (m, 2H), 4.26 (m, 1H), 3.02 – 2.94 (m, 1H), 2.56 (d, 1H, *J* = 2.9 Hz), 1.04 (d, 3H, *J* = 7.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) 172.6, 140.1, 129.3, 124.8, 124.8, 120.0, 117.0, 74.7, 40.6, 30.9, 11.5; HRMS (ESI): Exact mass calcd for $C_{12}H_{15}NO_2$ $[M+H]^+$, requires *m/z*: 206.1181, found *m/z*: 206.1185; Enantiomeric excess determined by HPLC with Chiracel AD-H column, 10% *i*-PrOH/hexanes, 0.9 mL/min, 254 nm; t_r (syn major) = 12.1, t_r (syn minor) = 9.3, 94% ee, t_r (anti major) = 13.8, t_r (anti minor) 8.6, 63% ee.

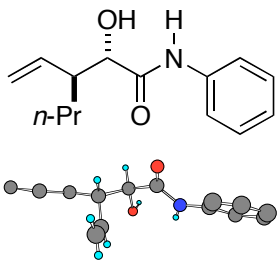


(2*S*,3*R*)-2-hydroxy-*N*,3-diphenylpent-4-enamide. The indicated compound was prepared according to General Procedure A as a 99:1 anti/syn mixture of diastereomers. Flash chromatography (15% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (67% yield); mp 125 – 128 °C; $[\alpha]_D$ –101.8° (*c* = 0.890, $CHCl_3$); IR (film) 3363, 3062, 1662, 1600, 1536, 1496, 1445, 1316, 1238, 1098, 925, 754, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (brs, 1H), 7.48 (dd, 2H, *J* = 8.8, 1.1 Hz), 7.38 – 7.35 (m, 3H), 7.34 – 7.28 (m, 4H), 7.15 – 7.10 (m, 1H), 6.22 (ddd, 1H, *J* = 16.8, 10.2, 8.4 Hz), 5.26 – 5.19 (m, 2H), 4.44 (brs, 1H), 4.05 (dd, 1H, *J* = 8.8, 3.7 Hz), 2.78 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 140.7, 137.2, 134.4, 129.3, 129.2, 128.5, 127.5, 124.8, 120.2, 119.6, 76.2, 53.1; HRMS (ESI): Exact mass calcd for $C_{17}H_{17}NO_2$ $[M+H]^+$, requires m/z: 268.1337, found m/z: 268.1344; Enantiomeric excess determined by HPLC with Whelk-(*S*) column, 10% *i*-PrOH/hexanes, 1.0 mL/min, 254 nm; t_r (anti major) = 10.7, t_r (anti minor) = 8.9, 99% ee, t_r (syn major) = 10.0, t_r (syn minor) 7.6.

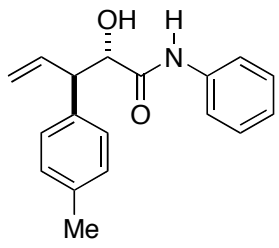


(2*S*,3*S*)-3-ethyl-2-hydroxy-*N*-phenylpent-4-enamide. The indicated compound was prepared according to General Procedure C as a 32:1 anti/syn mixture of diastereomers. Flash chromatography (15% EtOAc/hexanes) yielded a single diastereomer (anti) as a white

solid that was homogeneous as judged by ^{1}H NMR spectroscopy (76% yield); mp 49 – 51 °C; $[\alpha]_D$ –62.5° (*c* = 0.835, $CHCl_3$); IR (film) 3366, 2963, 1662, 1600, 1535, 1446, 1315, 1125, 1048, 999, 920, 754, 692 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (brs, 1H), 7.53 (d, 2H, *J* = 7.3 Hz), 7.33 (t, 2H, *J* = 7.7 Hz), 7.12 (t, 1H, *J* = 7.7 Hz), 5.71 (ddd, 1H, *J* = 17.2, 10.2, 8.4 Hz), 5.19 (dd, 1H, *J* = 10.6, 1.8 Hz), 5.17 (d, 1H, *J* = 17.6 Hz), 4.20 (brs, 1H), 2.74 (brs, 1H), 2.61 – 2.58 (m, 1H), 1.69 – 1.63 (m, 1H), 1.60 – 1.52 (m, 1H), 0.95 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 137.4, 136.3, 129.3, 124.7, 120.1, 119.2, 74.6, 49.3, 23.6, 12.1; HRMS (ESI): Exact mass calcd for $C_{13}H_{17}NO_2$ $[M+H]^+$, requires *m/z*: 220.1337, found *m/z*: 220.1338; Enantiomeric excess determined by HPLC with Chiracel OD-H column, 5% *i*-PrOH/hexanes, 0.8 mL/min, 254 nm; t_r (anti major) = 22.6, t_r (anti minor) = 34.0, 91% ee, t_r (syn major) = 21.3, t_r (syn minor) 27.1, 9% ee.

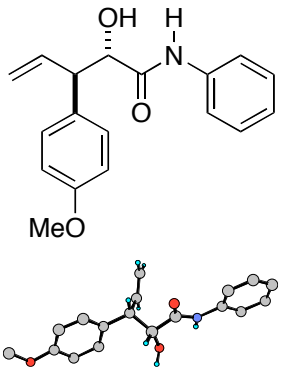


(2*S*,3*R*)-2-hydroxy-*N*,3-diphenylpent-4-enamide. The indicated compound was prepared according to General Procedure C as a 29:1 anti/syn mixture of diastereomers. Flash chromatography (15% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (71% yield); mp 58 – 61 °C; $[\alpha]_D$ –63.9° (*c* = 0.825, $CHCl_3$); IR (film) 3361, 2944, 1661, 1600, 1446, 1316, 1126, 1075, 999, 920, 754, 692 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.36 (brs, 1H), 7.53 (d, 2H, *J* = 7.8 Hz), 7.32 (t, 2H, *J* = 7.8 Hz), 7.12 (t, 1H, *J* = 7.3 Hz), 5.71 (ddd, 1H, *J* = 17.1, 10.3, 8.3 Hz), 5.19 – 5.14 (m, 2H), 4.18 (d, 1H, *J* = 3.4 Hz), 2.90 (brs, 1H), 2.73 – 2.67 (m, 1H), 1.59 – 1.49 (m, 2H), 1.44 – 1.37 (m, 1H), 1.37 – 1.29 (m, 1H), 0.93 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 137.3, 136.5, 129.2, 124.8, 120.1, 118.9, 75.0, 47.3, 32.8, 20.6, 14.2; HRMS (ESI): Exact mass calcd for $C_{14}H_{19}NO_2$ $[M+H]^+$, requires m/z: 234.1494, found m/z: 234.1490; Enantiomeric excess determined by HPLC with Chiracel OD-H column, 10% *i*-PrOH/hexanes, 0.9 mL/min, 254 nm; t_r (anti major) = 18.8, t_r (anti minor) = 26.4, 93% ee, t_r (syn major) = 19.9, t_r (syn minor) 22.9, 11% ee.

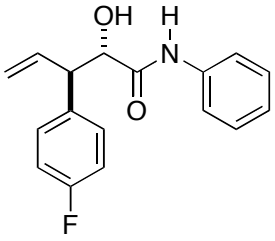


(2*S*,3*R*)-2-hydroxy-*N*-phenyl-3-*p*-tolylpent-4-enamide. The indicated compound was prepared according to General Procedure C as a 99:1 anti/syn mixture of diastereomers. Flash

chromatography (15% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (75% yield); mp 146 – 147 °C; $[\alpha]_D$ –94.5° (*c* = 1.05, $CHCl_3$); IR (film) 3360, 1660, 1600, 1531, 1444, 1316, 1099, 922, 821, 754, 692 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (brs, 1H), 7.48 (d, 2H, *J* = 7.7 Hz), 7.32 (t, 2H, *J* = 7.7 Hz), 7.25 (d, 2H, *J* = 8.1 Hz), 7.18 (d, 2H, *J* = 7.7 Hz), 7.12 (t, 1H, *J* = 7.3 Hz), 6.20 (ddd, 1H, *J* = 17.2, 10.2, 8.8 Hz), 5.23 (d, 1H, *J* = 9.9 Hz), 5.21 (d, 1H, *J* = 17.2 Hz), 4.42 (t, 1H, *J* = 4.0 Hz), 4.01 (dd, 1H, *J* = 8.4, 3.3 Hz), 2.76 (d, 1H, *J* = 4.8 Hz), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 137.6, 137.3, 137.1, 134.6, 129.9, 129.2, 128.3, 124.8, 120.1, 119.4, 76.2, 52.7, 21.3; HRMS (ESI): Exact mass calcd for $C_{18}H_{19}NO_2$ $[M+H]^+$, requires m/z: 282.1494, found m/z: 282.1486; Enantiomeric excess determined by HPLC with Whelk-(*S*) column, 10% *i*-PrOH/hexanes, 1.0 mL/min, 254 nm; t_r (anti major) = 12.0, t_r (anti minor) = 9.5, 99% ee.

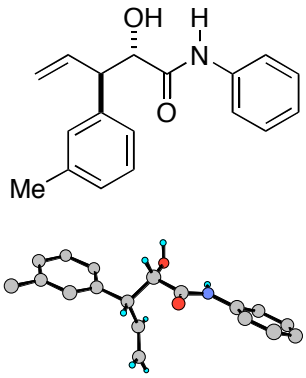


(2*S*,3*R*)-2-hydroxy-*N*-phenyl-3-*p*-methoxyphenylpent-4-enamide. The indicated compound was prepared according to General Procedure C as a 95:5 anti/syn mixture of diastereomers. Flash chromatography (30% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (64% yield); mp 135 – 136 °C; $[\alpha]_D$ –90.0° (*c* = 0.88, $CHCl_3$); IR (film) 3357, 1661, 1634, 1560, 1529, 1511, 1443, 1316, 1247, 1181, 1092, 1034, 924, 844, 754, 691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (brs, 1H), 7.48 (d, 2H, *J* = 7.3 Hz), 7.35 – 7.26 (m, 4H), 7.12 (t, 1H, *J* = 7.5 Hz), 6.90 (d, 2H, *J* = 8.8 Hz), 6.18 (ddd, 1H, *J* = 17.2, 10.0, 8.4 Hz), 5.24 – 5.21 (m, 2H), 4.38 (dd, 1H, *J* = 5.2, 3.7 Hz), 3.99 (dd, 1H, *J* = 8.6, 5.2 Hz), 3.79 (s, 3H), 2.89 (d, 1H, *J* = 4.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170. 0, 158.9, 137.3, 134.7, 132.6, 129.5, 129.2, 120.2, 119.3, 114.5, 76.3, 55.5, 52.3; HRMS (ESI): Exact mass calcd for $C_{18}H_{19}NO_3$ $[M+H]^+$, requires m/z: 298.1443, found m/z: 298.1448; Enantiomeric excess determined by HPLC with Whelk-(*S*) column, 10% *i*-PrOH/hexanes, 1.0 mL/min, 235 nm, t_r (anti major) = 23.3, t_r (anti minor) = 17.6, 97% ee.

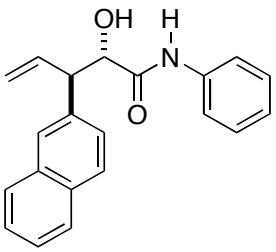


(2*S*,3*R*)-3-(4-fluorophenyl)-2-hydroxy-*N*-phenylpent-4-enamide. The indicated compound was prepared according to General Procedure C as a 99:1 anti/syn mixture of diastereomers.

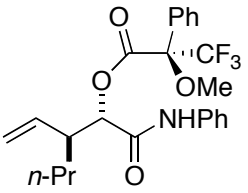
Flash chromatography (15% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (73% yield); mp 150 – 153 °C; $[\alpha]_D$ –93.1° (*c* = 1.035, $CHCl_3$); IR (film) 3363, 1652, 1600, 1537, 1508, 1445, 1318, 1220, 1094, 934, 834, 755, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (brs, 1H), 7.49 (d, 2H, *J* = 7.3 Hz), 7.34 – 7.30 (m, 4H), 7.13 (t, 1H, *J* = 7.3 Hz), 7.04 (t, 2H, *J* = 8.8 Hz), 6.16 (dq, 1H, *J* = 17.2, 10.2, 8.4 Hz), 5.25 (d, 1H, *J* = 10.2 Hz), 5.19 (d, 1H, *J* = 17.2 Hz), 4.40 (dd, 1H, *J* = 5.1, 3.7 Hz), 4.05 (dd, 1H, *J* = 8.4, 2.9 Hz), 2.83 (d, 1H, *J* = 5.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 137.1, 136.4, 134.3, 130.1, 130.0, 129.3, 125.0, 120.2, 119.8, 116.0, 115.8, 76.2, 52.3; HRMS (ESI): Exact mass calcd for $C_{17}H_{16}FNO_2$ $[M+H]^+$, requires m/z: 286.1243, found m/z: 286.1248; Enantiomeric excess determined by HPLC with Whelk-(*S*) column, 10% *i*-PrOH/hexanes, 0.8 mL/min, 254 nm; t_r (anti major) = 12.7, t_r (anti minor) = 10.3, 99% ee.



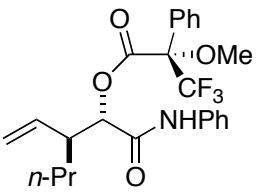
(2*S*,3*R*)-2-hydroxy-*N*-phenyl-3-*m*-tolylpent-4-enamide. The indicated compound was prepared according to General Procedure A as a 89:11 anti/syn mixture of diastereomers. Flash chromatography (15% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (64% yield); mp 88 – 91 °C; $[\alpha]_D$ –81.4° (*c* = 0.71, $CHCl_3$); IR (film) 3365, 3066, 3013, 2920, 2356, 1941, 1866, 1660, 1601, 1538, 1496, 1445, 1316, 1094, 1002, 926, 792, 754, 723, 691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (brs, 1H), 7.46 – 7.49 (dd, 2H), 7.35 – 7.25 (m, 3H), 7.17 – 7.10 (m, 4H), 6.22 (ddd, 1H, *J* = 17.2, 9.3, 8.4 Hz), 5.25 – 5.23 (m, 2H), 4.54 (dd, 1H, *J* = 4.9, 3.9 Hz), 4.02 (dd, 1H, *J* = 8.8, 3.9 Hz), 2.66 (d, 1H, *J* = 4.4 Hz), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 140.6, 139.0, 137.2, 134.4, 129.2, 129.1, 128.3, 125.4, 124.8, 120.1, 119.5, 76.2, 53.1, 21.7; HRMS (ESI): Exact mass calcd for $C_{18}H_{19}NO_2$ $[M+H]^+$, requires m/z: 282.1494, found m/z: 282.1490; Enantiomeric excess determined by HPLC with Whelk-(*S*) column, 10% *i*-PrOH/hexanes, 1.0 mL/min, 254 nm, t_r (anti major) = 10.6, t_r (anti minor) = 9.2, 99% ee.



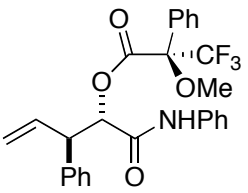
(2*S*,3*R*)-2-hydroxy-*N*-phenyl-3-*β*-napthylpent-4-enamide. The indicated compound was prepared according to General Procedure A as a 99:1 anti/syn mixture of diastereomers. Flash chromatography (10% EtOAc/hexanes) yielded a single diastereomer (anti) as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (89% yield); mp 159 – 161°C; $[\alpha]_D$ –131.0° (*c* = 1.25, $CHCl_3$); IR (film) 3364, 1657, 1632, 1599, 1531, 194, 1444, 1316, 1089, 1001, 927, 836, 754 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (brs, 1H), 7.87 – 7.81 (m, 4H), 7.51 – 7.48 (m, 5H), 7.33 (t, 2H, *J* = 7.7 Hz), 7.13 (t, 1H, *J* = 7.3 Hz), 6.33 (ddd, 1H, *J* = 17.1, 10.1, 8.2 Hz), 5.32 – 5.25 (m, 2H), 4.56 (dd, 1H, *J* = 4.8, 3.3 Hz), 4.27 (dd, 1H, *J* = 8.6, 4.8 Hz), 2.58 (d, 1H, *J* = 4.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 138.2, 137.2, 134.1, 133.7, 132.7, 129.3, 128.8, 128.0, 127.9, 127.1, 126.7, 126.2, 124.9, 120.2, 119.9, 76.1, 53.0; HRMS (ESI): Exact mass calcd for $C_{21}H_{19}NO_2$ $[M+H]^+$, requires m/z: 318.1494, found m/z: 318.1497; Enantiomeric excess determined by HPLC with Whelk-(*S*) column, 10% *i*-PrOH/hexanes, 1.0 mL/min, 225 nm, t_r (anti major) = 27.0, t_r (anti minor) = 21.0, 97% ee.



Mosher Ester Analysis of compound shown. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.581 – 7.560 (m, 2H), 7.455 – 7.399 (m, 3H), 7.291 – 7.201 (m, 5H), 7.126 – 7.083 (m, 1H), 5.582 (dq, 1H, *J* = 17.21, 10.25, 9.15 Hz), 5.578 (d, 1H, *J* = 3.30 Hz), 5.136 – 5.075 (m, 2H), 3.619 (s, 3H), 2.876 – 2.811 (m, 1H), 1.503 – 1.271 (m, 4H), 0.904 (t, 3H, *J* = 6.96 Hz).

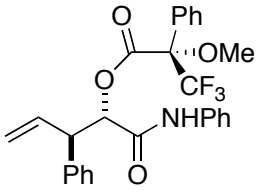


Mosher Ester Analysis of compound shown. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.585 – 7.565 (m, 2H), 7.480 – 7.427 (m, 3H), 7.380 (brs, 1H), 7.326 – 7.280 (m, 4H), 7.253 – 7.105 (m, 1H), 5.592 (d, 1H, *J* = 2.93 Hz), 5.554 (dq, 1H, *J* = 17.21, 10.25, 9.52 Hz), 5.121 – 5.067 (m, 2H), 3.522 (s, 3H), 2.833 – 2.803 (m, 1H), 1.446 – 1.254 (m, 4H), 0.889 (t, 3H, *J* = 6.96 Hz).

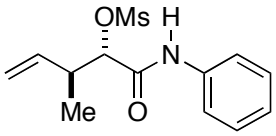


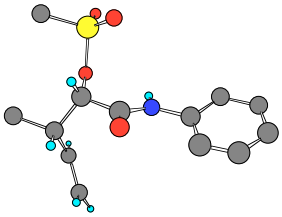
Mosher Ester Analysis of compound shown. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.437 – 7.401 (m, 1H), 7.355 – 7.254 (m, 14H), 7.162 – 7.126 (m, 1H), 6.180 (dt, 1H, *J* = 17.58, 9.52 Hz), 5.956 (d, 1H, *J* =

3.66 Hz), 5.327 (s, 1H), 5.292 (d, 1H, *J* = 4.03 Hz), 4.238 (dd, 1H, *J* = 9.52, 3.66 Hz), 3.378 (s, 3H).

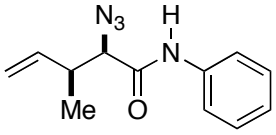


Mosher Ester Analysis of compound shown. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.477 – 7.384 (m, 5H), 7.359 (brs, 1H), 7.319 – 7.244 (m, 9H), 7.169 – 7.126 (m, 1H), 6.151 (dq, 1H, *J* = 17.21, 9.89, 8.79 Hz), 5.800 (d, 1H, *J* = 4.76 Hz), 5.264 (d, 1H, *J* = 4.39 Hz), 5.229 (s, 1H), 4.176 (dd, 1H, *J* = 9.15, 4.76 Hz), 3.404 (s, 3H).



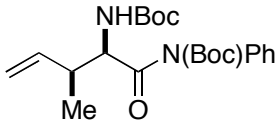


(2*S*,3*S*)-2-methanesulfonylhydroxy-3-methyl-*N*-phenylpent-4-enamide. The indicated compound was prepared according to General Procedure D and isolated as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (98% yield): mp 62 – 63 °C; $[\alpha]_D$ –95.3° (*c* = 1.0, $CHCl_3$); IR (film) 3033, 2977, 2940, 1688, 1668, 1599, 1532, 1501, 1445, 1351, 1181, 963, 924, 874, 849, 756, 689 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (brs, 1H), 7.51 (d, 2H, *J* = 7.7 Hz), 7.34 (t, 2H, *J* = 7.7 Hz), 7.13 (t, 1H, *J* = 7.3 Hz), 5.82 – 5.74 (m, 1H), 5.21-5.24 (m, 2H), 5.01 (d, 1H, *J* = 3.7 Hz), 3.16 (s, 3H), 3.07-3.03 (m, 1H), 1.23 (d, 3H, *J* = 7.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 136.7, 136.2, 129.3, 125.5, 120.7, 118.2, 83.8, 40.6, 38.9, 16.6; HRMS (ESI): Exact mass calcd for $C_{13}H_{17}NO_4S$ $[M+H]^+$, requires *m*/*z*: 284.0956, found *m*/*z*: 284.0966.

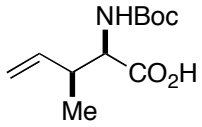


(2*R*,3*S*)-2-azido-3-methyl-*N*-phenylpent-4-enamide (5). The indicated compound was prepared according to General Procedure E and isolated as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (97% yield): mp 42-43 °C; $[\alpha]_D$ –19.3° (*c* = 0.67, $CHCl_3$); IR (film) 3283, 2969, 2104, 1664, 1601, 1529, 1500, 1445, 1261, 1087, 1029, 922, 802, 754, 691 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (brs, 1H), 7.55 (d, 2H, *J* = 7.3 Hz), 7.35 (t, 2H, *J* = 8.3 Hz), 7.16 (t, 1H, *J* = 7.3 Hz), 6.00 – 5.93 (m, 1H), 5.30 – 5.21 (m, 2H), 4.17 (d, 1H, *J* = 3.4 Hz), 3.13 – 3.07 (m, 1H), 1.11 (d, 3H, *J* = 6.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.6, 139.1, 137.0, 129.3, 125.2, 120.3, 117.4, 69.2, 40.1,

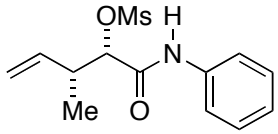
13.5; HRMS (ESI): Exact mass calcd for $C_{12}H_{14}N_4O$ $[M+H]^+$, requires *m*/*z*: 231.1246, found *m*/*z*: 231.3022.



(2*R*,3*S*)-2-(*tert*-butoxy)carbonylamino-3-methyl-*N*-phenyl-*N*-(*tert*-butoxy)carbonylpent-4-enamide (6). The indicated compound was prepared according to General Procedure F and isolated by flash chromatography (2% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (83% yield): $[\alpha]_D$ –31.0° (*c* = 0.43, $CHCl_3$); IR (film) 2975, 2926, 2362, 1739, 1716, 1497, 1369, 1293, 1257, 1152, 1083, 853, 759 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.32 (m, 3H), 7.06 – 7.08 (m, 1H), 5.67 – 5.65 (m, 1H,), 5.20 – 5.18 (d, 1H, *J* = 9.8 Hz), 1.44 (s, 9H), 1.38 (s, 9H), 1.32 – 1.28 (m, 2H), 0.96 (t, 3H, *J* = 7.3 Hz), 0.85 (d, 3H, *J* = 6.8 Hz) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 176.0, 156.1, 152.2, 138.9, 129.2, 128.4, 128.1, 83.8, 79.6, 57.4, 37.9, 28.6, 28.0, 13.7, 12.1; HRMS (ESI): Exact mass calcd for $C_{22}H_{34}N_2O_5$ $[M+H]^+$, requires *m*/*z*: 407.2546, found *m*/*z*: 407.2526.



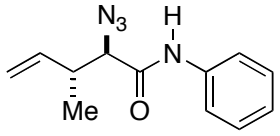
(2*R*,3*S*)-*N*-(*tert*-butoxycarbonyl)-alloisoleucine (7). The indicated compound was prepared according to General Procedure G and was isolated as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (quantitative yield); mp 66 – 67 °C; $[\alpha]_D$ –44.2° (*c* = 0.49, $CHCl_3$), lit.[18] $[\alpha]_D$ –40.7° (*c* = 2.06, $CHCl_3$); IR (film) 3332, 2968, 2928, 1718, 1516, 1400, 1367, 1254, 1163, 1091, 1004, 911, 856, 714 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.94 (d, 1H, *J* = 8.8 Hz), 4.41 – 4.40 (m, 1H), 2.06 – 1.90 (m, 1H), 1.45 (s, 9H), 1.32 – 1.22 (m, 2H), 0.96 (t, 3H, *J* = 7.6 Hz), 0.90 (d, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) 177.5, 156.1, 80.3, 56.9, 37.6, 28.5 26.5, 14.6, 11.9; HRMS (ESI): Exact mass calcd for $C_{11}H_{21}NO_4$ $[M+H]^+$, requires *m*/*z*: 232.1549, found *m*/*z*: 232.1560.



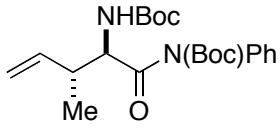
(2*S*,3*R*)-2-methanesulfonyloxyhydroxy-3-methyl-*N*-phenylpent-4-enamide. The indicated compound was prepared

(18) Rinehart, K. L.; Sakai, R.; Kishore, V.; Sullins, D. W.; Li, K. *J. Org. Chem.* **1992**, *57*, 3007. See also Lloyd-Williams, P.; Monerris, P.; Gonzalez, I.; Jou, G.; Giralt, E. *J. Chem. Soc. Perkin Trans. 1*. **1994**, *32*, 1969.

according to General Procedure D and isolated as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (96% yield): mp 99 – 100 °C; $[\alpha]_D$ –81.9° (*c* = 1.1, $CHCl_3$); IR (film) 3323, 2975, 1665, 1599, 1527, 1445, 1349, 1175, 986, 952, 912, 874, 850, 737, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (brs, 1H), 7.55 (d, 2H, *J* = 7.7 Hz), 7.35 (t, 2H, *J* = 7.7 Hz), 7.17 (t, 1H, *J* = 7.3 Hz), 5.95 – 5.87 (m, 1H), 5.28 – 5.22 (m, 2H), 5.10 (d, 1H, *J* = 3.7 Hz), 3.13 (s, 3H), 3.13-3.09 (m, 1H), 1.15 (d, 3H, *J* = 6.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 138.3, 136.8, 129.3, 125.5, 120.5, 117.5, 83.3, 39.7, 39.1, 13.0; HRMS (ESI): Exact mass calcd for $C_{13}H_{17}NO_4S$ $[M+H]^+$, requires *m*/*z*: 284.0956, found *m*/*z*: 284.0964.

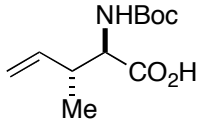


(2*S*,3*R*)-2-azido-3-methyl-*N*-phenylpent-4-enamide. The indicated compound was prepared according to General Procedure E and isolated as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (95% yield): mp 43-44 °C; $[\alpha]_D$ +84.1° (*c* = 0.84, $CHCl_3$); IR (film) 3308, 2965, 2873, 2112, 1679, 1602, 1542, 1453, 1288, 1244, 1125, 1075, 996, 922, 754, 691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (brs, 1H), 7.54 – 7.51 (m, 2H), 7.37 – 7.32 (m, 2H), 7.13 – 7.17 (m, 1H), 5.81 – 5.73 (m, 1H), 5.18 – 5.09 (m, 1H), 4.06 (d, 1H, *J* = 4.0 Hz), 3.06 – 3.01 (m, 1H), 1.27 (d, 3H, *J* = 7.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 137.1, 137.0, 70.4, 41.9, 17.4; HRMS (ESI): Exact mass calcd for $C_{12}H_{14}N_4O$ $[M+H]^+$, requires *m*/*z*: 231.1246, found *m*/*z*: 231.3023.



(2*S*,3*S*)-2-(*tert*-butoxy)carbonylamino-3-methyl-*N*-phenyl-*N*-(*tert*-butoxy)carbonylpent-4-enamide. The indicated compound was prepared according to General Procedure F and isolated by flash chromatography (2% EtOAc/hexanes) as a colorless oil that was homogeneous as judged by ^{1}H NMR spectroscopy (81% yield): $[\alpha]_D$ –22.0° (*c* = 0.15, $CHCl_3$); IR (film) 2975, 2926, 2362, 1739, 1716, 1497, 1369, 1293, 1257, 1152, 1083, 853, 759 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.33 (m, 3H), 7.07 – 7.05 (m, 1H), 5.52 – 5.57 (m, 1H,), 5.17 (d, 1H, *J* = 9.5 Hz), 1.90 (m, 1H), 1.68 (s, 9H), 1.44 (s, 9H), 0.97 – 0.92 (m, 3H), 1.05 (d, 3H, *J* = 6.8 Hz), 0.92 (t, 3H, *J* = 7.5 Hz) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.0, 148.1, 128.8, 128.0, 127.5, 87.0, 85.5, 82.6, 31.8, 28.1, 27.2, 27.4,

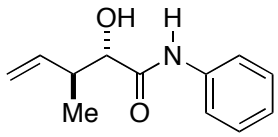
18.3, 11.6; HRMS (ESI): Exact mass calcd for $C_{22}H_{34}N_2O_5$ $[M+H]^+$, requires *m*/*z*: 407.2546, found *m*/*z*: 407.2526.



***N*-(*tert-butoxycarbonyl*)-D-isoleucine (8).** The indicated compound was prepared according to General Procedure G and was isolated as a white solid that was homogeneous as judged by ^{1}H NMR spectroscopy (quantitative yield); mp 63 – 65 °C; $[\alpha]_D$ +10.2° (*c* = 3.35, $CHCl_3$); IR (film) 3317, 2967, 2878, 2360, 1767, 1653, 1506, 1456, 1395, 1368, 1251, 1163, 1089, 1046, 1020, 859, 772 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.00 (d, 1H, *J* = 8.8 Hz), 4.43 – 4.29 (m, 1H), 2.05 – 1.80 (m, 1H), 1.45 (s, 9H), 1.26 – 1.17 (m, 2H), 0.98 (d, 3H, *J* = 6.8 Hz), 0.94 (t, 3H, *J* = 7.6 Hz) ; ^{13}C NMR (100 MHz, $CDCl_3$) 177.3, 156.0, 80.3, 58.1, 38.0, 28.5, 25.1 15.7, 11.8; HRMS (ESI): Exact mass calcd for $C_{11}H_{21}NO_4$ $[M+H]^+$, requires *m*/*z*: 232.1549, found *m*/*z*: 232.1557. [19]

(19) Data matched with that of Boc-L-isoleucine commercially available from Aldrich (CAS # 13139-16-7).

Single X-ray Crystals Data



(2*S*,3*S*)-2-hydroxy-3-methyl-*N*-phenylpent-4-enamide (4)

Table 1. Crystal data and structure refinement for ya1sad.

Identification code	ya1sad	
Empirical formula	C12 H15 N O2	
Formula weight	205.25	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	I4(1)/a	
Unit cell dimensions	a = 18.011(5) Å	a= 90°.
	b = 18.011(5) Å	b= 90°.
	c = 14.279(8) Å	g = 90°.
Volume	4632(3) $Å^3$	
Z	16	
Density (calculated)	1.177 Mg/m^3	
Absorption coefficient	0.080 mm^{-1}	
F(000)	1760	
Crystal size	0.04 x 0.03 x 0.02 mm^3	
Theta range for data collection	1.82 to 24.99°.	
Index ranges	-18<=h<=21, -21<=k<=21, -16<=l<=16	
Reflections collected	12935	
Independent reflections	2044 [R(int) = 0.0330]	
Completeness to theta = 24.99°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2044 / 0 / 196	
Goodness-of-fit on F^2	1.019	
Final R indices [I>2sigma(I)]	R1 = 0.0360, wR2 = 0.0835	
R indices (all data)	R1 = 0.0518, wR2 = 0.0925	

Largest diff. peak and hole	0.116 and -0.117 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ya1sad. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9284(1)	4166(1)	1480(1)	36(1)
C(2)	9479(1)	3778(1)	571(1)	36(1)
C(3)	8783(1)	3682(1)	-35(1)	44(1)
C(4)	8462(1)	4418(1)	-300(1)	52(1)
C(5)	7794(1)	4641(2)	-115(2)	72(1)
C(6)	8952(1)	3210(1)	-897(2)	67(1)
C(7)	9294(1)	5372(1)	2293(1)	36(1)
C(8)	9172(1)	6107(1)	2078(1)	54(1)
C(9)	8990(1)	6603(1)	2777(1)	69(1)
C(10)	8924(1)	6375(1)	3687(1)	62(1)
C(11)	9048(1)	5644(1)	3903(1)	57(1)
C(12)	9238(1)	5141(1)	3213(1)	49(1)
N(1)	9487(1)	4880(1)	1553(1)	36(1)
O(1)	8941(1)	3833(1)	2098(1)	51(1)
O(2)	10031(1)	4158(1)	57(1)	40(1)

Table 3. Bond lengths [Å] and angles [°] for ya1sad.

C(1)-O(1)	1.2331(17)
C(1)-N(1)	1.3396(19)
C(1)-C(2)	1.517(2)
C(2)-O(2)	1.4131(17)
C(2)-C(3)	1.533(2)
C(3)-C(4)	1.496(2)
C(3)-C(6)	1.526(3)
C(4)-C(5)	1.296(3)
C(7)-C(8)	1.376(2)
C(7)-C(12)	1.383(2)
C(7)-N(1)	1.4225(19)
C(8)-C(9)	1.379(3)
C(9)-C(10)	1.367(3)
C(10)-C(11)	1.370(3)
C(11)-C(12)	1.382(2)
O(1)-C(1)-N(1)	123.30(14)
O(1)-C(1)-C(2)	120.22(13)
N(1)-C(1)-C(2)	116.46(12)
O(2)-C(2)-C(1)	112.58(11)
O(2)-C(2)-C(3)	109.70(12)
C(1)-C(2)-C(3)	110.16(12)
C(4)-C(3)-C(6)	111.49(16)
C(4)-C(3)-C(2)	111.06(13)
C(6)-C(3)-C(2)	110.72(15)
C(5)-C(4)-C(3)	125.58(19)
C(8)-C(7)-C(12)	119.33(15)
C(8)-C(7)-N(1)	118.27(14)
C(12)-C(7)-N(1)	122.40(14)
C(7)-C(8)-C(9)	120.01(17)
C(10)-C(9)-C(8)	120.86(19)
C(9)-C(10)-C(11)	119.30(18)
C(10)-C(11)-C(12)	120.60(18)
C(11)-C(12)-C(7)	119.89(17)

C(1)-N(1)-C(7)	126.10(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for ya1sad. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	35(1)	37(1)	36(1)	6(1)	-2(1)	-1(1)
C(2)	41(1)	30(1)	39(1)	6(1)	3(1)	2(1)
C(3)	48(1)	45(1)	40(1)	2(1)	-2(1)	-11(1)
C(4)	47(1)	56(1)	52(1)	14(1)	-14(1)	-7(1)
C(5)	61(1)	81(2)	74(2)	16(1)	-10(1)	10(1)
C(6)	81(2)	70(1)	50(1)	-12(1)	-2(1)	-16(1)
C(7)	32(1)	41(1)	36(1)	-2(1)	1(1)	-2(1)
C(8)	74(1)	47(1)	41(1)	2(1)	7(1)	10(1)
C(9)	102(2)	50(1)	54(1)	-4(1)	10(1)	19(1)
C(10)	72(1)	64(1)	49(1)	-16(1)	6(1)	7(1)
C(11)	66(1)	68(1)	36(1)	-5(1)	2(1)	-8(1)
C(12)	59(1)	46(1)	42(1)	1(1)	-3(1)	-4(1)
N(1)	39(1)	36(1)	34(1)	3(1)	4(1)	-3(1)
O(1)	66(1)	44(1)	41(1)	2(1)	12(1)	-14(1)
O(2)	37(1)	40(1)	42(1)	9(1)	5(1)	4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for ya1sad.

	x	y	z	U(eq)
H(2)	9668(7)	3285(8)	744(10)	30(3)
H(3)	8415(8)	3431(8)	360(11)	42(4)
H(4)	8789(10)	4734(10)	-654(13)	67(6)
H(8)	9195(10)	6247(10)	1442(15)	65(5)
H(9)	8907(11)	7125(12)	2614(15)	83(6)
H(10)	8784(11)	6707(11)	4157(15)	76(6)
H(11)	9016(10)	5473(10)	4530(15)	65(5)
H(12)	9335(9)	4636(10)	3367(12)	56(5)
H(6A)	9297(11)	3458(11)	-1314(16)	77(6)
H(6B)	8493(12)	3109(11)	-1274(17)	86(7)
H(1N)	9674(9)	5060(8)	1060(12)	40(4)
H(5A)	7588(12)	5122(13)	-330(16)	89(7)
H(5B)	7469(14)	4323(13)	212(17)	96(8)
H(6C)	9181(11)	2727(12)	-717(15)	80(7)
H(2O)	10457(11)	3966(10)	235(13)	61(6)

Table 6. Torsion angles [°] for ya1sad.

O(1)-C(1)-C(2)-O(2)	-164.01(13)
N(1)-C(1)-C(2)-O(2)	17.55(17)
O(1)-C(1)-C(2)-C(3)	73.20(17)
N(1)-C(1)-C(2)-C(3)	-105.23(14)
O(2)-C(2)-C(3)-C(4)	-62.10(17)
C(1)-C(2)-C(3)-C(4)	62.36(17)
O(2)-C(2)-C(3)-C(6)	62.34(17)
C(1)-C(2)-C(3)-C(6)	-173.20(15)
C(6)-C(3)-C(4)-C(5)	114.1(2)
C(2)-C(3)-C(4)-C(5)	-121.9(2)
C(12)-C(7)-C(8)-C(9)	-0.4(3)
N(1)-C(7)-C(8)-C(9)	-179.50(17)
C(7)-C(8)-C(9)-C(10)	-0.4(3)
C(8)-C(9)-C(10)-C(11)	0.6(3)
C(9)-C(10)-C(11)-C(12)	0.0(3)
C(10)-C(11)-C(12)-C(7)	-0.9(3)
C(8)-C(7)-C(12)-C(11)	1.1(2)
N(1)-C(7)-C(12)-C(11)	-179.91(15)
O(1)-C(1)-N(1)-C(7)	-5.4(2)
C(2)-C(1)-N(1)-C(7)	172.94(13)
C(8)-C(7)-N(1)-C(1)	-144.91(15)
C(12)-C(7)-N(1)-C(1)	36.0(2)

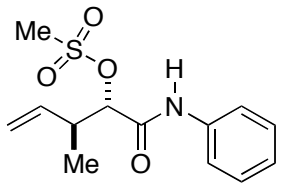
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for ya1sad [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...O(2)#1	0.845(17)	2.192(17)	3.0061(19)	161.6(14)
N(1)-H(1N)...O(2)	0.845(17)	2.260(15)	2.6859(19)	111.4(12)
O(2)-H(2O)...O(1)#2	0.88(2)	1.76(2)	2.6271(16)	170.9(18)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y+1,-z #2 y+3/4,-x+5/4,-z+1/4



(2*S*,3*S*)-2-methanesulfonylhydroxy-3-methyl-*N*-phenylpent-4-enamide.

Table 1. Crystal data and structure refinement for jwu101t.

Identification code	jwu101t	
Empirical formula	C13 H17 N O4 S	
Formula weight	283.34	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 5.3345(9) Å	α = 90°.
	b = 15.737(2) Å	β = 90°.
	c = 17.134(3) Å	γ = 90°.
Volume	1438.4(4) $Å^3$	
Z	4	
Density (calculated)	1.308 Mg/m^3	
Absorption coefficient	0.234 mm^{-1}	
F(000)	600	
Crystal size	0.02 x 0.01 x 0.01 mm^3	
Theta range for data collection	1.76 to 25.00°.	
Index ranges	-6<=h<=6, -17<=k<=18, -20<=l<=16	
Reflections collected	8232	
Independent reflections	2547 [R(int) = 0.0591]	
Completeness to theta = 25.00°	100.0 %	
Absorption correction	None	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2547 / 0 / 240
Goodness-of-fit on F^2	0.801
Final R indices [I>2sigma(I)]	R1 = 0.0394, wR2 = 0.0708
R indices (all data)	R1 = 0.0526, wR2 = 0.0752
Absolute structure parameter	-0.06(9)
Largest diff. peak and hole	0.180 and -0.199 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for jwu101t. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3404(5)	2344(2)	7906(2)	25(1)
C(2)	3091(5)	1701(2)	7252(2)	27(1)
C(3)	3617(7)	2108(2)	6463(2)	40(1)
C(4)	1791(9)	2785(2)	6264(2)	51(1)
C(5)	2336(12)	3595(3)	6178(3)	75(1)
C(6)	3777(13)	1432(3)	5822(3)	75(2)
C(7)	1256(5)	3142(2)	8959(2)	20(1)
C(8)	-784(5)	3064(2)	9456(2)	23(1)
C(9)	-996(6)	3588(2)	10096(2)	27(1)
C(10)	828(6)	4189(2)	10255(2)	31(1)
C(11)	2864(6)	4262(2)	9757(2)	32(1)
C(12)	3080(5)	3745(2)	9107(2)	27(1)
C(13)	1757(6)	-299(2)	7084(2)	38(1)
N(1)	1356(4)	2596(1)	8296(1)	23(1)
O(1)	5540(3)	2603(1)	8027(1)	35(1)
O(2)	583(3)	1325(1)	7225(1)	27(1)
O(3)	-2473(3)	303(1)	7585(1)	42(1)
O(4)	1250(4)	482(1)	8410(1)	38(1)
S(1)	146(1)	446(1)	7657(1)	28(1)

Table 3. Bond lengths [Å] and angles [°] for jwu101t.

C(1)-O(1)	1.228(3)
C(1)-N(1)	1.341(3)
C(1)-C(2)	1.519(4)
C(2)-O(2)	1.464(3)
C(2)-C(3)	1.523(4)
C(3)-C(4)	1.483(5)
C(3)-C(6)	1.531(5)
C(4)-C(5)	1.316(6)
C(7)-C(8)	1.388(4)
C(7)-C(12)	1.383(4)
C(7)-N(1)	1.424(3)
C(8)-C(9)	1.377(4)
C(9)-C(10)	1.383(4)
C(10)-C(11)	1.386(4)
C(11)-C(12)	1.384(4)
C(13)-S(1)	1.754(3)
O(2)-S(1)	1.5864(18)

O(3)-S(1)	1.4202(17)
O(4)-S(1)	1.419(2)
O(1)-C(1)-N(1)	125.0(2)
O(1)-C(1)-C(2)	116.6(2)
N(1)-C(1)-C(2)	118.4(2)
O(2)-C(2)-C(1)	113.2(2)
O(2)-C(2)-C(3)	108.0(2)
C(1)-C(2)-C(3)	110.8(2)
C(4)-C(3)-C(2)	112.6(3)
C(4)-C(3)-C(6)	111.8(4)
C(2)-C(3)-C(6)	110.8(3)
C(5)-C(4)-C(3)	125.2(5)
C(8)-C(7)-C(12)	120.0(2)
C(8)-C(7)-N(1)	117.7(2)
C(12)-C(7)-N(1)	122.3(2)
C(9)-C(8)-C(7)	120.0(3)
C(8)-C(9)-C(10)	120.6(3)
C(9)-C(10)-C(11)	119.2(3)
C(12)-C(11)-C(10)	120.8(3)
C(11)-C(12)-C(7)	119.5(3)
C(1)-N(1)-C(7)	127.3(2)
C(2)-O(2)-S(1)	118.15(15)
O(4)-S(1)-O(3)	119.56(13)
O(4)-S(1)-O(2)	109.19(10)
O(3)-S(1)-O(2)	104.04(10)
O(4)-S(1)-C(13)	109.34(15)
O(3)-S(1)-C(13)	109.12(14)
O(2)-S(1)-C(13)	104.49(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for jwu101t. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	23(2)	26(1)	25(2)	3(1)	-2(1)	3(1)
C(2)	19(1)	28(1)	34(2)	-4(1)	1(1)	3(1)
C(3)	46(2)	44(2)	30(2)	-4(2)	11(2)	-7(2)
C(4)	72(3)	51(2)	30(2)	6(2)	-2(2)	0(2)
C(5)	120(5)	51(3)	53(3)	-2(2)	-8(3)	11(3)
C(6)	120(5)	67(3)	37(2)	-15(2)	26(3)	-4(3)
C(7)	18(1)	19(1)	23(2)	2(1)	-4(1)	2(1)
C(8)	21(1)	20(1)	27(2)	3(1)	-6(1)	-4(1)
C(9)	27(2)	29(2)	25(2)	-1(1)	4(1)	3(1)

C(10)	35(2)	29(2)	30(2)	-7(1)	-2(1)	2(1)
C(11)	31(2)	23(2)	42(2)	-5(1)	-2(2)	-6(1)
C(12)	23(2)	29(2)	30(2)	1(1)	6(1)	-2(1)
C(13)	27(2)	31(2)	57(3)	-15(2)	-1(2)	1(1)
N(1)	16(1)	26(1)	26(1)	-3(1)	-2(1)	-3(1)
O(1)	19(1)	45(1)	41(1)	-13(1)	1(1)	-3(1)
O(2)	21(1)	27(1)	34(1)	-8(1)	-2(1)	2(1)
O(3)	22(1)	35(1)	68(2)	-7(1)	2(1)	0(1)
O(4)	45(1)	34(1)	34(1)	-1(1)	-4(1)	-4(1)
S(1)	21(1)	26(1)	38(1)	-8(1)	-2(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for jwu101t.

	x	y	z	U(eq)
H(3)	4290(40)	1283(13)	7360(14)	13(6)
H(4)	180(70)	2580(20)	6160(20)	67(12)
H(8)	-1930(50)	2630(16)	9372(16)	31(8)
H(9)	-2420(50)	3529(15)	10442(16)	27(8)
H(10)	610(40)	4535(15)	10709(14)	21(6)
H(11)	4160(50)	4646(17)	9859(15)	37(8)
H(12)	4420(50)	3791(15)	8762(14)	25(7)
H(5A)	4220(80)	3720(20)	6280(20)	79(14)
H(6A)	2030(70)	1250(20)	5700(20)	58(12)
H(13A)	1110(60)	-253(16)	6533(18)	42(9)
H(5B)	1100(90)	4050(30)	6080(20)	95(15)
H(6B)	4290(70)	1690(20)	5330(20)	75(12)
H(13B)	1470(60)	-830(18)	7346(18)	52(9)
H(3C)	5220(60)	2362(17)	6486(17)	36(8)
H(6C)	4880(90)	970(30)	5940(30)	112(19)
H(13C)	3520(60)	-189(17)	7121(16)	45(9)
H(1N)	10(50)	2371(15)	8174(15)	22(7)

Table 6. Torsion angles [°] for jwu101t.

O(1)-C(1)-C(2)-O(2)	171.0(2)
N(1)-C(1)-C(2)-O(2)	-10.1(3)
O(1)-C(1)-C(2)-C(3)	-67.5(3)
N(1)-C(1)-C(2)-C(3)	111.4(3)
O(2)-C(2)-C(3)-C(4)	60.2(3)
C(1)-C(2)-C(3)-C(4)	-64.4(4)
O(2)-C(2)-C(3)-C(6)	-65.9(4)

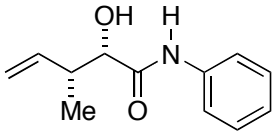
C(1)-C(2)-C(3)-C(6)	169.6(4)
C(2)-C(3)-C(4)-C(5)	115.6(4)
C(6)-C(3)-C(4)-C(5)	-118.8(5)
C(12)-C(7)-C(8)-C(9)	0.1(4)
N(1)-C(7)-C(8)-C(9)	-178.7(2)
C(7)-C(8)-C(9)-C(10)	-0.8(4)
C(8)-C(9)-C(10)-C(11)	0.6(4)
C(9)-C(10)-C(11)-C(12)	0.1(4)
C(10)-C(11)-C(12)-C(7)	-0.8(4)
C(8)-C(7)-C(12)-C(11)	0.6(4)
N(1)-C(7)-C(12)-C(11)	179.4(3)
O(1)-C(1)-N(1)-C(7)	-6.0(4)
C(2)-C(1)-N(1)-C(7)	175.2(2)
C(8)-C(7)-N(1)-C(1)	-154.8(3)
C(12)-C(7)-N(1)-C(1)	26.4(4)
C(1)-C(2)-O(2)-S(1)	-94.8(2)
C(3)-C(2)-O(2)-S(1)	142.18(18)
C(2)-O(2)-S(1)-O(4)	45.9(2)
C(2)-O(2)-S(1)-O(3)	174.56(18)
C(2)-O(2)-S(1)-C(13)	-71.0(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for jwu101t [Å and º].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...O(2)	0.83(3)	2.33(3)	2.746(3)	111(2)
N(1)-H(1N)...O(1)#1	0.83(3)	2.42(3)	3.137(3)	145(2)

Symmetry transformations used to generate equivalent atoms:
#1 x-1,y,z



(2*S*,3*R*)-2-hydroxy-3-methyl-*N*-phenylpent-4-enamide (10).

Table 1. Crystal data and structure refinement for jwu100sad.

Identification code	jwu100sad	
Empirical formula	C12 H15 N O2	
Formula weight	205.25	
Temperature	193(2) K	
Wavelength	0.71073 ≈	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 24.869(14) ≈	α= 90∞.
	b = 11.816(7) ≈	β= 103.348(11)∞.
	c = 8.085(5) ≈	γ = 90∞.
Volume	2312(2) ≈^{3}	
Z	8	
Density (calculated)	1.180 Mg/m^3	
Absorption coefficient	0.080 mm^{-1}	
F(000)	880	
Crystal size	0.03 x 0.02 x 0.01 mm^3	
Theta range for data collection	1.92 to 25.00∞.	
Index ranges	-29<=h<=29, -14<=k<=13, -9<=l<=8	
Reflections collected	6332	
Independent reflections	2031 [R(int) = 0.0216]	
Completeness to theta = 25.00∞	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2031 / 0 / 196	
Goodness-of-fit on F^2	1.069	
Final R indices [I>2sigma(I)]	R1 = 0.0355, wR2 = 0.0887	
R indices (all data)	R1 = 0.0433, wR2 = 0.0937	
Largest diff. peak and hole	0.168 and -0.136 e.≈$^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for jwu100sad. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2649(1)	4191(1)	2741(2)	32(1)
C(2)	2083(1)	4117(1)	1525(2)	31(1)
C(3)	1672(1)	3499(1)	2374(2)	41(1)
C(4)	1089(1)	3684(2)	1407(2)	54(1)
C(5)	714(1)	4232(2)	1972(3)	73(1)
C(6)	1811(1)	2244(2)	2598(3)	62(1)
C(7)	3607(1)	3472(1)	3334(2)	36(1)
C(8)	3903(1)	2508(2)	3150(2)	56(1)
C(9)	4449(1)	2411(2)	4021(3)	73(1)
C(10)	4703(1)	3258(2)	5078(2)	63(1)
C(11)	4408(1)	4207(2)	5271(2)	53(1)
C(12)	3864(1)	4329(1)	4401(2)	45(1)
N(1)	3050(1)	3537(1)	2399(1)	34(1)
O(1)	2711(1)	4824(1)	3983(1)	43(1)
O(2)	2095(1)	3583(1)	-35(1)	35(1)

Table 3. Bond lengths [≈] and angles [∞] for jwu100sad.

C(1)-O(1)	1.2330(15)
C(1)-N(1)	1.3395(17)
C(1)-C(2)	1.5212(19)
C(2)-O(2)	1.4171(16)
C(2)-C(3)	1.5393(19)
C(3)-C(4)	1.497(2)
C(3)-C(6)	1.524(2)
C(4)-C(5)	1.300(3)
C(7)-C(8)	1.382(2)
C(7)-C(12)	1.387(2)
C(7)-N(1)	1.4204(18)
C(8)-C(9)	1.383(2)
C(9)-C(10)	1.371(3)
C(10)-C(11)	1.369(3)
C(11)-C(12)	1.381(2)
O(1)-C(1)-N(1)	123.90(12)
O(1)-C(1)-C(2)	119.00(11)
N(1)-C(1)-C(2)	117.09(11)
O(2)-C(2)-C(1)	113.21(10)
O(2)-C(2)-C(3)	109.74(11)
C(1)-C(2)-C(3)	110.43(11)
C(4)-C(3)-C(6)	111.75(14)
C(4)-C(3)-C(2)	111.14(12)
C(6)-C(3)-C(2)	111.01(13)
C(5)-C(4)-C(3)	125.14(18)
C(8)-C(7)-C(12)	119.26(14)
C(8)-C(7)-N(1)	117.78(13)
C(12)-C(7)-N(1)	122.97(12)
C(7)-C(8)-C(9)	119.85(16)
C(10)-C(9)-C(8)	120.89(17)
C(11)-C(10)-C(9)	119.19(17)
C(10)-C(11)-C(12)	121.00(16)
C(11)-C(12)-C(7)	119.80(15)

C(1)-N(1)-C(7)	127.13(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for jwu100sad. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + \ldots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	36(1)	30(1)	31(1)	1(1)	12(1)	0(1)
C(2)	35(1)	29(1)	31(1)	-1(1)	10(1)	1(1)
C(3)	42(1)	47(1)	37(1)	-4(1)	17(1)	-8(1)
C(4)	43(1)	72(1)	51(1)	-9(1)	16(1)	-17(1)
C(5)	40(1)	93(2)	87(1)	-12(1)	17(1)	-5(1)
C(6)	79(1)	47(1)	70(1)	11(1)	36(1)	-11(1)
C(7)	35(1)	43(1)	30(1)	5(1)	9(1)	2(1)
C(8)	47(1)	61(1)	53(1)	-15(1)	0(1)	15(1)
C(9)	52(1)	76(1)	79(1)	-17(1)	-7(1)	25(1)
C(10)	38(1)	77(1)	66(1)	1(1)	-5(1)	7(1)
C(11)	42(1)	54(1)	59(1)	0(1)	3(1)	-9(1)
C(12)	38(1)	40(1)	56(1)	1(1)	9(1)	-2(1)
N(1)	35(1)	37(1)	30(1)	-4(1)	7(1)	3(1)
O(1)	40(1)	46(1)	41(1)	-13(1)	7(1)	4(1)
O(2)	40(1)	37(1)	30(1)	-2(1)	11(1)	-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for jwu100sad.

	x	y	z	U(eq)
H(1)	2955(6)	3108(13)	1548(19)	39(4)
H(2)	2263(7)	4062(15)	-570(20)	56(5)
H(3)	1707(6)	3850(12)	3490(20)	41(4)
H(4)	994(8)	3330(16)	320(30)	74(6)
H(2A)	1965(5)	4890(12)	1334(16)	32(3)
H(5A)	805(9)	4556(19)	3120(30)	90(7)
H(6A)	1545(8)	1865(16)	3150(20)	73(5)
H(5B)	333(9)	4326(17)	1280(30)	84(6)
H(6B)	1780(8)	1891(17)	1460(30)	78(6)
H(6C)	2186(10)	2123(19)	3300(30)	91(7)
H(12)	3664(7)	4957(15)	4550(20)	52(4)
H(8)	3724(8)	1907(16)	2430(20)	68(5)
H(11)	4578(7)	4797(17)	6000(20)	70(5)
H(10)	5072(9)	3174(16)	5660(20)	72(5)
H(9)	4637(9)	1696(19)	3910(30)	93(7)

Table 6. Torsion angles [∞] for jwu100sad.

O(1)-C(1)-C(2)-O(2)	165.89(11)
N(1)-C(1)-C(2)-O(2)	-15.21(15)
O(1)-C(1)-C(2)-C(3)	-70.62(15)
N(1)-C(1)-C(2)-C(3)	108.28(13)
O(2)-C(2)-C(3)-C(4)	-69.70(15)
C(1)-C(2)-C(3)-C(4)	164.82(12)
O(2)-C(2)-C(3)-C(6)	55.34(17)
C(1)-C(2)-C(3)-C(6)	-70.14(16)
C(6)-C(3)-C(4)-C(5)	121.0(2)
C(2)-C(3)-C(4)-C(5)	-114.4(2)
C(12)-C(7)-C(8)-C(9)	-0.2(3)
N(1)-C(7)-C(8)-C(9)	179.33(16)
C(7)-C(8)-C(9)-C(10)	0.3(3)
C(8)-C(9)-C(10)-C(11)	0.2(3)
C(9)-C(10)-C(11)-C(12)	-1.0(3)
C(10)-C(11)-C(12)-C(7)	1.1(2)
C(8)-C(7)-C(12)-C(11)	-0.5(2)
N(1)-C(7)-C(12)-C(11)	180.00(13)
O(1)-C(1)-N(1)-C(7)	-1.9(2)
C(2)-C(1)-N(1)-C(7)	179.29(11)
C(8)-C(7)-N(1)-C(1)	158.40(14)
C(12)-C(7)-N(1)-C(1)	-22.1(2)

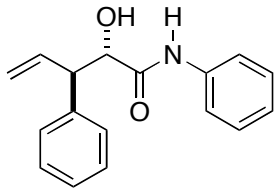
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for jwu100sad [≈ and ∞].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(2)#1	0.843(16)	2.331(16)	3.1208(19)	156.2(13)
O(2)-H(2)...O(1)#2	0.873(18)	1.816(19)	2.6620(16)	162.6(16)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2,-y+1/2,-z #2 x,-y+1,z-1/2



(2*S*,3*R*)-2-hydroxy-*N*,3-diphenylpent-4-enamide

Table 1. Crystal data and structure refinement for sad.

Identification code	sad	
Empirical formula	C17 H17 N O2	
Formula weight	267.32	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.4101(17) Å	α = 90°.
	b = 8.6372(18) Å	β = 90°.
	c = 20.288(4) Å	γ = 90°.
Volume	1473.7(5) $Å^3$	
Z	4	
Density (calculated)	1.205 Mg/m^3	
Absorption coefficient	0.079 mm^{-1}	
F(000)	568	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.01 to 22.49°.	
Index ranges	-9<=h<=7, -9<=k<=8, -21<=l<=21	
Reflections collected	6355	

Independent reflections	1922 [R(int) = 0.0408]
Completeness to theta = 22.49°	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1922 / 0 / 187
Goodness-of-fit on F^2	1.399
Final R indices [I>2sigma(I)]	R1 = 0.0954, wR2 = 0.1691
R indices (all data)	R1 = 0.0992, wR2 = 0.1703
Absolute structure parameter	0(5)
Largest diff. peak and hole	0.266 and -0.190 e.$Å^{-3}$

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

	x	y	z	U(eq)
C(1)	2757(7)	6820(7)	172(3)	29(1)
C(2)	3333(7)	7853(7)	735(2)	28(1)
C(3)	1945(7)	8397(6)	1176(3)	35(2)
C(4)	2353(6)	9828(6)	1564(3)	28(1)
C(5)	3378(7)	9799(7)	2099(3)	37(2)
C(6)	3723(8)	11127(9)	2446(3)	48(2)
C(7)	3087(9)	12526(9)	2267(3)	57(2)
C(8)	2095(9)	12570(7)	1730(4)	59(2)
C(9)	1722(8)	11241(8)	1381(3)	50(2)
C(10)	1348(8)	7108(7)	1593(3)	47(2)
C(11)	7(11)	6614(10)	1676(5)	112(4)
C(12)	2951(7)	4127(7)	-259(3)	36(2)
C(13)	3565(10)	2685(8)	-91(3)	58(2)
C(14)	3339(13)	1435(9)	-503(3)	81(3)
C(15)	2509(10)	1652(9)	-1078(4)	67(2)
C(16)	1934(10)	3060(9)	-1262(4)	70(2)
C(17)	2171(9)	4308(8)	-845(3)	55(2)
N(1)	3252(6)	5368(5)	191(2)	31(1)
O(1)	1899(6)	7397(5)	-253(2)	54(1)
O(2)	4525(4)	7139(5)	1123(2)	37(1)

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

C(1)-O(1)	1.229(6)
C(1)-N(1)	1.322(7)
C(1)-C(2)	1.529(8)
C(2)-O(2)	1.415(6)
C(2)-C(3)	1.544(8)
C(3)-C(10)	1.486(8)
C(3)-C(4)	1.505(8)
C(4)-C(9)	1.381(8)
C(4)-C(5)	1.387(8)
C(5)-C(6)	1.377(9)
C(6)-C(7)	1.371(10)
C(7)-C(8)	1.372(10)
C(8)-C(9)	1.385(9)
C(10)-C(11)	1.217(10)
C(12)-C(17)	1.368(9)
C(12)-C(13)	1.391(9)

C(12)-N(1)	1.429(7)
C(13)-C(14)	1.379(10)
C(14)-C(15)	1.371(11)
C(15)-C(16)	1.361(10)
C(16)-C(17)	1.385(9)

O(1)-C(1)-N(1)	126.2(6)
O(1)-C(1)-C(2)	118.2(5)
N(1)-C(1)-C(2)	115.6(5)
O(2)-C(2)-C(1)	112.7(5)
O(2)-C(2)-C(3)	110.3(4)
C(1)-C(2)-C(3)	111.8(5)
C(10)-C(3)-C(4)	113.2(5)
C(10)-C(3)-C(2)	111.0(5)
C(4)-C(3)-C(2)	112.3(5)
C(9)-C(4)-C(5)	117.8(6)
C(9)-C(4)-C(3)	119.8(5)
C(5)-C(4)-C(3)	122.4(5)
C(6)-C(5)-C(4)	121.1(6)
C(7)-C(6)-C(5)	121.1(6)
C(6)-C(7)-C(8)	118.2(6)
C(7)-C(8)-C(9)	121.4(7)
C(4)-C(9)-C(8)	120.5(6)
C(11)-C(10)-C(3)	130.9(8)
C(17)-C(12)-C(13)	119.5(6)
C(17)-C(12)-N(1)	123.6(6)
C(13)-C(12)-N(1)	116.7(6)
C(14)-C(13)-C(12)	120.1(7)
C(15)-C(14)-C(13)	118.6(8)
C(16)-C(15)-C(14)	122.5(7)
C(15)-C(16)-C(17)	118.5(7)
C(12)-C(17)-C(16)	120.7(7)
C(1)-N(1)-C(12)	129.6(5)

Symmetry transformations used to generate equivalent atoms:

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	29(3)	38(4)	22(3)	9(3)	-2(3)	4(3)
C(2)	36(4)	32(3)	16(3)	12(3)	-6(3)	4(3)
C(3)	19(3)	25(3)	59(4)	6(3)	3(3)	7(3)
C(4)	28(3)	28(3)	30(3)	6(3)	8(3)	-11(3)
C(5)	34(4)	41(4)	36(4)	0(3)	9(3)	4(3)

C(6)	43(4)	64(5)	38(4)	-6(4)	-2(3)	-22(4)
C(7)	56(5)	58(5)	57(4)	-24(4)	5(4)	-25(5)
C(8)	62(5)	24(4)	89(6)	-23(4)	4(5)	8(4)
C(9)	50(4)	43(4)	57(4)	4(4)	-10(4)	0(4)
C(10)	31(4)	37(4)	72(5)	5(4)	17(4)	0(3)
C(11)	85(7)	63(6)	188(11)	32(7)	81(8)	18(5)
C(12)	35(4)	41(4)	31(3)	-14(3)	14(3)	7(3)
C(13)	97(6)	47(5)	31(4)	-4(3)	4(4)	0(5)
C(14)	164(10)	34(4)	46(5)	-1(4)	8(6)	-2(6)
C(15)	92(7)	60(6)	50(5)	-18(4)	3(5)	-19(5)
C(16)	73(6)	65(5)	72(5)	-26(5)	-22(5)	17(5)
C(17)	67(5)	49(4)	50(5)	-8(4)	-25(4)	15(4)
N(1)	29(3)	36(3)	26(3)	0(3)	-7(3)	5(2)
O(1)	64(3)	43(3)	54(3)	-9(3)	-27(3)	14(3)
O(2)	27(2)	54(3)	28(2)	-5(2)	-1(2)	-4(2)

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

	x	y	z	U(eq)
H(2A)	3806	8779	536	34
H(3)	1074	8682	879	41
H(5)	3839	8866	2226	44
H(6)	4399	11074	2808	58
H(7)	3322	13421	2502	68
H(8)	1663	13512	1598	70
H(9)	1043	11300	1020	60
H(10)	2130	6592	1831	56
H(11A)	-845	7066	1457	134
H(11B)	-164	5784	1960	134
H(13)	4128	2563	300	70
H(14)	3741	465	-394	98
H(15)	2332	806	-1351	81
H(16)	1395	3180	-1659	84
H(17)	1796	5281	-966	66
H(1)	3920(50)	5110(50)	540(20)	4(12)
H(2)	5383	7201	932	55

Table 6. Torsion angles [°] for sad.

O(1)-C(1)-C(2)-O(2)	-169.5(5)
N(1)-C(1)-C(2)-O(2)	10.1(7)
O(1)-C(1)-C(2)-C(3)	65.7(7)

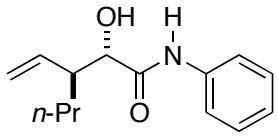
N(1)-C(1)-C(2)-C(3)	-114.7(5)
O(2)-C(2)-C(3)-C(10)	-53.0(7)
C(1)-C(2)-C(3)-C(10)	73.2(6)
O(2)-C(2)-C(3)-C(4)	74.9(6)
C(1)-C(2)-C(3)-C(4)	-158.9(5)
C(10)-C(3)-C(4)-C(9)	-127.8(6)
C(2)-C(3)-C(4)-C(9)	105.5(6)
C(10)-C(3)-C(4)-C(5)	53.5(7)
C(2)-C(3)-C(4)-C(5)	-73.2(7)
C(9)-C(4)-C(5)-C(6)	1.6(8)
C(3)-C(4)-C(5)-C(6)	-179.7(6)
C(4)-C(5)-C(6)-C(7)	-1.1(10)
C(5)-C(6)-C(7)-C(8)	0.0(11)
C(6)-C(7)-C(8)-C(9)	0.8(12)
C(5)-C(4)-C(9)-C(8)	-0.8(9)
C(3)-C(4)-C(9)-C(8)	-179.6(6)
C(7)-C(8)-C(9)-C(4)	-0.3(11)
C(4)-C(3)-C(10)-C(11)	104.1(10)
C(2)-C(3)-C(10)-C(11)	-128.5(9)
C(17)-C(12)-C(13)-C(14)	-2.2(12)
N(1)-C(12)-C(13)-C(14)	-179.3(7)
C(12)-C(13)-C(14)-C(15)	0.3(13)
C(13)-C(14)-C(15)-C(16)	1.6(14)
C(14)-C(15)-C(16)-C(17)	-1.4(14)
C(13)-C(12)-C(17)-C(16)	2.4(11)
N(1)-C(12)-C(17)-C(16)	179.3(7)
C(15)-C(16)-C(17)-C(12)	-0.6(13)
O(1)-C(1)-N(1)-C(12)	0.5(10)
C(2)-C(1)-N(1)-C(12)	-179.1(5)
C(17)-C(12)-N(1)-C(1)	6.5(10)
C(13)-C(12)-N(1)-C(1)	-176.5(6)

Symmetry transformations used to generate equivalent atoms:

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(2)...O(1)#1	0.82	1.91	2.695(6)	159.9
N(1)-H(1)...O(2)	0.94(5)	2.17(4)	2.658(6)	111(3)

Symmetry transformations used to generate equivalent atoms:
#1 x+1/2,-y+3/2,-z



(2*S*,3*R*)-2-hydroxy-*N*,3-diphenylpent-4-enamide

Table 1. Crystal data and structure refinement for jwu85sad.

Identification code	jwu85sad	
Empirical formula	C14 H19 N O2	
Formula weight	233.30	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 8.523(2) Å	α = 90°.
	b = 9.314(2) Å	β = 97.879(5)°.
	c = 17.357(4) Å	γ = 90°.
Volume	1364.8(6) $Å^3$	
Z	4	
Density (calculated)	1.135 Mg/m^3	
Absorption coefficient	0.075 mm^{-1}	
F(000)	504	
Crystal size	0.08 x 0.03 x 0.02 mm^3	
Theta range for data collection	1.18 to 25.00°.	
Index ranges	-10<=h<=6, -10<=k<=11, -19<=l<=20	
Reflections collected	7705	
Independent reflections	4670 [R(int) = 0.0251]	
Completeness to theta = 25.00°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4670 / 1 / 319	
Goodness-of-fit on F^2	1.047	
Final R indices [I>2sigma(I)]	R1 = 0.0473, wR2 = 0.0957	
R indices (all data)	R1 = 0.0641, wR2 = 0.1065	
Absolute structure parameter	-0.5(12)	
Largest diff. peak and hole	0.147 and -0.136 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for jwu85sad. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3112(3)	656(3)	2489(2)	43(1)
C(2)	1955(3)	155(3)	3023(2)	45(1)
C(3)	1351(3)	1425(3)	3465(2)	45(1)
C(4)	2690(3)	2065(3)	4010(2)	49(1)
C(5)	3081(4)	3428(3)	4064(2)	62(1)
C(6)	-29(3)	980(3)	3886(2)	55(1)
C(7)	-958(3)	2212(4)	4164(2)	67(1)
C(8)	-2332(4)	1741(5)	4572(2)	92(1)
C(9)	5826(3)	250(3)	2190(1)	43(1)
C(10)	6819(3)	-887(3)	2103(2)	50(1)
C(11)	8105(3)	-722(4)	1711(2)	60(1)
C(12)	8426(3)	591(4)	1404(2)	57(1)
C(13)	7434(3)	1727(3)	1485(2)	61(1)
C(14)	6143(3)	1574(3)	1876(2)	60(1)
C(15)	1134(3)	5682(3)	2395(2)	42(1)
C(16)	1991(3)	4866(3)	1819(1)	42(1)
C(17)	2653(3)	5918(3)	1264(1)	47(1)
C(18)	1355(3)	6616(3)	726(2)	56(1)
C(19)	1126(4)	8006(3)	638(2)	68(1)
C(20)	3867(3)	5190(3)	820(2)	58(1)
C(21)	4920(4)	6247(4)	467(2)	69(1)
C(22)	6176(4)	5548(4)	62(2)	92(1)
C(23)	-1462(3)	5789(3)	2909(1)	41(1)
C(24)	-1066(3)	6753(3)	3513(2)	53(1)
C(25)	-2220(3)	7202(4)	3949(2)	62(1)
C(26)	-3742(3)	6695(4)	3798(2)	64(1)
C(27)	-4117(3)	5736(4)	3203(2)	66(1)
C(28)	-2993(3)	5294(3)	2760(2)	56(1)
N(1)	4541(3)	35(2)	2611(1)	44(1)
N(2)	-356(3)	5282(2)	2433(1)	47(1)
O(1)	2730(2)	1549(2)	1973(1)	55(1)
O(2)	2633(2)	-893(2)	3563(1)	52(1)
O(3)	1844(2)	6613(2)	2806(1)	55(1)
O(4)	977(2)	3824(2)	1406(1)	54(1)

Table 3. Bond lengths [Å] and angles [°] for jwu85sad.

C(1)-O(1)	1.233(3)

C(1)-N(1)	1.338(3)
C(1)-C(2)	1.517(4)
C(2)-O(2)	1.421(3)
C(2)-C(3)	1.537(4)
C(3)-C(4)	1.502(4)
C(3)-C(6)	1.525(3)
C(4)-C(5)	1.312(4)
C(6)-C(7)	1.510(4)
C(7)-C(8)	1.515(4)
C(9)-C(10)	1.377(4)
C(9)-C(14)	1.389(4)
C(9)-N(1)	1.411(3)
C(10)-C(11)	1.376(4)
C(11)-C(12)	1.376(4)
C(12)-C(13)	1.374(4)
C(13)-C(14)	1.377(4)
C(15)-O(3)	1.229(3)
C(15)-N(2)	1.334(3)
C(15)-C(16)	1.520(4)
C(16)-O(4)	1.425(3)
C(16)-C(17)	1.536(4)
C(17)-C(18)	1.495(4)
C(17)-C(20)	1.530(4)
C(18)-C(19)	1.315(4)
C(20)-C(21)	1.516(4)
C(21)-C(22)	1.507(4)
C(23)-C(28)	1.375(4)
C(23)-C(24)	1.387(4)
C(23)-N(2)	1.416(3)
C(24)-C(25)	1.384(4)
C(25)-C(26)	1.372(4)
C(26)-C(27)	1.369(4)
C(27)-C(28)	1.372(4)

O(1)-C(1)-N(1)	123.4(3)
O(1)-C(1)-C(2)	121.5(2)
N(1)-C(1)-C(2)	115.1(2)
O(2)-C(2)-C(1)	112.0(2)
O(2)-C(2)-C(3)	109.43(19)
C(1)-C(2)-C(3)	111.0(2)
C(4)-C(3)-C(6)	112.1(2)
C(4)-C(3)-C(2)	110.1(2)
C(6)-C(3)-C(2)	111.3(2)
C(5)-C(4)-C(3)	126.3(3)
C(7)-C(6)-C(3)	114.8(2)
C(6)-C(7)-C(8)	113.7(3)

C(10)-C(9)-C(14)	118.9(3)
C(10)-C(9)-N(1)	118.7(2)
C(14)-C(9)-N(1)	122.4(2)
C(11)-C(10)-C(9)	120.8(3)
C(10)-C(11)-C(12)	120.3(3)
C(13)-C(12)-C(11)	119.2(3)
C(12)-C(13)-C(14)	121.0(3)
C(13)-C(14)-C(9)	119.8(3)
O(3)-C(15)-N(2)	124.4(3)
O(3)-C(15)-C(16)	119.5(2)
N(2)-C(15)-C(16)	116.1(2)
O(4)-C(16)-C(15)	111.0(2)
O(4)-C(16)-C(17)	111.6(2)
C(15)-C(16)-C(17)	110.2(2)
C(18)-C(17)-C(20)	111.8(2)
C(18)-C(17)-C(16)	111.4(2)
C(20)-C(17)-C(16)	111.4(2)
C(19)-C(18)-C(17)	125.9(3)
C(21)-C(20)-C(17)	113.2(2)
C(22)-C(21)-C(20)	113.9(3)
C(28)-C(23)-C(24)	119.1(3)
C(28)-C(23)-N(2)	117.9(3)
C(24)-C(23)-N(2)	123.0(2)
C(25)-C(24)-C(23)	119.4(2)
C(26)-C(25)-C(24)	121.0(3)
C(27)-C(26)-C(25)	119.0(3)
C(26)-C(27)-C(28)	120.7(3)
C(27)-C(28)-C(23)	120.8(3)
C(1)-N(1)-C(9)	127.6(2)
C(15)-N(2)-C(23)	130.6(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for jwu85sad. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	49(2)	31(1)	47(2)	-10(1)	4(1)	1(1)
C(2)	47(2)	35(2)	51(2)	-2(1)	3(1)	-4(1)
C(3)	48(2)	37(1)	51(2)	-1(1)	10(1)	2(1)
C(4)	57(2)	43(2)	50(2)	-3(1)	14(1)	2(1)
C(5)	80(2)	51(2)	57(2)	-6(1)	18(2)	-17(2)
C(6)	52(2)	53(2)	62(2)	3(2)	14(1)	-4(1)

C(7)	56(2)	71(2)	78(2)	5(2)	20(2)	12(2)
C(8)	65(2)	113(3)	104(3)	-10(3)	36(2)	2(2)
C(9)	48(2)	36(1)	43(1)	-2(1)	2(1)	2(1)
C(10)	54(2)	42(2)	53(2)	5(1)	10(1)	8(1)
C(11)	58(2)	60(2)	65(2)	6(2)	13(2)	19(2)
C(12)	50(2)	68(2)	52(2)	-1(2)	10(1)	-3(2)
C(13)	59(2)	45(2)	79(2)	6(2)	13(2)	-6(2)
C(14)	54(2)	34(2)	95(2)	-4(2)	21(2)	-1(1)
C(15)	46(2)	31(1)	50(2)	4(1)	1(1)	4(1)
C(16)	43(1)	31(1)	51(2)	0(1)	0(1)	3(1)
C(17)	52(2)	38(2)	50(2)	-1(1)	7(1)	3(1)
C(18)	63(2)	52(2)	55(2)	5(2)	13(1)	11(2)
C(19)	80(2)	54(2)	75(2)	17(2)	31(2)	22(2)
C(20)	62(2)	52(2)	59(2)	3(2)	10(2)	12(2)
C(21)	72(2)	67(2)	72(2)	1(2)	23(2)	7(2)
C(22)	91(3)	90(3)	105(3)	17(2)	47(2)	25(2)
C(23)	39(1)	39(1)	46(1)	12(1)	5(1)	0(1)
C(24)	49(2)	63(2)	48(2)	-1(2)	8(1)	-15(2)
C(25)	59(2)	81(2)	50(2)	-6(2)	17(1)	-15(2)
C(26)	53(2)	87(2)	54(2)	9(2)	19(1)	-3(2)
C(27)	44(2)	80(2)	75(2)	6(2)	9(2)	-14(2)
C(28)	52(2)	48(2)	68(2)	-4(2)	1(2)	-9(1)
N(1)	54(2)	31(1)	49(1)	3(1)	9(1)	3(1)
N(2)	46(1)	37(1)	58(2)	-8(1)	2(1)	-5(1)
O(1)	60(1)	48(1)	57(1)	9(1)	10(1)	12(1)
O(2)	64(1)	31(1)	61(1)	1(1)	9(1)	-4(1)
O(3)	53(1)	46(1)	67(1)	-18(1)	14(1)	-11(1)
O(4)	59(1)	34(1)	63(1)	-7(1)	-4(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for jwu85sad.

	x	y	z	U(eq)
H(2A)	1025	-290	2694	54
H(3)	957	2177	3075	54
H(4A)	3314	1421	4349	59
H(5A)	2493	4114	3738	74
H(5B)	3953	3728	4429	74
H(6A)	-761	374	3532	66
H(6B)	387	384	4341	66
H(7A)	-1370	2815	3711	81
H(7B)	-233	2813	4524	81
H(8A)	-3079	1178	4213	137
H(8B)	-2872	2589	4743	137

H(8C)	-1935	1153	5025	137
H(10)	6615	-1796	2317	59
H(11)	8774	-1518	1651	73
H(12)	9322	710	1140	68
H(13)	7642	2633	1268	73
H(14)	5472	2371	1932	72
H(16)	2904	4348	2119	51
H(17)	3219	6694	1589	56
H(18)	629	6000	421	68
H(19A)	1822	8662	931	82
H(19B)	266	8353	281	82
H(20A)	4539	4545	1179	69
H(20B)	3299	4592	399	69
H(21A)	4249	6860	89	83
H(21B)	5442	6879	885	83
H(22A)	6886	4984	438	138
H(22B)	6787	6290	-165	138
H(22C)	5673	4915	-351	138
H(24)	-13	7102	3627	64
H(25)	-1953	7872	4359	75
H(26)	-4525	7005	4102	76
H(27)	-5166	5373	3097	79
H(28)	-3276	4639	2344	68
H(1N)	4630(30)	-510(30)	3003(15)	57(9)
H(2N)	-690(30)	4650(30)	2114(13)	29(7)
H(2)	2378	-1717	3393	78
H(4)	1282	2997	1550	80

Table 6. Torsion angles [°] for jwu85sad.

O(1)-C(1)-C(2)-O(2)	178.9(2)
N(1)-C(1)-C(2)-O(2)	0.4(3)
O(1)-C(1)-C(2)-C(3)	-58.4(3)
N(1)-C(1)-C(2)-C(3)	123.1(2)
O(2)-C(2)-C(3)-C(4)	58.5(3)
C(1)-C(2)-C(3)-C(4)	-65.6(3)
O(2)-C(2)-C(3)-C(6)	-66.3(3)
C(1)-C(2)-C(3)-C(6)	169.5(2)
C(6)-C(3)-C(4)-C(5)	-106.4(3)
C(2)-C(3)-C(4)-C(5)	129.2(3)
C(4)-C(3)-C(6)-C(7)	71.0(3)
C(2)-C(3)-C(6)-C(7)	-165.3(2)
C(3)-C(6)-C(7)-C(8)	179.4(2)
C(14)-C(9)-C(10)-C(11)	-0.2(4)
N(1)-C(9)-C(10)-C(11)	-178.8(2)
C(9)-C(10)-C(11)-C(12)	0.6(4)

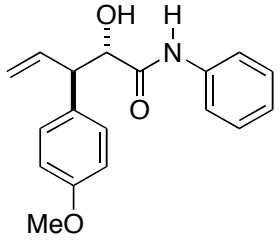
C(10)-C(11)-C(12)-C(13)	-0.9(4)
C(11)-C(12)-C(13)-C(14)	0.9(4)
C(12)-C(13)-C(14)-C(9)	-0.5(4)
C(10)-C(9)-C(14)-C(13)	0.2(4)
N(1)-C(9)-C(14)-C(13)	178.7(2)
O(3)-C(15)-C(16)-O(4)	179.2(2)
N(2)-C(15)-C(16)-O(4)	1.6(3)
O(3)-C(15)-C(16)-C(17)	-56.6(3)
N(2)-C(15)-C(16)-C(17)	125.7(2)
O(4)-C(16)-C(17)-C(18)	54.9(3)
C(15)-C(16)-C(17)-C(18)	-68.9(3)
O(4)-C(16)-C(17)-C(20)	-70.8(3)
C(15)-C(16)-C(17)-C(20)	165.4(2)
C(20)-C(17)-C(18)-C(19)	-110.1(3)
C(16)-C(17)-C(18)-C(19)	124.4(3)
C(18)-C(17)-C(20)-C(21)	72.8(3)
C(16)-C(17)-C(20)-C(21)	-161.8(2)
C(17)-C(20)-C(21)-C(22)	177.1(3)
C(28)-C(23)-C(24)-C(25)	-0.3(4)
N(2)-C(23)-C(24)-C(25)	179.6(3)
C(23)-C(24)-C(25)-C(26)	0.8(5)
C(24)-C(25)-C(26)-C(27)	-0.3(5)
C(25)-C(26)-C(27)-C(28)	-0.5(5)
C(26)-C(27)-C(28)-C(23)	0.9(5)
C(24)-C(23)-C(28)-C(27)	-0.5(4)
N(2)-C(23)-C(28)-C(27)	179.5(3)
O(1)-C(1)-N(1)-C(9)	-0.8(4)
C(2)-C(1)-N(1)-C(9)	177.7(2)
C(10)-C(9)-N(1)-C(1)	-146.9(3)
C(14)-C(9)-N(1)-C(1)	34.6(4)
O(3)-C(15)-N(2)-C(23)	1.1(4)
C(16)-C(15)-N(2)-C(23)	178.6(2)
C(28)-C(23)-N(2)-C(15)	172.3(3)
C(24)-C(23)-N(2)-C(15)	-7.7(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for jwu85sad [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4)...O(1)	0.84	1.91	2.699(3)	157.1
O(2)-H(2)...O(3)#1	0.84	1.88	2.707(3)	167.9
N(2)-H(2N)...O(4)	0.83(2)	2.15(2)	2.622(3)	116(2)
N(1)-H(1N)...O(2)	0.85(3)	2.10(3)	2.619(3)	119(2)

Symmetry transformations used to generate equivalent atoms:
#1 x,y-1,z



(2*S*,3*R*)-2-hydroxy-*N*-phenyl-3-*p*-methoxyphenylpent-4-enamide

Table 1. Crystal data and structure refinement for jwu89sad.

Identification code	jwu89sad	
Empirical formula	C18 H19 N O3	
Formula weight	297.34	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.4676(15) Å	α = 90°.
	b = 10.3486(19) Å	β = 90°.
	c = 18.041(3) Å	γ = 90°.
Volume	1580.9(5) $Å^3$	
Z	4	
Density (calculated)	1.249 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	632	
Crystal size	0.02 x 0.01 x 0.01 mm^3	
Theta range for data collection	2.26 to 24.99°.	
Index ranges	-10<=h<=10, -10<=k<=12, -17<=l<=21	
Reflections collected	8501	
Independent reflections	2783 [R(int) = 0.0418]	
Completeness to theta = 24.99°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2783 / 0 / 205	
Goodness-of-fit on F^2	1.400	
Final R indices [I>2sigma(I)]	R1 = 0.0867, wR2 = 0.1285	
R indices (all data)	R1 = 0.1014, wR2 = 0.1320	
Absolute structure parameter	1(2)	
Largest diff. peak and hole	0.223 and -0.223 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for jwu89sad. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2525(5)	-3450(4)	-1060(3)	52(1)
C(2)	3453(5)	-3401(4)	-435(3)	59(1)
C(3)	3788(5)	5329(3)	2257(2)	34(1)
C(4)	3649(5)	-2252(4)	-54(3)	47(1)
C(5)	1397(4)	1759(3)	1544(2)	38(1)
C(6)	-9(5)	1239(4)	1548(3)	55(1)
C(7)	1692(5)	6284(4)	1579(2)	47(1)
C(8)	2226(6)	8586(4)	2365(3)	63(1)
C(9)	1450(5)	5108(3)	1232(2)	44(1)
C(10)	3540(5)	4159(3)	1902(2)	33(1)
C(11)	2682(4)	1212(3)	-15(2)	31(1)
C(12)	1980(4)	-1185(3)	-930(2)	37(1)
C(13)	3314(4)	2228(3)	524(2)	33(1)
C(14)	1956(4)	2760(3)	1003(2)	32(1)
C(15)	2864(4)	6393(3)	2098(2)	34(1)
C(16)	2913(4)	-1143(3)	-300(2)	30(1)
C(17)	2344(4)	4039(3)	1383(2)	30(1)
O(3)	3203(3)	7487(2)	2492(2)	50(1)
C(18)	1797(5)	-2342(4)	-1303(2)	44(1)
O(1)	1738(3)	1512(2)	-497(2)	45(1)
N(1)	3212(4)	14(3)	96(2)	33(1)
O(2)	4547(3)	1711(2)	967(2)	40(1)

Table 3. Bond lengths [Å] and angles [°] for jwu89sad.

C(1)-C(18)	1.373(6)
C(1)-C(2)	1.376(6)
C(2)-C(4)	1.383(6)
C(3)-C(15)	1.381(5)
C(3)-C(10)	1.386(5)
C(4)-C(16)	1.379(5)
C(5)-C(6)	1.307(5)
C(5)-C(14)	1.500(5)
C(7)-C(15)	1.370(5)
C(7)-C(9)	1.384(5)
C(8)-O(3)	1.424(5)
C(9)-C(17)	1.368(5)
C(10)-C(17)	1.385(5)
C(11)-O(1)	1.221(4)

C(11)-N(1)	1.333(4)
C(11)-C(13)	1.529(5)
C(12)-C(18)	1.382(5)
C(12)-C(16)	1.385(5)
C(13)-O(2)	1.420(4)
C(13)-C(14)	1.540(5)
C(14)-C(17)	1.527(5)
C(15)-O(3)	1.367(4)
C(16)-N(1)	1.416(4)

C(18)-C(1)-C(2)	119.1(4)
C(1)-C(2)-C(4)	120.5(4)
C(15)-C(3)-C(10)	120.9(4)
C(16)-C(4)-C(2)	120.1(4)
C(6)-C(5)-C(14)	125.1(4)
C(15)-C(7)-C(9)	119.2(3)
C(17)-C(9)-C(7)	122.7(4)
C(17)-C(10)-C(3)	120.1(3)
O(1)-C(11)-N(1)	124.3(4)
O(1)-C(11)-C(13)	120.5(3)
N(1)-C(11)-C(13)	115.3(3)
C(18)-C(12)-C(16)	119.3(4)
O(2)-C(13)-C(11)	110.9(3)
O(2)-C(13)-C(14)	111.6(3)
C(11)-C(13)-C(14)	109.9(3)
C(5)-C(14)-C(17)	111.9(3)
C(5)-C(14)-C(13)	110.8(3)
C(17)-C(14)-C(13)	113.6(3)
O(3)-C(15)-C(7)	125.1(3)
O(3)-C(15)-C(3)	115.7(3)
C(7)-C(15)-C(3)	119.2(3)
C(4)-C(16)-C(12)	119.7(3)
C(4)-C(16)-N(1)	117.4(3)
C(12)-C(16)-N(1)	122.8(3)
C(9)-C(17)-C(10)	117.8(3)
C(9)-C(17)-C(14)	119.5(3)
C(10)-C(17)-C(14)	122.6(3)
C(15)-O(3)-C(8)	117.1(3)
C(1)-C(18)-C(12)	121.2(4)
C(11)-N(1)-C(16)	130.6(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for jwu89sad. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	50(3)	42(3)	64(3)	-21(2)	-2(3)	-1(2)
C(2)	71(3)	36(2)	70(4)	-3(2)	-9(3)	14(2)
C(3)	32(2)	42(2)	26(2)	-2(2)	-6(2)	-7(2)
C(4)	53(3)	39(2)	49(3)	-5(2)	-17(2)	6(2)
C(5)	37(2)	33(2)	44(3)	-7(2)	6(2)	-3(2)
C(6)	51(3)	51(3)	62(3)	-2(2)	14(2)	-16(2)
C(7)	51(3)	31(2)	58(3)	-4(2)	-11(2)	12(2)
C(8)	78(3)	33(2)	77(4)	-11(2)	1(3)	-3(2)
C(9)	42(2)	41(2)	48(3)	-8(2)	-17(2)	7(2)
C(10)	33(2)	31(2)	35(2)	6(2)	4(2)	5(2)
C(11)	27(2)	31(2)	34(2)	6(2)	3(2)	4(2)
C(12)	40(2)	32(2)	37(3)	2(2)	1(2)	5(2)
C(13)	27(2)	30(2)	44(2)	6(2)	-1(2)	-3(2)
C(14)	27(2)	30(2)	38(2)	-1(2)	-1(2)	-2(2)
C(15)	34(2)	30(2)	37(3)	-4(2)	1(2)	-4(2)
C(16)	29(2)	29(2)	33(2)	0(2)	7(2)	0(2)
C(17)	28(2)	30(2)	30(2)	1(2)	-1(2)	0(2)
O(3)	48(2)	34(2)	66(2)	-15(1)	-7(2)	-4(1)
C(18)	47(3)	51(2)	34(3)	-8(2)	1(2)	3(2)
O(1)	47(2)	43(2)	47(2)	-6(1)	-13(2)	20(1)
N(1)	31(2)	37(2)	32(2)	4(2)	-9(2)	2(2)
O(2)	28(1)	34(2)	58(2)	-3(1)	-4(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for jwu89sad.

	x	y	z	U(eq)
H(1)	2389	-4240	-1320	62
H(2)	3963	-4161	-263	71
H(3)	4606	5400	2615	40
H(4)	4293	-2226	377	56
H(5)	2122	1482	1913	46
H(6A)	-768	1491	1188	66
H(6B)	-272	610	1910	66
H(7)	1053	7009	1457	56
H(8A)	2328	8860	1847	94
H(8B)	2556	9293	2692	94
H(8C)	1124	8362	2468	94
H(9)	629	5040	875	52
H(10)	4191	3438	2015	40
H(12)	1470	-425	-1105	44

H(13)	3758	2959	227	40
H(14)	1054	2934	659	38
H(18)	1157	-2372	-1735	53
H(2A)	5378	2138	897	60
H(1N)	3830(30)	-80(30)	423(17)	0(8)

Table 6. Torsion angles [°] for jwu89sad.

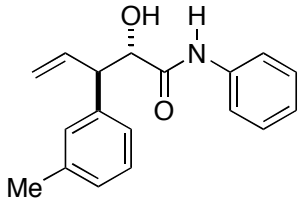
C(18)-C(1)-C(2)-C(4)	0.1(7)
C(1)-C(2)-C(4)-C(16)	0.0(7)
C(15)-C(7)-C(9)-C(17)	-0.5(7)
C(15)-C(3)-C(10)-C(17)	-0.5(5)
O(1)-C(11)-C(13)-O(2)	-172.0(3)
N(1)-C(11)-C(13)-O(2)	9.2(4)
O(1)-C(11)-C(13)-C(14)	64.1(4)
N(1)-C(11)-C(13)-C(14)	-114.6(3)
C(6)-C(5)-C(14)-C(17)	116.3(4)
C(6)-C(5)-C(14)-C(13)	-115.8(4)
O(2)-C(13)-C(14)-C(5)	-52.8(4)
C(11)-C(13)-C(14)-C(5)	70.6(4)
O(2)-C(13)-C(14)-C(17)	74.2(4)
C(11)-C(13)-C(14)-C(17)	-162.4(3)
C(9)-C(7)-C(15)-O(3)	-178.5(4)
C(9)-C(7)-C(15)-C(3)	0.8(6)
C(10)-C(3)-C(15)-O(3)	179.0(4)
C(10)-C(3)-C(15)-C(7)	-0.4(6)
C(2)-C(4)-C(16)-C(12)	-0.2(6)
C(2)-C(4)-C(16)-N(1)	-177.4(4)
C(18)-C(12)-C(16)-C(4)	0.2(6)
C(18)-C(12)-C(16)-N(1)	177.3(3)
C(7)-C(9)-C(17)-C(10)	-0.3(6)
C(7)-C(9)-C(17)-C(14)	177.3(4)
C(3)-C(10)-C(17)-C(9)	0.8(5)
C(3)-C(10)-C(17)-C(14)	-176.8(3)
C(5)-C(14)-C(17)-C(9)	-114.0(4)
C(13)-C(14)-C(17)-C(9)	119.6(4)
C(5)-C(14)-C(17)-C(10)	63.5(5)
C(13)-C(14)-C(17)-C(10)	-62.9(5)
C(7)-C(15)-O(3)-C(8)	2.0(6)
C(3)-C(15)-O(3)-C(8)	-177.3(4)
C(2)-C(1)-C(18)-C(12)	-0.1(7)
C(16)-C(12)-C(18)-C(1)	-0.1(6)
O(1)-C(11)-N(1)-C(16)	3.0(6)
C(13)-C(11)-N(1)-C(16)	-178.4(3)
C(4)-C(16)-N(1)-C(11)	179.5(4)
C(12)-C(16)-N(1)-C(11)	2.4(6)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for jwu89sad [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...O(2)	0.80(3)	2.18(3)	2.615(4)	114(2)
O(2)-H(2A)...O(1)#1	0.84	1.95	2.746(4)	157.9

Symmetry transformations used to generate equivalent atoms:
#1 x+1/2,-y+1/2,-z



(2*S*,3*R*)-2-hydroxy-*N*-phenyl-3-*m*-tolylpent-4-enamide

Table 1. Crystal data and structure refinement for jwu84sad.

Identification code	jwu84sad	
Empirical formula	C18 H19 N O2	
Formula weight	281.34	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	?	
Space group	?	
Unit cell dimensions	a = 8.6105(16) Å	α = 90°.
	b = 9.7462(18) Å	β = 90°.
	c = 18.860(3) Å	γ = 90°.
Volume	1582.7(5) $Å^3$	
Z	4	
Density (calculated)	1.181 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	600	
Crystal size	? x ? x ? mm^3	
Theta range for data collection	2.16 to 25.00°.	
Index ranges	-10<=h<=10, -11<=k<=9, -22<=l<=20	
Reflections collected	8525	
Independent reflections	2797 [R(int) = 0.0231]	
Completeness to theta = 25.00°	99.9 %	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2797 / 0 / 196
Goodness-of-fit on F^2	1.429
Final R indices [I>2sigma(I)]	R1 = 0.0723, wR2 = 0.1307
R indices (all data)	R1 = 0.0740, wR2 = 0.1314
Absolute structure parameter	1(2)
Largest diff. peak and hole	0.190 and -0.199 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for jwu84sad. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2801(3)	8696(3)	9996(2)	38(1)
C(2)	3440(4)	7681(3)	9452(2)	38(1)
C(3)	2098(4)	7138(3)	8981(2)	37(1)
C(4)	1441(4)	8270(3)	8526(2)	46(1)
C(5)	3(4)	8654(4)	8511(2)	57(1)
C(6)	2560(3)	5902(3)	8540(2)	36(1)
C(7)	1740(4)	4687(3)	8608(2)	41(1)
C(8)	2066(4)	3544(3)	8194(2)	48(1)
C(9)	1147(6)	2229(4)	8278(2)	77(1)
C(10)	3248(4)	3633(4)	7704(2)	51(1)
C(11)	4085(4)	4827(4)	7629(2)	55(1)
C(12)	3744(4)	5954(4)	8043(2)	51(1)
C(13)	2918(4)	11198(3)	10288(2)	39(1)
C(14)	2090(4)	11189(4)	10918(2)	58(1)
C(15)	1757(5)	12419(4)	11248(2)	66(1)
C(16)	2229(5)	13651(4)	10967(2)	62(1)
C(17)	3046(6)	13655(4)	10346(2)	69(1)
C(18)	3392(5)	12431(4)	10008(2)	59(1)
N(1)	3322(3)	9989(3)	9920(2)	40(1)
O(1)	1883(3)	8346(2)	10453(1)	51(1)
O(2)	4628(2)	8283(2)	9039(1)	42(1)

Table 3. Bond lengths [Å] and angles [°] for jwu84sad.

C(1)-O(1)	1.218(4)
C(1)-N(1)	1.346(4)
C(1)-C(2)	1.528(4)
C(2)-O(2)	1.413(4)
C(2)-C(3)	1.551(4)
C(3)-C(4)	1.507(4)
C(3)-C(6)	1.517(4)
C(4)-C(5)	1.294(5)
C(6)-C(7)	1.384(4)
C(6)-C(12)	1.386(4)
C(7)-C(8)	1.389(4)
C(8)-C(10)	1.378(5)
C(8)-C(9)	1.514(5)
C(10)-C(11)	1.375(5)
C(11)-C(12)	1.380(5)

C(13)-C(18)	1.374(5)
C(13)-C(14)	1.385(5)
C(13)-N(1)	1.411(4)
C(14)-C(15)	1.380(5)
C(15)-C(16)	1.374(5)
C(16)-C(17)	1.366(6)
C(17)-C(18)	1.384(5)

O(1)-C(1)-N(1)	123.6(3)
O(1)-C(1)-C(2)	121.9(3)
N(1)-C(1)-C(2)	114.5(3)
O(2)-C(2)-C(1)	111.2(2)
O(2)-C(2)-C(3)	111.4(2)
C(1)-C(2)-C(3)	109.7(2)
C(4)-C(3)-C(6)	111.6(2)
C(4)-C(3)-C(2)	110.9(2)
C(6)-C(3)-C(2)	113.0(2)
C(5)-C(4)-C(3)	125.7(3)
C(7)-C(6)-C(12)	118.0(3)
C(7)-C(6)-C(3)	119.7(3)
C(12)-C(6)-C(3)	122.3(3)
C(6)-C(7)-C(8)	122.1(3)
C(10)-C(8)-C(7)	118.4(3)
C(10)-C(8)-C(9)	120.7(3)
C(7)-C(8)-C(9)	121.0(3)
C(11)-C(10)-C(8)	120.7(3)
C(10)-C(11)-C(12)	120.2(3)
C(11)-C(12)-C(6)	120.7(3)
C(18)-C(13)-C(14)	119.2(3)
C(18)-C(13)-N(1)	117.9(3)
C(14)-C(13)-N(1)	122.9(3)
C(15)-C(14)-C(13)	119.2(3)
C(16)-C(15)-C(14)	121.6(4)
C(17)-C(16)-C(15)	119.0(3)
C(16)-C(17)-C(18)	120.2(4)
C(13)-C(18)-C(17)	120.8(4)
C(1)-N(1)-C(13)	130.3(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for jwu84sad. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	31(2)	41(2)	42(2)	9(1)	-6(2)	-4(1)
C(2)	33(2)	30(2)	50(2)	12(1)	-5(1)	-2(1)
C(3)	34(2)	35(2)	44(2)	6(1)	6(2)	-3(1)
C(4)	42(2)	42(2)	53(2)	5(2)	-5(2)	-5(2)
C(5)	48(2)	51(2)	71(2)	1(2)	-14(2)	7(2)
C(6)	33(2)	38(2)	38(2)	13(1)	-1(1)	3(1)
C(7)	39(2)	45(2)	40(2)	6(1)	9(2)	3(2)
C(8)	54(2)	42(2)	46(2)	7(2)	0(2)	6(2)
C(9)	109(4)	44(2)	77(3)	-11(2)	23(3)	-12(2)
C(10)	48(2)	54(2)	50(2)	-5(2)	-7(2)	19(2)
C(11)	42(2)	78(3)	45(2)	-3(2)	10(2)	6(2)
C(12)	47(2)	53(2)	52(2)	6(2)	10(2)	-7(2)
C(13)	35(2)	37(2)	45(2)	3(1)	-8(1)	-3(1)
C(14)	58(2)	54(2)	62(2)	-1(2)	13(2)	-18(2)
C(15)	61(3)	65(3)	72(3)	-17(2)	17(2)	-12(2)
C(16)	67(2)	51(2)	68(2)	-16(2)	-9(2)	4(2)
C(17)	111(4)	40(2)	55(2)	6(2)	-12(2)	-1(2)
C(18)	89(3)	45(2)	44(2)	4(2)	2(2)	-5(2)
N(1)	38(2)	43(2)	39(2)	6(1)	8(1)	-2(1)
O(1)	49(1)	53(1)	51(1)	2(1)	12(1)	-17(1)
O(2)	26(1)	37(1)	63(1)	11(1)	4(1)	0(1)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for jwu84sad.

	x	y	z	U(eq)
H(2A)	3896	6885	9714	45
H(3)	1250	6833	9307	45
H(4)	2144	8742	8224	55
H(5A)	-740	8211	8804	68
H(5B)	-309	9381	8207	68
H(7)	929	4634	8948	49
H(9A)	1004	2032	8784	115
H(9B)	1712	1471	8054	115
H(9C)	130	2333	8051	115
H(10)	3488	2864	7415	61
H(11)	4901	4875	7291	66
H(12)	4327	6774	7987	61

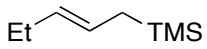
H(14)	1756	10347	11120	69
H(15)	1189	12413	11679	79
H(16)	1991	14488	11201	74
H(17)	3377	14499	10145	83
H(18)	3965	12443	9578	71
H(1N)	3970(30)	10080(30)	9608(14)	10(7)
H(2)	5488	7939	9151	63

Table 6. Torsion angles [°] for jwu84sad.

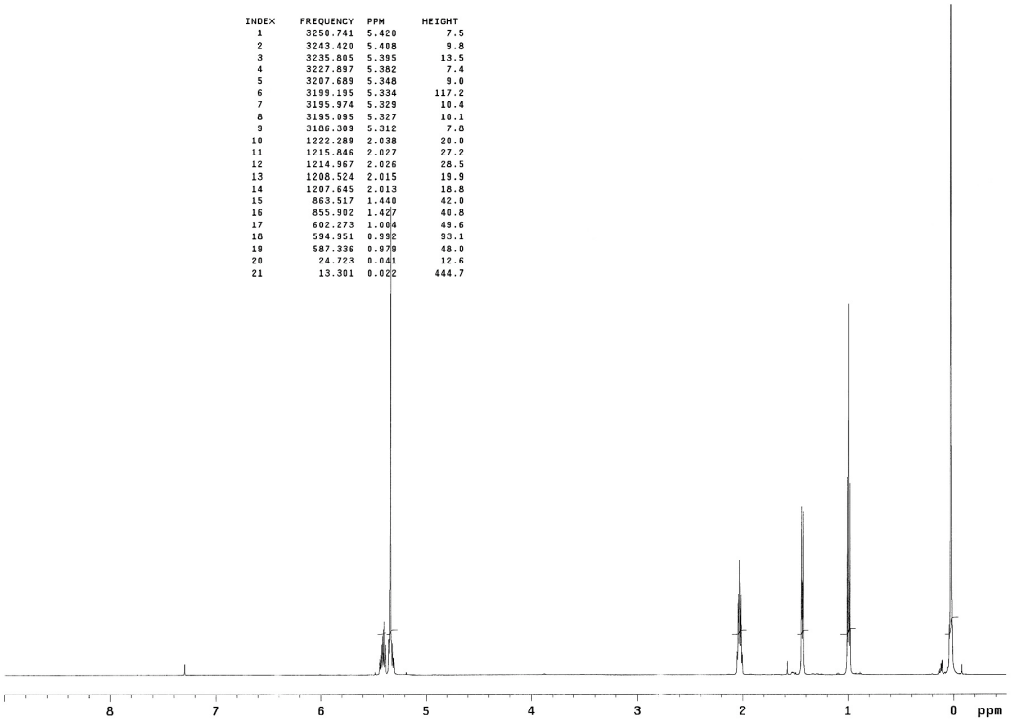
O(1)-C(1)-C(2)-O(2)	-172.3(3)
N(1)-C(1)-C(2)-O(2)	8.3(3)
O(1)-C(1)-C(2)-C(3)	63.9(4)
N(1)-C(1)-C(2)-C(3)	-115.4(3)
O(2)-C(2)-C(3)-C(4)	-56.8(3)
C(1)-C(2)-C(3)-C(4)	66.8(3)
O(2)-C(2)-C(3)-C(6)	69.2(3)
C(1)-C(2)-C(3)-C(6)	-167.1(2)
C(6)-C(3)-C(4)-C(5)	109.7(4)
C(2)-C(3)-C(4)-C(5)	-123.5(4)
C(4)-C(3)-C(6)-C(7)	-112.2(3)
C(2)-C(3)-C(6)-C(7)	122.1(3)
C(4)-C(3)-C(6)-C(12)	65.1(4)
C(2)-C(3)-C(6)-C(12)	-60.6(4)
C(12)-C(6)-C(7)-C(8)	-0.4(5)
C(3)-C(6)-C(7)-C(8)	176.9(3)
C(6)-C(7)-C(8)-C(10)	0.2(5)
C(6)-C(7)-C(8)-C(9)	-179.8(3)
C(7)-C(8)-C(10)-C(11)	0.1(5)
C(9)-C(8)-C(10)-C(11)	-179.8(4)
C(8)-C(10)-C(11)-C(12)	-0.3(5)
C(10)-C(11)-C(12)-C(6)	0.1(5)
C(7)-C(6)-C(12)-C(11)	0.3(5)
C(3)-C(6)-C(12)-C(11)	-177.0(3)
C(18)-C(13)-C(14)-C(15)	0.2(5)
N(1)-C(13)-C(14)-C(15)	179.3(3)
C(13)-C(14)-C(15)-C(16)	-0.1(6)
C(14)-C(15)-C(16)-C(17)	0.1(7)
C(15)-C(16)-C(17)-C(18)	-0.2(7)
C(14)-C(13)-C(18)-C(17)	-0.3(6)
N(1)-C(13)-C(18)-C(17)	-179.4(4)
C(16)-C(17)-C(18)-C(13)	0.3(7)
O(1)-C(1)-N(1)-C(13)	-3.9(5)
C(2)-C(1)-N(1)-C(13)	175.4(3)
C(18)-C(13)-N(1)-C(1)	-166.9(4)
C(14)-C(13)-N(1)-C(1)	14.0(5)

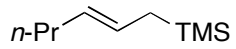
Symmetry transformations used to generate equivalent atoms:

1H NMR Spectra

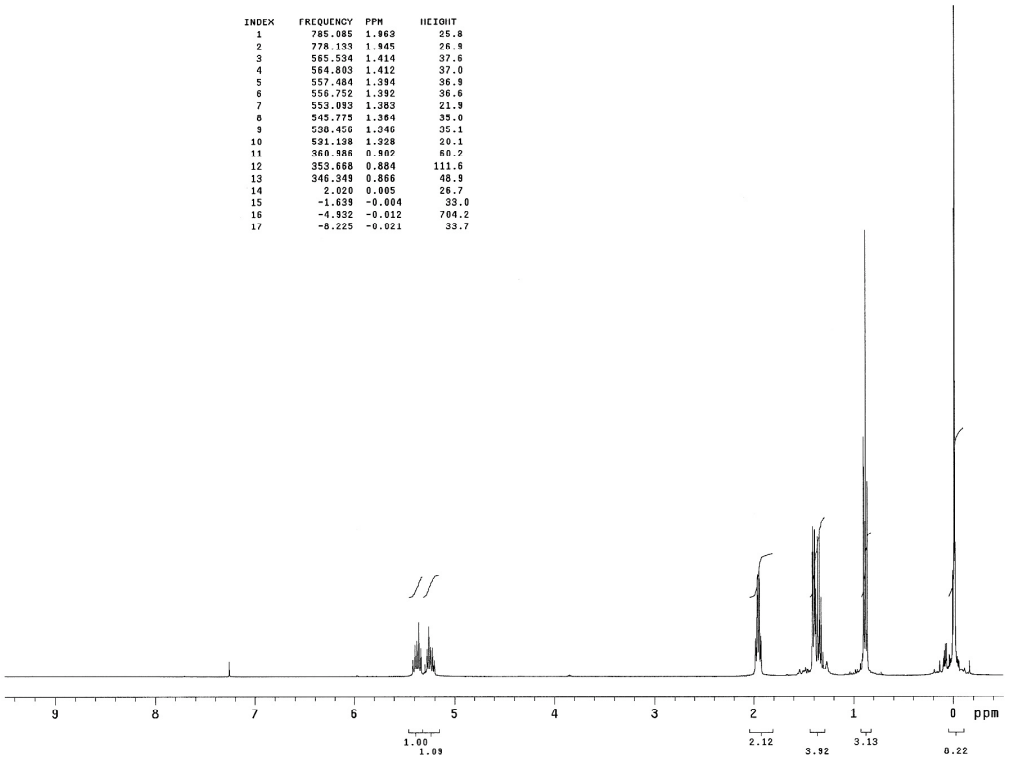


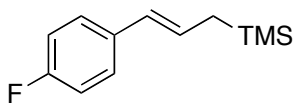
INDEX	FREQUENCY	PPM	HEIGHT
1	3250.741	5.420	7.5
2	3243.420	5.408	9.8
3	3235.805	5.395	13.5
4	3227.897	5.382	7.4
5	3207.689	5.348	9.0
6	3199.195	5.334	117.2
7	3195.974	5.329	10.4
8	3195.095	5.327	10.1
9	3186.309	5.312	7.0
10	1222.289	2.038	20.0
11	1215.846	2.027	27.2
12	1214.967	2.026	28.5
13	1208.524	2.015	19.9
14	1207.645	2.013	18.8
15	863.517	1.440	42.0
16	855.902	1.427	40.8
17	602.273	1.004	49.6
18	594.951	0.992	93.1
19	587.336	0.979	48.0
20	24.723	0.041	12.6
21	13.301	0.022	444.7



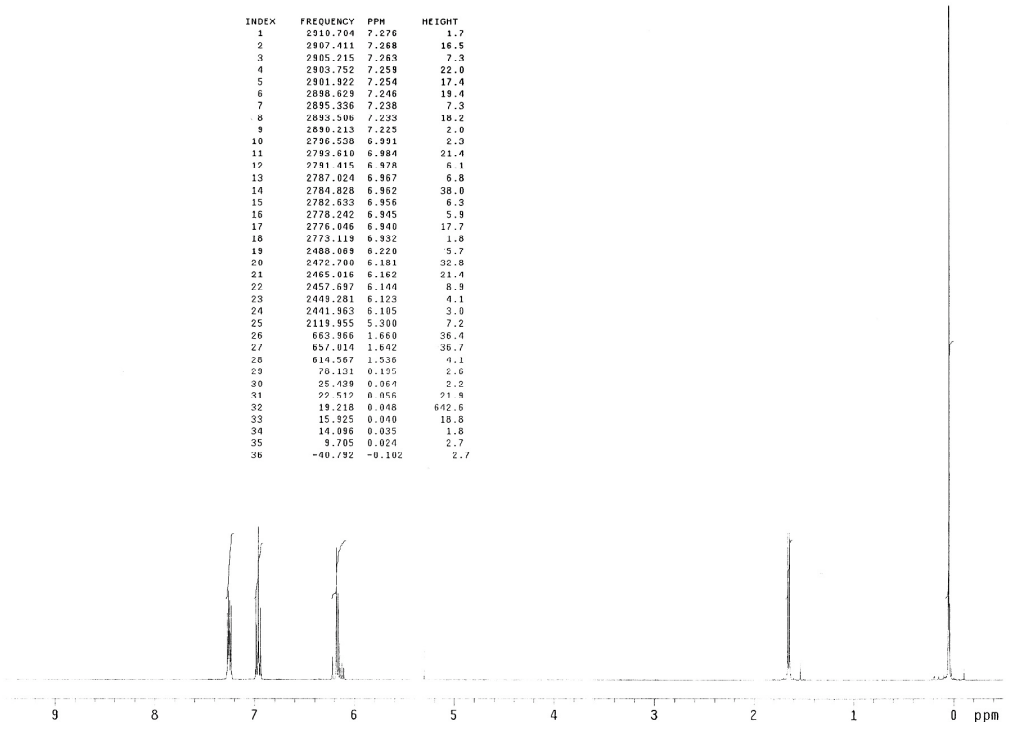


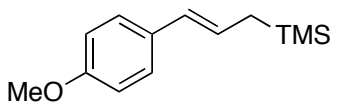
INDEX	FREQUENCY	PPM	HEIGHT
1	785.085	1.963	25.8
2	778.133	1.945	26.9
3	565.534	1.414	37.6
4	564.803	1.412	37.0
5	557.484	1.394	36.9
6	556.752	1.392	36.6
7	553.093	1.383	21.9
8	545.775	1.364	35.0
9	538.456	1.346	35.1
10	531.138	1.328	20.1
11	360.886	0.902	60.2
12	353.568	0.884	111.6
13	346.349	0.866	48.9
14	2.020	0.005	26.7
15	-1.639	-0.004	33.0
16	-4.932	-0.012	704.2
17	-8.225	-0.021	33.7



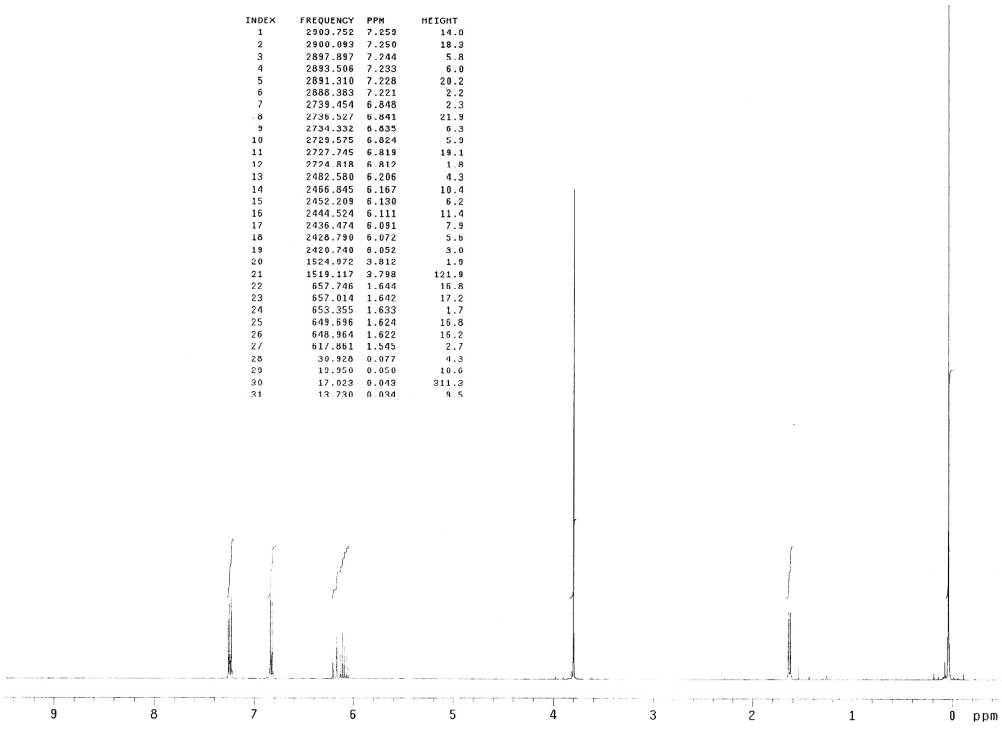


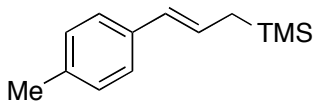
INDEX	FREQUENCY	PPM	HEIGHT
1	2910.704	7.276	1.7
2	2907.411	7.268	16.5
3	2905.215	7.263	7.3
4	2903.752	7.259	22.0
5	2901.922	7.254	17.4
6	2898.629	7.246	19.4
7	2895.336	7.238	7.3
8	2893.506	7.233	16.2
9	2890.213	7.225	2.0
10	2796.530	6.991	2.3
11	2793.610	6.984	21.4
12	2791.415	6.978	6.1
13	2787.024	6.967	6.8
14	2784.828	6.962	38.0
15	2782.633	6.956	6.3
16	2778.242	6.945	5.9
17	2776.046	6.940	17.7
18	2773.119	6.932	1.8
19	2488.069	6.220	5.7
20	2472.700	6.181	32.8
21	2465.016	6.162	21.4
22	2457.697	6.144	8.9
23	2449.281	6.123	4.1
24	2441.963	6.105	3.0
25	2119.955	5.300	7.2
26	663.966	1.660	36.4
27	657.014	1.642	36.7
28	614.567	1.536	4.1
29	78.131	0.195	2.6
30	25.439	0.064	2.2
31	22.512	0.056	21.8
32	19.218	0.048	642.6
33	15.925	0.040	18.8
34	14.096	0.035	1.8
35	9.705	0.024	2.7
36	-40.792	-0.102	2.7



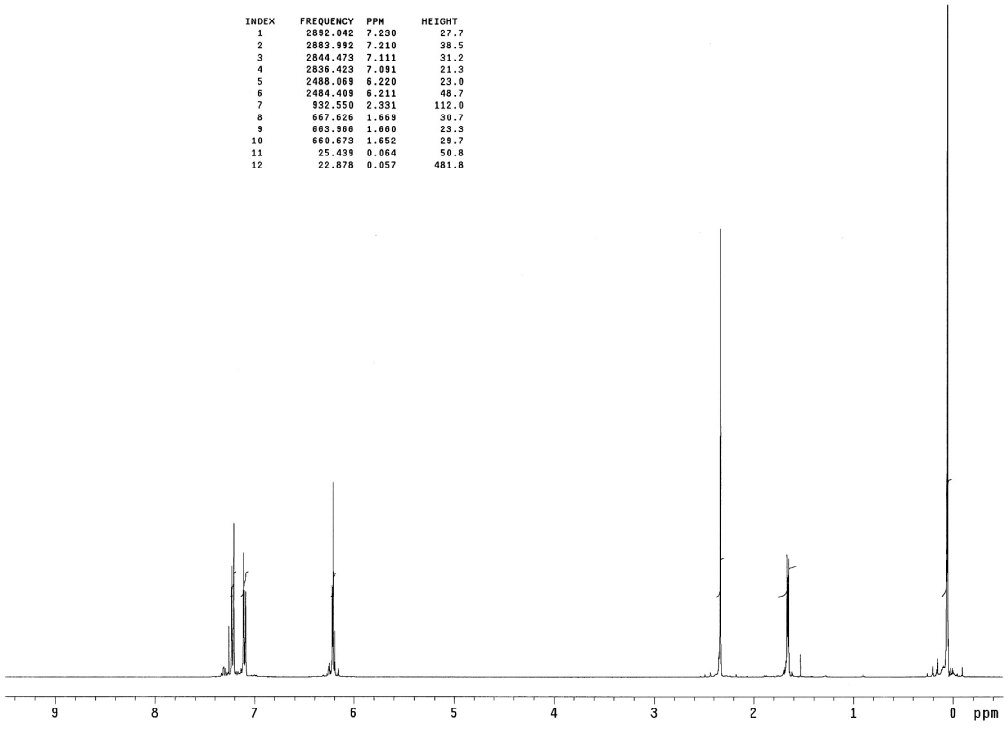


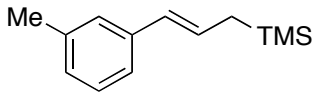
INDEX	FREQUENCY	PPM	HEIGHT
1	2903.752	7.259	14.0
2	2900.093	7.250	18.3
3	2897.887	7.244	5.8
4	2893.506	7.233	6.0
5	2891.310	7.228	20.2
6	2888.383	7.221	2.2
7	2739.454	6.848	2.3
8	2736.527	6.841	21.9
9	2734.332	6.835	6.3
10	2729.575	6.824	5.9
11	2727.745	6.819	19.1
12	2724.818	6.812	1.8
13	2482.580	6.206	4.3
14	2466.845	6.167	10.4
15	2452.209	6.130	6.2
16	2444.524	6.111	11.4
17	2436.474	6.091	7.9
18	2426.790	6.072	5.6
19	2420.740	6.052	3.0
20	1524.972	3.812	1.9
21	1519.117	3.798	121.9
22	657.746	1.644	16.8
23	657.014	1.642	17.2
24	653.355	1.633	1.7
25	649.696	1.624	15.8
26	648.964	1.622	15.2
27	617.861	1.545	2.7
28	30.928	0.077	4.3
29	19.950	0.050	10.6
30	17.023	0.043	311.3
31	13.730	0.034	9.5



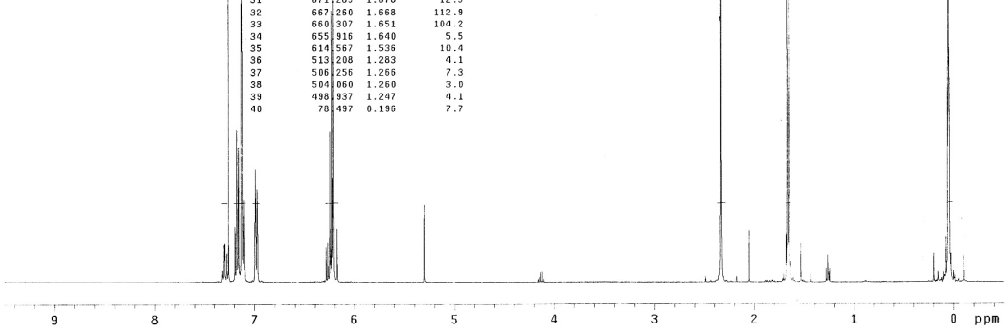


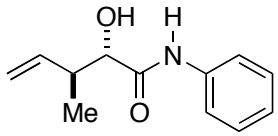
INDEX	FREQUENCY	PPM	HEIGHT
1	2892.042	7.230	27.7
2	2883.992	7.210	98.5
3	2844.473	7.111	31.2
4	2836.423	7.091	21.3
5	2488.069	6.220	23.0
6	2484.409	6.211	48.7
7	932.550	2.331	112.0
8	667.626	1.669	30.7
9	663.366	1.660	23.3
10	660.673	1.652	28.7
11	25.439	0.064	50.8
12	22.878	0.057	481.8



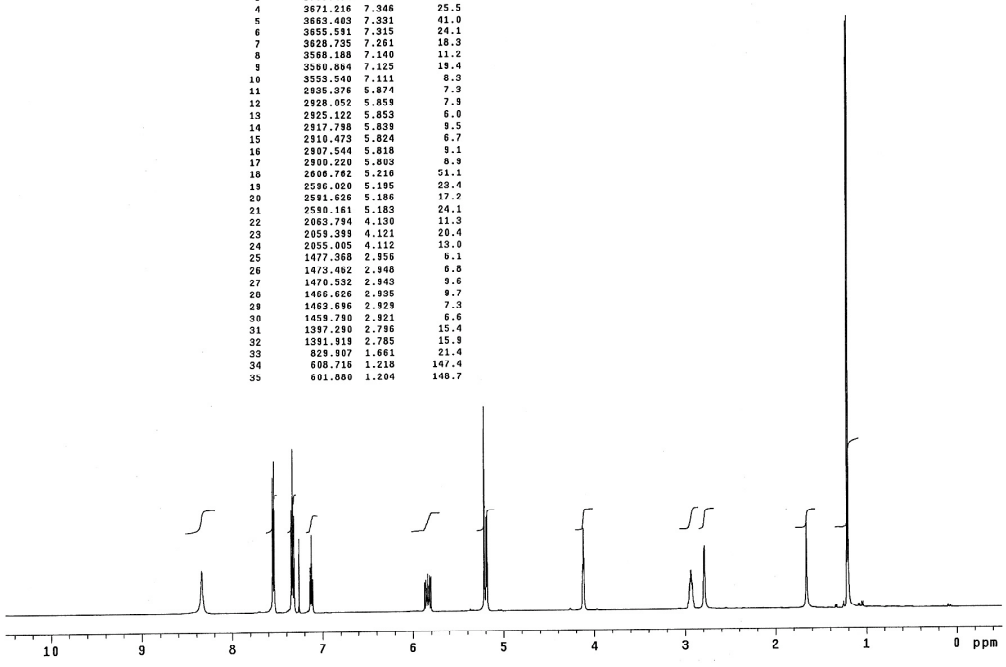


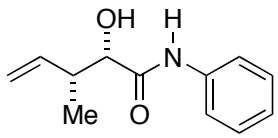
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2927.902	7.319	3.5	41	62.031	0.155	3.2
2	2921.316	7.303	9.8	42	40.076	0.100	3.1
3	2919.486	7.298	8.9	43	30.928	0.077	11.8
4	2918.388	7.296	10.2	44	22.878	0.057	54.1
5	2911.070	7.277	7.7	45	19.950	0.050	1439.5
6	2909.240	7.273	3.3	46	16.657	0.042	58.6
7	2903.752	7.259	83.4	47	10.436	0.026	7.7
8	2877.040	7.192	14.3	48	0.191	0.000	3.3
9	2869.721	7.174	37.7	49	-40.060	-0.100	7.1
10	2868.989	7.172	38.5				
11	2861.671	7.154	34.1				
12	2850.012	7.145	3.1				
13	2856.182	7.140	3.2				
14	2848.864	7.122	93.5				
15	2841.180	7.103	20.9				
16	2795.806	6.989	28.5				
17	2788.487	6.971	23.4				
18	2513.683	6.284	9.2				
19	2506.365	6.266	10.5				
20	2505.267	6.263	4.4				
21	2497.948	6.245	36.1				
22	2494.655	6.236	8.8				
23	2490.996	6.227	68.1				
24	2485.507	6.213	95.9				
25	2470.505	6.176	13.9				
26	2120.321	5.300	19.6				
27	1654.873	4.137	2.9				
28	1647.554	4.119	3.2				
29	934.014	2.335	291.7				
30	820.579	2.051	13.2				
31	671.205	1.678	12.5				
32	667.260	1.668	112.9				
33	660.307	1.651	104.2				
34	655.916	1.640	5.5				
35	614.567	1.536	10.4				
36	513.208	1.283	4.1				
37	506.256	1.266	7.3				
38	504.060	1.260	3.0				
39	498.937	1.247	4.1				
40	78.497	0.196	7.7				





INDEX	FREQUENCY	PPM	HEIGHT
1	4168.774	8.342	10.7
2	3773.266	7.550	33.7
3	3765.454	7.535	37.7
4	3671.216	7.346	25.5
5	3663.403	7.331	41.0
6	3655.591	7.315	24.1
7	3628.735	7.261	18.3
8	3568.188	7.140	11.2
9	3560.864	7.125	19.4
10	3553.540	7.111	8.3
11	2935.376	5.874	7.9
12	2928.052	5.859	7.9
13	2925.122	5.853	6.0
14	2917.798	5.839	9.5
15	2910.473	5.824	6.7
16	2907.544	5.818	9.1
17	2900.220	5.803	8.9
18	2606.762	5.216	51.1
19	2596.020	5.195	23.4
20	2591.626	5.186	17.2
21	2590.161	5.183	24.1
22	2063.794	4.130	11.3
23	2059.399	4.121	20.4
24	2055.005	4.112	13.0
25	1477.368	2.956	9.1
26	1473.492	2.948	8.8
27	1470.532	2.943	9.6
28	1466.626	2.935	9.7
29	1463.696	2.929	7.3
30	1459.790	2.921	6.6
31	1397.290	2.796	15.4
32	1391.919	2.785	15.9
33	828.907	1.661	21.4
34	608.716	1.218	147.4
35	601.880	1.204	148.7





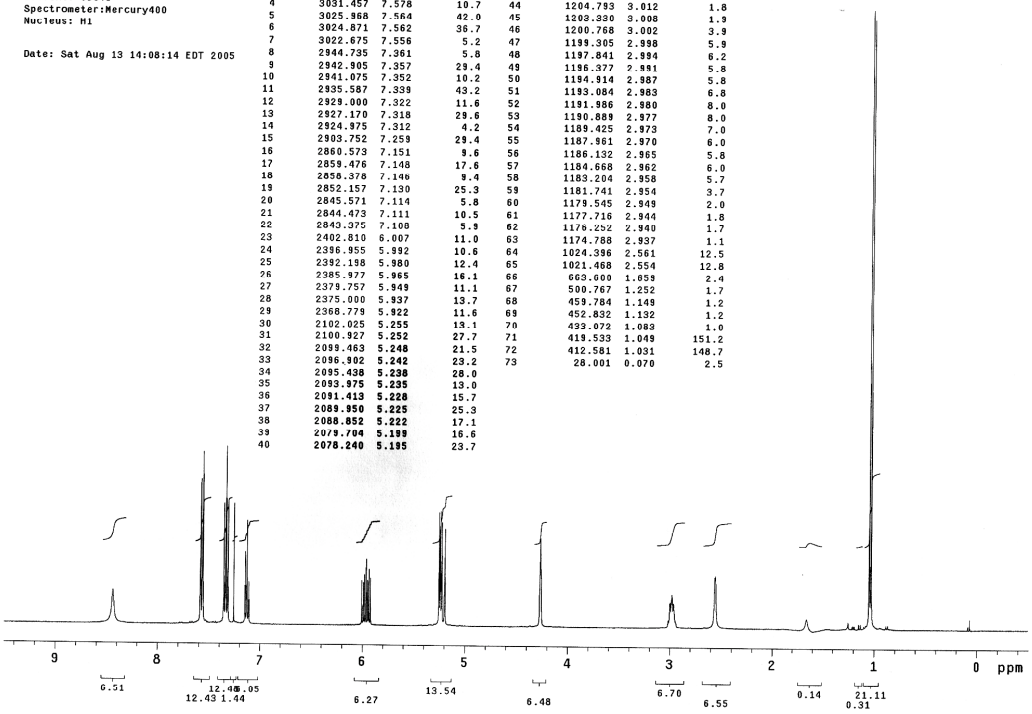
YA-I-225A-syn-ROH-ent

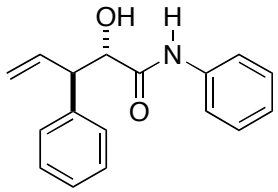
Pulse Sequence: s2pul

Solvent: CDCl3
Spectrometer:Mercury400
Nucleus: H1

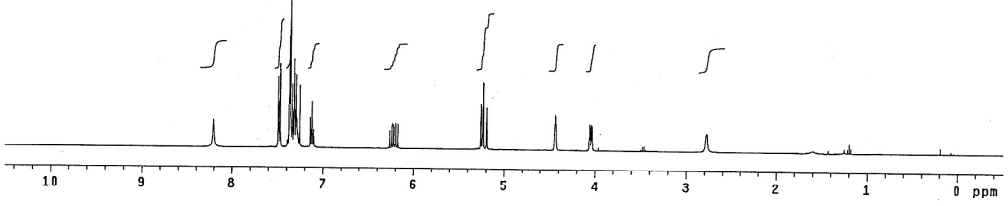
Date: Sat Aug 13 14:08:14 EDT 2005

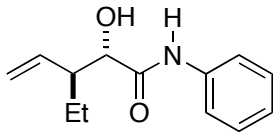
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3376.518	8.441	8.1	41	2076.777	5.192	12.0
2	3034.750	7.586	30.1	42	1705.735	4.264	21.4
3	3033.287	7.583	35.5	43	1206.257	3.015	1.7
4	3031.457	7.578	10.7	44	1204.793	3.012	1.8
5	3025.968	7.564	42.0	45	1203.330	3.008	1.9
6	3024.871	7.562	36.7	46	1200.768	3.002	3.9
7	3022.675	7.556	5.2	47	1199.305	2.998	5.9
8	2944.735	7.361	5.8	48	1197.841	2.994	6.2
9	2942.905	7.357	29.4	49	1196.377	2.991	5.8
10	2941.075	7.352	10.2	50	1194.914	2.987	5.8
11	2935.587	7.339	43.2	51	1193.084	2.983	6.8
12	2929.000	7.322	11.6	52	1191.986	2.980	8.0
13	2927.170	7.318	29.6	53	1190.889	2.977	8.0
14	2924.975	7.312	4.2	54	1189.425	2.973	7.0
15	2903.752	7.259	29.4	55	1187.961	2.970	6.0
16	2860.573	7.151	9.6	56	1186.132	2.965	5.8
17	2859.476	7.148	17.6	57	1184.668	2.962	6.0
18	2856.378	7.140	9.4	58	1183.204	2.958	5.7
19	2852.157	7.130	25.3	59	1181.741	2.954	3.7
20	2845.571	7.114	5.8	60	1179.545	2.949	2.0
21	2844.473	7.111	10.5	61	1177.716	2.944	1.8
22	2843.375	7.108	5.9	62	1176.252	2.940	1.7
23	2402.810	6.007	11.0	63	1174.788	2.937	1.1
24	2396.955	5.992	10.6	64	1024.396	2.561	12.5
25	2392.198	5.980	12.4	65	1021.468	2.554	12.8
26	2385.977	5.965	16.1	66	663.600	1.659	2.4
27	2379.757	5.949	11.1	67	500.767	1.252	1.7
28	2375.000	5.937	13.7	68	459.784	1.149	1.2
29	2368.779	5.922	11.6	69	452.832	1.132	1.2
30	2102.025	5.255	13.1	70	433.072	1.083	1.0
31	2100.927	5.252	27.7	71	419.533	1.049	151.2
32	2099.463	5.248	21.5	72	412.581	1.031	148.7
33	2096.902	5.242	23.2	73	28.001	0.070	2.5
34	2095.438	5.238	28.0				
35	2093.975	5.235	13.0				
36	2091.413	5.228	15.7				
37	2089.950	5.225	25.3				
38	2088.852	5.222	17.1				
39	2079.704	5.199	16.6				
40	2078.240	5.195	23.7				



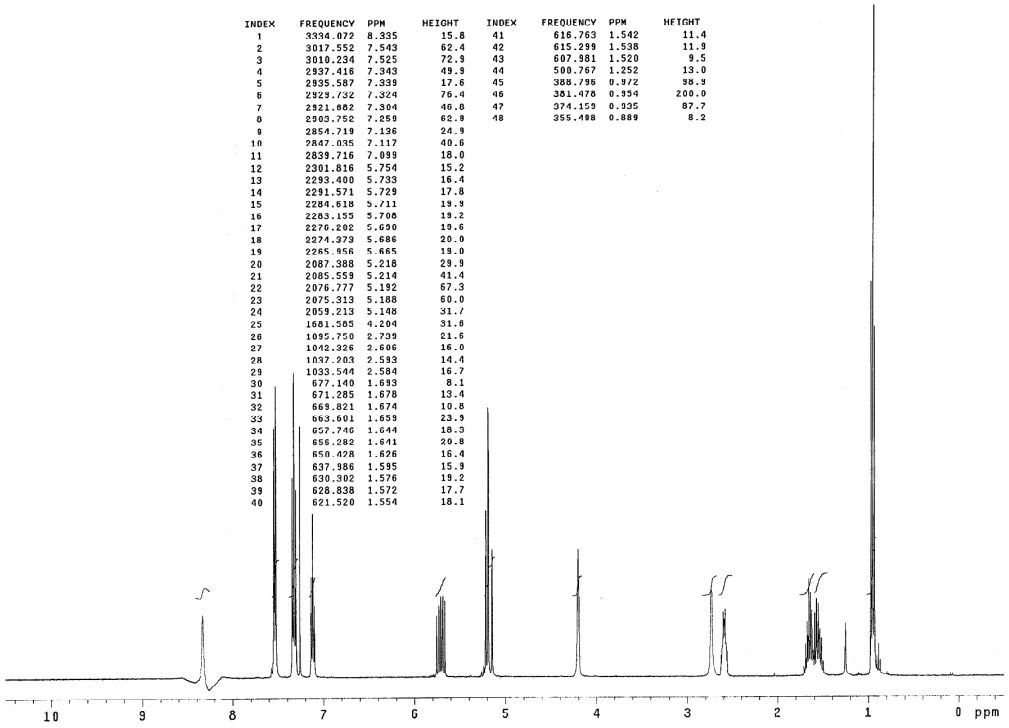


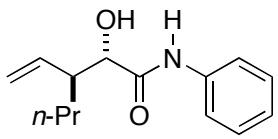
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3283.835	8.209	6.9	41	2103.677	5.259	11.5
2	2999.133	7.497	14.1	42	2096.724	5.242	10.8
3	2998.035	7.495	18.0	43	2095.260	5.238	16.8
4	2990.351	7.475	21.2	44	2094.162	5.235	16.6
5	2989.619	7.474	19.9	45	2079.525	5.199	8.7
6	2987.423	7.468	3.2	46	2078.427	5.196	10.7
7	2953.025	7.382	3.0	47	2076.963	5.192	7.8
8	2950.463	7.376	28.3	48	1775.794	4.439	9.0
9	2948.998	7.372	29.8	49	1626.490	4.066	8.8
10	2945.340	7.363	105.1	50	1622.891	4.057	6.5
11	2940.217	7.350	5.4	51	1617.706	4.044	6.5
12	2938.387	7.346	3.8	52	1614.048	4.035	6.2
13	2936.557	7.341	16.1	53	1114.539	2.786	4.4
14	2934.362	7.335	6.0	54	1111.612	2.778	4.4
15	2928.873	7.322	22.6	55	479.999	1.200	2.1
16	2925.213	7.313	7.1				
17	2922.286	7.305	10.6				
18	2920.456	7.301	18.4				
19	2917.528	7.293	6.2				
20	2916.431	7.291	7.3				
21	2914.235	7.285	4.5				
22	2912.405	7.281	3.1				
23	2910.942	7.277	3.7				
24	2908.014	7.270	2.3				
25	2903.985	7.260	15.8				
26	2858.246	7.145	4.2				
27	2857.148	7.142	7.6				
28	2856.050	7.140	4.5				
29	2849.829	7.124	11.8				
30	2843.243	7.108	9.0				
31	2842.145	7.105	4.7				
32	2841.047	7.102	2.8				
33	2505.479	6.263	4.7				
34	2497.063	6.242	5.8				
35	2495.233	6.238	6.3				
36	2488.646	6.221	6.1				
37	2486.816	6.217	5.7				
38	2479.864	6.199	6.4				
39	2478.034	6.195	6.2				
40	2469.617	6.174	6.2				



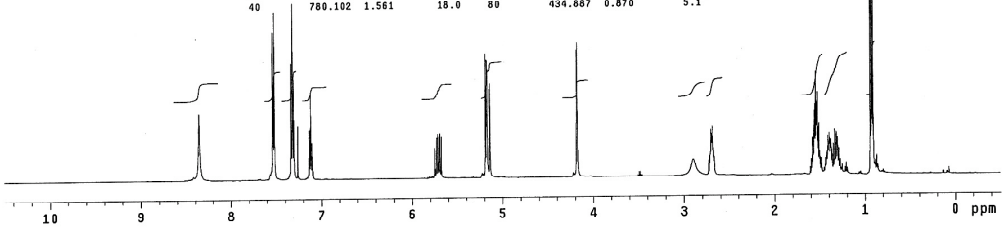


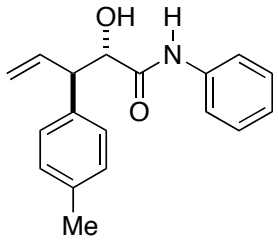
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3334.072	8.335	15.8	41	616.763	1.542	11.4
2	3017.552	7.543	62.4	42	615.299	1.538	11.9
3	3010.234	7.525	72.9	43	607.981	1.520	9.5
4	2937.416	7.343	49.9	44	500.767	1.252	13.0
5	2935.587	7.339	17.6	45	388.796	0.972	98.9
6	2929.732	7.324	76.4	46	381.478	0.954	200.0
7	2921.882	7.304	46.8	47	374.159	0.935	87.7
8	2903.752	7.259	62.9	48	355.498	0.889	8.2
9	2854.719	7.136	24.9				
10	2847.035	7.117	40.6				
11	2839.716	7.099	18.0				
12	2301.816	5.754	15.2				
13	2293.400	5.733	16.4				
14	2291.571	5.729	17.8				
15	2284.618	5.711	19.9				
16	2283.155	5.706	19.2				
17	2276.202	5.690	19.6				
18	2274.373	5.686	20.0				
19	2265.956	5.665	19.0				
20	2087.388	5.218	29.9				
21	2085.559	5.214	41.4				
22	2076.777	5.192	67.3				
23	2075.313	5.188	60.0				
24	2059.213	5.148	31.7				
25	1681.585	4.204	31.8				
26	1095.750	2.739	21.6				
27	1042.326	2.606	16.0				
28	1037.203	2.593	14.4				
29	1033.544	2.584	16.7				
30	677.140	1.693	8.1				
31	671.285	1.678	13.4				
32	669.821	1.674	10.8				
33	663.601	1.659	23.9				
34	657.746	1.644	18.3				
35	656.282	1.641	20.8				
36	650.428	1.626	16.4				
37	637.986	1.595	15.9				
38	630.302	1.576	19.2				
39	628.838	1.572	17.7				
40	621.520	1.554	18.1				



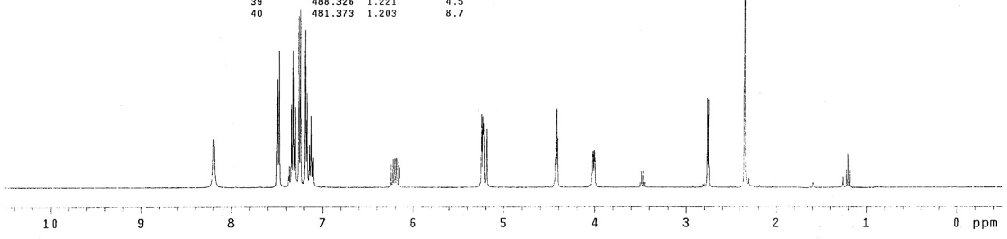


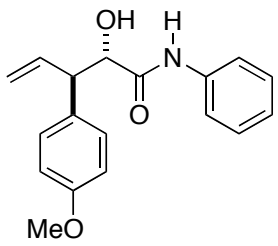
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	4177.075	8.358	16.9	41	777.661	1.556	12.4				
2	3765.942	7.536	37.2	42	773.755	1.548	26.0				
3	3758.130	7.520	42.4	43	769.848	1.540	18.6				
4	3665.645	7.335	23.4	44	767.407	1.536	15.2				
5	3650.032	7.320	44.3	45	764.966	1.531	20.9				
6	3650.220	7.304	25.5	46	760.571	1.522	12.2				
7	3628.735	7.261	13.6	47	755.688	1.512	13.4				
8	3564.282	7.132	12.6	48	751.782	1.504	6.3				
9	3556.470	7.117	21.3	49	747.387	1.496	4.6				
10	3549.145	7.102	9.3	50	742.505	1.486	4.4				
11	2872.387	5.748	7.8	51	720.044	1.441	4.3				
12	2864.087	5.731	9.6	52	714.673	1.430	5.7				
13	2862.134	5.727	10.3	53	712.720	1.426	6.0				
14	2855.298	5.713	11.3	54	706.860	1.414	10.0				
15	2853.833	5.711	10.4	55	701.489	1.404	8.7				
16	2846.997	5.697	11.0	56	699.048	1.399	10.8				
17	2845.044	5.693	11.4	57	697.095	1.395	9.3				
18	2836.255	5.675	10.4	58	694.165	1.389	8.2				
19	2591.137	5.185	31.4	59	691.723	1.384	8.9				
20	2585.278	5.173	29.8	60	689.770	1.380	9.1				
21	2582.837	5.168	29.7	61	686.352	1.373	4.3				
22	2580.884	5.164	27.2	62	684.399	1.369	6.5				
23	2587.700	5.130	29.7	63	676.098	1.353	8.0				
24	2089.673	4.181	32.1	64	668.774	1.338	11.6				
25	2086.255	4.175	33.8	65	666.333	1.333	8.1				
26	1449.536	2.901	4.3	66	661.938	1.325	11.0				
27	1361.645	2.725	4.0	67	659.497	1.320	10.9				
28	1356.274	2.714	5.9	68	655.102	1.311	8.6				
29	1352.856	2.707	11.9	69	652.661	1.306	9.6				
30	1349.438	2.700	10.8	70	648.266	1.297	5.8				
31	1347.973	2.697	10.8	71	645.825	1.292	6.9				
32	1344.067	2.689	12.6	72	630.909	1.279	4.1				
33	1341.137	2.684	7.1	73	604.809	1.210	3.3				
34	1335.278	2.672	5.0	74	470.532	0.942	67.9				
35	796.704	1.594	4.2	75	463.208	0.927	134.6				
36	793.286	1.587	2.9	76	458.790	0.920	16.0				
37	789.868	1.581	9.4	77	455.884	0.912	62.1				
38	787.427	1.576	6.0	78	445.630	0.892	3.2				
39	783.520	1.568	13.8	79	442.212	0.885	3.8				
40	780.102	1.561	18.0	80	434.887	0.870	5.1				



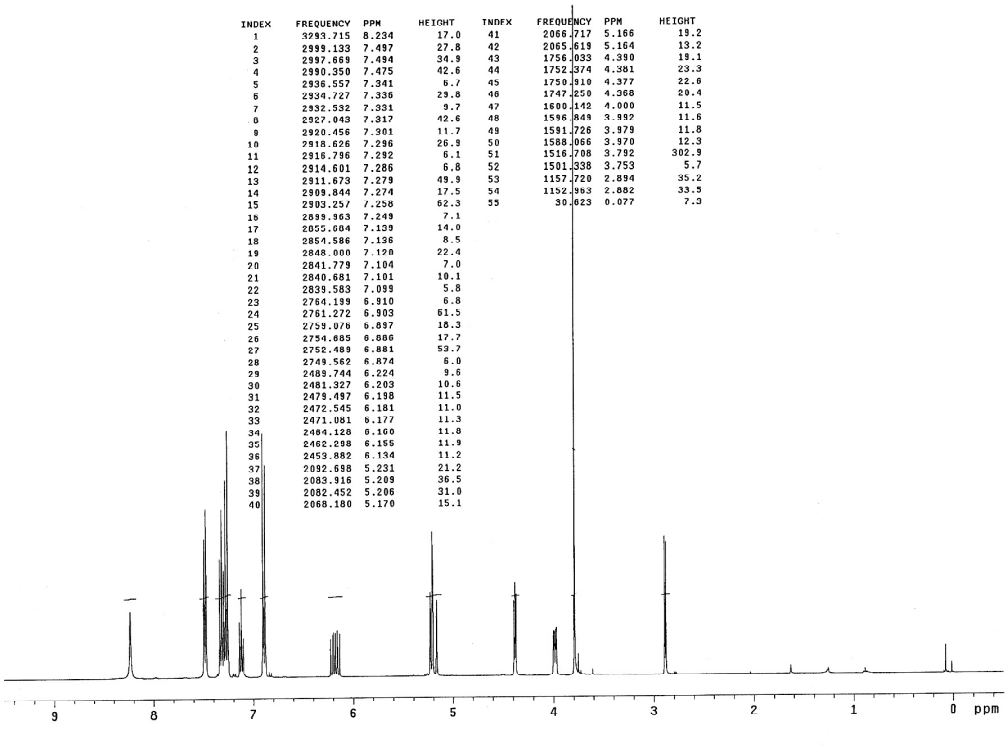


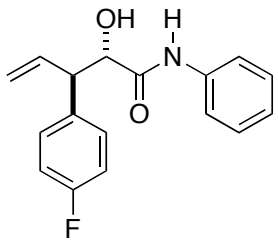
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3279.550	8.198	12.4	41	474.421	1.186	4.3
2	2997.793	7.494	27.0				
3	2990.109	7.475	34.4				
4	2945.467	7.363	5.5				
5	2935.587	7.339	20.6				
6	2927.903	7.319	34.6				
7	2919.852	7.299	20.0				
8	2903.752	7.259	43.1				
9	2895.702	7.239	44.8				
10	2875.576	7.189	39.6				
11	2867.892	7.169	23.6				
12	2855.817	7.139	10.8				
13	2848.498	7.121	17.9				
14	2841.180	7.103	7.7				
15	2497.948	6.245	6.0				
16	2489.167	6.223	7.3				
17	2487.703	6.219	7.4				
18	2480.750	6.202	7.7				
19	2478.921	6.197	7.5				
20	2472.334	6.180	7.6				
21	2470.505	6.176	7.7				
22	2462.089	6.155	6.6				
23	2096.902	5.242	16.3				
24	2091.040	5.227	17.9				
25	2087.022	5.217	16.1				
26	2079.849	5.184	14.8				
27	1772.699	4.431	12.7				
28	1768.674	4.421	19.6				
29	1764.649	4.411	12.8				
30	1610.231	4.025	9.1				
31	1606.938	4.017	9.2				
32	1601.815	4.004	9.5				
33	1598.156	3.995	8.9				
34	1393.242	3.483	4.1				
35	1386.289	3.466	4.2				
36	1105.630	2.764	22.3				
37	1100.873	2.752	22.0				
38	938.771	2.347	121.6				
39	488.326	1.221	4.5				
40	481.373	1.203	8.7				





INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3293.715	8.234	17.0	41	2066.717	5.166	19.2
2	2999.133	7.497	27.8	42	2065.619	5.164	13.2
3	2997.669	7.494	34.9	43	1756.033	4.390	19.1
4	2990.350	7.475	42.6	44	1752.374	4.381	23.3
5	2936.557	7.341	6.7	45	1750.910	4.377	22.6
6	2934.727	7.336	29.8	46	1747.250	4.368	20.4
7	2932.532	7.331	9.7	47	1600.142	4.000	11.5
8	2927.043	7.317	42.6	48	1596.848	3.992	11.6
9	2920.456	7.301	11.7	49	1591.726	3.979	11.8
10	2918.626	7.296	26.9	50	1588.066	3.970	12.3
11	2916.796	7.292	6.1	51	1516.708	3.792	302.9
12	2914.601	7.286	6.8	52	1501.338	3.753	5.7
13	2911.673	7.279	49.9	53	1157.720	2.894	35.2
14	2909.844	7.274	17.5	54	1152.983	2.882	33.5
15	2903.257	7.258	62.3	55	30.623	0.077	7.3
16	2899.963	7.249	7.1				
17	2855.604	7.139	14.0				
18	2854.586	7.136	8.5				
19	2848.000	7.120	22.4				
20	2841.779	7.104	7.0				
21	2840.681	7.101	10.1				
22	2839.583	7.099	5.8				
23	2764.199	6.910	6.8				
24	2761.272	6.903	61.5				
25	2759.078	6.897	18.3				
26	2754.685	6.886	17.7				
27	2752.489	6.881	53.7				
28	2749.562	6.874	6.0				
29	2493.744	6.224	9.6				
30	2481.327	6.203	10.6				
31	2479.497	6.198	11.5				
32	2472.545	6.181	11.0				
33	2471.081	6.177	11.3				
34	2464.128	6.160	11.8				
35	2462.298	6.155	11.9				
36	2459.882	6.134	11.2				
37	2092.698	5.231	21.2				
38	2083.916	5.209	36.5				
39	2082.452	5.206	31.0				
40	2068.180	5.170	15.1				





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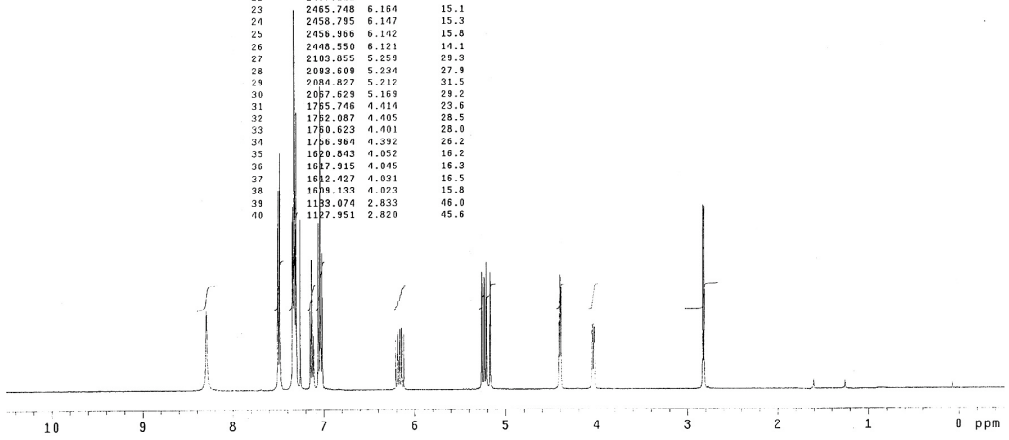
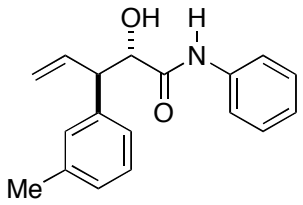
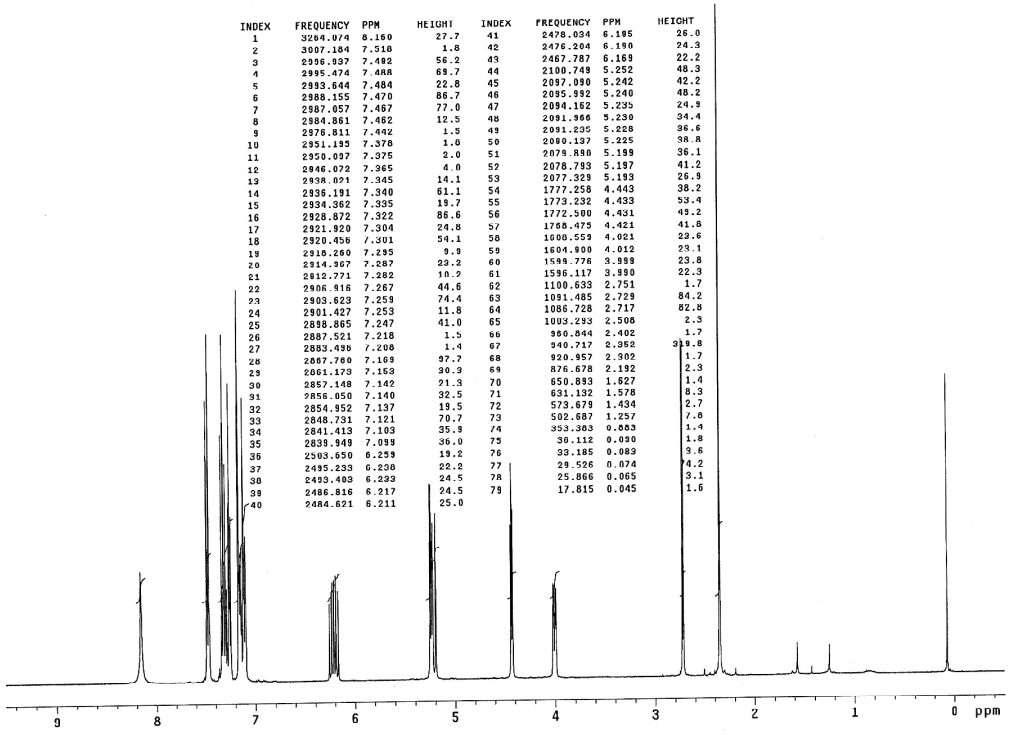
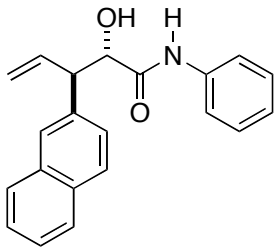
1	3316.874	8.292	20.0
2	3001.086	7.502	49.8
3	2993.768	7.484	58.6
4	2937.782	7.344	41.9
5	2935.507	7.339	45.7
6	2933.381	7.333	22.9
7	2930.098	7.325	94.6
8	2927.171	7.318	50.2
9	2921.682	7.304	69.1
10	2903.752	7.259	42.6
11	2860.208	7.150	19.9
12	2852.889	7.132	32.3
13	2845.571	7.114	13.7
14	2825.445	7.063	41.5
15	2823.250	7.058	14.7
16	2816.663	7.041	75.7
17	2810.077	7.025	12.6
18	2807.881	7.018	34.1
19	2484.410	6.211	12.6
20	2475.994	6.190	13.6
21	2474.164	6.185	14.8
22	2467.211	6.168	15.6
23	2465.748	6.164	15.1
24	2458.795	6.147	15.3
25	2456.966	6.142	15.8
26	2448.550	6.121	14.1
27	2103.055	5.259	29.9
28	2093.609	5.234	27.9
29	2084.827	5.212	31.5
30	2067.629	5.169	29.2
31	1765.746	4.414	23.6
32	1762.087	4.405	28.5
33	1760.623	4.401	28.0
34	1756.964	4.392	26.2
35	1620.843	4.052	16.2
36	1617.915	4.045	16.3
37	1612.427	4.031	16.5
38	1609.133	4.023	15.8
39	1133.074	2.833	46.0
40	1127.951	2.820	45.6



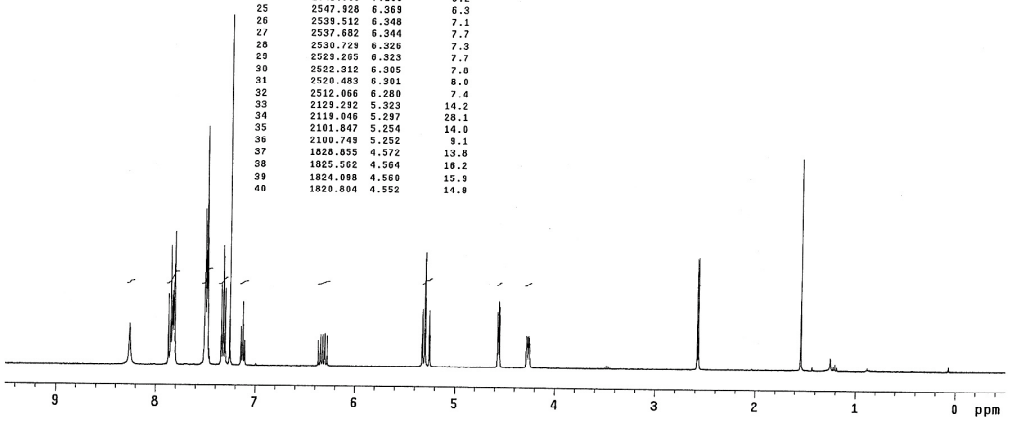


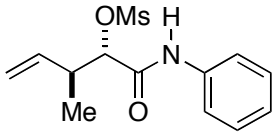


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3264.074	8.160	27.7	41	2478.034	6.195	26.0
2	3007.184	7.516	1.8	42	2476.204	6.190	24.3
3	2996.937	7.492	56.2	43	2467.787	6.169	22.2
4	2995.474	7.488	69.7	44	2100.749	5.252	48.3
5	2993.644	7.484	22.8	45	2097.090	5.242	42.2
6	2988.155	7.470	86.7	46	2095.992	5.240	48.2
7	2987.057	7.467	77.0	47	2094.162	5.235	24.9
8	2984.861	7.462	12.5	48	2091.966	5.230	34.4
9	2976.811	7.442	1.5	49	2091.235	5.228	36.6
10	2951.195	7.378	1.0	50	2090.137	5.225	38.8
11	2950.097	7.375	2.0	51	2079.880	5.199	36.1
12	2946.072	7.365	4.0	52	2078.793	5.197	41.2
13	2938.021	7.345	14.1	53	2077.329	5.193	26.9
14	2936.191	7.340	61.1	54	1777.258	4.443	38.2
15	2934.362	7.335	19.7	55	1773.232	4.433	53.4
16	2928.872	7.322	86.6	56	1772.500	4.431	49.2
17	2921.920	7.304	24.8	57	1768.475	4.421	41.8
18	2920.456	7.301	54.1	58	1608.559	4.021	23.6
19	2918.260	7.295	9.9	59	1604.900	4.012	23.1
20	2914.967	7.287	23.2	60	1599.776	3.999	23.8
21	2912.771	7.282	10.2	61	1596.117	3.990	22.3
22	2906.916	7.267	44.6	62	1100.633	2.751	1.7
23	2903.623	7.259	74.4	63	1091.485	2.729	84.2
24	2901.427	7.253	11.8	64	1086.728	2.717	82.8
25	2898.865	7.247	41.0	65	1003.293	2.508	2.3
26	2887.521	7.218	1.5	66	960.844	2.402	1.7
27	2883.498	7.208	1.4	67	940.717	2.352	319.8
28	2867.760	7.169	97.7	68	920.957	2.302	1.7
29	2861.173	7.153	30.9	69	876.678	2.192	2.3
30	2857.148	7.142	21.3	70	650.883	1.627	1.4
31	2856.050	7.140	32.5	71	631.132	1.578	8.3
32	2854.952	7.137	19.5	72	573.679	1.434	2.7
33	2848.731	7.121	70.7	73	502.687	1.257	7.8
34	2841.413	7.103	35.9	74	353.383	0.883	1.4
35	2839.949	7.099	36.0	75	36.112	0.090	1.8
36	2503.650	6.259	19.2	76	33.185	0.083	3.6
37	2495.233	6.238	22.2	77	29.526	0.074	74.2
38	2493.403	6.233	24.5	78	25.866	0.065	3.1
39	2486.816	6.217	24.5	79	17.815	0.045	1.6
40	2484.621	6.211	25.0				



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3304.693	8.261	10.1	41	1712.120	4.280	7.9
2	3149.169	7.872	17.3	42	1709.193	4.273	7.7
3	3140.752	7.851	29.5	43	1703.703	4.259	7.9
4	3134.897	7.837	17.1	44	1700.410	4.251	7.5
5	3131.603	7.829	18.1	45	1032.203	2.580	27.0
6	3125.382	7.813	33.1	46	1027.445	2.568	27.9
7	3009.013	7.522	3.0	47	618.324	1.546	52.2
8	3003.890	7.509	38.5	48	500.857	1.252	3.0
9	3002.426	7.506	35.5	49	482.194	1.205	1.6
10	3000.597	7.501	28.1				
11	2996.571	7.491	58.8				
12	2993.278	7.483	31.4				
13	2991.082	7.477	15.8				
14	2986.325	7.465	4.0				
15	2984.495	7.461	1.9				
16	2938.021	7.345	19.8				
17	2935.825	7.339	6.4				
18	2930.336	7.325	29.4				
19	2921.920	7.304	18.7				
20	2903.900	7.260	87.2				
21	2858.246	7.145	9.5				
22	2857.148	7.142	5.8				
23	2850.927	7.127	15.6				
24	2843.608	7.109	6.2				
25	2547.928	6.369	6.3				
26	2539.512	6.348	7.1				
27	2537.682	6.344	7.7				
28	2530.729	6.326	7.3				
29	2523.265	6.323	7.7				
30	2522.312	6.305	7.0				
31	2520.483	6.301	8.0				
32	2512.066	6.280	7.4				
33	2129.232	5.323	14.2				
34	2119.046	5.297	28.1				
35	2101.847	5.254	14.0				
36	2100.749	5.252	9.1				
37	1828.855	4.572	13.8				
38	1825.562	4.564	16.2				
39	1824.098	4.560	15.9				
40	1820.804	4.552	14.8				

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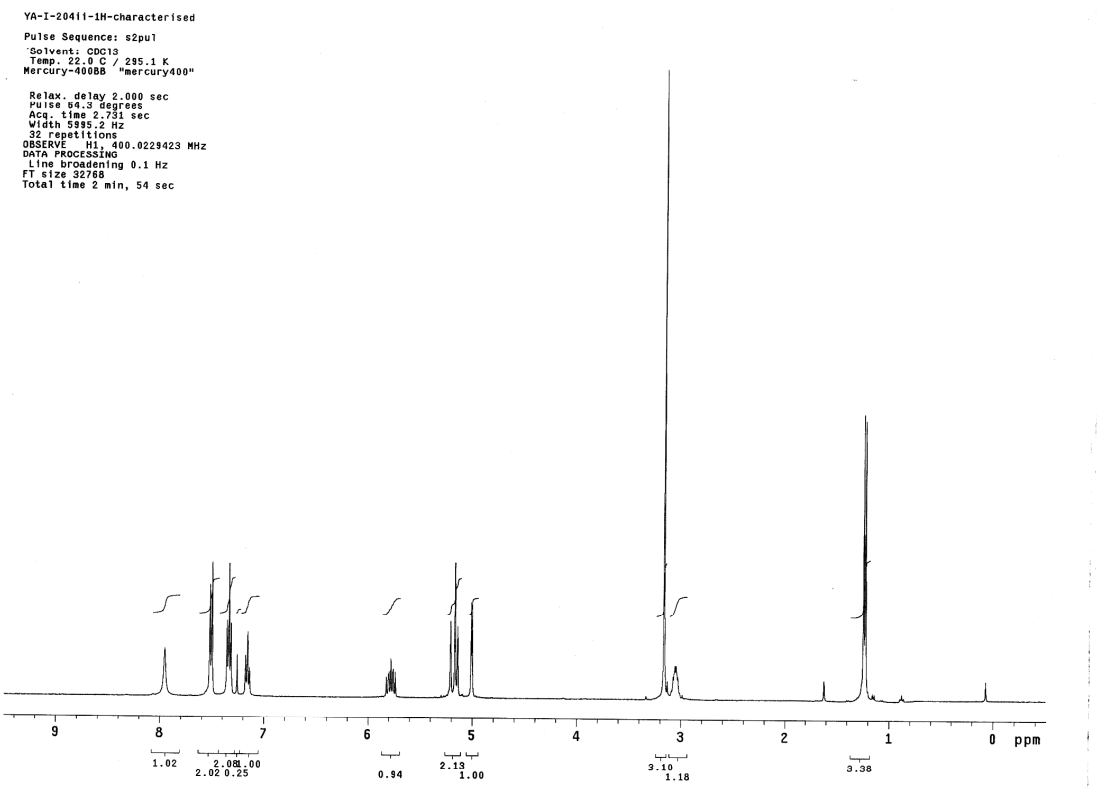
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Solvent: CDCl3
Temp. 22.0 C / 295.1 K
Mercury-400BB "mercury400"
Relax. delay 2.000 sec
Pulse 84.3 degrees
Acq. time 2.731 sec
Width 5995.2 Hz
32 repetitions
OBSERVE H1, 400.0229423 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 2 min, 54 sec
9
8
7
6
5
4
3
2
1
0 ppm
1.02
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3.10
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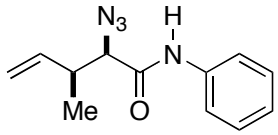
3.38



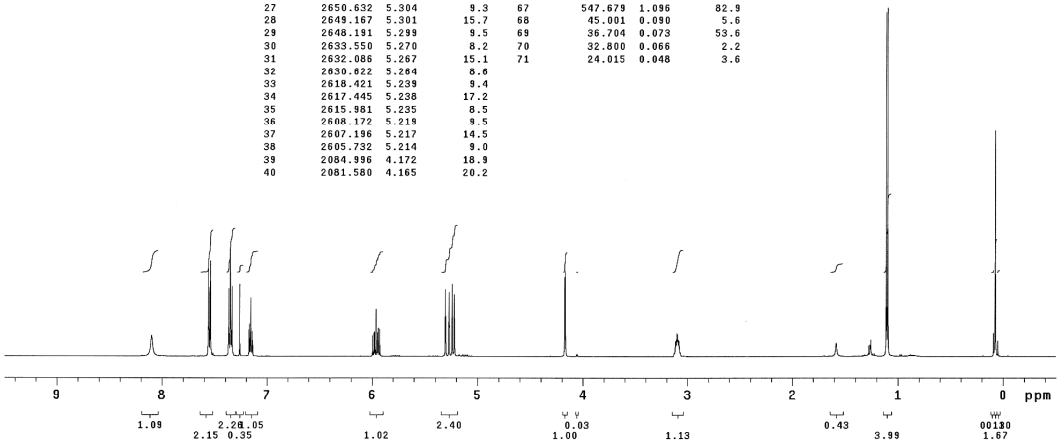
YA-I-226-ent-syn-azide

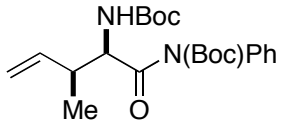
Pulse Sequence: s2pul
Solvent: CDCl3

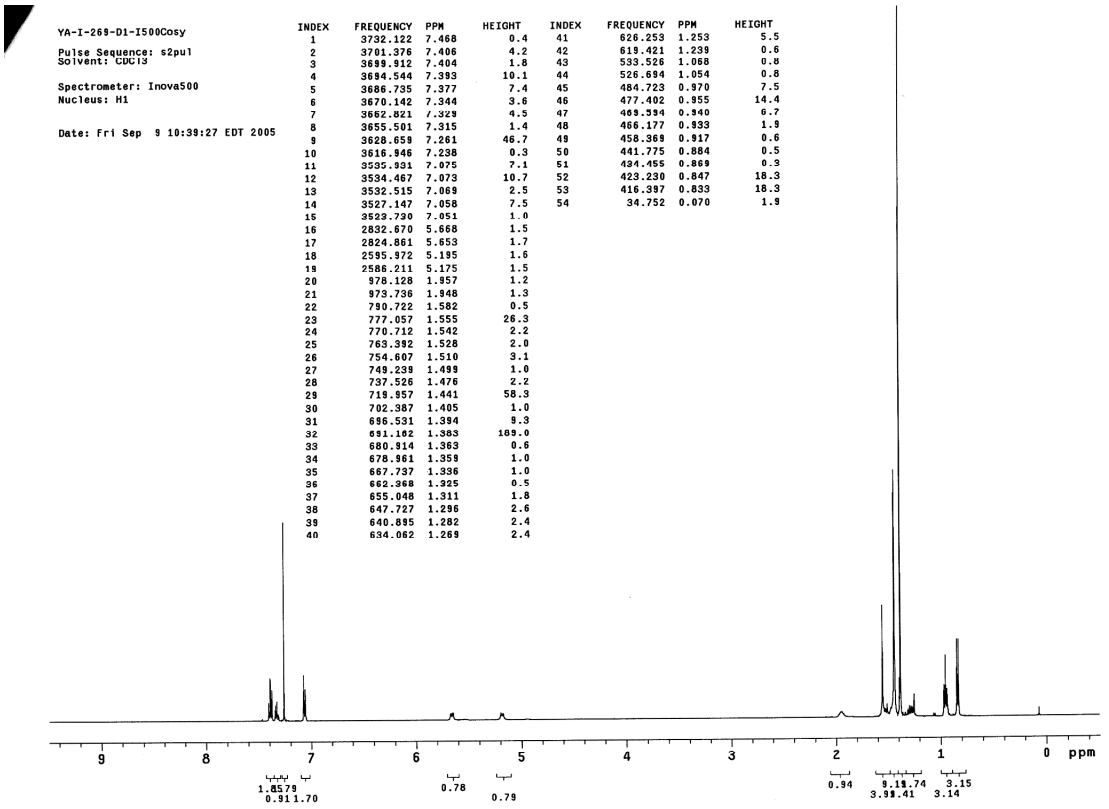
Spectrometer: Inova500
Nucleus: H1

Date: Sat Aug 13 09:12:18 EDT 2005

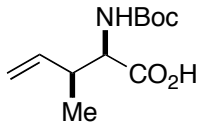
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	4047.394	8.099	5.1	41	1563.285	3.128	0.9
2	3777.510	7.559	21.4	42	1561.332	3.124	0.7
3	3775.557	7.555	6.5	43	1559.868	3.121	1.0
4	3770.189	7.544	22.7	44	1557.916	3.117	2.2
5	3757.500	7.519	0.7	45	1556.452	3.114	3.5
6	3682.342	7.368	15.9	46	1554.988	3.112	2.8
7	3680.390	7.364	5.5	47	1553.036	3.108	3.6
8	3674.534	7.353	24.3	48	1551.084	3.104	4.1
9	3668.189	7.340	6.3	49	1549.620	3.101	5.5
10	3666.237	7.336	16.7	50	1548.155	3.098	4.2
11	3664.285	7.332	2.5	51	1546.203	3.094	5.3
12	3661.845	7.327	0.9	52	1544.739	3.091	4.1
13	3629.146	7.262	17.1	53	1542.787	3.087	3.9
14	3583.759	7.171	7.7	54	1541.323	3.084	2.9
15	3582.783	7.169	4.9	55	1539.371	3.080	3.5
16	3580.343	7.164	1.1	56	1537.907	3.077	2.3
17	3576.438	7.156	13.8	57	1535.955	3.073	1.1
18	3570.094	7.144	3.6	58	1534.490	3.071	0.8
19	3569.118	7.142	6.0	59	1532.538	3.067	0.9
20	2997.138	5.997	5.0	60	791.210	1.583	3.0
21	2990.305	5.984	5.7	61	639.430	1.280	2.5
22	2986.401	5.976	5.9	62	632.598	1.266	2.7
23	2979.569	5.962	11.2	63	627.717	1.256	4.0
24	2973.224	5.949	5.6	64	617.468	1.236	0.7
25	2969.320	5.942	6.8	65	610.636	1.222	0.7
26	2962.487	5.928	6.5	66	554.512	1.110	81.8
27	2650.632	5.304	9.3	67	547.679	1.096	82.9
28	2649.167	5.301	15.7	68	45.001	0.090	5.6
29	2648.191	5.299	9.5	69	36.704	0.073	53.6
30	2633.550	5.270	8.2	70	32.800	0.066	2.2
31	2632.086	5.267	15.1	71	24.015	0.048	3.6
32	2630.622	5.264	8.6				
33	2618.421	5.239	9.4				
34	2617.445	5.238	17.2				
35	2615.981	5.235	8.5				
36	2608.172	5.219	9.5				
37	2607.196	5.217	14.5				
38	2605.732	5.214	9.0				
39	2084.996	4.172	18.9				
40	2081.580	4.165	20.2				

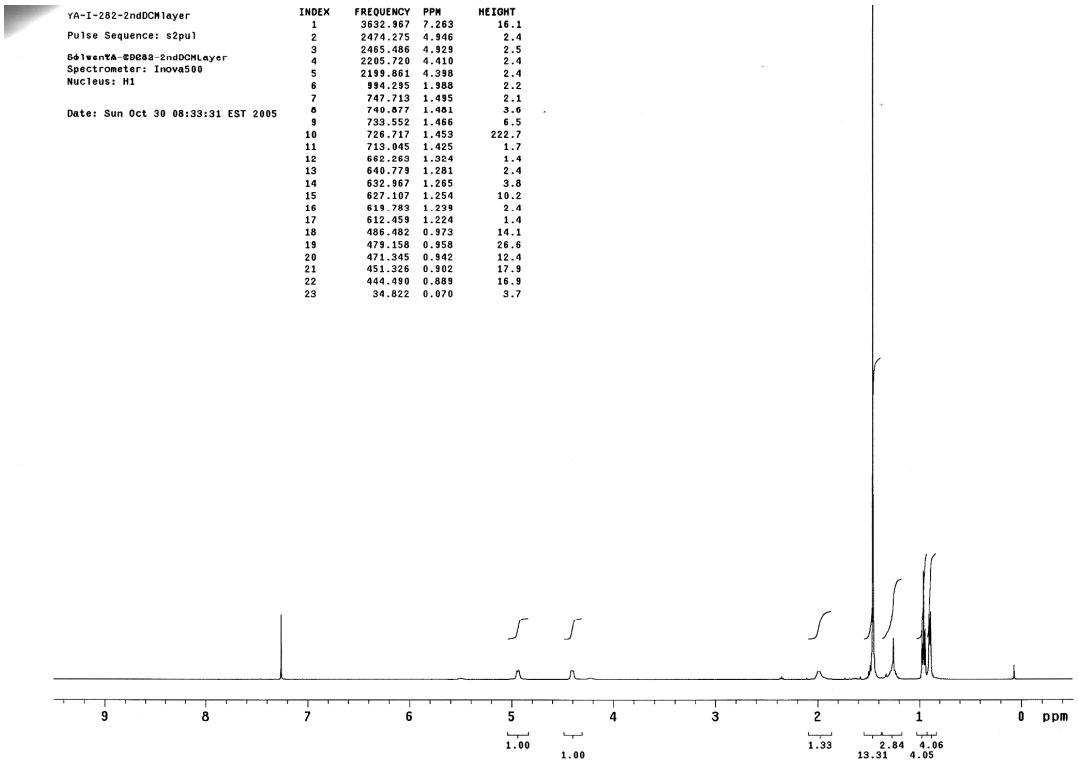


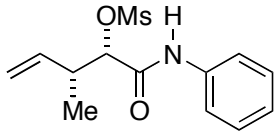




INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3732.122	7.468	0.4	41	626.253	1.253	5.5
2	3701.376	7.406	4.2	42	619.421	1.239	0.6
3	3689.912	7.404	1.8	43	533.526	1.068	0.8
4	3684.544	7.393	10.1	44	526.694	1.054	0.8
5	3686.735	7.377	7.4	45	484.723	0.970	7.5
6	3670.142	7.344	3.6	46	477.402	0.955	14.4
7	3662.821	7.329	4.5	47	469.534	0.940	6.7
8	3655.501	7.315	1.4	48	466.177	0.933	1.9
9	3628.658	7.261	46.7	49	458.369	0.917	0.6
10	3616.946	7.238	0.3	50	441.775	0.884	0.5
11	3535.931	7.075	7.1	51	434.455	0.869	0.9
12	3534.467	7.073	10.7	52	423.230	0.847	18.3
13	3532.515	7.069	2.5	53	416.397	0.833	18.3
14	3527.147	7.058	7.5	54	34.752	0.070	1.9
15	3523.730	7.051	1.0				
16	2832.670	5.668	1.5				
17	2824.861	5.653	1.7				
18	2595.972	5.195	1.6				
19	2586.211	5.175	1.5				
20	978.128	1.957	1.2				
21	973.736	1.948	1.3				
22	790.722	1.582	0.5				
23	777.057	1.555	26.3				
24	770.712	1.542	2.2				
25	763.392	1.528	2.0				
26	754.607	1.510	3.1				
27	749.238	1.499	1.0				
28	737.526	1.476	2.2				
29	719.957	1.441	58.3				
30	702.387	1.405	1.0				
31	696.531	1.394	9.3				
32	691.162	1.383	189.0				
33	680.914	1.363	0.6				
34	678.961	1.359	1.0				
35	667.737	1.336	1.0				
36	662.368	1.325	0.5				
37	655.048	1.311	1.8				
38	647.727	1.296	2.6				
39	640.885	1.282	2.4				
40	634.062	1.269	2.4				


NHBoc
CO2H
Me


YA-I-282-2ndDCMlayer
Pulse Sequence: s2pul
Solvent: CDCl3
Spectrometer: Inova500
Nucleus: H1
Date: Sun Oct 30 08:33:31 EST 2005
INDEX FREQUENCY PPM HEIGHT
1 3632.967 7.263 16.1
2 2474.275 4.946 2.4
3 2465.486 4.929 2.5
4 2205.720 4.410 2.4
5 2199.861 4.398 2.4
6 994.295 1.988 2.2
7 747.713 1.495 2.1
8 740.877 1.481 3.6
9 733.552 1.466 6.5
10 726.717 1.453 222.7
11 713.045 1.425 1.7
12 662.263 1.324 1.4
13 640.779 1.281 2.4
14 632.967 1.265 3.8
15 627.107 1.254 10.2
16 619.783 1.239 2.4
17 612.459 1.224 1.4
18 486.482 0.973 14.1
19 479.158 0.958 26.6
20 471.345 0.942 12.4
21 451.326 0.902 17.9
22 444.490 0.889 16.9
23 34.822 0.070 3.7
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.33
13.31
2.84
4.05
4.06



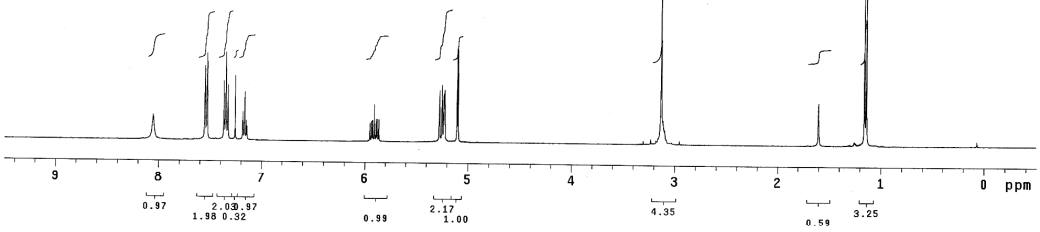
YA-I-232-1H-characterised

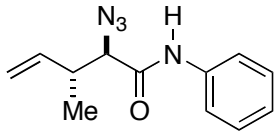
Pulse Sequence: s2pul

Solvent: CDCl3
Spectrometer:Mercury400
Nucleus: H1

Date: Mon Aug 15 10:04:05 EDT 2005

INDEX	FREQUENCY	PPM	HEIGHT
1	3222.101	8.055	6.1
2	3022.309	7.555	14.7
3	3021.211	7.553	18.1
4	3013.527	7.533	21.1
5	2948.028	7.370	14.3
6	2946.188	7.365	5.3
7	2940.344	7.350	21.4
8	2933.757	7.334	6.4
9	2932.293	7.330	13.3
10	2903.752	7.259	15.6
11	2873.746	7.184	6.9
12	2866.428	7.166	11.7
13	2860.207	7.150	3.3
14	2859.110	7.147	4.9
15	2380.469	5.951	4.3
16	2374.268	5.935	4.7
17	2370.243	5.925	5.1
18	2363.656	5.909	9.0
19	2357.070	5.892	5.5
20	2353.045	5.882	5.5
21	2346.458	5.866	5.4
22	2110.441	5.276	7.8
23	2109.345	5.273	12.4
24	2107.880	5.269	8.5
25	2099.829	5.249	9.3
26	2098.732	5.247	13.8
27	2091.779	5.228	11.7
28	2090.316	5.225	10.7
29	2089.584	5.224	10.8
30	2088.486	5.221	12.5
31	2041.648	5.104	21.0
32	2037.989	5.095	22.5
33	1251.997	3.130	151.2
34	1245.410	3.113	5.6
35	1242.483	3.106	4.0
36	1240.653	3.101	3.3
37	1238.824	3.097	3.4
38	641.645	1.604	10.3
39	463.077	1.158	58.6
40	456.491	1.141	58.4





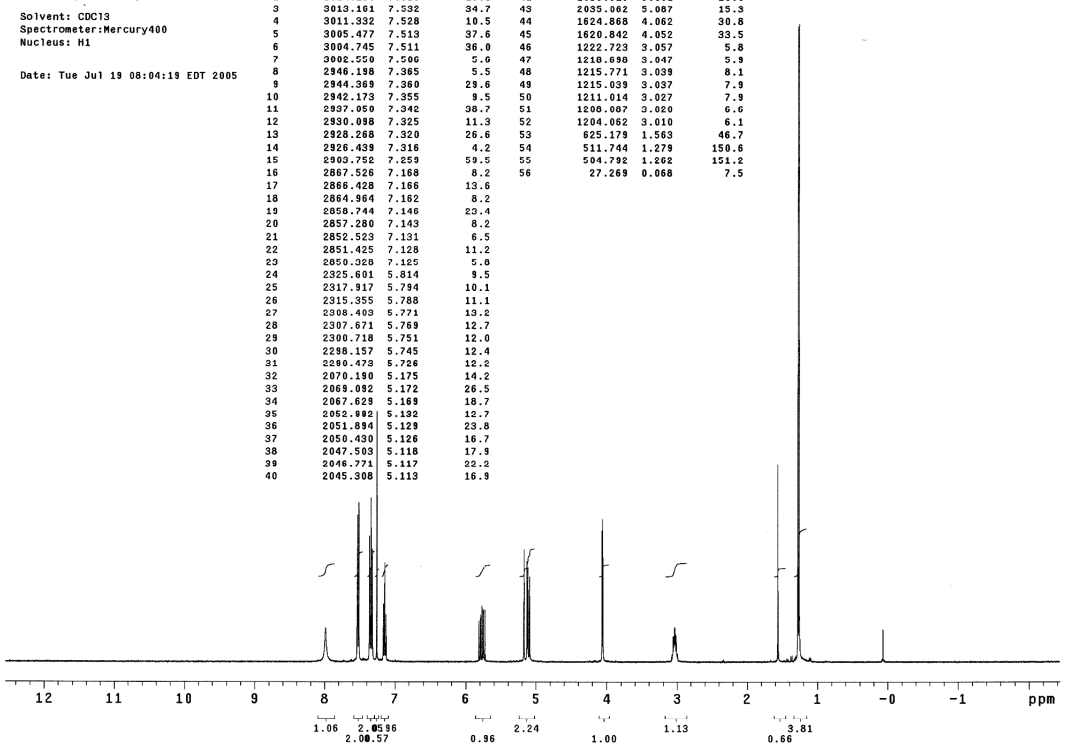
YA-I-217-azide-1H-characterised

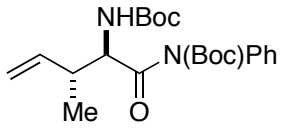
Pulse Sequence: s2pul

Solvent: CDCl3
Spectrometer:Mercury400
Nucleus: H1

Date: Tue Jul 19 08:04:19 EDT 2005

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3194.657	7.986	7.9	41	2037.257	5.093	17.3
2	3014.259	7.535	26.8	42	2036.526	5.091	20.0
3	3013.161	7.532	34.7	43	2035.062	5.087	15.3
4	3011.332	7.528	10.5	44	1624.868	4.062	30.8
5	3005.477	7.513	37.6	45	1620.842	4.052	33.5
6	3004.745	7.511	36.0	46	1222.723	3.057	5.8
7	3002.550	7.506	5.6	47	1210.698	3.047	5.9
8	2946.198	7.365	5.5	48	1215.771	3.039	8.1
9	2944.369	7.360	29.6	49	1215.039	3.037	7.9
10	2942.173	7.355	9.5	50	1211.014	3.027	7.9
11	2937.050	7.342	38.7	51	1208.087	3.020	6.6
12	2930.098	7.325	11.3	52	1204.062	3.010	6.1
13	2928.268	7.320	26.6	53	625.179	1.563	46.7
14	2926.439	7.316	4.2	54	511.744	1.279	150.6
15	2903.752	7.259	59.5	55	504.792	1.262	151.2
16	2867.526	7.168	8.2	56	27.269	0.068	7.5
17	2866.428	7.166	13.6				
18	2864.964	7.162	8.2				
19	2858.744	7.146	23.4				
20	2857.280	7.143	8.2				
21	2852.523	7.131	6.5				
22	2851.425	7.128	11.2				
23	2850.328	7.125	5.8				
24	2325.601	5.814	9.5				
25	2317.917	5.794	10.1				
26	2315.355	5.788	11.1				
27	2308.403	5.771	13.2				
28	2307.671	5.769	12.7				
29	2300.718	5.751	12.0				
30	2298.157	5.745	12.4				
31	2290.473	5.726	12.2				
32	2070.190	5.175	14.2				
33	2069.092	5.172	26.5				
34	2067.629	5.169	18.7				
35	2052.992	5.132	12.7				
36	2051.894	5.129	23.8				
37	2050.430	5.126	16.7				
38	2047.503	5.118	17.9				
39	2046.771	5.117	22.2				
40	2045.308	5.113	16.9				



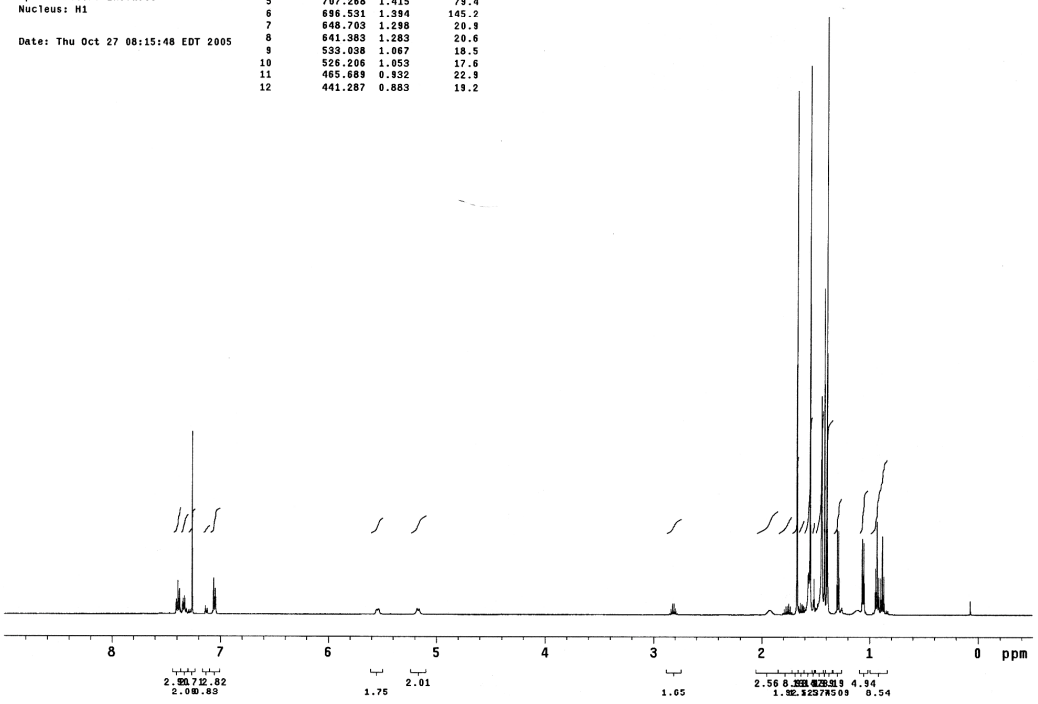


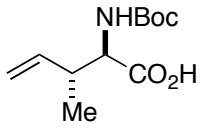
YA-I-254-column-1stfracs

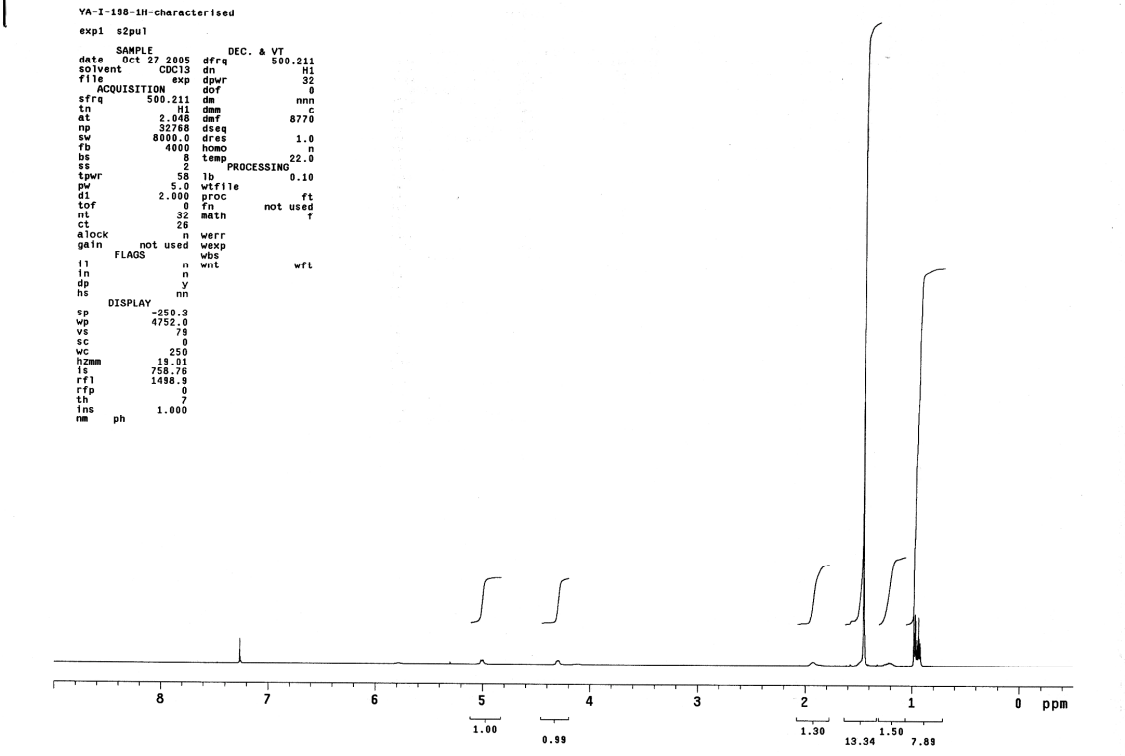
Pulse Sequence: s2pul
Solvent: CDCl3
File: YA-I-254-column-1stfracs
Spectrometer: Inova500
Nucleus: H1

Date: Thu Oct 27 08:15:48 EDT 2005

INDEX	FREQUENCY	PPM	HEIGHT
1	3629.147	7.262	44.6
2	836.621	1.672	127.1
3	775.105	1.551	133.3
4	720.933	1.443	53.1
5	707.268	1.415	79.4
6	696.531	1.394	145.2
7	648.703	1.298	20.8
8	641.383	1.283	20.6
9	533.038	1.067	18.5
10	526.206	1.053	17.6
11	465.689	0.932	22.9
12	441.287	0.883	19.2




NHBoc
CO2H
Me


YA-I-198-1H-characterised
exp1 s2pul
SAMPLE
date Oct 27 2005
solvent CDCl3
file exp
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tn H1
at 2.048
np 32768
sw 8000.0
fb 4000
bs 8
ss 2
tpwr 58
pw 5.0
d1 2.000
tof 0
nt 32
ct 26
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.3
wp 4752.0
vs 79
sc 0
wc 250
hzmm 19.01
is 758.76
rfl 1498.9
rfp 0
th 7
ins 1.000
nm ph
DEC. & VT
dfrq 500.211
dn H1
dpwr 32
dof 0
dm nnn
dmm c
dmf 8770
dseq
dres 1.0
homo n
temp 22.0
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
8
7
6
5
4
3
2
1
0
ppm
1.00
0.99
1.30
13.34
1.50
7.88