# Supporting Information

## Aza-Baylis-Hillman Reactions of *N*-Tosylated Imines with Activated Allenes and Alkynes in the Presence of Various Lewis Base Promoters

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**General Remarks**. Melting points are uncorrected. $^{1}H$ and $^{13}C$ NMR spectra were recorded at 300 and 75 MHz respectively. Mass spectra were recorded by EI, MALDI and ESI methods, and HRMS was measured by EI method. Organic solvents used were dried by standard methods when necessary. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC plates. Flash column chromatography was carried out using silica gel or $Al_2O_3$ at increased pressure. The starting materials such as *N*-tosylated imines,[1] ethyl 2,3-butadienoate,[2] and penta-3,4-dien-2-one[3] were prepared according to the literatures.


1) Love, B. E.; Raje, P. S.; Williams, T. C. *Synlett* **1994**, 493.

2) (a) Anderson, J. C.; Cubbon, R. J.; Harling, J. D. *Tetrahedron: Asymmetry* **2001**, *12*, 923.
   (b) Jansch, H.; Kannenberg, S.; Boche, G. *Eur. J. Org. Chem.* **2001**, 2923.

3) Buono, G. *Synthesis* **1981**, 272.

1) Typical Reaction Procedures.

**Aza-Baylis-Hillman reaction of *N*-tosylated aldimines with ethyl 2,3-butadienoate catalyzed by DABCO**.

**Typical reaction procedure of *N*-tosylated aldimines with ethyl 2,3-butadienoate at room temperature**.

To a Schlenk tube with *N*-(*p*-methylbenzenesulfonyl)benzaldimine (65 mg, 0.25 mmol) and DABCO (6 mg, 0.05 mmol) and MS 4A (100 mg) in benzene (0.5 mL) was added ethyl 2,3-butadienoate (34 mg, 0.30 mmol) and the reaction mixture was stirred for 1 h at room temperature (20 $^{o}$C). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/6~1/4) to give adduct **1a** (75 mg, yield 82%) as a white solid.

**Aza-Baylis-Hillman reaction of *N*-tosylated aldimines with ethyl 2,3-butadienoate catalyzed by DMAP**.

**Typical reaction procedure of *N*-tosylated aldimines with ethyl 2,3-butadienoate at room temperature**. To a Schlenk tube with *N*-(*p*-methylbenzenesulfonyl)benzaldimine (65 mg, 0.25 mmol) and DMAP (6 mg, 0.05 mmol) in DCM (0.5 mL) was added ethyl 2,3-butadienoate (34 mg, 0.30 mmol) and the reaction mixture was stirred for 10 min. at room temperature (20 $^{o}$C). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/5) to give adduct **3a** (43 mg, yield 60%) as a white solid.

**Aza-Baylis-Hillman reaction of *N*-tosylated aldimines with ethyl penta-2,3-dienoate catalyzed by $PPhMe_2$**.

**Typical reaction procedure of *N*-tosylated aldimines with ethyl penta-2,3-dienoate at room temperature**. To a Schlenk tube with *N*-(*p*-methylbenzenesulfonyl)benzaldimine (130 mg, 0.5 mmol) in DCM (0.5 mL) was added ethyl penta-2,3-dienoate (63 mg, 0.50 mmol) and

$PPhMe_2$ (7 μL, 0.05 mmol) under argon atmosphere. The reaction mixture was stirred for 24 h at room temperature (20 $^oC$). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/6) to give adduct **4a** (183 mg, yield 95%) as a mixture of *anti* and *syn*-configuration (1 : 13).

**Aza-Baylis-Hillman reaction of *N*-tosylated aldimines with penta-3,4-dien-2-one catalyzed by DABCO**.

**Typical reaction procedure of *N*-tosylated aldimines with penta-3,4-dien-2-one at room temperature**. To a Schlenk tube with *N*-(*p*-methylbenzenesulfonyl)benzaldimine (65 mg, 0.25 mmol) and DABCO (6 mg, 0.05 mmol) in DCM (0.5 mL) was added penta-3,4-dien-2-one (25 mg, 0.30 mmol) and the reaction mixture was stirred for 3 h at room temperature (20 $^oC$). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/4) to give adduct **5a** (37 mg, yield 43%) as a white solid.

**Reaction procedure of *N*-isobutylidene-4-methyl-benzenesulfonamide with ethyl 2,3-butadienoate at room temperature**. To a Schlenk tube with *N*-isobutylidene-4-methyl-benzenesulfonamide (112 mg, 0.50 mmol) and DMAP (6 mg, 0.05 mmol) in DCM (0.5 mL) was added ethyl 2,3-butadienoate (56 mg, 0.50 mmol) and the reaction mixture was stirred for 10 min. at room temperature (20 $^oC$). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/8) to give adduct **5** (34 mg, yield 20%) as a colorless oil.

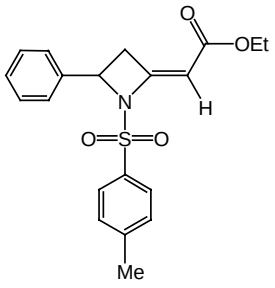
**Aza-Baylis-Hillman reaction of *N*-tosylated aldimines with methyl propiolate catalyzed by DABCO**.

**The reaction procedure of *N*-tosylated aldimines with methyl propiolate at room temperature**. To a Schlenk tube with *N*-(*p*-methylbenzenesulfonyl)benzaldimine (130 mg, 0.5 mmol) and DABCO (6 mg, 0.05 mmol) in DCM (0.5 mL) was added methyl propiolate (84 mg, 1 mmol) and the reaction mixture was stirred for 10 h at room temperature (20 $^{o}$C). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/4~1/2) to give adduct **8** (45 mg, yield 29%) and adduct **9** (52 mg, yield 34%) as white solids.

**Aza-Baylis-Hillman reaction of *N*-tosylated aldimines with but-3-yn-2-one catalyzed by DMAP**.

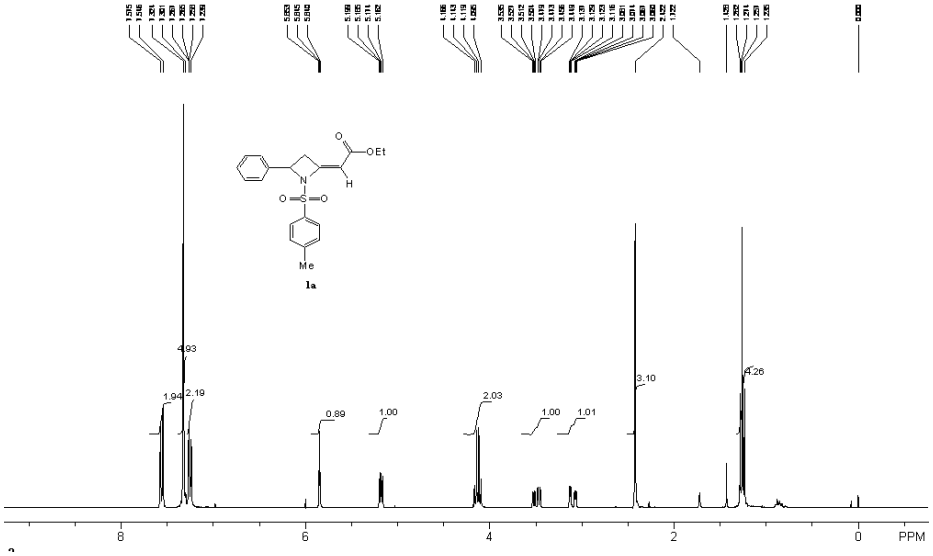
**Typical reaction procedure of *N*-tosylated aldimines with but-3-yn-2-one at room temperature**. To a Schlenk tube with *N*-(*p*-methylbenzenesulfonyl)benzaldimine (130 mg, 0.5 mmol) and DMAP (6 mg, 0.05 mmol) in $CH_3CN$ (0.5 mL) was added but-3-yn-2-one (68 mg, 1 mmol) and the reaction mixture was stirred for 24 h at room temperature (20 $^{o}$C). The reaction mixture was washed with water (3 x 10 mL) and extracted with dichloromethane (2 x 10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum= 1/4~1/3) to give adduct **12** (49 mg, yield 30%) and adduct **11** (19 mg, yield 13%) as white solids.

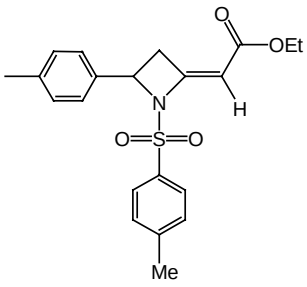
2) *The spectroscopic data of the compounds* **1-13**.




**1a**

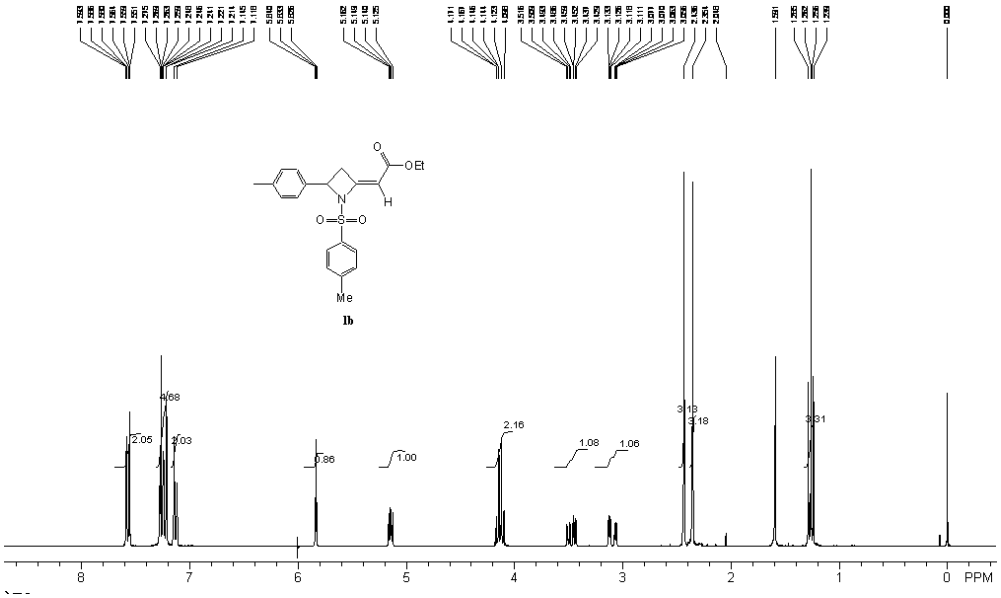
**[4-Phenyl-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]-acetic acid ethyl ester 1a**: a colorless solid. mp. 100-103 $^{o}$C. IR ($CH_2Cl_2$) ν 2982, 1703 (C=O), 1666, 1596, 1358, 1267, 1155, 1040 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.27 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.43 (3H, s, $CH_3$), 3.10 (1H, ddd, *J* = 16.8, 4.2, 2.4 Hz, $CH_2$), 3.49 (1H, ddd, *J* = 16.8, 6.9, 1.8 Hz, $CH_2$), 4.13 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.19 (1H, dd, *J* = 6.9, 4.2 Hz, CH), 5.84-5.85 (1H, m, =CH), 7.26 (2H, d, *J* = 8.4 Hz, ArH), 7.34 (5H, s, ArH), 7.56 (2H, d, *J* = 8.4 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 37.4, 59.7, 66.0, 94.7, 126.7, 127.3, 128.6, 128.8, 129.8, 134.1, 137.2, 144.8, 158.2, 167.2. MS (EI) *m/z* 371 ($M^+$, 2.44), 232 ($M^+$-139, 64.22), 216 ($M^+$-155, 44.63), 155 ($M^+$-216, 51.98), 91 ($M^+$-280, 100). Anal. Calcd. for $C_{20}H_{21}NO_4S$ requires C, 64.68; H, 5.70; N, 3.77%. Found: C, 64.66; H, 5.71; N, 3.56%.

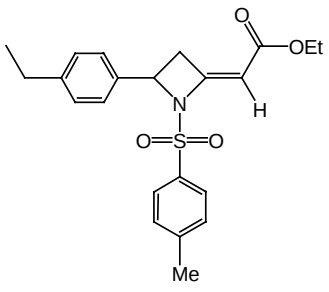





**1b**

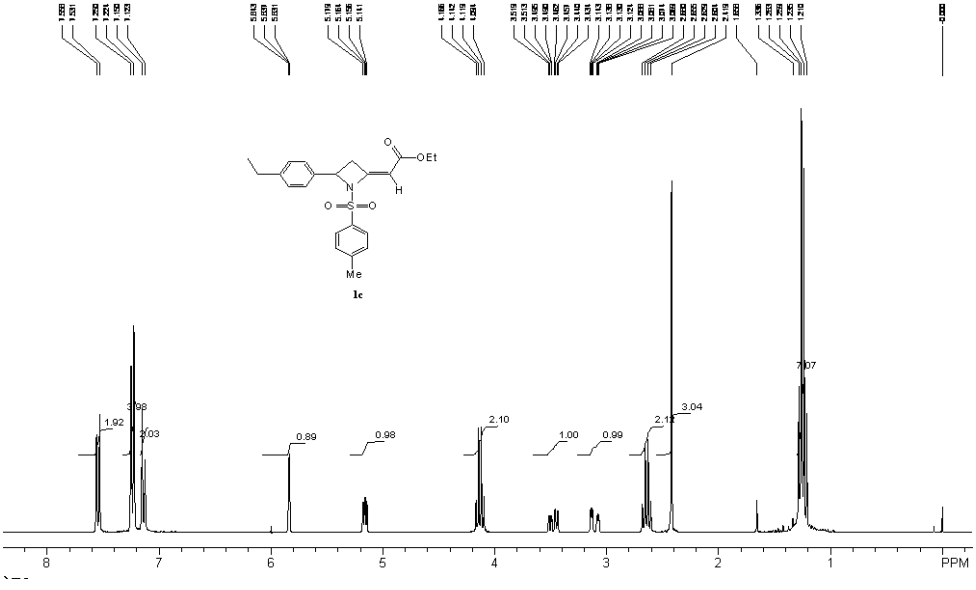
**[1-(Toluene-4-sulfonyl)-4-p-tolyl-azetidin-2-ylidene]acetic acid ethyl ester 1b**: a colorless solid. mp. 135-137 $^{o}$C. IR ($CH_2Cl_2$) ν 2981, 1708 (C=O), 1658, 1597, 1363, 1169, 1121, 1046 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.35 (3H, s, $CH_3$), 2.44 (3H, s, $CH_3$), 3.09 (1H, ddd, *J* = 17.1, 4.5, 2.1 Hz, $CH_2$), 3.47 (1H, ddd, *J* = 17.1, 6.9, 2.4 Hz, $CH_2$), 4.13 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.15 (1H, dd, *J* = 6.9, 4.5 Hz, CH), 5.83-5.84 (1H, m, =CH), 7.13 (2H, d, *J* = 8.1 Hz, ArH), 7.21-7.28 (4H, m, ArH), 7.57 (2H, d, *J* = 8.1 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.0, 21.4, 37.3, 59.5, 65.9, 94.4, 126.5, 127.2, 129.2, 129.6, 134.1, 134.1, 138.6, 144.7, 158.3, 167.1. MS (EI) *m/z* 385 ($M^+$, 0.63), 340 ($M^+$-45, 8.31), 230 ($M^+$-155, 63.12), 157 ($M^+$-228, 89.01), 155 ($M^+$-230, 25.70), 91 ($M^+$-294, 100). Anal. Calcd. for $C_{21}H_{23}NO_4S$ requires C, 65.44; H, 6.02; N, 3.64%. Found: C, 65.37; H, 6.25; N, 3.48%.

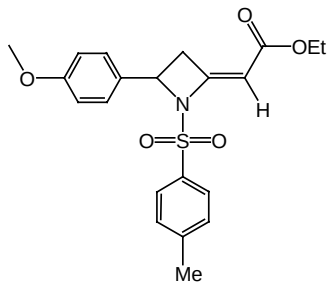





**1c**

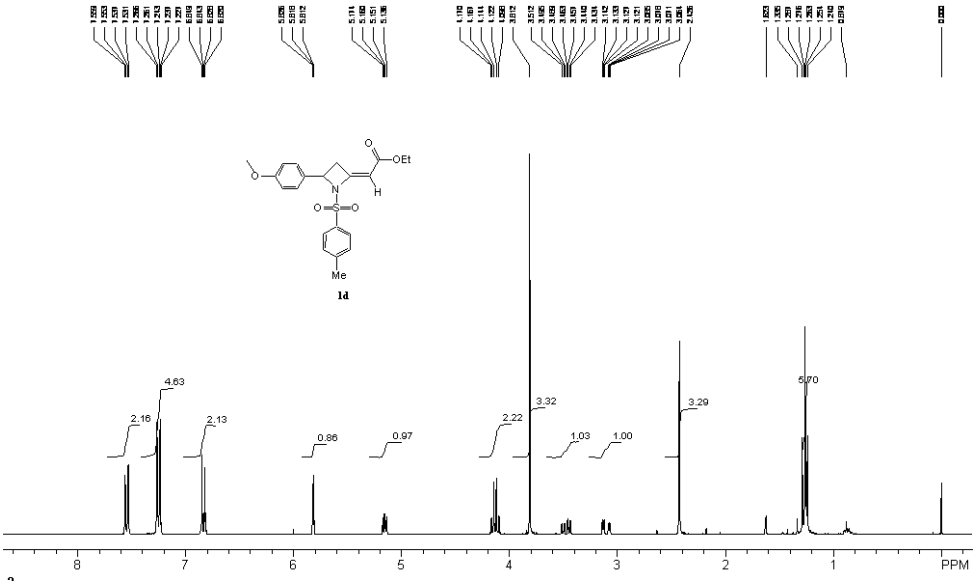
**[4-(4-Ethylphenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1c**: a colorless solid. mp. 106-109 $^{o}$C. IR ($CH_2Cl_2$) ν 2970, 1707 (C=O), 1657, 1598, 1364, 1168, 1127, 1048 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.17-1.25 (6H, m, $2CH_3$), 2.38 (3H, s, $CH_3$), 2.60 (2H, q, *J* = 7.5 Hz, $CH_2$), 3.07 (1H, ddd, *J* = 16.5, 4.5, 1.8 Hz, $CH_2$), 3.44 (1H, ddd, *J* = 16.5, 6.9, 1.8 Hz, $CH_2$), 4.09 (2H, q, *J* = 7.5 Hz, $CH_2$), 5.12 (1H, dd, *J* = 6.9, 4.5 Hz, CH), 5.79-5.81 (1H, m, =CH), 7.10 (2H, d, *J* = 8.1 Hz, ArH), 7.19-7.21 (4H, m, ArH), 7.51 (2H, d, *J* = 8.1 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 15.5, 21.6, 28.5, 37.4, 59.7, 66.1, 94.5, 126.8, 127.3, 128.1, 129.7, 134.3, 134.3, 144.7, 145.1, 158.4, 167.3. MS (EI) *m/z* 399 ($M^+$, 0.70), 244 ($M^+$-155, 74.77), 171 ($M^+$-228, 95.03), 155 ($M^+$-244, 23.38), 91 ($M^+$-308, 100). Anal. Calcd. for $C_{22}H_{25}NO_4S$ requires C, 66.15; H, 6.31; N, 3.51%. Found: C, 66.37; H, 6.49; N, 3.33%.

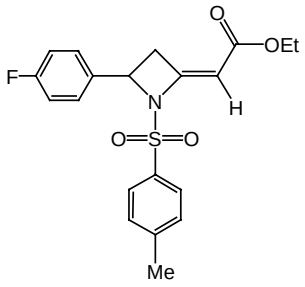





**1d**

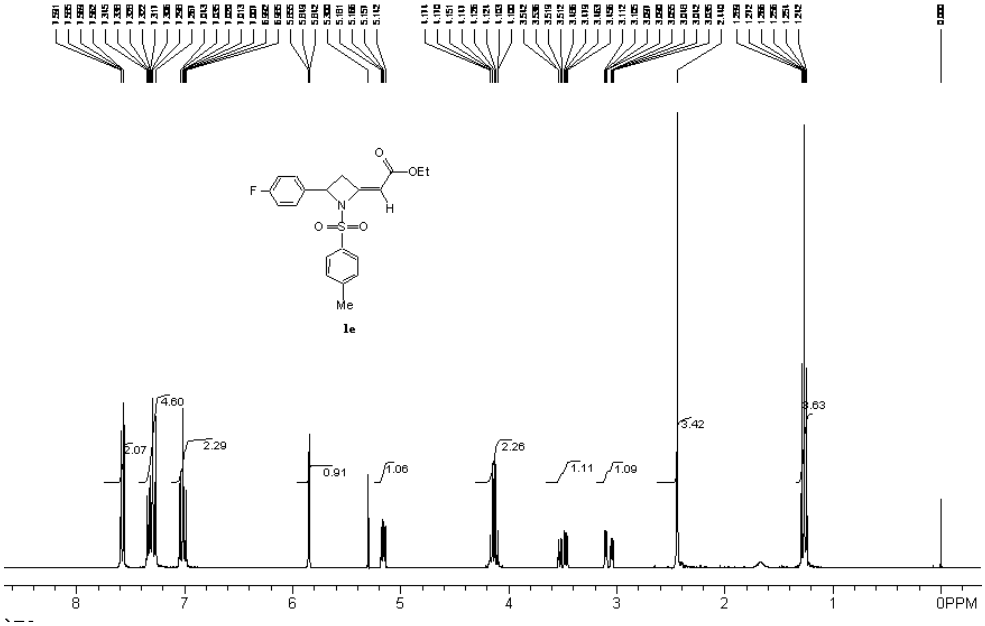
**[4-(4-Methoxyphenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1d**: a colorless solid. mp. 111-115 $^{o}$C. IR ($CH_2Cl_2$) ν 2980, 1706 (C=O), 1656, 1516, 1363, 1166, 1124, 1037 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.43 (3H, s, $CH_3$), 3.10 (1H, ddd, *J* = 17.1, 4.8, 2.1 Hz, $CH_2$), 3.48 (1H, ddd, *J* = 17.1, 6.9, 1.8 Hz, $CH_2$), 3.81 (3H, s, $OCH_3$), 4.13 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.16 (1H, dd, *J* = 6.9, 4.8 Hz, CH), 5.81-5.83 (1H, m, =CH), 6.84 (2H, d, *J* = 6.6 Hz, ArH), 7.23-7.26 (4H, m, ArH), 7.55 (2H, d, *J* = 6.6 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 37.4, 55.3, 59.7, 66.0, 94.4, 114.0, 127.3, 128.2, 129.1, 129.7, 134.4, 144.7, 158.3, 159.9, 167.3. MS (EI) *m/z* 401 ($M^+$, 0.65), 246 ($M^+$-155, 100), 173 ($M^+$-228, 95.87), 134 ($M^+$-267, 95.04), 91 ($M^+$-310, 86.40). Anal. Calcd. for $C_{21}H_{23}NO_5S$ requires C, 62.83; H, 5.78; N, 3.49%. Found: C, 62.69; H, 5.72; N, 3.34%.

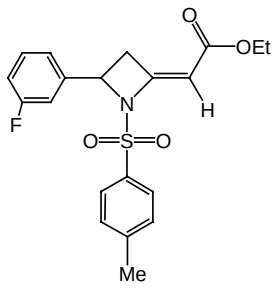





**1e**

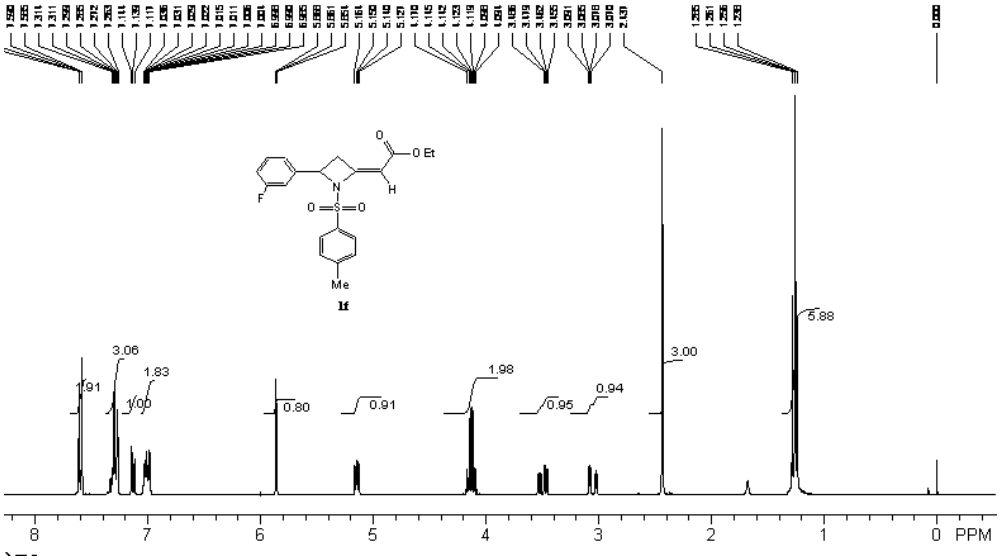
**[4-(4-Fluorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1e**: a colorless solid. mp. 103-105 $^{o}$C. IR ($CH_2Cl_2$) ν 2981, 1708 (C=O), 1659, 1606, 1363, 1159, 1113, 1047 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.27 (3H, t, $J$ = 7.2 Hz, $CH_3$), 2.44 (3H, s, $CH_3$), 3.07 (1H, ddd, $J$ = 17.1, 4.5, 2.1 Hz, $CH_2$), 3.50 (1H, ddd, $J$ = 17.1, 6.9, 2.1 Hz, $CH_2$), 4.13 (2H, q, $J$ = 7.2 Hz, $CH_2$), 5.16 (1H, dd, $J$ = 6.9, 4.5 Hz, CH), 5.84-5.86 (1H, m, =CH), 6.99-7.04 (2H, m, ArH), 7.27-7.35 (4H, m, ArH), 7.56-7.60 (2H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 37.5, 59.8, 65.3, 94.9, 115.6 (d, $^{2}J_{C\text{-}F}$ = 21.6 Hz), 127.3, 128.6 (d, $^{3}J_{C\text{-}F}$ = 8.6 Hz), 129.8, 133.1 (d, $^{4}J_{C\text{-}F}$ = 3.5 Hz), 134.1, 145.0, 157.9, 162.8 (d, $^{1}J_{C\text{-}F}$ = 246.6 Hz), 167.2. MS (EI) *m/z* 389 ($M^+$, 1.17), 344 ($M^+$-45, 7.17), 234 ($M^+$-155, 48.45), 162 ($M^+$-227, 57.57), 161 ($M^+$-228, 60.77), 155 ($M^+$-234, 27.29), 91 ($M^+$-298, 100). Anal. Calcd. for $C_{20}H_{20}FNO_4S$ requires C, 61.69; H, 5.18; N, 3.60%. Found: C, 61.60; H, 5.09; N, 3.40%.

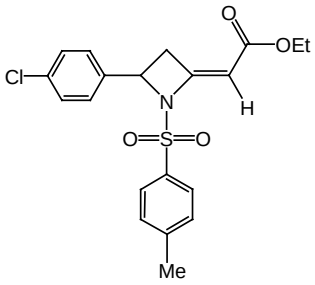





**1f**

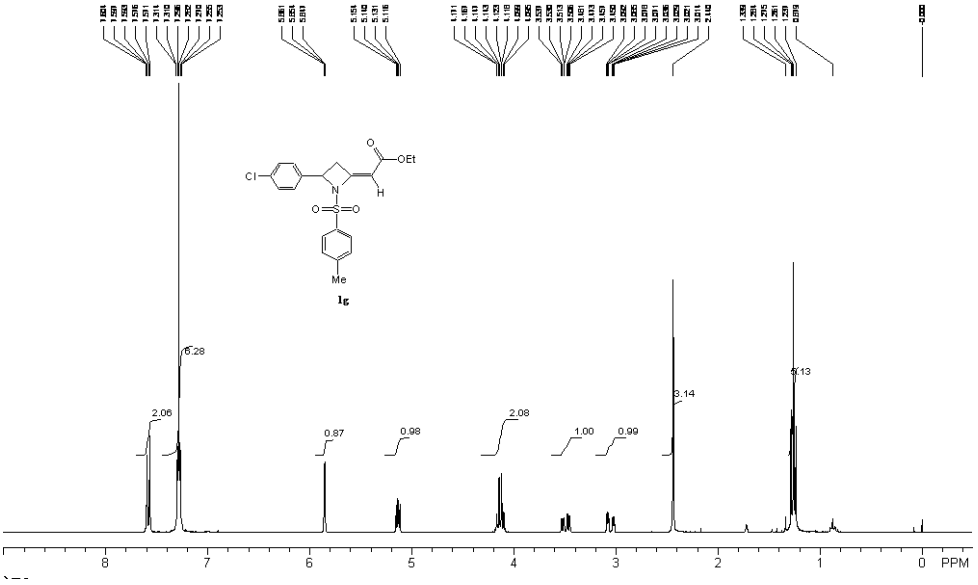
**[4-(3-Fluorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1f**: a colorless solid. mp. 107-109 $^{o}$C. IR ($CH_2Cl_2$) ν 2981, 1708 (C=O), 1658, 1596, 1364, 1168, 1124, 1046 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.29 (3H, t, $J$ = 7.5 Hz, $CH_3$), 2.44 (3H, s, $CH_3$), 3.05 (1H, ddd, $J$ = 16.5, 3.9, 1.8 Hz, $CH_2$), 3.50 (1H, ddd, $J$ = 16.5, 7.5, 2.1 Hz, $CH_2$), 4.13 (2H, q, $J$ = 7.5 Hz, $CH_2$), 5.15 (1H, dd, $J$ = 7.5, 3.9 Hz, CH), 5.85-5.87 (1H, m, =CH), 6.99-7.04 (2H, m, ArH), 7.11-7.14 (1H, m, ArH), 7.26-7.32 (3H, m, ArH), 7.58-7.61 (2H, m, ArH). $^{13}$C NMR ($CDCl_3$, TMS, 75 MHz) δ 14.3, 21.6, 37.4, 59.8, 65.2 (d, $^{4}J_{C\text{-}F}$ = 2.2 Hz), 95.2, 113.5 (d, $^{2}J_{C\text{-}F}$ = 22.7 Hz), 115.7 (d, $^{2}J_{C\text{-}F}$ = 21.1 Hz), 122.3 (d, $^{4}J_{C\text{-}F}$ = 2.8 Hz), 127.3, 129.9, 130.3 (d, $^{3}J_{C\text{-}F}$ = 8.6 Hz), 133.9, 139.9 (d, $^{3}J_{C\text{-}F}$ = 7.9 Hz), 145.1, 157.7, 162.8 (d, $^{1}J_{C\text{-}F}$ = 246.8 Hz), 167.1. MS (EI) *m/z* 389 ($M^+$, 7.17), 234 ($M^+$-155, 24.36), 161 ($M^+$-228, 71.39), 155 ($M^+$-234, 28.60), 91 ($M^+$-298, 100). Anal. Calcd. for $C_{20}H_{20}FNO_4S$ requires C, 61.69; H, 5.18; N, 3.60%. Found: C, 61.58; H, 5.24; N, 3.42%.

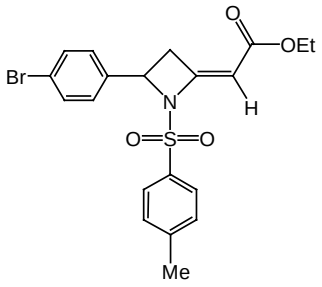





**1g**

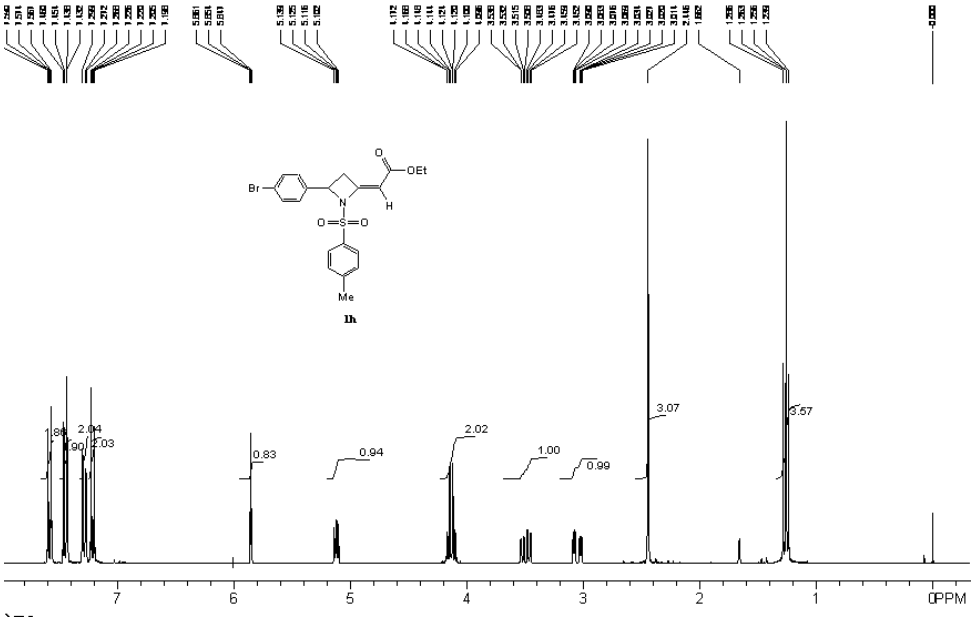
**[4-(4-Chlorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]-acetic acid ethyl ester 1g**: a colorless solid. mp. 108-110 $^{o}$C. IR ($CH_2Cl_2$) ν 2987, 1707 (C=O), 1659, 1598, 1365, 1169, 1127, 1048 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.28 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.44 (3H, s, $CH_3$), 3.05 (1H, ddd, *J* = 16.8, 4.2, 2.1 Hz, $CH_2$), 3.49 (1H, ddd, *J* = 16.8, 7.2, 2.1 Hz, $CH_2$), 4.13 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.14 (1H, ddd, *J* = 7.2, 4.2 Hz, CH), 5.85-5.86 (1H, m, =CH), 7.25-7.31 (6H, m, ArH), 7.58 (2H, d, *J* = 8.4 Hz, ArH). $^{13}$C NMR ($CDCl_3$, TMS, 75 MHz) δ 14.3, 21.6, 37.4, 59.8, 65.2, 95.0, 127.3, 128.0, 128.8, 129.9, 134.0, 134.6, 135.8, 145.1, 157.8, 167.1. MS (EI) *m/z* 405 ($M^+$, 1.16), 250 ($M^+$-155, 34.42), 178 ($M^+$-227, 43.19), 155 ($M^+$-250, 28.58), 91 ($M^+$-314, 100). Anal. Calcd. for $C_{20}H_{20}ClNO_4S$ requires C, 59.19; H, 4.97; N, 3.45%. Found: C, 59.35; H, 5.08; N, 3.26%.

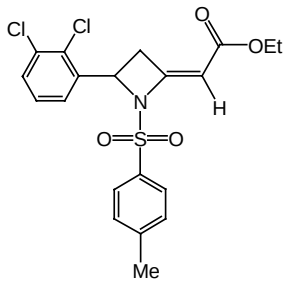





**1h**

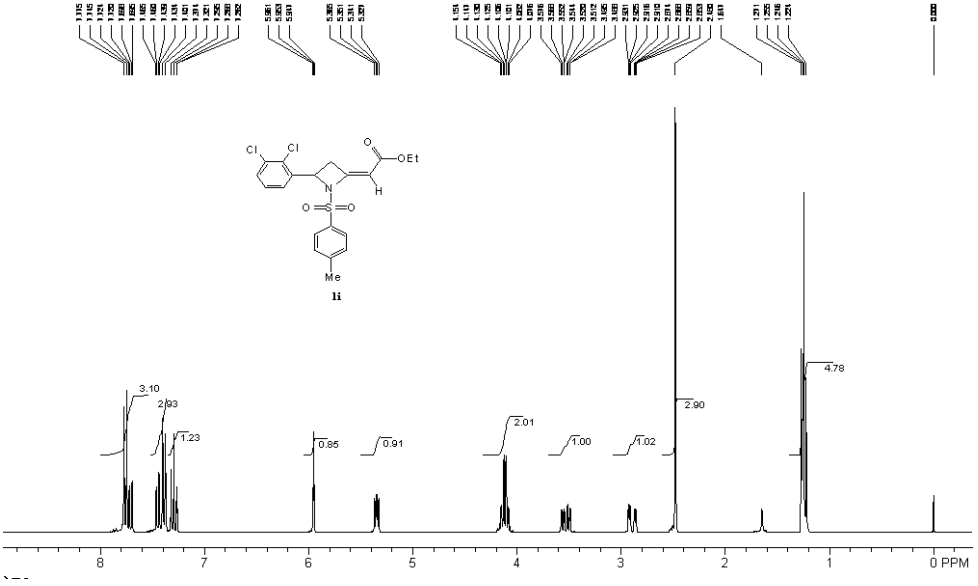
**[4-(4-Bromophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1h**: a colorless solid. mp. 135-137 $^{o}$C. IR ($CH_2Cl_2$) ν 2979, 1708 (C=O), 1658, 1596, 1364, 1167, 1125, 1046 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.45 (3H, s, $CH_3$), 3.05 (1H, ddd, *J* = 16.5, 3.9, 2.1 Hz, $CH_2$), 3.50 (1H, ddd, *J* = 16.5, 6.9, 1.8 Hz, $CH_2$), 4.13 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.12 (1H, dd, *J* = 6.9, 3.9 Hz, CH), 5.85-5.86 (1H, m, =CH), 7.20-7.23 (2H, m, ArH), 7.27-7.30 (2H, m, ArH), 7.42-7.46 (2H, m, ArH), 7.56-7.60 (2H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 37.3, 59.8, 65.2, 95.0, 122.8, 127.3, 128.3, 129.9, 131.8, 133.9, 136.3, 145.1, 157.7, 167.1. MS (EI) *m/z* 406 ($M^+$-43, 4.22), 404 ($M^+$-45, 4.54), 296 ($M^+$-153, 24.22), 294 ($M^+$-155, 25.01), 223 ($M^+$-226, 23.86), 221 ($M^+$-228, 23.54), 171 ($M^+$-278, 60.73), 155 ($M^+$-294, 31.75), 91 ($M^+$-358, 100). Anal. Calcd. for $C_{20}H_{20}BrNO_4S$ requires C, 53.35; H, 4.48; N, 3.11%. Found: C, 53.29; H, 4.55; N, 2.95%.

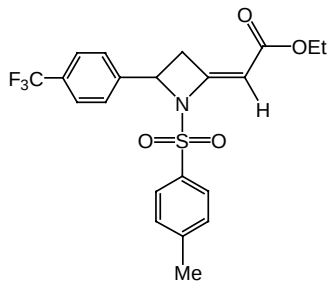





**1i**

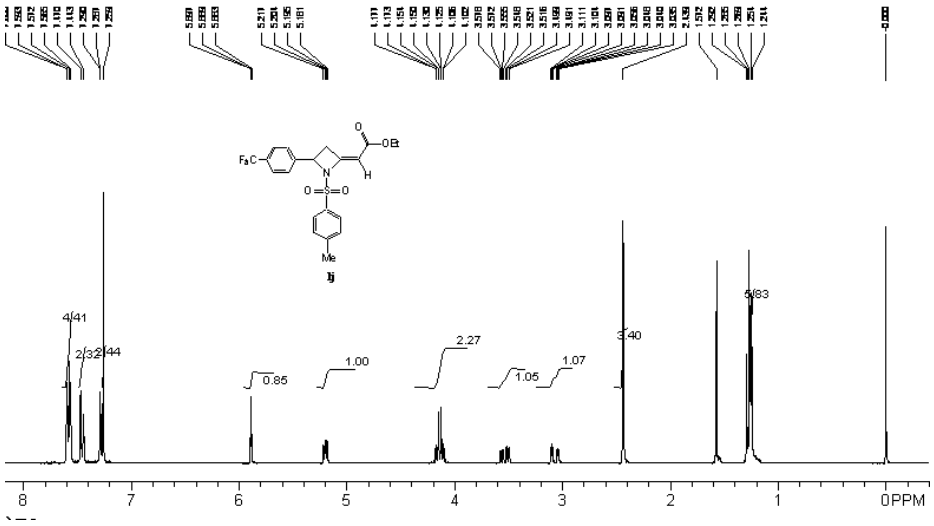
**[4-(2,3-Dichlorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1i**: a colorless solid. mp. 80-82 $^{o}$C. IR ($CH_2Cl_2$) ν 2981, 1710 (C=O), 1661, 1597, 1366, 1337, 1169, 1121, 1044 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.27 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.48 (3H, s, $CH_3$), 2.89 (1H, ddd, *J* = 17.1, 4.8, 1.8 Hz, $CH_2$), 3.53 (1H, ddd, *J* = 17.1, 7.5, 2.4 Hz, $CH_2$), 4.12 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.35 (1H, dd, *J* = 7.5, 4.8 Hz, CH), 5.95-5.96 (1H, m, =CH), 7.26-7.32 (1H, m, ArH), 7.38-7.47 (3H, m, ArH), 7.70-7.77 (3H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 37.1, 59.9, 62.9, 97.1, 125.3, 127.7, 127.7, 129.6, 130.0, 134.1, 132.6, 133.0, 138.1, 145.5, 157.5, 166.7. MS (EI) *m/z* 441 ($M^+$+2, 4.69), 440 ($M^+$+1, 1.58), 439 ($M^+$, 6.45), 394 ($M^+$-45, 4.89), 284 ($M^+$-155, 9.06), 211 ($M^+$-228, 31.35), 155 ($M^+$-284, 34.84), 91 ($M^+$-348, 100). Anal. Calcd. for $C_{20}H_{19}Cl_2NO_4S$ requires C, 54.56; H, 4.35; N, 3.18%. Found: C, 54.56; H, 4.29; N, 3.04%.

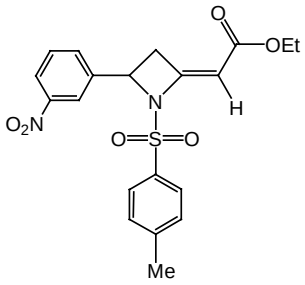




**1j**

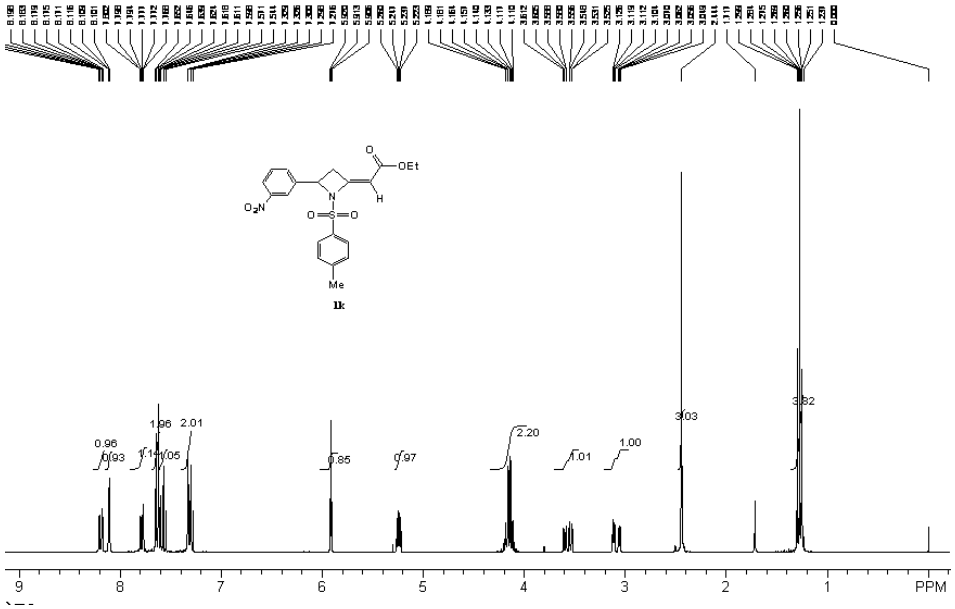
**[1-(Toluene-4-sulfonyl)-4-(4-trifluoromethylphenyl)-azetidin-2-ylidene]acetic acid ethyl ester 1j**: a colorless solid. mp. 135-136 $^{o}$C; IR ($CH_2Cl_2$) ν 2983, 1707 (C=O), 1660, 1326, 1266, 1169, 1128, 1069 cm$^{-1}$; $^{1}$H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.27 (3H, t, $J$ = 7.2 Hz, $CH_3$), 2.44 (3H, s, $CH_3$), 3.07 (1H, ddd, $J$ = 16.5, 4.2, 2.1 Hz, $CH_2$), 3.53 (1H, ddd, $J$ = 16.5, 6.9, 1.8 Hz, $CH_2$), 4.14 (2H, q, $J$ = 7.2 Hz, $CH_2$), 5.20 (1H, dd, $J$ = 6.9, 4.2 Hz, CH), 5.88-5.90 (1H, m, =CH), 7.26-7.29 (2H, m, Ar), 7.44-7.47 (2H, m, Ar), 7.57-7.60 (4H, m, Ar). $^{13}$C NMR ($CDCl_3$, TMS, 75 MHz) δ 14.3, 21.6, 37.4, 59.8, 65.0, 95.4, 122.0, 125.7 (q, $^{3}J_{C\text{-}F}$ = 3.1 Hz), 126.9, 127.3, 129.9, 130.8 (q, $^{2}J_{C\text{-}F}$ = 32.6 Hz), 133.8, 139.3 (q, $^{1}J_{C\text{-}F}$ = 294.8 Hz), 145.2, 157.6, 167.1. MS (EI) *m/z* 440 ($M^+$+1, 2.05), 439 ($M^+$, 8.23), 284 ($M^+$-155, 11.68), 240 ($M^+$-199, 16.47), 212 ($M^+$-227, 50.04), 155 ($M^+$-284, 30.78), 91 ($M^+$-348, 100). Anal. Calcd. for $C_{21}H_{20}F_3NO_4S$ requires C, 57.40; H, 4.59; N, 3.19%. Found: C, 57.38; H, 4.67; N, 2.99%.

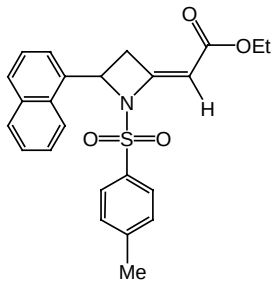




**1k**

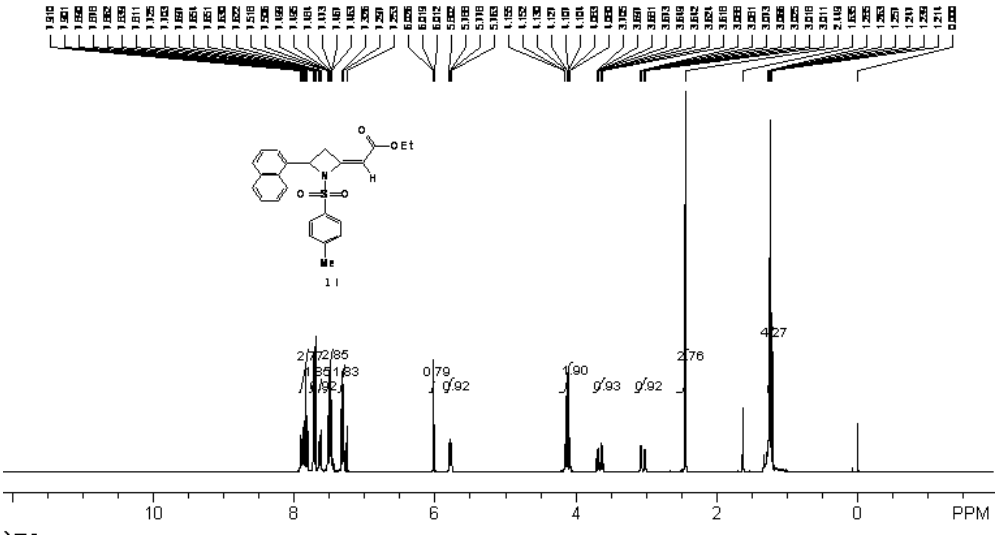
**[4-(3-Nitrophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1k**: a colorless solid. mp. 165-167 $^{o}$C. IR ($CH_2Cl_2$) ν 2982, 1708 (C=O), 1660, 1533, 1364, 1167, 1126, 1046 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.28 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.44 (3H, s, $CH_3$), 3.08 (1H, ddd, *J* = 16.8, 4.2, 2.1 Hz, $CH_2$), 3.57 (1H, ddd, *J* = 16.8, 7.5, 2.4 Hz, $CH_2$), 4.15 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.24 (1H, dd, *J* = 7.5, 4.2 Hz, CH), 5.91-5.92 (1H, m, =CH), 7.28-7.33 (2H, m, Ar), 7.54-7.65 (3H, m, Ar), 7.77-7.80 (1H, m, Ar), 8.10-8.12 (1H, m, Ar), 8.17-8.21 (1H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 37.4, 59.9, 64.5, 95.8, 121.4, 123.6, 127.3, 129.9, 130.0, 132.7, 133.6, 139.6, 145.4, 148.2, 157.1, 166.9. MS (EI) *m/z* 416 ($M^+$, 17.26), 371 ($M^+$-45, 12.05), 261 ($M^+$-155, 7.98), 215 ($M^+$-201, 43.39), 155 ($M^+$-261, 44.21), 91 ($M^+$-325, 100). Anal. Calcd. for $C_{20}H_{20}N_2O_6S$ requires C, 57.69; H, 4.84; N, 6.73%. Found: C, 57.68; H, 4.86; N, 6.29%.

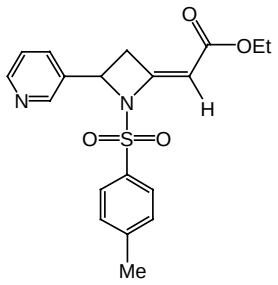





**1l**

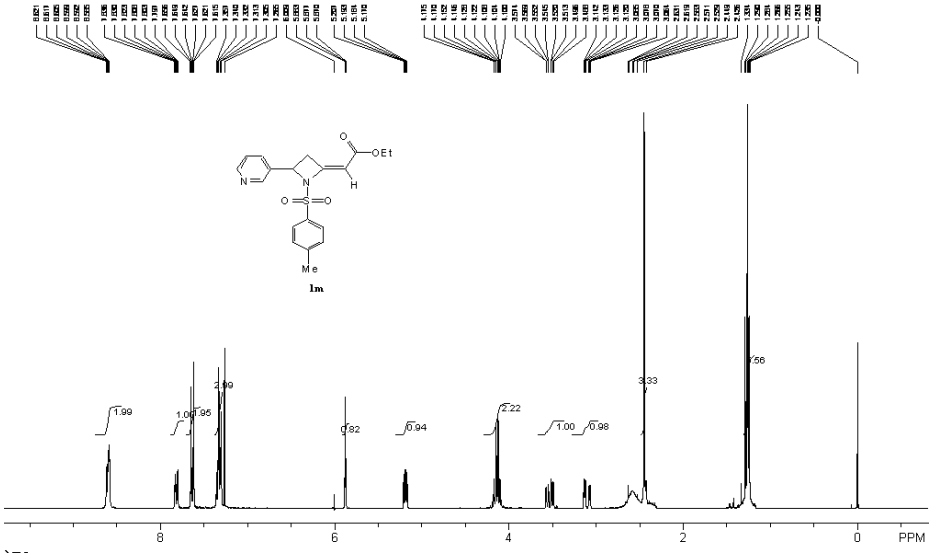
**[4-Naphthalen-1-yl-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1l**: a colorless oil; IR ($CH_2Cl_2$) ν 2978, 1708 (C=O), 1658, 1598, 1364, 1168, 1121, 1044 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, TMS, 300 MHz) δ 1.25 (3H, t, *J* = 7.5 Hz, $CH_3$), 2.45 (3H, s, $CH_3$), 3.05 (1H, ddd, *J* = 16.8, 4.8, 2.1 Hz, $CH_2$), 3.66 (1H, ddd, *J* = 16.8, 7.5, 2.1 Hz, $CH_2$), 4.12 (2H, q, *J* = 7.5 Hz, $CH_2$), 5.78 (1H, dd, *J* = 7.5, 4.8 Hz, CH), 6.01-6.03 (1H, m, =CH), 7.31 (2H, d, *J* = 8.4 Hz, Ar), 7.46-7.52 (3H, m, Ar), 7.62-7.65 (1H, m, Ar), 7.71 (2H, d, *J* = 8.4 Hz, Ar), 7.81-7.91 (3H, m, Ar). $^{13}C$ NMR ($CDCl_3$, TMS, 75 MHz) δ 14.3, 21.6, 37.8, 59.8, 63.8, 96.7, 121.8, 123.8, 125.4, 125.8, 126.4, 127.6, 128.7, 129.1, 129.3, 129.9, 133.1, 133.2, 133.5, 145.2, 158.3, 167.0. MS (EI) *m/z* 421 ($M^+$, 6.06), 376 ($M^+$-45, 7.40), 266 ($M^+$-155, 59.43), 193 ($M^+$-228, 100), 155 ($M^+$-266, 17.41), 91 ($M^+$-330, 35.70). HRMS calcd. for $C_{24}H_{23}NO_4S$: 421.1348, Found: 421.1320.

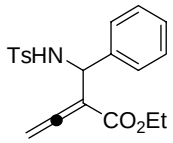





**1m**

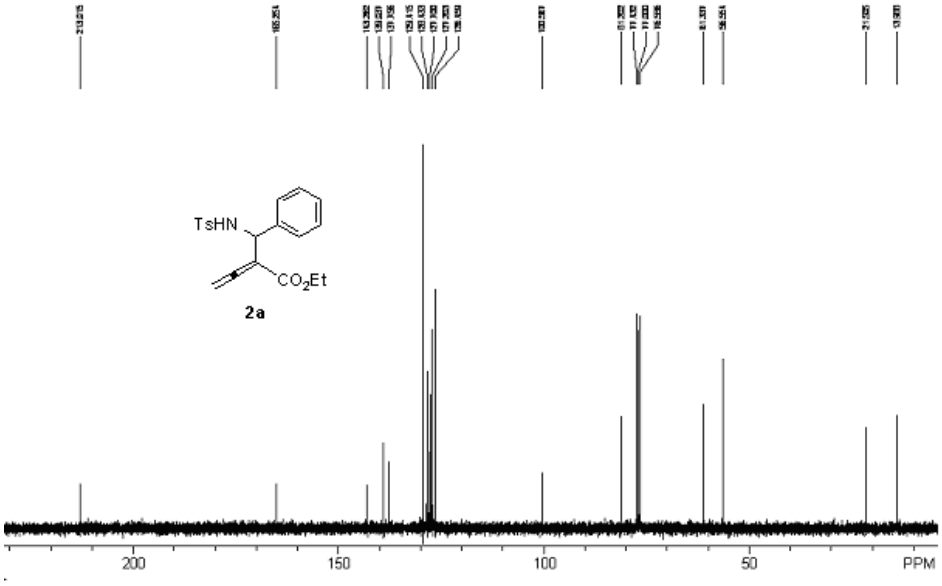
**[4-Pyridin-3-yl-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]acetic acid ethyl ester 1m**: a colorless oil. IR ($CH_2Cl_2$) ν 2980, 1708 (C=O), 1659, 1596, 1364, 1167, 1124, 1044 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, TMS, 300 MHz) δ 1.28 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.45 (3H, s, $CH_3$), 3.10 (1H, ddd, *J* = 17.1, 4.8, 2.7 Hz, $CH_2$), 3.53 (1H, ddd, *J* = 17.1, 6.6, 1.8 Hz, $CH_2$), 4.14 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.19 (1H, dd, *J* = 6.6, 4.8 Hz, CH), 5.87-5.88 (1H, m, =CH), 7.31-7.36 (3H, m, Ar), 7.62-7.65 (2H, m, Ar), 7.80-7.84 (1H, m, Ar), 8.58-8.62 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, TMS, 75 MHz) δ 14.3, 21.6, 37.2, 59.9, 63.3, 95.6, 123.7, 127.4, 130.0, 133.2, 133.6, 134.3, 145.3, 148.1, 150.0, 157.4, 167.0. MS (EI) *m/z* 372 ($M^+$, 8.02), 299 ($M^+$-73, 33.79), 217 ($M^+$-155, 15.56), 155 ($M^+$-217, 28.00), 145 ($M^+$-227, 44.45), 91 ($M^+$-281, 100). HRMS calcd. for $C_{19}H_{20}N_2O_4S$: 372.1144, Found: 372.1155.

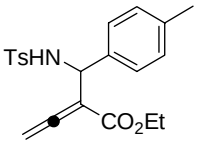





**2a**

**Ethyl 5-(4-Methylphenylsulfonamido)-5-phenylpenta-2,3-dienoate 2a**: a white solid. mp. 121-124 $^{o}$C. IR ($CH_2Cl_2$) ν 3261, 1702 (C=O), 1599, 1327, 1262, 1162, 1121, 1054 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.15 (3H, t, *J* = 6.9 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 4.05 (2H, q, *J* = 6.9 Hz, $CH_2$), 5.09-5.22 (2H, m, =CH), 5.31 (1H, d, *J* = 9.6 Hz, CH), 5.77 (1H, d, *J* = 9.6 Hz, NH), 7.21-7.27 (7H, m, Ar), 7.67-7.70 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 21.5, 56.6, 61.4, 81.2, 100.6, 126.5, 127.2, 127.7, 128.5, 129.4, 137.8, 139.1, 143.3, 165.3, 213.0. MS (EI) *m/z* 371 ($M^+$, 0.59), 260 ($M^+$-111, 100), 216 ($M^+$-155, 14.20), 155 ($M^+$-216, 53.64), 91 ($M^+$-280, 66.32). HRMS calcd. for $C_{20}H_{21}NO_4S$: 371.1191, Found: 371.1202.

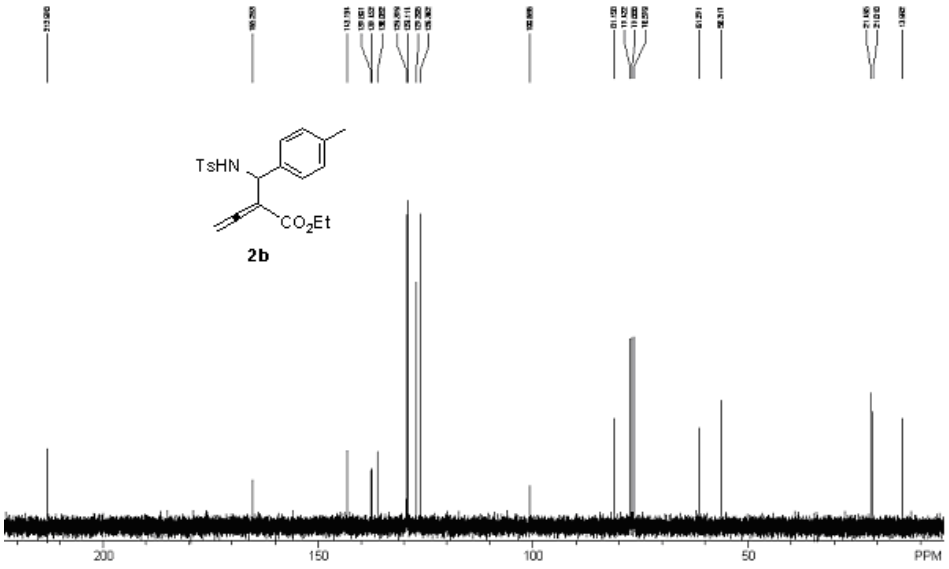


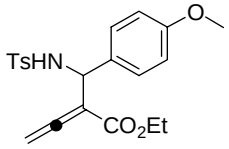

**2b**

**Ethyl 5-(4-Methylphenylsulfonamido)-5-*p*-tolylpenta-2,3-dienoate 2b**: a white solid. mp. 116-120 $^{o}$C. IR ($CH_2Cl_2$) ν 3285, 1708 (C=O), 1598, 1333, 1162, 1093, 1054 cm$^{-1}$. $^{1}$H NMR

($CDCl_3$, 300 MHz, TMS) δ 1.15 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.29 (3H, s, $CH_3$), 2.41 (3H, s, $CH_3$), 4.05 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.09-5.21 (2H, m, =CH), 5.27 (1H, d, *J* = 9.9 Hz, CH), 5.71 (1H, d, *J* = 9.9 Hz, NH), 7.02-7.07 (2H, m, Ar), 7.12-7.15 (2H, m, Ar), 7.22-7.26 (2H, m, Ar), 7.66-7.70 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 21.0, 21.5, 56.3, 61.3, 81.2, 100.7, 126.4, 127.2, 129.1, 129.4, 136.1, 137.5, 137.8, 143.2, 165.3, 213.0. MS (EI) *m/z* 385 ($M^+$, 0.56), 274 ($M^+$-111, 100), 230 ($M^+$-155, 26.94), 184 ($M^+$-201, 24.83), 155 ($M^+$-230, 48.02), 91 ($M^+$-294, 74.34). HRMS calcd. for $C_{21}H_{23}NO_4S$: 385.1348, Found: 385.1364.



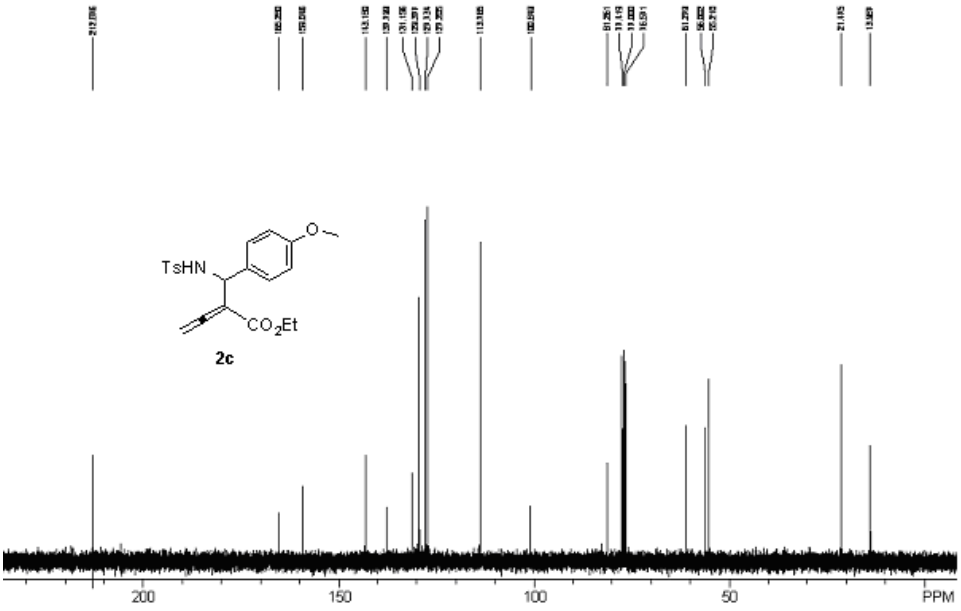

**2b**



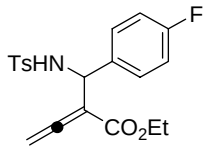

**2c**

**Ethyl 5-(4-Methoxyphenyl)-5-(4-methylphenylsulfonamido)penta-2,3-dienoate 2c**: a colorless oil. IR ($CH_2Cl_2$) ν 2922, 1708 (C=O), 1611, 1512, 1333, 1250, 1093, 1032 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 1.16 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 3.77 (3H, s, $OCH_3$), 4.07 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.09-5.21 (2H, m, =CH), 5.27 (1H, d, *J* = 9.6 Hz, CH), 5.67 (1H, d, *J* = 9.6 Hz, NH), 6.74-6.79 (2H, m, Ar), 7.15-7.26 (4H, m, Ar), 7.63-7.69 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 21.5, 56.2, 56.0, 61.3, 81.3, 100.9, 113.8, 127.2, 127.7, 129.4, 131.2, 137.9, 143.2, 159.1, 165.2, 212.9. MS (EI) *m/z* 292 ($M^+$-109, 7.82), 290

($M^+$-111, 100), 246 ($M^+$-155, 20.75), 155 ($M^+$-246, 27.20), 91 ($M^+$-310, 49.10). HRMS calcd. for $C_{21}H_{23}NO_5S$: 401.1297, Found: 401.1287.

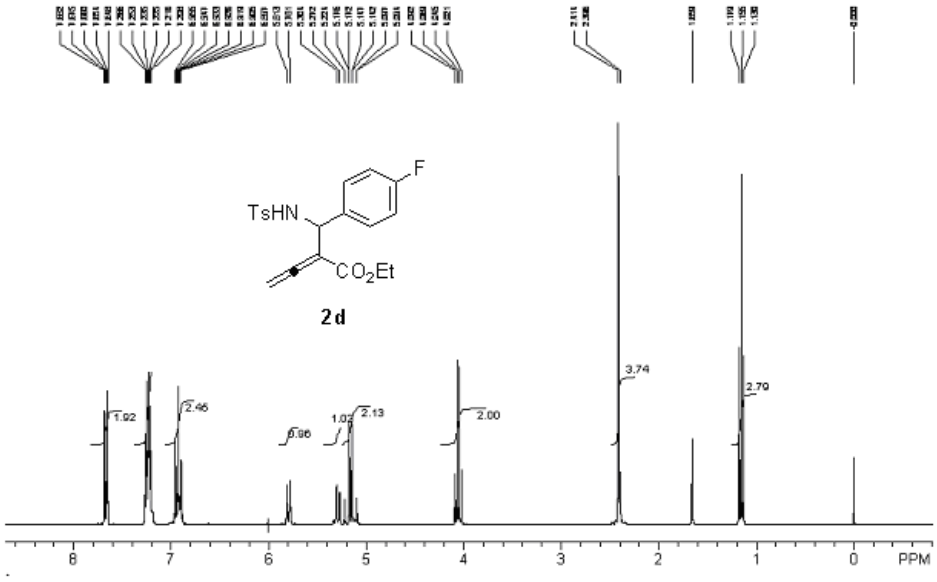



2c

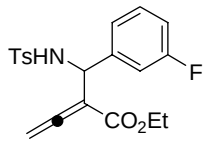



**2d**

**Ethyl 5-(4-Fluorophenyl)-5-(4-methylphenylsulfonamido)penta-2,3-dienoate 2d**: a colorless oil. IR ($CH_2Cl_2$) ν 3275, 1708 (C=O), 1509, 1334, 1162, 1093, 1055 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 1.16 (3H, t, *J* = 6.9 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 4.05 (2H, q, *J* = 6.9 Hz, $CH_2$), 5.09-5.22 (2H, m, =CH), 5.29 (1H, d, *J* = 9.6 Hz, CH), 5.80 (1H, d, *J* = 9.6 Hz, NH), 6.90-6.96 (2H, m, Ar), 7.20-7.27 (4H, m, Ar), 7.61-7.69 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 21.5, 56.0, 61.4, 81.4, 100.5, 115.2 (d, *J* = 21.8 Hz), 127.2, 128.3 (d, *J* = 8.6 Hz), 129.4, 134.9 (d, *J* = 3.3 Hz), 137.6, 143.4, 162.1 (d, *J* = 245.0 Hz), 165.2, 212.8. MS (EI) *m/z* 389 ($M^+$, 0.70), 278 ($M^+$-111, 100), 234 ($M^+$-155, 23.09), 188 ($M^+$-201, 14.96), 155 ($M^+$-234, 74.35), 91 ($M^+$-298, 98.61). HRMS calcd. for $C_{20}H_{20}NO_4SF$: 389.1097, Found: 389.1077.

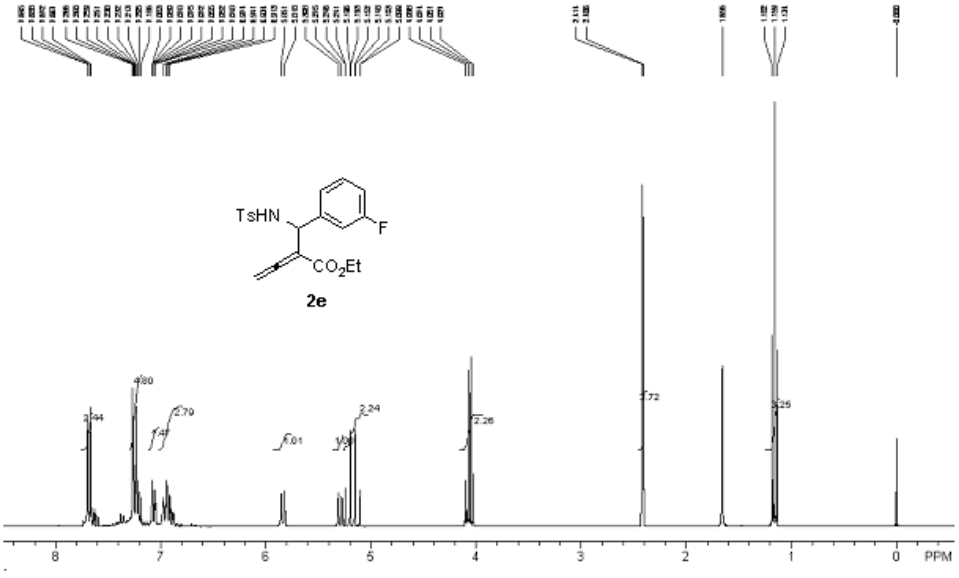


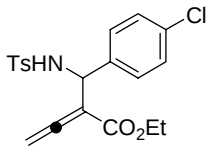

**2d**




**2e**

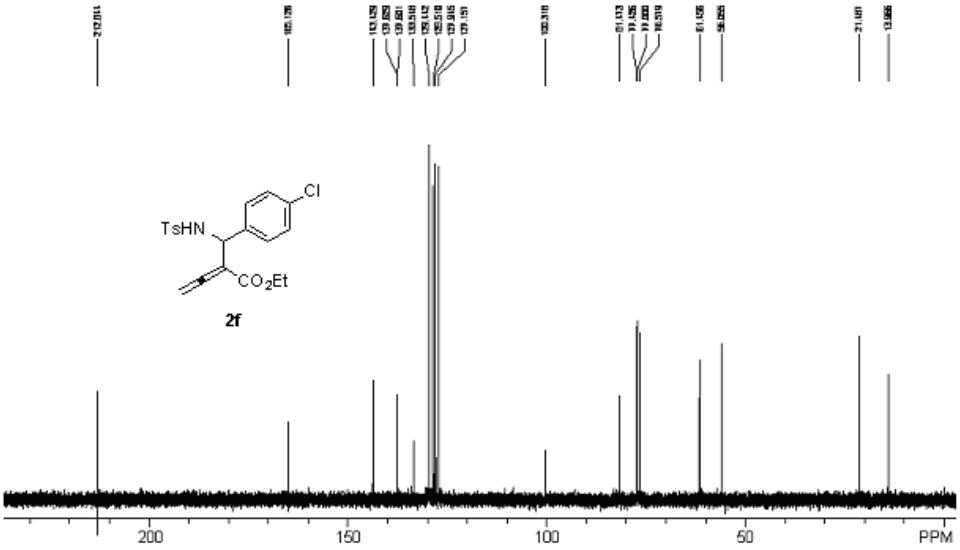
**Ethyl 5-(3-Fluorophenyl)-5-(4-methylphenylsulfonamido)penta-2,3-dienoate 2e** : a white solid; mp. 86-90 $^{o}$C. IR ($CH_2Cl_2$) ν 2987, 1707 (C=O), 1593, 1336, 1162, 1092, 1055 $cm^{-1}$. $^{1}H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 1.16 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 4.06 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.10-5.25 (2H, m, =CH), 5.29 (1H, d, *J* = 9.6 Hz, CH), 5.83 (1H, d, *J* = 9.6 Hz, NH), 6.91-6.97 (2H, m, Ar), 7.05-7.08 (1H, m, Ar), 7.19-7.27 (3H, m, Ar), 7.66-7.70 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 13.9, 21.5, 56.2 (d, *J* = 2.0 Hz), 61.5, 81.4, 100.2, 113.6 (d, *J* = 22.6 Hz), 114.6 (d, *J* = 21.2 Hz), 122.1 (d, *J* = 2.9 Hz), 127.2, 129.5, 130.0 (d, *J* = 8.3 Hz), 137.6, 141.7 (d, *J* = 7.2 Hz), 143.4, 162.7 (d, *J* = 245.3 Hz), 165.1, 213.0. MS (EI) *m/z* 389 ($M^+$, 0.62), 278 ($M^+$-111, 76.93), 234 ($M^+$-155, 23.51), 188 ($M^+$-201), 155 ($M^+$-234, 69.97), 91 ($M^+$-298, 100). HRMS calcd. for $C_{20}H_{20}NO_4SF$: 389.1097, Found: 389.1107.



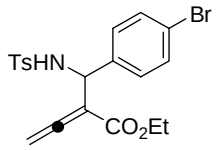




**2f**

**Ethyl 5-(4-Chlorophenyl)-5-(4-methylphenylsulfonamido)penta-2,3-dienoate 2f**: a colorless solid. mp. 107-112 $^{o}$C. IR ($CH_2Cl_2$) ν 2985, 1966, 1712 (C=O), 1597, 1337, 1163, 1091, 1053 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.16 (3H, t, *J* = 6.9 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 4.05 (2H, q, *J* = 6.9 Hz, $CH_2$), 5.10-5.24 (2H, m, =CH), 5.27 (1H, d, *J* = 9.6 Hz, CH), 5.80 (1H, d, *J* = 9.6 Hz, NH), 7.18-7.26 (6H, m, Ar), 7.60-7.67 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 21.5, 56.1, 61.5, 81.5, 100.3, 127.2, 128.0, 128.5, 129.4, 133.6, 137.6, 137.6, 143.4, 165.1, 212.9. MS (EI) *m/z* 296 ($M^+$-109, 37.45), 294 ($M^+$-111, 96.77), 250 ($M^+$-155, 21.06), 215 ($M^+$-190, 22.44), 155 ($M^+$-250, 84.47), 91 ($M^+$-314, 100). HRMS calcd. for $C_{20}H_{20}NO_4SCl$: 405.0802, Found: 405.0805.

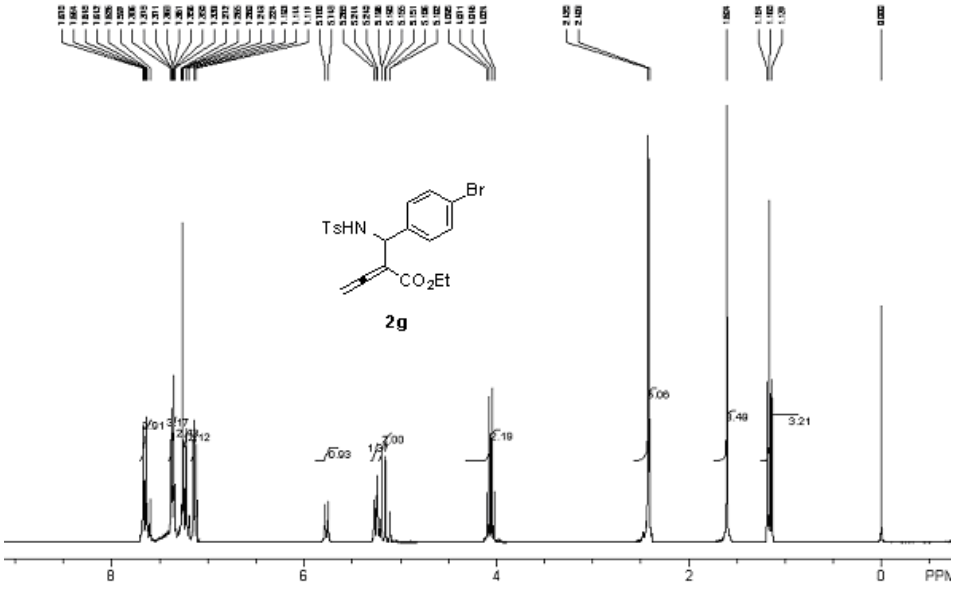


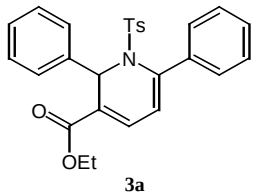

**2f**




**2g**

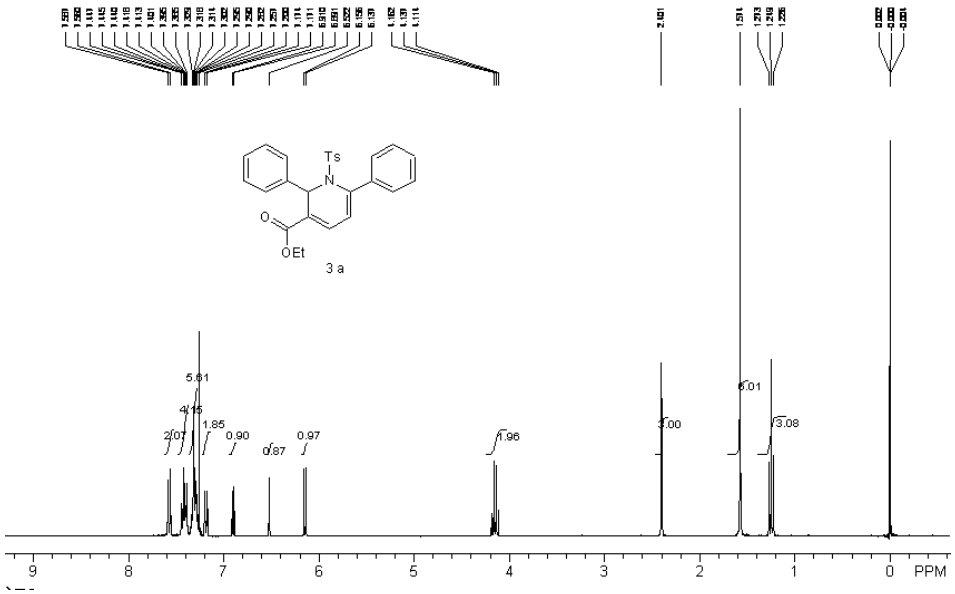
**Ethyl 5-(4-Bromophenyl)-5-(4-methylphenylsulfonamido)penta-2,3-dienoate 2g**: a colorless solid; mp. 100-103 $^{o}$C. IR ($CH_2Cl_2$) ν 2920, 1708 (C=O), 1596, 1335, 1162, 1092, 1060 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.16 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.42 (3H, s, $CH_3$), 4.06 (2H, q, *J* = 7.2 Hz, $CH_2$), 5.10-5.24 (2H, m, =CH), 5.25 (1H, d, *J* = 9.6 Hz, CH), 5.76 (1H, d, *J* = 9.6 Hz, NH), 7.12-7.15 (2H, m, Ar), 7.19-7.26 (2H, m, Ar), 7.34-7.38 (2H, m,Ar), 7.60-7.67 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 21.5, 56.2, 61.5, 81.4, 100.2, 121.8, 127.2, 128.3, 129.5, 131.5, 137.6, 138.1, 143.5, 154.9, 212.9. MS (EI) *m/z* 340 ($M^+$-109, 58.93), 338 ($M^+$-111, 58.57), 294 ($M^+$-155, 8.64), 215 ($M^+$-234, 30.80), 155 ($M^+$-294, 82.52), 91 ($M^+$-358, 100). HRMS calcd. for $C_{20}H_{20}NO_4SBr$: 449.0296, Found: 449.0299.

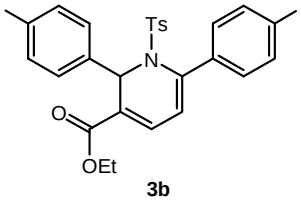

Br
TsHN
CO2Et
2g
8
6
4
2
0
PPM




**3a**

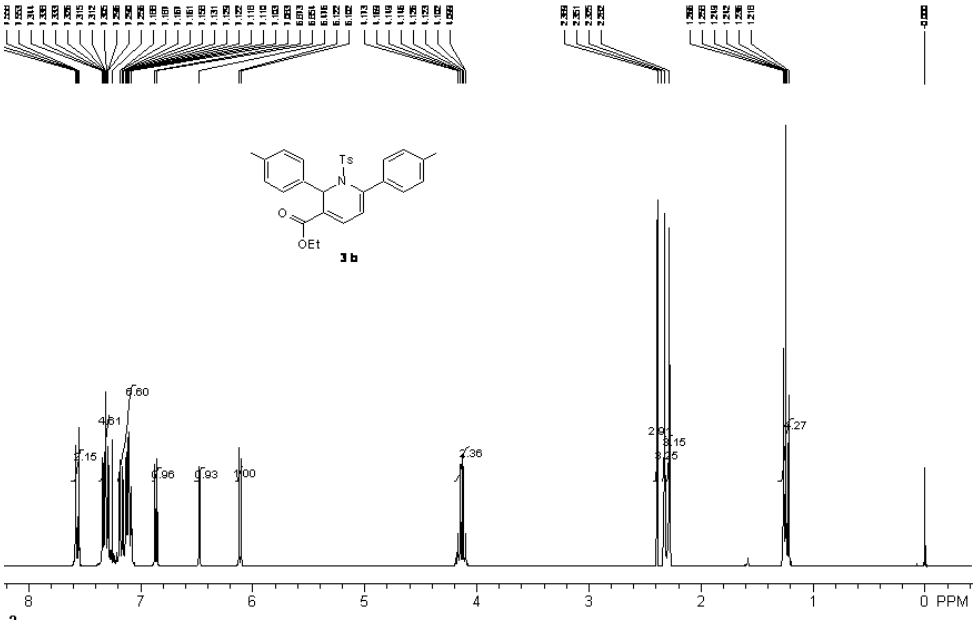
**2,6-Diphenyl-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3a**: a colorless solid. mp. 130-132 $^{o}$C. IR ($CH_2Cl_2$) ν 3056, 1703 (C=O), 1555, 1364, 1266, 1092, 739 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.25 (3H, t, *J* = 6.9 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 4.15 (2H, q, *J* = 6.9 Hz, $OCH_2$), 6.15 (1H, d, *J* = 5.7 Hz, =CH), 6.52 (1H, s, CH), 6.90 (1H, d, *J* = 5.7 Hz, =CH), 7.17-7.20 (2H, m, ArH), 7.26-7.34 (6H, m, ArH), 7.39-7.45 (4H, m, ArH), 7.56-7.59 (2H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 56.0, 60.7, 116.0, 123.9, 127.1, 127.2, 127.3, 128.1, 128.3, 128.4, 129.0, 129.4, 131.8, 135.8, 136.9, 137.6, 141.7, 144.0, 164.4. MS (EI) *m/z* 459 ($M^+$, 34.43), 382 ($M^+$-77, 72.12), 304 ($M^+$-155, 17.04), 274 ($M^+$-185, 77.13), 232 ($M^+$-227, 59.52), 155 ($M^+$-304, 63.89), 91 ($M^+$-368, 100). Anal. Calcd. for $C_{27}H_{25}NSO_4$ requires C, 70.57; H, 5.48; N, 3.05%. Found: C, 70.58; H, 5.56; N, 2.93%.

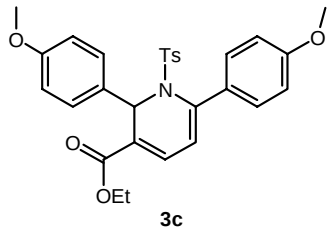



**3b**

**1-(Toluene-4-sulfonyl)-2,6-di-*p*-tolyl-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3b**: a colorless solid. mp. 144-146 $^{o}$C. IR ($CH_2Cl_2$) ν 2982, 1701 (C=O), 1553, 1354, 1167, 1092, 815 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.24 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.28 (3H, s, $CH_3$), 2.32 (3H, s, $CH_3$), 2.39 (3H, s, $CH_3$), 4.14 (2H, q, *J* = 7.2 Hz, $OCH_2$), 6.11 (1H, d, *J* = 5.7 Hz, =CH), 6.48 (1H, s, CH), 6.86 (1H, d, *J* = 5.7 Hz, =CH), 7.08-7.19 (6H, m, ArH), 7.29-7.34 (4H, m, ArH), 7.57 (2H, d, *J* = 6.3 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.1, 21.3, 21.6, 55.8, 60.6, 115.3, 123.8, 127.15, 127.18, 127.4, 128.9, 129.0, 129.1, 131.8, 133.9, 134.9, 135.9, 137.9, 139.6, 141.7, 143.9, 164.5. MS (EI) *m/z* 487 ($M^+$, 100), 396 ($M^+$-91, 66.09), 332 ($M^+$-155, 29.72), 288 ($M^+$-199, 55.63), 260 ($M^+$-227, 60.39), 155 ($M^+$-332, 23.72), 91 ($M^+$-396, 69.93). Anal. Calcd. for $C_{29}H_{29}NSO_4$ requires C, 71.43; H, 5.99; N, 2.87%. Found: C, 71.25; H, 6.10; N, 2.56%.

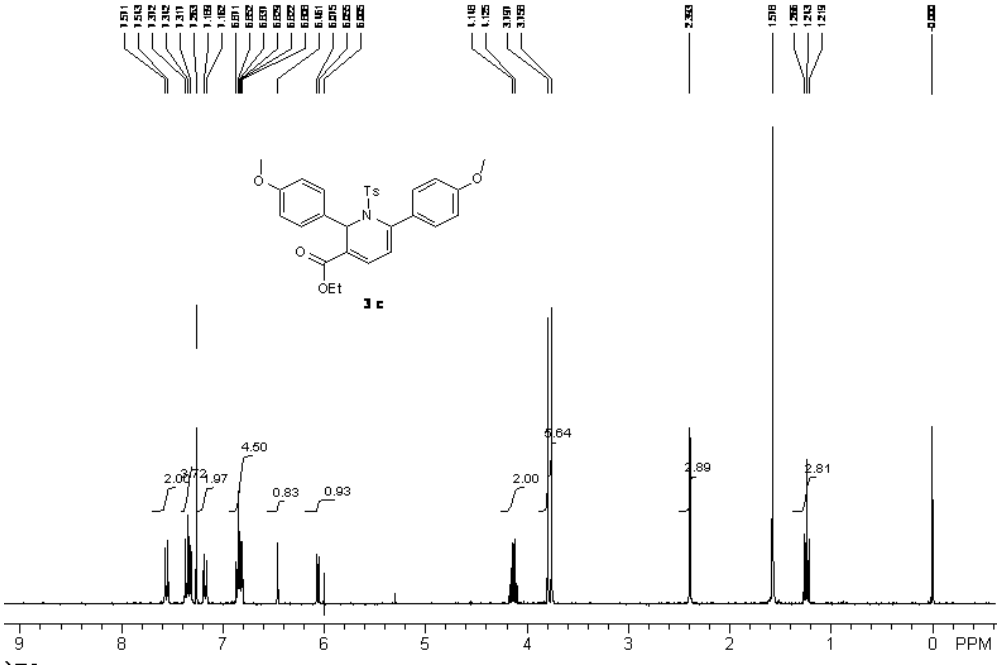


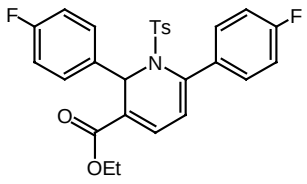




3c

**2,6-Bis(4-methoxyphenyl)-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3c**: a colorless solid. mp. 152-154 $^{o}$C. IR ($CH_2Cl_2$) ν 3055, 1702 (C=O), 1509, 1355, 1266, 1169, 739 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.24 (3H, t, *J* = 7.2 Hz, $CH_3$),

2.39 (3H, s, $CH_3$), 3.76 (3H, s, $OCH_3$), 3.80 (3H, s, $OCH_3$), 4.14 (2H, q, *J* = 7.2 Hz, $CH_2$), 6.07 (1H, d, *J* = 6.0 Hz, =CH), 6.46 (1H, s, CH), 6.81-6.84 (4H, m, ArH), 6.86 (1H, d, *J* = 6.0 Hz, =CH), 7.18 (2H, d, *J* = 8.1 Hz, ArH), 7.32-7.37 (4H, m, ArH), 7.56 (2H, d, *J* = 8.1 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.3, 21.6, 55.2, 55.3, 55.7, 60.6, 113.6, 113.8, 114.4, 123.4, 127.2, 128.6, 128.7, 128.9, 129.0, 130.3, 131.9, 135.9, 141.3, 143.9, 159.5, 160.8, 164.5. MS (EI) *m/z* 519 ($M^+$, 95.65), 364 ($M^+$-155, 64.33), 320 ($M^+$-199, 63.22), 291 ($M^+$-228, 100), 155 ($M^+$-364, 18.15), 91 ($M^+$-428, 63.93). Anal. Calcd. for $C_{29}H_{29}NSO_6$ requires C, 67.03; H, 5.63; N, 2.70%. Found: C, 67.82; H, 5.73; N, 2.64%.

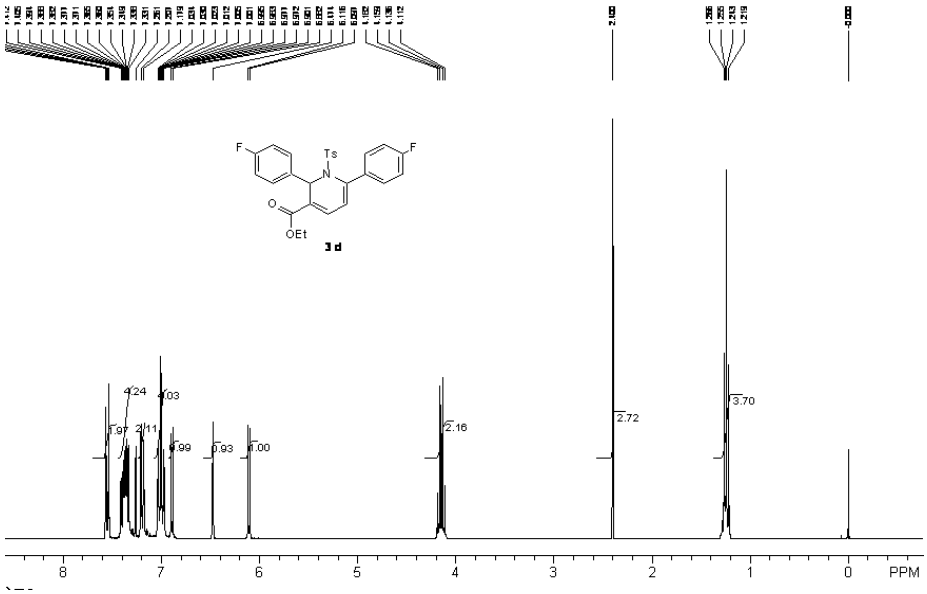


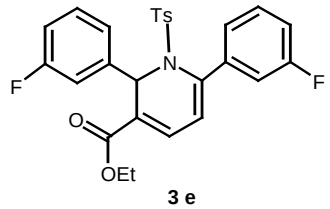




**3 d**

**2,6-Bis(4-fluorophenyl)-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3d**: a colorless solid. mp. 133-135 $^{o}$C. IR ($CH_2Cl_2$) ν 3067, 1702 (C=O), 1506, 1360, 1168, 1091 $cm^{-1}$. $^1$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 4.15 (2H, q, *J* = 7.2 Hz, $CH_2$), 6.11 (1H, d, *J* = 5.4 Hz, =CH), 6.47 (1H, s, CH), 6.86 (1H, d, *J* = 5.4 Hz, =CH), 6.97-7.03 (4H, m, ArH), 7.19 (2H, d, *J* = 8.4 Hz, ArH),

7.33-7.41 (4H, m, ArH), 7.55 (2H, d, $J$ = 8.4 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 55.4, 60.8, 115.3 (d, $^1J_{C\text{-}F}$ = 21.8 Hz), 115.4 (d, $^1J_{C\text{-}F}$ = 21.0 Hz), 123.81, 123.82, 127.1, 128.9 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 129.1 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 129.2, 131.7, 132.5 (d, $^4J_{C\text{-}F}$ = 3.1 Hz), 133.6 (d, $^4J_{C\text{-}F}$ = 2.7 Hz), 135.6, 140.5, 144.3, 162.6 (d, $^1J_{C\text{-}F}$ = 235.2 Hz), 163.1 (d, $^1J_{C\text{-}F}$ = 232.0 Hz), 165.2. MS (EI) *m/z* 495 ($M^+$, 57.74), 400 ($M^+$-95, 59.86), 340 ($M^+$-155, 31.13), 310 ($M^+$-185, 55.58), 296 ($M^+$-199, 76.44), 268 ($M^+$-227, 93.89), 155 ($M^+$-340, 76.37), 91 ($M^+$-404, 100). Anal. Calcd. for $C_{27}H_{23}NSO_4F_2$ requires C, 65.44; H, 4.68; N, 2.83%. Found: C, 65.43 ; H, 4.58; N, 2.71%.

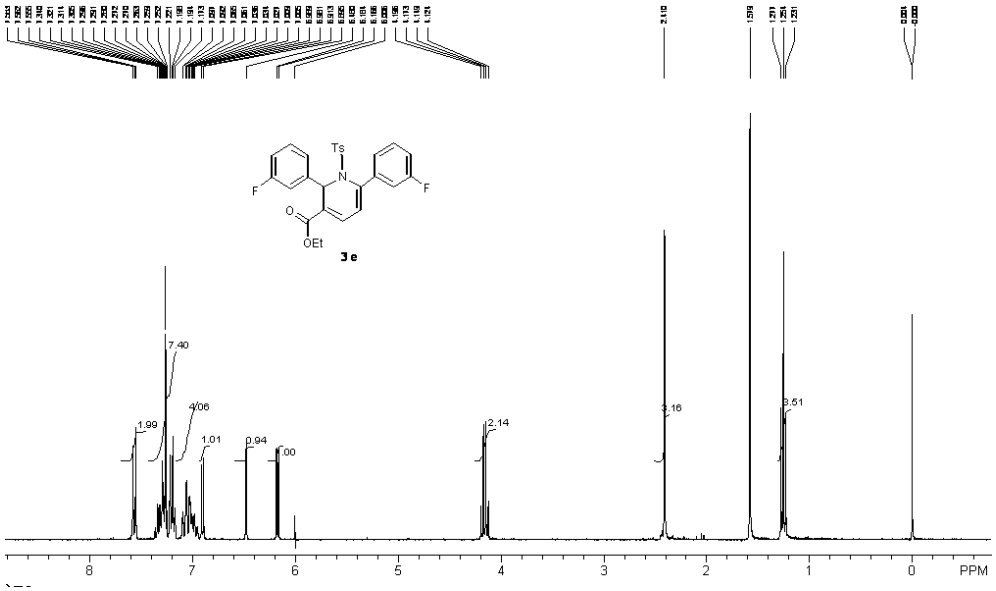


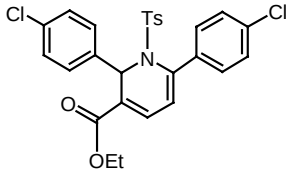

**3 e**

**2,6-Bis(3-fluorophenyl)-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3e**: a colorless solid. mp. 130-132 $^o$C. IR ($CH_2Cl_2$) ν 2983, 1705 (C=O), 1589, 1366, 1270, 1168, 1091, 737 cm$^{-1}$. $^1$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.25 (3H, t, $J$ = 7.2 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 4.16 (2H, q, $J$ = 7.2 Hz, $OCH_2$), 6.18 (1H, d, $J$ = 5.4 Hz, =CH), 6.48 (1H, s, CH), 6.90 (1H, d, $J$ = 5.4 Hz, =CH), 6.98-7.11 (4H, m, ArH), 7.17-7.22 (3H, m, ArH), 7.25-7.34 (3H, m, ArH), 7.56-7.58 (2H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 55.5 (d, $J_{C\text{-}F}$ = 2.0 Hz), 60.9, 113.9 (d, $^2J_{C\text{-}F}$ = 16.9 Hz), 114.2 (d, $^2J_{C\text{-}F}$ = 16.7 Hz), 115.4

(d, $^{2}J_{C\text{-}F}$ = 21.4 Hz), 116.4 (d, $^{2}J_{C\text{-}F}$ = 21.1 Hz), 116.6, 123.0 (d, $^{4}J_{C\text{-}F}$ = 3.0 Hz), 123.1 (d, $^{4}J_{C\text{-}F}$ = 3.1 Hz), 124.1, 127.1, 129.2, 129.7 (d, $^{3}J_{C\text{-}F}$ = 8.5 Hz), 130.1 (d, $^{3}J_{C\text{-}F}$ = 8.0 Hz), 131.6, 135.5, 139.5 (d, $^{3}J_{C\text{-}F}$ = 8.9 Hz), 139.6 (d, $^{3}J_{C\text{-}F}$ = 11.1 Hz), 140.5 (d, $^{4}J_{C\text{-}F}$ = 2.9 Hz), 144.4, 162.4 (d, $^{1}J_{C\text{-}F}$ = 244.4 Hz), 162.7 (d, $^{1}J_{C\text{-}F}$ = 245.1 Hz), 164.0. MS (EI) *m/z* 495 ($M^{+}$, 30.92), 400 ($M^{+}$-95, 61.79), 340 ($M^{+}$-155, 21.86), 268 ($M^{+}$-227, 76.18), 155 ($M^{+}$-340, 80.59), 91 ($M^{+}$-404, 100). Anal. Calcd. for $C_{27}H_{23}F_{2}NSO_{4}$ requires C, 65.44; H, 4.68; N, 2.83%. Found: C, 65.26; H, 4.38; N, 2.75%.



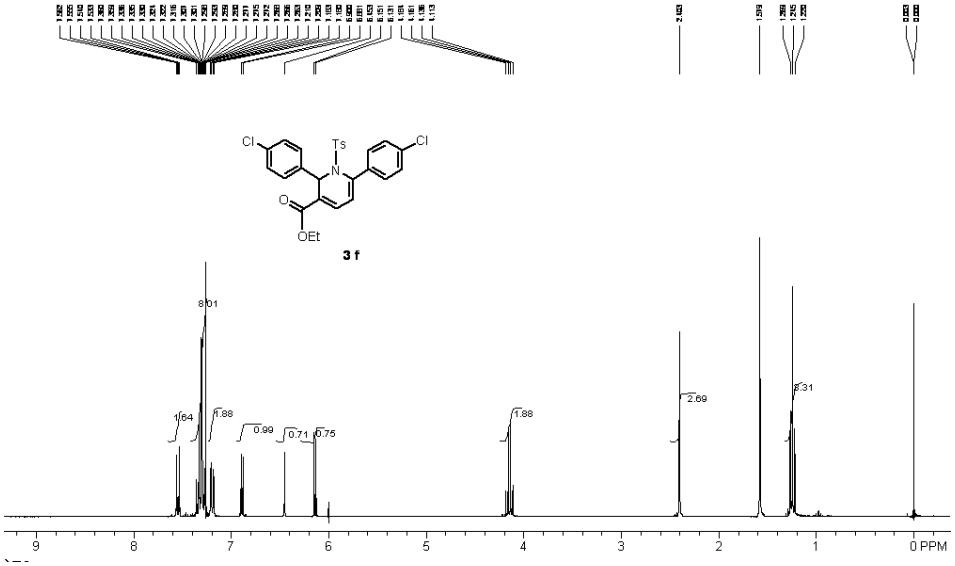

3 e

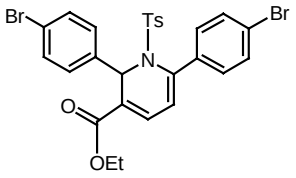



3 f

**2,6-Bis(4-chlorophenyl)-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3f**: a colorless solid. mp. 188-189 $^{o}$C. IR ($CH_2Cl_2$) ν 3064, 1704 (C=O), 1596, 1489, 1366, 1167, 1091, 829 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.25 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 4.14 (2H, q, *J* = 7.2 Hz, $OCH_2$), 6.14 (1H, d, *J* = 6.0 Hz, =CH), 6.45 (1H, s, CH), 6.89 (1H, d, *J* = 6.0 Hz, =CH), 7.20 (2H, d, *J* = 8.7 Hz, ArH), 7.26-7.36 (8H, m, ArH), 7.55 (2H, d, *J* = 8.7 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 55.4, 60.8, 116.1, 123.9, 127.1, 128.3, 128.4, 128.7, 128.7, 129.2, 131.7, 134.3, 135.3, 135.5, 135.6, 135.8, 140.5, 144.4, 164.0. MS (EI) *m/z* 531 ($M^{+}$+4, 4.24), 529 ($M^{+}$+2, 18.21), 527 ($M^{+}$, 25.24), 416

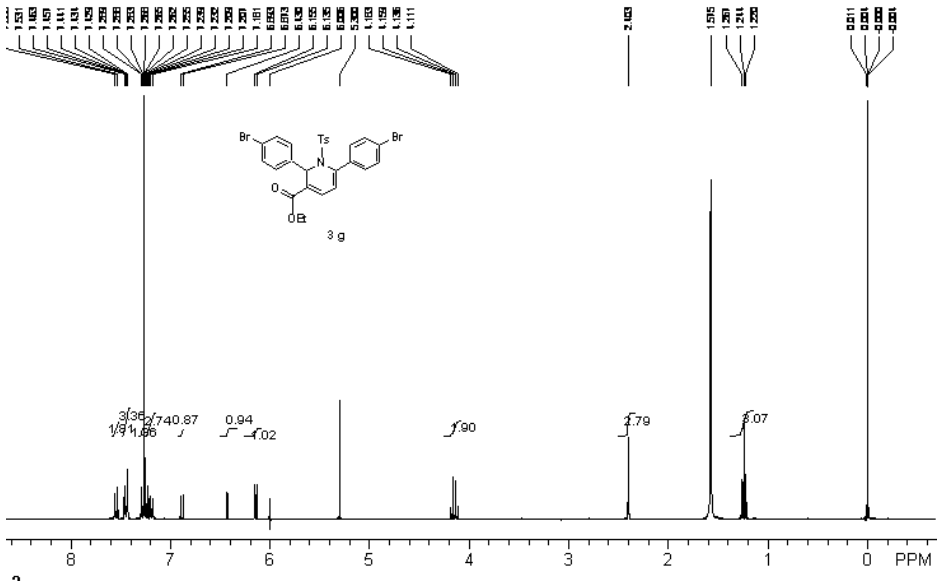
($M^+$-109, 53.93), 372 ($M^+$-155, 17.48), 300 ($M^+$-227, 53.41), 155 ($M^+$-372, 73.51), 91 ($M^+$-436, 100). Anal. Calcd. for $C_{27}H_{23}Cl_2NSO_4$ requires C, 61.37; H, 4.39; N, 2.65%. Found: C, 61.02; H, 4.31; N, 2.61%.

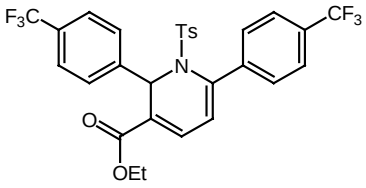



**3 g**

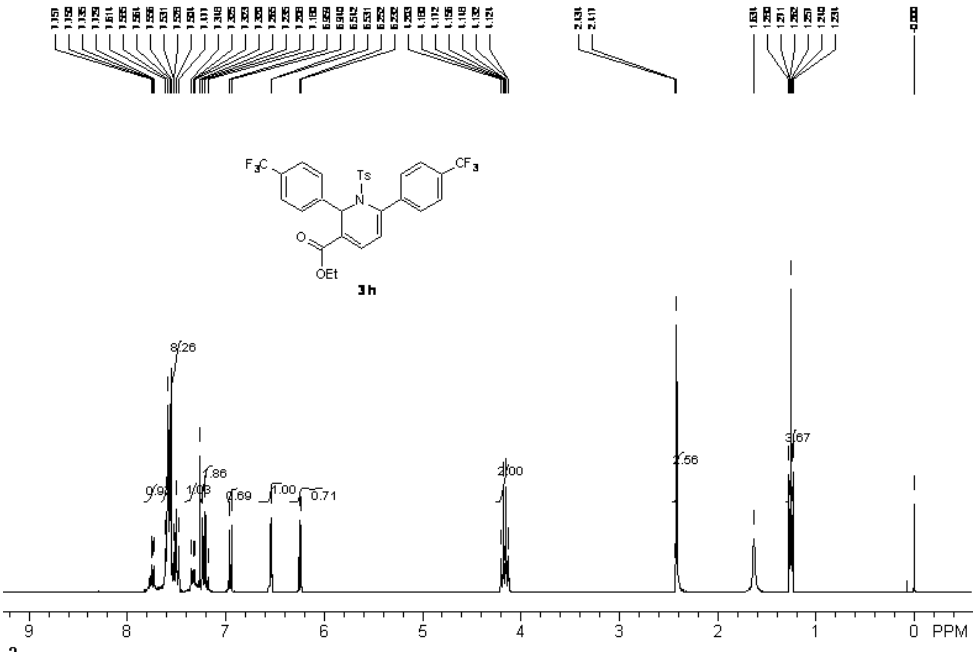
**2,6-Bis(4-bromophenyl)-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ester 3g**: a colorless solid. mp. 178-181 $^oC$. IR ($CH_2Cl_2$) ν 2983, 1702 (C=O), 1485, 1364, 1166, 1090, 1010 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 1.25 (3H, t, $J$ = 7.2 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 4.15 (2H, q, $J$ = 7.2 Hz, $OCH_2$), 6.15 (1H, d, $J$ = 5.7 Hz, =CH), 6.43 (1H, s, CH), 6.89 (1H, d, $J$ = 5.7 Hz, =CH), 7.18-7.29 (6H, m, ArH), 7.43-7.47 (4H, m, ArH), 7.53-7.56 (2H, m, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 55.5, 60.9, 116.1, 122.5, 123.9, 124.0, 127.1, 128.5, 129.0, 129.2, 131.4, 131.7, 131.7, 135.5, 135.8, 136.2, 140.6, 144.4, 164.0; MS (EI) *m/z* 617 ($M^+$+2, 74.62), 615 ($M^+$, 37.75), 462 ($M^+$-153, 98.13), 460 ($M^+$-155, 83.34), 432 ($M^+$-183, 31.16), 390 ($M^+$-225, 36.27), 155 ($M^+$-460, 78.72), 91 ($M^+$-524, 100). Anal. Calcd. for $C_{27}H_{23}Br_2NSO_4 \cdot CH_2Cl_2$ requires C, 47.88; H, 3.59; N, 1.99%; Found: C, 48.00; H, 3.34; N, 1.80%.

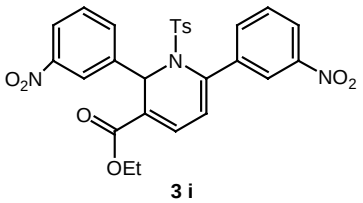



**3 h**

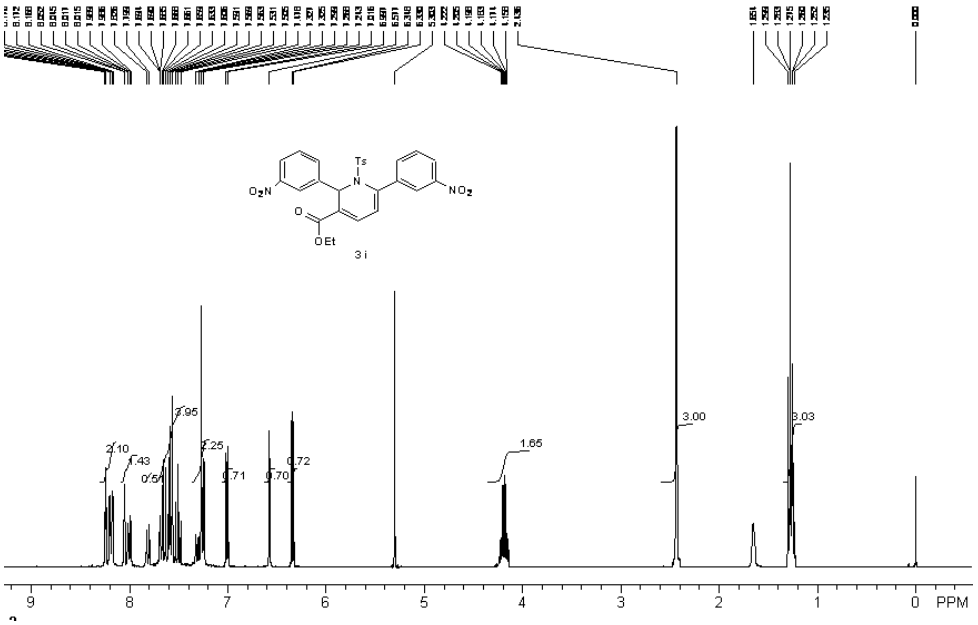
**1-(Toluene-4-sulfonyl)-2,6-bis(4-trifluorophenyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3h**: a colorless oil. IR ($CH_2Cl_2$) ν 2928, 1725 (C=O), 1619, 1326, 1168, 1068, 739 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 1.27 (3H, t, $J$ = 7.2 Hz, $CH_3$), 2.42 (3H, s, $CH_3$), 4.16 (2H, q, $J$ = 7.2 Hz, $OCH_2$), 6.24 (1H, d, $J$ = 6.0 Hz, =CH), 6.53 (1H, s, CH), 6.95 (1H, d, $J$ = 6.0 Hz, =CH), 7.18-7.26 (2H, m, ArH), 7.32-7.35 (1H, m, ArH), 7.48-7.61 (8H, m, ArH), 7.73-7.78 (1H, m, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 55.5, 61.1, 117.3, 124.4, 125.3 (q, $J_{C-F}$ = 3.6 Hz), 125.6 (q, $J_{C-F}$ = 3.8 Hz), 127.1, 127.4, 127.7, 129.2 (q, $J_{C-F}$ = 118.2 Hz), 129.4, 129.8 (q, $J_{C-F}$ =102.5 Hz), 131.0, 131.0, 131.6, 135.4, 140.4, 140.6 (q, $J_{C-F}$ = 1.8 Hz), 140.8 (q, $J_{C-F}$ = 1.4 Hz), 144.7, 163.9. MS (EI) *m/z* 595 ($M^+$, 15.39), 450 ($M^+$-145, 37.63), 396 ($M^+$-199, 24.58), 268 ($M^+$-327, 3.13), 155 ($M^+$-440, 97.35), 91 ($M^+$-504, 100). HRMS calcd. for $C_{29}H_{23}F_6NO_4S$: 595.1252, Found: 595.1251.

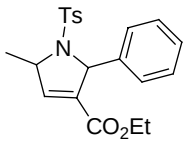



3 i

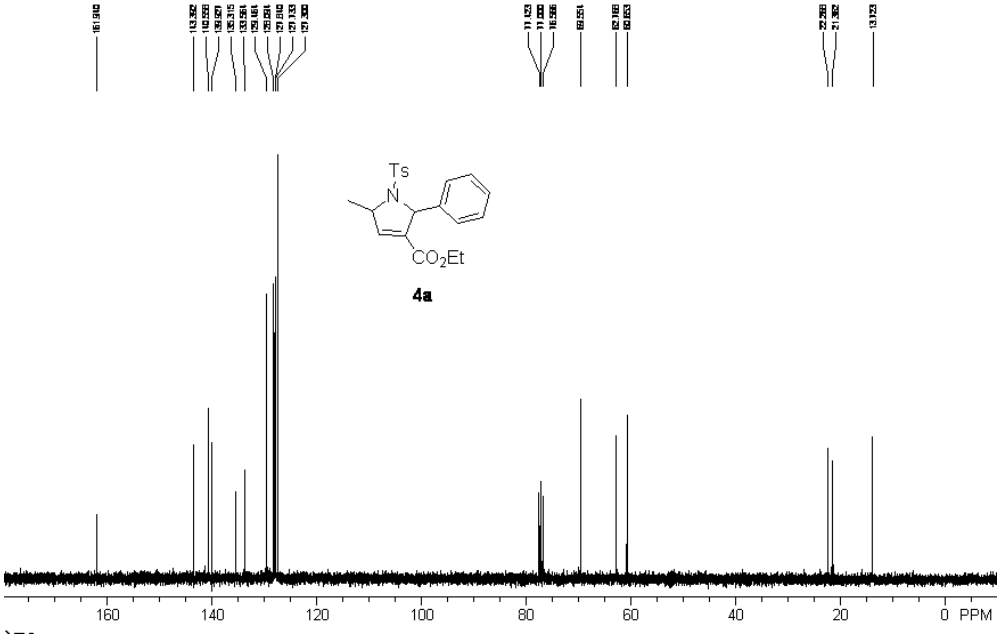
**2,6-Bis(3-nitrophenyl)-1-(toluene-4-sulfonyl)-1,2-dihydropyridine-3-carboxylic acid ethyl ester 3i**: a colorless solid. mp. 152-155 $^{o}$C. IR ($CH_2Cl_2$) ν 3056, 1703 (C=O), 1529, 1364, 1168, 738 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.28 (3H, t, *J* = 7.2 Hz, $CH_3$), 2.44 (3H, s, $CH_3$), 4.19 (2H, q, *J* = 7.2 Hz, $OCH_2$), 6.34 (1H, d, *J* = 5.4 Hz, =CH), 6.58 (1H, s, CH), 7.01 (1H, d, *J* = 5.4 Hz, =CH), 7.24-7.33 (1H, m, ArH), 7.48-7.69 (5H, m, ArH), 7.80-7.83 (1H, m, ArH), 7.99-8.05 (2H, m, ArH), 8.17-8.25 (3H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.2, 21.6, 55.3, 61.3, 117.6, 121.3, 121.7, 123.9, 124.2, 124.2, 126.4, 127.1, 129.3, 129.6, 129.7, 130.0, 131.8, 133.3, 134.0, 135.0, 138.7, 139.0, 139.3, 145.1, 163.7. MS (EI) *m/z* 549 ($M^+$, 18.63), 427 ($M^+$-122, 49.27), 394 ($M^+$-155, 20.41), 364 ($M^+$-185, 47.44), 322 ($M^+$-227, 46.41), 155 ($M^+$-394, 84.62), 91 ($M^+$-458, 100). Anal. Calcd. for $C_{27}H_{23}N_3O_8S \cdot CH_2Cl_2$ requires C, 53.04; H, 3.97; N, 6.63%. Found: C, 52.90; H, 4.19; N, 6.48%.

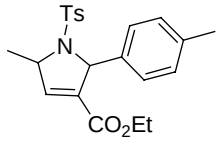

Ts
N
O2N
NO2
O
OEt
3i
2.10
1.43
0.54
2.95
2.25
0.71
0.70
0.72
1.65
3.00
3.03
9
8
7
6
5
4
3
2
1
0
PPM




**4a**

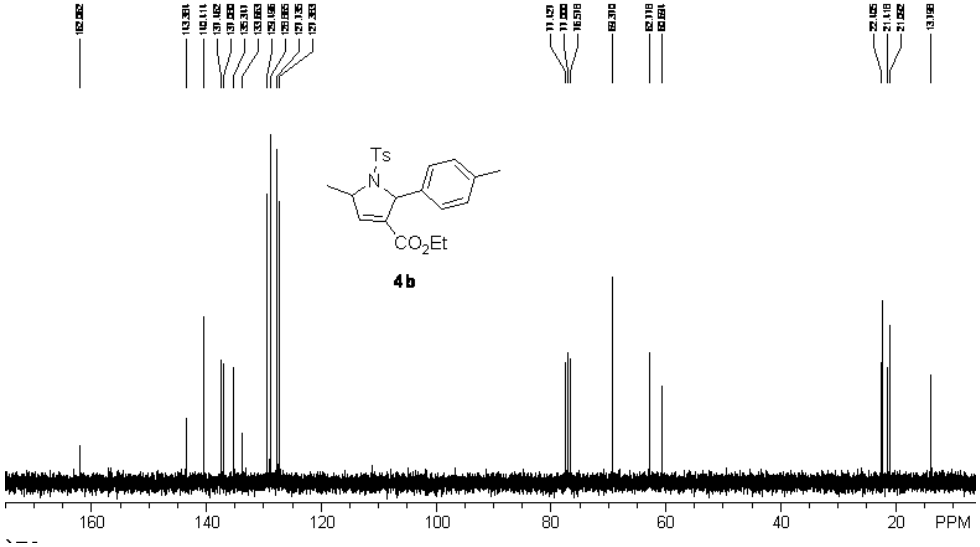
**Ethyl 5-Methyl-2-phenyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4a**: mp. 115-117 $^{o}$C. IR ($CH_2Cl_2$) ν 2981, 1721 (C=O), 1495, 1455, 1350, 1259, 1164, 1093 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4a** δ 1.08 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.55 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.38 (3H, s, $CH_3$), 3.94-4.02 (2H, m, $CH_2$), 4.77-4.80 (1H, m, CH), 5.66-5.67 (1H, m, CH), 6.63-6.65 (1H, m, =CH), 7.19 (2H, d, *J* = 7.5 Hz, ArH), 7.24-7.34 (5H, m, ArH), 7.56 (2H, d, *J* = 7.5 Hz, ArH); ***anti*-4a** δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.68 (3H, d, *J* = 6.3 Hz, $CH_3$), 2.32 (3H, s, $CH_3$), 4.02-4.13 (2H, m, $CH_2$), 4.77-4.80 (1H, m, CH), 5.78-5.81 (1H, m, CH), 6.70-6.71 (1H, m, =CH), 6.94-7.11 (9H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4a** δ 13.7, 21.4, 22.3, 60.7, 62.8, 69.6, 127.3, 127.7, 127.8, 128.1, 129.5, 133.6, 135.3, 139.9, 140.6, 143.4, 161.9. MS (EI) *m/z* 386 ($M^+$+1, 0.72), 385 ($M^+$, 2.85), 370 ($M^+$-15, 30.16), 298 ($M^+$-87, 27.25), 230 ($M^+$-155, 25.07), 155 ($M^+$-230, 39.85), 91 ($M^+$- 294, 100). Found: C, 65.44; H, 5.94; N, 3.54%. $C_{21}H_{23}O_4NS$ requires C, 65.43; H, 6.01; N, 3.63%.



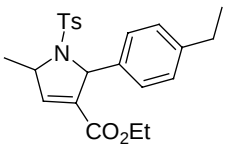



**4b**

**Ethyl 5-Methyl-2-*p*-tolyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4b**: mp. 88-91 $^{o}$C. IR ($CH_2Cl_2$) ν 2980, 1721 (C=O), 1349, 1258, 1164, 1093 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4b** δ 1.09 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.54 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.31 (3H, s, $CH_3$), 2.38 (3H, s, $CH_3$), 3.98-4.03 (2H, m, $CH_2$), 4.73-4.75 (1H, m, CH), 5.61-5.62 (1H, m, CH), 6.61-6.62 (1H, m, =CH), 7.08 (2H, d, *J* = 7.5 Hz, ArH), 7.18-7.22 (4H, m, ArH), 7.58 (2H, d, *J* = 7.5 Hz, ArH); ***anti*-4b** δ 1.10 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.66 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.29 (3H, s, $CH_3$), 2.36 (3H, s, $CH_3$), 3.98-4.03 (2H, m, $CH_2$), 4.81-4.85 (1H, m, CH), 5.74-5.77 (1H, m, CH), 6.68-6.69 (1H, m, =CH), 6.87-6.96 (6H, m, ArH), 7.05-7.07 (2H, m, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4b** δ 13.8, 21.1, 21.4, 22.4, 60.7, 62.8, 69.4, 127.4, 127.7, 128.9, 129.5, 133.7, 135.4, 137.1, 137.5, 140.4, 143.4, 162.1. MS (EI) *m/z* 399 ($M^+$, 1.54), 384 ($M^+$-15, 13.13), 312 ($M^+$-87, 18.85), 244 ($M^+$-155, 35.21), 155 ($M^+$-244, 26.65), 91 ($M^+$-308, 100). Found: C, 66.15; H, 6.22; N, 3.55%. $C_{22}H_{25}O_4NS$ requires C, 66.14; H, 6.31; N, 3.51%.



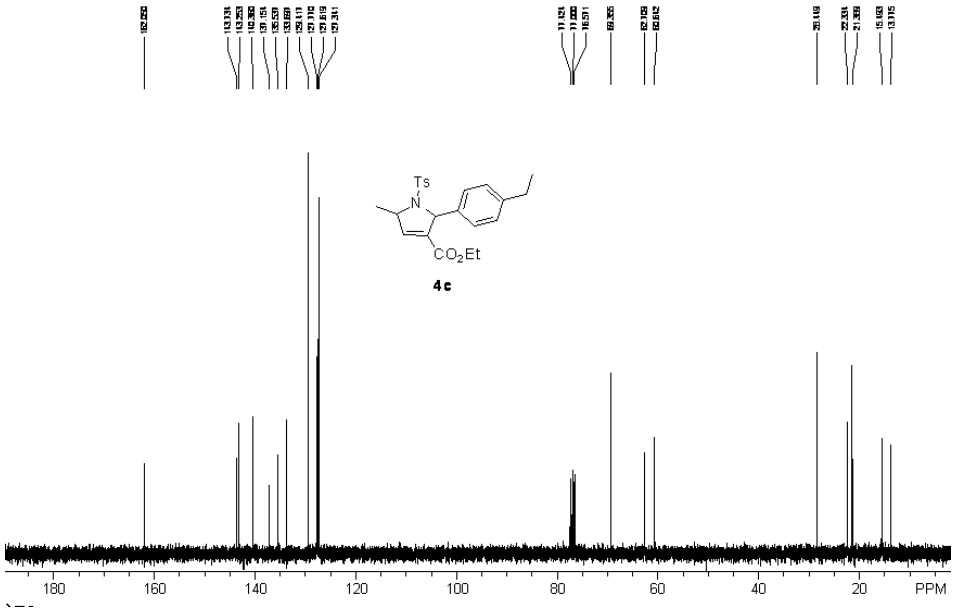

**4b**

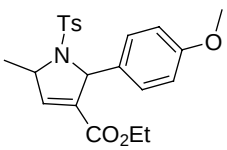



**4c**

**Ethyl 2-(4-Ethylphenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4c**: mp.

115-118 $^{o}$C. IR ($CH_2Cl_2$) ν 2966, 1721 (C=O), 1349, 1259, 1164, 1093, 666 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4c** δ 1.09 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.21 (3H, t, *J* = 7.5 Hz, $CH_3$), 1.55 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.37 (3H, s, $CH_3$), 2.61 (2H, q, *J* = 7.5 Hz, $CH_2$), 3.98-4.04 (2H, m, $CH_2$), 4.77-4.79 (1H, m, CH), 5.64-5.65 (1H, m, CH), 6.62-6.63 (1H, m, =CH), 7.09 (2H, d, *J* = 7.8 Hz, ArH), 7.16-7.23 (4H, m, ArH), 8.54 (2H, d, *J* = 7.8 Hz, ArH); ***anti*-4c** δ 1.07 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.23 (3H, t, *J* = 7.5 Hz, $CH_3$), 1.68 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.30 (3H, s, $CH_3$), 2.60 (2H, q, *J* = 7.5 Hz, $CH_2$), 3.94-4.07 (2H, m, $CH_2$), 4.75-4.79 (1H, m, CH), 5.76-5.78 (1H, m, CH), 6.68-6.69 (1H, m, =CH), 6.89-7.08 (4H, m, ArH), 7.16-7.26 (4H, m, ArH); $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4c** δ 13.8, 15.5, 21.4, 22.3, 28.5, 60.6, 62.7, 69.4, 127.3, 127.6, 127.8, 129.4, 133.7, 135.5, 137.2, 140.4, 143.3, 143.7, 162.1. MS (EI) *m/z* 413 ($M^+$, 1.38), 398 ($M^+$-15, 12.58), 326 ($M^+$-87, 20.29), 258 ($M^+$-155, 41.62), 170 ($M^+$-243, 31.80), 91 ($M^+$-322, 100). Found: C, 66.67; H, 6.61; N, 3.23%. $C_{23}H_{27}O_4NS$ requires C, 66.80; H, 6.58; N, 3.39%.

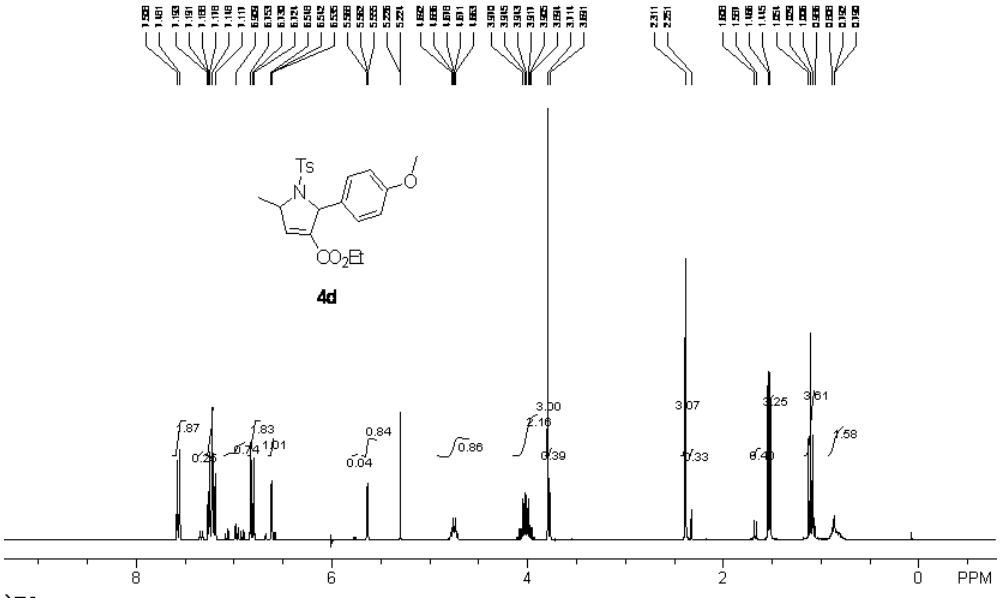


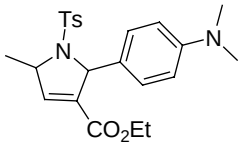

**4d**

**Ethyl 2-(4-Methoxyphenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4d**: a colorless oil. IR ($CH_2Cl_2$) ν 2979, 1720 (C=O), 1596, 1513, 1262, 1157, 1090, 667 cm$^{-1}$. $^{1}$H

NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4d** δ 1.10 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.52 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.38 (3H, s, $CH_3$), 3.79 (3H, s, $OCH_3$), 3.95-4.06 (2H, m, $CH_2$), 4.73-4.76 (1H, m, CH), 5.63-5.64 (1H, m, CH), 6.61-6.62 (1H, m, =CH), 6.81 (2H, d, *J* = 8.4 Hz, ArH), 7.19-7.26 (4H, m, ArH), 7.56 (2H, d, *J* = 8.4 Hz, ArH); ***anti*-4d** δ 1.08 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.67 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.32 (3H, s, $CH_3$), 3.76 (3H, s, $OCH_3$), 3.95-4.06 (2H, m, $CH_2$), 4.73-4.76 (1H, m, CH), 5.75-5.78 (1H, m, CH), 6.67-6.69 (1H, m, =CH), 6.79-6.84 (2H, m, ArH), 6.90-7.10 (6H, m, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4d** δ 13.9, 21.5, 22.5, 55.2, 60.7, 62.7, 69.1, 113.6, 127.4, 128.8, 129.0, 129.5, 132.3, 133.7, 135.6, 140.4, 143.4, 159.2. MS (EI) *m/z* 415 ($M^+$, 1.90), 400 ($M^+$-15, 1.49), 328 ($M^+$-87, 3.27), 259 ($M^+$-156, 15.84), 155 ($M^+$-260, 19.46), 134 ($M^+$-281, 45.89), 91 ($M^+$-324, 100). HRMS calcd. for $C_{22}H_{25}NO_5SNa^{+1}$: 438.1346, Found: 438.1364.




**4d**

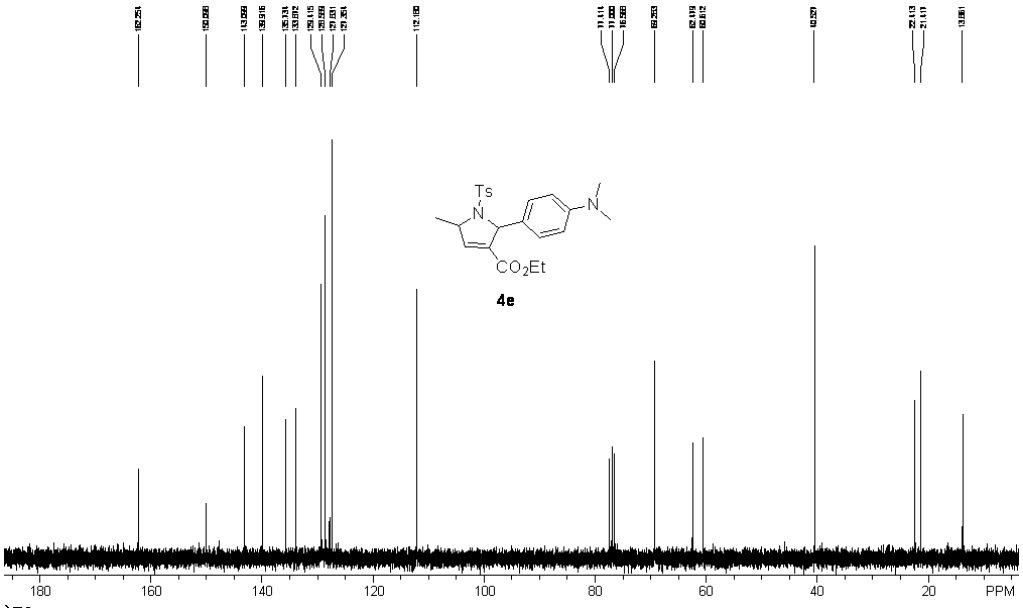



**4e**

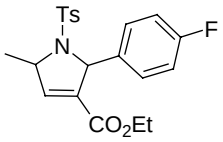
**Ethyl**

**2-(4-(Dimethylamino)phenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4e**: mp. 125-127 $^oC$. IR ($CH_2Cl_2$) ν 2981, 1720 (C=O), 1614, 1523, 1348, 1261, 1165, 1093, 666 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4e** δ 1.11 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.51 (3H, d, *J*

= 6.3 Hz, $CH_3$), 2.37 (3H, s, $CH_3$), 2.92 (6H, s, $CH_3$), 3.99-4.03 (2H, m, $CH_2$), 4.70-4.73 (1H, m, CH), 5.60-5.61 (1H, m, CH), 6.58-6.59 (1H, m, =CH), 6.63 (2H, d, *J* = 7.2 Hz, ArH), 7.15-7.20 (4H, m, ArH), 7.56 (2H, d, *J* = 7.2 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4e** δ 13.9, 21.4, 22.4, 40.5, 60.6, 62.5, 69.3, 112.2, 127.4, 127.8, 128.6, 129.4, 133.9, 135.7, 139.9, 143.1, 150.1, 162.3. MS (EI) *m/z* 428 ($M^+$, 43.88), 272 ($M^+$-156, 50.84), 227 ($M^+$-201, 33.46), 199 ($M^+$-229, 41.39), 91 ($M^+$-337, 100). Found: C, 64.45; H, 6.54; N, 6.43%. $C_{23}H_{28}O_4N_2S$ requires C, 64.46; H, 6.59; N, 6.54%.



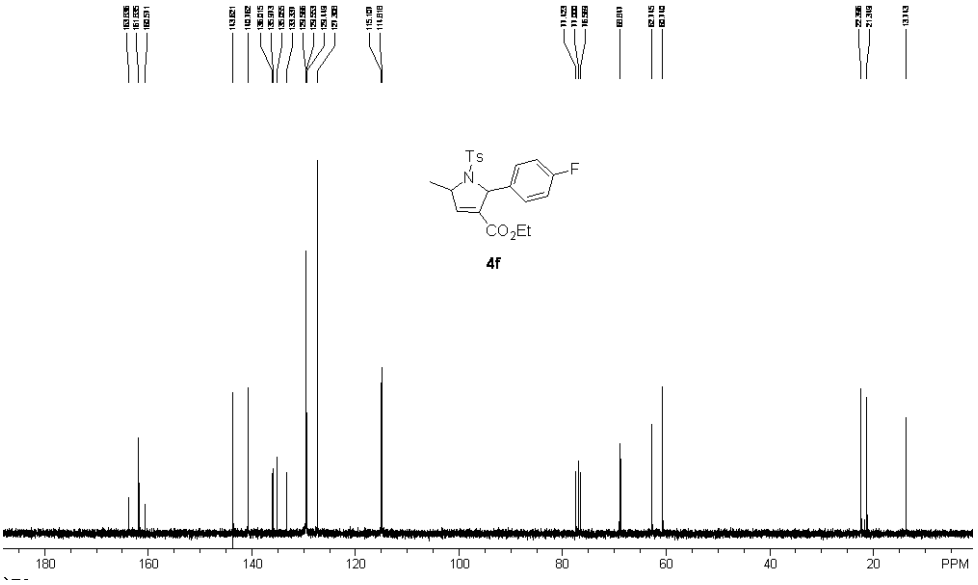

**4e**



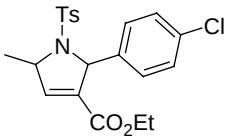

**4f**

**Ethyl 2-(4-Fluorophenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4f**: mp. 78-80 $^o$C. IR ($CH_2Cl_2$) ν 2982, 1721 (C=O), 1509, 1344, 1165, 1093, 666 cm$^{-1}$. $^1$H NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4f** δ 1.10 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.55 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.39 (3H, s, $CH_3$), 3.99-4.06 (2H, m, $CH_2$), 4.75-4.83 (1H, m, CH), 5.63-5.64 (1H, m, CH), 6.63-6.65 (1H, m, =CH), 6.94-7.00 (2H, m, ArH), 7.22 (2H, d, *J* = 8.4 Hz, ArH), 7.27-7.32 (2H, m, ArH), 7.57 (2H, d, *J* = 8.4 Hz, ArH); ***anti*-4f** δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.67 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.34 (3H, s, $CH_3$), 4.05-4.14 (2H, m, $CH_2$), 4.83-4.87 (1H, m, CH), 5.77-5.79 (1H, m, CH), 6.70-6.71 (1H, m, =CH), 6.74-6.80 (2H, m, ArH), 7.00-7.11 (6H, m, ArH). $^{13}$C

NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4f** δ 13.7, 21.3, 22.4, 60.7, 62.7, 68.8, 115.0 (d, *J* = 21.7 Hz), 127.3, 129.5 (d, *J* = 7.8 Hz), 129.6, 133.3, 135.1, 136.0 (d, *J* = 3.2 Hz), 140.8, 143.6, 161.8, 162.2 (d, *J* = 244.9 Hz). MS (EI) *m/z* 388 ($M^+$-15, 20.17), 316 ($M^+$-87, 19.50), 248 ($M^+$-155, 24.41), 205 ($M^+$-198, 73.38), 155 ($M^+$-248, 38.66), 91 ($M^+$-312, 100). Found: C, 62.41; H, 5.51; N, 3.37%. $C_{21}H_{22}O_4NSF$ requires C, 62.51; H, 5.50; N, 3.47%.



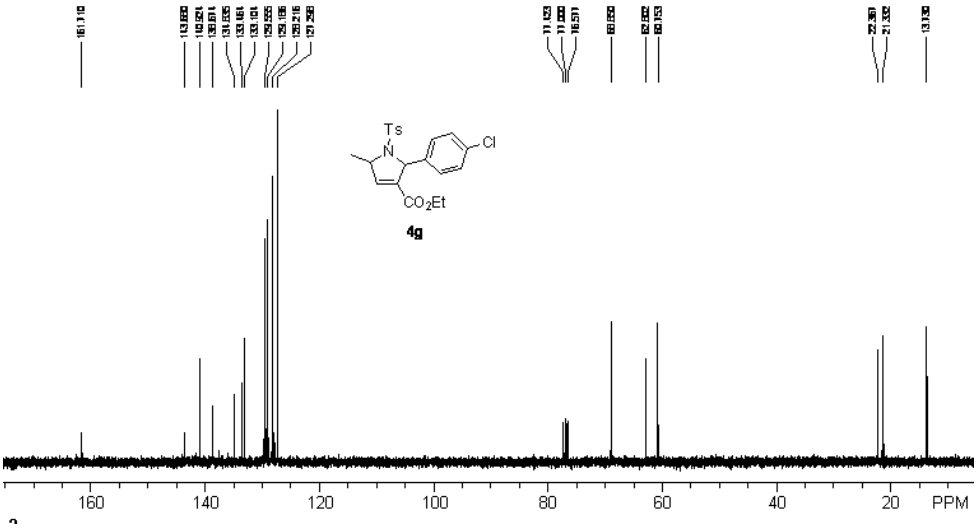

**4f**

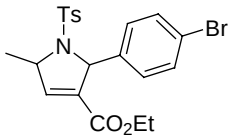



**4g**

**Ethyl 2-(4-Chlorophenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4g**: mp. 90-93 °C. IR ($CH_2Cl_2$) ν 2982, 1720 (C=O), 1491, 1267, 1164, 1092, 666 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4g** δ 1.11 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.56 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 4.00-4.05 (2H, m, $CH_2$), 4.75-4.77 (1H, m, CH), 5.60-5.62 (1H, m, CH), 6.63-6.65 (1H, m, =CH), 7.21-7.27 (6H, m, ArH), 7.58 (2H, d, *J* = 7.5 Hz, ArH); ***anti*-4g** δ 1.23 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.68 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.36 (3H, s, $CH_3$), 3.96-4.08 (2H, m, $CH_2$), 4.86-4.88 (1H, m, CH), 5.74-5.76 (1H, m, CH), 6.75-6.77 (1H, m, =CH), 6.95-7.10 (8H, m, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4g** δ 13.7, 21.3, 22.4, 60.8, 62.8, 68.9, 127.3, 128.2, 129.2, 129.6, 133.1, 133.5, 134.8, 138.7, 140.9, 143.7, 161.7. MS (EI) *m/z* 419 ($M^+$, 1.50), 404 ($M^+$-15, 16.45), 332 ($M^+$-87, 17.66), 264 ($M^+$-155, 26.32), 155 ($M^+$-264, 45.14), 91

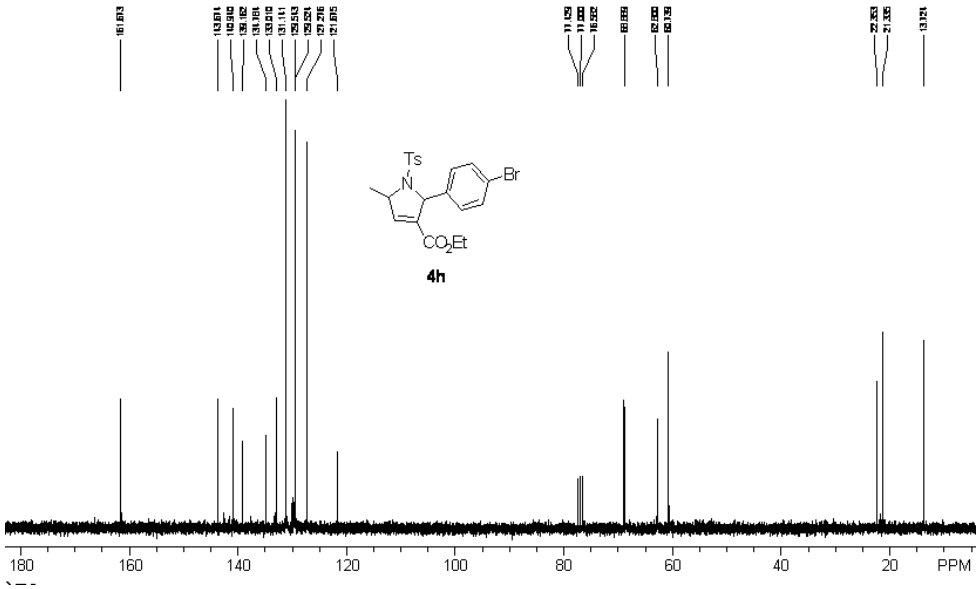
($M^+$-328, 100). Found: C, 60.26; H, 5.33; N, 3.32%. $C_{21}H_{22}O_4NSCl$ requires C, 60.06; H, 5.28; N, 3.34%.



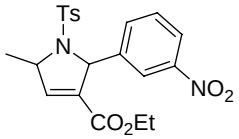




**4h**

**Ethyl 2-(4-Bromophenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4h**: mp. 91-93 $^oC$. IR ($CH_2Cl_2$) ν 2983, 1720 (C=O), 1350, 1266, 1164, 1093, 666 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4h** δ 1.12 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.57 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 3.97-4.09 (2H, m, $CH_2$), 4.75-4.79 (1H, m, CH), 5.60-5.62 (1H, m, CH), 6.65-6.66 (1H, m, =CH), 7.24 (4H, d, *J* = 8.4 Hz, ArH), 7.42 (2H, d, *J* = 8.4 Hz, ArH), 7.61 (2H, d, *J* = 8.4 Hz, ArH); ***anti*-4h** δ 1.23 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.68 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.36 (3H, s, $CH_3$), 4.08-4.14 (2H, m, $CH_2$), 4.88-4.93 (1H, m, CH), 5.73-5.76 (1H, m, CH), 6.73-6.74 (1H, m, =CH), 6.90 (2H, d, *J* = 8.4 Hz, ArH), 7.02 (2H, d, *J* = 8.4 Hz, ArH), 7.12-7.20 (4H, m, ArH); $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4h** δ 13.7, 21.3, 22.4, 60.7, 62.8, 68.9, 121.7, 127.3, 129.5, 129.5, 131.1, 133.0, 134.8, 139.2, 140.9, 143.7, 161.7. MS (EI) *m/z* 465 ($M^+$+2, 0.93), 450 ($M^+$-13, 9.58), 448 ($M^+$-15, 8.98), 378 ($M^+$-85, 8.63), 376 ($M^+$-87, 8.61), 308 ($M^+$-155, 17.99), 155 ($M^+$-308, 44.02), 91 ($M^+$-372, 100). Found: C, 54.45; H, 4.73; N, 2.93%. $C_{21}H_{22}O_4NSBr$ requires C, 54.32; H, 4.78; N, 3.02%.

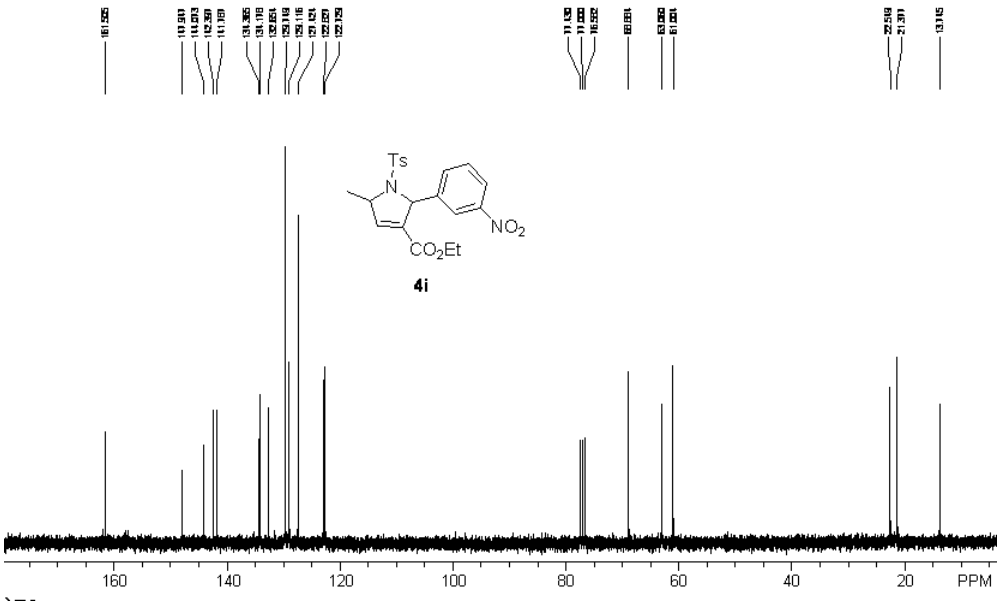


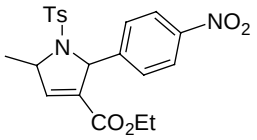

**4h**




**4i**

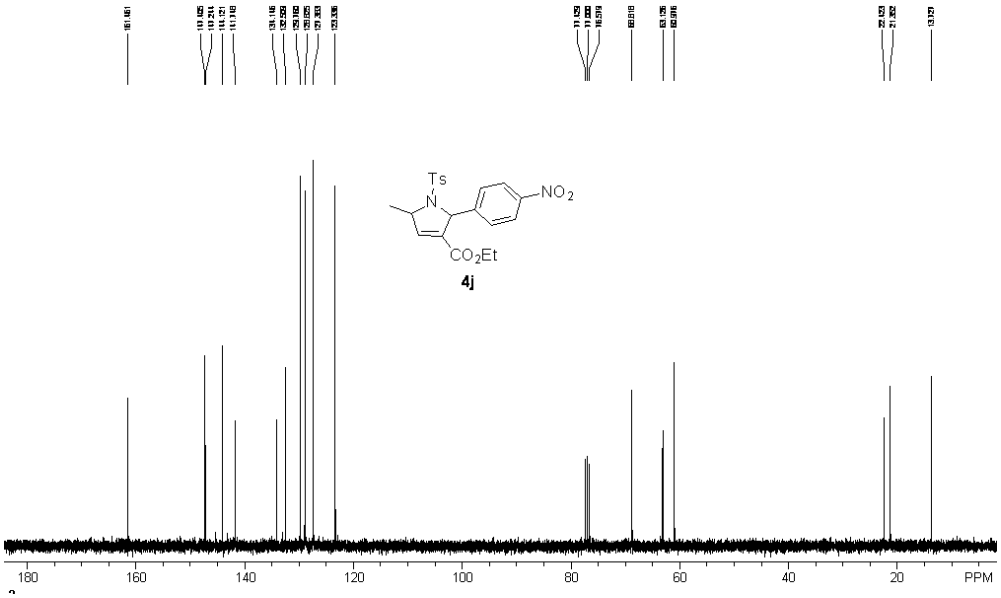
**Ethyl 5-Methyl-2-(3-nitrophenyl)-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4i**: mp. 138-140 $^{o}$C. IR ($CH_2Cl_2$) ν 2966, 1721 (C=O), 1349, 1259, 1164, 1093, 666 $cm^{-1}$. $^{1}H$ NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4i** δ 1.12 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.66 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.38 (3H, s, $CH_3$), 3.99-4.05 (2H, m, $CH_2$), 4.78-4.81 (1H, m, CH), 5.69-5.70 (1H, m, CH), 6.71-6.72 (1H, m, =CH), 7.25 (2H, d, *J* = 8.4 Hz, ArH), 7.46-7.52 (1H, m, ArH), 7.63 (2H, d, *J* = 8.4 Hz, ArH), 7.74-7.76 (1H, m, ArH), 8.10-8.15 (2H, m, ArH); ***anti*-4i** δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.72 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.28 (3H, s, $CH_3$), 3.99-4.05 (2H, m, $CH_2$), 5.02-5.08 (1H, m, CH), 5.85-5.87 (1H, m, CH), 6.79-6.81 (1H, m, =CH), 6.99 (2H, d, *J* = 8.4 Hz, ArH), 7.14 (2H, d, *J* = 8.4 Hz, ArH), 7.45-7.52 (2H, m, ArH), 8.05-8.09 (2H, m, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4i** δ 13.8, 21.4, 22.6, 61.0, 63.1, 68.9, 122.7, 122.8, 127.4, 129.1, 129.8, 132.7, 134.2, 134.4, 141.8, 142.4, 144.1, 148.0, 161.5. MS (EI) *m/z* 415 ($M^+$-15, 24.85), 343 ($M^+$-87, 14.44), 275 ($M^+$-155, 3.69), 155 ($M^+$-275, 53.48), 91 ($M^+$-339, 100). Found: C, 58.56; H, 5.09; N, 6.45%. $C_{21}H_{22}O_6N_2S$ requires C, 58.59; H, 5.15; N, 6.51%.



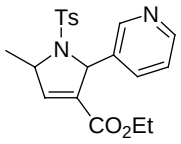




**4j**

**Ethyl 5-Methyl-2-(4-nitrophenyl)-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4j**: mp. 108-110 $^{o}$C. IR ($CH_2Cl_2$) ν 2986, 1720 (C=O), 1525, 1349, 1266, 1165, 1093, 666 $cm^{-1}$. $^{1}H$ NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4j** δ 1.12 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.62 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 3.97-4.07 (2H, m, $CH_2$), 4.76-4.79 (1H, m, CH), 5.68-5.70 (1H, m, CH), 6.68-6.70 (1H, m, =CH), 7.26 (2H, d, *J* = 7.8 Hz, ArH), 7.54 (2H, d, *J* = 9.3 Hz, ArH), 7.62 (2H, d, *J* = 7.8 Hz, ArH), 8.16 (2H, d, *J* = 9.3 Hz, ArH); ***anti*-4j** δ 1.26 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.67 (3H, d, *J* = 6.9 Hz, $CH_3$), 2.35 (3H, s, $CH_3$), 4.03-4.07 (2H, m, $CH_2$), 4.97-5.03 (1H, m, CH), 5.82-5.83 (1H, m, CH), 6.77-6.79 (1H, m, =CH), 7.03 (2H, d, *J* = 7.8 Hz, ArH), 7.19-7.22 (2H, m, ArH), 7.52-7.56 (2H, m, ArH), 7.96 (2H, d, *J* = 8.7 Hz, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4j** δ 13.7, 21.4, 22.4, 61.0, 63.1, 68.8, 123.3, 127.4, 128.8, 129.8, 132.5, 134.2, 141.8, 144.1, 147.2, 147.4, 161.5. MS (EI) *m/z* 430 ($M^+$, 1.14), 415 ($M^+$-15, 25.62), 343 ($M^+$-87, 11.09), 275 ($M^+$-155, 8.63), 155 ($M^+$-275, 55.24), 91 ($M^+$-339, 100). Found: C, 58.71; H, 5.08; N, 6.51%. $C_{21}H_{22}O_6N_2S$ requires C, 58.59; H, 5.15; N, 6.51%.

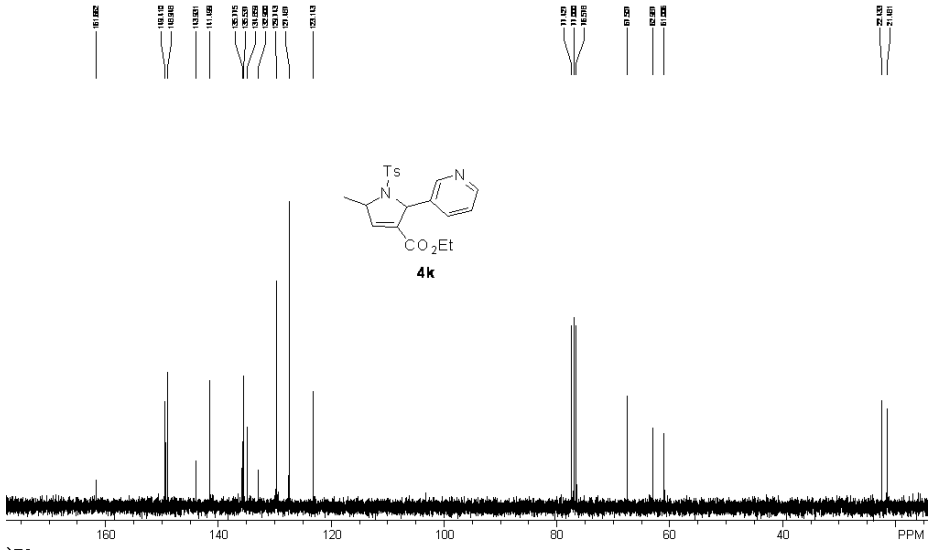



**4j**

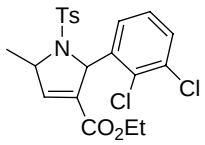



**4k**

**Ethyl 5-Methyl-2-(pyridin-3-yl)-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4k**: a colorless oil. IR ($CH_2Cl_2$) ν 2869, 1720 (C=O), 1597, 1343, 1262, 1164, 1093, 666 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4k** δ 1.09 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.58 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.38 (3H, s, $CH_3$), 3.95-4.04 (2H, m, $CH_2$), 4.77-4.79 (1H, m, CH), 5.63-5.64 (1H, m, CH), 6.67-6.68 (1H, m, =CH), 7.19-7.24 (3H, m, ArH), 7.58-7.66 (3H, m, ArH), 8.48-8.50 (1H, m, ArH), 8.56-8.57 (1H, m, ArH); ***anti*-4k** δ 1.24 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.67 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 4.05-4.12 (2H, m, $CH_2$), 4.86-4.91 (1H, m, CH), 5.77-5.79 (1H, m, CH), 6.75-6.76 (1H, m, =CH), 7.01-7.04 (2H, m, ArH), 7.11-7.14 (2H, m, ArH), 7.33-7.36 (2H, m, ArH), 8.28-8.29 (1H, m, ArH), 8.41-8.43 (1H, m, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4k** δ 13.8, 21.5, 22.4, 61.0, 63.0, 67.5, 123.1, 127.5, 129.7, 132.9, 134.9, 135.5, 135.8, 141.5, 143.9, 149.0, 149.4, 161.7. MS (EI) *m/z* 386 ($M^+$, 2.27), 371 ($M^+$-15, 9.53), 299 ($M^+$-87, 3.80), 231 ($M^+$-155, 8.78), 155 ($M^+$-231, 24.31), 108 ($M^+$-278, 77.17), 91 ($M^+$-295, 100). HRMS calcd. for $C_{20}H_{23}N_2O_4S^{+1}$: 387.1373, Found: 387.1380.

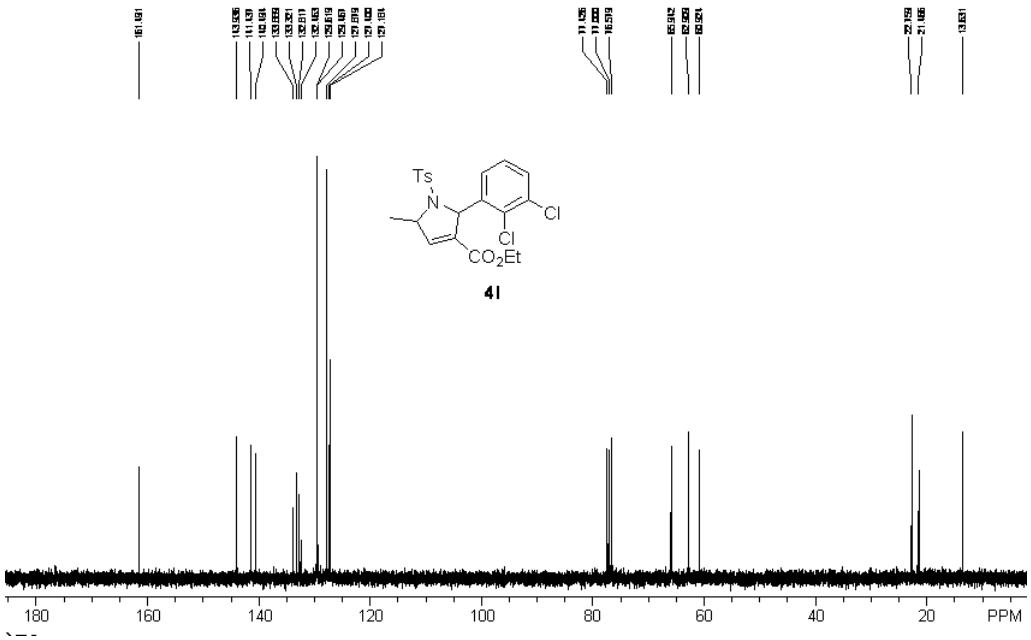



4k




**4l**

**Ethyl 2-(2,3-Dichlorophenyl)-5-methyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 4l**: mp. 103-105 $^{o}$C. IR ($CH_2Cl_2$) ν 2981, 1720 (C=O), 1598, 1351, 1268, 1166, 1092, 665 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) ***syn*-4l** δ 1.05 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.70 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.41 (3H, s, $CH_3$), 3.91-4.04 (2H, m, $CH_2$), 4.74-4.78 (1H, m, CH), 6.16-6.17 (1H, m, CH), 6.67-6.68 (1H, m, =CH), 7.17-7.19 (2H, m, ArH), 7.25-7.35 (3H, m, ArH), 7.72 (2H, d, *J* = 7.2 Hz, ArH); ***anti*-4l** δ 1.05 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.58 (3H, d, *J* = 6.6 Hz, $CH_3$), 2.28 (3H, s, $CH_3$), 3.94-4.05 (2H, m, $CH_2$), 4.91-4.96 (1H, m, CH), 6.16-6.17 (1H, m, CH), 6.69-6.70 (1H, m, =CH), 7.14-7.17 (2H, m, ArH), 7.25-7.35 (3H, m, ArH), 7.72 (2H, d, *J* = 7.2 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) ***syn*-4l** δ 13.6, 21.5, 22.8, 60.9, 62.9, 65.9, 127.2, 127.4, 127.9, 129.5, 129.6, 132.5, 132.8, 133.3, 133.9, 140.5, 141.4, 143.9, 161.5. MS (EI) *m/z* 453 ($M^+$, 1.90), 438 ($M^+$-15, 10.53), 300 ($M^+$-153, 2.64), 155 ($M^+$-298, 38.02), 91 ($M^+$-362, 100). Found: C, 55.66; H, 4.61; N, 3.18%. $C_{21}H_{21}O_4NSCl_2$ requires C, 55.51; H, 4.66; N, 3.08%.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CO2Et
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160
140
120
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80
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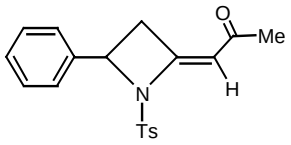
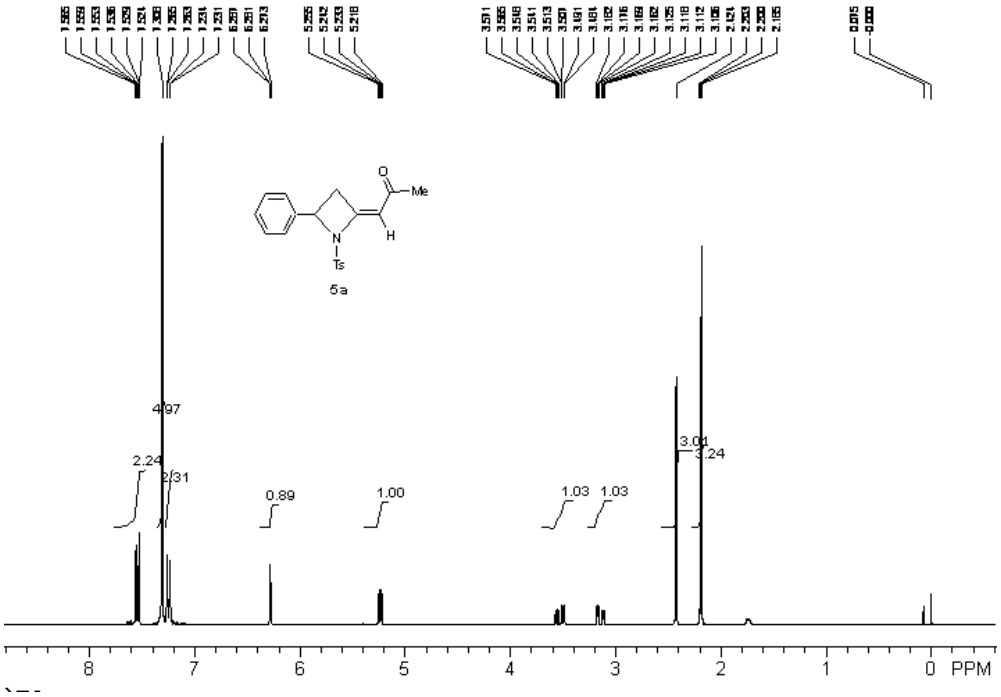
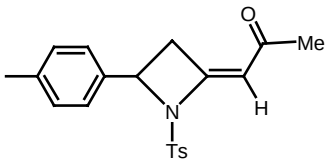
20
PPM




**5 a**

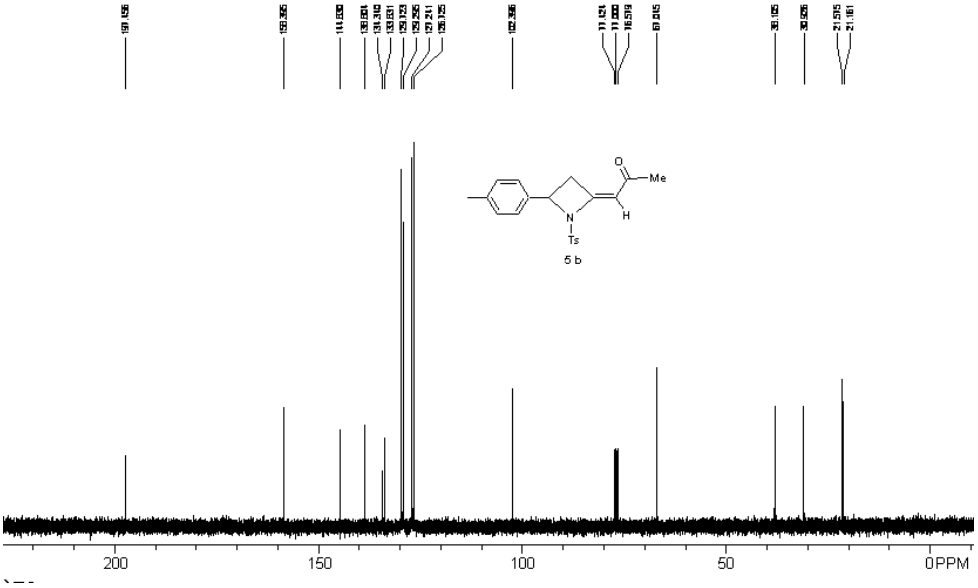
**1-[4-Phenyl-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5a**: mp. 123-125 $^{o}$C. IR ($CH_2Cl_2$) ν 2928, 1690 (C=O), 1495, 1425, 1163, 922, 740 cm$^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.19 (3H, s, $CH_3$), 2.43 (3H, s, $CH_3$), 3.15 (1H, ddd, *J* = 17.1, 3.9, 1.8 Hz, $CH_2$), 3.53 (1H, ddd, *J* = 17.1, 6.6, 1.8 Hz, $CH_2$), 5.23 (1H, dd, *J* = 6.6, 3.9 Hz, CH), 6.27-6.28 (1H, m, =CH), 7.25-7.29 (2H, m, Ar), 7.31 (5H, s, Ar), 7.53-7.56 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.1, 67.1, 102.5, 126.7, 127.2, 128.7, 128.9, 129.8, 134.3, 136.8, 144.9, 158.3, 197.5. MS (EI) *m/z* 341 ($M^+$, 0.78), 260 ($M^+$-81, 1.77), 186 ($M^+$-155, 3.95), 155 ($M^+$-186, 14.63), 144 ($M^+$-197,31.76), 104 ($M^+$-237, 29.18), 91 ($M^+$-250, 100). Anal. Calcd. for $C_{19}H_{19}NO_3S$ requires C, 66.85; H, 5.61; N, 4.10%. Found: C, 66.57; H, 5.60; N, 3.95%.

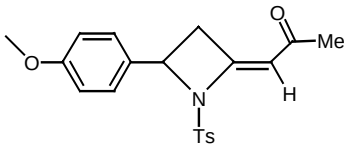



**5 b**

**1-[1-(Toluene-4-sulfonyl)-4-*p*-tolyl-azetidin-2-ylidene]propan-2-one 5b**: a colorless oil. IR ($CH_2Cl_2$) ν 2923, 1692 (C=O), 1608, 1359, 1166, 1089, 815 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.20 (3H, s, $CH_3$), 2.34 (3H, s, $CH_3$), 2.42 (3H, s, $CH_3$), 3.13 (1H, ddd, J = 17.1, 3.9, 2.4 Hz, $CH_2$), 3.50 (1H, ddd, *J* = 17.1, 6.6, 2.1 Hz, $CH_2$), 5.20 (1H, dd, *J* = 6.6, 3.9 Hz, CH), 6.26-6.27 (1H, m, =CH), 7.11 (2H, d, *J* = 6.9 Hz, Ar), 7.18-7.27 (4H, m, Ar), 7.55 (2H, dd, *J* = 6.9 Hz, 1.8 Hz, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.2, 21.6, 30.9, 38.1, 67.1, 102.4, 126.7, 127.2, 129.3, 129.7, 133.8, 134.3, 138.8, 144.8, 158.4, 197.5. MS (EI) *m/z* 355 ($M^+$, 0.66), 200 ($M^+$-155, 11.68), 158 ($M^+$-197, 100), 155 ($M^+$-200, 16.17), 118 ($M^+$-237), 91 ($M^+$-264, 63.57). HRMS calcd. for $C_{20}H_{21}NO_3S$: 355.1242, Found: 355.1246.

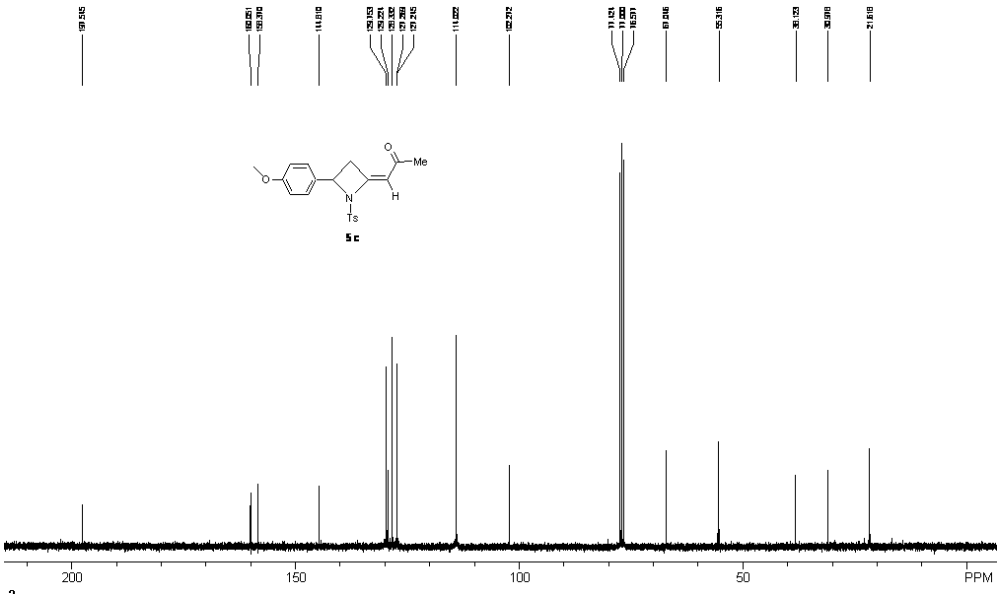


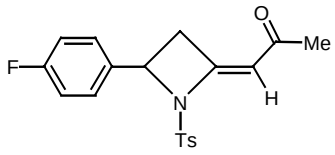




**5 c**

**1-[4-(4-Methoxyphenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5c**: a white solid. mp. 95-97 $^oC$. IR ($CH_2Cl_2$) ν 2925, 1701 (C=O), 1604, 1360, 1165, 1089, 910 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.19 (3H, s, $CH_3$), 2.43 (3H, s, $CH_3$), 3.15 (1H, ddd, *J* = 16.8, 4.2, 2.1 Hz, $CH_2$), 3.51 (1H, ddd, *J* = 16.8, 6.9, 2.4 Hz, $CH_2$), 3.81 (3H, s, $OCH_3$), 5.21 (1H, dd, *J* = 6.9, 4.2 Hz, CH), 6.25-6.26 (1H, m, =CH), 6.82 (2H, d, *J* = 6.9 Hz, Ar), 7.20-7.27 (4H, m, Ar), 7.51-7.55 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.1, 55.4,

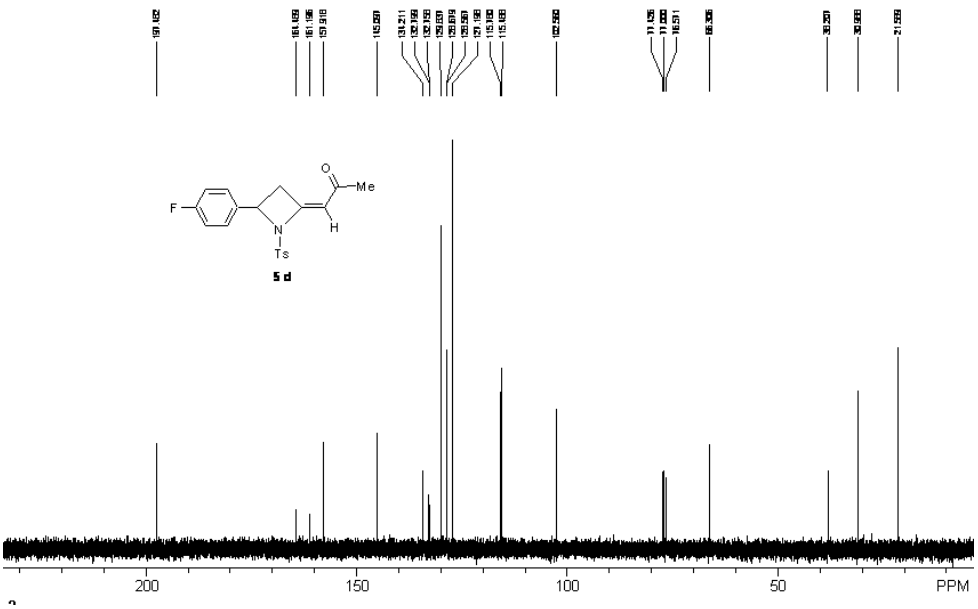
67.1, 102.3, 114.0, 127.3, 127.3, 128.3, 129.2, 129.8, 144.8, 158.4, 160.1, 197.6. MS (EI) *m/z* 371 ($M^+$, 3.07), 216 ($M^+$-155, 20.73), 174 ($M^+$-197, 100), 155 ($M^+$-216, 9.46), 91 ($M^+$-280, 51.30). Anal. Calcd. for $C_{20}H_{21}NO_4S$ requires C, 64.68; H, 5.70; N, 3.77%. Found: C, 64.33; H, 5.65; N, 3.53%.

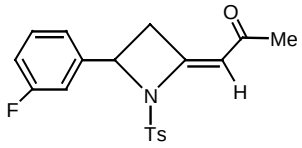



**5 d**

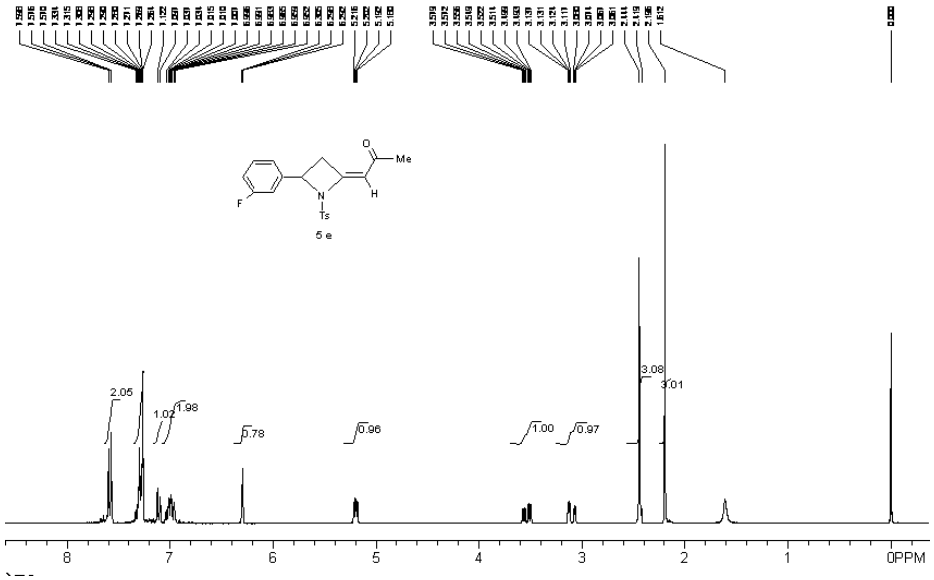
**1-[4-(4-Fluorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5d**: a white solid. mp. 140-142 $^oC$. IR ($CH_2Cl_2$) ν 2922, 1692 (C=O), 1608, 1360, 1167, 1089, 836 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.22 (3H, s, $CH_3$), 2.44 (3H, s, $CH_3$), 3.12 (1H, ddd, *J* = 17.1, 4.2, 1.8 Hz, $CH_2$), 3.53 (1H, ddd, *J* = 17.1, 6.9, 2.1 Hz, $CH_2$), 5.21 (1H, dd, *J* = 6.9, 4.2 Hz, CH), 6.28-6.29 (1H, m, =CH), 6.97-7.03 (2H, m, Ar), 7.26-7.32 (4H, m, Ar), 7.54-7.62 (2H, m, Ar). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.2, 66.3, 102.6, 115.6 (d, $^2J_{C-F}$ = 21.9 Hz), 127.2, 128.6 (d, $^3J_{C-F}$ = 8.4 Hz), 129.9, 132.8 (d, $^4J_{C-F}$ = 3.1 Hz), 134.2, 145.1, 157.9, 162.8 (d, $^1J_{C-F}$ = 247.0 Hz), 197.5. MS (EI) *m/z* 359 ($M^+$, 2.05), 204 ($M^+$-155, 13.14), 162 ($M^+$-197, 100), 155 ($M^+$-204, 22.06), 91 ($M^+$-268, 75.49). Anal. Calcd. for $C_{19}H_{18}FNO_3S$ requires C, 63.50; H, 5.05; N, 3.90%. Found: C, 63.35; H, 4.94; N, 3.62%.

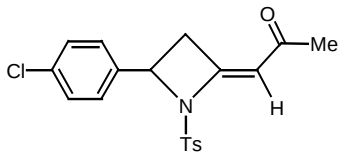



**5 e**

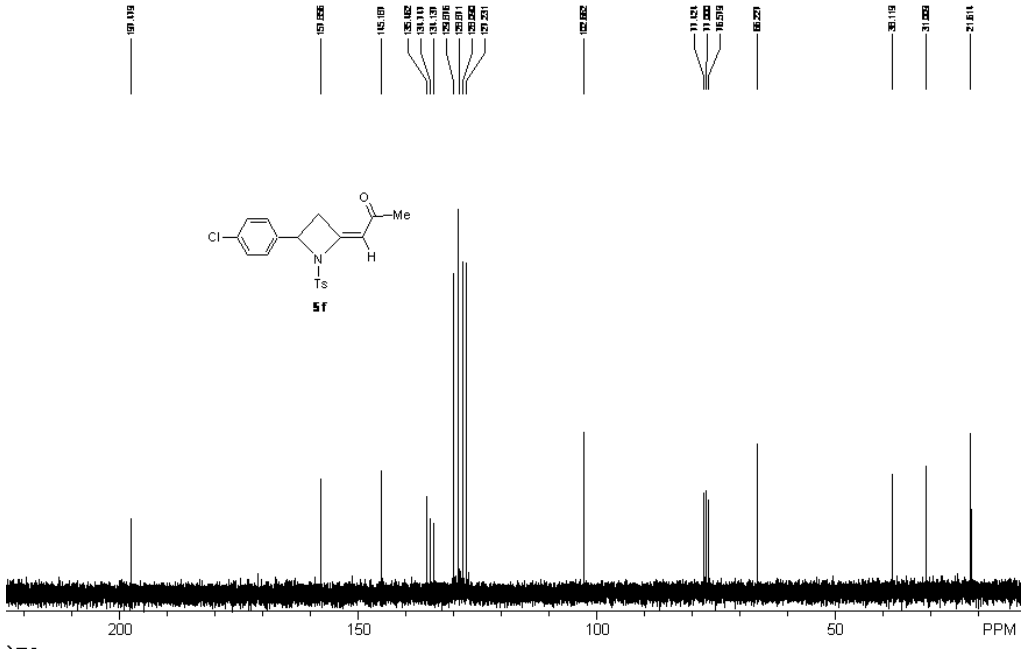
**1-[4-(3-Fluorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5e**: a white solid. mp. 125-127 $^{o}$C. IR ($CH_2Cl_2$) ν 2925, 1692 (C=O), 1611, 1359, 1167, 1088, 674 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.20 (3H, s, $CH_3$), 2.44 (3H, s, $CH_3$), 3.10 (1H, ddd, *J* = 17.4, 3.9, 1.2 Hz, $CH_2$), 3.54 (1H, ddd, *J* = 17.4, 7.2, 1.8 Hz, $CH_2$), 5.20 (1H, dd, *J* = 7.2, 3.9 Hz, CH), 6.29-6.31 (1H, m, =CH), 6.95-7.04 (2H, m, Ar), 7.10-7.12 (1H, m, Ar), 7.26-7.32 (3H, m, Ar), 7.57-7.60 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.2, 66.2 (d, *J* = 2.3 Hz), 102.8, 113.5 (d, *J* = 22.5 Hz), 115.8 (d, *J* = 21.1 Hz), 122.4 (d, *J* = 3.0 Hz), 127.3, 129.9, 130.4 (d, *J* = 8.0 Hz), 134.1, 139.5 (d, *J* = 7.0 Hz), 145.2, 157.8, 162.8 (d, *J* = 246.2 Hz), 197.5. MS (EI) *m/z* 359 ($M^+$, 5.07), 204 ($M^+$-155, 12.52), 162 ($M^+$-197, 71.41), 155 ($M^+$-204, 31.54), 91 ($M^+$-268, 100). Anal. Calcd. for $C_{19}H_{18}FNO_3S$ requires C, 63.50; H, 5.05; N, 3.90%. Found: C, 63.67; H, 5.21; N, 3.63%.

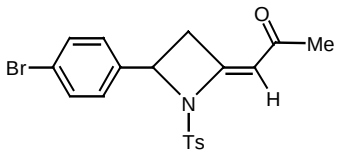



**5 f**

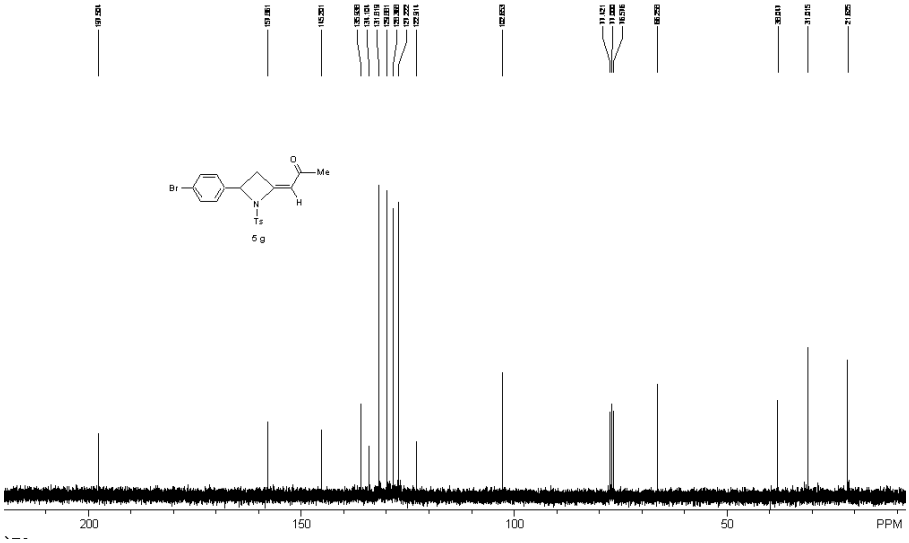
**1-[4-(4-Chlorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]-propan-2-one 5f**: a white solid. mp. 152-153 $^{o}$C. IR ($CH_2Cl_2$) ν 1695 (C=O), 1609, 1360, 1166, 1088, 677 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.19 (3H, s, $CH_3$), 2.44 (3H, s, $CH_3$), 3.09 (1H, ddd, *J* = 17.1, 4.2, 2.1 Hz, $CH_2$), 3.52 (1H, ddd, *J* = 17.1, 7.2, 2.1 Hz, $CH_2$), 5.19 (1H, dd, *J* = 7.2, 4.2 Hz, CH), 6.28-6.29 (1H, m, =CH), 7.03-7.18 (2H, m, Ar), 7.21-7.30 (4H, m, Ar), 7.54-7.64 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.1, 66.2, 102.7, 127.2, 128.1, 128.9, 129.9, 134.1, 134.8, 135.5, 145.2, 157.9, 197.5. MS (EI) *m/z* 375 ($M^+$, 2.07), 220 ($M^+$-155, 15.13), 180 ($M^+$-195, 32.27), 178 ($M^+$-197, 100), 155 ($M^+$-220, 31.05), 91 ($M^+$-284, 95.73). Anal. Calcd. for $C_{19}H_{18}ClNO_3S$ requires C, 60.72; H, 4.83; N, 3.73%. Found: C, 60.74; H, 4.92; N, 3.54%.

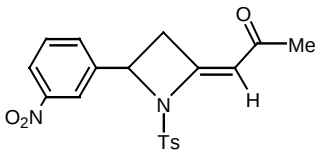



**5 g**

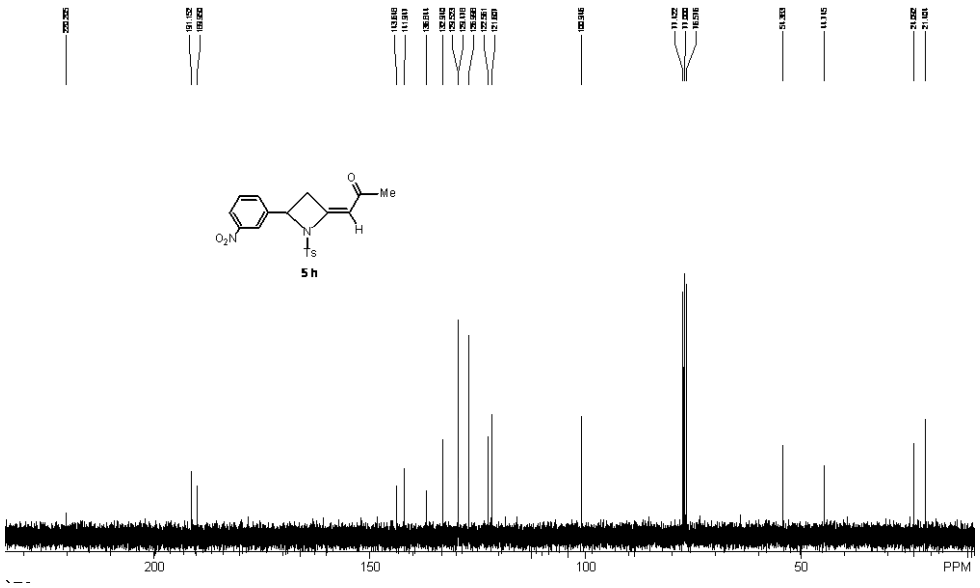
**1-[4-(4-Bromophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5g**: a white solid. mp. 110-113 $^{o}$C. IR ($CH_2Cl_2$) ν 2922, 1719 (C=O), 1598, 1333, 1159, 1093, 813 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.22 (3H, s, $CH_3$), 2.45 (3H, s, $CH_3$), 3.09 (1H, ddd, *J* = 17.1, 3.9, 1.8 Hz, $CH_2$), 3.53 (1H, ddd, *J* = 17.1, 6.9, 1.5 Hz, $CH_2$), 5.17 (1H, dd, *J* = 6.9, 3.9 Hz, CH), 6.28-6.29 (1H, m, =CH), 7.16-7.24 (2H, m, Ar), 7.26-7.34 (2H, m, Ar), 7.42-7.46 (2H, m, Ar), 7.55-7.60 (2H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.1, 66.3, 102.7, 122.9, 127.2, 128.4, 129.9, 131.8, 134.1, 135.9, 145.2, 157.9, 197.5. MS (EI) *m/z* 422 ($M^+$+3, 3.56), 420 ($M^+$+1, 1.44), 340 ($M^+$-79, 83.68), 338 ($M^+$-81, 84.39), 282 ($M^+$-137, 26.95), 155 ($M^+$-264, 84.54), 91 ($M^+$-328, 100). Anal. Calcd. for $C_{19}H_{18}BrNO_3S$ requires C, 54.30; H, 4.32; N, 3.33%. Found: C, 54.93; H, 4.47; N, 2.88%.

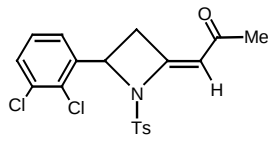



**5 h**

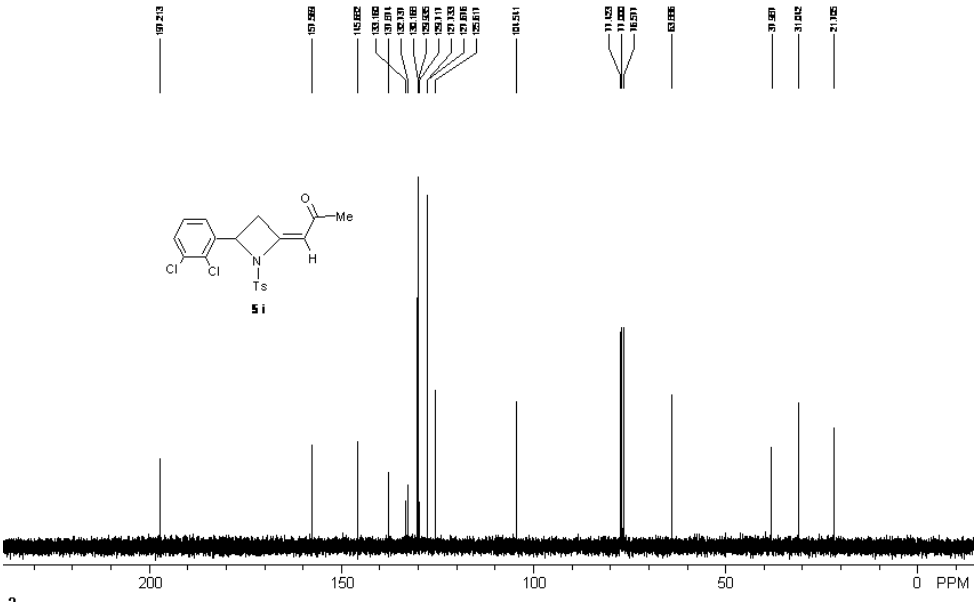
**1-[4-(3-Nitrophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5h**: a white solid. mp. 103-105 $^{o}$C. IR ($CH_2Cl_2$) ν 2924, 1701 (C=O), 1598, 1530, 1349, 1160, 1093, 814 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.21 (3H, s, $CH_3$), 2.44 (3H, s, $CH_3$), 3.12 (1H, ddd, *J* = 17.1, 4.2, 2.1 Hz, $CH_2$), 3.60 (1H, ddd, *J* = 17.1, 6.6, 1.2 Hz, $CH_2$), 5.27-5.31 (1H, m, CH), 6.34 (1H, s, =CH), 7.29-7.31 (2H, m, Ar), 7.53-7.62 (1H, m, Ar), 7.73-7.82 (3H, m, Ar), 8.07 (1H, s, Ar), 8.17-8.20 (1H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.7, 31.3, 38.5, 65.8, 103.5, 121.7, 124.0, 126.6, 129.9, 133.1, 134.1, 139.4, 139.6, 143.7, 145.9, 157.6, 197.9. MS (EI) *m/z* 386 ($M^+$, 6.02), 231 ($M^+$-155, 6.31), 171 ($M^+$-215, 35.90), 155 ($M^+$-231, 39.15), 91 ($M^+$-295, 100). HRMS calcd. for $C_{19}H_{18}N_2O_4S$: 386.0936, Found: 386.0920.

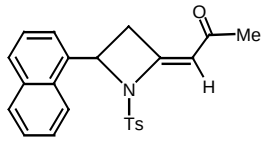



**5 i**

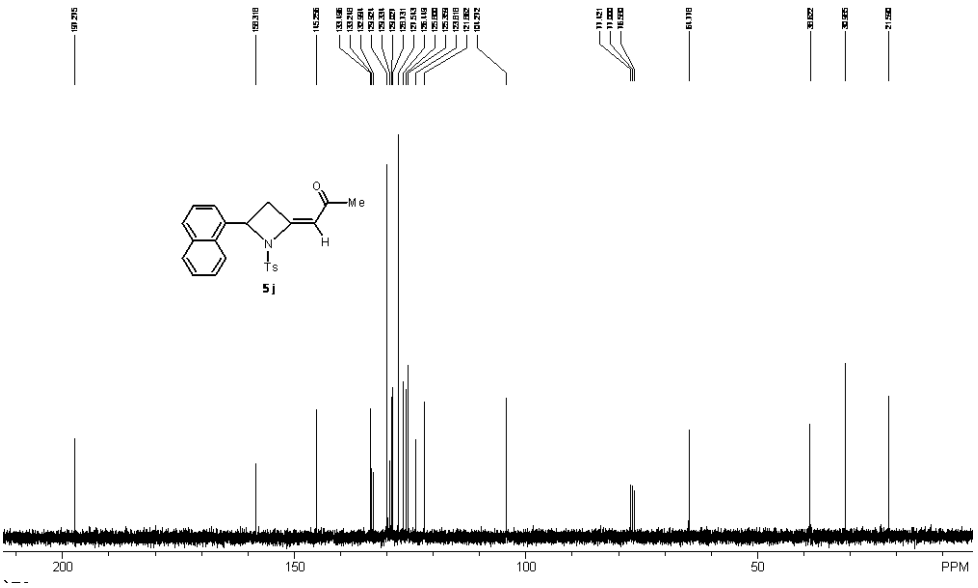
**1-[4-(2,3-Dichlorophenyl)-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5i**: a white solid. mp. 135-137 $^{o}$C. IR ($CH_2Cl_2$) ν 1695 (C=O), 1614, 1360, 1166, 1133, 1088, 609 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.18 (3H, s, $CH_3$), 2.48 (3H, s, $CH_3$), 2.93 (1H, ddd, *J* = 17.1, 4.2, 2.1 Hz, $CH_2$), 3.57 (1H, ddd, *J* = 17.1, 7.2, 1.8 Hz, $CH_2$), 5.40 (1H, dd, *J* = 4.2 Hz, 7.2 Hz, CH), 6.37-6.38 (1H, m, =CH), 7.26-7.31 (1H, m, Ar), 7.37-7.47 (3H, m, Ar), 7.66-7.77 (3H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.7, 31.0, 38.0, 63.9, 104.5, 125.6, 127.7, 127.7, 129.7, 129.9, 130.2, 132.7, 133.2, 137.9, 145.7, 157.6, 197.2. MS (EI) *m/z* 411 ($M^{+}$+2, 3.49), 409 ($M^{+}$, 4.86), 254 ($M^{+}$-155, 10.03), 212 ($M^{+}$-197, 26.08), 155 ($M^{+}$-254, 31.55), 91 ($M^{+}$-318, 100). Anal. Calcd. for $C_{19}H_{17}Cl_2NO_3S$ requires C, 55.62; H, 4.18; N, 3.42%. Found: C, 55.66; H, 4.31; N, 3.17%.

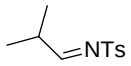



**5 j**

**1-[4-Naphthalen-1-yl-1-(toluene-4-sulfonyl)-azetidin-2-ylidene]propan-2-one 5j**: a white solid. mp. 108-110 $^{o}$C. IR ($CH_2Cl_2$) ν 1696 (C=O), 1599, 1359, 1165, 1088, 777 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.20 (3H, s, $CH_3$), 2.46 (3H, s, $CH_3$), 3.09 (1H, ddd, *J* = 17.1, 4.2, 2.4 Hz, $CH_2$), 3.70 (1H, ddd, *J* = 17.1, 7.2, 2.4 Hz, $CH_2$), 5.82 (1H, dd, *J* = 4.2 Hz, 7.2 Hz, CH), 6.44-6.45 (1H, m, =CH), 7.31 (2H, d, *J* = 7.2 Hz, Ar), 7.46-7.51 (3H, m, Ar), 7.61-7.65 (1H, m, Ar), 7.70 (2H, dd, *J* = 7.2 Hz, 1.8 Hz, Ar), 7.81-7.91 (3H, m, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 31.0, 38.6, 64.8, 104.3, 121.9, 123.8, 125.4, 125.8, 126.5, 127.5, 128.7, 129.0, 129.3, 129.9, 133.0, 133.3, 133.5, 145.3, 158.3, 197.3. MS (EI) *m/z* 393 ($M^+$+2, 0.63), 391 ($M^+$, 3.31), 236 ($M^+$-155, 16.87), 194 ($M^+$-197, 100), 155 ($M^+$-236, 22.61), 153 ($M^+$-238, 51.33), 91 ($M^+$-300, 56.29). Anal. Calcd. for $C_{23}H_{21}NO_3S$ requires C, 70.57; H, 5.41; N, 3.58%. Found: C, 70.28; H, 5.31; N, 3.31%.

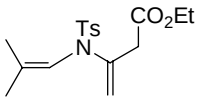



**6**

***N*-Isobutylidene-4-methylbenzenesulfonamide 6**:[1] $^1$H NMR (300 MHz, $CDCl_3$, TMS): δ 1.21 (6H, d, *J* = 7.2 Hz, Me), 2.40 (3H, s, $CH_3$), 2.72 (1H, hept, *J* = 2.6 Hz, CH), 7.31 (2H, d, *J* = 8.2 Hz, ArH), 7.80 (2H, d, *J* = 8.2 Hz, ArH), 8.50 (1H, d, *J* = 4.2 Hz, =CH).

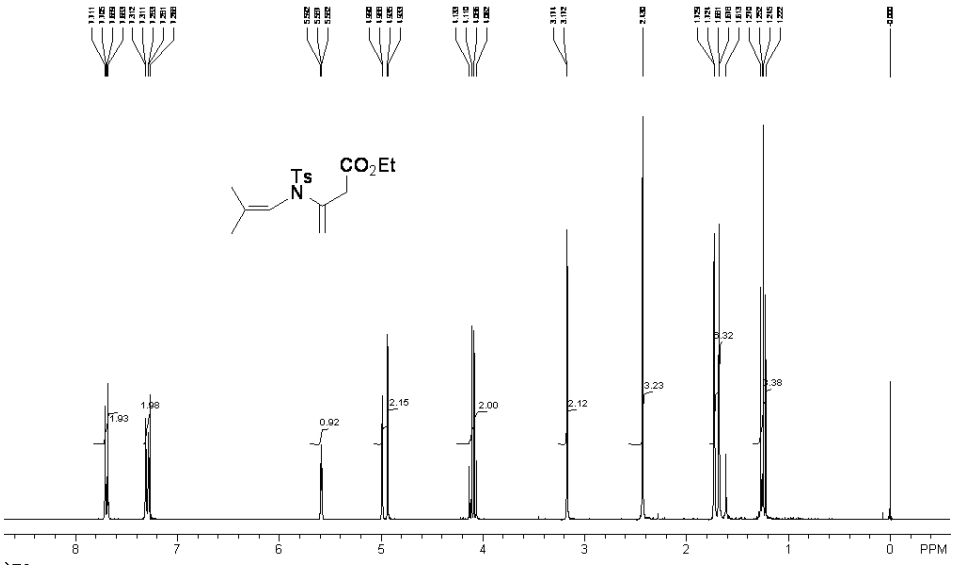
1. Chemla, F.; Hebbe, V.; Normant, J.-F. *Synlett* **2000**, 75-77.

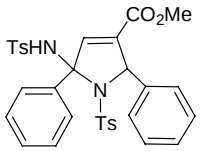



**7**

**3-[(2-Methylpropenyl)-(toluene-4-sulfonyl)amino]-but-3-enoic acid ethyl ester 7**: a colorless oil; IR ($CH_2Cl_2$) ν 3064, 1736 (C=O), 1610, 1354, 1276, 1164, 1090, 1033 cm$^{-1}$; $^1$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.25 (3H, t, *J* = 7.2 Hz, $CH_3$), 1.68 (3H, d, *J* = 1.5 Hz, $CH_3$), 1.73 (3H, d, *J* = 1.5 Hz, $CH_3$), 2.43 (3H, s, $CH_3$), 3.17 (2H, d, *J* = 0.6 Hz, $CH_2$), 4.10 (2H, q, *J* = 7.2 Hz , $CH_2$), 4.94 (1H, d, *J* = 0.9 Hz, =CH), 4.99 (1H, d, *J* = 0.9 Hz, =CH), 5.58-5.59 (1H, m, =CH), 7.30 (2H, dd, *J* = 7.2 Hz, 0.6 Hz, Ar), 7.70 (2H, dd, *J* = 7.2 Hz, 0.6 Hz, Ar). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 14.0, 17.7, 21.4, 22.1, 41.0, 60.8, 112.5, 119.9, 127.8, 129.3, 135.3, 140.3, 140.4, 143.6, 169.8. MS (EI) m/z 337 ($M^+$, 1.80), 273 ($M^+$-64, 10.34), 182 ($M^+$-155, 16.41), 168 ($M^+$-169, 59.11), 155 ($M^+$-182, 19.32), 108 ($M^+$-229, 100), 91 ($M^+$-246, 48.73);

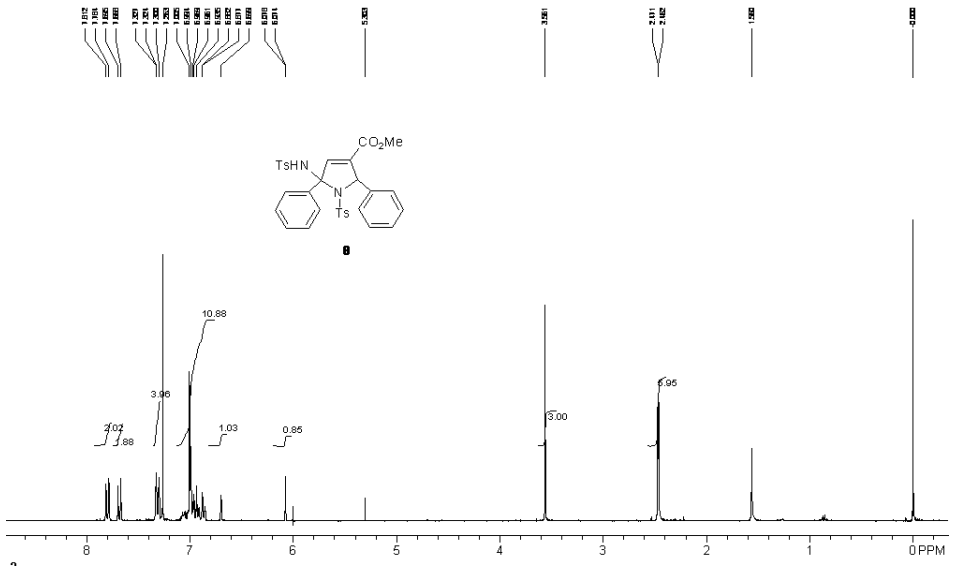
HRMS calcd. for $C_{17}H_{23}NO_4S$: 337.1348, Found: 337.1339.

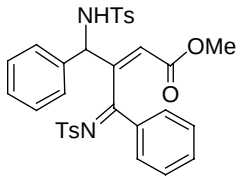



**8**

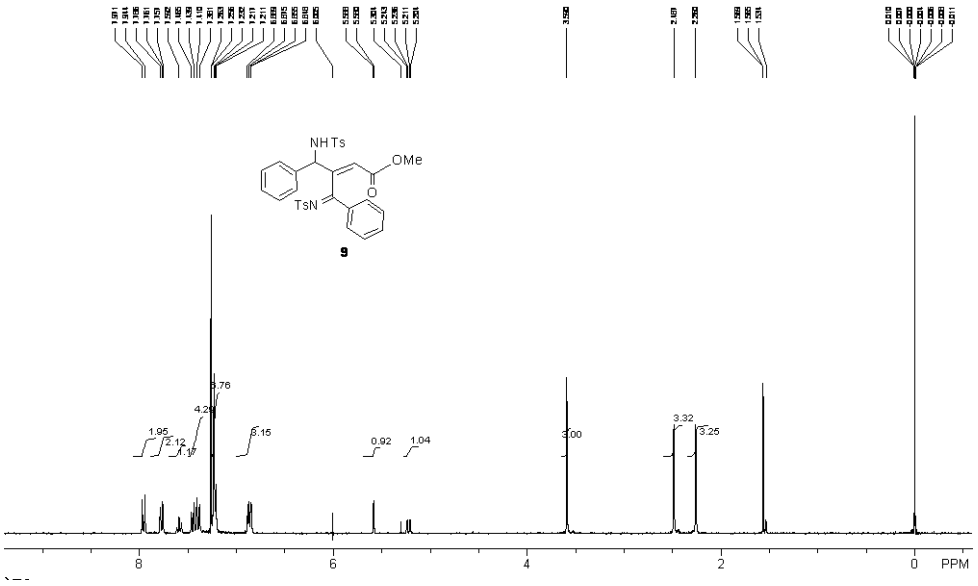
**Methyl-5-(4-methylphenylsulfonamido)-2,5-diphenyl-1-tosyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate 8**: mp. 148-152 $^{o}$C. IR ($CH_2Cl_2$) ν 2952, 1728 (C=O), 1636, 1345, 1161, 1089 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.46 (3H, s, $CH_3$), 2.47 (3H, s, $CH_3$), 3.56 (3H, s, $OCH_3$), 6.08 (1H, d, *J* = 1.2 Hz, CH), 6.70 (1H, s, =CH), 6.84-7.09 (11H, m, ArH, NH), 7.31 (4H, d, *J* = 8.1 Hz, ArH), 7.68 (2H, d, *J* = 8.1 Hz, ArH), 7.80 (2H, d, *J* = 8.1 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.7, 21.8, 51.3, 65.3, 78.6, 96.3, 126.8, 126.9, 127.4, 127.6, 127.7, 128.2, 128.2, 128.3, 129.8, 130.1, 133.9, 134.0, 135.2, 136.9, 145.0, 145.6, 150.6, 166.1. MS (EI) *m/z* 601 ($M^+$-1, 2.02), 447 ($M^+$-155, 72.83), 415 ($M^+$-187, 29.61), 383 ($M^+$-219, 100), 260 ($M^+$-342, 41.83), 194 ($M^+$-408, 56.39), 91 ($M^+$-511, 69.90); HRMS (MALDI) calcd. for $C_{32}H_{30}N_2O_6S_2Na^{+1}$: 625.1438, Found: 625.1434.

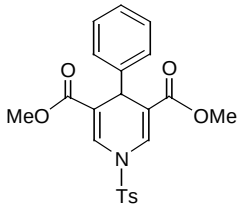



**9**

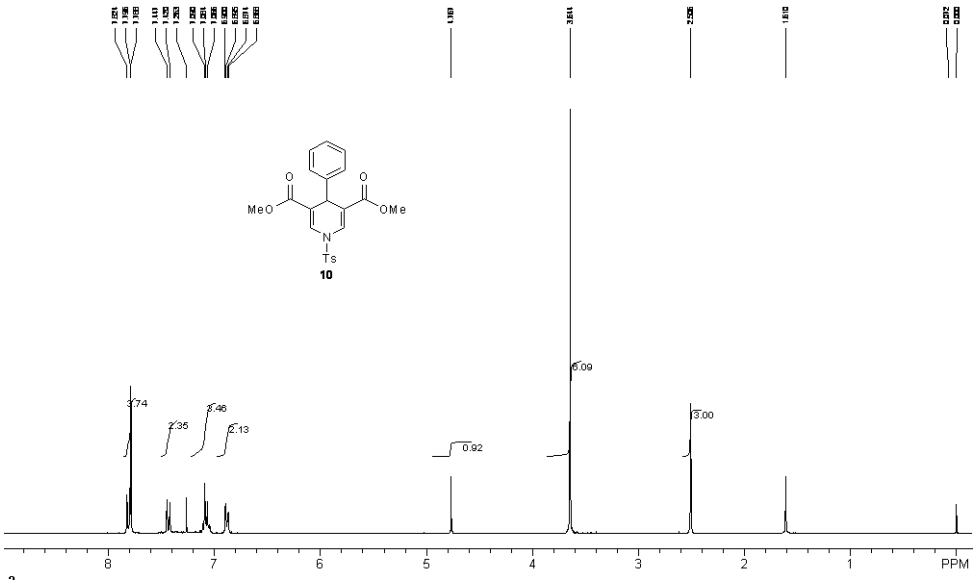
**(*Z*)-Methyl-4-(4-methylphenylsulfonamido)-3-((4-methylphenylsulfonamido)(phenyl)methyl)-4-phenylbut-2-enoate 9**: mp. 215-219 $^{o}$C. IR ($CH_2Cl_2$) ν 3298, 1727 (C=O), 1560, 1335, 1159, 1088 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.26 (3H, s, $CH_3$), 2.49 (3H, s, $CH_3$), 3.59 (3H, s, $OCH_3$), 5.22 (1H, dd, *J* = 6.6, 1.2 Hz, CH), 5.58 (1H, d, *J* = 1.2 Hz, =CH), 6.85-6.89 (3H, m, NH, ArH), 7.21-7.26 (7H, m, ArH), 7.38-7.47 (4H, m, ArH), 7.57-7.59 (1H, m, ArH), 7.77 (2H, d, *J* = 8.4 Hz, ArH), 7.96 (2H, d, *J* = 8.4 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.4, 21.7, 52.1, 61.5, 125.2, 126.6, 127.2, 128.2, 128.4, 128.9, 128.9, 129.0, 129.3, 129.6, 133.3, 133.9, 136.4, 137.4, 137.5, 142.6, 144.1, 153.4, 164.5, 178.1. MS (EI) *m/z* 447 ($M^+$-155, 54.09), 415 ($M^+$-187, 2.86), 260 ($M^+$-342, 14.81), 155 ($M^+$-447, 38.26), 91 ($M^+$-511, 100). HRMS (MALDI) calcd. for $C_{32}H_{30}N_2O_6S_2Na^{+1}$: 625.1438, Found: 625.1409.

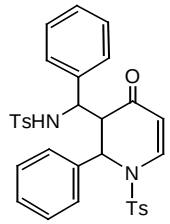



**10**

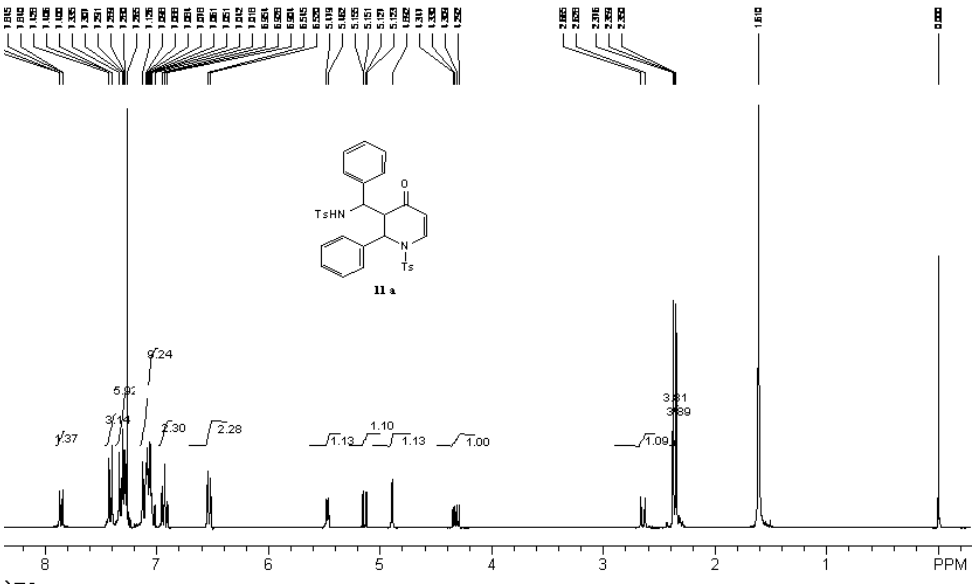
**Dimethyl-4-phenyl-1-tosyl-1,4-dihydropyridine-3,5-dicarboxylate 10**: mp. 146-148 $^{o}$C. IR ($CH_2Cl_2$) ν 2953, 1720 (C=O), 1614, 1386, 1226, 1175 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.51 (3H, s, $CH_3$), 3.65 (6H, s, $2OCH_3$), 4.77 (1H, s, CH), 6.87-6.90 (2H, m, ArH), 7.07-7.09 (3H, m, ArH), 7.43 (2H, d, *J* = 8.1 Hz, ArH), 7.79 (2H, s, 2=CH), 7.81 (2H, d, *J* = 8.1 Hz, ArH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.7, 37.7, 51.8, 115.2, 126.9, 127.2, 128.0, 128.2, 129.6, 130.6, 134.1, 143.1, 145.8, 165.6. MS (EI) *m/z* 427 ($M^+$, 14.85), 350 ($M^+$-77, 80.39), 277 ($M^+$-150, 52.60), 221 ($M^+$-206, 74.19), 155 ($M^+$-272, 54.80), 91 ($M^+$-336, 66.26), 84 ($M^+$-343, 100). Anal. Calcd. for $C_{22}H_{21}NO_6S$ requires C, 61.82; H, 4.95; N, 3.28%. Found: C, 61.61; H, 5.04; N, 3.07%.

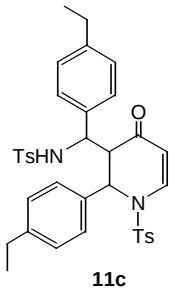



**11a**

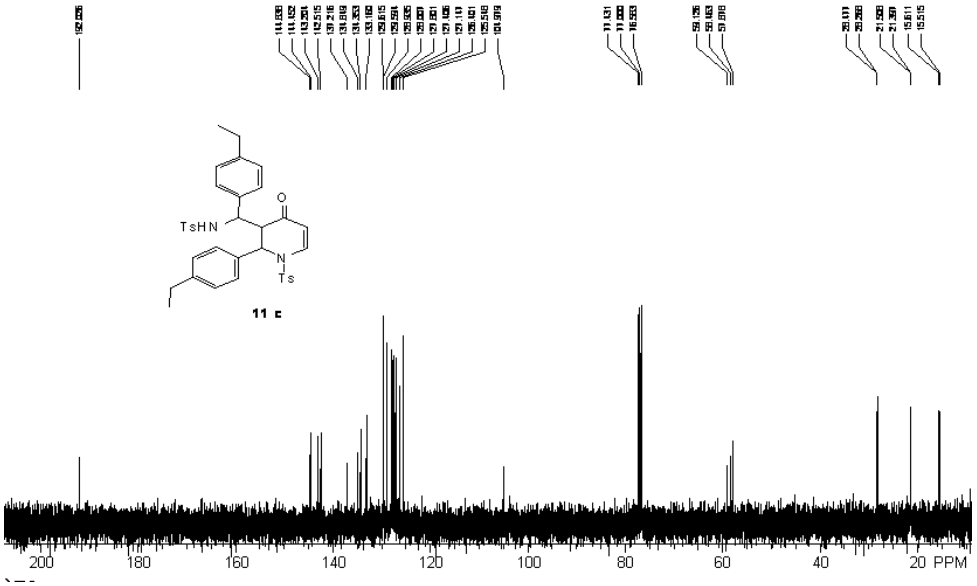
**4-Methyl-*N*-[(4-oxo-2-phenyl-1-tosyl-1,2,3,4-tetrahydropyridin-3-yl)(phenyl)methyl]benzenesulfonamide 11a**: mp. 156-157 $^{o}$C. IR ($CH_2Cl_2$) ν 1658 (C=O), 1597, 1333, 1164, 1057, 703 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.35 (3H, s, $CH_3$), 2.38 (3H, s, $CH_3$), 2.65 (1H, d, *J* = 11.1, Hz, CH), 4.32 (1H, dd, *J* = 11.1, 5.1 Hz, CH), 4.89 (1H, s, CH), 5.14 (1H, d, *J* = 8.1 Hz, =CH), 5.47 (1H, d, *J* = 5.1 Hz, NH), 6.53 (2H, d, *J* = 7.5 Hz, ArH), 6.90-6.95 (2H, m, Ar), 7.02-7.13 (8H, m, ArH), 7.26–7.34 (4H, m, ArH), 7.41 (2H, d, *J* = 7.5 Hz, ArH), 7.85 (1H, d, *J* = 8.1 Hz, =CH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.4, 21.5, 58.1, 58.4, 59.1, 105.0, 125.5, 126.4, 127.2, 127.5, 127.9, 128.6, 128.8, 129.1, 129.7, 129.7, 134.7, 135.9, 137.1, 137.4, 142.8, 143.2, 145.1, 191.7. MS (MALDI) *m/z* 609 ($M^{+}$+Na, 100). HRMS (MALDI) calcd. for $C_{32}H_{30}N_2O_5S_2Na^{+1}$: 609.1488, Found: 609.1493.

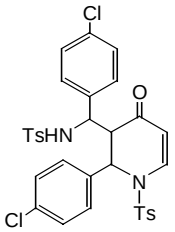



**11c**

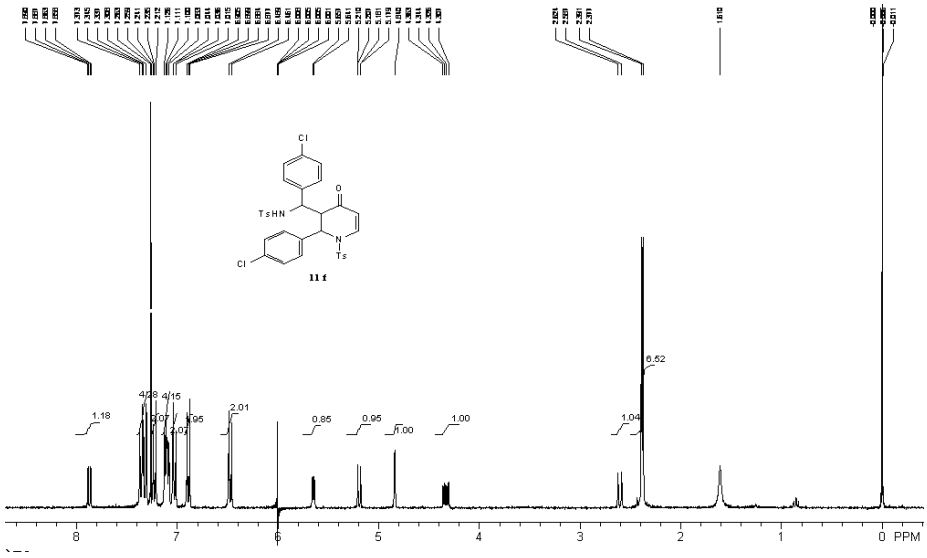
***N*-((4-Ethylphenyl)(2-(4-ethylphenyl)-4-oxo-1-tosyl-1,2,3,4-tetrahydropyridin-3-yl)methyl)-4-methylbenzenesulfonamide 11c**: mp. 194-197 $^{o}$C. IR ($CH_2Cl_2$) ν 1664 (C=O), 1597, 1334, 1166, 1055 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.08 (3H, t, *J* = 7.5 Hz, $CH_3$), 1.23 (3H, t, *J* = 7.5 Hz, $CH_3$), 2.33 (3H, s, $CH_3$), 2.35 (3H, s, $CH_3$), 2.44 (2H, q, *J* = 7.5 Hz, $CH_2$), 2.62 (2H, t, *J* = 7.5 Hz, $CH_2$), 2.70 (1H, d, *J* = 11.4, Hz, CH), 4.32 (1H, dd, *J* = 11.4, 6.0 Hz, CH), 4.88 (1H, s, CH), 5.21 (1H, d, *J* = 8.4 Hz, =CH), 5.70 (1H, d, *J* = 6.0 Hz, NH), 6.48 (2H, d, *J* = 8.1 Hz, ArH), 6.75 (2H, d, *J* = 8.1 Hz, ArH), 6.91 (2H, d, *J* = 8.1 Hz, ArH), 7.01-7.08 (6H, m, ArH), 7.31–7.36 (4H, m, ArH), 7.89 (1H, d, *J* = 8.4 Hz, =CH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 15.5, 15.6, 21.4, 21.5, 57.9, 58.5, 59.1, 105.0, 125.5, 126.4, 127.2, 127.4, 127.8, 128.0, 128.9, 129.6, 129.6, 133.2, 134.4, 134.9, 137.2, 142.5, 143.2, 144.5, 144.8, 192.0. MS (MALDI) *m/z* 665 ($M^{+}$+Na, 100). HRMS (MALDI) calcd. for $C_{36}H_{39}N_2O_5S_2^{+1}$: 643.2295, Found: 643.2296.

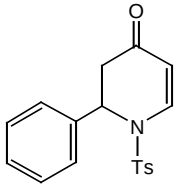



**11f**

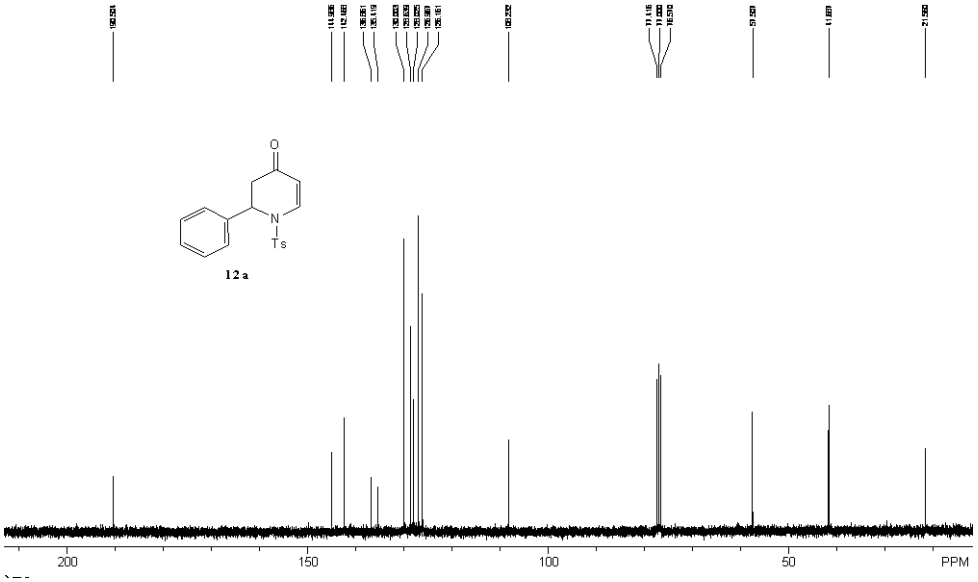
***N*-[(4-Chlorophenyl)(2-(4-chlorophenyl)-4-oxo-1-tosyl-1,2,3,4-tetrahydropyridin-3-yl)methyl]-4-methylbenzenesulfonamide 11f**: mp. 223-226 $^{o}$C. IR ($CH_2Cl_2$) ν 1655 (C=O), 1596, 1492, 1168, 1056 $cm^{-1}$. $^{1}$H NMR ($CDCl_3$, 300 MHz, TMS) δ 2.38 (3H, s, $CH_3$), 2.39 (3H, s, $CH_3$), 2.61 (1H, d, *J* = 11.1, Hz, CH), 4.34 (1H, dd, *J* = 11.1, 5.4 Hz, CH), 4.84 (1H, s, CH), 5.19 (1H, d, *J* = 8.4 Hz, =CH), 5.65 (1H, d, *J* = 5.4 Hz, NH), 6.48 (2H, d, *J* = 8.1 Hz, ArH), 6.89 (2H, m, ArH), 7.03 (2H, d, *J* = 8.1 Hz, ArH), 7.08-7.13 (4H, m, ArH), 7.21-7.24 (2H, m, ArH), 7.31–7.37 (4H, m, ArH), 7.88 (1H, d, *J* = 8.4 Hz, =CH). $^{13}$C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.5, 21.6, 57.4, 58.0, 58.4, 104.9, 114.0, 120.5, 127.0, 127.2, 127.4, 128.8, 129.1, 129.2, 129.4, 129.8, 134.1, 134.4, 134.6, 135.8, 136.9, 143.3, 145.7, 191.0. MS (MALDI) *m/z* 678 ($M^{+}$+1+Na, 100). HRMS (MALDI) calcd. for $C_{32}H_{28}N_2O_5S_2Cl_2Na^{+1}$: 677.0709, Found: 677.0709.

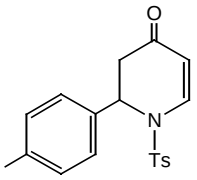



**12a**

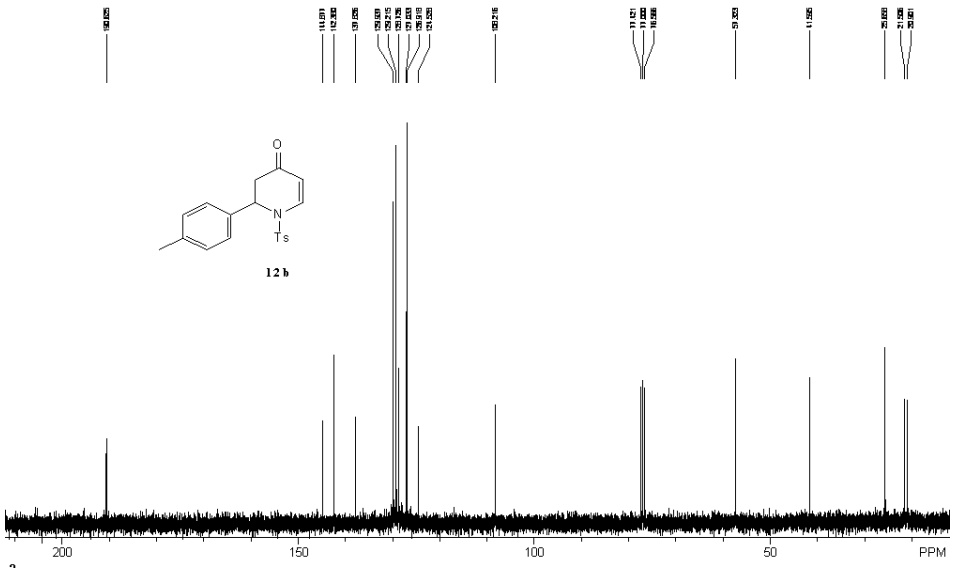
**2-Phenyl-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12a**: a colorless oil. IR ($CH_2Cl_2$) ν 1691 (C=O), 1676, 1596, 1364, 1226, 1169, 1052 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.40 (3H, s, $CH_3$), 2.63-2.70 (1H, m, $CH_2$), 2.84 (1H, dd, *J* = 16.5, 6.9 Hz, $CH_2$), 5.41 (1H, dd, *J* = 8.4, 1.6 Hz, =CH), 5.52 (1H, d, *J* = 6.9 Hz, CH), 7.15-7.26 (7H, m, ArH), 7.59 (2H, d, *J* = 8.4 Hz, ArH), 7.81 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 41.7, 57.5, 108.2, 126.2, 127.0, 128.0, 128.7, 130.0, 135.4, 136.9, 142.5, 145.0, 190.5. MS (EI) *m/z* 327 ($M^+$, 6.04), 204 ($M^+$-123, 20.93), 189 ($M^+$-138, 100), 121 ($M^+$-206, 29.89), 84 ($M^+$-243, 94.35), 43 ($M^+$-284, 99.38). HRMS (MALDI) calcd. for $C_{18}H_{18}NO_3S^{+1}$: 328.1002, Found: 328.0988.

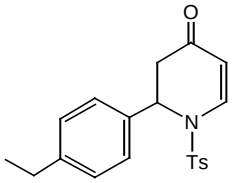



**12b**

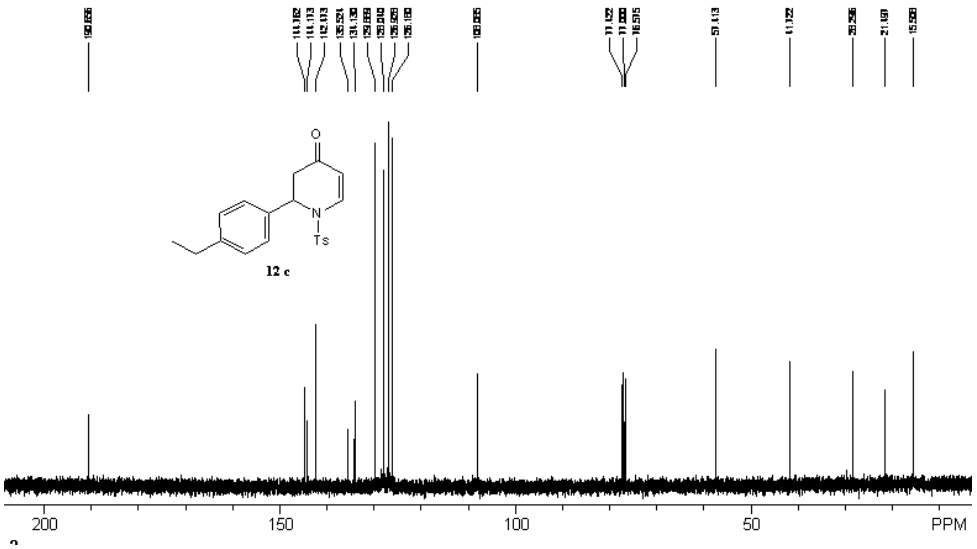
**2-*p*-Tolyl-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12b**: a colorless oil. IR ($CH_2Cl_2$) ν 1673 (C=O), 1597, 1513, 1386, 1309, 1172, 1053 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.27 (3H, s, $CH_3$), 2.40 (3H, s, $CH_3$), 2.62-2.68 (1H, m, $CH_2$), 2.80 (1H, dd, *J* = 16.5, 6.9 Hz, $CH_2$), 5.39 (1H, dd, *J* = 8.4, 1.6 Hz, =CH), 5.47 (1H, d, *J* = 6.9 Hz, CH), 6.97-7.07 (4H, m, ArH), 7.23 (2H, d, *J* = 8.4 Hz, ArH), 7.60 (2H, d, *J* = 8.4 Hz, ArH), 7.78 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 20.9, 21.5, 41.6, 57.3, 108.2, 124.5, 126.9, 127.0, 128.7, 129.2, 130.0, 137.8, 142.4, 144.9, 190.6. MS (EI) *m/z* 341 ($M^+$, 7.38), 250 ($M^+$-91, 8.44), 186 ($M^+$-155, 31.88), 158 ($M^+$-183, 25.07), 118 ($M^+$-223, 100), 91 ($M^+$-250, 89.33). HRMS (MALDI) calcd. for $C_{19}H_{20}NO_3S^{+1}$: 342.1158, Found: 342.1156.

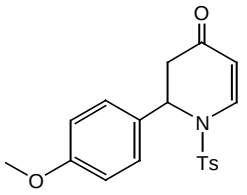



**12c**

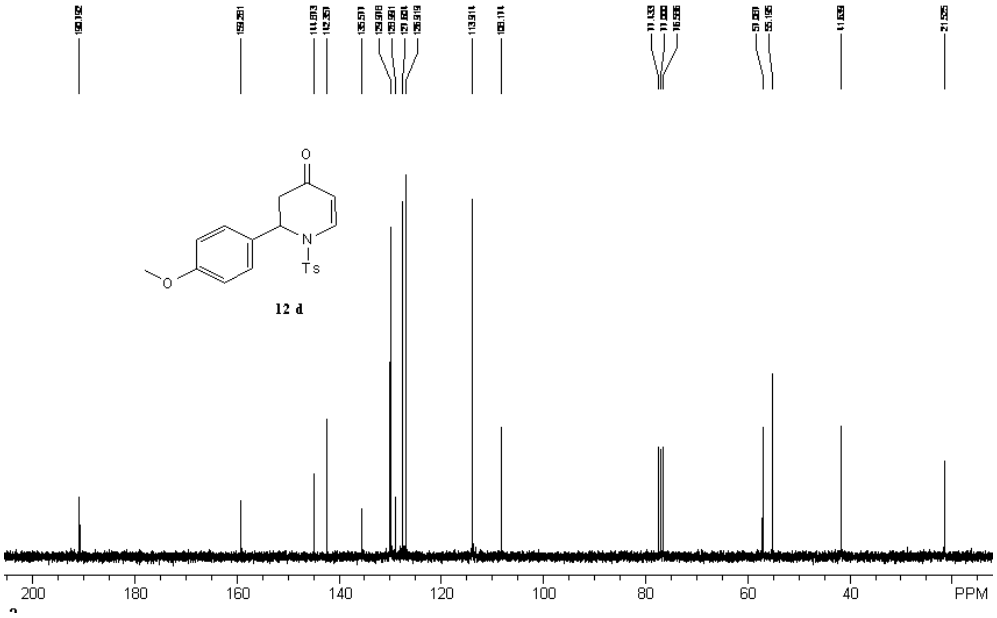
**2-(4-Ethylphenyl)-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12c**: a colorless oil. IR ($CH_2Cl_2$) ν 1670 (C=O), 1596, 1513, 1364, 1168, 1172, 1052 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 1.18 (3H, t, *J* = 7.5 Hz, $CH_3$), 2.40 (3H, s, $CH_3$), 2.56 (2H, q, *J* = 7.5 Hz, $CH_2$), 2.63-2.68 (1H, m, $CH_2$), 2.84 (1H, dd, *J* = 16.5, 7.2 Hz, $CH_2$), 5.41 (1H, d, *J* = 8.4 Hz, =CH), 5.49 (1H, d, *J* = 7.2 Hz, CH), 6.99-7.08 (4H, m, ArH), 7.21 (2H, d, *J* = 7.8 Hz, ArH), 7.58 (2H, d, *J* = 7.8 Hz, ArH),7.80 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 15.5, 21.5, 28.3, 41.7, 57.4, 108.1, 126.2, 126.9, 128.1, 129.9, 134.1, 135.5, 142.5, 144.2, 144.8, 190.7. MS (EI) *m/z* 355 ($M^+$, 80.52), 250 ($M^+$-105, 41.11), 200 ($M^+$-155, 58.70), 132 ($M^+$-223, 100), 117 ($M^+$-238, 99.55), 91 ($M^+$-264, 97.91). HRMS (MALDI) calcd. for $C_{20}H_{22}NO_3S^{+1}$: 356.1315, Found: 356.1314.

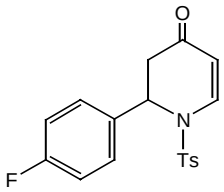



**12d**

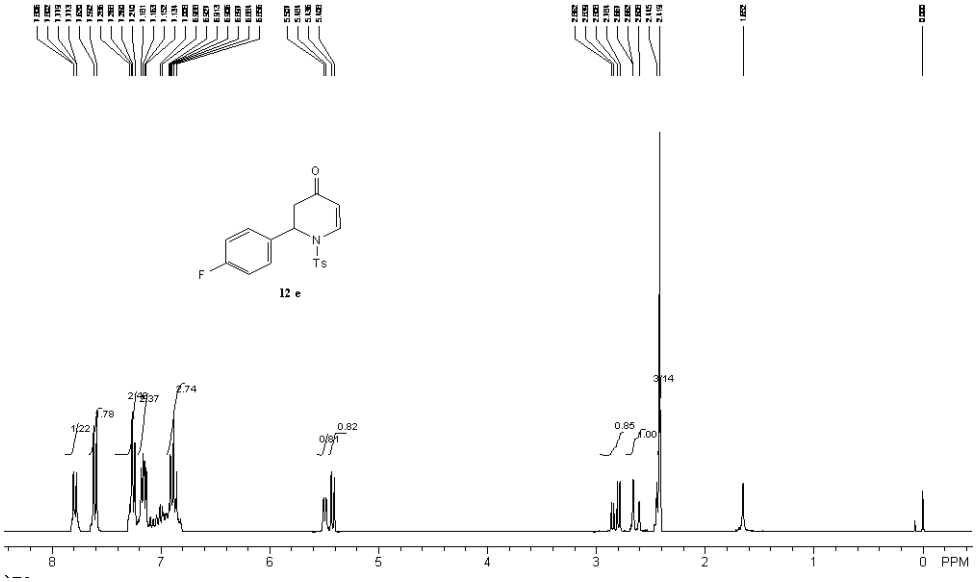
**2-(4-Methoxyphenyl)-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12d**: a colorless oil. IR ($CH_2Cl_2$) ν 1665 (C=O), 1596, 1513, 1364, 1252, 1168, 1052 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.40 (3H, s, $CH_3$), 2.61-2.66 (1H, m, $CH_2$), 2.79 (1H, dd, *J* = 16.5, 6.9 Hz, $CH_2$), 3.74 (3H, s, OMe), 5.40 (1H, d, *J* = 8.4 Hz, =CH), 5.46 (1H, d, *J* = 6.9 Hz, CH), 6.71 (2H, d, *J* = 8.7 Hz, ArH), 7.10 (2H, d, *J* = 8.7 Hz, ArH), 7.23 (2H, d, *J* = 8.4 Hz, ArH), 7.60 (2H, d, *J* = 8.4 Hz, ArH), 7.76 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.5, 41.6, 55.2, 57.1, 108.2, 113.9, 126.9, 127.6, 129.0, 130.0, 135.6, 142.4, 144.9, 159.3, 190.8. MS (EI) *m/z* 357 ($M^+$, 20.02), 250 ($M^+$-107, 12.48), 202 ($M^+$-155, 26.99), 134 ($M^+$-223, 100), 91 ($M^+$-266, 78.32); HRMS (MALDI) calcd. for $C_{19}H_{20}NO_4S^{+1}$: 358.1108, Found: 358.1105.

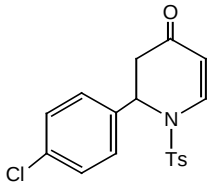



**12e**

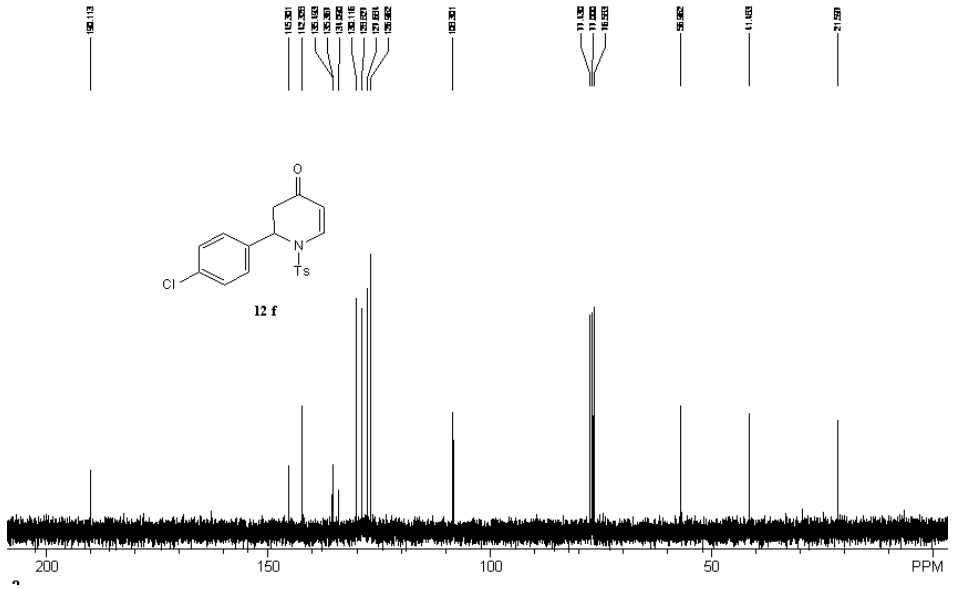
**2-(4-Fluorophenyl)-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12e**: a colorless oil. IR ($CH_2Cl_2$) ν 1671 (C=O), 1596, 1510, 1365, 1168, 1052 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.42 (3H, s, $CH_3$), 2.61-2.67 (1H, m, $CH_2$), 2.82 (1H, dd, *J* = 16.5, 6.9 Hz, $CH_2$), 5.42 (1H, d, *J* = 8.4 Hz, =CH), 5.49 (1H, d, *J* = 6.9 Hz, CH), 6.86-6.93 (2H, m, ArH), 7.13-7.18 (2H, m, ArH), 7.24-7.29 (2H, m, ArH), 7.60 (2H, d, *J* = 8.1 Hz, ArH), 7.79 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 41.6, 56.9, 108.3, 115.6 (d, *J* = 21.2 Hz), 127.0, 128.1 (d, *J* = 8.0 Hz), 130.1, 132.9 (d, *J* = 3.5 Hz), 135.5, 142.4, 145.2, 162.4 (d, *J* = 246.2 Hz), 190.4. MS (EI) *m/z* 345 ($M^+$, 38.96), 250 ($M^+$-95, 25.13), 190 ($M^+$-155, 49.63), 162 ($M^+$-183, 23.80), 155 ($M^+$-190, 30.67), 122 ($M^+$-223, 100), 91 ($M^+$-254, 62.35). HRMS (MALDI) calcd. for $C_{18}H_{17}NO_3SF^{+1}$: 346.0908, Found: 346.0910.

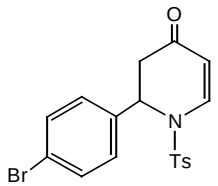



**12f**

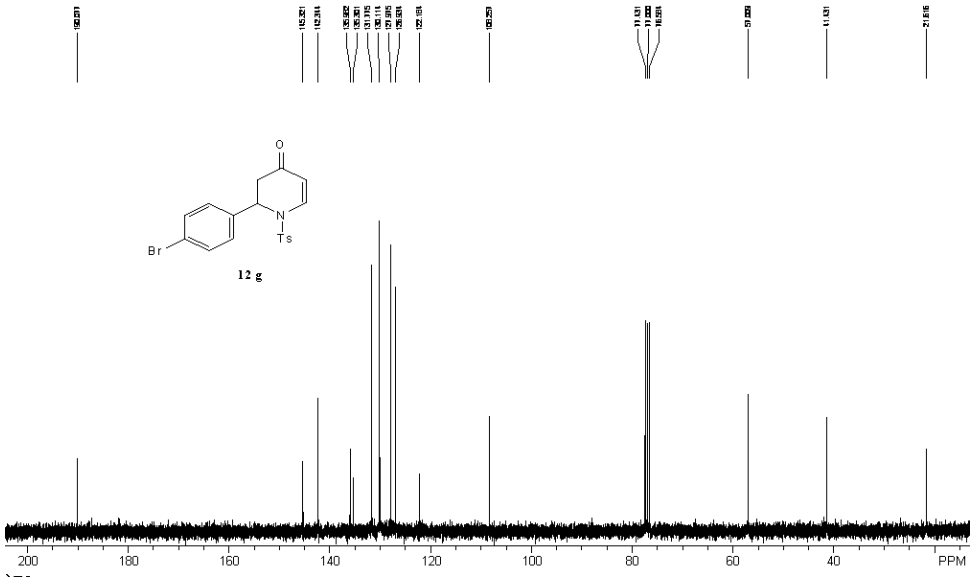
**2-(4-Chlorophenyl)-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12f**: a colorless oil. IR ($CH_2Cl_2$) ν 1677 (C=O), 1596, 1366, 1169, 1089, 1052 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.42 (3H, s, $CH_3$), 2.60-2.65 (1H, m, $CH_2$), 2.82 (1H, dd, *J* = 16.5, 6.9 Hz, $CH_2$), 5.41 (1H, d, *J* = 8.4 Hz, =CH), 5.47 (1H, d, *J* = 6.9 Hz, CH), 7.08-7.18 (4H, m, ArH), 7.25 (2H, d, *J* = 8.4 Hz, ArH), 7.60 (2H, d, *J* = 8.4 Hz, ArH), 7.80 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 41.5, 57.0, 108.3, 127.0, 127.7, 128.8, 130.1, 134.1, 135.4, 135.5, 142.3, 145.3, 190.1. MS (EI) *m/z* 363 ($M^+$+2, 12.31), 361 ($M^+$, 31.46), 250 ($M^+$-111, 26.90), 206 ($M^+$-155, 55.24), 178 ($M^+$-183, 31.73), 155 ($M^+$-206, 40.56), 138 ($M^+$-223, 100), 91 ($M^+$-270, 90.69). HRMS (MALDI) calcd. for $C_{18}H_{17}NO_3SCl^{+1}$: 362.0612, Found: 362.0615.

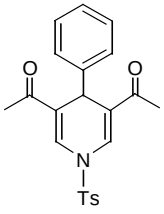



**12g**

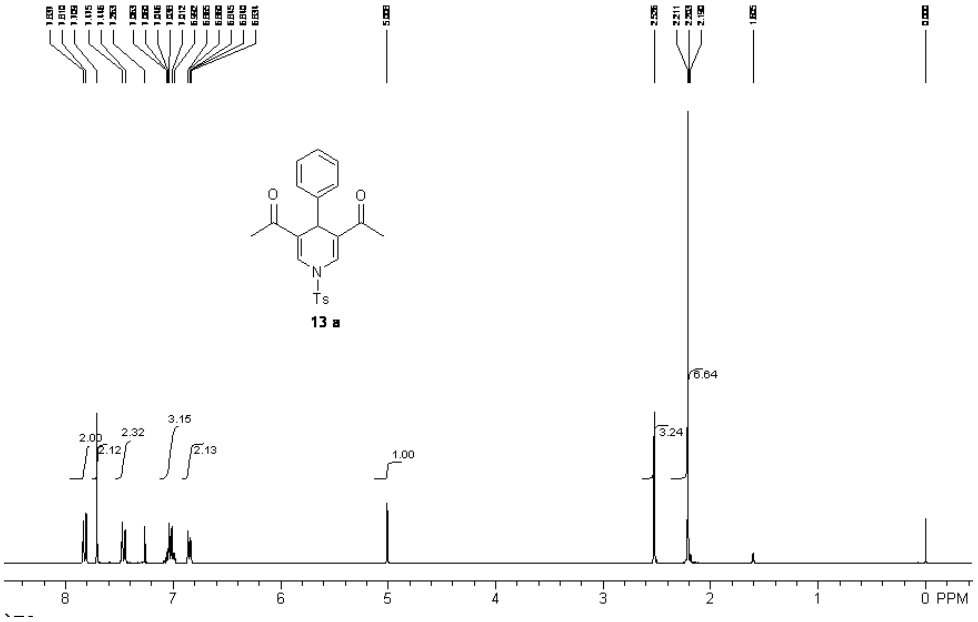
**2-(4-Bromophenyl)-1-tosyl-2,3-dihydropyridin-4(1*H*)-one 12g**: a colorless oil. IR ($CH_2Cl_2$) ν 1676 (C=O), 1596, 1366, 1169, 1089, 1052 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.43 (3H, s, $CH_3$), 2.59-2.65 (1H, m, $CH_2$), 2.83 (1H, dd, *J* = 16.5, 6.9 Hz, $CH_2$), 5.41 (1H, d, *J* = 8.4 Hz, =CH), 5.45 (1H, d, *J* = 6.9 Hz, CH), 7.03 (2H, d, *J* = 8.1 Hz, ArH), 7.24-7.33 (4H, m, ArH), 7.59 (2H, d, *J* = 8.1 Hz, ArH), 7.80 (1H, dd, *J* = 8.4, 1.6 Hz, =CH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 41.4, 57.0, 108.3, 122.2, 126.9, 128.0, 130.1, 131.8, 135.3, 136.0, 142.4, 145.3, 190.1. MS (EI) *m/z* 407 ($M^+$+2, 8.85), 405 ($M^+$, 8.28), 252 ($M^+$-153, 10.36), 250 ($M^+$-155, 17.22), 189 ($M^+$-216, 15.63), 155 ($M^+$-250, 13.74), 91 ($M^+$-314, 70.57), 86 ($M^+$-319, 67.32), 84 ($M^+$-321, 100). HRMS (MALDI) calcd. for $C_{18}H_{17}NO_3SBr^{+1}$: 406.0107, Found: 406.0109.




**13a**

**Compound 13a**: a colorless oil. IR ($CH_2Cl_2$) ν 2925, 1672 (C=O), 1597, 1387, 1220, 1174 $cm^{-1}$. $^1H$ NMR ($CDCl_3$, 300 MHz, TMS) δ 2.22 (6H, s, $2CH_3$), 2.53 (3H, s, $CH_3$), 5.01 (1H, s, CH), 6.83-6.86 (2H, m, ArH), 7.01-7.04 (3H, m, ArH), 7.46 (2H, d, *J* = 8.4 Hz, ArH), 7.71 (2H, s, =CH), 7.83 (2H, d, *J* = 8.4 Hz, ArH). $^{13}C$ NMR ($CDCl_3$, 75 MHz, TMS) δ 21.8, 25.7, 35.8, 124.5, 126.8, 127.1, 128.1, 128.2, 129.9, 130.7, 134.0, 142.9, 146.2, 194.7. MS (EI) *m/z* 395 ($M^+$, 7.55), 318 ($M^+$-77, 58.31), 240 ($M^+$-155, 100), 222 ($M^+$-173, 36.62), 155 ($M^+$-240, 68.50), 91 ($M^+$-304, 58.27). HRMS (MALDI) calcd. for $C_{22}H_{22}NO_4S^{+1}$: 396.1264, Found: 396.1270.


O
O
N
Ts
13 a
2.00
2.12
2.32
3.15
2.13
1.00
3.24
6.64
8
7
6
5
4
3
2
1
0 PPM

3) The determination of the configuration of compound **11a** by $^{1}$H NMR spectroscopy.

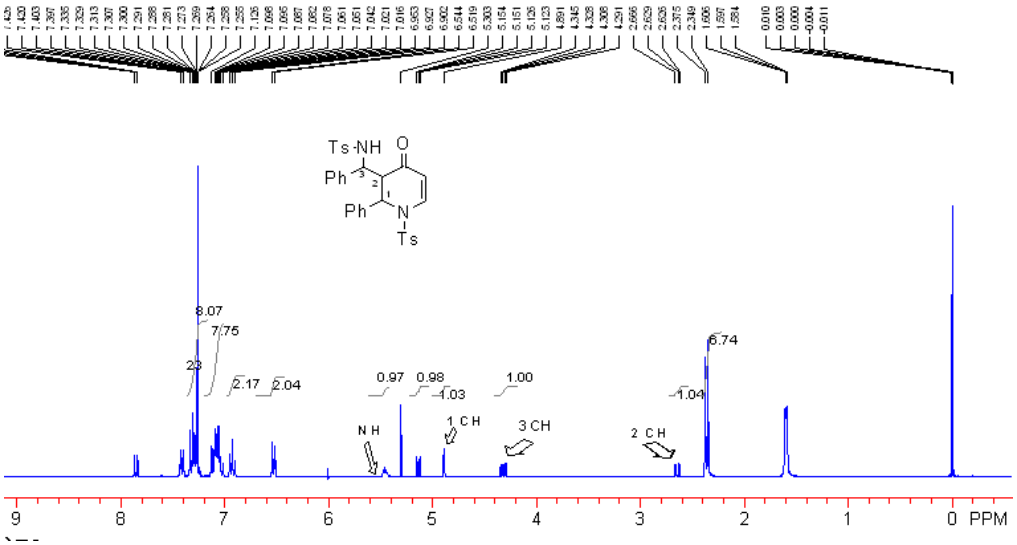



Figure 1. The $^{1}$H NMR spectroscopy of the product **11a** in $CDCl_3$.

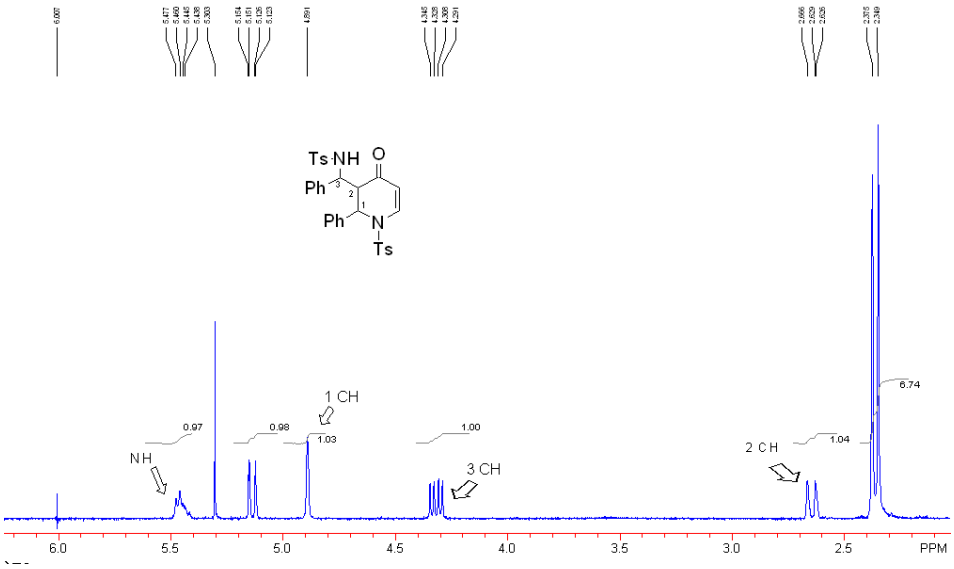



Figure 2. A part of the enlarged $^{1}$H NMR spectroscopy of the product **11a** in $CDCl_3$.

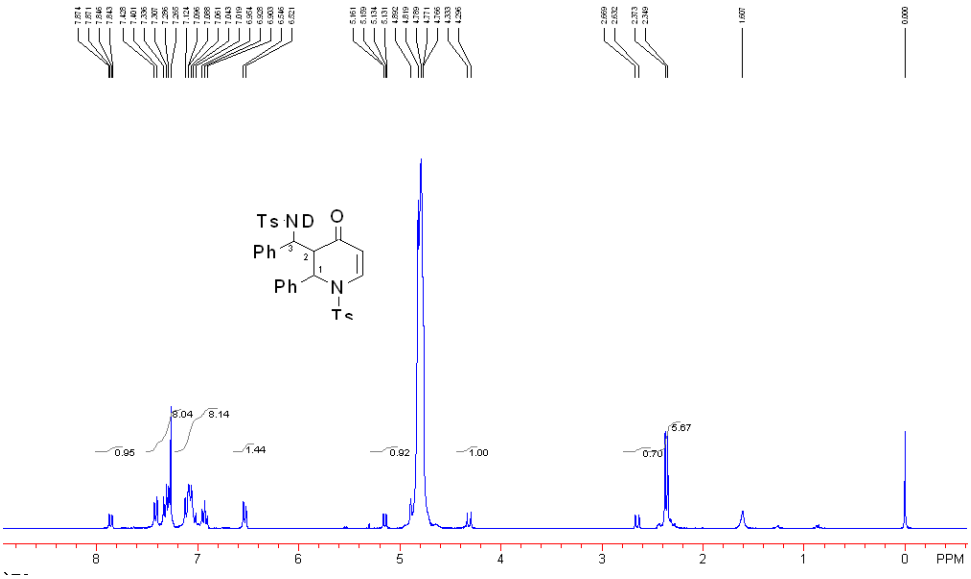
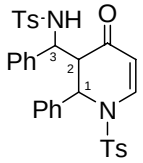
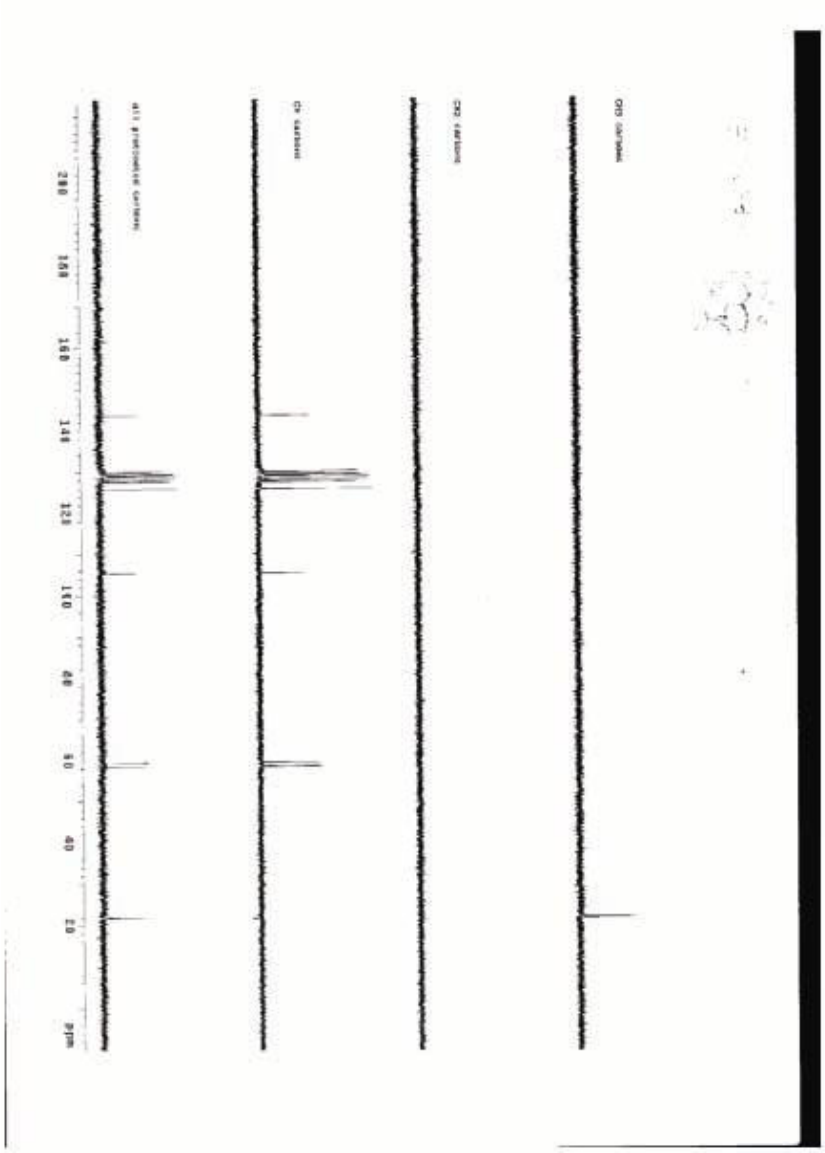


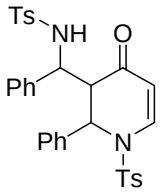

Figure 3. The $^{1}$H NMR spectroscopy of the product **11a** + $D_2O$ in $CDCl_3$.




From Figures 1-3, we can find out $J_{C1\text{-}C2}$ = 0 Hz, $J_{C2\text{-}C3}$ = 11.4 Hz. On the basis of the well-known Karplus's rule, the dihedral angle of H-$C_1$-$C_2$-H is about 90$^o$ and H-$C_2$-$C_3$-H is about 160$^o$. Thus, both configuration of H-$C_1$-$C_2$-H and H-$C_2$-$C_3$-H should be *anti*.

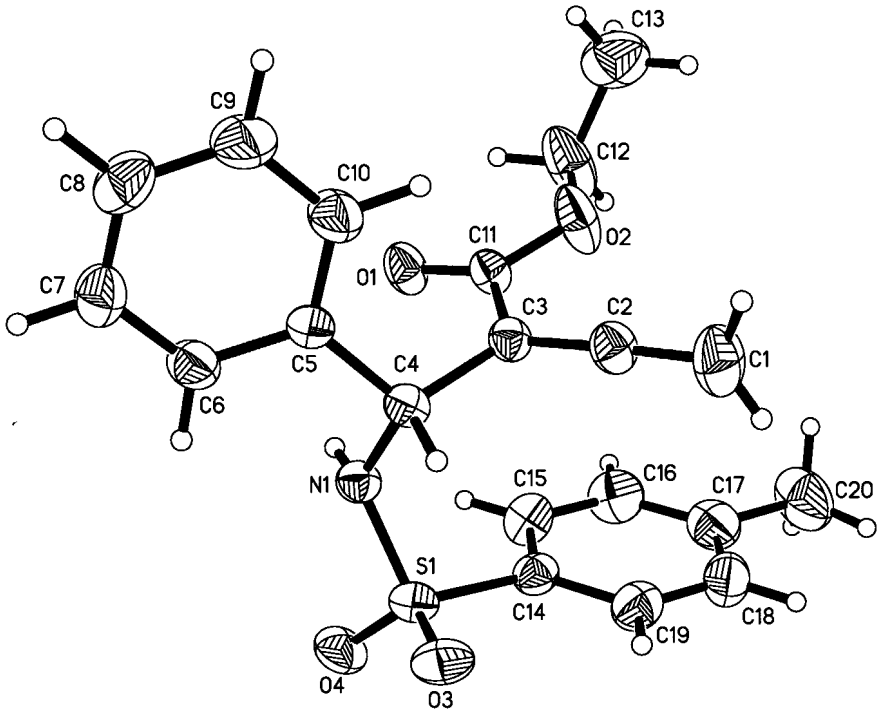
The DEPT spectroscopy of the product **11a**:






**11a**

4) X-ray crystal structures and data for the compounds **2a**, **3a**, **4j**, **5d**, **8**, **9**, and **10**.




The crystal data of **2a** has been deposited in CCDC with number 266291. Empirical Formula: $C_{20}H_{21}NO_4S$; Formula Weight: 371.44; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.492 x 0.403 x 0.357 mm; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 11.0446(10)Å, b = 12.4582(12)Å, c = 14.1435(13)Å, $\alpha = 90^o$, $\beta = 91.214(2)^o$, $\gamma = 90^o$, V = 1945.6(3)Å$^3$; Space group: P2(1)/n; Z = 4; $D_{calc}$ = 1.268 g/cm$^3$; $F_{000}$ = 784; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0442, 0.1055.

Table 1. Crystal data and structure refinement for cd2591.

| | |
|---|---|
| Identification code | cd2591 |
| Empirical formula | C20 H21 N O4 S |
| Formula weight | 371.44 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/n |
| Unit cell dimensions | a = 11.0446(10) A alpha = 90 deg.<br>b = 12.4582(12) A beta = 91.214(2) deg.<br>c = 14.1435(13) A gamma = 90 deg. |
| Volume | 1945.6(3) A^3 |
| Z, Calculated density | 4, 1.268 Mg/m^3 |
| Absorption coefficient | 0.190 mm^-1 |
| F(000) | 784 |
| Crystal size | 0.492 x 0.403 x 0.357 mm |
| Theta range for data collection | 2.18 to 27.00 deg. |
| Limiting indices | -14<=h<=14, -9<=k<=15, -16<=l<=18 |
| Reflections collected / unique | 11196 / 4225 [R(int) = 0.0364] |
| Completeness to theta = 27.00 | 99.6 % |
| Absorption correction | Empirical |
| Max. and min. transmission | 1.00000 and 0.74331 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 4225 / 0 / 273 |
| Goodness-of-fit on F^2 | 0.913 |
| Final R indices [I>2sigma(I)] | R1 = 0.0442, wR2 = 0.1055 |
| R indices (all data) | R1 = 0.0697, wR2 = 0.1142 |
| Largest diff. peak and hole | 0.217 and -0.269 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd2591.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 3254(1) | 7300(1) | 10921(1) | 53(1) |
| N(1) | 3102(2) | 6091(1) | 10527(1) | 47(1) |
| O(1) | 4468(1) | 4979(1) | 9031(1) | 62(1) |
| O(2) | 4257(1) | 5815(1) | 7651(1) | 89(1) |
| O(3) | 2078(1) | 7759(1) | 10980(1) | 69(1) |
| O(4) | 4004(1) | 7217(1) | 11750(1) | 71(1) |
| C(1) | 1731(3) | 7504(3) | 7676(2) | 88(1) |
| C(2) | 2254(2) | 6799(2) | 8207(1) | 57(1) |
| C(3) | 2780(2) | 6108(1) | 8775(1) | 46(1) |
| C(4) | 2263(2) | 5843(2) | 9736(1) | 45(1) |
| C(5) | 1816(2) | 4695(1) | 9816(1) | 44(1) |
| C(6) | 1712(2) | 4204(2) | 10683(2) | 62(1) |
| C(7) | 1207(2) | 3197(2) | 10767(2) | 75(1) |
| C(8) | 809(2) | 2663(2) | 9987(2) | 80(1) |
| C(9) | 922(2) | 3124(2) | 9123(2) | 90(1) |
| C(10) | 1414(2) | 4138(2) | 9038(2) | 71(1) |
| C(11) | 3921(2) | 5569(2) | 8513(1) | 49(1) |
| C(12) | 5412(2) | 5400(3) | 7329(2) | 106(1) |
| C(13) | 5268(3) | 4972(2) | 6389(2) | 111(1) |
| C(14) | 4051(2) | 8035(2) | 10076(1) | 52(1) |
| C(15) | 5215(2) | 7729(2) | 9858(2) | 68(1) |
| C(16) | 5819(2) | 8277(2) | 9169(2) | 81(1) |
| C(17) | 5297(3) | 9130(2) | 8693(2) | 80(1) |
| C(18) | 4152(3) | 9428(2) | 8924(2) | 79(1) |
| C(19) | 3517(2) | 8886(2) | 9613(2) | 67(1) |
| C(20) | 5982(3) | 9722(2) | 7933(2) | 117(1) |

Table 3. Bond lengths [A] and angles [deg] for cd2591.

| | |
|---|---|
| S(1)-O(3) | 1.4228(14) |
| S(1)-O(4) | 1.4239(14) |
| S(1)-N(1) | 1.6135(16) |
| S(1)-C(14) | 1.758(2) |
| N(1)-C(4) | 1.470(2) |
| N(1)-H(1) | 0.833(18) |
| O(1)-C(11) | 1.193(2) |
| O(2)-C(11) | 1.318(2) |
| O(2)-C(12) | 1.459(2) |
| C(1)-C(2) | 1.286(3) |
| C(1)-H(1A) | 0.95(2) |
| C(1)-H(1B) | 0.90(2) |
| C(2)-C(3) | 1.305(2) |
| C(3)-C(11) | 1.482(3) |
| C(3)-C(4) | 1.522(2) |
| C(4)-C(5) | 1.518(2) |
| C(4)-H(4) | 0.973(16) |
| C(5)-C(10) | 1.367(3) |
| C(5)-C(6) | 1.378(3) |
| C(6)-C(7) | 1.379(3) |
| C(6)-H(6) | 0.94(2) |
| C(7)-C(8) | 1.354(3) |
| C(7)-H(7) | 0.96(3) |
| C(8)-C(9) | 1.359(3) |
| C(8)-H(8) | 0.96(3) |
| C(9)-C(10) | 1.381(3) |
| C(9)-H(9) | 0.9300 |
| C(10)-H(10) | 0.99(2) |
| C(12)-C(13) | 1.438(3) |
| C(12)-H(12A) | 0.9700 |
| C(12)-H(12B) | 0.9700 |
| C(13)-H(13A) | 0.9600 |
| C(13)-H(13B) | 0.9600 |
| C(13)-H(13C) | 0.9600 |
| C(14)-C(19) | 1.373(3) |
| C(14)-C(15) | 1.382(3) |
| C(15)-C(16) | 1.373(3) |
| C(15)-H(15) | 0.9300 |
| C(16)-C(17) | 1.378(3) |
| C(16)-H(16) | 0.9300 |
| C(17)-C(18) | 1.365(3) |
| C(17)-C(20) | 1.518(3) |
| C(18)-C(19) | 1.387(3) |
| C(18)-H(18) | 0.9300 |
| C(19)-H(19) | 0.92(2) |
| C(20)-H(20A) | 0.9600 |
| C(20)-H(20B) | 0.9600 |
| C(20)-H(20C) | 0.9600 |

| | |
|---|---|
| O(3)-S(1)-O(4) | 119.79(9) |
| O(3)-S(1)-N(1) | 107.87(9) |
| O(4)-S(1)-N(1) | 105.70(9) |
| O(3)-S(1)-C(14) | 107.56(9) |
| O(4)-S(1)-C(14) | 107.84(9) |
| N(1)-S(1)-C(14) | 107.54(8) |
| C(4)-N(1)-S(1) | 121.21(13) |
| C(4)-N(1)-H(1) | 113.3(12) |
| S(1)-N(1)-H(1) | 115.3(13) |
| C(11)-O(2)-C(12) | 118.12(16) |
| C(2)-C(1)-H(1A) | 112.8(18) |
| C(2)-C(1)-H(1B) | 117.7(17) |
| H(1A)-C(1)-H(1B) | 129(2) |
| C(1)-C(2)-C(3) | 177.7(3) |
| C(2)-C(3)-C(11) | 120.97(17) |
| C(2)-C(3)-C(4) | 121.50(17) |
| C(11)-C(3)-C(4) | 117.52(15) |
| N(1)-C(4)-C(5) | 110.03(14) |
| N(1)-C(4)-C(3) | 113.06(15) |

| | |
|---|---|
| C(5)-C(4)-C(3) | 113.52(14) |
| N(1)-C(4)-H(4) | 102.2(9) |
| C(5)-C(4)-H(4) | 111.0(9) |
| C(3)-C(4)-H(4) | 106.4(9) |
| C(10)-C(5)-C(6) | 117.36(19) |
| C(10)-C(5)-C(4) | 121.29(17) |
| C(6)-C(5)-C(4) | 121.16(16) |
| C(5)-C(6)-C(7) | 121.5(2) |
| C(5)-C(6)-H(6) | 115.0(12) |
| C(7)-C(6)-H(6) | 123.6(12) |
| C(8)-C(7)-C(6) | 120.1(3) |
| C(8)-C(7)-H(7) | 120.8(15) |
| C(6)-C(7)-H(7) | 119.0(15) |
| C(7)-C(8)-C(9) | 119.4(2) |
| C(7)-C(8)-H(8) | 118.4(14) |
| C(9)-C(8)-H(8) | 121.9(14) |
| C(8)-C(9)-C(10) | 120.6(2) |
| C(8)-C(9)-H(9) | 119.7 |
| C(10)-C(9)-H(9) | 119.7 |
| C(5)-C(10)-C(9) | 121.0(2) |
| C(5)-C(10)-H(10) | 120.0(12) |
| C(9)-C(10)-H(10) | 118.9(12) |
| O(1)-C(11)-O(2) | 124.23(17) |
| O(1)-C(11)-C(3) | 123.23(16) |
| O(2)-C(11)-C(3) | 112.54(16) |
| C(13)-C(12)-O(2) | 109.8(2) |
| C(13)-C(12)-H(12A) | 109.7 |
| O(2)-C(12)-H(12A) | 109.7 |
| C(13)-C(12)-H(12B) | 109.7 |
| O(2)-C(12)-H(12B) | 109.7 |
| H(12A)-C(12)-H(12B) | 108.2 |
| C(12)-C(13)-H(13A) | 109.5 |
| C(12)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 |
| C(12)-C(13)-H(13C) | 109.5 |
| H(13A)-C(13)-H(13C) | 109.5 |
| H(13B)-C(13)-H(13C) | 109.5 |
| C(19)-C(14)-C(15) | 119.9(2) |
| C(19)-C(14)-S(1) | 120.71(17) |
| C(15)-C(14)-S(1) | 119.36(16) |
| C(16)-C(15)-C(14) | 119.3(2) |
| C(16)-C(15)-H(15) | 120.4 |
| C(14)-C(15)-H(15) | 120.4 |
| C(15)-C(16)-C(17) | 121.7(2) |
| C(15)-C(16)-H(16) | 119.1 |
| C(17)-C(16)-H(16) | 119.1 |
| C(18)-C(17)-C(16) | 118.2(2) |
| C(18)-C(17)-C(20) | 121.1(3) |
| C(16)-C(17)-C(20) | 120.7(3) |
| C(17)-C(18)-C(19) | 121.4(2) |
| C(17)-C(18)-H(18) | 119.3 |
| C(19)-C(18)-H(18) | 119.3 |
| C(14)-C(19)-C(18) | 119.5(2) |
| C(14)-C(19)-H(19) | 117.2(13) |
| C(18)-C(19)-H(19) | 123.3(13) |
| C(17)-C(20)-H(20A) | 109.5 |
| C(17)-C(20)-H(20B) | 109.5 |
| H(20A)-C(20)-H(20B) | 109.5 |
| C(17)-C(20)-H(20C) | 109.5 |
| H(20A)-C(20)-H(20C) | 109.5 |
| H(20B)-C(20)-H(20C) | 109.5 |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd2591.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

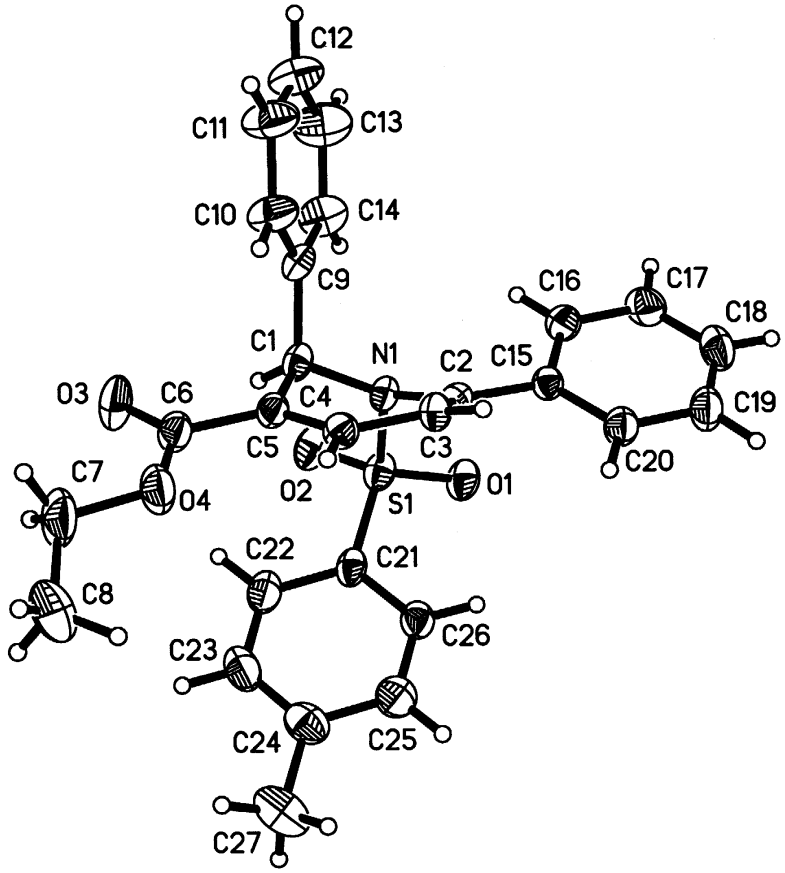
| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 54(1) | 55(1) | 49(1) | -11(1) | 1(1) | -1(1) |
| N(1) | 45(1) | 50(1) | 45(1) | -3(1) | -4(1) | 4(1) |
| O(1) | 58(1) | 79(1) | 48(1) | 13(1) | 3(1) | 24(1) |
| O(2) | 76(1) | 136(1) | 54(1) | 37(1) | 22(1) | 42(1) |
| O(3) | 58(1) | 68(1) | 80(1) | -19(1) | 13(1) | 7(1) |
| O(4) | 83(1) | 80(1) | 49(1) | -11(1) | -11(1) | -10(1) |
| C(1) | 79(2) | 100(2) | 85(2) | 27(2) | -11(2) | 22(2) |
| C(2) | 49(1) | 67(1) | 54(1) | 6(1) | -4(1) | 6(1) |
| C(3) | 44(1) | 49(1) | 44(1) | 2(1) | -5(1) | 1(1) |
| C(4) | 40(1) | 52(1) | 42(1) | -3(1) | -2(1) | 6(1) |
| C(5) | 37(1) | 49(1) | 45(1) | -5(1) | 3(1) | 1(1) |
| C(6) | 68(1) | 66(1) | 53(1) | -6(1) | 10(1) | -10(1) |
| C(7) | 79(2) | 71(2) | 75(2) | 12(1) | 11(1) | -13(1) |
| C(8) | 76(2) | 61(2) | 104(2) | 0(2) | 10(1) | -19(1) |
| C(9) | 113(2) | 82(2) | 76(2) | -18(1) | -5(2) | -38(2) |
| C(10) | 86(2) | 73(2) | 55(1) | -4(1) | -3(1) | -24(1) |
| C(11) | 49(1) | 59(1) | 40(1) | 4(1) | -1(1) | 3(1) |
| C(12) | 76(2) | 177(3) | 65(2) | 34(2) | 28(1) | 44(2) |
| C(13) | 116(2) | 100(2) | 119(2) | -21(2) | 34(2) | 10(2) |
| C(14) | 55(1) | 47(1) | 55(1) | -6(1) | -4(1) | -3(1) |
| C(15) | 56(1) | 64(1) | 84(2) | 4(1) | 1(1) | -1(1) |
| C(16) | 65(2) | 85(2) | 94(2) | -3(2) | 16(1) | -11(1) |
| C(17) | 93(2) | 75(2) | 72(2) | -7(1) | 5(1) | -29(2) |
| C(18) | 103(2) | 63(2) | 70(2) | 9(1) | -10(1) | -5(1) |
| C(19) | 68(2) | 61(1) | 73(2) | -3(1) | -2(1) | 6(1) |
| C(20) | 146(3) | 120(2) | 86(2) | 12(2) | 19(2) | -55(2) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd2591.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(9) | 665 | 2754 | 8584 | 108 |
| H(12A) | 6009 | 5971 | 7331 | 127 |
| H(12B) | 5700 | 4841 | 7756 | 127 |
| H(13A) | 4728 | 4368 | 6400 | 167 |
| H(13B) | 6042 | 4746 | 6164 | 167 |
| H(13C) | 4938 | 5515 | 5977 | 167 |
| H(15) | 5586 | 7159 | 10174 | 81 |
| H(16) | 6599 | 8066 | 9020 | 97 |
| H(18) | 3789 | 10007 | 8614 | 95 |
| H(20A) | 5507 | 10318 | 7708 | 176 |
| H(20B) | 6135 | 9242 | 7418 | 176 |
| H(20C) | 6737 | 9979 | 8193 | 176 |
| H(1) | 3736(17) | 5728(15) | 10522(12) | 54(6) |
| H(1A) | 1170(20) | 7200(20) | 7234(19) | 114(10) |
| H(1B) | 1950(20) | 8190(20) | 7753(17) | 91(9) |
| H(4) | 1608(15) | 6350(12) | 9836(10) | 37(4) |
| H(6) | 1993(18) | 4615(16) | 11205(14) | 71(6) |
| H(7) | 1190(20) | 2866(19) | 11380(18) | 100(8) |
| H(8) | 540(20) | 1940(20) | 10057(15) | 92(8) |
| H(10) | 1449(17) | 4471(16) | 8399(15) | 74(6) |
| H(19) | 2740(20) | 9056(15) | 9771(13) | 67(6) |

Table 7. Hydrogen bonds for cd2591 [A and deg.].

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




The crystal data of **3a** has been deposited in CCDC with number 211894. Empirical Formula: $C_{27}H_{25}NO_4S$; Formula Weight: 459.54; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.468 x 0.375 x 0.245 mm; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: a = 7.6675(8)Å, b = 13.8140(15)Å, c = 22.914(3)Å, $\alpha$ = 92.491(2)$^o$, $\beta$ = 93.885(2)$^o$, $\gamma$ = 98.992(2)$^o$, V = 2388.1(4)Å$^3$; Space group: P-1; Z = 4; $D_{calc}$ = 1.278 g/cm$^3$; $F_{000}$ = 968; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0605, 0.1271.

Table 1. Crystal data and structure refinement for cd23203.

| | |
|---|---|
| Identification code | cd23203 |
| Empirical formula | C27 H25 N O4 S |
| Formula weight | 459.54 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Triclinic, P-1 |
| Unit cell dimensions | a = 7.6675(8) A alpha = 92.491(2) deg.<br>b = 13.8140(15) A beta = 93.885(2) deg.<br>c = 22.914(3) A gamma = 98.992(2) deg. |
| Volume | 2388.1(4) A^3 |
| Z, Calculated density | 4, 1.278 Mg/m^3 |
| Absorption coefficient | 0.169 mm^-1 |
| F(000) | 968 |
| Crystal size | 0.468 x 0.375 x 0.245 mm |
| Theta range for data collection | 1.49 to 28.33 deg. |
| Limiting indices | -10<=h<=10, -17<=k<=17, -29<=l<=27 |
| Reflections collected / unique | 14712 / 10597 [R(int) = 0.0692] |
| Completeness to theta = 28.33 | 88.9 % |
| Absorption correction | Sadabs |
| Max. and min. transmission | 1.00000 and 0.59800 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 10597 / 3 / 660 |
| Goodness-of-fit on F^2 | 0.818 |
| Final R indices [I>2sigma(I)] | R1 = 0.0605, wR2 = 0.1271 |
| R indices (all data) | R1 = 0.1232, wR2 = 0.1463 |
| Extinction coefficient | 0.0018(4) |
| Largest diff. peak and hole | 0.553 and -0.296 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd23203.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 758(1) | 4721(1) | 1313(1) | 49(1) |
| S(2) | 3884(1) | -259(1) | 3630(1) | 49(1) |
| N(1) | 2853(3) | 5200(2) | 1535(1) | 42(1) |
| N(2) | 1731(3) | -666(2) | 3471(1) | 43(1) |
| O(1) | 83(3) | 5417(2) | 959(1) | 56(1) |
| O(2) | -58(3) | 4437(2) | 1834(1) | 63(1) |
| O(3) | 4360(4) | 2845(2) | 2436(1) | 83(1) |
| O(4) | 5636(4) | 2417(2) | 1636(1) | 73(1) |
| O(6) | 4633(3) | -175(2) | 3077(1) | 67(1) |
| O(5) | 4490(3) | -889(2) | 4047(1) | 62(1) |
| O(7) | 407(4) | 1583(2) | 2422(1) | 79(1) |
| O(8) | -780(4) | 2182(2) | 3203(1) | 77(1) |
| C(1) | 3751(5) | 4671(3) | 1989(1) | 45(1) |
| C(2) | 3967(4) | 5603(2) | 1096(1) | 40(1) |
| C(3) | 5351(4) | 5164(2) | 976(1) | 45(1) |
| C(4) | 5612(4) | 4259(2) | 1227(1) | 46(1) |
| C(5) | 4802(4) | 3976(2) | 1702(1) | 44(1) |
| C(6) | 4898(5) | 3038(3) | 1967(2) | 56(1) |
| C(7) | 5693(8) | 1464(4) | 1861(2) | 107(2) |
| C(8) | 5675(15) | 760(4) | 1393(3) | 302(7) |
| C(9) | 4798(5) | 5400(3) | 2442(1) | 47(1) |
| C(10) | 6546(6) | 5386(4) | 2605(2) | 71(1) |
| C(11) | 7459(6) | 6030(4) | 3035(2) | 90(2) |
| C(12) | 6662(7) | 6717(4) | 3302(2) | 89(2) |
| C(13) | 4934(7) | 6766(4) | 3143(2) | 95(2) |
| C(14) | 3990(5) | 6102(3) | 2714(2) | 70(1) |
| C(15) | 3613(4) | 6512(2) | 832(1) | 40(1) |
| C(16) | 3043(4) | 7254(3) | 1159(2) | 51(1) |
| C(17) | 2819(6) | 8123(3) | 917(2) | 65(1) |
| C(18) | 3172(6) | 8270(3) | 346(2) | 69(1) |
| C(19) | 3731(6) | 7545(3) | 14(2) | 65(1) |
| C(20) | 3945(5) | 6679(3) | 258(2) | 55(1) |
| C(21) | 888(4) | 3668(3) | 874(2) | 47(1) |
| C(22) | 840(5) | 2761(3) | 1113(2) | 64(1) |
| C(23) | 1076(5) | 1964(3) | 760(2) | 72(1) |
| C(24) | 1344(5) | 2044(3) | 177(2) | 67(1) |
| C(25) | 1342(5) | 2951(3) | -55(2) | 64(1) |
| C(26) | 1115(4) | 3754(3) | 285(2) | 52(1) |
| C(28) | 852(5) | -184(3) | 2982(1) | 43(1) |
| C(27) | 1622(6) | 1170(3) | -201(2) | 99(2) |
| C(29) | 633(5) | -929(2) | 3940(1) | 44(1) |
| C(30) | -672(5) | -413(2) | 4036(1) | 49(1) |
| C(31) | -856(5) | 449(3) | 3727(2) | 48(1) |
| C(32) | -73(4) | 604(2) | 3232(1) | 43(1) |
| C(33) | -103(5) | 1491(3) | 2902(2) | 54(1) |
| C(34) | -826(8) | 3093(3) | 2932(2) | 104(2) |
| C(35) | 141(9) | 3885(4) | 3289(3) | 155(3) |
| C(36) | -316(5) | -960(3) | 2586(1) | 44(1) |
| C(37) | -2080(5) | -929(3) | 2455(2) | 61(1) |
| C(38) | -3093(5) | -1622(3) | 2080(2) | 75(1) |
| C(39) | -2383(6) | -2365(3) | 1830(2) | 74(1) |
| C(40) | -638(6) | -2418(3) | 1962(2) | 82(1) |
| C(41) | 378(5) | -1725(3) | 2333(2) | 68(1) |
| C(42) | 917(5) | -1802(2) | 4262(1) | 45(1) |
| C(43) | 1312(5) | -2636(3) | 3976(2) | 58(1) |
| C(44) | 1458(6) | -3459(3) | 4286(2) | 75(1) |
| C(45) | 1202(7) | -3469(4) | 4867(2) | 84(2) |

| | | | | |
|---|---|---|---|---|
| C(46) | 837(7) | -2647(4) | 5152(2) | 85(2) |
| C(47) | 681(6) | -1820(3) | 4849(2) | 68(1) |
| C(48) | 4043(4) | 913(3) | 3964(2) | 47(1) |
| C(49) | 4221(5) | 1720(3) | 3623(2) | 64(1) |
| C(50) | 4247(6) | 2642(3) | 3893(2) | 77(1) |
| C(51) | 4112(6) | 2767(3) | 4481(2) | 75(1) |
| C(52) | 3965(6) | 1954(3) | 4817(2) | 71(1) |
| C(53) | 3918(5) | 1032(3) | 4559(2) | 59(1) |
| C(54) | 4111(7) | 3771(3) | 4775(2) | 119(2) |

Table 3. Bond lengths [A] and angles [deg] for cd23203.

| | |
|---|---|
| S(1)-O(1) | 1.420(2) |
| S(1)-O(2) | 1.427(2) |
| S(1)-N(1) | 1.675(3) |
| S(1)-C(21) | 1.751(4) |
| S(2)-O(5) | 1.424(2) |
| S(2)-O(6) | 1.429(2) |
| S(2)-N(2) | 1.669(3) |
| S(2)-C(48) | 1.742(4) |
| N(1)-C(2) | 1.438(4) |
| N(1)-C(1) | 1.488(4) |
| N(2)-C(29) | 1.434(4) |
| N(2)-C(28) | 1.502(4) |
| O(3)-C(6) | 1.203(4) |
| O(4)-C(6) | 1.335(4) |
| O(4)-C(7) | 1.439(5) |
| O(7)-C(33) | 1.199(4) |
| O(8)-C(33) | 1.342(4) |
| O(8)-C(34) | 1.433(4) |
| C(1)-C(5) | 1.504(5) |
| C(1)-C(9) | 1.509(5) |
| C(1)-H(1) | 0.90(2) |
| C(2)-C(3) | 1.340(4) |
| C(2)-C(15) | 1.473(4) |
| C(3)-C(4) | 1.435(4) |
| C(3)-H(2) | 0.9300 |
| C(4)-C(5) | 1.334(4) |
| C(4)-H(3) | 0.9300 |
| C(5)-C(6) | 1.464(5) |
| C(7)-C(8) | 1.413(7) |
| C(7)-H(5) | 0.9700 |
| C(7)-H(4) | 0.9700 |
| C(8)-H(8) | 0.9600 |
| C(8)-H(7) | 0.9600 |
| C(8)-H(6) | 0.9600 |
| C(9)-C(10) | 1.371(5) |
| C(9)-C(14) | 1.380(5) |
| C(10)-C(11) | 1.373(6) |
| C(10)-H(9) | 0.90(3) |
| C(11)-C(12) | 1.354(5) |
| C(11)-H(10) | 0.9300 |
| C(12)-C(13) | 1.363(6) |
| C(12)-H(11) | 0.9300 |
| C(13)-C(14) | 1.395(6) |
| C(13)-H(12) | 0.9300 |
| C(14)-H(13) | 0.9300 |
| C(15)-C(20) | 1.379(4) |
| C(15)-C(16) | 1.387(4) |
| C(16)-C(17) | 1.374(5) |
| C(16)-H(14) | 0.9300 |
| C(17)-C(18) | 1.371(6) |
| C(17)-H(15) | 0.94(3) |
| C(18)-C(19) | 1.370(6) |
| C(18)-H(16) | 0.92(4) |
| C(19)-C(20) | 1.370(5) |
| C(19)-H(17) | 0.96(3) |
| C(20)-H(18) | 0.98(3) |
| C(21)-C(26) | 1.381(4) |
| C(21)-C(22) | 1.386(5) |
| C(22)-C(23) | 1.379(6) |
| C(22)-H(19) | 0.97(3) |
| C(23)-C(24) | 1.371(5) |
| C(23)-H(20) | 0.9300 |
| C(24)-C(25) | 1.384(5) |
| C(24)-C(27) | 1.506(5) |

| | |
|---|---|
| C(25)-C(26) | 1.367(5) |
| C(25)-H(21) | 0.9300 |
| C(26)-H(22) | 0.9300 |
| C(28)-C(32) | 1.503(4) |
| C(28)-C(36) | 1.510(5) |
| C(28)-H(26) | 0.93(3) |
| C(27)-H(23) | 0.9600 |
| C(27)-H(24) | 0.9600 |
| C(27)-H(25) | 0.9600 |
| C(29)-C(30) | 1.340(4) |
| C(29)-C(42) | 1.478(4) |
| C(30)-C(31) | 1.433(4) |
| C(30)-H(27) | 0.9300 |
| C(31)-C(32) | 1.329(4) |
| C(31)-H(28) | 0.93(3) |
| C(32)-C(33) | 1.469(5) |
| C(34)-C(35) | 1.415(6) |
| C(34)-H(29) | 0.9700 |
| C(34)-H(30) | 0.9700 |
| C(35)-H(32) | 0.9600 |
| C(35)-H(31) | 0.9600 |
| C(35)-H(33) | 0.9600 |
| C(36)-C(37) | 1.373(5) |
| C(36)-C(41) | 1.380(5) |
| C(37)-C(38) | 1.365(5) |
| C(37)-H(34) | 0.94(3) |
| C(38)-C(39) | 1.358(5) |
| C(38)-H(35) | 0.9300 |
| C(39)-C(40) | 1.365(5) |
| C(39)-H(36) | 0.9300 |
| C(40)-C(41) | 1.360(5) |
| C(40)-H(37) | 0.9300 |
| C(41)-H(38) | 0.9300 |
| C(42)-C(47) | 1.371(5) |
| C(42)-C(43) | 1.385(4) |
| C(43)-C(44) | 1.382(5) |
| C(43)-H(39) | 0.9300 |
| C(44)-C(45) | 1.361(6) |
| C(44)-H(40) | 0.9300 |
| C(45)-C(46) | 1.362(6) |
| C(45)-H(41) | 0.95(4) |
| C(46)-C(47) | 1.378(5) |
| C(46)-H(42) | 0.94(3) |
| C(47)-H(43) | 0.99(4) |
| C(48)-C(53) | 1.379(5) |
| C(48)-C(49) | 1.384(5) |
| C(49)-C(50) | 1.388(5) |
| C(49)-H(44) | 0.9300 |
| C(50)-C(51) | 1.365(6) |
| C(50)-H(45) | 0.9300 |
| C(51)-C(52) | 1.383(6) |
| C(51)-C(54) | 1.515(6) |
| C(52)-C(53) | 1.374(5) |
| C(52)-H(46) | 0.94(4) |
| C(53)-H(47) | 0.94(3) |
| C(54)-H(49) | 0.9600 |
| C(54)-H(50) | 0.9600 |
| C(54)-H(48) | 0.9600 |

| | |
|---|---|
| O(1)-S(1)-O(2) | 120.04(15) |
| O(1)-S(1)-N(1) | 106.81(14) |
| O(2)-S(1)-N(1) | 105.23(14) |
| O(1)-S(1)-C(21) | 108.88(15) |
| O(2)-S(1)-C(21) | 109.22(17) |
| N(1)-S(1)-C(21) | 105.70(14) |
| O(5)-S(2)-O(6) | 120.31(15) |
| O(5)-S(2)-N(2) | 106.68(15) |
| O(6)-S(2)-N(2) | 105.23(14) |

| | |
|---|---|
| O(5)-S(2)-C(48) | 108.65(15) |
| O(6)-S(2)-C(48) | 108.59(17) |
| N(2)-S(2)-C(48) | 106.55(14) |
| C(2)-N(1)-C(1) | 114.8(2) |
| C(2)-N(1)-S(1) | 117.1(2) |
| C(1)-N(1)-S(1) | 116.5(2) |
| C(29)-N(2)-C(28) | 114.5(3) |
| C(29)-N(2)-S(2) | 118.8(2) |
| C(28)-N(2)-S(2) | 116.0(2) |
| C(6)-O(4)-C(7) | 116.1(3) |
| C(33)-O(8)-C(34) | 116.8(3) |
| N(1)-C(1)-C(5) | 110.0(3) |
| N(1)-C(1)-C(9) | 109.9(3) |
| C(5)-C(1)-C(9) | 114.9(3) |
| N(1)-C(1)-H(1) | 104.4(18) |
| C(5)-C(1)-H(1) | 105.9(19) |
| C(9)-C(1)-H(1) | 111.2(19) |
| C(3)-C(2)-N(1) | 117.9(3) |
| C(3)-C(2)-C(15) | 123.2(3) |
| N(1)-C(2)-C(15) | 118.7(3) |
| C(2)-C(3)-C(4) | 121.4(3) |
| C(2)-C(3)-H(2) | 119.3 |
| C(4)-C(3)-H(2) | 119.3 |
| C(5)-C(4)-C(3) | 120.1(3) |
| C(5)-C(4)-H(3) | 120.0 |
| C(3)-C(4)-H(3) | 120.0 |
| C(4)-C(5)-C(6) | 124.0(3) |
| C(4)-C(5)-C(1) | 117.9(3) |
| C(6)-C(5)-C(1) | 118.2(3) |
| O(3)-C(6)-O(4) | 123.3(3) |
| O(3)-C(6)-C(5) | 123.2(4) |
| O(4)-C(6)-C(5) | 113.5(3) |
| C(8)-C(7)-O(4) | 110.1(4) |
| C(8)-C(7)-H(5) | 109.6 |
| O(4)-C(7)-H(5) | 109.6 |
| C(8)-C(7)-H(4) | 109.6 |
| O(4)-C(7)-H(4) | 109.6 |
| H(5)-C(7)-H(4) | 108.2 |
| C(7)-C(8)-H(8) | 109.5 |
| C(7)-C(8)-H(7) | 109.5 |
| H(8)-C(8)-H(7) | 109.5 |
| C(7)-C(8)-H(6) | 109.5 |
| H(8)-C(8)-H(6) | 109.5 |
| H(7)-C(8)-H(6) | 109.5 |
| C(10)-C(9)-C(14) | 117.6(4) |
| C(10)-C(9)-C(1) | 122.4(3) |
| C(14)-C(9)-C(1) | 120.0(3) |
| C(9)-C(10)-C(11) | 121.6(4) |
| C(9)-C(10)-H(9) | 114(2) |
| C(11)-C(10)-H(9) | 124(2) |
| C(12)-C(11)-C(10) | 120.7(4) |
| C(12)-C(11)-H(10) | 119.7 |
| C(10)-C(11)-H(10) | 119.7 |
| C(11)-C(12)-C(13) | 119.4(4) |
| C(11)-C(12)-H(11) | 120.3 |
| C(13)-C(12)-H(11) | 120.3 |
| C(12)-C(13)-C(14) | 120.2(4) |
| C(12)-C(13)-H(12) | 119.9 |
| C(14)-C(13)-H(12) | 119.9 |
| C(9)-C(14)-C(13) | 120.5(4) |
| C(9)-C(14)-H(13) | 119.7 |
| C(13)-C(14)-H(13) | 119.7 |
| C(20)-C(15)-C(16) | 117.7(3) |
| C(20)-C(15)-C(2) | 120.8(3) |
| C(16)-C(15)-C(2) | 121.4(3) |
| C(17)-C(16)-C(15) | 120.8(4) |
| C(17)-C(16)-H(14) | 119.6 |
| C(15)-C(16)-H(14) | 119.6 |

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|---|---|
| C(18)-C(17)-C(16) | 120.2(4) |
| C(18)-C(17)-H(15) | 123(2) |
| C(16)-C(17)-H(15) | 117(2) |
| C(19)-C(18)-C(17) | 120.0(4) |
| C(19)-C(18)-H(16) | 121(3) |
| C(17)-C(18)-H(16) | 119(3) |
| C(20)-C(19)-C(18) | 119.6(4) |
| C(20)-C(19)-H(17) | 117(2) |
| C(18)-C(19)-H(17) | 123(2) |
| C(19)-C(20)-C(15) | 121.8(4) |
| C(19)-C(20)-H(18) | 122(2) |
| C(15)-C(20)-H(18) | 116.5(19) |
| C(26)-C(21)-C(22) | 119.6(4) |
| C(26)-C(21)-S(1) | 119.3(3) |
| C(22)-C(21)-S(1) | 121.0(3) |
| C(23)-C(22)-C(21) | 119.2(4) |
| C(23)-C(22)-H(19) | 120(2) |
| C(21)-C(22)-H(19) | 121(2) |
| C(24)-C(23)-C(22) | 121.8(4) |
| C(24)-C(23)-H(20) | 119.1 |
| C(22)-C(23)-H(20) | 119.1 |
| C(23)-C(24)-C(25) | 118.0(4) |
| C(23)-C(24)-C(27) | 121.2(4) |
| C(25)-C(24)-C(27) | 120.8(4) |
| C(26)-C(25)-C(24) | 121.4(4) |
| C(26)-C(25)-H(21) | 119.3 |
| C(24)-C(25)-H(21) | 119.3 |
| C(25)-C(26)-C(21) | 119.9(4) |
| C(25)-C(26)-H(22) | 120.0 |
| C(21)-C(26)-H(22) | 120.0 |
| N(2)-C(28)-C(32) | 109.6(3) |
| N(2)-C(28)-C(36) | 109.3(3) |
| C(32)-C(28)-C(36) | 114.9(3) |
| N(2)-C(28)-H(26) | 102(2) |
| C(32)-C(28)-H(26) | 112(2) |
| C(36)-C(28)-H(26) | 108(2) |
| C(24)-C(27)-H(23) | 109.5 |
| C(24)-C(27)-H(24) | 109.5 |
| H(23)-C(27)-H(24) | 109.5 |
| C(24)-C(27)-H(25) | 109.5 |
| H(23)-C(27)-H(25) | 109.5 |
| H(24)-C(27)-H(25) | 109.5 |
| C(30)-C(29)-N(2) | 118.4(3) |
| C(30)-C(29)-C(42) | 123.4(3) |
| N(2)-C(29)-C(42) | 118.0(3) |
| C(29)-C(30)-C(31) | 121.4(3) |
| C(29)-C(30)-H(27) | 119.3 |
| C(31)-C(30)-H(27) | 119.3 |
| C(32)-C(31)-C(30) | 119.9(3) |
| C(32)-C(31)-H(28) | 119.0(18) |
| C(30)-C(31)-H(28) | 120.7(18) |
| C(31)-C(32)-C(33) | 123.9(3) |
| C(31)-C(32)-C(28) | 118.4(3) |
| C(33)-C(32)-C(28) | 117.6(3) |
| O(7)-C(33)-O(8) | 124.1(3) |
| O(7)-C(33)-C(32) | 124.1(4) |
| O(8)-C(33)-C(32) | 111.8(3) |
| C(35)-C(34)-O(8) | 110.8(4) |
| C(35)-C(34)-H(29) | 109.5 |
| O(8)-C(34)-H(29) | 109.5 |
| C(35)-C(34)-H(30) | 109.5 |
| O(8)-C(34)-H(30) | 109.5 |
| H(29)-C(34)-H(30) | 108.1 |
| C(34)-C(35)-H(32) | 109.5 |
| C(34)-C(35)-H(31) | 109.5 |
| H(32)-C(35)-H(31) | 109.5 |
| C(34)-C(35)-H(33) | 109.5 |
| H(32)-C(35)-H(33) | 109.5 |

| | |
|---|---|
| H(31)-C(35)-H(33) | 109.5 |
| C(37)-C(36)-C(41) | 117.4(4) |
| C(37)-C(36)-C(28) | 122.2(3) |
| C(41)-C(36)-C(28) | 120.4(3) |
| C(38)-C(37)-C(36) | 121.0(4) |
| C(38)-C(37)-H(34) | 122(2) |
| C(36)-C(37)-H(34) | 117(2) |
| C(39)-C(38)-C(37) | 120.8(4) |
| C(39)-C(38)-H(35) | 119.6 |
| C(37)-C(38)-H(35) | 119.6 |
| C(38)-C(39)-C(40) | 119.1(4) |
| C(38)-C(39)-H(36) | 120.4 |
| C(40)-C(39)-H(36) | 120.4 |
| C(41)-C(40)-C(39) | 120.3(4) |
| C(41)-C(40)-H(37) | 119.9 |
| C(39)-C(40)-H(37) | 119.9 |
| C(40)-C(41)-C(36) | 121.4(4) |
| C(40)-C(41)-H(38) | 119.3 |
| C(36)-C(41)-H(38) | 119.3 |
| C(47)-C(42)-C(43) | 118.6(3) |
| C(47)-C(42)-C(29) | 120.0(3) |
| C(43)-C(42)-C(29) | 121.3(3) |
| C(44)-C(43)-C(42) | 119.6(4) |
| C(44)-C(43)-H(39) | 120.2 |
| C(42)-C(43)-H(39) | 120.2 |
| C(45)-C(44)-C(43) | 121.1(4) |
| C(45)-C(44)-H(40) | 119.5 |
| C(43)-C(44)-H(40) | 119.5 |
| C(44)-C(45)-C(46) | 119.5(4) |
| C(44)-C(45)-H(41) | 123(3) |
| C(46)-C(45)-H(41) | 117(3) |
| C(45)-C(46)-C(47) | 120.1(5) |
| C(45)-C(46)-H(42) | 119(2) |
| C(47)-C(46)-H(42) | 121(2) |
| C(42)-C(47)-C(46) | 121.1(4) |
| C(42)-C(47)-H(43) | 119(2) |
| C(46)-C(47)-H(43) | 120(2) |
| C(53)-C(48)-C(49) | 120.2(4) |
| C(53)-C(48)-S(2) | 120.3(3) |
| C(49)-C(48)-S(2) | 119.5(3) |
| C(48)-C(49)-C(50) | 118.5(4) |
| C(48)-C(49)-H(44) | 120.7 |
| C(50)-C(49)-H(44) | 120.7 |
| C(51)-C(50)-C(49) | 121.7(4) |
| C(51)-C(50)-H(45) | 119.1 |
| C(49)-C(50)-H(45) | 119.1 |
| C(50)-C(51)-C(52) | 118.9(4) |
| C(50)-C(51)-C(54) | 121.8(5) |
| C(52)-C(51)-C(54) | 119.3(5) |
| C(53)-C(52)-C(51) | 120.5(4) |
| C(53)-C(52)-H(46) | 120(3) |
| C(51)-C(52)-H(46) | 119(3) |
| C(52)-C(53)-C(48) | 120.1(4) |
| C(52)-C(53)-H(47) | 118(2) |
| C(48)-C(53)-H(47) | 122(2) |
| C(51)-C(54)-H(49) | 109.5 |
| C(51)-C(54)-H(50) | 109.5 |
| H(49)-C(54)-H(50) | 109.5 |
| C(51)-C(54)-H(48) | 109.5 |
| H(49)-C(54)-H(48) | 109.5 |
| H(50)-C(54)-H(48) | 109.5 |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd23203. The anisotropic displacement factor exponent takes the form: -2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 36(1) | 62(1) | 49(1) | 17(1) | 4(1) | 9(1) |
| S(2) | 42(1) | 59(1) | 47(1) | 3(1) | 2(1) | 14(1) |
| N(1) | 40(2) | 48(2) | 39(2) | 13(1) | 2(1) | 9(1) |
| N(2) | 43(2) | 50(2) | 36(2) | 2(1) | 1(1) | 10(1) |
| O(1) | 44(2) | 67(2) | 61(2) | 20(1) | -4(1) | 16(1) |
| O(2) | 44(2) | 93(2) | 55(2) | 26(1) | 14(1) | 11(1) |
| O(3) | 108(3) | 85(2) | 65(2) | 40(2) | 17(2) | 26(2) |
| O(4) | 104(2) | 53(2) | 71(2) | 20(2) | 13(2) | 29(2) |
| O(6) | 49(2) | 99(2) | 55(2) | -1(1) | 15(1) | 16(2) |
| O(5) | 54(2) | 60(2) | 72(2) | 10(1) | -12(1) | 20(1) |
| O(7) | 97(2) | 86(2) | 61(2) | 32(2) | 18(2) | 24(2) |
| O(8) | 121(3) | 53(2) | 64(2) | 17(1) | 9(2) | 30(2) |
| C(1) | 38(2) | 57(3) | 39(2) | 17(2) | 6(2) | 6(2) |
| C(2) | 38(2) | 46(2) | 35(2) | 3(2) | 1(2) | 5(2) |
| C(3) | 46(2) | 46(2) | 44(2) | 11(2) | 10(2) | 7(2) |
| C(4) | 47(2) | 45(2) | 46(2) | 3(2) | 3(2) | 13(2) |
| C(5) | 43(2) | 46(2) | 43(2) | 10(2) | -3(2) | 6(2) |
| C(6) | 55(3) | 57(3) | 55(3) | 16(2) | -7(2) | 8(2) |
| C(7) | 185(6) | 72(4) | 82(3) | 27(3) | 8(4) | 70(4) |
| C(8) | 620(20) | 58(5) | 208(8) | 10(5) | -174(11) | 75(8) |
| C(9) | 49(2) | 60(3) | 33(2) | 8(2) | 2(2) | 15(2) |
| C(10) | 64(3) | 81(3) | 67(3) | -21(3) | -16(2) | 25(3) |
| C(11) | 76(3) | 99(4) | 90(4) | -28(3) | -31(3) | 24(3) |
| C(12) | 88(4) | 110(4) | 62(3) | -23(3) | -9(3) | 13(3) |
| C(13) | 92(4) | 116(4) | 79(4) | -35(3) | 20(3) | 26(3) |
| C(14) | 55(3) | 94(4) | 64(3) | -11(3) | 11(2) | 18(3) |
| C(15) | 37(2) | 41(2) | 42(2) | 7(2) | 1(2) | 7(2) |
| C(16) | 49(2) | 53(2) | 51(2) | 5(2) | 3(2) | 12(2) |
| C(17) | 72(3) | 50(3) | 75(3) | -2(2) | 1(2) | 22(2) |
| C(18) | 71(3) | 50(3) | 87(4) | 24(3) | 1(3) | 12(2) |
| C(19) | 87(3) | 54(3) | 58(3) | 19(2) | 11(2) | 17(2) |
| C(20) | 67(3) | 49(3) | 50(2) | 8(2) | 6(2) | 15(2) |
| C(21) | 34(2) | 56(2) | 51(2) | 19(2) | -5(2) | 3(2) |
| C(22) | 55(3) | 70(3) | 61(3) | 23(3) | -6(2) | -3(2) |
| C(23) | 64(3) | 49(3) | 98(4) | 18(3) | -15(3) | 2(2) |
| C(24) | 49(3) | 58(3) | 89(3) | -1(3) | -2(2) | 1(2) |
| C(25) | 56(3) | 66(3) | 66(3) | 6(2) | 5(2) | 3(2) |
| C(26) | 47(2) | 52(3) | 54(2) | 12(2) | -1(2) | 0(2) |
| C(28) | 44(2) | 53(2) | 33(2) | 8(2) | 5(2) | 10(2) |
| C(27) | 92(4) | 64(3) | 138(4) | -10(3) | 16(3) | 5(3) |
| C(29) | 52(2) | 42(2) | 36(2) | 2(2) | 4(2) | 6(2) |
| C(30) | 59(2) | 49(2) | 45(2) | 7(2) | 18(2) | 16(2) |
| C(31) | 52(2) | 48(2) | 48(2) | 3(2) | 8(2) | 17(2) |
| C(32) | 43(2) | 46(2) | 40(2) | 4(2) | -2(2) | 8(2) |
| C(33) | 55(3) | 55(3) | 50(2) | 13(2) | -7(2) | 5(2) |
| C(34) | 165(5) | 57(3) | 94(4) | 33(3) | -11(4) | 31(3) |
| C(35) | 237(8) | 70(4) | 139(5) | 39(4) | -71(5) | -7(5) |
| C(36) | 42(2) | 56(2) | 34(2) | 4(2) | 2(2) | 9(2) |
| C(37) | 51(3) | 61(3) | 68(3) | -10(2) | -8(2) | 15(2) |
| C(38) | 53(3) | 80(3) | 86(3) | -12(3) | -16(2) | 11(2) |
| C(39) | 64(3) | 89(4) | 60(3) | -21(2) | -4(2) | -1(3) |
| C(40) | 73(3) | 95(4) | 76(3) | -40(3) | 12(3) | 16(3) |
| C(41) | 44(2) | 93(3) | 66(3) | -24(2) | 5(2) | 19(2) |
| C(42) | 53(2) | 42(2) | 43(2) | 4(2) | 3(2) | 11(2) |
| C(43) | 63(3) | 54(3) | 58(2) | 2(2) | 3(2) | 16(2) |
| C(44) | 84(3) | 48(3) | 95(4) | 0(3) | -2(3) | 23(2) |
| C(45) | 99(4) | 62(3) | 93(4) | 35(3) | 4(3) | 16(3) |
| C(46) | 127(5) | 74(4) | 61(3) | 22(3) | 16(3) | 28(3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(47) | 105(4) | 55(3) | 47(3) | 14(2) | 10(2) | 23(3) |
| C(48) | 42(2) | 59(3) | 41(2) | 6(2) | -3(2) | 9(2) |
| C(49) | 68(3) | 68(3) | 54(3) | 16(2) | 0(2) | 4(2) |
| C(50) | 79(3) | 56(3) | 92(4) | 20(3) | -10(3) | 3(2) |
| C(51) | 72(3) | 58(3) | 90(4) | -14(3) | -20(3) | 14(2) |
| C(52) | 87(4) | 66(3) | 59(3) | -6(3) | -4(3) | 17(3) |
| C(53) | 65(3) | 61(3) | 51(3) | 8(2) | 0(2) | 12(2) |
| C(54) | 135(5) | 66(4) | 152(5) | -20(3) | -24(4) | 25(3) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd23203.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(2) | 6159 | 5452 | 725 | 54 |
| H(3) | 6347 | 3869 | 1057 | 55 |
| H(5) | 6759 | 1486 | 2118 | 129 |
| H(4) | 4680 | 1280 | 2087 | 129 |
| H(8) | 5543 | 1063 | 1027 | 453 |
| H(7) | 4703 | 237 | 1418 | 453 |
| H(6) | 6767 | 500 | 1417 | 453 |
| H(10) | 8637 | 5993 | 3143 | 108 |
| H(11) | 7288 | 7152 | 3592 | 106 |
| H(12) | 4382 | 7243 | 3320 | 114 |
| H(13) | 2806 | 6134 | 2612 | 85 |
| H(14) | 2809 | 7162 | 1548 | 61 |
| H(20) | 1052 | 1357 | 922 | 86 |
| H(21) | 1498 | 3017 | -451 | 76 |
| H(22) | 1115 | 4357 | 119 | 62 |
| H(23) | 2205 | 738 | 33 | 149 |
| H(24) | 2341 | 1388 | -511 | 149 |
| H(25) | 497 | 828 | -364 | 149 |
| H(27) | -1474 | -620 | 4310 | 59 |
| H(29) | -325 | 3068 | 2555 | 125 |
| H(30) | -2045 | 3196 | 2865 | 125 |
| H(32) | -417 | 3946 | 3648 | 232 |
| H(31) | 167 | 4481 | 3087 | 232 |
| H(33) | 1329 | 3765 | 3374 | 232 |
| H(35) | -4283 | -1584 | 1995 | 89 |
| H(36) | -3076 | -2831 | 1572 | 88 |
| H(37) | -143 | -2929 | 1798 | 98 |
| H(38) | 1566 | -1768 | 2417 | 81 |
| H(39) | 1478 | -2641 | 3577 | 69 |
| H(40) | 1736 | -4016 | 4093 | 90 |
| H(44) | 4322 | 1647 | 3222 | 77 |
| H(45) | 4360 | 3188 | 3667 | 92 |
| H(49) | 4821 | 4260 | 4569 | 178 |
| H(50) | 4592 | 3783 | 5173 | 178 |
| H(48) | 2919 | 3906 | 4768 | 178 |
| H(1) | 2860(30) | 4290(19) | 2148(12) | 41(9) |
| H(9) | 7040(40) | 4940(20) | 2397(13) | 53(11) |
| H(15) | 2470(40) | 8600(30) | 1165(14) | 62(12) |
| H(16) | 2960(50) | 8840(30) | 184(17) | 95(15) |
| H(17) | 4030(50) | 7620(30) | -386(15) | 70(12) |
| H(18) | 4400(40) | 6160(30) | 39(14) | 62(11) |
| H(19) | 680(50) | 2680(20) | 1526(15) | 69(12) |
| H(26) | 1810(40) | 80(30) | 2777(14) | 67(12) |
| H(28) | -1410(40) | 940(20) | 3891(12) | 41(9) |
| H(34) | -2550(40) | -410(20) | 2626(13) | 54(11) |
| H(41) | 1360(60) | -4010(30) | 5100(19) | 110(16) |
| H(42) | 750(50) | -2640(30) | 5558(16) | 69(12) |
| H(43) | 460(50) | -1210(30) | 5058(17) | 94(15) |
| H(46) | 3910(50) | 2040(30) | 5224(18) | 96(16) |
| H(47) | 3830(50) | 500(30) | 4801(15) | 71(12) |

Table 6. Torsion angles [deg] for cd23203.

| | |
|---|---|
| O(1)-S(1)-N(1)-C(2) | -47.9(3) |
| O(2)-S(1)-N(1)-C(2) | -176.5(2) |
| C(21)-S(1)-N(1)-C(2) | 68.0(3) |
| O(1)-S(1)-N(1)-C(1) | 170.7(2) |
| O(2)-S(1)-N(1)-C(1) | 42.1(3) |
| C(21)-S(1)-N(1)-C(1) | -73.5(3) |
| O(5)-S(2)-N(2)-C(29) | 44.4(3) |
| O(6)-S(2)-N(2)-C(29) | 173.3(2) |
| C(48)-S(2)-N(2)-C(29) | -71.5(3) |
| O(5)-S(2)-N(2)-C(28) | -172.8(2) |
| O(6)-S(2)-N(2)-C(28) | -44.0(3) |
| C(48)-S(2)-N(2)-C(28) | 71.2(2) |
| C(2)-N(1)-C(1)-C(5) | -47.4(4) |
| S(1)-N(1)-C(1)-C(5) | 94.9(3) |
| C(2)-N(1)-C(1)-C(9) | 80.0(3) |
| S(1)-N(1)-C(1)-C(9) | -137.7(3) |
| C(1)-N(1)-C(2)-C(3) | 27.2(4) |
| S(1)-N(1)-C(2)-C(3) | -114.8(3) |
| C(1)-N(1)-C(2)-C(15) | -148.5(3) |
| S(1)-N(1)-C(2)-C(15) | 69.5(3) |
| N(1)-C(2)-C(3)-C(4) | 6.8(5) |
| C(15)-C(2)-C(3)-C(4) | -177.7(3) |
| C(2)-C(3)-C(4)-C(5) | -18.9(5) |
| C(3)-C(4)-C(5)-C(6) | 176.0(3) |
| C(3)-C(4)-C(5)-C(1) | -5.0(5) |
| N(1)-C(1)-C(5)-C(4) | 36.6(4) |
| C(9)-C(1)-C(5)-C(4) | -88.0(4) |
| N(1)-C(1)-C(5)-C(6) | -144.4(3) |
| C(9)-C(1)-C(5)-C(6) | 91.1(4) |
| C(7)-O(4)-C(6)-O(3) | 2.6(6) |
| C(7)-O(4)-C(6)-C(5) | -177.2(4) |
| C(4)-C(5)-C(6)-O(3) | 170.0(4) |
| C(1)-C(5)-C(6)-O(3) | -9.0(6) |
| C(4)-C(5)-C(6)-O(4) | -10.2(5) |
| C(1)-C(5)-C(6)-O(4) | 170.8(3) |
| C(6)-O(4)-C(7)-C(8) | 152.0(6) |
| N(1)-C(1)-C(9)-C(10) | -128.7(4) |
| C(5)-C(1)-C(9)-C(10) | -4.1(5) |
| N(1)-C(1)-C(9)-C(14) | 52.5(4) |
| C(5)-C(1)-C(9)-C(14) | 177.1(3) |
| C(14)-C(9)-C(10)-C(11) | 1.5(7) |
| C(1)-C(9)-C(10)-C(11) | -177.4(4) |
| C(9)-C(10)-C(11)-C(12) | -1.4(8) |
| C(10)-C(11)-C(12)-C(13) | 0.1(8) |
| C(11)-C(12)-C(13)-C(14) | 1.0(8) |
| C(10)-C(9)-C(14)-C(13) | -0.3(6) |
| C(1)-C(9)-C(14)-C(13) | 178.6(4) |
| C(12)-C(13)-C(14)-C(9) | -0.9(7) |
| C(3)-C(2)-C(15)-C(20) | 36.8(5) |
| N(1)-C(2)-C(15)-C(20) | -147.8(3) |
| C(3)-C(2)-C(15)-C(16) | -139.1(4) |
| N(1)-C(2)-C(15)-C(16) | 36.4(5) |
| C(20)-C(15)-C(16)-C(17) | 0.1(5) |
| C(2)-C(15)-C(16)-C(17) | 176.0(3) |
| C(15)-C(16)-C(17)-C(18) | -0.5(6) |
| C(16)-C(17)-C(18)-C(19) | 0.7(7) |
| C(17)-C(18)-C(19)-C(20) | -0.4(7) |
| C(18)-C(19)-C(20)-C(15) | 0.0(7) |
| C(16)-C(15)-C(20)-C(19) | 0.2(6) |
| C(2)-C(15)-C(20)-C(19) | -175.8(4) |
| O(1)-S(1)-C(21)-C(26) | 27.6(3) |
| O(2)-S(1)-C(21)-C(26) | 160.4(3) |
| N(1)-S(1)-C(21)-C(26) | -86.9(3) |
| O(1)-S(1)-C(21)-C(22) | -155.2(3) |

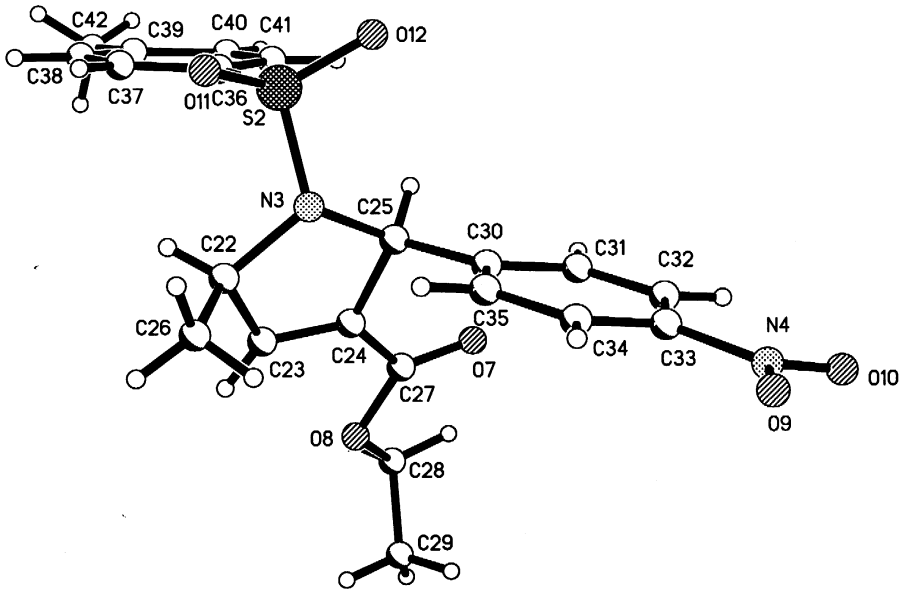
| | |
|---|---|
| O(2)-S(1)-C(21)-C(22) | -22.4(3) |
| N(1)-S(1)-C(21)-C(22) | 90.4(3) |
| C(26)-C(21)-C(22)-C(23) | 1.9(6) |
| S(1)-C(21)-C(22)-C(23) | -175.3(3) |
| C(21)-C(22)-C(23)-C(24) | -0.5(6) |
| C(22)-C(23)-C(24)-C(25) | -1.1(6) |
| C(22)-C(23)-C(24)-C(27) | 179.2(4) |
| C(23)-C(24)-C(25)-C(26) | 1.3(6) |
| C(27)-C(24)-C(25)-C(26) | -179.1(4) |
| C(24)-C(25)-C(26)-C(21) | 0.2(6) |
| C(22)-C(21)-C(26)-C(25) | -1.8(5) |
| S(1)-C(21)-C(26)-C(25) | 175.5(3) |
| C(29)-N(2)-C(28)-C(32) | 46.9(4) |
| S(2)-N(2)-C(28)-C(32) | -97.5(3) |
| C(29)-N(2)-C(28)-C(36) | -79.9(3) |
| S(2)-N(2)-C(28)-C(36) | 135.7(2) |
| C(28)-N(2)-C(29)-C(30) | -26.8(4) |
| S(2)-N(2)-C(29)-C(30) | 116.5(3) |
| C(28)-N(2)-C(29)-C(42) | 148.7(3) |
| S(2)-N(2)-C(29)-C(42) | -68.0(4) |
| N(2)-C(29)-C(30)-C(31) | -7.0(5) |
| C(42)-C(29)-C(30)-C(31) | 177.8(3) |
| C(29)-C(30)-C(31)-C(32) | 18.8(6) |
| C(30)-C(31)-C(32)-C(33) | -176.5(3) |
| C(30)-C(31)-C(32)-C(28) | 5.5(5) |
| N(2)-C(28)-C(32)-C(31) | -36.7(4) |
| C(36)-C(28)-C(32)-C(31) | 86.9(4) |
| N(2)-C(28)-C(32)-C(33) | 145.1(3) |
| C(36)-C(28)-C(32)-C(33) | -91.3(4) |
| C(34)-O(8)-C(33)-O(7) | -2.2(6) |
| C(34)-O(8)-C(33)-C(32) | 178.2(4) |
| C(31)-C(32)-C(33)-O(7) | -170.3(4) |
| C(28)-C(32)-C(33)-O(7) | 7.7(6) |
| C(31)-C(32)-C(33)-O(8) | 9.3(5) |
| C(28)-C(32)-C(33)-O(8) | -172.7(3) |
| C(33)-O(8)-C(34)-C(35) | -121.3(5) |
| N(2)-C(28)-C(36)-C(37) | 125.3(4) |
| C(32)-C(28)-C(36)-C(37) | 1.6(5) |
| N(2)-C(28)-C(36)-C(41) | -55.9(4) |
| C(32)-C(28)-C(36)-C(41) | -179.6(3) |
| C(41)-C(36)-C(37)-C(38) | -1.1(6) |
| C(28)-C(36)-C(37)-C(38) | 177.7(4) |
| C(36)-C(37)-C(38)-C(39) | 0.5(7) |
| C(37)-C(38)-C(39)-C(40) | 0.6(7) |
| C(38)-C(39)-C(40)-C(41) | -1.0(7) |
| C(39)-C(40)-C(41)-C(36) | 0.4(7) |
| C(37)-C(36)-C(41)-C(40) | 0.6(6) |
| C(28)-C(36)-C(41)-C(40) | -178.2(4) |
| C(30)-C(29)-C(42)-C(47) | -38.9(6) |
| N(2)-C(29)-C(42)-C(47) | 145.9(4) |
| C(30)-C(29)-C(42)-C(43) | 137.0(4) |
| N(2)-C(29)-C(42)-C(43) | -38.2(5) |
| C(47)-C(42)-C(43)-C(44) | 0.0(6) |
| C(29)-C(42)-C(43)-C(44) | -175.9(4) |
| C(42)-C(43)-C(44)-C(45) | 0.7(7) |
| C(43)-C(44)-C(45)-C(46) | -1.5(8) |
| C(44)-C(45)-C(46)-C(47) | 1.7(8) |
| C(43)-C(42)-C(47)-C(46) | 0.2(7) |
| C(29)-C(42)-C(47)-C(46) | 176.2(4) |
| C(45)-C(46)-C(47)-C(42) | -1.1(8) |
| O(5)-S(2)-C(48)-C(53) | -28.2(4) |
| O(6)-S(2)-C(48)-C(53) | -160.7(3) |
| N(2)-S(2)-C(48)-C(53) | 86.4(3) |
| O(5)-S(2)-C(48)-C(49) | 154.5(3) |
| O(6)-S(2)-C(48)-C(49) | 22.0(3) |
| N(2)-S(2)-C(48)-C(49) | -90.9(3) |
| C(53)-C(48)-C(49)-C(50) | -0.8(6) |
| S(2)-C(48)-C(49)-C(50) | 176.5(3) |

| | |
|---|---|
| C(48)-C(49)-C(50)-C(51) | 0.4(6) |
| C(49)-C(50)-C(51)-C(52) | 0.7(7) |
| C(49)-C(50)-C(51)-C(54) | -179.0(4) |
| C(50)-C(51)-C(52)-C(53) | -1.3(7) |
| C(54)-C(51)-C(52)-C(53) | 178.4(4) |
| C(51)-C(52)-C(53)-C(48) | 0.9(7) |
| C(49)-C(48)-C(53)-C(52) | 0.2(6) |
| S(2)-C(48)-C(53)-C(52) | -177.1(3) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23203 [A and deg.].

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




The crystal data of **4j** has been deposited in CCDC with number 235749. Empirical Formula: $C_{21}H_{22}N_2O_6S$; Formula Weight: 430.47; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.368 x 0.327 x 0.163 mm; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 20.5357(16)Å, b = 11.4427(9)Å, c = 20.5964(15)Å, $\alpha = 90^o$, $\beta = 117.564(2)^o$, $\gamma = 90^o$, V= 4290.5(6)Å$^3$; Space group: P2(1)/n; Z = 8; $D_{calc} = 1.333$ g/cm$^3$; $F_{000} = 1808$; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0632, 0.1559.

Table 1. Crystal data and structure refinement for cd24138.

| | |
|---|---|
| Identification code | cd24138 |
| Empirical formula | C21 H22 N2 O6 S |
| Formula weight | 430.47 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/n |
| Unit cell dimensions | a = 20.5357(16) A alpha = 90 deg.<br>b = 11.4427(9) A beta = 117.564(2) deg.<br>c = 20.5964(15) A gamma = 90 deg. |
| Volume | 4290.5(6) A^3 |
| Z, Calculated density | 8, 1.333 Mg/m^3 |
| Absorption coefficient | 0.190 mm^-1 |
| F(000) | 1808 |
| Crystal size | 0.368 x 0.327 x 0.163 mm |
| Theta range for data collection | 1.16 to 28.32 deg. |
| Limiting indices | -26<=h<=27, -14<=k<=14, -27<=l<=18 |
| Reflections collected / unique | 25789 / 9877 [R(int) = 0.0730] |
| Completeness to theta = 28.32 | 92.4 % |
| Absorption correction | Empirical |
| Max. and min. transmission | 1.00000 and 0.51549 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 9877 / 6 / 584 |
| Goodness-of-fit on F^2 | 0.740 |
| Final R indices [I>2sigma(I)] | R1 = 0.0632, wR2 = 0.1559 |
| R indices (all data) | R1 = 0.1798, wR2 = 0.1880 |
| Largest diff. peak and hole | 0.669 and -0.377 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd24138.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 7447(1) | 2072(1) | 7042(1) | 58(1) |
| S(2) | 2418(1) | 1521(1) | 7047(1) | 54(1) |
| N(1) | 6712(2) | 1639(3) | 6314(2) | 49(1) |
| N(2) | 5862(3) | -3856(5) | 5955(3) | 85(2) |
| N(3) | 1677(2) | 1066(3) | 6335(2) | 47(1) |
| N(4) | 819(3) | -4432(5) | 5963(3) | 89(2) |
| O(1) | 6668(2) | 179(3) | 4361(2) | 80(1) |
| O(2) | 5841(2) | 1555(4) | 3816(2) | 110(2) |
| O(3) | 5450(2) | -4015(4) | 6225(3) | 111(2) |
| O(4) | 6077(3) | -4630(4) | 5697(3) | 125(2) |
| O(5) | 7184(2) | 2599(3) | 7507(2) | 74(1) |
| O(6) | 7933(2) | 1093(3) | 7284(2) | 74(1) |
| O(7) | 1609(2) | -369(3) | 4357(2) | 84(1) |
| O(8) | 740(2) | 960(3) | 3817(2) | 107(2) |
| O(9) | 429(3) | -4599(4) | 6251(3) | 128(2) |
| O(10) | 999(3) | -5193(4) | 5662(3) | 129(2) |
| O(11) | 2171(2) | 2061(3) | 7518(2) | 69(1) |
| O(12) | 2905(2) | 541(3) | 7292(2) | 68(1) |
| C(1) | 6149(3) | 2552(4) | 5914(3) | 54(1) |
| C(2) | 5990(2) | 2331(4) | 5148(2) | 52(1) |
| C(3) | 6320(2) | 1405(4) | 5075(2) | 46(1) |
| C(4) | 6797(3) | 816(4) | 5806(2) | 48(1) |
| C(5) | 5475(4) | 2473(7) | 6029(4) | 77(2) |
| C(6) | 6298(3) | 957(4) | 4391(3) | 59(1) |
| C(7) | 5828(5) | 1268(7) | 3096(4) | 180(4) |
| C(8) | 5153(8) | 669(13) | 2673(9) | 401(12) |
| C(9) | 6563(2) | -425(4) | 5874(2) | 47(1) |
| C(10) | 6833(2) | -1356(4) | 5639(2) | 60(1) |
| C(11) | 6608(3) | -2482(5) | 5672(3) | 68(2) |
| C(12) | 6123(3) | -2657(4) | 5943(3) | 60(1) |
| C(13) | 5861(3) | -1772(5) | 6189(2) | 64(1) |
| C(14) | 6086(3) | -642(5) | 6159(3) | 58(1) |
| C(15) | 7845(2) | 3175(4) | 6755(2) | 51(1) |
| C(16) | 7736(3) | 4329(5) | 6867(3) | 57(1) |
| C(17) | 8008(3) | 5196(4) | 6594(3) | 69(1) |
| C(18) | 8394(3) | 4944(5) | 6213(3) | 68(1) |
| C(19) | 8523(3) | 3785(5) | 6140(3) | 76(2) |
| C(20) | 8252(3) | 2890(4) | 6407(3) | 66(1) |
| C(21) | 8664(3) | 5905(5) | 5894(3) | 111(2) |
| C(22) | 1105(3) | 1964(5) | 5941(3) | 56(1) |
| C(23) | 933(2) | 1739(4) | 5166(2) | 56(1) |
| C(24) | 1263(2) | 816(4) | 5083(2) | 48(1) |
| C(25) | 1756(2) | 249(4) | 5814(2) | 46(1) |
| C(26) | 444(3) | 1872(5) | 6086(3) | 85(2) |
| C(27) | 1226(3) | 390(5) | 4394(3) | 59(1) |
| C(28) | 831(5) | 740(8) | 3117(4) | 203(5) |
| C(29) | 112(7) | 529(12) | 2580(8) | 350(9) |
| C(30) | 1532(2) | -991(4) | 5897(2) | 46(1) |
| C(31) | 1762(3) | -1930(5) | 5620(2) | 64(1) |
| C(32) | 1545(3) | -3044(5) | 5648(3) | 67(1) |
| C(33) | 1091(3) | -3234(5) | 5956(3) | 62(1) |
| C(34) | 872(2) | -2349(5) | 6255(2) | 61(1) |
| C(35) | 1092(2) | -1231(4) | 6221(2) | 56(1) |
| C(36) | 2811(2) | 2618(4) | 6741(2) | 49(1) |
| C(37) | 2692(2) | 3773(5) | 6829(2) | 60(1) |
| C(38) | 2972(3) | 4622(5) | 6550(3) | 67(2) |
| C(39) | 3372(3) | 4353(5) | 6189(3) | 73(2) |

| | | | | |
|---|---|---|---|---|
| C(40) | 3498(3) | 3180(5) | 6127(3) | 80(2) |
| C(41) | 3229(3) | 2308(4) | 6407(3) | 66(1) |
| C(42) | 3649(3) | 5283(5) | 5863(3) | 108(2) |

Table 3. Bond lengths [A] and angles [deg] for cd24138.

| | |
|---|---|
| S(1)-O(6) | 1.429(3) |
| S(1)-O(5) | 1.430(3) |
| S(1)-N(1) | 1.636(3) |
| S(1)-C(15) | 1.749(5) |
| S(2)-O(11) | 1.427(3) |
| S(2)-O(12) | 1.431(3) |
| S(2)-N(3) | 1.634(3) |
| S(2)-C(36) | 1.759(4) |
| N(1)-C(4) | 1.477(5) |
| N(1)-C(1) | 1.491(5) |
| N(2)-O(4) | 1.217(6) |
| N(2)-O(3) | 1.221(5) |
| N(2)-C(12) | 1.477(6) |
| N(3)-C(25) | 1.486(5) |
| N(3)-C(22) | 1.487(5) |
| N(4)-O(9) | 1.213(6) |
| N(4)-O(10) | 1.221(6) |
| N(4)-C(33) | 1.482(6) |
| O(1)-C(6) | 1.191(5) |
| O(2)-C(6) | 1.312(5) |
| O(2)-C(7) | 1.509(6) |
| O(7)-C(27) | 1.197(5) |
| O(8)-C(27) | 1.318(5) |
| O(8)-C(28) | 1.558(7) |
| C(1)-C(2) | 1.477(6) |
| C(1)-C(5) | 1.511(7) |
| C(1)-H(1) | 0.97(2) |
| C(2)-C(3) | 1.305(5) |
| C(2)-H(2) | 0.9300 |
| C(3)-C(6) | 1.479(6) |
| C(3)-C(4) | 1.521(6) |
| C(4)-C(9) | 1.527(6) |
| C(4)-H(3) | 0.91(4) |
| C(5)-H(4) | 0.97(4) |
| C(5)-H(5) | 0.97(4) |
| C(5)-H(6) | 0.94(2) |
| C(7)-C(8) | 1.425(14) |
| C(7)-H(7) | 0.9700 |
| C(7)-H(8) | 0.9700 |
| C(8)-H(9) | 0.9600 |
| C(8)-H(10) | 0.9600 |
| C(8)-H(11) | 0.9600 |
| C(9)-C(14) | 1.377(6) |
| C(9)-C(10) | 1.387(5) |
| C(10)-C(11) | 1.381(6) |
| C(10)-H(12) | 0.9300 |
| C(11)-C(12) | 1.365(7) |
| C(11)-H(13) | 0.96(4) |
| C(12)-C(13) | 1.350(6) |
| C(13)-C(14) | 1.384(6) |
| C(13)-H(14) | 0.9300 |
| C(14)-H(15) | 0.96(4) |
| C(15)-C(20) | 1.370(5) |
| C(15)-C(16) | 1.376(6) |
| C(16)-C(17) | 1.380(6) |
| C(16)-H(16) | 0.96(4) |
| C(17)-C(18) | 1.380(6) |
| C(17)-H(17) | 0.9300 |
| C(18)-C(19) | 1.373(7) |
| C(18)-C(21) | 1.512(6) |
| C(19)-C(20) | 1.394(6) |
| C(19)-H(18) | 0.94(4) |
| C(20)-H(19) | 0.9300 |
| C(21)-H(21) | 0.9600 |

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|---|---|
| C(21)-H(20) | 0.9600 |
| C(21)-H(22) | 0.9600 |
| C(22)-C(23) | 1.489(6) |
| C(22)-C(26) | 1.523(6) |
| C(22)-H(23) | 0.91(4) |
| C(23)-C(24) | 1.308(5) |
| C(23)-H(24) | 0.9300 |
| C(24)-C(27) | 1.469(6) |
| C(24)-C(25) | 1.518(6) |
| C(25)-C(30) | 1.526(6) |
| C(25)-H(25) | 0.986(19) |
| C(26)-H(27) | 0.9600 |
| C(26)-H(26) | 0.9600 |
| C(26)-H(28) | 0.9600 |
| C(28)-C(29) | 1.398(12) |
| C(28)-H(29) | 0.9700 |
| C(28)-H(30) | 0.9700 |
| C(29)-H(33) | 0.9600 |
| C(29)-H(32) | 0.9600 |
| C(29)-H(31) | 0.9600 |
| C(30)-C(35) | 1.377(5) |
| C(30)-C(31) | 1.397(6) |
| C(31)-C(32) | 1.361(6) |
| C(31)-H(34) | 0.9300 |
| C(32)-C(33) | 1.366(6) |
| C(32)-H(35) | 0.9300 |
| C(33)-C(34) | 1.364(6) |
| C(34)-C(35) | 1.369(6) |
| C(34)-H(36) | 0.9300 |
| C(35)-H(37) | 0.9300 |
| C(36)-C(37) | 1.371(6) |
| C(36)-C(41) | 1.374(5) |
| C(37)-C(38) | 1.381(6) |
| C(37)-H(38) | 0.9300 |
| C(38)-C(39) | 1.374(7) |
| C(38)-H(39) | 0.94(4) |
| C(39)-C(40) | 1.384(7) |
| C(39)-C(42) | 1.504(6) |
| C(40)-C(41) | 1.389(6) |
| C(40)-H(40) | 0.9300 |
| C(41)-H(41) | 0.9300 |
| C(42)-H(42) | 0.9600 |
| C(42)-H(44) | 0.9600 |
| C(42)-H(43) | 0.9600 |

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|---|---|
| O(6)-S(1)-O(5) | 121.1(2) |
| O(6)-S(1)-N(1) | 106.36(19) |
| O(5)-S(1)-N(1) | 105.55(19) |
| O(6)-S(1)-C(15) | 108.8(2) |
| O(5)-S(1)-C(15) | 107.6(2) |
| N(1)-S(1)-C(15) | 106.59(18) |
| O(11)-S(2)-O(12) | 120.70(19) |
| O(11)-S(2)-N(3) | 105.85(18) |
| O(12)-S(2)-N(3) | 105.96(19) |
| O(11)-S(2)-C(36) | 107.5(2) |
| O(12)-S(2)-C(36) | 108.9(2) |
| N(3)-S(2)-C(36) | 107.21(18) |
| C(4)-N(1)-C(1) | 111.7(3) |
| C(4)-N(1)-S(1) | 118.8(3) |
| C(1)-N(1)-S(1) | 116.4(3) |
| O(4)-N(2)-O(3) | 123.9(6) |
| O(4)-N(2)-C(12) | 117.4(6) |
| O(3)-N(2)-C(12) | 118.6(6) |
| C(25)-N(3)-C(22) | 111.3(3) |
| C(25)-N(3)-S(2) | 118.8(3) |
| C(22)-N(3)-S(2) | 116.2(3) |
| O(9)-N(4)-O(10) | 124.0(6) |
| O(9)-N(4)-C(33) | 119.1(6) |

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|---|---|
| O(10)-N(4)-C(33) | 116.8(6) |
| C(6)-O(2)-C(7) | 117.0(4) |
| C(27)-O(8)-C(28) | 112.9(4) |
| C(2)-C(1)-N(1) | 101.7(3) |
| C(2)-C(1)-C(5) | 113.0(4) |
| N(1)-C(1)-C(5) | 113.8(5) |
| C(2)-C(1)-H(1) | 118(2) |
| N(1)-C(1)-H(1) | 105(2) |
| C(5)-C(1)-H(1) | 105(2) |
| C(3)-C(2)-C(1) | 113.1(4) |
| C(3)-C(2)-H(2) | 123.4 |
| C(1)-C(2)-H(2) | 123.4 |
| C(2)-C(3)-C(6) | 127.1(4) |
| C(2)-C(3)-C(4) | 112.1(4) |
| C(6)-C(3)-C(4) | 120.8(4) |
| N(1)-C(4)-C(3) | 100.8(4) |
| N(1)-C(4)-C(9) | 112.5(4) |
| C(3)-C(4)-C(9) | 114.7(4) |
| N(1)-C(4)-H(3) | 110(2) |
| C(3)-C(4)-H(3) | 112(2) |
| C(9)-C(4)-H(3) | 107(2) |
| C(1)-C(5)-H(4) | 109(3) |
| C(1)-C(5)-H(5) | 107(3) |
| H(4)-C(5)-H(5) | 102(4) |
| C(1)-C(5)-H(6) | 111(3) |
| H(4)-C(5)-H(6) | 104(4) |
| H(5)-C(5)-H(6) | 123(4) |
| O(1)-C(6)-O(2) | 123.4(5) |
| O(1)-C(6)-C(3) | 124.6(4) |
| O(2)-C(6)-C(3) | 111.9(4) |
| C(8)-C(7)-O(2) | 106.0(10) |
| C(8)-C(7)-H(7) | 110.5 |
| O(2)-C(7)-H(7) | 110.5 |
| C(8)-C(7)-H(8) | 110.5 |
| O(2)-C(7)-H(8) | 110.5 |
| H(7)-C(7)-H(8) | 108.7 |
| C(7)-C(8)-H(9) | 109.4 |
| C(7)-C(8)-H(10) | 109.5 |
| H(9)-C(8)-H(10) | 109.5 |
| C(7)-C(8)-H(11) | 109.5 |
| H(9)-C(8)-H(11) | 109.5 |
| H(10)-C(8)-H(11) | 109.5 |
| C(14)-C(9)-C(10) | 119.2(5) |
| C(14)-C(9)-C(4) | 121.5(4) |
| C(10)-C(9)-C(4) | 119.3(4) |
| C(11)-C(10)-C(9) | 120.3(5) |
| C(11)-C(10)-H(12) | 119.9 |
| C(9)-C(10)-H(12) | 119.9 |
| C(12)-C(11)-C(10) | 118.6(5) |
| C(12)-C(11)-H(13) | 115(3) |
| C(10)-C(11)-H(13) | 126(3) |
| C(13)-C(12)-C(11) | 122.5(5) |
| C(13)-C(12)-N(2) | 118.7(5) |
| C(11)-C(12)-N(2) | 118.8(5) |
| C(12)-C(13)-C(14) | 119.0(5) |
| C(12)-C(13)-H(14) | 120.5 |
| C(14)-C(13)-H(14) | 120.5 |
| C(9)-C(14)-C(13) | 120.3(5) |
| C(9)-C(14)-H(15) | 119(3) |
| C(13)-C(14)-H(15) | 120(3) |
| C(20)-C(15)-C(16) | 120.2(4) |
| C(20)-C(15)-S(1) | 120.0(4) |
| C(16)-C(15)-S(1) | 119.8(4) |
| C(15)-C(16)-C(17) | 119.5(5) |
| C(15)-C(16)-H(16) | 119(3) |
| C(17)-C(16)-H(16) | 122(3) |
| C(16)-C(17)-C(18) | 122.0(5) |
| C(16)-C(17)-H(17) | 119.0 |

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|---|---|
| C(18)-C(17)-H(17) | 119.0 |
| C(19)-C(18)-C(17) | 117.0(5) |
| C(19)-C(18)-C(21) | 121.8(5) |
| C(17)-C(18)-C(21) | 121.2(5) |
| C(18)-C(19)-C(20) | 122.3(5) |
| C(18)-C(19)-H(18) | 118(3) |
| C(20)-C(19)-H(18) | 120(3) |
| C(15)-C(20)-C(19) | 118.9(5) |
| C(15)-C(20)-H(19) | 120.6 |
| C(19)-C(20)-H(19) | 120.6 |
| C(18)-C(21)-H(21) | 109.5 |
| C(18)-C(21)-H(20) | 109.5 |
| H(21)-C(21)-H(20) | 109.5 |
| C(18)-C(21)-H(22) | 109.5 |
| H(21)-C(21)-H(22) | 109.5 |
| H(20)-C(21)-H(22) | 109.5 |
| N(3)-C(22)-C(23) | 101.5(4) |
| N(3)-C(22)-C(26) | 113.4(4) |
| C(23)-C(22)-C(26) | 114.0(4) |
| N(3)-C(22)-H(23) | 112(2) |
| C(23)-C(22)-H(23) | 109(2) |
| C(26)-C(22)-H(23) | 106(2) |
| C(24)-C(23)-C(22) | 113.6(4) |
| C(24)-C(23)-H(24) | 123.2 |
| C(22)-C(23)-H(24) | 123.2 |
| C(23)-C(24)-C(27) | 126.7(4) |
| C(23)-C(24)-C(25) | 111.3(4) |
| C(27)-C(24)-C(25) | 122.0(4) |
| N(3)-C(25)-C(24) | 101.6(3) |
| N(3)-C(25)-C(30) | 111.5(3) |
| C(24)-C(25)-C(30) | 114.3(4) |
| N(3)-C(25)-H(25) | 108(2) |
| C(24)-C(25)-H(25) | 112(2) |
| C(30)-C(25)-H(25) | 109(2) |
| C(22)-C(26)-H(27) | 109.5 |
| C(22)-C(26)-H(26) | 109.5 |
| H(27)-C(26)-H(26) | 109.5 |
| C(22)-C(26)-H(28) | 109.5 |
| H(27)-C(26)-H(28) | 109.5 |
| H(26)-C(26)-H(28) | 109.5 |
| O(7)-C(27)-O(8) | 123.3(5) |
| O(7)-C(27)-C(24) | 124.1(5) |
| O(8)-C(27)-C(24) | 112.6(5) |
| C(29)-C(28)-O(8) | 103.0(10) |
| C(29)-C(28)-H(29) | 111.1 |
| O(8)-C(28)-H(29) | 111.2 |
| C(29)-C(28)-H(30) | 111.3 |
| O(8)-C(28)-H(30) | 111.2 |
| H(29)-C(28)-H(30) | 109.1 |
| C(28)-C(29)-H(33) | 109.3 |
| C(28)-C(29)-H(32) | 109.5 |
| H(33)-C(29)-H(32) | 109.5 |
| C(28)-C(29)-H(31) | 109.5 |
| H(33)-C(29)-H(31) | 109.5 |
| H(32)-C(29)-H(31) | 109.5 |
| C(35)-C(30)-C(31) | 117.8(4) |
| C(35)-C(30)-C(25) | 122.6(4) |
| C(31)-C(30)-C(25) | 119.6(4) |
| C(32)-C(31)-C(30) | 121.5(5) |
| C(32)-C(31)-H(34) | 119.2 |
| C(30)-C(31)-H(34) | 119.2 |
| C(31)-C(32)-C(33) | 118.5(5) |
| C(31)-C(32)-H(35) | 120.8 |
| C(33)-C(32)-H(35) | 120.8 |
| C(34)-C(33)-C(32) | 121.9(5) |
| C(34)-C(33)-N(4) | 118.6(5) |
| C(32)-C(33)-N(4) | 119.5(5) |
| C(33)-C(34)-C(35) | 119.0(5) |

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|---|---|
| C(33)-C(34)-H(36) | 120.5 |
| C(35)-C(34)-H(36) | 120.5 |
| C(34)-C(35)-C(30) | 121.2(5) |
| C(34)-C(35)-H(37) | 119.4 |
| C(30)-C(35)-H(37) | 119.4 |
| C(37)-C(36)-C(41) | 120.4(4) |
| C(37)-C(36)-S(2) | 120.1(4) |
| C(41)-C(36)-S(2) | 119.5(4) |
| C(36)-C(37)-C(38) | 119.3(5) |
| C(36)-C(37)-H(38) | 120.4 |
| C(38)-C(37)-H(38) | 120.4 |
| C(39)-C(38)-C(37) | 122.4(5) |
| C(39)-C(38)-H(39) | 119(3) |
| C(37)-C(38)-H(39) | 119(3) |
| C(38)-C(39)-C(40) | 116.9(5) |
| C(38)-C(39)-C(42) | 121.8(5) |
| C(40)-C(39)-C(42) | 121.3(5) |
| C(39)-C(40)-C(41) | 122.1(5) |
| C(39)-C(40)-H(40) | 119.0 |
| C(41)-C(40)-H(40) | 119.0 |
| C(36)-C(41)-C(40) | 118.9(5) |
| C(36)-C(41)-H(41) | 120.5 |
| C(40)-C(41)-H(41) | 120.5 |
| C(39)-C(42)-H(42) | 109.5 |
| C(39)-C(42)-H(44) | 109.5 |
| H(42)-C(42)-H(44) | 109.5 |
| C(39)-C(42)-H(43) | 109.5 |
| H(42)-C(42)-H(43) | 109.5 |
| H(44)-C(42)-H(43) | 109.5 |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd24138.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 68(1) | 56(1) | 43(1) | 1(1) | 20(1) | -6(1) |
| S(2) | 62(1) | 53(1) | 42(1) | 1(1) | 20(1) | -6(1) |
| N(1) | 56(2) | 41(2) | 47(2) | -5(2) | 21(2) | -3(2) |
| N(2) | 69(4) | 54(4) | 96(4) | 17(3) | 8(3) | -3(3) |
| N(3) | 47(2) | 46(2) | 45(2) | -2(2) | 19(2) | 2(2) |
| N(4) | 80(4) | 57(4) | 97(4) | 11(3) | 14(3) | -7(3) |
| O(1) | 98(3) | 82(3) | 68(2) | 4(2) | 45(2) | 29(2) |
| O(2) | 153(4) | 130(4) | 53(2) | 23(2) | 53(3) | 75(3) |
| O(3) | 87(3) | 79(3) | 159(4) | 35(3) | 52(3) | -13(2) |
| O(4) | 154(4) | 41(3) | 177(5) | -2(3) | 75(4) | -5(3) |
| O(5) | 101(3) | 81(3) | 45(2) | -12(2) | 39(2) | -17(2) |
| O(6) | 72(2) | 60(2) | 65(2) | 14(2) | 12(2) | 3(2) |
| O(7) | 101(3) | 92(3) | 71(3) | 5(2) | 49(2) | 26(2) |
| O(8) | 155(4) | 117(3) | 46(2) | 17(2) | 43(3) | 62(3) |
| O(9) | 120(4) | 88(4) | 182(5) | 20(3) | 74(4) | -33(3) |
| O(10) | 161(5) | 51(3) | 175(5) | 4(3) | 77(4) | 2(3) |
| O(11) | 90(2) | 78(2) | 46(2) | -9(2) | 38(2) | -10(2) |
| O(12) | 67(2) | 58(2) | 60(2) | 16(2) | 13(2) | 4(2) |
| C(1) | 59(3) | 45(3) | 59(3) | -5(3) | 27(3) | 5(3) |
| C(2) | 53(3) | 48(3) | 54(3) | 13(2) | 25(2) | 6(2) |
| C(3) | 48(3) | 46(3) | 49(3) | 4(2) | 27(2) | 5(2) |
| C(4) | 47(3) | 49(3) | 49(3) | -2(2) | 22(3) | -1(2) |
| C(5) | 79(5) | 89(6) | 74(5) | -1(4) | 45(4) | 13(4) |
| C(6) | 65(3) | 58(4) | 56(3) | 9(3) | 29(3) | 20(3) |
| C(7) | 272(11) | 204(9) | 108(6) | 26(6) | 125(7) | 129(9) |
| C(9) | 51(3) | 45(3) | 38(3) | 1(2) | 16(2) | 1(2) |
| C(10) | 69(3) | 48(3) | 70(3) | 9(3) | 39(3) | 10(3) |
| C(11) | 77(4) | 37(4) | 79(4) | 6(3) | 28(3) | 11(3) |
| C(12) | 57(3) | 43(3) | 61(3) | 13(3) | 11(3) | -4(3) |
| C(13) | 61(3) | 56(4) | 75(4) | 7(3) | 32(3) | -4(3) |
| C(14) | 66(4) | 50(4) | 64(3) | -1(3) | 36(3) | -3(3) |
| C(15) | 53(3) | 51(3) | 45(3) | -9(2) | 18(2) | -6(2) |
| C(16) | 65(3) | 51(4) | 57(3) | -10(3) | 31(3) | -9(3) |
| C(17) | 79(4) | 51(4) | 80(4) | -10(3) | 39(3) | -3(3) |
| C(18) | 76(4) | 60(4) | 71(4) | -8(3) | 36(3) | -18(3) |
| C(19) | 85(4) | 77(5) | 85(4) | -19(3) | 56(4) | -13(3) |
| C(20) | 74(4) | 51(4) | 82(4) | -11(3) | 43(3) | -8(3) |
| C(21) | 120(5) | 93(5) | 143(6) | 4(4) | 80(5) | -28(4) |
| C(22) | 57(3) | 51(4) | 58(3) | -2(3) | 24(3) | 2(3) |
| C(23) | 54(3) | 55(3) | 53(3) | 6(2) | 21(3) | 3(3) |
| C(24) | 48(3) | 51(3) | 47(3) | 3(2) | 24(2) | 0(2) |
| C(25) | 46(3) | 45(3) | 46(3) | 0(2) | 21(2) | -1(2) |
| C(26) | 72(4) | 105(5) | 94(4) | 4(3) | 52(3) | 20(3) |
| C(27) | 71(4) | 60(4) | 48(3) | 1(3) | 31(3) | -1(3) |
| C(28) | 268(11) | 214(10) | 84(6) | -17(6) | 44(7) | 149(8) |
| C(30) | 44(3) | 45(3) | 39(3) | 2(2) | 11(2) | 3(2) |
| C(31) | 75(4) | 55(4) | 69(3) | 11(3) | 39(3) | 15(3) |
| C(32) | 81(4) | 44(3) | 69(3) | 3(3) | 30(3) | 13(3) |
| C(33) | 54(3) | 48(4) | 61(3) | 14(3) | 7(3) | 0(3) |
| C(34) | 52(3) | 58(4) | 71(3) | 9(3) | 26(3) | -2(3) |
| C(35) | 55(3) | 55(4) | 57(3) | 3(2) | 26(3) | -1(3) |
| C(36) | 52(3) | 50(3) | 40(3) | -5(2) | 18(2) | -5(2) |
| C(37) | 68(3) | 58(4) | 62(3) | -5(3) | 37(3) | -2(3) |
| C(38) | 77(4) | 49(4) | 75(4) | -7(3) | 35(3) | -6(3) |
| C(39) | 82(4) | 60(4) | 81(4) | -10(3) | 42(3) | -14(3) |
| C(40) | 88(4) | 84(5) | 92(4) | -22(4) | 61(4) | -18(4) |
| C(41) | 73(4) | 54(4) | 84(4) | -13(3) | 47(3) | -11(3) |
| C(42) | 136(5) | 86(5) | 130(5) | -5(4) | 86(5) | -42(4) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd24138.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(2) | 5685 | 2804 | 4758 | 62 |
| H(7) | 5851 | 1977 | 2849 | 216 |
| H(8) | 6242 | 776 | 3172 | 216 |
| H(9) | 5081 | 92 | 2972 | 601 |
| H(10) | 5170 | 293 | 2264 | 601 |
| H(11) | 4755 | 1218 | 2497 | 601 |
| H(12) | 7166 | -1221 | 5459 | 72 |
| H(14) | 5535 | -1919 | 6376 | 76 |
| H(17) | 7928 | 5973 | 6669 | 83 |
| H(19) | 8346 | 2112 | 6349 | 80 |
| H(21) | 8857 | 6535 | 6239 | 167 |
| H(20) | 8263 | 6185 | 5449 | 167 |
| H(22) | 9043 | 5604 | 5791 | 167 |
| H(24) | 621 | 2209 | 4778 | 67 |
| H(27) | 107 | 2495 | 5838 | 127 |
| H(26) | 604 | 1929 | 6603 | 127 |
| H(28) | 204 | 1135 | 5909 | 127 |
| H(29) | 1037 | 1419 | 2996 | 244 |
| H(30) | 1144 | 69 | 3179 | 244 |
| H(33) | -28 | -253 | 2631 | 524 |
| H(32) | 82 | 621 | 2104 | 524 |
| H(31) | -214 | 1075 | 2636 | 524 |
| H(34) | 2071 | -1790 | 5411 | 77 |
| H(35) | 1702 | -3662 | 5463 | 80 |
| H(36) | 578 | -2503 | 6478 | 74 |
| H(37) | 942 | -624 | 6420 | 67 |
| H(38) | 2426 | 3983 | 7074 | 72 |
| H(40) | 3772 | 2970 | 5890 | 96 |
| H(41) | 3331 | 1526 | 6368 | 80 |
| H(42) | 3276 | 5461 | 5376 | 162 |
| H(44) | 4081 | 5008 | 5844 | 162 |
| H(43) | 3765 | 5975 | 6160 | 162 |
| H(1) | 6380(20) | 3290(20) | 6140(20) | 67(14) |
| H(3) | 7280(20) | 790(30) | 5900(20) | 56(14) |
| H(4) | 5630(20) | 2440(40) | 6550(20) | 74(16) |
| H(5) | 5230(20) | 3220(40) | 5890(20) | 77(17) |
| H(6) | 5230(20) | 1770(30) | 5850(20) | 80(20) |
| H(13) | 6780(20) | -3170(40) | 5540(20) | 65(14) |
| H(15) | 5940(20) | -10(40) | 6370(20) | 65(14) |
| H(16) | 7440(20) | 4510(40) | 7110(20) | 81(16) |
| H(18) | 8800(20) | 3610(30) | 5890(20) | 56(13) |
| H(23) | 1280(20) | 2710(30) | 6068(19) | 47(13) |
| H(25) | 2276(12) | 250(30) | 5922(18) | 49(12) |
| H(39) | 2870(20) | 5410(40) | 6590(20) | 70(16) |

Table 6. Torsion angles [deg] for cd24138.

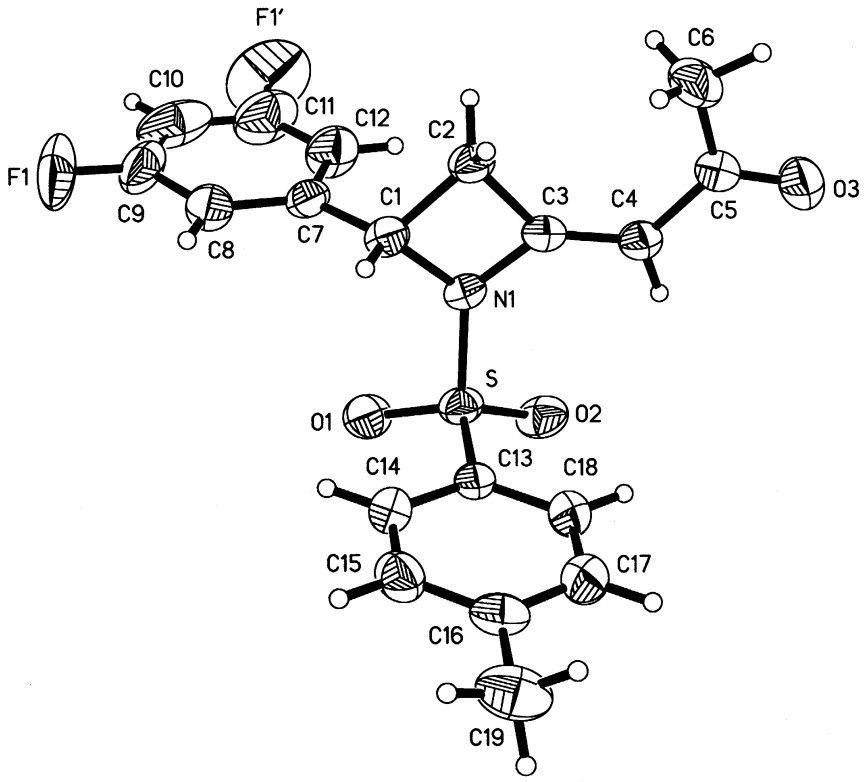
| | |
|---|---|
| O(6)-S(1)-N(1)-C(4) | -35.9(4) |
| O(5)-S(1)-N(1)-C(4) | -165.7(3) |
| C(15)-S(1)-N(1)-C(4) | 80.1(4) |
| O(6)-S(1)-N(1)-C(1) | -174.0(3) |
| O(5)-S(1)-N(1)-C(1) | 56.2(3) |
| C(15)-S(1)-N(1)-C(1) | -58.0(3) |
| O(11)-S(2)-N(3)-C(25) | -167.4(3) |
| O(12)-S(2)-N(3)-C(25) | -38.1(3) |
| C(36)-S(2)-N(3)-C(25) | 78.1(3) |
| O(11)-S(2)-N(3)-C(22) | 55.5(3) |
| O(12)-S(2)-N(3)-C(22) | -175.2(3) |
| C(36)-S(2)-N(3)-C(22) | -59.0(3) |
| C(4)-N(1)-C(1)-C(2) | -8.2(5) |
| S(1)-N(1)-C(1)-C(2) | 132.8(3) |
| C(4)-N(1)-C(1)-C(5) | 113.6(5) |
| S(1)-N(1)-C(1)-C(5) | -105.4(5) |
| N(1)-C(1)-C(2)-C(3) | 5.7(5) |
| C(5)-C(1)-C(2)-C(3) | -116.7(5) |
| C(1)-C(2)-C(3)-C(6) | -178.1(4) |
| C(1)-C(2)-C(3)-C(4) | -1.3(5) |
| C(1)-N(1)-C(4)-C(3) | 7.5(4) |
| S(1)-N(1)-C(4)-C(3) | -132.4(3) |
| C(1)-N(1)-C(4)-C(9) | -115.1(4) |
| S(1)-N(1)-C(4)-C(9) | 105.0(4) |
| C(2)-C(3)-C(4)-N(1) | -3.8(5) |
| C(6)-C(3)-C(4)-N(1) | 173.2(4) |
| C(2)-C(3)-C(4)-C(9) | 117.2(4) |
| C(6)-C(3)-C(4)-C(9) | -65.7(6) |
| C(7)-O(2)-C(6)-O(1) | -3.7(9) |
| C(7)-O(2)-C(6)-C(3) | 173.6(6) |
| C(2)-C(3)-C(6)-O(1) | 171.8(5) |
| C(4)-C(3)-C(6)-O(1) | -4.7(8) |
| C(2)-C(3)-C(6)-O(2) | -5.4(7) |
| C(4)-C(3)-C(6)-O(2) | 178.0(4) |
| C(6)-O(2)-C(7)-C(8) | 107.9(10) |
| N(1)-C(4)-C(9)-C(14) | 22.5(6) |
| C(3)-C(4)-C(9)-C(14) | -91.9(5) |
| N(1)-C(4)-C(9)-C(10) | -157.9(4) |
| C(3)-C(4)-C(9)-C(10) | 87.7(5) |
| C(14)-C(9)-C(10)-C(11) | 1.9(7) |
| C(4)-C(9)-C(10)-C(11) | -177.7(4) |
| C(9)-C(10)-C(11)-C(12) | -0.5(7) |
| C(10)-C(11)-C(12)-C(13) | -0.8(8) |
| C(10)-C(11)-C(12)-N(2) | 177.9(4) |
| O(4)-N(2)-C(12)-C(13) | 176.4(5) |
| O(3)-N(2)-C(12)-C(13) | -4.0(7) |
| O(4)-N(2)-C(12)-C(11) | -2.4(7) |
| O(3)-N(2)-C(12)-C(11) | 177.2(5) |
| C(11)-C(12)-C(13)-C(14) | 0.7(7) |
| N(2)-C(12)-C(13)-C(14) | -178.1(4) |
| C(10)-C(9)-C(14)-C(13) | -2.1(7) |
| C(4)-C(9)-C(14)-C(13) | 177.5(4) |
| C(12)-C(13)-C(14)-C(9) | 0.8(7) |
| O(6)-S(1)-C(15)-C(20) | 36.0(4) |
| O(5)-S(1)-C(15)-C(20) | 168.8(4) |
| N(1)-S(1)-C(15)-C(20) | -78.4(4) |
| O(6)-S(1)-C(15)-C(16) | -145.3(4) |
| O(5)-S(1)-C(15)-C(16) | -12.4(4) |
| N(1)-S(1)-C(15)-C(16) | 100.4(4) |
| C(20)-C(15)-C(16)-C(17) | 3.3(7) |
| S(1)-C(15)-C(16)-C(17) | -175.5(4) |
| C(15)-C(16)-C(17)-C(18) | -0.5(7) |
| C(16)-C(17)-C(18)-C(19) | -2.6(8) |
| C(16)-C(17)-C(18)-C(21) | 177.8(5) |

| | |
|---|---|
| C(17)-C(18)-C(19)-C(20) | 2.9(8) |
| C(21)-C(18)-C(19)-C(20) | -177.4(5) |
| C(16)-C(15)-C(20)-C(19) | -2.9(7) |
| S(1)-C(15)-C(20)-C(19) | 175.8(4) |
| C(18)-C(19)-C(20)-C(15) | -0.3(8) |
| C(25)-N(3)-C(22)-C(23) | -8.0(5) |
| S(2)-N(3)-C(22)-C(23) | 132.2(3) |
| C(25)-N(3)-C(22)-C(26) | 114.7(4) |
| S(2)-N(3)-C(22)-C(26) | -105.1(4) |
| N(3)-C(22)-C(23)-C(24) | 6.1(5) |
| C(26)-C(22)-C(23)-C(24) | -116.2(5) |
| C(22)-C(23)-C(24)-C(27) | -177.7(4) |
| C(22)-C(23)-C(24)-C(25) | -1.9(6) |
| C(22)-N(3)-C(25)-C(24) | 7.1(4) |
| S(2)-N(3)-C(25)-C(24) | -131.9(3) |
| C(22)-N(3)-C(25)-C(30) | -115.1(4) |
| S(2)-N(3)-C(25)-C(30) | 105.9(4) |
| C(23)-C(24)-C(25)-N(3) | -3.2(5) |
| C(27)-C(24)-C(25)-N(3) | 172.8(4) |
| C(23)-C(24)-C(25)-C(30) | 117.0(4) |
| C(27)-C(24)-C(25)-C(30) | -66.9(5) |
| C(28)-O(8)-C(27)-O(7) | -12.4(8) |
| C(28)-O(8)-C(27)-C(24) | 165.5(5) |
| C(23)-C(24)-C(27)-O(7) | 170.1(5) |
| C(25)-C(24)-C(27)-O(7) | -5.3(7) |
| C(23)-C(24)-C(27)-O(8) | -7.8(7) |
| C(25)-C(24)-C(27)-O(8) | 176.8(4) |
| C(27)-O(8)-C(28)-C(29) | 134.0(8) |
| N(3)-C(25)-C(30)-C(35) | 20.1(5) |
| C(24)-C(25)-C(30)-C(35) | -94.5(5) |
| N(3)-C(25)-C(30)-C(31) | -161.5(4) |
| C(24)-C(25)-C(30)-C(31) | 83.9(5) |
| C(35)-C(30)-C(31)-C(32) | 1.8(6) |
| C(25)-C(30)-C(31)-C(32) | -176.7(4) |
| C(30)-C(31)-C(32)-C(33) | 0.1(7) |
| C(31)-C(32)-C(33)-C(34) | -2.2(7) |
| C(31)-C(32)-C(33)-N(4) | 177.2(4) |
| O(9)-N(4)-C(33)-C(34) | -2.1(7) |
| O(10)-N(4)-C(33)-C(34) | 176.8(5) |
| O(9)-N(4)-C(33)-C(32) | 178.4(5) |
| O(10)-N(4)-C(33)-C(32) | -2.7(7) |
| C(32)-C(33)-C(34)-C(35) | 2.4(7) |
| N(4)-C(33)-C(34)-C(35) | -177.0(4) |
| C(33)-C(34)-C(35)-C(30) | -0.4(7) |
| C(31)-C(30)-C(35)-C(34) | -1.6(6) |
| C(25)-C(30)-C(35)-C(34) | 176.8(4) |
| O(11)-S(2)-C(36)-C(37) | -14.4(4) |
| O(12)-S(2)-C(36)-C(37) | -146.8(4) |
| N(3)-S(2)-C(36)-C(37) | 99.0(4) |
| O(11)-S(2)-C(36)-C(41) | 166.3(3) |
| O(12)-S(2)-C(36)-C(41) | 33.9(4) |
| N(3)-S(2)-C(36)-C(41) | -80.3(4) |
| C(41)-C(36)-C(37)-C(38) | 2.8(7) |
| S(2)-C(36)-C(37)-C(38) | -176.5(4) |
| C(36)-C(37)-C(38)-C(39) | -0.5(8) |
| C(37)-C(38)-C(39)-C(40) | -1.1(8) |
| C(37)-C(38)-C(39)-C(42) | 177.6(5) |
| C(38)-C(39)-C(40)-C(41) | 0.6(8) |
| C(42)-C(39)-C(40)-C(41) | -178.2(5) |
| C(37)-C(36)-C(41)-C(40) | -3.3(7) |
| S(2)-C(36)-C(41)-C(40) | 176.0(4) |
| C(39)-C(40)-C(41)-C(36) | 1.5(8) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd24138 [A and deg.].

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




The crystal data of **5d** has been deposited in CCDC with number 211895. Empirical Formula: $C_{19}H_{18}NO_3FS$; Formula Weight: 359.40; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.478 x 0.237 x 0.226 mm; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 6.3576(9)Å, b = 8.9534(13)Å, c = 31.589(5)Å, $\alpha = 90^o$, $\beta = 91.829(3)^o$, $\gamma = 90^o$, V = 1797.2(4)Å$^3$; Space group: P2(1)/n; Z = 4; $D_{calc}$ = 1.328 g/cm$^3$; $F_{000}$ = 752; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0568, 0.1278.

Table 1. Crystal data and structure refinement for cd23228.

| | |
|---|---|
| Identification code | cd23228 |
| Empirical formula | C19 H18 N O3 F S |
| Formula weight | 359.40 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/n |
| Unit cell dimensions | a = 6.3576(9) A alpha = 90 deg.<br>b = 8.9534(13) A beta = 91.829(3) deg.<br>c = 31.589(5) A gamma = 90 deg. |
| Volume | 1797.2(4) A^3 |
| Z, Calculated density | 4, 1.328 Mg/m^3 |
| Absorption coefficient | 0.207 mm^-1 |
| F(000) | 752 |
| Crystal size | 0.478 x 0.237 x 0.226 mm |
| Theta range for data collection | 1.29 to 28.27 deg. |
| Limiting indices | -5<=h<=8, -11<=k<=11, -41<=l<=42 |
| Reflections collected / unique | 10360 / 4132 [R(int) = 0.0711] |
| Completeness to theta = 28.27 | 92.6 % |
| Absorption correction | Sadabs |
| Max. and min. transmission | 1.00000 and 0.55366 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 4132 / 5 / 260 |
| Goodness-of-fit on F^2 | 0.837 |
| Final R indices [I>2sigma(I)] | R1 = 0.0568, wR2 = 0.1278 |
| R indices (all data) | R1 = 0.1350, wR2 = 0.1544 |
| Largest diff. peak and hole | 0.299 and -0.211 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd23228.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S | -1003(1) | 3203(1) | 853(1) | 61(1) |
| F(1) | 6209(8) | -1736(6) | 1249(2) | 136(2) |
| F(1') | 569(11) | -2582(7) | 1828(2) | 196(3) |
| N(1) | -162(4) | 2999(3) | 1355(1) | 57(1) |
| O(1) | -126(4) | 1959(2) | 639(1) | 83(1) |
| O(2) | -3233(3) | 3428(3) | 869(1) | 77(1) |
| O(3) | -4417(4) | 5899(3) | 2277(1) | 101(1) |
| C(1) | 2003(5) | 2572(4) | 1507(1) | 58(1) |
| C(2) | 1469(5) | 3342(4) | 1932(1) | 59(1) |
| C(3) | -626(5) | 3808(3) | 1724(1) | 53(1) |
| C(4) | -2343(5) | 4592(3) | 1822(1) | 61(1) |
| C(5) | -2694(6) | 5315(4) | 2221(1) | 71(1) |
| C(6) | -989(8) | 5353(7) | 2557(1) | 97(1) |
| C(7) | 2449(5) | 939(4) | 1506(1) | 60(1) |
| C(8) | 4291(6) | 388(4) | 1352(1) | 79(1) |
| C(9) | 4653(10) | -1177(6) | 1375(1) | 116(2) |
| C(10) | 3204(12) | -2117(6) | 1543(2) | 133(3) |
| C(11) | 1451(11) | -1540(6) | 1693(2) | 130(2) |
| C(12) | 1036(6) | -60(4) | 1677(1) | 92(1) |
| C(13) | 156(5) | 4844(3) | 674(1) | 54(1) |
| C(14) | 2118(6) | 4783(4) | 502(1) | 70(1) |
| C(15) | 3038(5) | 6090(5) | 363(1) | 78(1) |
| C(16) | 2008(6) | 7459(4) | 391(1) | 72(1) |
| C(17) | 53(6) | 7494(4) | 568(1) | 76(1) |
| C(18) | -859(5) | 6202(4) | 707(1) | 67(1) |
| C(19) | 3040(6) | 8871(4) | 244(1) | 105(1) |

Table 3. Bond lengths [A] and angles [deg] for cd23228.

| | |
|---|---|
| S-O(1) | 1.427(2) |
| S-O(2) | 1.434(2) |
| S-N(1) | 1.665(2) |
| S-C(13) | 1.747(3) |
| F(1)-C(9) | 1.188(5) |
| F(1')-C(11) | 1.176(7) |
| N(1)-C(3) | 1.413(3) |
| N(1)-C(1) | 1.493(3) |
| O(3)-C(5) | 1.232(3) |
| C(1)-C(7) | 1.489(4) |
| C(1)-C(2) | 1.558(4) |
| C(1)-H(1) | 0.912(15) |
| C(2)-C(3) | 1.525(4) |
| C(2)-H(2) | 0.97(2) |
| C(2)-H(3) | 0.995(17) |
| C(3)-C(4) | 1.342(4) |
| C(4)-C(5) | 1.440(4) |
| C(4)-H(4) | 0.9300 |
| C(5)-C(6) | 1.494(5) |
| C(6)-H(5) | 0.957(18) |
| C(6)-H(6) | 0.988(19) |
| C(6)-H(7) | 0.949(18) |
| C(7)-C(8) | 1.374(4) |
| C(7)-C(12) | 1.389(4) |
| C(8)-C(9) | 1.421(6) |
| C(8)-H(8) | 0.9300 |
| C(9)-C(10) | 1.367(6) |
| C(10)-C(11) | 1.330(7) |
| C(10)-H(9) | 0.9300 |
| C(11)-C(12) | 1.351(6) |
| C(12)-H(10) | 0.9300 |
| C(13)-C(14) | 1.378(4) |
| C(13)-C(18) | 1.382(4) |
| C(14)-C(15) | 1.386(4) |
| C(14)-H(11) | 0.9300 |
| C(15)-C(16) | 1.394(4) |
| C(15)-H(12) | 0.9300 |
| C(16)-C(17) | 1.379(4) |
| C(16)-C(19) | 1.505(4) |
| C(17)-C(18) | 1.372(4) |
| C(17)-H(13) | 0.9300 |
| C(18)-H(14) | 0.9300 |
| C(19)-H(16) | 0.9600 |
| C(19)-H(15) | 0.9600 |
| C(19)-H(17) | 0.9600 |
| | |
| O(1)-S-O(2) | 121.85(15) |
| O(1)-S-N(1) | 104.35(13) |
| O(2)-S-N(1) | 105.75(12) |
| O(1)-S-C(13) | 109.07(14) |
| O(2)-S-C(13) | 108.70(14) |
| N(1)-S-C(13) | 105.94(13) |
| C(3)-N(1)-C(1) | 94.6(2) |
| C(3)-N(1)-S | 131.3(2) |
| C(1)-N(1)-S | 126.84(19) |
| C(7)-C(1)-N(1) | 115.1(3) |
| C(7)-C(1)-C(2) | 118.9(3) |
| N(1)-C(1)-C(2) | 86.6(2) |
| C(7)-C(1)-H(1) | 114.8(16) |
| N(1)-C(1)-H(1) | 112.6(16) |
| C(2)-C(1)-H(1) | 105.4(15) |
| C(3)-C(2)-C(1) | 87.7(2) |
| C(3)-C(2)-H(2) | 111.4(15) |
| C(1)-C(2)-H(2) | 115.1(15) |

| | |
|---|---|
| C(3)-C(2)-H(3) | 114.1(16) |
| C(1)-C(2)-H(3) | 118.5(15) |
| H(2)-C(2)-H(3) | 109(2) |
| C(4)-C(3)-N(1) | 130.6(3) |
| C(4)-C(3)-C(2) | 138.6(3) |
| N(1)-C(3)-C(2) | 90.8(2) |
| C(3)-C(4)-C(5) | 125.9(3) |
| C(3)-C(4)-H(4) | 117.1 |
| C(5)-C(4)-H(4) | 117.1 |
| O(3)-C(5)-C(4) | 118.8(3) |
| O(3)-C(5)-C(6) | 121.0(3) |
| C(4)-C(5)-C(6) | 120.2(3) |
| C(5)-C(6)-H(5) | 115(2) |
| C(5)-C(6)-H(6) | 103(3) |
| H(5)-C(6)-H(6) | 112(4) |
| C(5)-C(6)-H(7) | 104(2) |
| H(5)-C(6)-H(7) | 111(3) |
| H(6)-C(6)-H(7) | 111(3) |
| C(8)-C(7)-C(12) | 118.3(3) |
| C(8)-C(7)-C(1) | 121.2(3) |
| C(12)-C(7)-C(1) | 120.4(3) |
| C(7)-C(8)-C(9) | 118.4(4) |
| C(7)-C(8)-H(8) | 120.8 |
| C(9)-C(8)-H(8) | 120.8 |
| F(1)-C(9)-C(10) | 116.7(6) |
| F(1)-C(9)-C(8) | 122.2(7) |
| C(10)-C(9)-C(8) | 121.1(5) |
| C(11)-C(10)-C(9) | 118.8(6) |
| C(11)-C(10)-H(9) | 120.6 |
| C(9)-C(10)-H(9) | 120.6 |
| F(1')-C(11)-C(10) | 103.6(7) |
| F(1')-C(11)-C(12) | 134.2(8) |
| C(10)-C(11)-C(12) | 122.2(6) |
| C(11)-C(12)-C(7) | 121.2(5) |
| C(11)-C(12)-H(10) | 119.4 |
| C(7)-C(12)-H(10) | 119.4 |
| C(14)-C(13)-C(18) | 119.7(3) |
| C(14)-C(13)-S | 119.4(3) |
| C(18)-C(13)-S | 120.9(3) |
| C(13)-C(14)-C(15) | 119.2(3) |
| C(13)-C(14)-H(11) | 120.4 |
| C(15)-C(14)-H(11) | 120.4 |
| C(14)-C(15)-C(16) | 121.2(3) |
| C(14)-C(15)-H(12) | 119.4 |
| C(16)-C(15)-H(12) | 119.4 |
| C(17)-C(16)-C(15) | 118.5(3) |
| C(17)-C(16)-C(19) | 120.9(4) |
| C(15)-C(16)-C(19) | 120.5(4) |
| C(18)-C(17)-C(16) | 120.3(3) |
| C(18)-C(17)-H(13) | 119.8 |
| C(16)-C(17)-H(13) | 119.8 |
| C(17)-C(18)-C(13) | 121.0(3) |
| C(17)-C(18)-H(14) | 119.5 |
| C(13)-C(18)-H(14) | 119.5 |
| C(16)-C(19)-H(16) | 109.5 |
| C(16)-C(19)-H(15) | 109.5 |
| H(16)-C(19)-H(15) | 109.5 |
| C(16)-C(19)-H(17) | 109.5 |
| H(16)-C(19)-H(17) | 109.5 |
| H(15)-C(19)-H(17) | 109.5 |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd23228.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S | 70(1) | 64(1) | 48(1) | 2(1) | -11(1) | 4(1) |
| F(1) | 103(4) | 125(4) | 180(5) | -46(4) | 14(3) | 59(3) |
| F(1') | 218(7) | 96(5) | 274(8) | 71(5) | 4(6) | -64(4) |
| N(1) | 58(2) | 62(2) | 49(2) | 7(1) | -7(1) | 8(1) |
| O(1) | 122(2) | 64(2) | 62(1) | -11(1) | -7(1) | 16(1) |
| O(2) | 57(2) | 98(2) | 73(2) | 15(1) | -16(1) | -6(1) |
| O(3) | 94(2) | 131(2) | 77(2) | -13(2) | 5(2) | 40(2) |
| C(1) | 50(2) | 63(2) | 60(2) | 12(2) | -1(2) | -3(2) |
| C(2) | 62(2) | 63(2) | 51(2) | 1(2) | -11(2) | 2(2) |
| C(3) | 62(2) | 52(2) | 44(2) | 4(1) | -3(2) | -5(2) |
| C(4) | 60(2) | 72(2) | 51(2) | 2(2) | -5(2) | 5(2) |
| C(5) | 81(3) | 79(3) | 53(2) | 7(2) | 7(2) | 15(2) |
| C(6) | 96(4) | 134(5) | 58(3) | -27(3) | -11(3) | 16(3) |
| C(7) | 67(2) | 59(2) | 54(2) | 5(2) | -9(2) | 9(2) |
| C(8) | 82(3) | 88(3) | 66(2) | -7(2) | -5(2) | 13(2) |
| C(9) | 145(5) | 110(4) | 90(3) | -38(3) | -39(3) | 79(4) |
| C(10) | 227(8) | 56(3) | 111(4) | 4(3) | -69(5) | 1(4) |
| C(11) | 179(6) | 77(4) | 132(4) | 21(3) | -18(4) | 15(4) |
| C(12) | 103(3) | 63(3) | 109(3) | 22(2) | 12(2) | -2(2) |
| C(13) | 55(2) | 63(2) | 43(2) | 3(1) | -3(2) | 7(2) |
| C(14) | 71(3) | 73(3) | 67(2) | 5(2) | 7(2) | 18(2) |
| C(15) | 61(2) | 105(3) | 68(2) | 4(2) | 11(2) | 4(2) |
| C(16) | 89(3) | 77(3) | 48(2) | 6(2) | -7(2) | -15(2) |
| C(17) | 84(3) | 68(3) | 77(2) | 5(2) | 12(2) | 9(2) |
| C(18) | 65(2) | 70(2) | 66(2) | 4(2) | 13(2) | 9(2) |
| C(19) | 125(4) | 110(4) | 80(3) | 8(2) | 3(2) | -40(3) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd23228.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(4) | -3401 | 4676 | 1613 | 73 |
| H(8) | 5276 | 1022 | 1236 | 94 |
| H(9) | 3439 | -3142 | 1551 | 160 |
| H(10) | -217 | 296 | 1783 | 110 |
| H(11) | 2816 | 3875 | 479 | 84 |
| H(12) | 4367 | 6054 | 249 | 94 |
| H(13) | -651 | 8399 | 594 | 91 |
| H(14) | -2180 | 6241 | 825 | 80 |
| H(16) | 2418 | 9168 | -24 | 158 |
| H(15) | 4518 | 8698 | 213 | 158 |
| H(17) | 2844 | 9648 | 448 | 158 |
| H(1) | 3020(30) | 3150(20) | 1393(7) | 46(8) |
| H(2) | 1270(40) | 2670(30) | 2168(8) | 48(8) |
| H(3) | 2360(40) | 4190(20) | 2033(8) | 62(9) |
| H(5) | 350(40) | 5690(40) | 2466(11) | 109(16) |
| H(6) | -950(70) | 4310(30) | 2656(14) | 160(20) |
| H(7) | -1520(50) | 6000(30) | 2766(9) | 102(13) |

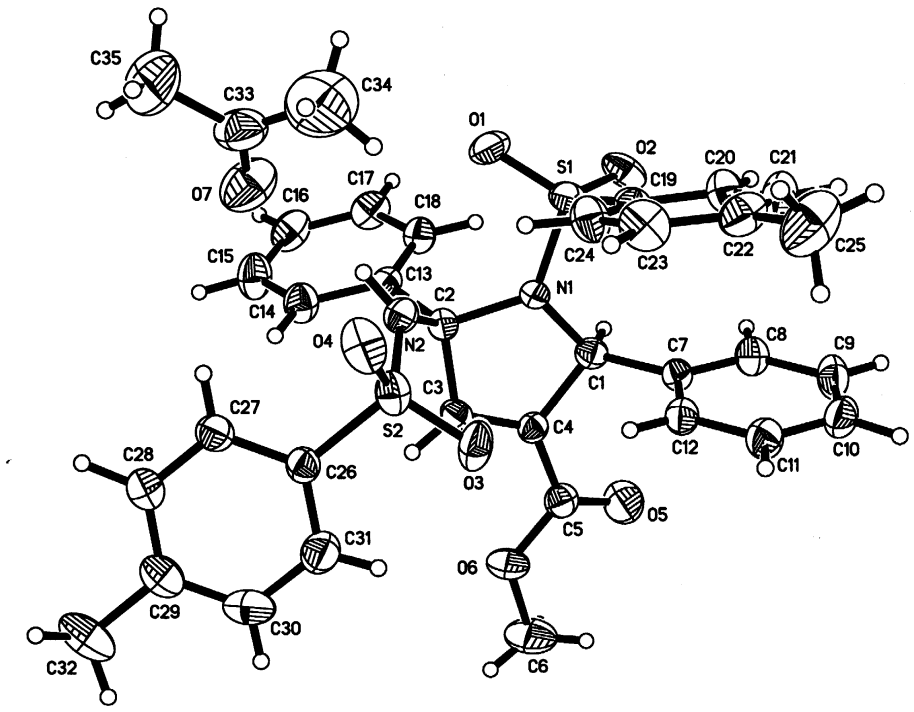
Table 6. Torsion angles [deg] for cd23228.

| | |
|---|---|
| O(1)-S-N(1)-C(3) | -174.6(3) |
| O(2)-S-N(1)-C(3) | -45.0(3) |
| C(13)-S-N(1)-C(3) | 70.3(3) |
| O(1)-S-N(1)-C(1) | 43.2(3) |
| O(2)-S-N(1)-C(1) | 172.8(2) |
| C(13)-S-N(1)-C(1) | -71.9(3) |
| C(3)-N(1)-C(1)-C(7) | 124.9(3) |
| S-N(1)-C(1)-C(7) | -82.6(3) |
| C(3)-N(1)-C(1)-C(2) | 4.4(2) |
| S-N(1)-C(1)-C(2) | 156.9(2) |
| C(7)-C(1)-C(2)-C(3) | -121.0(3) |
| N(1)-C(1)-C(2)-C(3) | -4.1(2) |
| C(1)-N(1)-C(3)-C(4) | 177.7(3) |
| S-N(1)-C(3)-C(4) | 27.1(5) |
| C(1)-N(1)-C(3)-C(2) | -4.5(2) |
| S-N(1)-C(3)-C(2) | -155.0(2) |
| C(1)-C(2)-C(3)-C(4) | -178.2(4) |
| C(1)-C(2)-C(3)-N(1) | 4.3(2) |
| N(1)-C(3)-C(4)-C(5) | 176.5(3) |
| C(2)-C(3)-C(4)-C(5) | -0.3(6) |
| C(3)-C(4)-C(5)-O(3) | -174.4(3) |
| C(3)-C(4)-C(5)-C(6) | 5.8(6) |
| N(1)-C(1)-C(7)-C(8) | 134.8(3) |
| C(2)-C(1)-C(7)-C(8) | -124.6(3) |
| N(1)-C(1)-C(7)-C(12) | -47.8(4) |
| C(2)-C(1)-C(7)-C(12) | 52.8(4) |
| C(12)-C(7)-C(8)-C(9) | 0.2(5) |
| C(1)-C(7)-C(8)-C(9) | 177.6(3) |
| C(7)-C(8)-C(9)-F(1) | -180.0(4) |
| C(7)-C(8)-C(9)-C(10) | 0.5(6) |
| F(1)-C(9)-C(10)-C(11) | 179.1(5) |
| C(8)-C(9)-C(10)-C(11) | -1.3(7) |
| C(9)-C(10)-C(11)-F(1') | -177.4(6) |
| C(9)-C(10)-C(11)-C(12) | 1.5(9) |
| F(1')-C(11)-C(12)-C(7) | 177.6(8) |
| C(10)-C(11)-C(12)-C(7) | -0.8(8) |
| C(8)-C(7)-C(12)-C(11) | -0.1(6) |
| C(1)-C(7)-C(12)-C(11) | -177.5(4) |
| O(1)-S-C(13)-C(14) | -23.9(3) |
| O(2)-S-C(13)-C(14) | -158.9(2) |
| N(1)-S-C(13)-C(14) | 87.9(3) |
| O(1)-S-C(13)-C(18) | 156.7(2) |
| O(2)-S-C(13)-C(18) | 21.8(3) |
| N(1)-S-C(13)-C(18) | -91.5(3) |
| C(18)-C(13)-C(14)-C(15) | -0.1(5) |
| S-C(13)-C(14)-C(15) | -179.5(2) |
| C(13)-C(14)-C(15)-C(16) | -0.6(5) |
| C(14)-C(15)-C(16)-C(17) | 1.1(5) |
| C(14)-C(15)-C(16)-C(19) | 179.3(3) |
| C(15)-C(16)-C(17)-C(18) | -0.9(5) |
| C(19)-C(16)-C(17)-C(18) | -179.1(3) |
| C(16)-C(17)-C(18)-C(13) | 0.2(5) |
| C(14)-C(13)-C(18)-C(17) | 0.3(5) |
| S-C(13)-C(18)-C(17) | 179.7(2) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23228 [A and deg.].

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




The crystal data of **8** has been deposited in CCDC with number 216649. Empirical Formula: $C_{35}H_{36}N_2O_7S_2$; Formula Weight: 660.78; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.439 x 0.262 x 0.206 mm; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 11.5923(8)Å, b = 17.0433(12)Å, c = 17.9971(13)Å, $\alpha = 90^o$, $\beta = 108.321(2)^o$, $\gamma = 90^o$, V = 3375.5(4)Å$^3$; Space group: P2(1)/n; Z = 4; $D_{calc}$ = 1.300 g/cm$^3$; $F_{000}$ = 1392; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0469, 0.0806.

Table 1. Crystal data and structure refinement for cd23138.

| | |
|---|---|
| Identification code | cd23138 |
| Empirical formula | C35 H36 N2 O7 S2 |
| Formula weight | 660.78 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/n |
| Unit cell dimensions | a = 11.5923(8) A alpha = 90 deg.<br>b = 17.0433(12) A beta = 108.321(2) deg.<br>c = 17.9971(13) A gamma = 90 deg. |
| Volume | 3375.5(4) A^3 |
| Z, Calculated density | 4, 1.300 Mg/m^3 |
| Absorption coefficient | 0.208 mm^-1 |
| F(000) | 1392 |
| Crystal size | 0.439 x 0.262 x 0.206 mm |
| Theta range for data collection | 1.86 to 28.32 deg. |
| Limiting indices | -8<=h<=15, -21<=k<=21, -22<=l<=23 |
| Reflections collected / unique | 20523 / 7909 [R(int) = 0.0592] |
| Completeness to theta = 28.32 | 94.1 % |
| Absorption correction | Sadabs |
| Max. and min. transmission | 1.00000 and 0.60909 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 7909 / 0 / 515 |
| Goodness-of-fit on F^2 | 0.748 |
| Final R indices [I>2sigma(I)] | R1 = 0.0469, wR2 = 0.0806 |
| R indices (all data) | R1 = 0.1190, wR2 = 0.0934 |
| Largest diff. peak and hole | 0.209 and -0.261 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd23138.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 10303(1) | 1947(1) | 610(1) | 49(1) |
| S(2) | 7898(1) | 3672(1) | 1226(1) | 55(1) |
| O(1) | 9237(2) | 1505(1) | 559(1) | 63(1) |
| O(2) | 11429(2) | 1557(1) | 687(1) | 64(1) |
| O(3) | 8830(2) | 4197(1) | 1182(1) | 70(1) |
| O(4) | 6798(2) | 3596(1) | 586(1) | 73(1) |
| O(5) | 13445(2) | 3810(1) | 2970(1) | 90(1) |
| O(6) | 11958(2) | 4247(1) | 3396(1) | 66(1) |
| O(7) | 6221(2) | 1771(1) | 1003(2) | 109(1) |
| N(1) | 10526(2) | 2519(1) | 1366(1) | 40(1) |
| N(2) | 8465(2) | 2795(1) | 1376(1) | 47(1) |
| C(1) | 11722(2) | 2898(1) | 1738(1) | 44(1) |
| C(2) | 9692(2) | 2584(1) | 1844(1) | 40(1) |
| C(3) | 10362(2) | 3195(1) | 2417(1) | 43(1) |
| C(4) | 11456(2) | 3334(1) | 2391(1) | 40(1) |
| C(5) | 12399(2) | 3814(1) | 2936(1) | 52(1) |
| C(6) | 12834(5) | 4735(3) | 3963(3) | 89(1) |
| C(7) | 12156(2) | 3421(1) | 1197(1) | 43(1) |
| C(8) | 13247(2) | 3272(2) | 1070(2) | 56(1) |
| C(9) | 13643(3) | 3757(2) | 580(2) | 66(1) |
| C(10) | 12953(3) | 4377(2) | 218(2) | 65(1) |
| C(11) | 11880(3) | 4535(2) | 350(2) | 62(1) |
| C(12) | 11480(3) | 4059(1) | 834(2) | 53(1) |
| C(13) | 9644(2) | 1849(1) | 2328(1) | 41(1) |
| C(14) | 8855(2) | 1841(2) | 2770(2) | 60(1) |
| C(15) | 8879(3) | 1242(2) | 3286(2) | 74(1) |
| C(16) | 9695(3) | 640(2) | 3371(2) | 68(1) |
| C(17) | 10469(3) | 633(2) | 2937(2) | 63(1) |
| C(18) | 10454(2) | 1239(1) | 2422(2) | 53(1) |
| C(19) | 9978(2) | 2545(1) | -225(1) | 45(1) |
| C(20) | 10805(3) | 2635(2) | -620(2) | 61(1) |
| C(21) | 10521(3) | 3113(2) | -1268(2) | 74(1) |
| C(22) | 9436(3) | 3500(2) | -1538(2) | 68(1) |
| C(23) | 8621(3) | 3397(2) | -1139(2) | 76(1) |
| C(24) | 8878(3) | 2926(2) | -490(2) | 63(1) |
| C(25) | 9159(3) | 4035(2) | -2243(2) | 113(1) |
| C(26) | 7497(2) | 3940(1) | 2053(1) | 47(1) |
| C(27) | 6567(2) | 3548(2) | 2221(2) | 54(1) |
| C(28) | 6212(3) | 3771(2) | 2851(2) | 61(1) |
| C(29) | 6768(3) | 4378(2) | 3325(2) | 63(1) |
| C(30) | 7703(3) | 4761(2) | 3160(2) | 78(1) |
| C(31) | 8076(3) | 4545(2) | 2525(2) | 70(1) |
| C(32) | 6348(3) | 4642(2) | 3995(2) | 95(1) |
| C(33) | 5496(3) | 1649(2) | 378(2) | 88(1) |
| C(34) | 5748(4) | 1840(3) | -334(3) | 193(2) |
| C(35) | 4352(3) | 1292(3) | 310(3) | 224(3) |

Table 3. Bond lengths [A] and angles [deg] for cd23138.

| | |
|---|---|
| S(1)-O(1) | 1.4258(14) |
| S(1)-O(2) | 1.4311(14) |
| S(1)-N(1) | 1.6270(17) |
| S(1)-C(19) | 1.757(2) |
| S(2)-O(3) | 1.4246(15) |
| S(2)-O(4) | 1.4308(17) |
| S(2)-N(2) | 1.6206(19) |
| S(2)-C(26) | 1.754(2) |
| O(5)-C(5) | 1.195(2) |
| O(6)-C(5) | 1.326(3) |
| O(6)-C(6) | 1.454(4) |
| O(7)-C(33) | 1.191(3) |
| N(1)-C(1) | 1.485(3) |
| N(1)-C(2) | 1.488(2) |
| N(2)-C(2) | 1.452(3) |
| C(1)-C(4) | 1.501(3) |
| C(1)-C(7) | 1.518(3) |
| C(2)-C(3) | 1.499(3) |
| C(2)-C(13) | 1.536(3) |
| C(3)-C(4) | 1.306(3) |
| C(4)-C(5) | 1.467(3) |
| C(7)-C(8) | 1.377(3) |
| C(7)-C(12) | 1.379(3) |
| C(8)-C(9) | 1.387(3) |
| C(9)-C(10) | 1.362(4) |
| C(10)-C(11) | 1.365(3) |
| C(11)-C(12) | 1.374(3) |
| C(13)-C(18) | 1.376(3) |
| C(13)-C(14) | 1.388(3) |
| C(14)-C(15) | 1.373(3) |
| C(15)-C(16) | 1.372(4) |
| C(16)-C(17) | 1.362(3) |
| C(17)-C(18) | 1.384(3) |
| C(19)-C(20) | 1.371(3) |
| C(19)-C(24) | 1.375(3) |
| C(20)-C(21) | 1.374(4) |
| C(21)-C(22) | 1.368(4) |
| C(22)-C(23) | 1.366(4) |
| C(22)-C(25) | 1.512(3) |
| C(23)-C(24) | 1.371(4) |
| C(26)-C(31) | 1.370(3) |
| C(26)-C(27) | 1.379(3) |
| C(27)-C(28) | 1.376(3) |
| C(28)-C(29) | 1.365(3) |
| C(29)-C(30) | 1.376(4) |
| C(29)-C(32) | 1.504(3) |
| C(30)-C(31) | 1.391(4) |
| C(33)-C(35) | 1.428(4) |
| C(33)-C(34) | 1.439(5) |

| | |
|---|---|
| O(1)-S(1)-O(2) | 120.50(10) |
| O(1)-S(1)-N(1) | 106.09(9) |
| O(2)-S(1)-N(1) | 107.30(10) |
| O(1)-S(1)-C(19) | 107.61(11) |
| O(2)-S(1)-C(19) | 107.22(11) |
| N(1)-S(1)-C(19) | 107.53(9) |
| O(3)-S(2)-O(4) | 120.73(11) |
| O(3)-S(2)-N(2) | 108.31(10) |
| O(4)-S(2)-N(2) | 105.26(11) |
| O(3)-S(2)-C(26) | 106.96(12) |
| O(4)-S(2)-C(26) | 107.46(11) |
| N(2)-S(2)-C(26) | 107.52(10) |
| C(5)-O(6)-C(6) | 115.8(3) |
| C(1)-N(1)-C(2) | 113.03(17) |

| | |
|---|---|
| C(1)-N(1)-S(1) | 121.22(14) |
| C(2)-N(1)-S(1) | 124.54(14) |
| C(2)-N(2)-S(2) | 126.68(17) |
| N(1)-C(1)-C(4) | 100.14(17) |
| N(1)-C(1)-C(7) | 114.59(19) |
| C(4)-C(1)-C(7) | 113.79(18) |
| N(2)-C(2)-N(1) | 112.20(19) |
| N(2)-C(2)-C(3) | 115.32(18) |
| N(1)-C(2)-C(3) | 99.09(17) |
| N(2)-C(2)-C(13) | 109.16(17) |
| N(1)-C(2)-C(13) | 114.46(17) |
| C(3)-C(2)-C(13) | 106.33(18) |
| C(4)-C(3)-C(2) | 113.7(2) |
| C(3)-C(4)-C(5) | 127.3(2) |
| C(3)-C(4)-C(1) | 112.2(2) |
| C(5)-C(4)-C(1) | 120.4(2) |
| O(5)-C(5)-O(6) | 123.8(2) |
| O(5)-C(5)-C(4) | 124.2(2) |
| O(6)-C(5)-C(4) | 112.0(2) |
| C(8)-C(7)-C(12) | 118.8(2) |
| C(8)-C(7)-C(1) | 120.4(2) |
| C(12)-C(7)-C(1) | 120.8(2) |
| C(7)-C(8)-C(9) | 120.0(3) |
| C(10)-C(9)-C(8) | 120.3(3) |
| C(9)-C(10)-C(11) | 120.0(3) |
| C(10)-C(11)-C(12) | 120.1(3) |
| C(11)-C(12)-C(7) | 120.7(3) |
| C(18)-C(13)-C(14) | 118.0(2) |
| C(18)-C(13)-C(2) | 122.7(2) |
| C(14)-C(13)-C(2) | 118.7(2) |
| C(15)-C(14)-C(13) | 121.2(3) |
| C(16)-C(15)-C(14) | 119.9(3) |
| C(17)-C(16)-C(15) | 119.8(3) |
| C(16)-C(17)-C(18) | 120.4(3) |
| C(13)-C(18)-C(17) | 120.7(2) |
| C(20)-C(19)-C(24) | 119.4(3) |
| C(20)-C(19)-S(1) | 120.8(2) |
| C(24)-C(19)-S(1) | 119.80(19) |
| C(19)-C(20)-C(21) | 119.1(3) |
| C(22)-C(21)-C(20) | 122.4(3) |
| C(23)-C(22)-C(21) | 117.5(3) |
| C(23)-C(22)-C(25) | 121.3(3) |
| C(21)-C(22)-C(25) | 121.2(3) |
| C(22)-C(23)-C(24) | 121.5(3) |
| C(23)-C(24)-C(19) | 120.0(3) |
| C(31)-C(26)-C(27) | 119.5(2) |
| C(31)-C(26)-S(2) | 120.9(2) |
| C(27)-C(26)-S(2) | 119.6(2) |
| C(28)-C(27)-C(26) | 120.2(3) |
| C(29)-C(28)-C(27) | 121.3(3) |
| C(28)-C(29)-C(30) | 118.3(3) |
| C(28)-C(29)-C(32) | 121.2(3) |
| C(30)-C(29)-C(32) | 120.5(3) |
| C(29)-C(30)-C(31) | 121.3(3) |
| C(26)-C(31)-C(30) | 119.4(3) |
| O(7)-C(33)-C(35) | 121.1(4) |
| O(7)-C(33)-C(34) | 121.3(4) |
| C(35)-C(33)-C(34) | 117.6(4) |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd23138.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 66(1) | 40(1) | 42(1) | -4(1) | 19(1) | 0(1) |
| S(2) | 52(1) | 61(1) | 57(1) | 17(1) | 23(1) | 12(1) |
| O(1) | 81(1) | 52(1) | 55(1) | -8(1) | 19(1) | -28(1) |
| O(2) | 84(1) | 53(1) | 56(1) | -2(1) | 25(1) | 30(1) |
| O(3) | 63(1) | 61(1) | 100(2) | 29(1) | 45(1) | 7(1) |
| O(4) | 55(1) | 109(2) | 52(1) | 18(1) | 10(1) | 24(1) |
| O(5) | 60(1) | 124(2) | 84(2) | -36(1) | 21(1) | -28(1) |
| O(6) | 76(1) | 60(1) | 56(1) | -22(1) | 14(1) | -7(1) |
| O(7) | 97(2) | 116(2) | 95(2) | 4(2) | 4(1) | -27(1) |
| N(1) | 45(1) | 39(1) | 38(1) | -6(1) | 18(1) | -6(1) |
| N(2) | 41(1) | 49(1) | 47(1) | 2(1) | 11(1) | -1(1) |
| C(1) | 45(2) | 41(1) | 43(2) | 1(1) | 13(1) | 2(1) |
| C(2) | 38(2) | 44(1) | 39(2) | 1(1) | 13(1) | 0(1) |
| C(3) | 55(2) | 39(1) | 38(2) | -2(1) | 20(1) | 3(1) |
| C(4) | 42(2) | 39(1) | 40(2) | 2(1) | 13(1) | -2(1) |
| C(5) | 58(2) | 53(2) | 45(2) | 3(1) | 16(2) | -4(2) |
| C(6) | 95(3) | 86(3) | 70(3) | -34(2) | 1(3) | -8(3) |
| C(7) | 45(2) | 46(1) | 40(2) | -2(1) | 16(1) | -3(1) |
| C(8) | 50(2) | 62(2) | 58(2) | 5(2) | 20(2) | 2(2) |
| C(9) | 52(2) | 91(2) | 64(2) | -1(2) | 30(2) | -10(2) |
| C(10) | 75(2) | 67(2) | 58(2) | 5(2) | 30(2) | -18(2) |
| C(11) | 74(2) | 55(2) | 64(2) | 8(2) | 32(2) | 1(2) |
| C(12) | 57(2) | 50(2) | 58(2) | 3(1) | 27(2) | -1(1) |
| C(13) | 42(2) | 43(1) | 38(2) | 1(1) | 11(1) | -6(1) |
| C(14) | 60(2) | 59(2) | 65(2) | 12(2) | 26(2) | 6(2) |
| C(15) | 77(2) | 80(2) | 76(2) | 25(2) | 39(2) | 3(2) |
| C(16) | 71(2) | 63(2) | 63(2) | 24(2) | 11(2) | -8(2) |
| C(17) | 60(2) | 52(2) | 71(2) | 13(2) | 13(2) | 7(2) |
| C(18) | 51(2) | 55(2) | 54(2) | 7(1) | 20(1) | 1(1) |
| C(19) | 55(2) | 41(1) | 38(2) | -4(1) | 14(1) | -2(1) |
| C(20) | 61(2) | 73(2) | 52(2) | 6(2) | 22(2) | 2(2) |
| C(21) | 73(3) | 97(2) | 58(2) | 10(2) | 29(2) | -18(2) |
| C(22) | 78(2) | 62(2) | 52(2) | 9(2) | 6(2) | -14(2) |
| C(23) | 73(2) | 83(2) | 62(2) | 12(2) | 9(2) | 15(2) |
| C(24) | 60(2) | 81(2) | 49(2) | 6(2) | 18(2) | 5(2) |
| C(25) | 124(3) | 116(3) | 77(3) | 42(2) | 1(2) | -31(2) |
| C(26) | 43(2) | 44(2) | 57(2) | 4(1) | 19(1) | 5(1) |
| C(27) | 55(2) | 52(2) | 56(2) | -4(2) | 18(2) | 2(1) |
| C(28) | 61(2) | 65(2) | 60(2) | 3(2) | 26(2) | 7(2) |
| C(29) | 68(2) | 65(2) | 55(2) | 7(2) | 16(2) | 24(2) |
| C(30) | 81(3) | 57(2) | 83(3) | -21(2) | 10(2) | 6(2) |
| C(31) | 60(2) | 61(2) | 95(3) | 1(2) | 31(2) | -3(2) |
| C(32) | 121(3) | 104(2) | 60(2) | -8(2) | 29(2) | 42(2) |
| C(33) | 75(3) | 85(2) | 92(3) | -25(2) | 9(2) | -10(2) |
| C(34) | 249(6) | 239(5) | 92(4) | -33(4) | 55(4) | -88(4) |
| C(35) | 100(3) | 343(7) | 244(6) | -162(5) | 74(4) | -119(4) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd23138.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(25A) | 8330 | 3963 | -2562 | 169 |
| H(25B) | 9689 | 3910 | -2542 | 169 |
| H(25C) | 9282 | 4571 | -2072 | 169 |
| H(32A) | 5479 | 4695 | 3818 | 143 |
| H(32B) | 6712 | 5138 | 4186 | 143 |
| H(32C) | 6583 | 4261 | 4408 | 143 |
| H(34A) | 6482 | 2142 | -213 | 290 |
| H(34B) | 5086 | 2140 | -668 | 290 |
| H(34C) | 5843 | 1365 | -596 | 290 |
| H(35A) | 4267 | 1235 | 821 | 336 |
| H(35B) | 4314 | 784 | 72 | 336 |
| H(35C) | 3708 | 1616 | -8 | 336 |
| H(1) | 12362(16) | 2487(10) | 1961(11) | 40(6) |
| H(2) | 10027(15) | 3407(9) | 2766(11) | 27(5) |
| H(3) | 12380(30) | 4975(19) | 4190(20) | 143(17) |
| H(4) | 13530(30) | 4434(17) | 4339(19) | 129(14) |
| H(5) | 13340(40) | 4910(20) | 3710(20) | 180(20) |
| H(6) | 13690(17) | 2809(11) | 1307(12) | 51(7) |
| H(7) | 14370(20) | 3635(14) | 500(15) | 93(10) |
| H(8) | 13227(19) | 4736(12) | -107(13) | 70(8) |
| H(9) | 11350(20) | 4959(13) | 99(13) | 68(8) |
| H(10) | 10740(20) | 4166(12) | 906(13) | 66(8) |
| H(11) | 8284(18) | 2250(11) | 2707(12) | 53(7) |
| H(12) | 8340(20) | 1264(13) | 3584(14) | 74(9) |
| H(13) | 9719(19) | 225(13) | 3744(14) | 71(8) |
| H(14) | 11000(20) | 231(13) | 2988(13) | 68(8) |
| H(15) | 10991(17) | 1243(10) | 2132(12) | 44(6) |
| H(16) | 11560(20) | 2356(12) | -443(13) | 67(8) |
| H(17) | 11070(20) | 3126(13) | -1493(14) | 65(9) |
| H(18) | 7860(20) | 3697(14) | -1282(15) | 94(10) |
| H(19) | 8340(20) | 2833(12) | -212(14) | 72(8) |
| H(23) | 6185(16) | 3145(11) | 1903(11) | 39(6) |
| H(24) | 5500(20) | 3480(13) | 2929(14) | 81(9) |
| H(25) | 8090(20) | 5142(14) | 3469(15) | 79(10) |
| H(26) | 8660(20) | 4765(12) | 2384(13) | 66(9) |
| H(36) | 7920(20) | 2435(13) | 1267(15) | 78(10) |

Table 6. Torsion angles [deg] for cd23138.

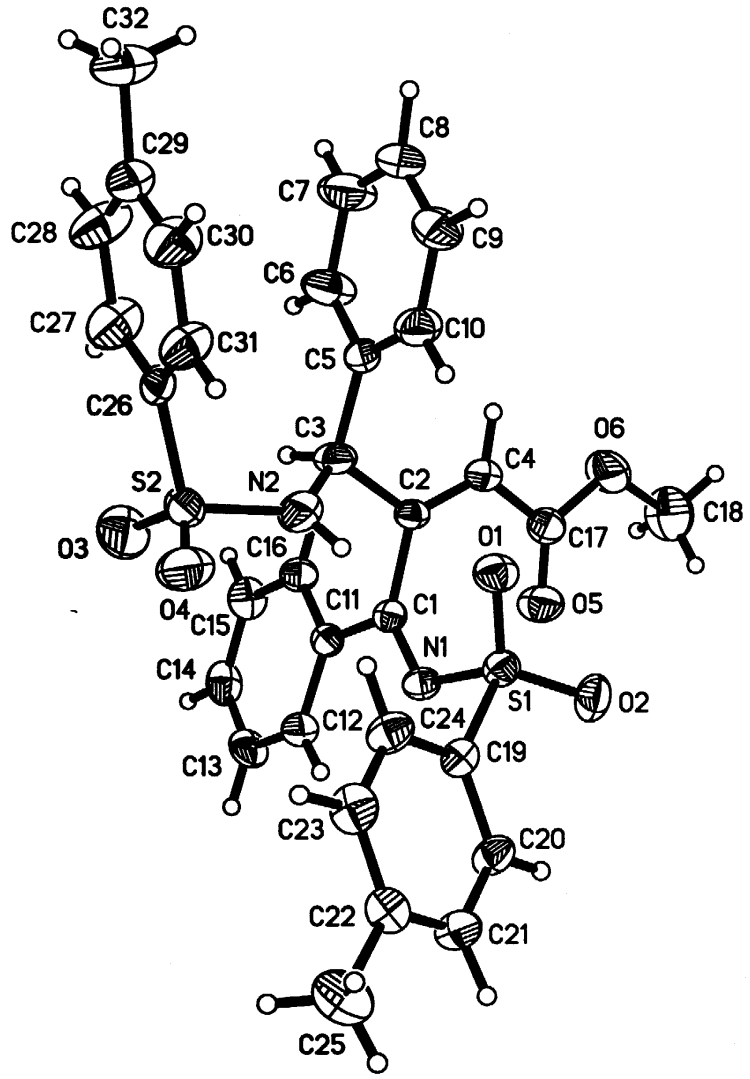
| | |
|---|---|
| O(1)-S(1)-N(1)-C(1) | 162.37(15) |
| O(2)-S(1)-N(1)-C(1) | 32.35(18) |
| C(19)-S(1)-N(1)-C(1) | -82.71(18) |
| O(1)-S(1)-N(1)-C(2) | -4.19(19) |
| O(2)-S(1)-N(1)-C(2) | -134.21(16) |
| C(19)-S(1)-N(1)-C(2) | 110.73(18) |
| O(3)-S(2)-N(2)-C(2) | 35.5(2) |
| O(4)-S(2)-N(2)-C(2) | 165.86(18) |
| C(26)-S(2)-N(2)-C(2) | -79.8(2) |
| C(2)-N(1)-C(1)-C(4) | -11.0(2) |
| S(1)-N(1)-C(1)-C(4) | -179.03(14) |
| C(2)-N(1)-C(1)-C(7) | -133.21(19) |
| S(1)-N(1)-C(1)-C(7) | 58.8(2) |
| S(2)-N(2)-C(2)-N(1) | -93.8(2) |
| S(2)-N(2)-C(2)-C(3) | 18.6(3) |
| S(2)-N(2)-C(2)-C(13) | 138.22(17) |
| C(1)-N(1)-C(2)-N(2) | 135.84(18) |
| S(1)-N(1)-C(2)-N(2) | -56.6(2) |
| C(1)-N(1)-C(2)-C(3) | 13.6(2) |
| S(1)-N(1)-C(2)-C(3) | -178.84(14) |
| C(1)-N(1)-C(2)-C(13) | -99.0(2) |
| S(1)-N(1)-C(2)-C(13) | 68.5(2) |
| N(2)-C(2)-C(3)-C(4) | -131.4(2) |
| N(1)-C(2)-C(3)-C(4) | -11.5(2) |
| C(13)-C(2)-C(3)-C(4) | 107.4(2) |
| C(2)-C(3)-C(4)-C(5) | -171.2(2) |
| C(2)-C(3)-C(4)-C(1) | 5.5(3) |
| N(1)-C(1)-C(4)-C(3) | 3.3(2) |
| C(7)-C(1)-C(4)-C(3) | 126.0(2) |
| N(1)-C(1)-C(4)-C(5) | -179.80(18) |
| C(7)-C(1)-C(4)-C(5) | -57.1(3) |
| C(6)-O(6)-C(5)-O(5) | 0.1(4) |
| C(6)-O(6)-C(5)-C(4) | 179.3(3) |
| C(3)-C(4)-C(5)-O(5) | 166.0(2) |
| C(1)-C(4)-C(5)-O(5) | -10.4(4) |
| C(3)-C(4)-C(5)-O(6) | -13.2(3) |
| C(1)-C(4)-C(5)-O(6) | 170.37(19) |
| N(1)-C(1)-C(7)-C(8) | -121.5(2) |
| C(4)-C(1)-C(7)-C(8) | 124.0(2) |
| N(1)-C(1)-C(7)-C(12) | 59.6(3) |
| C(4)-C(1)-C(7)-C(12) | -54.8(3) |
| C(12)-C(7)-C(8)-C(9) | -0.4(4) |
| C(1)-C(7)-C(8)-C(9) | -179.4(2) |
| C(7)-C(8)-C(9)-C(10) | -0.6(4) |
| C(8)-C(9)-C(10)-C(11) | 1.5(4) |
| C(9)-C(10)-C(11)-C(12) | -1.5(4) |
| C(10)-C(11)-C(12)-C(7) | 0.4(4) |
| C(8)-C(7)-C(12)-C(11) | 0.5(4) |
| C(1)-C(7)-C(12)-C(11) | 179.4(2) |
| N(2)-C(2)-C(13)-C(18) | 137.7(2) |
| N(1)-C(2)-C(13)-C(18) | 11.0(3) |
| C(3)-C(2)-C(13)-C(18) | -97.3(2) |
| N(2)-C(2)-C(13)-C(14) | -50.8(3) |
| N(1)-C(2)-C(13)-C(14) | -177.5(2) |
| C(3)-C(2)-C(13)-C(14) | 74.2(3) |
| C(18)-C(13)-C(14)-C(15) | -0.1(4) |
| C(2)-C(13)-C(14)-C(15) | -172.0(2) |
| C(13)-C(14)-C(15)-C(16) | 0.1(5) |
| C(14)-C(15)-C(16)-C(17) | -0.7(5) |
| C(15)-C(16)-C(17)-C(18) | 1.2(5) |
| C(14)-C(13)-C(18)-C(17) | 0.6(4) |
| C(2)-C(13)-C(18)-C(17) | 172.2(2) |
| C(16)-C(17)-C(18)-C(13) | -1.2(4) |
| O(1)-S(1)-C(19)-C(20) | -137.61(19) |

| | |
|---|---|
| O(2)-S(1)-C(19)-C(20) | -6.6(2) |
| N(1)-S(1)-C(19)-C(20) | 108.5(2) |
| O(1)-S(1)-C(19)-C(24) | 41.9(2) |
| O(2)-S(1)-C(19)-C(24) | 172.93(19) |
| N(1)-S(1)-C(19)-C(24) | -72.0(2) |
| C(24)-C(19)-C(20)-C(21) | 0.9(4) |
| S(1)-C(19)-C(20)-C(21) | -179.6(2) |
| C(19)-C(20)-C(21)-C(22) | -0.4(4) |
| C(20)-C(21)-C(22)-C(23) | -0.2(4) |
| C(20)-C(21)-C(22)-C(25) | 178.7(3) |
| C(21)-C(22)-C(23)-C(24) | 0.3(4) |
| C(25)-C(22)-C(23)-C(24) | -178.6(3) |
| C(22)-C(23)-C(24)-C(19) | 0.2(4) |
| C(20)-C(19)-C(24)-C(23) | -0.8(4) |
| S(1)-C(19)-C(24)-C(23) | 179.7(2) |
| O(3)-S(2)-C(26)-C(31) | -1.4(2) |
| O(4)-S(2)-C(26)-C(31) | -132.4(2) |
| N(2)-S(2)-C(26)-C(31) | 114.7(2) |
| O(3)-S(2)-C(26)-C(27) | 177.06(19) |
| O(4)-S(2)-C(26)-C(27) | 46.1(2) |
| N(2)-S(2)-C(26)-C(27) | -66.8(2) |
| C(31)-C(26)-C(27)-C(28) | 0.9(4) |
| S(2)-C(26)-C(27)-C(28) | -177.6(2) |
| C(26)-C(27)-C(28)-C(29) | -0.3(4) |
| C(27)-C(28)-C(29)-C(30) | -0.4(4) |
| C(27)-C(28)-C(29)-C(32) | 177.5(2) |
| C(28)-C(29)-C(30)-C(31) | 0.3(4) |
| C(32)-C(29)-C(30)-C(31) | -177.6(3) |
| C(27)-C(26)-C(31)-C(30) | -0.9(4) |
| S(2)-C(26)-C(31)-C(30) | 177.6(2) |
| C(29)-C(30)-C(31)-C(26) | 0.3(5) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23138 [A and deg.].

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

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The crystal data of **9** has been deposited in CCDC with number 216648. Empirical Formula: $C_{32}H_{30}N_2O_6S_2$; Formula Weight: 602.70; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.450 x 0.339 x 0.226 mm; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 15.3098(13)Å, b = 15.6606(10)Å, c = 12.3382(10)Å, $\alpha = 90^o$, $\beta = 91.245(2)^o$, $\gamma = 90^o$, V = 2957.5(4)Å$^3$; Space group: P2(1)/n; Z = 4; $D_{calc} = 1.354$ g/cm$^3$; $F_{000} = 1264$; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0581, 0.1411.

Table 1. Crystal data and structure refinement for cd23172.

| | |
|---|---|
| Identification code | cd23172 |
| Empirical formula | C32 H30 N2 O6 S2 |
| Formula weight | 602.70 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/c |
| Unit cell dimensions | a = 15.3098(13) A alpha = 90 deg.<br>b = 15.6606(13) A beta = 91.245(2) deg.<br>c = 12.3382(10) A gamma = 90 deg. |
| Volume | 2957.5(4) A^3 |
| Z, Calculated density | 4, 1.354 Mg/m^3 |
| Absorption coefficient | 0.228 mm^-1 |
| F(000) | 1264 |
| Crystal size | 0.450 x 0.339 x 0.226 mm |
| Theta range for data collection | 1.33 to 28.32 deg. |
| Limiting indices | -13<=h<=19, -20<=k<=19, -16<=l<=16 |
| Reflections collected / unique | 17841 / 6883 [R(int) = 0.0697] |
| Completeness to theta = 28.32 | 93.5 % |
| Absorption correction | Sadabs |
| Max. and min. transmission | 1.00000 and 0.71803 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 6883 / 2 / 413 |
| Goodness-of-fit on F^2 | 0.881 |
| Final R indices [I>2sigma(I)] | R1 = 0.0581, wR2 = 0.1411 |
| R indices (all data) | R1 = 0.1175, wR2 = 0.1668 |
| Largest diff. peak and hole | 0.880 and -0.277 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd23172.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 1894(1) | 10298(1) | 779(1) | 45(1) |
| S(2) | 3759(1) | 7781(1) | 876(1) | 71(1) |
| N(1) | 1481(1) | 9468(1) | 1427(2) | 40(1) |
| N(2) | 3418(2) | 8687(2) | 1360(2) | 70(1) |
| O(1) | 2827(1) | 10366(1) | 904(2) | 56(1) |
| O(2) | 1380(1) | 11023(1) | 1053(2) | 62(1) |
| O(3) | 3343(2) | 7077(2) | 1359(3) | 103(1) |
| O(4) | 3634(2) | 7923(2) | -275(3) | 103(1) |
| O(5) | 1213(2) | 10373(2) | 3639(2) | 70(1) |
| O(6) | 2168(2) | 11071(2) | 4696(2) | 83(1) |
| C(1) | 1826(2) | 9147(2) | 2293(2) | 37(1) |
| C(2) | 2676(2) | 9433(2) | 2809(2) | 38(1) |
| C(3) | 3463(2) | 8881(2) | 2501(3) | 52(1) |
| C(4) | 2721(2) | 10041(2) | 3551(2) | 46(1) |
| C(5) | 4335(2) | 9225(2) | 2899(3) | 47(1) |
| C(6) | 4723(2) | 8886(2) | 3801(3) | 78(1) |
| C(7) | 5545(3) | 9150(3) | 4152(4) | 96(1) |
| C(8) | 5980(3) | 9752(3) | 3601(4) | 75(1) |
| C(9) | 5593(2) | 10103(2) | 2709(4) | 79(1) |
| C(10) | 4778(2) | 9844(2) | 2358(3) | 66(1) |
| C(11) | 1363(2) | 8434(2) | 2828(2) | 38(1) |
| C(12) | 601(2) | 8099(2) | 2367(3) | 49(1) |
| C(13) | 156(2) | 7465(2) | 2898(3) | 60(1) |
| C(14) | 459(2) | 7152(2) | 3867(3) | 59(1) |
| C(15) | 1215(2) | 7461(2) | 4312(3) | 57(1) |
| C(16) | 1669(2) | 8102(2) | 3804(2) | 47(1) |
| C(17) | 1946(2) | 10501(2) | 3932(2) | 53(1) |
| C(18) | 1445(3) | 11552(3) | 5144(4) | 109(2) |
| C(19) | 1652(2) | 10003(2) | -561(2) | 41(1) |
| C(20) | 862(2) | 10221(2) | -1042(3) | 51(1) |
| C(21) | 678(2) | 9975(2) | -2081(3) | 57(1) |
| C(22) | 1266(2) | 9523(2) | -2680(2) | 52(1) |
| C(23) | 2051(2) | 9314(2) | -2192(3) | 60(1) |
| C(24) | 2255(2) | 9546(2) | -1143(3) | 56(1) |
| C(25) | 1053(3) | 9234(3) | -3814(3) | 80(1) |
| C(26) | 4879(2) | 7711(2) | 1165(3) | 50(1) |
| C(27) | 5194(3) | 7255(3) | 2032(4) | 87(1) |
| C(28) | 6071(3) | 7261(3) | 2301(4) | 92(1) |
| C(29) | 6644(2) | 7720(2) | 1740(3) | 70(1) |
| C(30) | 6327(2) | 8154(2) | 866(4) | 85(1) |
| C(31) | 5454(2) | 8147(2) | 571(3) | 72(1) |
| C(32) | 7601(3) | 7793(3) | 2079(4) | 126(2) |

Table 3. Bond lengths [A] and angles [deg] for cd23172.

| | |
|---|---|
| S(1)-O(2) | 1.426(2) |
| S(1)-O(1) | 1.437(2) |
| S(1)-N(1) | 1.657(2) |
| S(1)-C(19) | 1.748(3) |
| S(2)-O(3) | 1.411(3) |
| S(2)-O(4) | 1.446(3) |
| S(2)-N(2) | 1.631(3) |
| S(2)-C(26) | 1.747(3) |
| N(1)-C(1) | 1.285(3) |
| N(2)-C(3) | 1.440(4) |
| N(2)-H(30) | 0.8600 |
| O(5)-C(17) | 1.189(3) |
| O(6)-C(17) | 1.338(4) |
| O(6)-C(18) | 1.457(4) |
| C(1)-C(11) | 1.485(4) |
| C(1)-C(2) | 1.504(4) |
| C(2)-C(4) | 1.322(4) |
| C(2)-C(3) | 1.537(4) |
| C(3)-C(5) | 1.511(4) |
| C(3)-H(1) | 0.984(18) |
| C(4)-C(17) | 1.473(4) |
| C(4)-H(2) | 0.9300 |
| C(5)-C(6) | 1.358(4) |
| C(5)-C(10) | 1.366(4) |
| C(6)-C(7) | 1.386(5) |
| C(6)-H(3) | 0.9300 |
| C(7)-C(8) | 1.346(5) |
| C(7)-H(4) | 0.9300 |
| C(8)-C(9) | 1.355(5) |
| C(8)-H(5) | 0.98(4) |
| C(9)-C(10) | 1.374(4) |
| C(9)-H(6) | 0.9300 |
| C(10)-H(7) | 0.9300 |
| C(11)-C(16) | 1.383(4) |
| C(11)-C(12) | 1.389(4) |
| C(12)-C(13) | 1.379(4) |
| C(12)-H(8) | 0.91(3) |
| C(13)-C(14) | 1.364(5) |
| C(13)-H(9) | 0.9300 |
| C(14)-C(15) | 1.360(5) |
| C(14)-H(10) | 0.95(3) |
| C(15)-C(16) | 1.380(4) |
| C(15)-H(11) | 0.97(3) |
| C(16)-H(12) | 0.9300 |
| C(18)-H(13) | 0.9600 |
| C(18)-H(15) | 0.9600 |
| C(18)-H(14) | 0.9600 |
| C(19)-C(20) | 1.379(4) |
| C(19)-C(24) | 1.382(4) |
| C(20)-C(21) | 1.363(4) |
| C(20)-H(16) | 0.95(3) |
| C(21)-C(22) | 1.374(4) |
| C(21)-H(17) | 0.95(3) |
| C(22)-C(23) | 1.371(4) |
| C(22)-C(25) | 1.500(4) |
| C(23)-C(24) | 1.374(4) |
| C(23)-H(18) | 0.91(3) |
| C(24)-H(19) | 0.9300 |
| C(25)-H(22) | 0.9600 |
| C(25)-H(21) | 0.9600 |
| C(25)-H(20) | 0.9600 |
| C(26)-C(31) | 1.345(4) |
| C(26)-C(27) | 1.365(5) |
| C(27)-C(28) | 1.377(5) |

| | |
|---|---|
| C(27)-H(23) | 0.9300 |
| C(28)-C(29) | 1.339(5) |
| C(28)-H(24) | 0.9300 |
| C(29)-C(30) | 1.356(5) |
| C(29)-C(32) | 1.518(5) |
| C(30)-C(31) | 1.377(5) |
| C(30)-H(25) | 0.9300 |
| C(31)-H(26) | 0.9300 |
| C(32)-H(27) | 0.9600 |
| C(32)-H(29) | 0.9600 |
| C(32)-H(28) | 0.9600 |

| | |
|---|---|
| O(2)-S(1)-O(1) | 117.85(13) |
| O(2)-S(1)-N(1) | 106.95(13) |
| O(1)-S(1)-N(1) | 113.30(11) |
| O(2)-S(1)-C(19) | 109.09(13) |
| O(1)-S(1)-C(19) | 108.13(13) |
| N(1)-S(1)-C(19) | 100.00(12) |
| O(3)-S(2)-O(4) | 118.98(18) |
| O(3)-S(2)-N(2) | 111.95(17) |
| O(4)-S(2)-N(2) | 100.88(17) |
| O(3)-S(2)-C(26) | 108.35(17) |
| O(4)-S(2)-C(26) | 108.58(17) |
| N(2)-S(2)-C(26) | 107.46(14) |
| C(1)-N(1)-S(1) | 123.63(19) |
| C(3)-N(2)-S(2) | 122.2(2) |
| C(3)-N(2)-H(30) | 118.9 |
| S(2)-N(2)-H(30) | 118.9 |
| C(17)-O(6)-C(18) | 115.4(3) |
| N(1)-C(1)-C(11) | 118.2(2) |
| N(1)-C(1)-C(2) | 124.9(2) |
| C(11)-C(1)-C(2) | 116.9(2) |
| C(4)-C(2)-C(1) | 122.6(3) |
| C(4)-C(2)-C(3) | 123.1(2) |
| C(1)-C(2)-C(3) | 113.8(2) |
| N(2)-C(3)-C(5) | 114.6(3) |
| N(2)-C(3)-C(2) | 109.8(2) |
| C(5)-C(3)-C(2) | 114.3(2) |
| N(2)-C(3)-H(1) | 111.7(18) |
| C(5)-C(3)-H(1) | 105(2) |
| C(2)-C(3)-H(1) | 100(3) |
| C(2)-C(4)-C(17) | 122.9(3) |
| C(2)-C(4)-H(2) | 118.6 |
| C(17)-C(4)-H(2) | 118.6 |
| C(6)-C(5)-C(10) | 117.7(3) |
| C(6)-C(5)-C(3) | 119.7(3) |
| C(10)-C(5)-C(3) | 122.6(3) |
| C(5)-C(6)-C(7) | 121.2(3) |
| C(5)-C(6)-H(3) | 119.4 |
| C(7)-C(6)-H(3) | 119.4 |
| C(8)-C(7)-C(6) | 120.5(4) |
| C(8)-C(7)-H(4) | 119.8 |
| C(6)-C(7)-H(4) | 119.8 |
| C(7)-C(8)-C(9) | 118.8(4) |
| C(7)-C(8)-H(5) | 119(2) |
| C(9)-C(8)-H(5) | 122(2) |
| C(8)-C(9)-C(10) | 121.0(4) |
| C(8)-C(9)-H(6) | 119.5 |
| C(10)-C(9)-H(6) | 119.5 |
| C(5)-C(10)-C(9) | 120.9(3) |
| C(5)-C(10)-H(7) | 119.6 |
| C(9)-C(10)-H(7) | 119.6 |
| C(16)-C(11)-C(12) | 118.7(3) |
| C(16)-C(11)-C(1) | 120.9(2) |
| C(12)-C(11)-C(1) | 120.4(3) |
| C(13)-C(12)-C(11) | 119.8(3) |
| C(13)-C(12)-H(8) | 125.2(17) |
| C(11)-C(12)-H(8) | 115.0(17) |

| | |
|---|---|
| C(14)-C(13)-C(12) | 120.8(3) |
| C(14)-C(13)-H(9) | 119.6 |
| C(12)-C(13)-H(9) | 119.6 |
| C(15)-C(14)-C(13) | 119.8(3) |
| C(15)-C(14)-H(10) | 124.3(17) |
| C(13)-C(14)-H(10) | 115.7(17) |
| C(14)-C(15)-C(16) | 120.5(3) |
| C(14)-C(15)-H(11) | 118.0(19) |
| C(16)-C(15)-H(11) | 122(2) |
| C(15)-C(16)-C(11) | 120.3(3) |
| C(15)-C(16)-H(12) | 119.8 |
| C(11)-C(16)-H(12) | 119.8 |
| O(5)-C(17)-O(6) | 123.4(3) |
| O(5)-C(17)-C(4) | 125.5(3) |
| O(6)-C(17)-C(4) | 111.0(3) |
| O(6)-C(18)-H(13) | 109.5 |
| O(6)-C(18)-H(15) | 109.5 |
| H(13)-C(18)-H(15) | 109.5 |
| O(6)-C(18)-H(14) | 109.5 |
| H(13)-C(18)-H(14) | 109.5 |
| H(15)-C(18)-H(14) | 109.5 |
| C(20)-C(19)-C(24) | 119.6(3) |
| C(20)-C(19)-S(1) | 120.5(2) |
| C(24)-C(19)-S(1) | 119.9(2) |
| C(21)-C(20)-C(19) | 119.8(3) |
| C(21)-C(20)-H(16) | 120.2(18) |
| C(19)-C(20)-H(16) | 120.0(18) |
| C(20)-C(21)-C(22) | 121.8(3) |
| C(20)-C(21)-H(17) | 115.9(19) |
| C(22)-C(21)-H(17) | 122.2(19) |
| C(23)-C(22)-C(21) | 117.8(3) |
| C(23)-C(22)-C(25) | 120.5(3) |
| C(21)-C(22)-C(25) | 121.7(3) |
| C(22)-C(23)-C(24) | 122.0(3) |
| C(22)-C(23)-H(18) | 118.0(19) |
| C(24)-C(23)-H(18) | 119.7(19) |
| C(23)-C(24)-C(19) | 119.1(3) |
| C(23)-C(24)-H(19) | 120.5 |
| C(19)-C(24)-H(19) | 120.5 |
| C(22)-C(25)-H(22) | 109.5 |
| C(22)-C(25)-H(21) | 109.5 |
| H(22)-C(25)-H(21) | 109.5 |
| C(22)-C(25)-H(20) | 109.5 |
| H(22)-C(25)-H(20) | 109.5 |
| H(21)-C(25)-H(20) | 109.5 |
| C(31)-C(26)-C(27) | 117.9(3) |
| C(31)-C(26)-S(2) | 120.6(3) |
| C(27)-C(26)-S(2) | 121.5(3) |
| C(26)-C(27)-C(28) | 120.8(4) |
| C(26)-C(27)-H(23) | 119.6 |
| C(28)-C(27)-H(23) | 119.6 |
| C(29)-C(28)-C(27) | 121.7(4) |
| C(29)-C(28)-H(24) | 119.1 |
| C(27)-C(28)-H(24) | 119.1 |
| C(28)-C(29)-C(30) | 117.0(4) |
| C(28)-C(29)-C(32) | 122.4(4) |
| C(30)-C(29)-C(32) | 120.5(4) |
| C(29)-C(30)-C(31) | 122.3(4) |
| C(29)-C(30)-H(25) | 118.9 |
| C(31)-C(30)-H(25) | 118.9 |
| C(26)-C(31)-C(30) | 120.3(3) |
| C(26)-C(31)-H(26) | 119.9 |
| C(30)-C(31)-H(26) | 119.9 |
| C(29)-C(32)-H(27) | 109.5 |
| C(29)-C(32)-H(29) | 109.5 |
| H(27)-C(32)-H(29) | 109.5 |
| C(29)-C(32)-H(28) | 109.5 |
| H(27)-C(32)-H(28) | 109.5 |

| | |
|---|---|
| H(29)-C(32)-H(28) | 109.5 |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd23172. The anisotropic displacement factor exponent takes the form: -2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S(1) | 45(1) | 45(1) | 45(1) | 3(1) | -2(1) | 2(1) |
| S(2) | 41(1) | 64(1) | 107(1) | -38(1) | -2(1) | 4(1) |
| N(1) | 33(1) | 49(1) | 39(1) | 2(1) | 2(1) | 0(1) |
| N(2) | 54(2) | 73(2) | 83(2) | -16(2) | -6(2) | 16(1) |
| O(1) | 49(1) | 60(1) | 59(1) | 13(1) | -4(1) | -11(1) |
| O(2) | 79(2) | 47(1) | 61(2) | -5(1) | 2(1) | 19(1) |
| O(3) | 62(2) | 83(2) | 163(3) | -5(2) | 18(2) | -22(1) |
| O(4) | 78(2) | 121(2) | 107(2) | -32(2) | -34(2) | 26(2) |
| O(5) | 51(2) | 90(2) | 68(2) | -28(1) | -5(1) | 14(1) |
| O(6) | 85(2) | 84(2) | 81(2) | -43(2) | -14(1) | 8(1) |
| C(1) | 31(2) | 43(2) | 37(2) | -6(1) | 5(1) | 4(1) |
| C(2) | 32(2) | 41(2) | 40(2) | 4(1) | -2(1) | -4(1) |
| C(3) | 33(2) | 66(2) | 55(2) | -9(2) | -5(1) | 1(2) |
| C(4) | 41(2) | 50(2) | 48(2) | -3(2) | -7(1) | -2(1) |
| C(5) | 33(2) | 44(2) | 63(2) | -5(2) | 1(1) | 1(1) |
| C(6) | 66(2) | 84(3) | 81(3) | 25(2) | -26(2) | -30(2) |
| C(7) | 77(3) | 102(3) | 107(4) | 22(3) | -44(3) | -27(3) |
| C(8) | 43(2) | 77(3) | 105(3) | -6(2) | -16(2) | -10(2) |
| C(9) | 51(2) | 73(3) | 112(3) | 10(2) | 0(2) | -22(2) |
| C(10) | 48(2) | 67(2) | 84(3) | 17(2) | -8(2) | -7(2) |
| C(11) | 33(2) | 40(2) | 41(2) | -5(1) | 5(1) | 1(1) |
| C(12) | 42(2) | 51(2) | 55(2) | -3(2) | -3(2) | 0(2) |
| C(13) | 41(2) | 55(2) | 85(3) | -10(2) | 6(2) | -15(2) |
| C(14) | 59(2) | 46(2) | 74(3) | 4(2) | 22(2) | -8(2) |
| C(15) | 66(2) | 56(2) | 50(2) | 8(2) | 11(2) | -4(2) |
| C(16) | 42(2) | 52(2) | 46(2) | 2(2) | 2(1) | -3(1) |
| C(17) | 61(2) | 55(2) | 43(2) | -6(2) | -6(2) | 3(2) |
| C(18) | 117(4) | 105(3) | 104(4) | -59(3) | 8(3) | 30(3) |
| C(19) | 40(2) | 44(2) | 40(2) | 7(1) | 6(1) | 2(1) |
| C(20) | 48(2) | 60(2) | 43(2) | -1(2) | 1(2) | 17(2) |
| C(21) | 50(2) | 69(2) | 51(2) | 1(2) | -4(2) | 9(2) |
| C(22) | 58(2) | 55(2) | 43(2) | 1(2) | 9(2) | -7(2) |
| C(23) | 55(2) | 70(2) | 55(2) | -7(2) | 21(2) | 5(2) |
| C(24) | 41(2) | 71(2) | 55(2) | 2(2) | 8(2) | 11(2) |
| C(25) | 92(3) | 100(3) | 49(2) | -11(2) | 12(2) | -15(2) |
| C(26) | 41(2) | 41(2) | 67(2) | -7(2) | 5(2) | 5(1) |
| C(27) | 63(3) | 88(3) | 112(3) | 39(3) | 18(2) | -2(2) |
| C(28) | 65(3) | 118(4) | 93(3) | 39(3) | -5(2) | 20(2) |
| C(29) | 50(2) | 75(3) | 86(3) | -10(2) | -5(2) | 6(2) |
| C(30) | 50(2) | 87(3) | 119(4) | 23(3) | 19(2) | -9(2) |
| C(31) | 59(2) | 77(3) | 78(3) | 23(2) | 2(2) | 9(2) |
| C(32) | 58(3) | 145(4) | 174(5) | -30(4) | -35(3) | 9(3) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd23172.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(30) | 3204 | 9062 | 920 | 84 |
| H(2) | 3265 | 10185 | 3846 | 56 |
| H(3) | 4432 | 8469 | 4190 | 93 |
| H(4) | 5799 | 8910 | 4773 | 115 |
| H(6) | 5884 | 10525 | 2329 | 95 |
| H(7) | 4524 | 10093 | 1744 | 80 |
| H(9) | -358 | 7247 | 2591 | 72 |
| H(12) | 2184 | 8311 | 4118 | 56 |
| H(13) | 1094 | 11788 | 4563 | 163 |
| H(15) | 1671 | 12006 | 5591 | 163 |
| H(14) | 1094 | 11179 | 5572 | 163 |
| H(19) | 2791 | 9397 | -829 | 67 |
| H(22) | 491 | 9450 | -4034 | 121 |
| H(21) | 1045 | 8621 | -3838 | 121 |
| H(20) | 1488 | 9445 | -4295 | 121 |
| H(23) | 4812 | 6936 | 2445 | 105 |
| H(24) | 6271 | 6938 | 2888 | 111 |
| H(25) | 6712 | 8468 | 450 | 102 |
| H(26) | 5262 | 8445 | -41 | 86 |
| H(27) | 7681 | 8284 | 2536 | 189 |
| H(29) | 7949 | 7853 | 1446 | 189 |
| H(28) | 7776 | 7289 | 2468 | 189 |
| H(1) | 3360(30) | 8371(19) | 2946(18) | 151 |
| H(5) | 6580(30) | 9900(20) | 3840(30) | 112(14) |
| H(8) | 432(17) | 8339(17) | 1720(20) | 45(8) |
| H(10) | 133(18) | 6691(18) | 4160(20) | 54(9) |
| H(11) | 1420(20) | 7220(20) | 5000(30) | 74(11) |
| H(16) | 454(19) | 10550(17) | -660(20) | 54(9) |
| H(17) | 130(20) | 10165(18) | -2380(20) | 62(9) |
| H(18) | 2416(19) | 8963(19) | -2560(20) | 63(10) |

Table 6. Torsion angles [deg] for cd23172.

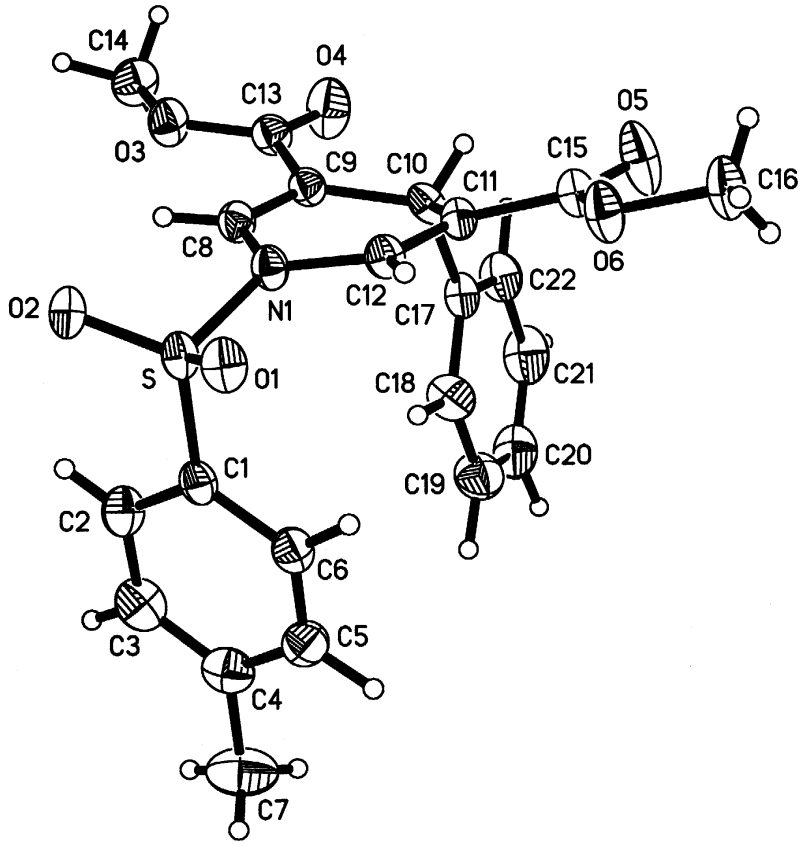
| | |
|---|---|
| O(2)-S(1)-N(1)-C(1) | -102.5(2) |
| O(1)-S(1)-N(1)-C(1) | 29.1(3) |
| C(19)-S(1)-N(1)-C(1) | 143.9(2) |
| O(3)-S(2)-N(2)-C(3) | 56.3(3) |
| O(4)-S(2)-N(2)-C(3) | -176.1(2) |
| C(26)-S(2)-N(2)-C(3) | -62.5(3) |
| S(1)-N(1)-C(1)-C(11) | 176.64(18) |
| S(1)-N(1)-C(1)-C(2) | -4.1(4) |
| N(1)-C(1)-C(2)-C(4) | 90.0(4) |
| C(11)-C(1)-C(2)-C(4) | -90.7(3) |
| N(1)-C(1)-C(2)-C(3) | -97.2(3) |
| C(11)-C(1)-C(2)-C(3) | 82.2(3) |
| S(2)-N(2)-C(3)-C(5) | 84.0(3) |
| S(2)-N(2)-C(3)-C(2) | -145.8(2) |
| C(4)-C(2)-C(3)-N(2) | -144.8(3) |
| C(1)-C(2)-C(3)-N(2) | 42.4(3) |
| C(4)-C(2)-C(3)-C(5) | -14.5(4) |
| C(1)-C(2)-C(3)-C(5) | 172.7(3) |
| C(1)-C(2)-C(4)-C(17) | -0.7(4) |
| C(3)-C(2)-C(4)-C(17) | -172.8(3) |
| N(2)-C(3)-C(5)-C(6) | -131.5(3) |
| C(2)-C(3)-C(5)-C(6) | 100.6(4) |
| N(2)-C(3)-C(5)-C(10) | 45.4(4) |
| C(2)-C(3)-C(5)-C(10) | -82.6(4) |
| C(10)-C(5)-C(6)-C(7) | -0.9(6) |
| C(3)-C(5)-C(6)-C(7) | 176.1(4) |
| C(5)-C(6)-C(7)-C(8) | -0.1(7) |
| C(6)-C(7)-C(8)-C(9) | 1.0(7) |
| C(7)-C(8)-C(9)-C(10) | -0.9(6) |
| C(6)-C(5)-C(10)-C(9) | 0.9(5) |
| C(3)-C(5)-C(10)-C(9) | -176.0(3) |
| C(8)-C(9)-C(10)-C(5) | 0.0(6) |
| N(1)-C(1)-C(11)-C(16) | -175.4(2) |
| C(2)-C(1)-C(11)-C(16) | 5.2(4) |
| N(1)-C(1)-C(11)-C(12) | 3.2(4) |
| C(2)-C(1)-C(11)-C(12) | -176.2(3) |
| C(16)-C(11)-C(12)-C(13) | 1.7(4) |
| C(1)-C(11)-C(12)-C(13) | -176.9(3) |
| C(11)-C(12)-C(13)-C(14) | -0.8(5) |
| C(12)-C(13)-C(14)-C(15) | -0.7(5) |
| C(13)-C(14)-C(15)-C(16) | 1.4(5) |
| C(14)-C(15)-C(16)-C(11) | -0.5(5) |
| C(12)-C(11)-C(16)-C(15) | -1.1(4) |
| C(1)-C(11)-C(16)-C(15) | 177.6(3) |
| C(18)-O(6)-C(17)-O(5) | -1.3(5) |
| C(18)-O(6)-C(17)-C(4) | -178.9(3) |
| C(2)-C(4)-C(17)-O(5) | 1.3(5) |
| C(2)-C(4)-C(17)-O(6) | 178.8(3) |
| O(2)-S(1)-C(19)-C(20) | -23.8(3) |
| O(1)-S(1)-C(19)-C(20) | -153.1(2) |
| N(1)-S(1)-C(19)-C(20) | 88.2(3) |
| O(2)-S(1)-C(19)-C(24) | 156.4(2) |
| O(1)-S(1)-C(19)-C(24) | 27.1(3) |
| N(1)-S(1)-C(19)-C(24) | -91.6(2) |
| C(24)-C(19)-C(20)-C(21) | 0.9(5) |
| S(1)-C(19)-C(20)-C(21) | -178.9(3) |
| C(19)-C(20)-C(21)-C(22) | -1.2(5) |
| C(20)-C(21)-C(22)-C(23) | 0.8(5) |
| C(20)-C(21)-C(22)-C(25) | 178.5(3) |
| C(21)-C(22)-C(23)-C(24) | -0.2(5) |
| C(25)-C(22)-C(23)-C(24) | -177.9(3) |
| C(22)-C(23)-C(24)-C(19) | 0.0(5) |
| C(20)-C(19)-C(24)-C(23) | -0.3(5) |
| S(1)-C(19)-C(24)-C(23) | 179.5(2) |

| | |
|---|---|
| O(3)-S(2)-C(26)-C(31) | 161.3(3) |
| O(4)-S(2)-C(26)-C(31) | 30.7(3) |
| N(2)-S(2)-C(26)-C(31) | -77.6(3) |
| O(3)-S(2)-C(26)-C(27) | -22.5(4) |
| O(4)-S(2)-C(26)-C(27) | -153.0(3) |
| N(2)-S(2)-C(26)-C(27) | 98.6(3) |
| C(31)-C(26)-C(27)-C(28) | 1.6(6) |
| S(2)-C(26)-C(27)-C(28) | -174.8(3) |
| C(26)-C(27)-C(28)-C(29) | 1.0(7) |
| C(27)-C(28)-C(29)-C(30) | -2.5(6) |
| C(27)-C(28)-C(29)-C(32) | 174.9(4) |
| C(28)-C(29)-C(30)-C(31) | 1.5(6) |
| C(32)-C(29)-C(30)-C(31) | -175.9(4) |
| C(27)-C(26)-C(31)-C(30) | -2.6(5) |
| S(2)-C(26)-C(31)-C(30) | 173.8(3) |
| C(29)-C(30)-C(31)-C(26) | 1.1(6) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23172 [A and deg.].

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

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The crystal data of **10** has been deposited in CCDC with number 216650. Empirical Formula: $C_{22}H_{21}NO_6S$; Formula Weight: 427.46; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.478 x 0.365 x 0.310 mm; Crystal System: Orthorhombic; Lattice Type: Primitive; Lattice Parameters: a = 10.1562(8)Å, b = 10.4485(8)Å, c = 19.7286(15)Å, $\alpha = 90^o$, $\beta = 90^o$, $\gamma = 90^o$, V = 2093.5(3)Å$^3$; Space group: P2(1)2(1)2(1); Z = 4; $D_{calc}$ = 1.356 g/cm$^3$; $F_{000}$ = 896; Diffractometer: Rigaku AFC7R; Residuals: R; Rw: 0.0383, 0.0632.

Table 1. Crystal data and structure refinement for cd23110.

| | |
|---|---|
| Identification code | cd23110 |
| Empirical formula | C22 H21 N O6 S |
| Formula weight | 427.46 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Orthorhombic, P2(1)2(1)2(1) |
| Unit cell dimensions | a = 10.1562(8) A alpha = 90 deg.<br>b = 10.4485(8) A beta = 90 deg.<br>c = 19.7286(15) A gamma = 90 deg. |
| Volume | 2093.5(3) A^3 |
| Z, Calculated density | 4, 1.356 Mg/m^3 |
| Absorption coefficient | 0.193 mm^-1 |
| F(000) | 896 |
| Crystal size | 0.478 x 0.365 x 0.310 mm |
| Theta range for data collection | 2.06 to 28.27 deg. |
| Limiting indices | -13<=h<=13, -6<=k<=13, -26<=l<=25 |
| Reflections collected / unique | 12886 / 4879 [R(int) = 0.0405] |
| Completeness to theta = 28.27 | 96.4 % |
| Absorption correction | Sadabs |
| Max. and min. transmission | 1.00000 and 0.48776 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 4879 / 0 / 356 |
| Goodness-of-fit on F^2 | 0.837 |
| Final R indices [I>2sigma(I)] | R1 = 0.0383, wR2 = 0.0632 |
| R indices (all data) | R1 = 0.0558, wR2 = 0.0668 |
| Absolute structure parameter | 0.00(15) |
| Largest diff. peak and hole | 0.301 and -0.279 e.A^-3 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd23110.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S | 6674(1) | 8262(1) | 9840(1) | 41(1) |
| O(1) | 7627(1) | 7664(1) | 10260(1) | 49(1) |
| O(2) | 5386(1) | 8526(1) | 10083(1) | 51(1) |
| O(3) | 4921(1) | 12607(1) | 9032(1) | 55(1) |
| O(4) | 6546(2) | 13564(2) | 8475(1) | 76(1) |
| O(5) | 11163(2) | 11976(2) | 9035(1) | 79(1) |
| O(6) | 11279(1) | 10314(1) | 9733(1) | 58(1) |
| N(1) | 7325(2) | 9689(1) | 9641(1) | 40(1) |
| C(1) | 6562(2) | 7465(2) | 9064(1) | 40(1) |
| C(2) | 5379(2) | 7411(2) | 8726(1) | 52(1) |
| C(3) | 5319(3) | 6794(3) | 8108(1) | 62(1) |
| C(4) | 6403(3) | 6243(2) | 7825(1) | 57(1) |
| C(5) | 7590(2) | 6323(2) | 8168(1) | 54(1) |
| C(6) | 7681(2) | 6924(2) | 8782(1) | 48(1) |
| C(7) | 6328(6) | 5502(4) | 7165(2) | 88(1) |
| C(8) | 6519(2) | 10661(2) | 9380(1) | 41(1) |
| C(9) | 7004(2) | 11624(2) | 9027(1) | 39(1) |
| C(10) | 8426(2) | 11660(2) | 8805(1) | 41(1) |
| C(11) | 9227(2) | 10828(2) | 9273(1) | 39(1) |
| C(12) | 8688(2) | 9868(2) | 9617(1) | 40(1) |
| C(13) | 6164(2) | 12699(2) | 8812(1) | 46(1) |
| C(14) | 4042(3) | 13588(3) | 8787(2) | 64(1) |
| C(15) | 10638(2) | 11110(2) | 9326(1) | 46(1) |
| C(16) | 12681(2) | 10516(3) | 9815(2) | 62(1) |
| C(17) | 8567(2) | 11252(2) | 8063(1) | 43(1) |
| C(18) | 8563(3) | 9988(3) | 7880(1) | 58(1) |
| C(19) | 8658(3) | 9609(3) | 7211(1) | 67(1) |
| C(20) | 8748(3) | 10504(4) | 6716(1) | 72(1) |
| C(21) | 8760(3) | 11778(4) | 6883(1) | 74(1) |
| C(22) | 8669(2) | 12158(3) | 7553(1) | 59(1) |

Table 3. Bond lengths [A] and angles [deg] for cd23110.

| | |
|---|---|
| S-O(1) | 1.4179(13) |
| S-O(2) | 1.4208(13) |
| S-N(1) | 1.6774(16) |
| S-C(1) | 1.7464(19) |
| O(3)-C(13) | 1.339(2) |
| O(3)-C(14) | 1.443(3) |
| O(4)-C(13) | 1.187(2) |
| O(5)-C(15) | 1.196(2) |
| O(6)-C(15) | 1.327(2) |
| O(6)-C(16) | 1.449(3) |
| N(1)-C(12) | 1.397(2) |
| N(1)-C(8) | 1.402(2) |
| C(1)-C(2) | 1.375(3) |
| C(1)-C(6) | 1.387(3) |
| C(2)-C(3) | 1.380(3) |
| C(2)-H(1) | 0.897(19) |
| C(3)-C(4) | 1.362(3) |
| C(3)-H(2) | 0.93(2) |
| C(4)-C(5) | 1.385(3) |
| C(4)-C(7) | 1.518(4) |
| C(5)-C(6) | 1.367(3) |
| C(5)-H(3) | 0.98(2) |
| C(6)-H(4) | 0.985(18) |
| C(7)-H(5) | 0.87(3) |
| C(7)-H(6) | 0.89(3) |
| C(7)-H(7) | 0.90(4) |
| C(8)-C(9) | 1.318(3) |
| C(8)-H(8) | 0.900(18) |
| C(9)-C(13) | 1.474(3) |
| C(9)-C(10) | 1.510(3) |
| C(10)-C(11) | 1.507(3) |
| C(10)-C(17) | 1.532(3) |
| C(10)-H(9) | 0.923(16) |
| C(11)-C(12) | 1.329(3) |
| C(11)-C(15) | 1.467(3) |
| C(12)-H(10) | 0.888(16) |
| C(14)-H(11) | 0.94(2) |
| C(14)-H(12) | 1.02(3) |
| C(14)-H(13) | 0.93(2) |
| C(16)-H(14) | 0.88(3) |
| C(16)-H(15) | 0.93(2) |
| C(16)-H(16) | 0.89(3) |
| C(17)-C(18) | 1.368(3) |
| C(17)-C(22) | 1.384(3) |
| C(18)-C(19) | 1.381(3) |
| C(18)-H(17) | 0.892(19) |
| C(19)-C(20) | 1.355(4) |
| C(19)-H(18) | 0.99(2) |
| C(20)-C(21) | 1.371(4) |
| C(20)-H(19) | 0.98(2) |
| C(21)-C(22) | 1.383(3) |
| C(21)-H(20) | 0.91(2) |
| C(22)-H(21) | 0.965(18) |
| | |
| O(1)-S-O(2) | 121.12(8) |
| O(1)-S-N(1) | 105.04(8) |
| O(2)-S-N(1) | 105.62(8) |
| O(1)-S-C(1) | 110.24(9) |
| O(2)-S-C(1) | 109.16(9) |
| N(1)-S-C(1) | 104.16(8) |
| C(13)-O(3)-C(14) | 115.08(19) |
| C(15)-O(6)-C(16) | 117.3(2) |
| C(12)-N(1)-C(8) | 117.91(17) |
| C(12)-N(1)-S | 121.17(14) |

| | |
|---|---|
| C(20)-C(19)-H(18) | 119.9(13) |
| C(18)-C(19)-H(18) | 120.4(13) |
| C(19)-C(20)-C(21) | 119.9(3) |
| C(19)-C(20)-H(19) | 115.7(15) |
| C(21)-C(20)-H(19) | 124.0(15) |
| C(20)-C(21)-C(22) | 120.4(3) |
| C(20)-C(21)-H(20) | 119.3(15) |
| C(22)-C(21)-H(20) | 120.0(15) |
| C(21)-C(22)-C(17) | 120.2(3) |
| C(21)-C(22)-H(21) | 124.4(12) |
| C(17)-C(22)-H(21) | 115.5(12) |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd23110.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| S | 34(1) | 45(1) | 44(1) | 10(1) | 1(1) | -4(1) |
| O(1) | 45(1) | 54(1) | 49(1) | 15(1) | -3(1) | 0(1) |
| O(2) | 38(1) | 62(1) | 53(1) | 6(1) | 10(1) | -5(1) |
| O(3) | 39(1) | 47(1) | 78(1) | 11(1) | 0(1) | 8(1) |
| O(4) | 61(1) | 59(1) | 109(1) | 37(1) | 16(1) | 9(1) |
| O(5) | 44(1) | 89(1) | 104(1) | 51(1) | -14(1) | -23(1) |
| O(6) | 33(1) | 63(1) | 77(1) | 22(1) | -9(1) | -5(1) |
| N(1) | 32(1) | 40(1) | 49(1) | 9(1) | -1(1) | 0(1) |
| C(1) | 38(1) | 38(1) | 43(1) | 10(1) | 1(1) | -5(1) |
| C(2) | 45(1) | 60(2) | 51(1) | 9(1) | -2(1) | 1(1) |
| C(3) | 59(2) | 68(2) | 59(2) | 9(2) | -20(1) | -1(2) |
| C(4) | 78(2) | 47(1) | 46(1) | 10(1) | -4(1) | -2(1) |
| C(5) | 60(2) | 46(1) | 57(1) | 6(1) | 7(1) | 3(1) |
| C(6) | 43(1) | 46(1) | 54(1) | 5(1) | -1(1) | -3(1) |
| C(7) | 142(4) | 70(2) | 53(2) | -4(2) | -17(2) | 8(3) |
| C(8) | 31(1) | 45(1) | 46(1) | 0(1) | -3(1) | 1(1) |
| C(9) | 35(1) | 39(1) | 44(1) | 1(1) | -3(1) | -1(1) |
| C(10) | 40(1) | 36(1) | 49(1) | 6(1) | -8(1) | -8(1) |
| C(11) | 33(1) | 45(1) | 40(1) | 2(1) | 1(1) | -4(1) |
| C(12) | 34(1) | 44(1) | 42(1) | 4(1) | -7(1) | 2(1) |
| C(13) | 45(1) | 41(1) | 53(1) | 3(1) | -3(1) | 0(1) |
| C(14) | 57(2) | 53(2) | 82(2) | 7(2) | -1(2) | 16(1) |
| C(15) | 36(1) | 52(1) | 49(1) | 6(1) | -1(1) | -5(1) |
| C(16) | 31(1) | 75(2) | 81(2) | 16(2) | -6(2) | -5(1) |
| C(17) | 33(1) | 54(1) | 43(1) | 10(1) | -1(1) | -4(1) |
| C(18) | 67(2) | 59(2) | 47(1) | 6(1) | -4(1) | -9(1) |
| C(19) | 71(2) | 77(2) | 54(2) | -6(2) | -5(1) | -8(2) |
| C(20) | 61(2) | 108(3) | 48(2) | -1(2) | -1(1) | -8(2) |
| C(21) | 70(2) | 103(3) | 50(2) | 27(2) | 2(1) | -8(2) |
| C(22) | 56(2) | 64(2) | 56(2) | 14(1) | -2(1) | -5(1) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd23110.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 4660(20) | 7757(19) | 8917(9) | 53(7) |
| H(2) | 4510(20) | 6760(20) | 7889(11) | 83(8) |
| H(3) | 8400(20) | 5970(20) | 7975(9) | 61(7) |
| H(4) | 8503(18) | 7006(17) | 9044(8) | 48(6) |
| H(5) | 6510(30) | 4700(30) | 7225(13) | 101(11) |
| H(6) | 5500(30) | 5610(30) | 7018(17) | 127(15) |
| H(7) | 6770(40) | 5870(40) | 6821(18) | 172(18) |
| H(8) | 5670(19) | 10577(18) | 9503(8) | 38(6) |
| H(9) | 8766(15) | 12476(15) | 8844(7) | 23(4) |
| H(10) | 9164(16) | 9340(16) | 9870(8) | 29(5) |
| H(11) | 4320(20) | 14370(20) | 8977(11) | 64(8) |
| H(12) | 3280(30) | 13440(30) | 9116(13) | 114(10) |
| H(13) | 4020(20) | 13600(20) | 8317(13) | 88(10) |
| H(14) | 13040(30) | 10440(30) | 9413(13) | 101(12) |
| H(15) | 12820(20) | 11390(20) | 9857(12) | 94(10) |
| H(16) | 13000(30) | 9980(30) | 10124(13) | 102(11) |
| H(17) | 8410(20) | 9385(18) | 8191(9) | 51(6) |
| H(18) | 8620(20) | 8690(20) | 7091(11) | 86(9) |
| H(19) | 8920(20) | 10180(20) | 6261(12) | 85(8) |
| H(20) | 8740(20) | 12370(20) | 6546(12) | 79(8) |
| H(21) | 8672(18) | 13038(18) | 7702(9) | 46(6) |

Table 6. Torsion angles [deg] for cd23110.

| | |
|---|---|
| O(1)-S-N(1)-C(12) | 27.87(16) |
| O(2)-S-N(1)-C(12) | 156.98(14) |
| C(1)-S-N(1)-C(12) | -88.06(16) |
| O(1)-S-N(1)-C(8) | -163.46(14) |
| O(2)-S-N(1)-C(8) | -34.35(16) |
| C(1)-S-N(1)-C(8) | 80.61(16) |
| O(1)-S-C(1)-C(2) | 147.89(17) |
| O(2)-S-C(1)-C(2) | 12.55(19) |
| N(1)-S-C(1)-C(2) | -99.89(17) |
| O(1)-S-C(1)-C(6) | -34.19(17) |
| O(2)-S-C(1)-C(6) | -169.54(15) |
| N(1)-S-C(1)-C(6) | 78.03(16) |
| C(6)-C(1)-C(2)-C(3) | 1.1(3) |
| S-C(1)-C(2)-C(3) | 178.97(19) |
| C(1)-C(2)-C(3)-C(4) | -0.3(4) |
| C(2)-C(3)-C(4)-C(5) | -0.7(4) |
| C(2)-C(3)-C(4)-C(7) | 176.7(3) |
| C(3)-C(4)-C(5)-C(6) | 0.9(3) |
| C(7)-C(4)-C(5)-C(6) | -176.5(3) |
| C(4)-C(5)-C(6)-C(1) | -0.2(3) |
| C(2)-C(1)-C(6)-C(5) | -0.8(3) |
| S-C(1)-C(6)-C(5) | -178.75(16) |
| C(12)-N(1)-C(8)-C(9) | 10.5(3) |
| S-N(1)-C(8)-C(9) | -158.56(15) |
| N(1)-C(8)-C(9)-C(13) | -174.17(17) |
| N(1)-C(8)-C(9)-C(10) | 7.9(3) |
| C(8)-C(9)-C(10)-C(11) | -23.9(3) |
| C(13)-C(9)-C(10)-C(11) | 158.08(16) |
| C(8)-C(9)-C(10)-C(17) | 99.9(2) |
| C(13)-C(9)-C(10)-C(17) | -78.1(2) |
| C(9)-C(10)-C(11)-C(12) | 24.6(3) |
| C(17)-C(10)-C(11)-C(12) | -98.7(2) |
| C(9)-C(10)-C(11)-C(15) | -156.34(18) |
| C(17)-C(10)-C(11)-C(15) | 80.4(2) |
| C(15)-C(11)-C(12)-N(1) | 171.59(18) |
| C(10)-C(11)-C(12)-N(1) | -9.4(3) |
| C(8)-N(1)-C(12)-C(11) | -9.7(3) |
| S-N(1)-C(12)-C(11) | 159.20(15) |
| C(14)-O(3)-C(13)-O(4) | 4.6(3) |
| C(14)-O(3)-C(13)-C(9) | -175.2(2) |
| C(8)-C(9)-C(13)-O(4) | -177.4(2) |
| C(10)-C(9)-C(13)-O(4) | 0.6(3) |
| C(8)-C(9)-C(13)-O(3) | 2.5(3) |
| C(10)-C(9)-C(13)-O(3) | -179.49(16) |
| C(16)-O(6)-C(15)-O(5) | -0.6(3) |
| C(16)-O(6)-C(15)-C(11) | 179.9(2) |
| C(12)-C(11)-C(15)-O(5) | -178.7(2) |
| C(10)-C(11)-C(15)-O(5) | 2.2(3) |
| C(12)-C(11)-C(15)-O(6) | 0.8(3) |
| C(10)-C(11)-C(15)-O(6) | -178.23(16) |
| C(11)-C(10)-C(17)-C(18) | 42.0(3) |
| C(9)-C(10)-C(17)-C(18) | -80.0(3) |
| C(11)-C(10)-C(17)-C(22) | -139.7(2) |
| C(9)-C(10)-C(17)-C(22) | 98.3(2) |
| C(22)-C(17)-C(18)-C(19) | 0.1(4) |
| C(10)-C(17)-C(18)-C(19) | 178.4(2) |
| C(17)-C(18)-C(19)-C(20) | -0.5(4) |
| C(18)-C(19)-C(20)-C(21) | 0.7(4) |
| C(19)-C(20)-C(21)-C(22) | -0.5(4) |
| C(20)-C(21)-C(22)-C(17) | 0.1(4) |
| C(18)-C(17)-C(22)-C(21) | 0.1(3) |
| C(10)-C(17)-C(22)-C(21) | -178.2(2) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23110 [A and deg.].

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