A facile approach to spirocyclic 2-azido indolines via azidation of indoles with ceric ammonium nitrate

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Supporting Information

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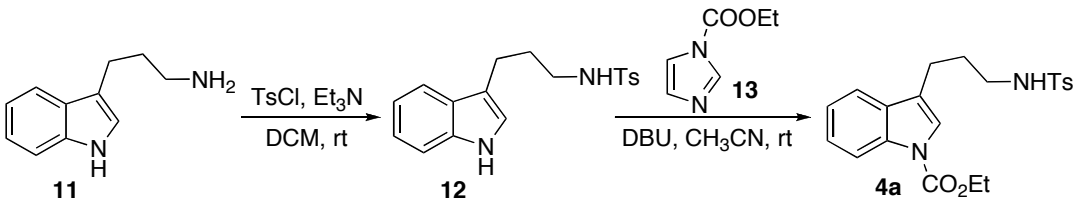
General Methods. All commercially available reagents were used without further purification unless otherwise noted. All solvents were freshly distilled under nitrogen from appropriate drying agents before use. Tetrahydrofuran and ethyl ether were distilled from sodium-benzophenone. Dichloromethane was distilled from CaH_2. Column chromatography was performed on silica gel (200-300 mesh). ^1H NMR spectra were recorded on a 400 MHz NMR spectrometer and ^{13}C NMR spectra were recorded on a 100 MHz NMR spectrometer. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected.

Preparation of compound 4a



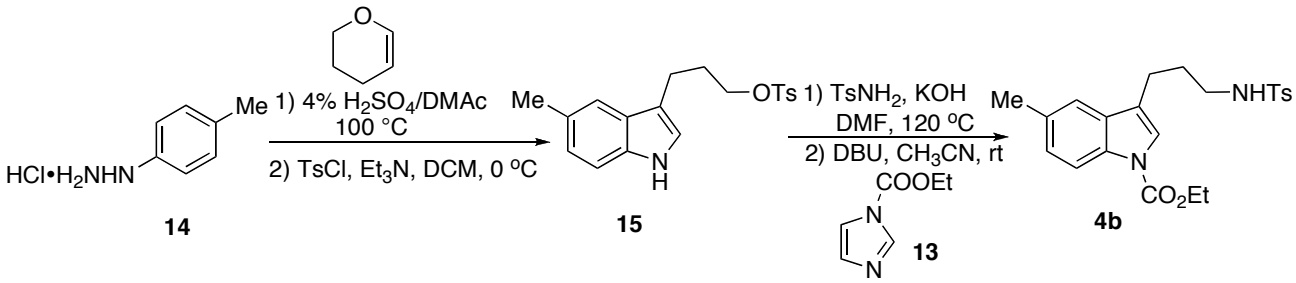
Scheme 4

To a solution of homotryptamine (**11**) (3.10 g, 17.8 mmol) in DCM (89 mL) were added Et_3N (8.2 mL, 58.8 mmol) and TsCl (3.80 g, 19.6 mmol) successively at rt. Upon stirring at rt overnight, the reaction mixture was concentrated and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 2/1) to give compound **12** as white solid (4.00 g, 68% yield).

To a solution of **12** (2.95 g, 9.0 mmol) in CH_3CN (27 mL) were added compound **13** (1.68 g, 12.0 mmol) and DBU (0.273 g, 1.8 mmol) at rt.[1] Upon stirring at rt overnight, the reaction mixture was concentrated and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 8/1-4/1) to give compound **4a** as white solid (2.20 g, 61% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 8.22-8.05 (m, 1H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.37-7.15 (m, 5H), 4.96 (t, *J* = 5.6 Hz, 1H), 4.46 (q, *J* = 6.8 Hz, 2H), 3.02 (q, *J* = 6.4 Hz, 2H), 2.67 (t, *J* = 7.2 Hz, 2H), 2.39 (s, 3H), 1.91-1.82 (m, 2H), 1.45 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 143.6, 137.1, 135.8, 130.5, 129.9, 127.2, 124.7, 122.8, 122.4, 120.5, 119.1, 115.4, 63.2, 42.9, 29.1, 22.0, 21.7, 14.6.

(1) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.

Preparation of compound 4b



Scheme 5

To a solution of *p*-tolyl hydrazine hydrocloride (**14**) (3.16 g, 20.0 mmol) in 4% aqueous H_2SO_4 (30 mL) and *N*,*N*-dimethyl acetamide (30 mL) was added dihydropyran (2.4 mL, 26.0 mmol) dropwise at 100 ^oC.[1] Upon stirring at 100 ^oC for 2 h, the reaction mixture was cooled to rt, extracted with ether (3 x 40 mL), dried over $MgSO_4$, filtered, concentrated, and dissolved in DCM (40 mL). To the resulting solution were added Et_3N (7.0 mL, 50.0 mmol) and TsCl (1.90 g, 10.0 mmol) at 0 ^oC.[2] Upon stirring at 0 ^oC overnight, the reaction mixture was concentrated and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 4/1) to give tosylate **15** as yellow oil (0.665 g, 19% yield).

To a solution of $TsNH_2$ (0.410 g, 2.4 mmol) in DMF (6 mL) was added KOH (0.134 g, 2.4 mmol). After the reaction mixture was stirred at 120 ^oC for 40 min, a solution of tosylate **15** (0.665 g, 1.96 mmol) in DMF (6 mL) was added at 120 ^oC via syringe pump over 30 min. Upon stirring at 120 ^oC for 2 h, the reaction mixture was cooled to rt, extracted with ether (3 x 10 mL), washed with water (3 x 8 mL), dried over $MgSO_4$, filtered, concentrated, and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 2/1) to give the sulfonamide as yellow solid (0.445 g, 66% yield).

To a solution of the above yellow solid (0.445 g, 1.3 mmol) in CH_3CN (6 mL) were added compound **13** (0.237 g, 1.69 mmol) and DBU (0.197 g, 1.3 mmol) at rt.[3] Upon stirring at rt overnight, the reaction mixture was concentrated and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 4/1) to give compound **4b** as white solid (0.337 g, 63% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 8.05-7.93 (m, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.33-7.24 (m,

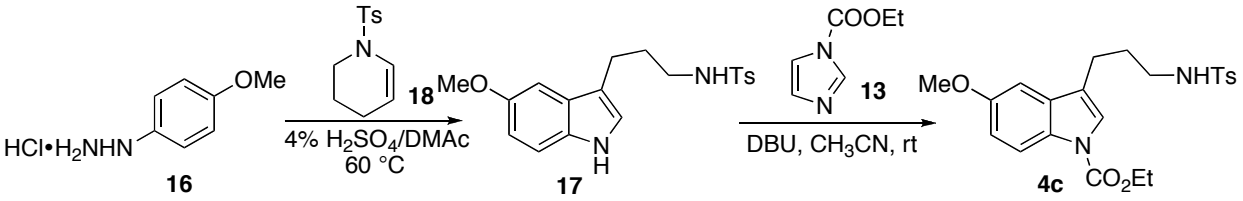
3H), 7.22 (s, 1H), 7.13 (d, J = 8.4 Hz, 1H), 4.88 (t, J = 6.4 Hz, 1H), 4.45 (q, J = 7.2 Hz, 2H), 3.02 (q, J = 6.8 Hz, 2H), 2.65 (t, J = 7.6 Hz, 2H), 2.43 (s, 3H), 2.40 (s, 3H), 1.94-1.80 (m, 2H), 1.45 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 143.6, 137.1, 134.0, 132.3, 130.7, 129.9, 127.2, 126.1, 122.4, 120.2, 119.0, 115.1, 63.1, 43.0, 29.1, 22.0, 21.7, 21.5, 14.6.

(1) K. R. Campos, J. C. S. Woo, S. Lee and R. D. Tillyer, *Org. Lett*., 2004, **6**, 79.

(2) A. L. Beck, M. Mascal, C. J. Moody and W. J. Coates, *J. Chem. Soc., Perkin Trans. 1,* 1992, 813.

(3) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.

Preparation of compound 4c



Scheme 6

To a mixture of 4-methoxy phenyl hydrazine hydrochloride (**16**) (0.454 g, 2.6 mmol) and compound **18**[1] (0.474 g, 2.0 mmol) were added 4% aqueous H_2SO_4 (3 mL) and *N*,*N*-dimethyl acetamide (3 mL) at 60 ^{o}C.[2] Upon stirring at 60 ^{o}C for 2 h, the reaction mixture was cooled to rt, extracted with DCM (3 x 6 mL), washed with water (3 x 6 mL), dried over $MgSO_4$, filtered, concentrated, and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 2/1) to give compound **17** as yellow oil (0.688 g, 99% yield).

To a solution of **17** (0.684 g, 2.0 mmol) in CH_3CN (6 mL) were added compound **13** (0.364 g, 2.6 mmol) and DBU (0.304 g, 2.0 mmol) at rt.[3] Upon stirring at rt overnight, the reaction mixture was concentrated and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 4/1) to give compound **4c** as yellow solid (0.375 g, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08-7.94 (m, 1H), 7.72 (d, J = 8.4 Hz, 2H), 7.31 (s, 1H), 7.27 (d, J = 7.2 Hz, 2H), 6.93 (dd, J = 8.8, 2.4 Hz, 1H), 6.88 (d, J = 2.0 Hz, 1H), 4.60 (t, J = 6.4 Hz, 1H), 4.45 (q, J = 7.2 Hz, 2H), 3.85 (s, 3H), 3.03 (q, J = 6.8 Hz, 2H), 2.66 (t, J = 7.6 Hz, 2H), 2.41 (s, 3H),

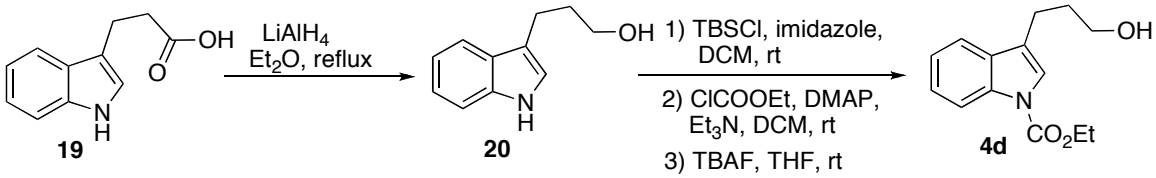
1.94-1.80 (m, 2H), 1.45 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 150.9, 143.4, 136.9, 131.3, 130.2, 129.7, 127.1, 122.8, 120.3, 115.9, 113.0, 101.9, 63.0, 55.7, 42.8, 28.7, 21.9, 21.5, 14.5.

(1) S. Yamasaki, M. Kanai and M. Shibasaki, *J. Am. Chem. Soc*., 2001, **123**, 1256.

(2) K. R. Campos, J. C. S. Woo, S. Lee and R. D. Tillyer, *Org. Lett*., 2004, **6**, 79.

(3) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.

Preparation of compound 4d



Scheme 7

To a solution of 3-indolepropanoic acid (**19**) (3.0 g, 15.8 mmol) in dry ethyl ether (100 mL) was added $LiAlH_4$ (1.5 g, 39.5 mmol) portionwise at reflux.[1] Upon stirring at reflux overnight, the reaction mixture was quenched with saturated NH_4Cl, filtered, extracted with ethyl ether (3 x 50 mL), dried over $MgSO_4$, filtered, and concentrated to give crude alcohol **20** (1.7 g), which was dissolved in DCM (80 mL), followed by addition of imidazole (1.32 g, 19.4 mmol) and TBSCl (2.2 g, 14.6 mmol) at rt.[2] Upon stirring at rt for 2 h, the reaction mixture was washed with water, brine, dried over $MgSO_4$, filtered, and concentrated to give the crude TBS ether (2.8 g), which was dissolved in DCM (80 mL). To the resulting solution were added DMAP (1.2 g, 9.7 mmol), Et_3N (6.7 mL, 48.5 mmol), and ClCOOEt (4.6 mL, 48.5 mmol) at rt, successively. Upon stirring at rt overnight, the reaction mixture was concentrated and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 60/1) to give the carbamate as colorless liquid (0.707 g, 12% yield over three steps).

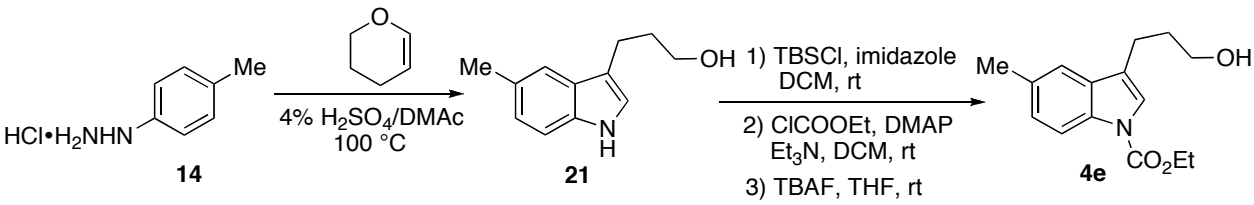
To a solution of the above carbamate (0.707 g, 1.96 mmol) in THF (30 mL) was added TBAF (1.0 M in THF) (2.3 mL, 2.3 mmol) at rt. Upon stirring at rt for 6 h, the reaction mixture was quenched with water, extracted with ethyl ether (3 x 30 mL), washed with water, dried over

$MgSO_4$, filtered, concentrated, and purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate = 2/1) to give alcohol **4d** as colorless liquid (0.420 g, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22-8.10 (m, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.40 (s, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.24 (t, *J* = 7.2 Hz, 1H), 4.46 (q, *J* = 7.2 Hz, 2H), 3.72 (t, *J* = 6.4 Hz, 2H), 2.78 (t, *J* = 7.6 Hz, 2H), 2.03-1.92 (m, 3H), 1.45 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.2, 135.8, 130.8, 124.7, 122.8, 122.2, 121.4, 119.2, 115.4, 63.2, 62.3, 32.0, 21.2, 14.6.

(1) M. G. Mamolo, D. Zampieri, C. Zanette, C. Florio, S. Collina, M. Urbano, O. Azzolina and L. Vio, *Eur. J. Med, Chem*., 2008, **43**, 2073.

(2) Q. Li, G. Li, S. Ma, P. Feng and Y. Shi, *Org. Lett*., 2013, **15**, 2601.

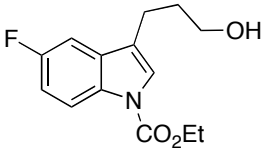
Preparation of compound 4e



Scheme 8

Indole **21** was prepared from *p*-tolyl hydrazine hydrocloride as described in Scheme 5, and was converted to alcohol **4e** in a manner similar to alcohol **4d** as shown in Scheme 7.

4e: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10-7.96 (m, 1H), 7.37 (s, 1H), 7.32 (s, 1H), 7.15 (d, *J* = 8.4 Hz, 1H), 4.46 (q, *J* = 7.2 Hz, 2H), 3.75 (t, *J* = 6.4 Hz, 2H), 2.77 (t, *J* = 7.6 Hz, 2H), 2.46 (s, 3H), 2.04-1.92 (m, 2H), 1.46 (t, *J* = 7.2 Hz, 3H), 1.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.9, 133.6, 131.8, 130.7, 125.5, 121.7, 121.0, 118.8, 114.6, 62.7, 61.7, 31.7, 21.1, 20.9, 14.1.

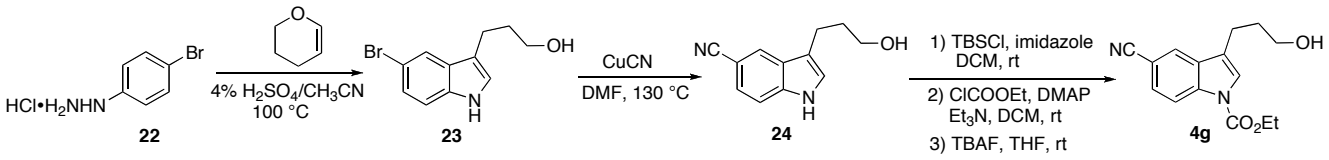


4f

Prepared in a manner similar to **4e**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20-8.00 (m, 1H), 7.44 (s, 1H), 7.18 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.03 (td, *J* = 9.2, 2.8 Hz, 1H), 4.46 (q, *J* = 7.2 Hz, 2H), 3.80-3.68 (m, 2H), 2.74 (t, *J* = 7.6 Hz, 2H), 2.03-1.90 (m, 2H), 1.56 (s, 1H), 1.46 (t, *J* = 7.2 Hz,

3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 158.3, 151.0, 131.8, 123.8, 121.2, 121.1, 116.4, 116.3, 112.5, 112.2, 105.0, 104.8, 63.4, 62.3, 32.0, 21.2, 14.6.

Preparation of compound 4g

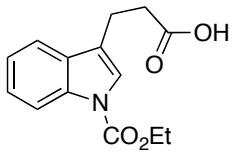


Scheme 9

Indole **23** was prepared from 4-bromo phenyl hydrazine hydrocloride using a Fischer indole synthesis as described in Scheme 5. Compound **23** was converted to **24** according to the reported procedure.[1] Alcohol **4g** was prepared from **24** using the same reaction sequence from **20** to **4d** as described in Scheme 7.

4g: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.4 Hz, 1H), 7.87 (s, 1H), 7.56 (dd, *J* = 8.8, 1.6 Hz, 1H), 7.51 (s, 1H), 4.50 (q, *J* = 7.2 Hz, 2H), 3.74 (t, *J* = 6.4 Hz, 2H), 2.80 (t, *J* = 7.6 Hz, 2H), 2.02-1.92 (m, 2H), 1.65 (br s, 1H), 1.47 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 137.7, 131.0, 127.9, 124.4, 124.2, 121.3, 119.9, 116.3, 106.2, 63.9, 62.1, 32.1, 21.0, 14.6.

(1) N. T. Hatzenbuhler, R. Baudy, D. A. Evrard, A. Failli, B. L. Harrison, S. Lenicek, R. E. Mewshaw, A. Saab, U. Shah, J. Sze, M. Zhang, D. Zhou, M. Chlenov, M. Kagan, J. Golembieski, G. Hornby, M. Lai, D. L. Smith, K. M. Sullivan, L. E. Schechter, T. H. Andree, *J. Med. Chem*., 2008, **51**, 6980.

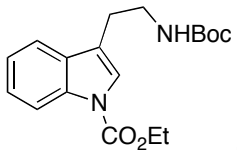


4h

Compound **4h** was prepared from 3-indolepropanoic acid according to the reported procedure.[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.86 (br s, 1H), 8.16 (d, *J* = 5.2 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.44 (s, 1H), 7.35 (t, *J* = 8.0 Hz, 1H), 7.30-7.22 (m, 1H), 4.48 (q, *J* = 7.2 Hz, 2H), 3.05 (t, *J* = 7.6 Hz, 2H), 2.80 (t, *J* = 8.0 Hz, 2H), 1.47 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ

173.9, 150.4, 134.9, 130.1, 124.6, 122.8, 122.2, 120.4, 119.3, 114.8, 63.1, 33.3, 19.8, 14.3.

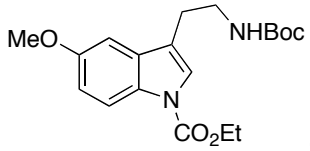
(1) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.



4i

Compound **4i** was prepared from trytamine according to the reported procedure.[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23-8.09 (m, 1H), 7.54 (d, *J* = 7.6 Hz, 1H), 7.45 (s, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 4.69 (br s, 1H), 4.47 (q, *J* = 7.2 Hz, 2H), 3.46 (q, *J* = 6.4 Hz, 2H), 2.90 (t, *J* = 6.8 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 151.1, 135.8, 130.6, 124.8, 122.9, 119.2, 118.6, 115.4, 79.4, 63.2, 40.3, 28.6, 25.7, 14.6.

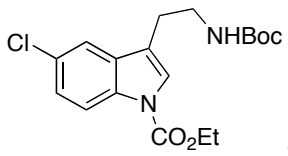
(1) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.



4j

Compound **4j** was prepared from 5-methoxytrytamine according to the reported procedure.[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15-7.95 (m, 1H), 7.43 (s, 1H), 7.02-6.91 (m, 2H), 4.63 (br s, 1H), 4.46 (q, *J* = 7.2 Hz, 2H), 3.87 (s, 3H), 3.52-3.40 (m, 2H), 2.86 (t, *J* = 6.8 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.04, 155.98, 150.9, 131.4, 130.3, 123.4, 118.4, 116.0, 113.1, 101.9, 79.2, 63.0, 55.7, 40.0, 28.5, 25.6, 14.5.

(1) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.



4k

Compound **4k** was prepared from 5-chlorotrytamine hydrochloride according to the reported procedure.[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14-8.04 (m, 1H), 7.50 (d, *J* = 1.6 Hz, 1H), 7.47 (s, 1H), 7.29 (dd, *J* = 8.8, 2.0 Hz, 1H), 4.61 (br s, 1H), 4.48 (q, *J* = 7.2 Hz, 2H), 3.60-3.40 (m, 2H),

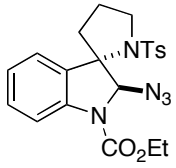
2.86 (t, J = 6.8 Hz, 2H), 1.46 (t, J = 7.2 Hz, 3H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 150.5, 133.8, 131.7, 128.4, 124.6, 124.0, 118.6, 118.1, 116.2, 79.2, 63.3, 40.2, 28.4, 25.4, 14.3.

(1) S. T. Heller, E. E. Schultz and R. Sarpong, *Angew. Chem. Int. Ed*., 2012, **51**, 8304.

Representative procedure for Table 2 (entry 1). To a suspension of **4a** (0.200 g, 0.50 mmol) and NaN_3 (0.049 g, 0.75 mmol) in acetonitrile (5 mL) was added a solution of ceric ammonium nitrate (0.08 M in acetonitrile) (19.0 mL, 1.50 mmol) under N_2 at 0 ^oC via syringe pump over 60 min. Upon stirring at 0 ^oC for 2 h, the reaction mixture was quenched with water (30 mL), extracted with Et_2O (3 x 30 mL), washed with water, dried over $MgSO_4$, filtered, concentrated, and purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 8/1-4/1) to give **5a** as white solid (major isomer 0.140 g, 63% yield, minor isomer 0.0453 g, 20% yield).

Representative procedure for Table 2 (entry 9). To a suspension of **4i** (0.166 g, 0.50 mmol) and NaN_3 (0.098 g, 1.50 mmol) in acetonitrile (5 mL) was added a solution of ceric ammonium nitrate (0.08 M in acetonitrile) (37.5 mL, 3.0 mmol) under N_2 at 0 ^oC via syringe pump over 60 min. Upon stirring at 0 ^oC for 3 h, the reaction mixture was quenched with water (60 mL), extracted with Et_2O (3 x 30 mL), washed with water (3 x 20 mL), dried over $MgSO_4$, filtered, concentrated, and purified by column chromatography (silica gel, dichloromethane/ethyl acetate = 5/1) to give **5i** as white solid (0.0781 g, 44% yield).

Table 2, entry 1

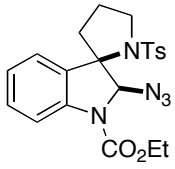


5a

(X-ray structure)

Major isomer: white solid; mp. 108-110 ^oC; IR (film) 2114, 1722, 1488 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.80 (m, 1H), 7.27 (t, J = 7.6 Hz, 1H), 7.05-6.93 (m, 4H), 6.64 (t, J

= 6.8 Hz, 1H), 6.55-6.35 (m, 1H), 6.24 (s, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 4.02-3.90 (m, 1H), 3.52-3.42 (m, 1H), 2.88-2.78 (m, 1H), 2.34 (s, 3H), 2.10-1.96 (m, 3H), 1.44 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 142.6, 142.4, 136.8, 129.8, 128.9, 128.5, 126.6, 124.1, 122.9, 115.6, 84.8, 73.5, 62.4, 50.0, 37.5, 24.0, 21.3, 14.4; HRMS Calcd for $C_{21}H_{23}N_5NaO_4S$ (M+Na): 464.1363; Found: 464.1352.

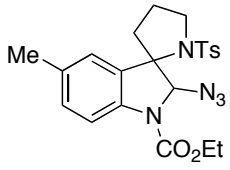


5a

(X-ray structure)

Minor isomer: white solid; mp. 49-51 °C; IR (film) 2113, 1720, 1486 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03-7.67 (m, 1H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.35-7.27 (m, 1H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.10-6.94 (m, 2H), 5.33 (br s, 1H), 4.50-4.30 (m, 2H), 3.69 (t, *J* = 8.0 Hz, 1H), 3.62-3.53 (m, 1H), 2.42 (s, 3H), 2.25-1.93 (m, 4H), 1.43 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.0, 143.2, 140.4, 138.1, 130.2, 129.7, 129.3, 127.6, 124.1, 123.3, 115.1, 83.3, 75.1, 62.8, 49.4, 44.2, 22.6, 21.6, 14.6; HRMS Calcd for $C_{21}H_{23}N_5NaO_4S$ (M+Na): 464.1363; Found: 464.1351.

Table 2, entry 2

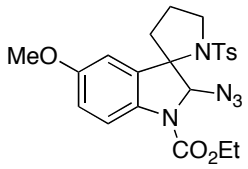


5b

Major isomer: white solid; mp. 118-120 °C; IR (film) 2115, 1721, 1498 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.70 (m, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 7.02-6.89 (m, 4H), 6.22 (s, 1H), 6.11 (s, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 4.09-3.90 (m, 1H), 3.55-3.45 (m, 1H), 2.89-2.72 (m, 1H), 2.35 (s, 3H), 2.09-1.98 (m, 3H), 1.95 (s, 3H), 1.43 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.0, 142.6, 140.5, 137.2, 132.5, 130.7, 129.0, 128.4, 126.9, 124.9, 115.7, 85.2, 73.6, 62.5, 50.5, 37.7, 24.2, 21.4, 20.6, 14.6; HRMS Calcd for $C_{22}H_{25}N_5NaO_4S$ (M+Na): 478.1520; Found: 478.1505.

Minor isomer: white solid; mp. 138-140 °C; IR (film) 2114, 1719, 1497 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90-7.60 (m, 1H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 1H), 6.67 (s, 1H), 5.32 (br s, 1H), 4.49-4.28 (m, 2H), 3.76 (t, *J* = 7.2 Hz, 1H), 3.65-3.48 (m, 1H), 2.42 (s, 3H), 2.24 (s, 3H), 2.19-1.98 (m, 4H), 1.41 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.0, 143.1, 138.4, 132.5, 130.5, 129.9, 129.2, 127.5, 127.2, 124.8, 114.9, 83.5, 74.9, 62.7, 49.5, 44.3, 22.6, 21.6, 21.1, 14.7; HRMS Calcd for $C_{22}H_{25}N_5NaO_4S$ (M+Na): 478.1520; Found: 478.1510.

Table 2, entry 3

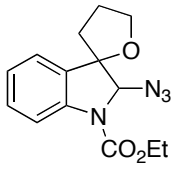


5c

Major isomer: white solid; mp. 113-115 °C; IR (film) 2114, 1717, 1498 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.6 Hz, 1H), 7.08-6.93 (m, 4H), 6.79 (dd, *J* = 8.8, 2.0 Hz, 1H), 6.23 (br s, 1H), 5.81 (s, 1H), 4.53-4.30 (m, 2H), 4.06-3.92 (m, 1H), 3.55-3.47 (m, 1H), 3.45 (s, 3H), 2.88-2.78 (m, 1H), 2.34 (s, 3H), 2.08-1.95 (m, 3H), 1.42 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 152.9, 142.8, 137.2, 136.4, 129.4, 129.1, 126.8, 116.8, 116.0, 109.1, 85.2, 73.7, 62.5, 55.1, 50.6, 37.6, 24.2, 21.5, 14.6; HRMS Calcd for $C_{22}H_{25}N_5NaO_5S$ (M+Na): 494.1469; Found: 494.1454.

Minor isomer: white solid; mp. 156-158 °C; IR (film) 2114, 1717, 1498 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.97-7.69 (m, 1H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.84 (dd, *J* = 9.2, 2.8 Hz, 1H), 6.54 (br s, 1H), 5.32 (s, 1H), 4.49-4.26 (m, 2H), 3.81-3.66 (m, 1H), 3.71 (s, 3H), 3.64-3.52 (m, 1H), 2.42 (s, 3H), 2.27-1.94 (m, 4H), 1.41 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 143.3, 138.3, 129.4, 129.3, 127.7, 127.6, 116.0, 115.3, 109.7, 83.5, 75.2, 62.7, 55.7, 49.5, 44.2, 22.7, 21.7, 14.7; HRMS Calcd for $C_{22}H_{25}N_5NaO_5S$ (M+Na): 494.1469; Found: 494.1472.

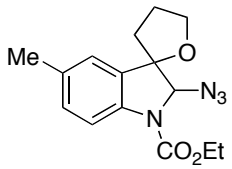
Table 2, entry 4



5d

Major and minor isomers: yellow oil; IR (film) 2111, 1720, 1484 cm^{-1}; Major: ^1H NMR (400 MHz, $CDCl_3$) δ 7.97-7.78 (m, 1H), 7.36-7.27 (m, 1H), 7.25-7.20 (m, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 5.58 (s, 1H), 4.42-4.26 (m, 2H), 4.04-3.88 (m, 2H), 2.63-2.46 (m, 1H), 2.26-2.03 (m, 3H), 1.38 (t, *J* = 7.2 Hz, 3H); Major and minor isomers: ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9, 152.4, 141.4, 139.6, 132.8, 131.4, 130.5, 129.7, 124.2, 123.8, 123.5, 123.2, 115.6, 115.1, 89.2, 88.5, 82.7, 82.0, 70.1, 68.2, 62.6, 62.5, 39.5, 30.6, 26.8, 25.4, 14.52, 14.47; HRMS Calcd for $C_{14}H_{16}N_4NaO_3$ (M+Na): 311.1115; Found: 311.1119.

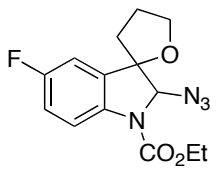
Table 2, entry 5



5e

Major and minor isomers: yellow oil; IR (film) 2110, 1722, 1495 cm^{-1}; Major: ^1H NMR (400 MHz, $CDCl_3$) δ 7.84-7.73 (m, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.06 (s, 1H), 5.59 (br s, 1H), 4.45-4.28 (m, 2H), 4.07-3.90 (m, 2H), 2.61-2.52 (m, 1H), 2.33 (s, 3H), 2.27-2.05 (m, 3H), 1.40 (t, *J* = 7.2 Hz, 3H); Major and minor isomers: ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 152.1, 139.2, 137.2, 133.7, 133.3, 132.7, 131.2, 131.0, 130.1, 123.8, 123.5, 115.2, 114.7, 89.1, 88.4, 82.7, 82.05, 82.02, 69.9, 68.1, 62.3, 39.2, 30.4, 26.7, 25.3, 20.91, 20.89, 14.4, 14.3; HRMS Calcd for $C_{15}H_{18}N_4NaO_3$ (M+Na): 325.1271; Found: 325.1268.

Table 2, entry 6



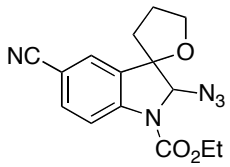
5f

Major isomer: yellow oil; IR (film) 2113, 1721, 1491 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.00-7.67 (m, 1H), 7.10-6.98 (m, 1H), 6.94 (dd, *J* = 8.0, 2.4 Hz, 1H), 5.61 (br s, 1H), 4.45-4.24 (m, 2H), 4.05-3.87 (m, 2H), 2.66-2.50 (m, 1H), 2.30-2.04 (m, 3H), 1.39 (t, *J* = 7.2

Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 158.3, 152.4, 137.6, 133.4, 117.2, 117.0, 116.83, 116.75, 110.9, 110.7, 89.0, 83.2, 68.5, 62.8, 30.8, 26.9, 14.6; HRMS Calcd for $C_{14}H_{15}FN_4NaO_3$ (M+Na): 329.1020; Found: 329.1018.

Minor isomer: yellow oil; IR (film) 2113, 1721, 1491 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-7.50 (m, 1H), 7.06-6.89 (m, 2H), 5.24 (br s, 1H), 4.48-4.27 (m, 2H), 4.26-4.10 (m, 2H), 2.34-2.02 (m, 3H), 2.00-1.87 (m, 1H), 1.40 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 158.7, 152.9, 135.6, 134.9, 116.4, 116.34, 116.26, 116.2, 110.7, 110.4, 88.2, 82.5, 70.3, 62.8, 39.5, 25.5, 14.6; HRMS Calcd for $C_{14}H_{15}FN_4NaO_3$ (M+Na): 329.1020; Found: 329.1017.

Table 2, entry 7

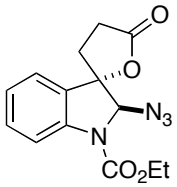


5g

Major isomer: white solid; mp. 151-153 °C; IR (film) 2225, 2114, 1727, 1487 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10-7.75 (m, 1H), 7.62 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.51 (s, 1H), 5.63 (br s, 1H), 4.46-4.32 (m, 2H), 4.05-3.93 (m, 2H), 2.65-2.54 (m, 1H), 2.32-2.09 (m, 3H), 1.41 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.2, 145.0, 135.1, 133.1, 127.8, 118.9, 116.2, 107.0, 88.5, 83.3, 68.7, 63.4, 30.9, 26.9, 14.5; HRMS Calcd for $C_{15}H_{16}N_5O_3$ (M+H): 314.1248; Found: 314.1257.

Minor isomer: white solid; mp. 122-124 °C; IR (film) 2225, 2114, 1725, 1487 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.73 (m, 1H), 7.58 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.53 (d, *J* = 1.6 Hz, 1H), 5.25 (br s, 1H), 4.45-4.31 (m, 2H), 4.28-4.13 (m, 2H), 2.33-2.06 (m, 3H), 1.99-1.89 (m, 1H), 1.41 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 143.4, 134.7, 134.4, 127.3, 118.9, 115.7, 107.5, 87.9, 82.3, 70.5, 63.4, 39.8, 25.5, 14.5; HRMS Calcd for $C_{15}H_{16}N_5O_3$ (M+H): 314.1248; Found: 314.1254.

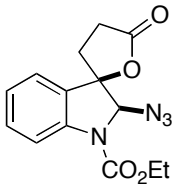
Table 2, entry 8



5h

(X-ray structure)

Major isomer: white solid; mp. 78-80 °C; IR (film) 2118, 1782, 1724, 1485 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.12-7.70 (m, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 7.6 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 5.87 (br s, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 2.95-2.74 (m, 3H), 2.64-2.53 (m, 1H), 1.42 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.9, 151.8, 142.0, 132.0, 128.2, 124.2, 123.8, 115.9, 90.1, 82.0, 63.0, 29.4, 26.9, 14.4; HRMS Calcd for $C_{14}H_{14}N_4NaO_4$ (M+Na): 325.0907; Found: 325.0906.

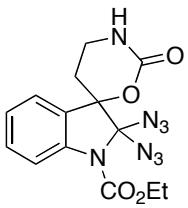


5h

(X-ray structure)

Minor isomer: white solid; mp. 102-104 °C; IR (film) 2117, 1782, 1724, 1485 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.84-7.70 (m, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 7.6 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 5.50 (s, 1H), 4.48-4.33 (m, 2H), 2.86-2.72 (m, 2H), 2.57-2.48 (m, 1H), 2.47-2.34 (m, 1H), 1.43 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.0, 152.9, 139.9, 131.3, 129.1, 124.6, 123.4, 115.5, 88.0, 81.2, 63.2, 35.0, 28.2, 14.6; HRMS Calcd for $C_{14}H_{14}N_4NaO_4$ (M+Na): 325.0907; Found: 325.0910.

Table 2, entry 9



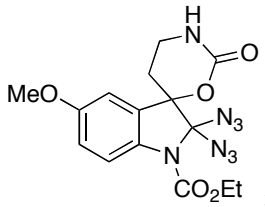
5i

(X-ray structure)

White solid; mp. 146-148 °C; IR (film) 2135, 1725, 1485 cm^{-1}; ^1H NMR (400 MHz,

$CDCl_3$) δ 7.88 (d, *J* = 8.0 Hz, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.15 (br s, 1H), 4.57-4.41 (m, 2H), 3.61-3.44 (m, 2H), 2.52-2.37 (m, 2H), 1.47 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.1, 152.2, 140.9, 132.1, 126.5, 124.6, 124.1, 115.6, 97.5, 88.1, 63.6, 36.8, 24.2, 14.4; HRMS Calcd for $C_{14}H_{14}N_8NaO_4$ (M+Na): 381.1030; Found: 381.1027.

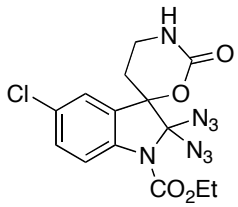
Table 2, entry 10



5j

White solid; mp. 168-170 °C; IR (film) 2136, 1723, 1495 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.73 (m, 1H), 6.96 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.86 (d, *J* = 2.8 Hz, 1H), 6.05 (br s, 1H), 4.54-4.39 (m, 2H), 3.80 (s, 3H), 3.63-3.44 (m, 2H), 2.52-2.33 (m, 2H), 1.46 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 153.0, 152.2, 134.4, 127.5, 117.5, 116.7, 109.5, 97.6, 88.2, 63.5, 56.0, 36.8, 24.3, 14.5; HRMS Calcd for $C_{15}H_{16}N_8NaO_5$ (M+Na): 411.1136; Found: 411.1131.

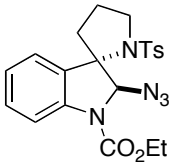
Table 2, entry 11

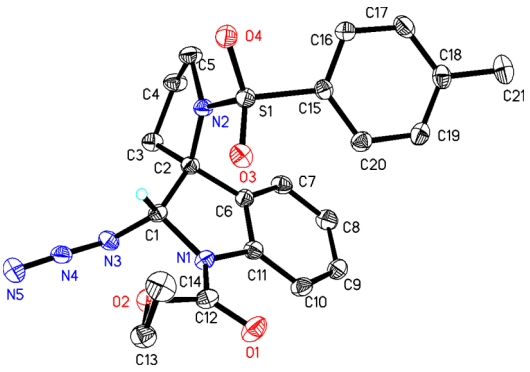


5k

White solid; mp. 123-125 °C; IR (film) 2135, 1725, 1481 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.4 Hz, 1H), 7.40 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.32 (d, *J* = 2.0 Hz, 1H), 5.60 (br s, 1H), 4.56-4.41 (m, 2H), 3.62-3.47 (m, 2H), 2.53-2.33 (m, 2H) 1.47 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.2, 152.0, 139.6, 132.2, 129.8, 128.1, 124.5, 117.1, 97.7, 87.8, 64.0, 37.0, 24.3, 14.5; HRMS Calcd for $C_{14}H_{13}ClN_8NaO_4$ (M+Na): 415.0641; Found: 415.0644.

The X-ray structure of compound *anti*-**5a**





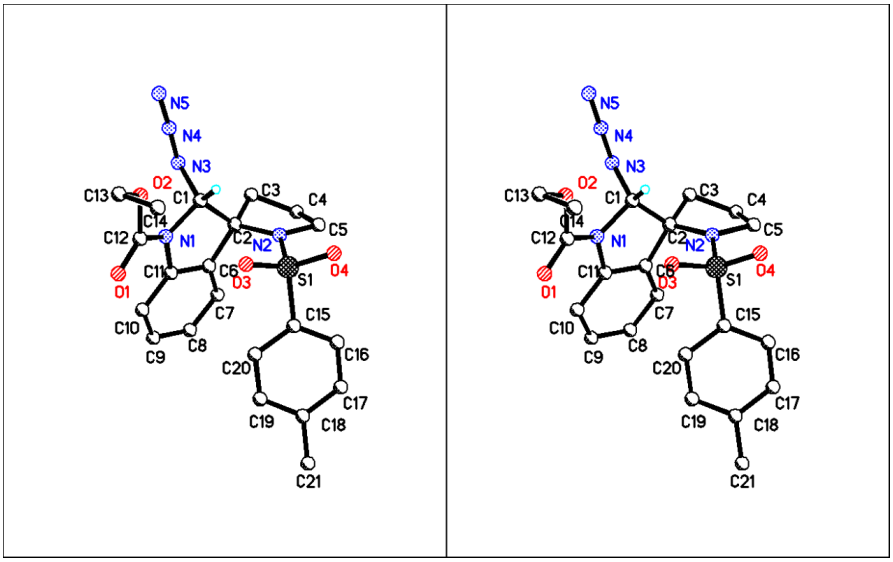


Table 1. Crystal data and structure refinement for *anti*-**5a**.

Identification code	*anti*-**5a**
Empirical formula	$C_{21}H_{23}N_5O_4S$
Formula weight	441.50
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 18.126(5) A alpha = 90 deg. b = 14.074(4) A beta = 95.045(4) deg. c = 16.468(6) A gamma = 90 deg.
Volume	4185(2) A^3
Z, Calculated density	8, 1.402 Mg/m^3
Absorption coefficient	0.194 mm^-1
F(000)	1856
Crystal size	0.37 x 0.26 x 0.13 mm
Theta range for data collection	2.48 to 27.48 deg.
Limiting indices	-23<=h<=23, -18<=k<=18, -21<=l<=21
Reflections collected / unique	30549 / 9533 [R(int) = 0.0648]
Completeness to theta = 27.48	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6838
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	9533 / 0 / 563
Goodness-of-fit on F^2	1.242
Final R indices [I>2sigma(I)]	R1 = 0.0834, wR2 = 0.1577
R indices (all data)	R1 = 0.0995, wR2 = 0.1659
Largest diff. peak and hole	0.388 and -0.405 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for *anti*-**5a**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S(1)	506(1)	4468(1)	3802(1)	30(1)
S(2)	4278(1)	2813(1)	1527(1)	31(1)
O(1)	-1586(2)	2905(2)	2342(1)	47(1)
O(2)	-1948(1)	3315(2)	3583(1)	36(1)
O(3)	-28(1)	3732(2)	3635(1)	37(1)
O(4)	993(1)	4440(2)	4537(1)	40(1)
O(5)	6421(2)	4558(2)	2712(2)	59(1)
O(6)	6706(1)	3958(2)	1504(2)	48(1)
O(7)	3813(1)	2820(2)	776(1)	41(1)
O(8)	4783(1)	3582(2)	1716(1)	39(1)
N(1)	-1408(1)	4400(2)	2855(1)	30(1)
N(2)	63(1)	5468(2)	3833(1)	29(1)
N(3)	-1967(1)	5526(2)	3712(2)	33(1)
N(4)	-2331(1)	5190(2)	4244(2)	35(1)
N(5)	-2717(2)	4957(2)	4712(2)	49(1)
N(6)	6263(1)	2987(2)	2402(1)	32(1)
N(7)	4769(1)	1853(2)	1506(1)	30(1)
N(8)	6759(2)	1707(2)	1629(2)	41(1)
N(9)	7171(2)	2032(2)	1138(2)	41(1)
N(10)	7593(2)	2235(3)	709(2)	60(1)
C(1)	-1283(2)	5001(2)	3582(2)	27(1)
C(2)	-666(2)	5708(2)	3376(2)	29(1)
C(3)	-774(2)	6731(2)	3683(2)	34(1)
C(4)	6(2)	7132(2)	3831(2)	43(1)
C(5)	468(2)	6313(2)	4161(2)	40(1)
C(6)	-670(2)	5606(2)	2455(2)	30(1)
C(7)	-339(2)	6152(2)	1892(2)	37(1)
C(8)	-408(2)	5888(3)	1075(2)	43(1)
C(9)	-801(2)	5076(3)	830(2)	42(1)
C(10)	-1144(2)	4525(2)	1381(2)	38(1)
C(11)	-1078(2)	4813(2)	2192(2)	31(1)
C(12)	-1639(2)	3481(2)	2878(2)	35(1)
C(13)	-2109(2)	2322(2)	3743(2)	38(1)
C(14)	-1439(2)	1858(3)	4155(2)	49(1)
C(15)	1077(2)	4505(2)	2983(2)	29(1)
C(16)	1789(2)	4857(2)	3112(2)	40(1)
C(17)	2230(2)	4878(3)	2465(2)	43(1)
C(18)	1979(2)	4536(2)	1699(2)	37(1)
C(19)	1263(2)	4185(2)	1590(2)	38(1)
C(20)	811(2)	4164(2)	2222(2)	34(1)

C(21)	2476(2)	4542(3)	1016(2)	53(1)
C(22)	6103(2)	2298(2)	1745(2)	30(1)
C(23)	5467(2)	1655(2)	2038(2)	28(1)
C(24)	5549(2)	580(2)	1919(2)	35(1)
C(25)	5159(2)	370(2)	1084(2)	41(1)
C(26)	4477(2)	994(2)	1066(2)	40(1)
C(27)	5456(2)	1945(2)	2919(2)	30(1)
C(28)	5072(2)	1534(3)	3518(2)	41(1)
C(29)	5144(2)	1934(3)	4295(2)	51(1)
C(30)	5591(2)	2713(3)	4457(2)	50(1)
C(31)	5985(2)	3124(2)	3872(2)	41(1)
C(32)	5911(2)	2720(2)	3097(2)	33(1)
C(33)	6459(2)	3909(2)	2246(2)	40(1)
C(34)	6798(2)	4910(3)	1162(3)	62(1)
C(35)	6252(3)	5024(3)	446(3)	70(1)
C(36)	3687(2)	2736(2)	2325(2)	31(1)
C(37)	3040(2)	2213(2)	2211(2)	36(1)
C(38)	2589(2)	2139(2)	2845(2)	40(1)
C(39)	2767(2)	2593(2)	3589(2)	36(1)
C(40)	3417(2)	3117(2)	3683(2)	41(1)
C(41)	3879(2)	3194(2)	3060(2)	36(1)
C(42)	2268(2)	2511(3)	4274(2)	50(1)

Table 3. Bond lengths [A] and angles [deg] for *anti*-**5a**.

S(1)-O(3)	1.428(2)
S(1)-O(4)	1.435(2)
S(1)-N(2)	1.622(2)
S(1)-C(15)	1.772(3)
S(2)-O(7)	1.434(2)
S(2)-O(8)	1.434(2)
S(2)-N(7)	1.622(3)
S(2)-C(36)	1.770(3)
O(1)-C(12)	1.208(4)
O(2)-C(12)	1.353(4)
O(2)-C(13)	1.457(4)
O(5)-C(33)	1.198(4)
O(6)-C(33)	1.339(4)
O(6)-C(34)	1.468(4)
N(1)-C(12)	1.360(4)
N(1)-C(11)	1.416(4)
N(1)-C(1)	1.467(3)
N(2)-C(5)	1.475(4)
N(2)-C(2)	1.500(3)
N(3)-N(4)	1.235(4)
N(3)-C(1)	1.476(4)
N(4)-N(5)	1.135(4)
N(6)-C(33)	1.376(4)
N(6)-C(32)	1.410(4)
N(6)-C(22)	1.463(4)
N(7)-C(26)	1.483(4)
N(7)-C(23)	1.499(3)
N(8)-N(9)	1.236(4)
N(8)-C(22)	1.479(4)
N(9)-N(10)	1.121(4)
C(1)-C(2)	1.557(4)
C(1)-H(1)	1.0000
C(2)-C(6)	1.523(4)
C(2)-C(3)	1.543(4)
C(3)-C(4)	1.522(4)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(5)	1.499(5)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.382(4)

C(6)-C(11)	1.387(4)
C(7)-C(8)	1.392(4)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.382(4)
C(9)-H(9)	0.9500
C(10)-C(11)	1.391(4)
C(10)-H(10)	0.9500
C(13)-C(14)	1.489(5)
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-C(16)	1.381(4)
C(15)-C(20)	1.388(4)
C(16)-C(17)	1.388(5)
C(16)-H(16)	0.9500
C(17)-C(18)	1.388(5)
C(17)-H(17)	0.9500
C(18)-C(19)	1.385(5)
C(18)-C(21)	1.503(5)
C(19)-C(20)	1.380(4)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
C(21)-H(21A)	0.9800
C(21)-H(21B)	0.9800
C(21)-H(21C)	0.9800
C(22)-C(23)	1.574(4)
C(22)-H(22)	1.0000
C(23)-C(27)	1.509(4)
C(23)-C(24)	1.534(4)
C(24)-C(25)	1.519(4)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900
C(25)-C(26)	1.516(5)
C(25)-H(25A)	0.9900
C(25)-H(25B)	0.9900
C(26)-H(26A)	0.9900
C(26)-H(26B)	0.9900
C(27)-C(32)	1.384(4)
C(27)-C(28)	1.384(4)
C(28)-C(29)	1.394(5)
C(28)-H(28)	0.9500
C(29)-C(30)	1.375(5)
C(29)-H(29)	0.9500

C(30)-C(31)	1.376(5)
C(30)-H(30)	0.9500
C(31)-C(32)	1.393(4)
C(31)-H(31)	0.9500
C(34)-C(35)	1.481(6)
C(34)-H(34A)	0.9900
C(34)-H(34B)	0.9900
C(35)-H(35A)	0.9800
C(35)-H(35B)	0.9800
C(35)-H(35C)	0.9800
C(36)-C(37)	1.384(4)
C(36)-C(41)	1.388(4)
C(37)-C(38)	1.384(4)
C(37)-H(37)	0.9500
C(38)-C(39)	1.394(4)
C(38)-H(38)	0.9500
C(39)-C(40)	1.386(5)
C(39)-C(42)	1.512(5)
C(40)-C(41)	1.384(4)
C(40)-H(40)	0.9500
C(41)-H(41)	0.9500
C(42)-H(42A)	0.9800
C(42)-H(42B)	0.9800
C(42)-H(42C)	0.9800
O(3)-S(1)-O(4)	119.88(14)
O(3)-S(1)-N(2)	107.88(13)
O(4)-S(1)-N(2)	105.47(13)
O(3)-S(1)-C(15)	107.82(14)
O(4)-S(1)-C(15)	106.60(14)
N(2)-S(1)-C(15)	108.81(13)
O(7)-S(2)-O(8)	119.89(14)
O(7)-S(2)-N(7)	105.59(13)
O(8)-S(2)-N(7)	106.99(13)
O(7)-S(2)-C(36)	107.05(14)
O(8)-S(2)-C(36)	107.54(14)
N(7)-S(2)-C(36)	109.53(14)
C(12)-O(2)-C(13)	115.3(2)
C(33)-O(6)-C(34)	117.1(3)
C(12)-N(1)-C(11)	124.2(3)
C(12)-N(1)-C(1)	123.5(2)
C(11)-N(1)-C(1)	110.3(2)
C(5)-N(2)-C(2)	112.9(2)
C(5)-N(2)-S(1)	118.65(19)
C(2)-N(2)-S(1)	126.61(19)
N(4)-N(3)-C(1)	114.8(2)
N(5)-N(4)-N(3)	172.7(3)

C(33)-N(6)-C(32)	123.0(3)
C(33)-N(6)-C(22)	121.7(3)
C(32)-N(6)-C(22)	110.5(2)
C(26)-N(7)-C(23)	112.4(2)
C(26)-N(7)-S(2)	121.1(2)
C(23)-N(7)-S(2)	125.38(19)
N(9)-N(8)-C(22)	114.6(3)
N(10)-N(9)-N(8)	172.0(4)
N(1)-C(1)-N(3)	109.6(2)
N(1)-C(1)-C(2)	104.7(2)
N(3)-C(1)-C(2)	109.9(2)
N(1)-C(1)-H(1)	110.8
N(3)-C(1)-H(1)	110.8
C(2)-C(1)-H(1)	110.8
N(2)-C(2)-C(6)	113.9(2)
N(2)-C(2)-C(3)	100.3(2)
C(6)-C(2)-C(3)	115.1(2)
N(2)-C(2)-C(1)	111.4(2)
C(6)-C(2)-C(1)	102.4(2)
C(3)-C(2)-C(1)	114.2(2)
C(4)-C(3)-C(2)	104.8(2)
C(4)-C(3)-H(3A)	110.8
C(2)-C(3)-H(3A)	110.8
C(4)-C(3)-H(3B)	110.8
C(2)-C(3)-H(3B)	110.8
H(3A)-C(3)-H(3B)	108.9
C(5)-C(4)-C(3)	104.7(3)
C(5)-C(4)-H(4A)	110.8
C(3)-C(4)-H(4A)	110.8
C(5)-C(4)-H(4B)	110.8
C(3)-C(4)-H(4B)	110.8
H(4A)-C(4)-H(4B)	108.9
N(2)-C(5)-C(4)	104.1(2)
N(2)-C(5)-H(5A)	110.9
C(4)-C(5)-H(5A)	110.9
N(2)-C(5)-H(5B)	110.9
C(4)-C(5)-H(5B)	110.9
H(5A)-C(5)-H(5B)	108.9
C(7)-C(6)-C(11)	119.3(3)
C(7)-C(6)-C(2)	130.6(3)
C(11)-C(6)-C(2)	110.1(3)
C(6)-C(7)-C(8)	119.5(3)
C(6)-C(7)-H(7)	120.2
C(8)-C(7)-H(7)	120.2
C(9)-C(8)-C(7)	120.1(3)
C(9)-C(8)-H(8)	119.9

C(7)-C(8)-H(8)	119.9
C(10)-C(9)-C(8)	121.3(3)
C(10)-C(9)-H(9)	119.3
C(8)-C(9)-H(9)	119.3
C(9)-C(10)-C(11)	117.5(3)
C(9)-C(10)-H(10)	121.3
C(11)-C(10)-H(10)	121.3
C(6)-C(11)-C(10)	122.2(3)
C(6)-C(11)-N(1)	109.8(2)
C(10)-C(11)-N(1)	128.0(3)
O(1)-C(12)-O(2)	125.1(3)
O(1)-C(12)-N(1)	125.1(3)
O(2)-C(12)-N(1)	109.8(3)
O(2)-C(13)-C(14)	109.6(3)
O(2)-C(13)-H(13A)	109.8
C(14)-C(13)-H(13A)	109.8
O(2)-C(13)-H(13B)	109.8
C(14)-C(13)-H(13B)	109.8
H(13A)-C(13)-H(13B)	108.2
C(13)-C(14)-H(14A)	109.5
C(13)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(13)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(16)-C(15)-C(20)	120.6(3)
C(16)-C(15)-S(1)	119.6(2)
C(20)-C(15)-S(1)	119.8(2)
C(15)-C(16)-C(17)	118.9(3)
C(15)-C(16)-H(16)	120.6
C(17)-C(16)-H(16)	120.6
C(18)-C(17)-C(16)	121.6(3)
C(18)-C(17)-H(17)	119.2
C(16)-C(17)-H(17)	119.2
C(19)-C(18)-C(17)	118.1(3)
C(19)-C(18)-C(21)	121.4(3)
C(17)-C(18)-C(21)	120.5(3)
C(20)-C(19)-C(18)	121.4(3)
C(20)-C(19)-H(19)	119.3
C(18)-C(19)-H(19)	119.3
C(19)-C(20)-C(15)	119.4(3)
C(19)-C(20)-H(20)	120.3
C(15)-C(20)-H(20)	120.3
C(18)-C(21)-H(21A)	109.5
C(18)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5

C(18)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5
N(6)-C(22)-N(8)	111.0(2)
N(6)-C(22)-C(23)	104.9(2)
N(8)-C(22)-C(23)	109.6(2)
N(6)-C(22)-H(22)	110.4
N(8)-C(22)-H(22)	110.4
C(23)-C(22)-H(22)	110.4
N(7)-C(23)-C(27)	115.5(2)
N(7)-C(23)-C(24)	101.3(2)
C(27)-C(23)-C(24)	113.5(2)
N(7)-C(23)-C(22)	108.5(2)
C(27)-C(23)-C(22)	102.2(2)
C(24)-C(23)-C(22)	116.5(2)
C(25)-C(24)-C(23)	105.3(2)
C(25)-C(24)-H(24A)	110.7
C(23)-C(24)-H(24A)	110.7
C(25)-C(24)-H(24B)	110.7
C(23)-C(24)-H(24B)	110.7
H(24A)-C(24)-H(24B)	108.8
C(26)-C(25)-C(24)	102.6(3)
C(26)-C(25)-H(25A)	111.2
C(24)-C(25)-H(25A)	111.2
C(26)-C(25)-H(25B)	111.2
C(24)-C(25)-H(25B)	111.2
H(25A)-C(25)-H(25B)	109.2
N(7)-C(26)-C(25)	101.9(2)
N(7)-C(26)-H(26A)	111.4
C(25)-C(26)-H(26A)	111.4
N(7)-C(26)-H(26B)	111.4
C(25)-C(26)-H(26B)	111.4
H(26A)-C(26)-H(26B)	109.3
C(32)-C(27)-C(28)	120.4(3)
C(32)-C(27)-C(23)	111.0(3)
C(28)-C(27)-C(23)	128.5(3)
C(27)-C(28)-C(29)	118.3(3)
C(27)-C(28)-H(28)	120.9
C(29)-C(28)-H(28)	120.9
C(30)-C(29)-C(28)	120.3(3)
C(30)-C(29)-H(29)	119.8
C(28)-C(29)-H(29)	119.8
C(29)-C(30)-C(31)	122.3(3)
C(29)-C(30)-H(30)	118.9
C(31)-C(30)-H(30)	118.9
C(30)-C(31)-C(32)	117.1(3)

C(30)-C(31)-H(31)	121.5
C(32)-C(31)-H(31)	121.5
C(27)-C(32)-C(31)	121.6(3)
C(27)-C(32)-N(6)	110.0(2)
C(31)-C(32)-N(6)	128.4(3)
O(5)-C(33)-O(6)	126.0(3)
O(5)-C(33)-N(6)	124.6(3)
O(6)-C(33)-N(6)	109.3(3)
O(6)-C(34)-C(35)	108.4(3)
O(6)-C(34)-H(34A)	110.0
C(35)-C(34)-H(34A)	110.0
O(6)-C(34)-H(34B)	110.0
C(35)-C(34)-H(34B)	110.0
H(34A)-C(34)-H(34B)	108.4
C(34)-C(35)-H(35A)	109.5
C(34)-C(35)-H(35B)	109.5
H(35A)-C(35)-H(35B)	109.5
C(34)-C(35)-H(35C)	109.5
H(35A)-C(35)-H(35C)	109.5
H(35B)-C(35)-H(35C)	109.5
C(37)-C(36)-C(41)	120.6(3)
C(37)-C(36)-S(2)	119.5(2)
C(41)-C(36)-S(2)	119.8(2)
C(36)-C(37)-C(38)	119.1(3)
C(36)-C(37)-H(37)	120.4
C(38)-C(37)-H(37)	120.4
C(37)-C(38)-C(39)	121.5(3)
C(37)-C(38)-H(38)	119.3
C(39)-C(38)-H(38)	119.3
C(40)-C(39)-C(38)	118.1(3)
C(40)-C(39)-C(42)	121.0(3)
C(38)-C(39)-C(42)	120.9(3)
C(41)-C(40)-C(39)	121.5(3)
C(41)-C(40)-H(40)	119.3
C(39)-C(40)-H(40)	119.3
C(40)-C(41)-C(36)	119.2(3)
C(40)-C(41)-H(41)	120.4
C(36)-C(41)-H(41)	120.4
C(39)-C(42)-H(42A)	109.5
C(39)-C(42)-H(42B)	109.5
H(42A)-C(42)-H(42B)	109.5
C(39)-C(42)-H(42C)	109.5
H(42A)-C(42)-H(42C)	109.5
H(42B)-C(42)-H(42C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for *anti*-**5a**. 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)	30(1)	31(1)	29(1)	1(1)	1(1)	5(1)
S(2)	33(1)	29(1)	31(1)	5(1)	0(1)	2(1)
O(1)	69(2)	36(1)	37(1)	-8(1)	6(1)	-11(1)
O(2)	42(1)	28(1)	40(1)	2(1)	8(1)	-3(1)
O(3)	37(1)	29(1)	45(1)	3(1)	6(1)	3(1)
O(4)	37(1)	51(1)	30(1)	3(1)	-2(1)	16(1)
O(5)	74(2)	37(1)	68(2)	-14(1)	12(1)	-14(1)
O(6)	55(2)	31(1)	60(2)	7(1)	18(1)	-7(1)
O(7)	41(1)	49(1)	31(1)	7(1)	-1(1)	7(1)
O(8)	39(1)	28(1)	49(1)	4(1)	5(1)	-2(1)
N(1)	36(1)	28(1)	27(1)	-2(1)	0(1)	-3(1)
N(2)	27(1)	28(1)	31(1)	-3(1)	-2(1)	2(1)
N(3)	28(1)	33(1)	39(1)	5(1)	2(1)	2(1)
N(4)	26(1)	38(2)	38(1)	-5(1)	-3(1)	-1(1)
N(5)	40(2)	57(2)	51(2)	-3(2)	12(1)	-2(2)
N(6)	32(1)	31(1)	32(1)	-3(1)	2(1)	-4(1)
N(7)	32(1)	29(1)	28(1)	0(1)	-6(1)	0(1)
N(8)	39(2)	39(2)	47(2)	2(1)	16(1)	7(1)
N(9)	38(2)	49(2)	37(1)	-7(1)	8(1)	0(1)
N(10)	51(2)	84(3)	46(2)	-6(2)	19(2)	-4(2)
C(1)	29(1)	26(2)	26(1)	0(1)	-1(1)	1(1)
C(2)	26(1)	29(2)	29(1)	1(1)	-1(1)	3(1)
C(3)	35(2)	26(2)	40(2)	0(1)	-2(1)	0(1)
C(4)	39(2)	36(2)	54(2)	-7(2)	-5(2)	-4(2)
C(5)	35(2)	37(2)	47(2)	-9(1)	-6(1)	-2(1)
C(6)	28(1)	32(2)	29(1)	1(1)	-1(1)	5(1)
C(7)	33(2)	37(2)	39(2)	7(1)	1(1)	-6(1)
C(8)	40(2)	56(2)	33(2)	13(2)	2(1)	-7(2)
C(9)	42(2)	58(2)	25(1)	1(1)	1(1)	-2(2)
C(10)	40(2)	42(2)	29(2)	0(1)	-4(1)	-4(2)
C(11)	30(2)	35(2)	27(1)	5(1)	-1(1)	-1(1)
C(12)	35(2)	37(2)	32(2)	-1(1)	-4(1)	-1(1)
C(13)	42(2)	30(2)	41(2)	3(1)	3(1)	-8(1)
C(14)	51(2)	40(2)	56(2)	7(2)	2(2)	5(2)
C(15)	29(1)	26(2)	32(1)	0(1)	4(1)	3(1)
C(16)	31(2)	49(2)	38(2)	-10(2)	-2(1)	2(2)
C(17)	29(2)	47(2)	55(2)	-3(2)	9(1)	-1(2)
C(18)	40(2)	34(2)	39(2)	2(1)	8(1)	10(2)
C(19)	45(2)	36(2)	32(2)	-4(1)	5(1)	5(2)
C(20)	36(2)	31(2)	36(2)	-4(1)	0(1)	0(1)
C(21)	53(2)	56(2)	54(2)	6(2)	21(2)	13(2)

C(22)	34(2)	28(2)	29(1)	-1(1)	4(1)	4(1)
C(23)	31(1)	26(2)	24(1)	2(1)	-2(1)	0(1)
C(24)	42(2)	26(2)	36(2)	4(1)	-4(1)	0(1)
C(25)	56(2)	29(2)	36(2)	-1(1)	-5(2)	1(2)
C(26)	47(2)	29(2)	40(2)	-2(1)	-11(1)	-4(2)
C(27)	34(2)	33(2)	24(1)	4(1)	1(1)	3(1)
C(28)	47(2)	44(2)	32(2)	7(1)	5(1)	0(2)
C(29)	64(2)	60(2)	32(2)	10(2)	12(2)	12(2)
C(30)	68(2)	56(2)	25(2)	-6(2)	-1(2)	13(2)
C(31)	48(2)	39(2)	35(2)	-6(1)	-8(1)	3(2)
C(32)	33(2)	35(2)	29(1)	-3(1)	-2(1)	2(1)
C(33)	34(2)	32(2)	54(2)	-3(2)	0(1)	-3(1)
C(34)	60(3)	35(2)	92(3)	15(2)	23(2)	-14(2)
C(35)	88(3)	53(3)	73(3)	20(2)	32(3)	15(2)
C(36)	31(2)	29(2)	32(1)	1(1)	2(1)	0(1)
C(37)	38(2)	36(2)	34(2)	-3(1)	-2(1)	-3(1)
C(38)	34(2)	40(2)	45(2)	0(1)	-2(1)	-5(2)
C(39)	36(2)	35(2)	38(2)	4(1)	2(1)	7(1)
C(40)	39(2)	45(2)	37(2)	-12(1)	0(1)	2(2)
C(41)	34(2)	32(2)	41(2)	-7(1)	-1(1)	-5(1)
C(42)	45(2)	59(2)	46(2)	8(2)	9(2)	5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for *anti*-**5a**.

	x	y	z	U(eq)
H(1)	-1117	4611	4072	33
H(3A)	-1073	7110	3268	41
H(3B)	-1025	6728	4193	41
H(4A)	184	7369	3317	52
H(4B)	20	7660	4230	52
H(5A)	971	6339	3973	48
H(5B)	509	6310	4765	48
H(7)	-65	6704	2062	44
H(8)	-186	6264	684	52
H(9)	-834	4895	272	50
H(10)	-1415	3971	1212	45
H(13A)	-2259	1992	3224	45
H(13B)	-2524	2281	4095	45
H(14A)	-1044	1851	3786	74
H(14B)	-1559	1204	4301	74
H(14C)	-1273	2213	4650	74
H(16)	1973	5080	3635	48
H(17)	2716	5134	2548	52
H(19)	1080	3953	1069	45
H(20)	322	3918	2136	41
H(21A)	2819	5081	1084	80
H(21B)	2176	4601	494	80
H(21C)	2759	3948	1023	80
H(22)	5930	2630	1226	36
H(24A)	6078	400	1940	42
H(24B)	5315	226	2348	42
H(25A)	5473	545	645	49
H(25B)	5024	-310	1030	49
H(26A)	4283	1147	500	48
H(26B)	4080	689	1351	48
H(28)	4767	993	3403	49
H(29)	4883	1667	4716	62
H(30)	5628	2976	4990	60
H(31)	6295	3659	3992	49
H(34A)	7306	4985	997	74
H(34B)	6715	5400	1576	74
H(35A)	6353	4557	28	105
H(35B)	6289	5666	224	105
H(35C)	5751	4923	611	105
H(37)	2906	1909	1704	44
H(38)	2149	1771	2771	48

H(40)	3548	3429	4186	49
H(41)	4322	3556	3135	43
H(42A)	1985	3100	4311	74
H(42B)	2570	2402	4789	74
H(42C)	1926	1978	4166	74

Table 6. Torsion angles [deg] for *anti*-**5a**.

O(3)-S(1)-N(2)-C(5)	167.5(2)
O(4)-S(1)-N(2)-C(5)	38.2(3)
C(15)-S(1)-N(2)-C(5)	-75.8(2)
O(3)-S(1)-N(2)-C(2)	-29.2(3)
O(4)-S(1)-N(2)-C(2)	-158.4(2)
C(15)-S(1)-N(2)-C(2)	87.5(3)
C(1)-N(3)-N(4)-N(5)	-178(100)
O(7)-S(2)-N(7)-C(26)	-30.2(3)
O(8)-S(2)-N(7)-C(26)	-158.9(2)
C(36)-S(2)-N(7)-C(26)	84.8(3)
O(7)-S(2)-N(7)-C(23)	162.9(2)
O(8)-S(2)-N(7)-C(23)	34.1(3)
C(36)-S(2)-N(7)-C(23)	-82.2(3)
C(22)-N(8)-N(9)-N(10)	-178(100)
C(12)-N(1)-C(1)-N(3)	-91.6(3)
C(11)-N(1)-C(1)-N(3)	103.7(3)
C(12)-N(1)-C(1)-C(2)	150.5(3)
C(11)-N(1)-C(1)-C(2)	-14.2(3)
N(4)-N(3)-C(1)-N(1)	101.0(3)
N(4)-N(3)-C(1)-C(2)	-144.4(2)
C(5)-N(2)-C(2)-C(6)	106.8(3)
S(1)-N(2)-C(2)-C(6)	-57.4(3)
C(5)-N(2)-C(2)-C(3)	-16.7(3)
S(1)-N(2)-C(2)-C(3)	179.1(2)
C(5)-N(2)-C(2)-C(1)	-138.0(3)
S(1)-N(2)-C(2)-C(1)	57.8(3)
N(1)-C(1)-C(2)-N(2)	-106.3(2)
N(3)-C(1)-C(2)-N(2)	136.1(2)
N(1)-C(1)-C(2)-C(6)	15.8(3)
N(3)-C(1)-C(2)-C(6)	-101.8(2)
N(1)-C(1)-C(2)-C(3)	141.0(2)
N(3)-C(1)-C(2)-C(3)	23.3(3)
N(2)-C(2)-C(3)-C(4)	32.0(3)
C(6)-C(2)-C(3)-C(4)	-90.7(3)
C(1)-C(2)-C(3)-C(4)	151.2(2)
C(2)-C(3)-C(4)-C(5)	-37.0(3)
C(2)-N(2)-C(5)-C(4)	-5.3(3)
S(1)-N(2)-C(5)-C(4)	160.2(2)
C(3)-C(4)-C(5)-N(2)	25.8(3)
N(2)-C(2)-C(6)-C(7)	-71.8(4)
C(3)-C(2)-C(6)-C(7)	43.3(4)
C(1)-C(2)-C(6)-C(7)	167.9(3)
N(2)-C(2)-C(6)-C(11)	107.4(3)
C(3)-C(2)-C(6)-C(11)	-137.5(3)

C(1)-C(2)-C(6)-C(11)	-12.9(3)
C(11)-C(6)-C(7)-C(8)	-1.2(5)
C(2)-C(6)-C(7)-C(8)	178.0(3)
C(6)-C(7)-C(8)-C(9)	-0.7(5)
C(7)-C(8)-C(9)-C(10)	1.5(5)
C(8)-C(9)-C(10)-C(11)	-0.3(5)
C(7)-C(6)-C(11)-C(10)	2.4(5)
C(2)-C(6)-C(11)-C(10)	-176.9(3)
C(7)-C(6)-C(11)-N(1)	-175.9(3)
C(2)-C(6)-C(11)-N(1)	4.8(3)
C(9)-C(10)-C(11)-C(6)	-1.6(5)
C(9)-C(10)-C(11)-N(1)	176.4(3)
C(12)-N(1)-C(11)-C(6)	-158.2(3)
C(1)-N(1)-C(11)-C(6)	6.4(3)
C(12)-N(1)-C(11)-C(10)	23.6(5)
C(1)-N(1)-C(11)-C(10)	-171.8(3)
C(13)-O(2)-C(12)-O(1)	10.5(4)
C(13)-O(2)-C(12)-N(1)	-170.5(2)
C(11)-N(1)-C(12)-O(1)	0.6(5)
C(1)-N(1)-C(12)-O(1)	-162.0(3)
C(11)-N(1)-C(12)-O(2)	-178.3(2)
C(1)-N(1)-C(12)-O(2)	19.1(4)
C(12)-O(2)-C(13)-C(14)	86.1(3)
O(3)-S(1)-C(15)-C(16)	-154.4(2)
O(4)-S(1)-C(15)-C(16)	-24.5(3)
N(2)-S(1)-C(15)-C(16)	88.9(3)
O(3)-S(1)-C(15)-C(20)	24.4(3)
O(4)-S(1)-C(15)-C(20)	154.3(2)
N(2)-S(1)-C(15)-C(20)	-92.4(3)
C(20)-C(15)-C(16)-C(17)	1.1(5)
S(1)-C(15)-C(16)-C(17)	179.8(3)
C(15)-C(16)-C(17)-C(18)	-1.5(5)
C(16)-C(17)-C(18)-C(19)	1.3(5)
C(16)-C(17)-C(18)-C(21)	-178.2(3)
C(17)-C(18)-C(19)-C(20)	-0.6(5)
C(21)-C(18)-C(19)-C(20)	178.8(3)
C(18)-C(19)-C(20)-C(15)	0.2(5)
C(16)-C(15)-C(20)-C(19)	-0.5(5)
S(1)-C(15)-C(20)-C(19)	-179.2(2)
C(33)-N(6)-C(22)-N(8)	96.9(3)
C(32)-N(6)-C(22)-N(8)	-107.1(3)
C(33)-N(6)-C(22)-C(23)	-144.8(3)
C(32)-N(6)-C(22)-C(23)	11.2(3)
N(9)-N(8)-C(22)-N(6)	-89.8(3)
N(9)-N(8)-C(22)-C(23)	154.7(3)
C(26)-N(7)-C(23)-C(27)	-126.1(3)

S(2)-N(7)-C(23)-C(27)	41.8(3)
C(26)-N(7)-C(23)-C(24)	-3.1(3)
S(2)-N(7)-C(23)-C(24)	164.8(2)
C(26)-N(7)-C(23)-C(22)	120.0(3)
S(2)-N(7)-C(23)-C(22)	-72.1(3)
N(6)-C(22)-C(23)-N(7)	111.1(2)
N(8)-C(22)-C(23)-N(7)	-129.7(2)
N(6)-C(22)-C(23)-C(27)	-11.3(3)
N(8)-C(22)-C(23)-C(27)	107.9(3)
N(6)-C(22)-C(23)-C(24)	-135.6(2)
N(8)-C(22)-C(23)-C(24)	-16.3(3)
N(7)-C(23)-C(24)-C(25)	26.6(3)
C(27)-C(23)-C(24)-C(25)	151.0(3)
C(22)-C(23)-C(24)-C(25)	-90.7(3)
C(23)-C(24)-C(25)-C(26)	-40.5(3)
C(23)-N(7)-C(26)-C(25)	-21.3(3)
S(2)-N(7)-C(26)-C(25)	170.2(2)
C(24)-C(25)-C(26)-N(7)	36.8(3)
N(7)-C(23)-C(27)-C(32)	-109.3(3)
C(24)-C(23)-C(27)-C(32)	134.4(3)
C(22)-C(23)-C(27)-C(32)	8.2(3)
N(7)-C(23)-C(27)-C(28)	71.4(4)
C(24)-C(23)-C(27)-C(28)	-44.9(4)
C(22)-C(23)-C(27)-C(28)	-171.1(3)
C(32)-C(27)-C(28)-C(29)	1.3(5)
C(23)-C(27)-C(28)-C(29)	-179.5(3)
C(27)-C(28)-C(29)-C(30)	-0.4(5)
C(28)-C(29)-C(30)-C(31)	-0.5(6)
C(29)-C(30)-C(31)-C(32)	0.5(5)
C(28)-C(27)-C(32)-C(31)	-1.3(5)
C(23)-C(27)-C(32)-C(31)	179.3(3)
C(28)-C(27)-C(32)-N(6)	177.7(3)
C(23)-C(27)-C(32)-N(6)	-1.7(3)
C(30)-C(31)-C(32)-C(27)	0.4(5)
C(30)-C(31)-C(32)-N(6)	-178.4(3)
C(33)-N(6)-C(32)-C(27)	149.3(3)
C(22)-N(6)-C(32)-C(27)	-6.4(3)
C(33)-N(6)-C(32)-C(31)	-31.8(5)
C(22)-N(6)-C(32)-C(31)	172.5(3)
C(34)-O(6)-C(33)-O(5)	-13.3(5)
C(34)-O(6)-C(33)-N(6)	167.9(3)
C(32)-N(6)-C(33)-O(5)	7.8(5)
C(22)-N(6)-C(33)-O(5)	160.8(3)
C(32)-N(6)-C(33)-O(6)	-173.2(3)
C(22)-N(6)-C(33)-O(6)	-20.3(4)
C(33)-O(6)-C(34)-C(35)	-113.3(4)

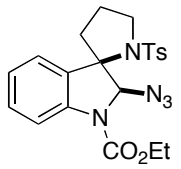
O(7)-S(2)-C(36)-C(37)	34.6(3)
O(8)-S(2)-C(36)-C(37)	164.6(2)
N(7)-S(2)-C(36)-C(37)	-79.5(3)
O(7)-S(2)-C(36)-C(41)	-145.9(3)
O(8)-S(2)-C(36)-C(41)	-15.8(3)
N(7)-S(2)-C(36)-C(41)	100.1(3)
C(41)-C(36)-C(37)-C(38)	-1.0(5)
S(2)-C(36)-C(37)-C(38)	178.6(2)
C(36)-C(37)-C(38)-C(39)	1.1(5)
C(37)-C(38)-C(39)-C(40)	-0.7(5)
C(37)-C(38)-C(39)-C(42)	179.6(3)
C(38)-C(39)-C(40)-C(41)	0.1(5)
C(42)-C(39)-C(40)-C(41)	179.8(3)
C(39)-C(40)-C(41)-C(36)	0.0(5)
C(37)-C(36)-C(41)-C(40)	0.5(5)
S(2)-C(36)-C(41)-C(40)	-179.1(2)

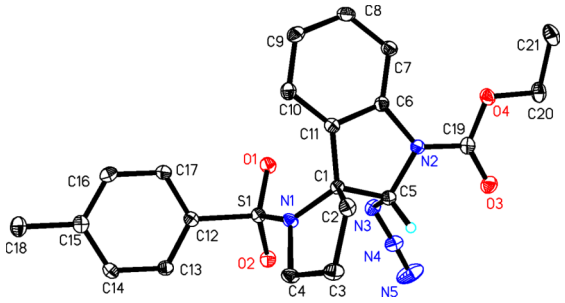
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for *anti*-**5a** [A and deg.].

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

The X-ray structure of compound *syn*-**5a**





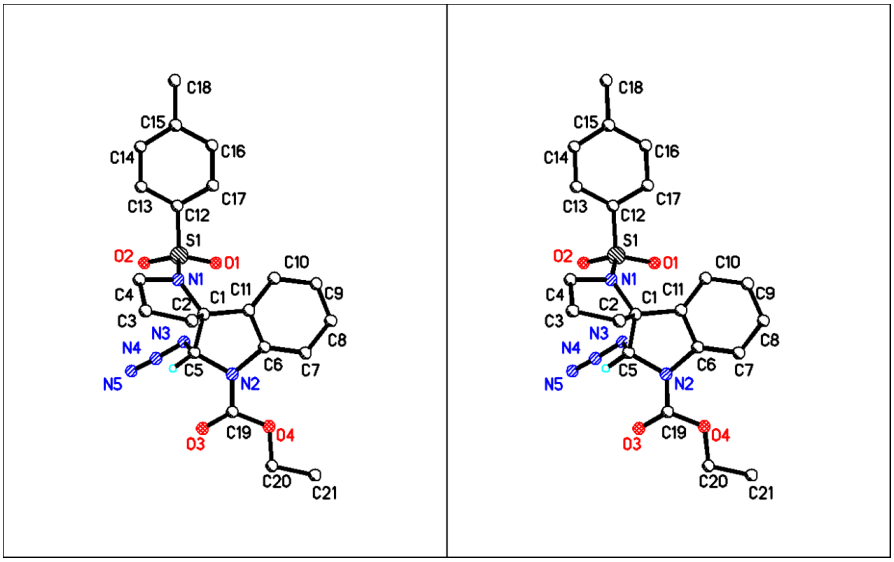


Table 1. Crystal data and structure refinement for *syn*-**5a**.

Identification code	*syn*-**5a**	
Empirical formula	$C_{21}H_{23}N_5O_4S$	
Formula weight	526.43	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 10.8728(15) Å	a= 73.171(6)°.
	b = 14.6282(18) Å	b= 81.536(7)°.
	c = 16.068(2) Å	g = 77.112(7)°.
Volume	2375.2(6) $Å^3$	
Z	4	
Density (calculated)	1.472 Mg/m^3	
Absorption coefficient	0.402 mm^{-1}	
F(000)	1096	
Crystal size	0.26 x 0.15 x 0.06 mm^3	
Theta range for data collection	1.707 to 27.484°.	
Index ranges	-14<=h<=14, -18<=k<=13, -20<=l<=20	
Reflections collected	23787	
Independent reflections	10808 [R(int) = 0.0407]	
Completeness to theta = 26.000°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.7456	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10808 / 0 / 617	
Goodness-of-fit on F^2	1.126	
Final R indices [I>2sigma(I)]	R1 = 0.0641, wR2 = 0.1301	
R indices (all data)	R1 = 0.0759, wR2 = 0.1373	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.562 and -0.461 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)for *syn*-**5a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl1A	8991(1)	8416(1)	4732(1)	40(1)
Cl2A	6309(1)	8686(1)	4536(1)	61(1)
C22A	7813(3)	8806(2)	3995(2)	38(1)
Cl1	5297(1)	3544(1)	4981(1)	66(1)
Cl2	7929(1)	3479(1)	5161(1)	49(1)
C22	6760(3)	3782(3)	4435(2)	46(1)
S1A	6422(1)	8730(1)	1532(1)	22(1)
O1A	7014(2)	8733(1)	2268(1)	27(1)
O2A	6546(2)	9466(1)	727(1)	28(1)
O3A	11836(2)	7119(1)	742(1)	32(1)
O4A	12207(2)	6452(1)	2163(1)	28(1)
N1A	6870(2)	7638(1)	1372(1)	20(1)
N2A	10219(2)	6782(2)	1779(1)	24(1)
N3A	9174(2)	8331(2)	957(1)	26(1)
N4A	9662(2)	8733(2)	240(1)	27(1)
N5A	10064(3)	9198(2)	-388(2)	43(1)
C1A	8044(2)	6954(2)	1638(1)	20(1)
C2A	7852(2)	6066(2)	1376(2)	23(1)
C3A	7216(2)	6491(2)	529(2)	27(1)
C4A	6473(2)	7496(2)	582(2)	27(1)
C5A	9290(2)	7272(2)	1141(2)	22(1)
C6A	9601(2)	6561(2)	2637(2)	22(1)
C7A	10104(2)	6252(2)	3438(2)	26(1)
C8A	9271(2)	6086(2)	4183(2)	28(1)
C9A	7982(2)	6210(2)	4135(2)	27(1)
C10A	7488(2)	6514(2)	3328(2)	24(1)
C11A	8306(2)	6696(2)	2583(2)	21(1)
C12A	4780(2)	8799(2)	1825(2)	24(1)
C13A	3943(2)	9189(2)	1184(2)	27(1)
C14A	2656(3)	9245(2)	1412(2)	32(1)
C15A	2183(2)	8922(2)	2277(2)	30(1)
C16A	3045(3)	8540(2)	2908(2)	32(1)

C17A	4332(2)	8475(2)	2692(2)	28(1)
C18A	784(3)	8995(2)	2525(2)	42(1)
C19A	11471(2)	6810(2)	1505(2)	24(1)
C20A	13550(2)	6462(2)	1922(2)	30(1)
C21A	14199(2)	6031(2)	2750(2)	34(1)
S1	5466(1)	3839(1)	1929(1)	22(1)
O1	6081(2)	3823(1)	2661(1)	28(1)
O2	5530(2)	4612(1)	1142(1)	28(1)
O3	10910(2)	2260(1)	1126(1)	33(1)
O4	11280(2)	1431(1)	2518(1)	29(1)
N1	5950(2)	2781(1)	1718(1)	22(1)
N2	9291(2)	1872(2)	2137(1)	23(1)
N3	8260(2)	3459(2)	1387(1)	26(1)
N4	8644(2)	3906(2)	658(1)	30(1)
N5	8968(3)	4401(2)	26(2)	57(1)
C1	7111(2)	2077(2)	1981(1)	20(1)
C2	6915(2)	1210(2)	1685(2)	26(1)
C3	6278(3)	1671(2)	840(2)	30(1)
C4	5526(2)	2660(2)	928(2)	27(1)
C5	8358(2)	2409(2)	1515(2)	22(1)
C6	8668(2)	1600(2)	2991(2)	22(1)
C7	9171(2)	1218(2)	3793(2)	26(1)
C8	8335(2)	1020(2)	4532(2)	30(1)
C9	7041(2)	1178(2)	4479(2)	30(1)
C10	6545(2)	1554(2)	3670(2)	26(1)
C11	7367(2)	1768(2)	2930(2)	21(1)
C12	3841(2)	3847(2)	2256(2)	22(1)
C13	2952(2)	4213(2)	1644(2)	25(1)
C14	1683(2)	4199(2)	1920(2)	26(1)
C15	1283(2)	3842(2)	2795(2)	26(1)
C16	2193(2)	3492(2)	3398(2)	28(1)
C17	3461(2)	3484(2)	3137(2)	26(1)
C18	-98(2)	3840(2)	3088(2)	33(1)
C19	10544(2)	1884(2)	1870(2)	25(1)
C20	12632(2)	1409(2)	2298(2)	34(1)
C21	13260(3)	934(2)	3126(2)	40(1)

Table 3. Bond lengths [Å] and angles [°] for *syn*-**5a**.

Cl1A-C22A	1.760(3)
Cl2A-C22A	1.756(3)
Cl1-C22	1.755(3)
Cl2-C22	1.749(3)
S1A-O1A	1.4277(16)
S1A-O2A	1.4345(17)
S1A-N1A	1.6456(19)
S1A-C12A	1.767(3)
O3A-C19A	1.217(3)
O4A-C19A	1.332(3)
O4A-C20A	1.457(3)
N1A-C1A	1.469(3)
N1A-C4A	1.482(3)
N2A-C5A	1.463(3)
N2A-C6A	1.422(3)
N2A-C19A	1.373(3)
N3A-N4A	1.234(3)
N3A-C5A	1.471(3)
N4A-N5A	1.130(3)
C1A-C2A	1.540(3)
C1A-C5A	1.560(3)
C1A-C11A	1.509(3)
C2A-C3A	1.521(3)
C3A-C4A	1.530(3)
C6A-C7A	1.386(3)
C6A-C11A	1.389(3)
C7A-C8A	1.387(4)
C8A-C9A	1.383(3)
C9A-C10A	1.390(3)
C10A-C11A	1.380(3)
C12A-C13A	1.385(3)
C12A-C17A	1.387(3)
C13A-C14A	1.384(4)
C14A-C15A	1.392(4)
C15A-C16A	1.391(4)
C15A-C18A	1.502(4)
C16A-C17A	1.380(4)
C20A-C21A	1.502(4)

S1-O1	1.4288(17)
S1-O2	1.4356(18)
S1-N1	1.636(2)
S1-C12	1.767(2)
O3-C19	1.211(3)
O4-C19	1.331(3)
O4-C20	1.456(3)
N1-C1	1.469(3)
N1-C4	1.481(3)
N2-C5	1.461(3)
N2-C6	1.425(3)
N2-C19	1.369(3)
N3-N4	1.229(3)
N3-C5	1.471(3)
N4-N5	1.124(3)
C1-C2	1.542(3)
C1-C5	1.555(3)
C1-C11	1.509(3)
C2-C3	1.523(3)
C3-C4	1.530(3)
C6-C7	1.388(3)
C6-C11	1.394(3)
C7-C8	1.386(3)
C8-C9	1.385(4)
C9-C10	1.394(3)
C10-C11	1.381(3)
C12-C13	1.390(3)
C12-C17	1.394(3)
C13-C14	1.388(3)
C14-C15	1.390(3)
C15-C16	1.397(3)
C15-C18	1.506(3)
C16-C17	1.379(3)
C20-C21	1.498(4)

Cl2A-C22A-Cl1A	111.49(16)
Cl2-C22-Cl1	111.50(16)
O1A-S1A-O2A	119.77(11)
O1A-S1A-N1A	107.68(10)
O1A-S1A-C12A	108.73(11)

O2A-S1A-N1A	110.58(10)
O2A-S1A-C12A	106.22(11)
N1A-S1A-C12A	102.49(10)
C19A-O4A-C20A	115.3(2)
C1A-N1A-S1A	125.55(15)
C1A-N1A-C4A	110.75(18)
C4A-N1A-S1A	117.06(15)
C6A-N2A-C5A	110.29(18)
C19A-N2A-C5A	117.6(2)
C19A-N2A-C6A	130.3(2)
N4A-N3A-C5A	115.6(2)
N5A-N4A-N3A	172.0(3)
N1A-C1A-C2A	100.18(17)
N1A-C1A-C5A	115.23(18)
N1A-C1A-C11A	117.84(19)
C2A-C1A-C5A	108.97(18)
C11A-C1A-C2A	111.80(18)
C11A-C1A-C5A	102.88(17)
C3A-C2A-C1A	104.85(18)
C2A-C3A-C4A	105.14(18)
N1A-C4A-C3A	104.55(18)
N2A-C5A-N3A	109.31(18)
N2A-C5A-C1A	102.52(18)
N3A-C5A-C1A	110.38(18)
C7A-C6A-N2A	129.9(2)
C7A-C6A-C11A	121.1(2)
C11A-C6A-N2A	109.0(2)
C6A-C7A-C8A	117.7(2)
C9A-C8A-C7A	121.5(2)
C8A-C9A-C10A	120.3(2)
C11A-C10A-C9A	118.7(2)
C6A-C11A-C1A	109.5(2)
C10A-C11A-C1A	129.6(2)
C10A-C11A-C6A	120.7(2)
C13A-C12A-S1A	119.51(19)
C13A-C12A-C17A	120.3(2)
C17A-C12A-S1A	120.19(19)
C14A-C13A-C12A	119.7(2)
C13A-C14A-C15A	121.1(2)
C14A-C15A-C18A	121.1(3)

C16A-C15A-C14A	118.0(2)
C16A-C15A-C18A	120.9(3)
C17A-C16A-C15A	121.7(2)
C16A-C17A-C12A	119.3(2)
O3A-C19A-O4A	125.4(2)
O3A-C19A-N2A	122.4(2)
O4A-C19A-N2A	112.2(2)
O4A-C20A-C21A	106.6(2)
O1-S1-O2	119.75(11)
O1-S1-N1	107.70(10)
O1-S1-C12	108.60(11)
O2-S1-N1	110.52(10)
O2-S1-C12	106.44(11)
N1-S1-C12	102.45(10)
C19-O4-C20	115.9(2)
C1-N1-S1	126.83(15)
C1-N1-C4	111.24(18)
C4-N1-S1	117.24(15)
C6-N2-C5	109.76(18)
C19-N2-C5	118.2(2)
C19-N2-C6	130.5(2)
N4-N3-C5	115.3(2)
N5-N4-N3	172.4(3)
N1-C1-C2	100.51(17)
N1-C1-C5	114.53(19)
N1-C1-C11	118.54(19)
C2-C1-C5	109.95(19)
C11-C1-C2	110.97(19)
C11-C1-C5	102.46(17)
C3-C2-C1	104.78(19)
C2-C3-C4	105.19(19)
N1-C4-C3	104.59(19)
N2-C5-N3	109.20(18)
N2-C5-C1	103.12(18)
N3-C5-C1	110.92(19)
C7-C6-N2	129.9(2)
C7-C6-C11	121.1(2)
C11-C6-N2	109.1(2)
C8-C7-C6	117.8(2)
C9-C8-C7	121.6(2)

C8-C9-C10	120.2(2)
C11-C10-C9	118.7(2)
C6-C11-C1	109.3(2)
C10-C11-C1	129.7(2)
C10-C11-C6	120.6(2)
C13-C12-S1	120.38(19)
C13-C12-C17	120.4(2)
C17-C12-S1	119.18(18)
C14-C13-C12	119.1(2)
C13-C14-C15	121.5(2)
C14-C15-C16	118.3(2)
C14-C15-C18	121.0(2)
C16-C15-C18	120.7(2)
C17-C16-C15	121.2(2)
C16-C17-C12	119.5(2)
O3-C19-O4	125.5(2)
O3-C19-N2	122.7(2)
O4-C19-N2	111.9(2)
O4-C20-C21	106.9(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for *syn*-**5a**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl1A	41(1)	44(1)	35(1)	-5(1)	-13(1)	-13(1)
Cl2A	36(1)	100(1)	56(1)	-28(1)	2(1)	-24(1)
C22A	36(2)	48(2)	31(1)	-7(1)	-6(1)	-11(1)
Cl1	36(1)	123(1)	52(1)	-36(1)	-2(1)	-23(1)
Cl2	39(1)	77(1)	33(1)	-7(1)	-6(1)	-25(1)
C22	46(2)	67(2)	31(2)	-3(2)	-11(1)	-27(2)
S1A	25(1)	18(1)	21(1)	-4(1)	-5(1)	-3(1)
O1A	32(1)	26(1)	26(1)	-10(1)	-8(1)	-6(1)
O2A	32(1)	22(1)	28(1)	0(1)	-3(1)	-6(1)
O3A	27(1)	40(1)	29(1)	-7(1)	1(1)	-9(1)
O4A	17(1)	32(1)	34(1)	-6(1)	-4(1)	-5(1)
N1A	20(1)	20(1)	22(1)	-7(1)	-7(1)	-2(1)
N2A	19(1)	29(1)	24(1)	-4(1)	-3(1)	-6(1)
N3A	26(1)	23(1)	27(1)	-4(1)	1(1)	-8(1)
N4A	29(1)	21(1)	31(1)	-7(1)	-2(1)	-4(1)
N5A	62(2)	25(1)	35(1)	-5(1)	8(1)	-10(1)
C1A	20(1)	18(1)	21(1)	-3(1)	-3(1)	-4(1)
C2A	25(1)	21(1)	25(1)	-6(1)	-3(1)	-5(1)
C3A	28(1)	28(1)	28(1)	-12(1)	-2(1)	-7(1)
C4A	29(1)	31(1)	25(1)	-13(1)	-9(1)	-1(1)
C5A	23(1)	20(1)	23(1)	-6(1)	-3(1)	-5(1)
C6A	23(1)	22(1)	22(1)	-5(1)	-4(1)	-4(1)
C7A	23(1)	28(1)	28(1)	-6(1)	-8(1)	-4(1)
C8A	31(1)	27(1)	24(1)	-1(1)	-9(1)	-5(1)
C9A	29(1)	29(1)	23(1)	-5(1)	-2(1)	-4(1)
C10A	21(1)	25(1)	25(1)	-3(1)	-4(1)	-4(1)
C11A	23(1)	16(1)	26(1)	-5(1)	-6(1)	-3(1)
C12A	28(1)	19(1)	25(1)	-7(1)	-2(1)	-2(1)
C13A	26(1)	28(1)	25(1)	-7(1)	-5(1)	3(1)
C14A	31(1)	30(1)	35(1)	-9(1)	-9(1)	0(1)
C15A	29(1)	22(1)	40(2)	-13(1)	1(1)	-2(1)
C16A	37(1)	29(1)	28(1)	-9(1)	4(1)	-7(1)

C17A	34(1)	25(1)	24(1)	-7(1)	-3(1)	-2(1)
C18A	32(2)	43(2)	53(2)	-18(2)	3(1)	-6(1)
C19A	21(1)	24(1)	29(1)	-8(1)	-1(1)	-5(1)
C20A	17(1)	30(1)	46(2)	-16(1)	0(1)	-6(1)
C21A	22(1)	36(2)	52(2)	-22(1)	-9(1)	-3(1)
S1	22(1)	20(1)	25(1)	-8(1)	-3(1)	-4(1)
O1	28(1)	30(1)	32(1)	-16(1)	-6(1)	-5(1)
O2	29(1)	21(1)	31(1)	-3(1)	0(1)	-6(1)
O3	28(1)	40(1)	29(1)	-7(1)	3(1)	-12(1)
O4	19(1)	34(1)	32(1)	-3(1)	-2(1)	-6(1)
N1	23(1)	21(1)	24(1)	-9(1)	-8(1)	-2(1)
N2	20(1)	27(1)	21(1)	-3(1)	-2(1)	-5(1)
N3	30(1)	23(1)	27(1)	-6(1)	2(1)	-9(1)
N4	42(1)	24(1)	27(1)	-8(1)	-2(1)	-9(1)
N5	107(3)	31(1)	32(1)	-3(1)	7(2)	-22(2)
C1	20(1)	21(1)	19(1)	-5(1)	-1(1)	-3(1)
C2	26(1)	22(1)	32(1)	-12(1)	-3(1)	-4(1)
C3	39(1)	29(1)	29(1)	-13(1)	-7(1)	-8(1)
C4	31(1)	29(1)	26(1)	-12(1)	-10(1)	-3(1)
C5	24(1)	22(1)	22(1)	-6(1)	-2(1)	-5(1)
C6	22(1)	20(1)	23(1)	-4(1)	1(1)	-5(1)
C7	24(1)	28(1)	25(1)	-4(1)	-5(1)	-6(1)
C8	32(1)	31(1)	23(1)	-2(1)	-7(1)	-6(1)
C9	32(1)	32(1)	24(1)	-1(1)	1(1)	-10(1)
C10	23(1)	28(1)	29(1)	-7(1)	0(1)	-8(1)
C11	23(1)	17(1)	24(1)	-4(1)	-4(1)	-5(1)
C12	24(1)	17(1)	25(1)	-6(1)	-4(1)	-2(1)
C13	27(1)	23(1)	23(1)	-4(1)	-3(1)	-5(1)
C14	24(1)	25(1)	29(1)	-5(1)	-8(1)	-2(1)
C15	27(1)	21(1)	31(1)	-9(1)	-1(1)	-5(1)
C16	34(1)	28(1)	22(1)	-4(1)	-1(1)	-8(1)
C17	27(1)	28(1)	22(1)	-6(1)	-5(1)	-4(1)
C18	28(1)	32(1)	40(2)	-9(1)	1(1)	-10(1)
C19	24(1)	23(1)	30(1)	-9(1)	0(1)	-6(1)
C20	21(1)	37(2)	46(2)	-15(1)	1(1)	-8(1)
C21	26(1)	42(2)	58(2)	-19(2)	-12(1)	-1(1)

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

	x	y	z	U(eq)
H22A	7805	9482	3681	46
H22B	8011	8425	3572	46
H22C	7019	3410	4007	56
H22D	6668	4467	4128	56
H2AA	8658	5645	1288	28
H2AB	7318	5694	1823	28
H3AA	6651	6085	479	32
H3AB	7843	6547	30	32
H4AA	6684	7995	66	32
H4AB	5568	7513	638	32
H5A	9515	7055	606	26
H7A	10971	6158	3475	32
H8A	9586	5887	4728	33
H9A	7444	6091	4645	33
H10A	6623	6592	3291	29
H13A	4245	9412	604	33
H14A	2097	9502	979	39
H16A	2745	8323	3489	38
H17A	4892	8216	3124	33
H18A	480	9508	2808	63
H18B	626	8390	2916	63
H18C	353	9134	2009	63
H20A	13682	7122	1658	36
H20B	13883	6079	1509	36
H21A	15094	6012	2619	52
H21B	14047	5383	3010	52
H21C	13872	6424	3148	52
H2A	7720	793	1586	31
H2B	6380	828	2121	31
H3A	5719	1272	769	36
H3B	6905	1750	341	36
H4A	5711	3176	418	33
H4B	4623	2667	999	33

H5	8581	2235	959	27
H7	10039	1099	3833	32
H8	8650	774	5077	35
H9	6501	1033	4985	36
H10	5678	1657	3629	32
H13	3203	4464	1058	30
H14	1087	4434	1511	31
H16	1940	3260	3987	34
H17	4058	3239	3544	31
H18D	-450	4387	3320	50
H18E	-182	3249	3531	50
H18F	-541	3883	2599	50
H20C	12793	2065	2054	40
H20D	12955	1041	1871	40
H21D	14161	884	3004	61
H21E	13068	295	3371	61
H21F	12955	1317	3535	61

Table 6. Torsion angles [°] for *syn*-**5a**.

S1A-N1A-C1A-C2A	175.67(16)
S1A-N1A-C1A-C5A	-67.6(2)
S1A-N1A-C1A-C11A	54.2(3)
S1A-N1A-C4A-C3A	170.18(16)
S1A-C12A-C13A-C14A	-180.0(2)
S1A-C12A-C17A-C16A	179.67(19)
O1A-S1A-N1A-C1A	-25.2(2)
O1A-S1A-N1A-C4A	-173.88(17)
O1A-S1A-C12A-C13A	154.32(19)
O1A-S1A-C12A-C17A	-25.1(2)
O2A-S1A-N1A-C1A	107.32(19)
O2A-S1A-N1A-C4A	-41.3(2)
O2A-S1A-C12A-C13A	24.2(2)
O2A-S1A-C12A-C17A	-155.19(19)
N1A-S1A-C12A-C13A	-91.9(2)
N1A-S1A-C12A-C17A	88.7(2)
N1A-C1A-C2A-C3A	37.5(2)
N1A-C1A-C5A-N2A	152.71(18)
N1A-C1A-C5A-N3A	36.4(2)
N1A-C1A-C11A-C6A	-145.4(2)
N1A-C1A-C11A-C10A	40.6(3)
N2A-C6A-C7A-C8A	-179.9(2)
N2A-C6A-C11A-C1A	4.2(3)
N2A-C6A-C11A-C10A	178.9(2)
N4A-N3A-C5A-N2A	106.6(2)
N4A-N3A-C5A-C1A	-141.4(2)
C1A-N1A-C4A-C3A	17.1(3)
C1A-C2A-C3A-C4A	-28.5(2)
C2A-C1A-C5A-N2A	-95.7(2)
C2A-C1A-C5A-N3A	147.99(18)
C2A-C1A-C11A-C6A	99.4(2)
C2A-C1A-C11A-C10A	-74.6(3)
C2A-C3A-C4A-N1A	7.9(3)
C4A-N1A-C1A-C2A	-34.0(2)
C4A-N1A-C1A-C5A	82.7(2)
C4A-N1A-C1A-C11A	-155.5(2)
C5A-N2A-C6A-C7A	-168.2(2)
C5A-N2A-C6A-C11A	12.1(3)

C5A-N2A-C19A-O3A	-8.8(3)
C5A-N2A-C19A-O4A	171.96(19)
C5A-C1A-C2A-C3A	-83.8(2)
C5A-C1A-C11A-C6A	-17.4(2)
C5A-C1A-C11A-C10A	168.6(2)
C6A-N2A-C5A-N3A	94.9(2)
C6A-N2A-C5A-C1A	-22.2(2)
C6A-N2A-C19A-O3A	-171.5(2)
C6A-N2A-C19A-O4A	9.3(4)
C6A-C7A-C8A-C9A	0.9(4)
C7A-C6A-C11A-C1A	-175.5(2)
C7A-C6A-C11A-C10A	-0.8(4)
C7A-C8A-C9A-C10A	-0.4(4)
C8A-C9A-C10A-C11A	-0.7(4)
C9A-C10A-C11A-C1A	174.7(2)
C9A-C10A-C11A-C6A	1.3(4)
C11A-C1A-C2A-C3A	163.20(19)
C11A-C1A-C5A-N2A	23.1(2)
C11A-C1A-C5A-N3A	-93.2(2)
C11A-C6A-C7A-C8A	-0.3(4)
C12A-S1A-N1A-C1A	-139.81(19)
C12A-S1A-N1A-C4A	71.55(19)
C12A-C13A-C14A-C15A	0.5(4)
C13A-C12A-C17A-C16A	0.3(4)
C13A-C14A-C15A-C16A	-0.1(4)
C13A-C14A-C15A-C18A	179.2(3)
C14A-C15A-C16A-C17A	-0.2(4)
C15A-C16A-C17A-C12A	0.1(4)
C17A-C12A-C13A-C14A	-0.6(4)
C18A-C15A-C16A-C17A	-179.5(2)
C19A-O4A-C20A-C21A	-179.9(2)
C19A-N2A-C5A-N3A	-71.1(3)
C19A-N2A-C5A-C1A	171.79(19)
C19A-N2A-C6A-C7A	-4.5(4)
C19A-N2A-C6A-C11A	175.8(2)
C20A-O4A-C19A-O3A	1.1(3)
C20A-O4A-C19A-N2A	-179.7(2)
S1-N1-C1-C2	173.30(17)
S1-N1-C1-C5	-68.9(2)
S1-N1-C1-C11	52.3(3)

S1-N1-C4-C3	171.96(17)
S1-C12-C13-C14	179.23(18)
S1-C12-C17-C16	179.72(19)
O1-S1-N1-C1	-21.0(2)
O1-S1-N1-C4	-174.45(17)
O1-S1-C12-C13	156.25(19)
O1-S1-C12-C17	-23.6(2)
O2-S1-N1-C1	111.5(2)
O2-S1-N1-C4	-41.9(2)
O2-S1-C12-C13	26.1(2)
O2-S1-C12-C17	-153.78(19)
N1-S1-C12-C13	-90.0(2)
N1-S1-C12-C17	90.2(2)
N1-C1-C2-C3	36.7(2)
N1-C1-C5-N2	154.06(18)
N1-C1-C5-N3	37.3(3)
N1-C1-C11-C6	-146.2(2)
N1-C1-C11-C10	41.1(3)
N2-C6-C7-C8	179.8(2)
N2-C6-C11-C1	5.8(3)
N2-C6-C11-C10	179.3(2)
N4-N3-C5-N2	112.8(2)
N4-N3-C5-C1	-134.2(2)
C1-N1-C4-C3	14.5(3)
C1-C2-C3-C4	-29.2(3)
C2-C1-C5-N2	-93.7(2)
C2-C1-C5-N3	149.54(19)
C2-C1-C11-C6	98.3(2)
C2-C1-C11-C10	-74.4(3)
C2-C3-C4-N1	9.8(3)
C4-N1-C1-C2	-31.9(2)
C4-N1-C1-C5	85.9(2)
C4-N1-C1-C11	-152.9(2)
C5-N2-C6-C7	-169.2(2)
C5-N2-C6-C11	11.3(3)
C5-N2-C19-O3	-5.3(4)
C5-N2-C19-O4	175.20(19)
C5-C1-C2-C3	-84.4(2)
C5-C1-C11-C6	-19.0(2)
C5-C1-C11-C10	168.3(2)

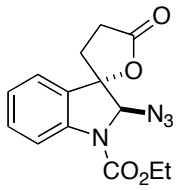
C6-N2-C5-N3	95.5(2)
C6-N2-C5-C1	-22.5(2)
C6-N2-C19-O3	-169.6(2)
C6-N2-C19-O4	11.0(4)
C6-C7-C8-C9	1.1(4)
C7-C6-C11-C1	-173.8(2)
C7-C6-C11-C10	-0.3(4)
C7-C8-C9-C10	-0.6(4)
C8-C9-C10-C11	-0.4(4)
C9-C10-C11-C1	172.9(2)
C9-C10-C11-C6	0.9(4)
C11-C1-C2-C3	162.9(2)
C11-C1-C5-N2	24.4(2)
C11-C1-C5-N3	-92.4(2)
C11-C6-C7-C8	-0.7(4)
C12-S1-N1-C1	-135.4(2)
C12-S1-N1-C4	71.14(19)
C12-C13-C14-C15	1.0(4)
C13-C12-C17-C16	-0.1(4)
C13-C14-C15-C16	0.0(4)
C13-C14-C15-C18	179.3(2)
C14-C15-C16-C17	-1.1(4)
C15-C16-C17-C12	1.1(4)
C17-C12-C13-C14	-0.9(4)
C18-C15-C16-C17	179.6(2)
C19-O4-C20-C21	176.1(2)
C19-N2-C5-N3	-71.9(3)
C19-N2-C5-C1	170.12(19)
C19-N2-C6-C7	-3.9(4)
C19-N2-C6-C11	176.5(2)
C20-O4-C19-O3	0.9(4)
C20-O4-C19-N2	-179.7(2)

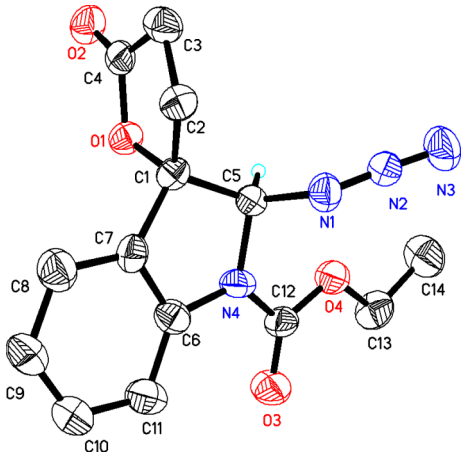
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for *syn*-**5a** [Å and °].

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

The X-ray structure of compound *anti*-**5h**





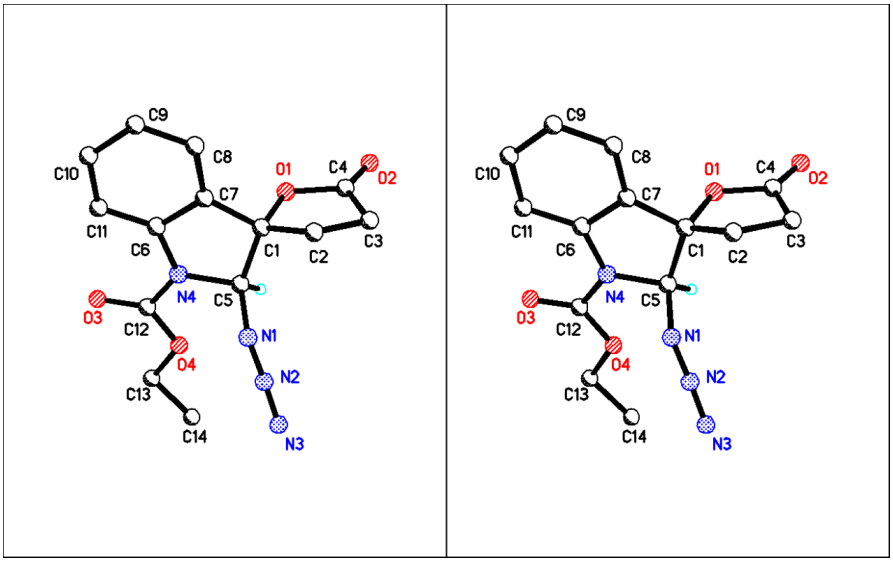


Table 1. Crystal data and structure refinement for *anti*-**5h**.

Identification code	*anti*-**5h**	
Empirical formula	$C_{14}H_{14}N_4O_4$	
Formula weight	302.29	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 2	
Unit cell dimensions	a = 18.769(4) Å	a= 90°.
	b = 12.495(3) Å	b= 90°.
	c = 13.680(3) Å	g = 90°.
Volume	3208.1(11) $Å^3$	
Z	8	
Density (calculated)	1.252 Mg/m^3	
Absorption coefficient	0.094 mm^{-1}	
F(000)	1264	
Crystal size	0.35 x 0.2 x 0.12 mm^3	
Theta range for data collection	2.714 to 27.468°.	
Index ranges	-24<=h<=23, -16<=k<=16, -16<=l<=17	
Reflections collected	26174	
Independent reflections	7275 [R(int) = 0.0732]	
Completeness to theta = 26.000°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.5174	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7275 / 0 / 399	
Goodness-of-fit on F^2	1.139	
Final R indices [I>2sigma(I)]	R1 = 0.0825, wR2 = 0.2014	
R indices (all data)	R1 = 0.0890, wR2 = 0.2066	
Absolute structure parameter	-0.2(8)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.566 and -0.296 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)for *anti*-**5h**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O1A	4453(2)	1122(3)	1498(2)	29(1)
O2A	5352(2)	1243(3)	437(3)	39(1)
O3A	2841(2)	2505(3)	4405(3)	42(1)
O4A	3067(2)	3664(3)	3172(3)	42(1)
N1A	2810(3)	2566(4)	1275(3)	38(1)
N2A	2801(3)	3552(4)	1153(3)	40(1)
N3A	2721(3)	4427(4)	969(4)	53(1)
N4A	3146(2)	1930(3)	2868(3)	35(1)
C1A	3671(3)	1072(4)	1489(3)	30(1)
C2A	3482(3)	797(4)	431(4)	33(1)
C3A	4127(3)	1194(4)	-152(4)	35(1)
C4A	4716(3)	1195(4)	579(4)	31(1)
C5A	3403(2)	2180(4)	1886(3)	29(1)
C6A	3120(3)	809(4)	3032(3)	27(1)
C7A	3434(3)	280(4)	2241(4)	32(1)
C8A	3494(3)	-810(4)	2225(5)	43(1)
C9A	3210(4)	-1387(4)	3013(5)	50(2)
C10A	2896(3)	-877(5)	3784(5)	46(1)
C11A	2851(3)	241(4)	3807(4)	40(1)
C12A	3005(3)	2690(4)	3568(4)	35(1)
C13A	2927(3)	4550(4)	3842(4)	42(1)
C14A	2962(4)	5584(5)	3278(6)	60(2)
O1	5171(2)	3533(3)	9212(3)	33(1)
O2	4495(2)	3708(3)	10528(3)	44(1)
O3	5950(3)	4832(3)	5781(3)	49(1)
O4	5975(2)	6044(3)	7018(3)	42(1)
N1	6816(2)	4960(4)	8634(3)	38(1)
N2	6873(2)	5942(4)	8744(3)	36(1)
N3	7009(3)	6807(4)	8865(4)	55(1)
N4	6023(3)	4311(4)	7377(3)	37(1)
C1	5927(2)	3511(4)	8941(4)	29(1)
C2	6313(3)	3305(4)	9919(4)	34(1)

C3	5798(3)	3677(4)	10694(4)	39(1)
C4	5087(3)	3642(4)	10199(4)	34(1)
C5	6076(3)	4583(4)	8418(3)	31(1)
C6	6052(3)	3182(4)	7238(4)	35(1)
C7	6015(3)	2694(4)	8150(4)	34(1)
C8	6027(3)	1576(5)	8217(5)	45(1)
C9	6080(4)	992(5)	7367(5)	49(2)
C10	6112(4)	1500(5)	6468(5)	52(2)
C11	6100(3)	2618(5)	6372(5)	46(1)
C12	5986(3)	5058(5)	6651(4)	37(1)
C13	5910(4)	6902(5)	6309(5)	50(2)
C14	5925(5)	7936(5)	6850(5)	62(2)

Table 3. Bond lengths [Å] and angles [°] for *anti*-**5h.**

O1A-C1A	1.468(6)
O1A-C4A	1.353(6)
O2A-C4A	1.211(6)
O3A-C12A	1.209(6)
O4A-C12A	1.336(7)
O4A-C13A	1.461(6)
N1A-N2A	1.243(6)
N1A-C5A	1.473(6)
N2A-N3A	1.131(7)
N4A-C5A	1.461(6)
N4A-C6A	1.419(6)
N4A-C12A	1.375(7)
C1A-C2A	1.530(7)
C1A-C5A	1.570(7)
C1A-C7A	1.496(7)
C2A-C3A	1.532(7)
C3A-C4A	1.491(7)
C6A-C7A	1.399(7)
C6A-C11A	1.371(7)
C7A-C8A	1.367(7)
C8A-C9A	1.402(9)
C9A-C10A	1.366(9)
C10A-C11A	1.399(8)
C13A-C14A	1.506(9)
O1-C1	1.467(5)
O1-C4	1.366(6)
O2-C4	1.201(6)
O3-C12	1.224(6)
O4-C12	1.331(7)
O4-C13	1.451(7)
N1-N2	1.240(7)
N1-C5	1.496(7)
N2-N3	1.124(7)
N4-C5	1.467(6)
N4-C6	1.425(7)
N4-C12	1.364(7)
C1-C2	1.543(7)
C1-C5	1.544(7)

C1-C7	1.497(7)
C2-C3	1.508(8)
C3-C4	1.498(8)
C6-C7	1.390(8)
C6-C11	1.381(8)
C7-C8	1.400(8)
C8-C9	1.376(9)
C9-C10	1.385(10)
C10-C11	1.404(9)
C13-C14	1.489(9)

C4A-O1A-C1A	111.1(4)
C12A-O4A-C13A	114.8(4)
N2A-N1A-C5A	114.2(4)
N3A-N2A-N1A	171.3(6)
C6A-N4A-C5A	111.6(4)
C12A-N4A-C5A	123.8(4)
C12A-N4A-C6A	124.4(4)
O1A-C1A-C2A	104.5(4)
O1A-C1A-C5A	106.3(4)
O1A-C1A-C7A	108.6(4)
C2A-C1A-C5A	116.8(4)
C7A-C1A-C2A	115.7(4)
C7A-C1A-C5A	104.5(4)
C1A-C2A-C3A	103.6(4)
C4A-C3A-C2A	103.7(4)
O1A-C4A-C3A	110.6(4)
O2A-C4A-O1A	120.8(5)
O2A-C4A-C3A	128.5(5)
N1A-C5A-C1A	109.5(4)
N4A-C5A-N1A	110.0(4)
N4A-C5A-C1A	103.6(4)
C7A-C6A-N4A	109.2(4)
C11A-C6A-N4A	130.3(5)
C11A-C6A-C7A	120.5(5)
C6A-C7A-C1A	110.2(4)
C8A-C7A-C1A	128.6(5)
C8A-C7A-C6A	121.2(5)
C7A-C8A-C9A	117.9(6)
C10A-C9A-C8A	121.2(5)

C9A-C10A-C11A	120.5(5)
C6A-C11A-C10A	118.6(5)
O3A-C12A-O4A	125.5(5)
O3A-C12A-N4A	125.2(5)
O4A-C12A-N4A	109.3(4)
O4A-C13A-C14A	108.7(5)
C4-O1-C1	111.4(4)
C12-O4-C13	115.7(4)
N2-N1-C5	114.5(4)
N3-N2-N1	171.6(6)
C6-N4-C5	110.9(4)
C12-N4-C5	123.5(4)
C12-N4-C6	125.6(4)
O1-C1-C2	103.7(4)
O1-C1-C5	106.1(4)
O1-C1-C7	107.6(4)
C2-C1-C5	117.5(4)
C7-C1-C2	117.4(4)
C7-C1-C5	103.7(4)
C3-C2-C1	104.9(4)
C4-C3-C2	104.1(4)
O1-C4-C3	110.3(4)
O2-C4-O1	119.0(5)
O2-C4-C3	130.8(5)
N1-C5-C1	110.5(4)
N4-C5-N1	109.1(4)
N4-C5-C1	103.7(4)
C7-C6-N4	108.2(5)
C11-C6-N4	128.4(5)
C11-C6-C7	123.3(5)
C6-C7-C1	110.8(4)
C6-C7-C8	119.7(5)
C8-C7-C1	129.4(5)
C9-C8-C7	118.3(6)
C8-C9-C10	120.7(6)
C9-C10-C11	122.6(6)
C6-C11-C10	115.3(6)
O3-C12-O4	125.3(5)
O3-C12-N4	123.6(5)
O4-C12-N4	111.1(4)

O4-C13-C14	107.9(5)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for *anti*-**5h**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1A	30(2)	27(2)	29(2)	2(1)	2(1)	0(1)
O2A	34(2)	39(2)	43(2)	5(2)	10(2)	-1(2)
O3A	60(2)	37(2)	28(2)	-1(2)	9(2)	1(2)
O4A	65(3)	25(2)	35(2)	1(2)	7(2)	9(2)
N1A	39(2)	34(2)	41(2)	5(2)	-5(2)	1(2)
N2A	45(3)	34(2)	39(2)	3(2)	-1(2)	9(2)
N3A	67(4)	41(3)	51(3)	16(2)	-4(3)	10(2)
N4A	48(3)	27(2)	29(2)	0(2)	7(2)	1(2)
C1A	32(2)	35(3)	25(2)	-2(2)	3(2)	1(2)
C2A	33(2)	35(3)	32(3)	-8(2)	5(2)	1(2)
C3A	41(3)	35(3)	27(2)	0(2)	-2(2)	1(2)
C4A	35(2)	21(2)	37(3)	3(2)	9(2)	-2(2)
C5A	27(2)	31(2)	29(2)	4(2)	-1(2)	-4(2)
C6A	31(2)	25(2)	27(2)	5(2)	-5(2)	1(2)
C7A	33(2)	20(2)	43(3)	-3(2)	3(2)	-3(2)
C8A	55(3)	29(3)	44(3)	-5(2)	7(3)	-3(2)
C9A	66(4)	21(2)	62(4)	6(3)	11(3)	3(3)
C10A	57(3)	34(3)	47(3)	6(2)	14(3)	-9(3)
C11A	50(3)	32(3)	37(3)	1(2)	6(3)	-2(2)
C12A	37(3)	38(3)	30(2)	-4(2)	4(2)	-4(2)
C13A	54(3)	36(3)	35(3)	-13(2)	2(3)	4(2)
C14A	84(5)	34(3)	62(4)	2(3)	13(4)	3(3)
O1	30(2)	37(2)	33(2)	1(2)	2(1)	1(2)
O2	40(2)	40(2)	53(2)	-3(2)	17(2)	0(2)
O3	71(3)	47(2)	30(2)	5(2)	-6(2)	-2(2)
O4	55(2)	34(2)	37(2)	3(2)	2(2)	-1(2)
N1	39(2)	32(2)	42(2)	1(2)	1(2)	-4(2)
N2	36(2)	38(3)	34(2)	0(2)	0(2)	-4(2)
N3	72(4)	36(3)	58(3)	-1(2)	-4(3)	-13(3)
N4	50(3)	37(2)	25(2)	-3(2)	3(2)	-1(2)
C1	24(2)	31(2)	32(2)	2(2)	3(2)	4(2)
C2	33(2)	36(3)	33(2)	2(2)	0(2)	2(2)

C3	51(3)	31(3)	36(3)	-2(2)	-4(2)	5(2)
C4	44(3)	20(2)	39(3)	-3(2)	10(2)	1(2)
C5	36(3)	30(2)	28(2)	-1(2)	0(2)	-3(2)
C6	34(3)	37(3)	35(3)	-3(2)	-3(2)	1(2)
C7	29(2)	34(3)	40(3)	2(2)	2(2)	-5(2)
C8	46(3)	39(3)	51(3)	-3(3)	2(3)	-1(3)
C9	62(4)	35(3)	50(3)	-10(3)	3(3)	1(3)
C10	63(4)	45(3)	49(3)	-12(3)	8(3)	-5(3)
C11	50(3)	48(3)	40(3)	-8(3)	2(3)	2(3)
C12	40(3)	45(3)	26(2)	1(2)	-2(2)	-5(2)
C13	61(4)	45(3)	45(3)	17(3)	-2(3)	3(3)
C14	90(5)	40(3)	57(4)	9(3)	-12(4)	0(4)

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

	x	y	z	U(eq)
H2AA	3050	1162	228	40
H2AB	3417	32	350	40
H3AA	4233	716	-691	42
H3AB	4044	1908	-407	42
H5A	3792	2704	1908	35
H8A	3716	-1159	1707	51
H9A	3235	-2130	3011	60
H10A	2710	-1276	4299	55
H11A	2644	590	4335	48
H13A	3278	4554	4362	50
H13B	2459	4468	4133	50
H14A	2598	5589	2785	90
H14B	3421	5648	2974	90
H14C	2890	6174	3716	90
H2A	6419	2550	9996	40
H2B	6754	3708	9951	40
H3A	5808	3203	11255	47
H3B	5911	4398	10907	47
H5	5723	5126	8599	38
H8	6001	1236	8821	54
H9	6094	249	7397	59
H10	6142	1083	5906	62
H11	6123	2956	5767	55
H13C	5466	6834	5951	60
H13D	6300	6872	5845	60
H14D	5860	8517	6399	94
H14E	6376	8013	7174	94
H14F	5549	7945	7325	94

Table 6. Torsion angles [°] for *anti*-**5h.**

O1A-C1A-C2A-C3A	-24.3(5)
O1A-C1A-C5A-N1A	136.1(4)
O1A-C1A-C5A-N4A	-106.5(4)
O1A-C1A-C7A-C6A	108.7(5)
O1A-C1A-C7A-C8A	-71.1(7)
N2A-N1A-C5A-N4A	102.9(5)
N2A-N1A-C5A-C1A	-143.9(5)
N4A-C6A-C7A-C1A	-1.4(6)
N4A-C6A-C7A-C8A	178.4(5)
N4A-C6A-C11A-C10A	-179.8(6)
C1A-O1A-C4A-O2A	-179.9(4)
C1A-O1A-C4A-C3A	-0.4(5)
C1A-C2A-C3A-C4A	23.9(5)
C1A-C7A-C8A-C9A	-178.2(5)
C2A-C1A-C5A-N1A	20.1(6)
C2A-C1A-C5A-N4A	137.4(4)
C2A-C1A-C7A-C6A	-134.3(5)
C2A-C1A-C7A-C8A	45.9(8)
C2A-C3A-C4A-O1A	-15.3(5)
C2A-C3A-C4A-O2A	164.1(5)
C4A-O1A-C1A-C2A	16.1(5)
C4A-O1A-C1A-C5A	-108.1(4)
C4A-O1A-C1A-C7A	140.0(4)
C5A-N4A-C6A-C7A	7.3(6)
C5A-N4A-C6A-C11A	-173.1(5)
C5A-N4A-C12A-O3A	-174.5(5)
C5A-N4A-C12A-O4A	6.6(7)
C5A-C1A-C2A-C3A	92.7(5)
C5A-C1A-C7A-C6A	-4.4(5)
C5A-C1A-C7A-C8A	175.7(6)
C6A-N4A-C5A-N1A	107.4(5)
C6A-N4A-C5A-C1A	-9.7(5)
C6A-N4A-C12A-O3A	-0.2(9)
C6A-N4A-C12A-O4A	-179.1(5)
C6A-C7A-C8A-C9A	2.0(9)
C7A-C1A-C2A-C3A	-143.6(4)
C7A-C1A-C5A-N1A	-109.1(4)
C7A-C1A-C5A-N4A	8.2(5)

C7A-C6A-C11A-C10A	-0.3(8)
C7A-C8A-C9A-C10A	-1.3(10)
C8A-C9A-C10A-C11A	-0.1(11)
C9A-C10A-C11A-C6A	0.9(10)
C11A-C6A-C7A-C1A	179.0(5)
C11A-C6A-C7A-C8A	-1.2(8)
C12A-O4A-C13A-C14A	-175.8(5)
C12A-N4A-C5A-N1A	-77.7(6)
C12A-N4A-C5A-C1A	165.3(5)
C12A-N4A-C6A-C7A	-167.6(5)
C12A-N4A-C6A-C11A	12.0(9)
C13A-O4A-C12A-O3A	0.8(8)
C13A-O4A-C12A-N4A	179.7(5)
O1-C1-C2-C3	-22.9(5)
O1-C1-C5-N1	145.9(4)
O1-C1-C5-N4	-97.3(4)
O1-C1-C7-C6	100.4(5)
O1-C1-C7-C8	-76.3(7)
N2-N1-C5-N4	104.6(5)
N2-N1-C5-C1	-142.0(5)
N4-C6-C7-C1	2.4(6)
N4-C6-C7-C8	179.5(5)
N4-C6-C11-C10	-179.4(6)
C1-O1-C4-O2	177.6(4)
C1-O1-C4-C3	-1.5(6)
C1-C2-C3-C4	22.0(5)
C1-C7-C8-C9	176.7(5)
C2-C1-C5-N1	30.5(6)
C2-C1-C5-N4	147.3(4)
C2-C1-C7-C6	-143.2(5)
C2-C1-C7-C8	40.1(8)
C2-C3-C4-O1	-13.5(6)
C2-C3-C4-O2	167.6(5)
C4-O1-C1-C2	15.4(5)
C4-O1-C1-C5	-109.0(4)
C4-O1-C1-C7	140.5(4)
C5-N4-C6-C7	8.8(6)
C5-N4-C6-C11	-172.0(6)
C5-N4-C12-O3	179.1(6)
C5-N4-C12-O4	-2.6(8)

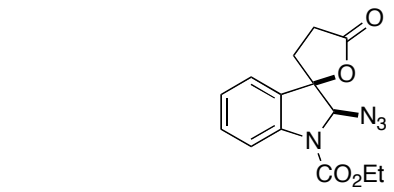
C5-C1-C2-C3	93.8(5)
C5-C1-C7-C6	-11.7(5)
C5-C1-C7-C8	171.6(6)
C6-N4-C5-N1	102.2(5)
C6-N4-C5-C1	-15.6(6)
C6-N4-C12-O3	2.4(9)
C6-N4-C12-O4	-179.3(5)
C6-C7-C8-C9	0.3(9)
C7-C1-C2-C3	-141.4(4)
C7-C1-C5-N1	-100.9(4)
C7-C1-C5-N4	15.9(5)
C7-C6-C11-C10	-0.2(9)
C7-C8-C9-C10	-0.7(10)
C8-C9-C10-C11	0.7(11)
C9-C10-C11-C6	-0.2(10)
C11-C6-C7-C1	-176.9(5)
C11-C6-C7-C8	0.2(8)
C12-O4-C13-C14	-178.6(6)
C12-N4-C5-N1	-74.9(6)
C12-N4-C5-C1	167.3(5)
C12-N4-C6-C7	-174.2(5)
C12-N4-C6-C11	5.1(10)
C13-O4-C12-O3	0.4(9)
C13-O4-C12-N4	-177.9(5)

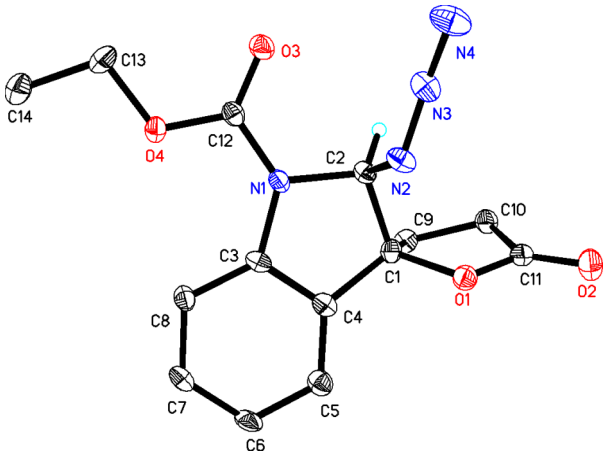
Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for *anti*-**5h** [Å and °].

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

The X-ray structure of compound *syn*-**5h**





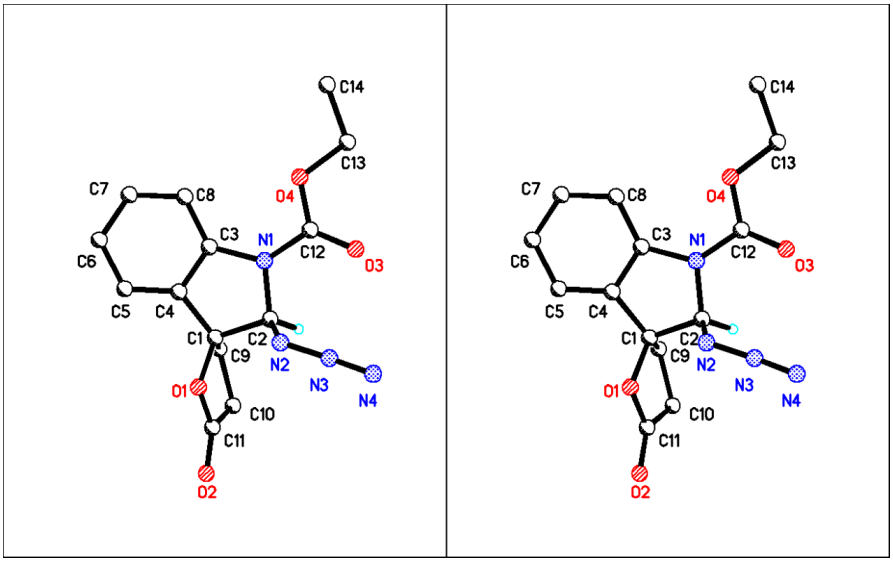


Table 1. Crystal data and structure refinement for *syn*-**5h**.

Identification code	*syn*-**5h**	
Empirical formula	$C_{14}H_{14}N_4O_4$	
Formula weight	302.29	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 7.764(3) Å	a= 80.083(16)°.
	b = 9.345(4) Å	b= 86.694(16)°.
	c = 20.472(9) Å	g = 79.180(13)°.
Volume	1436.7(11) $Å^3$	
Z	4	
Density (calculated)	1.398 Mg/m^3	
Absorption coefficient	0.105 mm^{-1}	
F(000)	632	
Crystal size	0.37 x 0.19 x 0.04 mm^3	
Theta range for data collection	1.010 to 25.198°.	
Index ranges	-9<=h<=9, -10<=k<=11, -8<=l<=24	
Reflections collected	5105	
Independent reflections	5105 [R(int) = ?]	
Completeness to theta = 25.242°	98.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.6022	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5105 / 24 / 419	
Goodness-of-fit on F^2	1.123	
Final R indices [I>2sigma(I)]	R1 = 0.1109, wR2 = 0.3066	
R indices (all data)	R1 = 0.1272, wR2 = 0.3194	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.445 and -0.495 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for *syn*-**5h**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O1A	1920(5)	3522(4)	8154(2)	32(1)
O2A	1867(7)	5424(6)	7330(2)	54(1)
O3A	-511(5)	3220(4)	10574(2)	35(1)
O4A	871(5)	859(4)	10866(2)	30(1)
N1A	1326(6)	2055(5)	9849(2)	29(1)
N2A	-718(6)	3444(5)	9052(3)	34(1)
N3A	-1479(15)	4667(14)	8769(9)	39(3)
N4A	-2207(19)	5724(15)	8495(9)	68(5)
C1A	2542(7)	3089(6)	8820(3)	26(1)
C2A	1002(7)	3372(6)	9334(3)	27(1)
C3A	2473(7)	891(6)	9599(3)	25(1)
C4A	3158(7)	1449(6)	8980(3)	27(1)
C5A	4329(7)	530(6)	8636(3)	31(1)
C6A	4774(8)	-944(7)	8898(3)	36(1)
C7A	4078(8)	-1497(7)	9516(3)	33(1)
C8A	2899(7)	-587(6)	9870(3)	28(1)
C9A	3945(7)	4016(6)	8864(3)	29(1)
C10A	3395(8)	5388(6)	8344(3)	32(1)
C11A	2346(8)	4850(7)	7872(3)	37(1)
C12A	489(7)	2111(6)	10451(3)	26(1)
C13A	-42(8)	834(7)	11515(3)	34(1)
C14A	607(9)	-638(7)	11914(3)	38(1)
O1	6579(5)	2145(4)	6833(2)	31(1)
O2	6310(7)	3631(6)	7593(2)	50(1)
O3	4744(5)	3668(4)	4382(2)	32(1)
O4	6165(5)	1534(4)	4084(2)	31(1)
N1	6382(6)	1937(5)	5126(2)	26(1)
N2	4155(6)	2766(5)	5917(3)	31(1)
N3	3046(6)	3887(6)	5742(3)	33(1)
N4	1920(7)	4828(6)	5616(3)	49(2)
C1	7335(7)	2116(6)	6180(3)	26(1)
C2	5951(7)	2849(6)	5644(3)	24(1)

C3	7371(6)	544(6)	5391(3)	24(1)
C4	7886(7)	579(6)	6025(3)	28(1)
C5	8864(8)	-640(6)	6391(3)	33(1)
C6	9327(8)	-1931(6)	6117(3)	34(1)
C7	8826(7)	-1953(6)	5483(3)	31(1)
C8	7835(7)	-736(6)	5108(3)	28(1)
C9	8857(7)	2980(6)	6148(3)	28(1)
C10	8211(8)	4077(6)	6619(3)	34(1)
C11	6962(8)	3325(7)	7076(3)	35(1)
C12	5682(7)	2467(6)	4512(3)	26(1)
C13	5488(9)	2048(7)	3425(3)	39(1)
C14	6234(10)	889(8)	3010(3)	46(2)
N3B	-1764(14)	4517(12)	9177(8)	37(3)
N4B	-2907(16)	5422(14)	9274(8)	67(5)

Table 3. Bond lengths [Å] and angles [°] for *syn*-**5h**.

O1A-C1A	1.438(7)
O1A-C11A	1.367(7)
O2A-C11A	1.193(8)
O3A-C12A	1.227(7)
O4A-C12A	1.317(7)
O4A-C13A	1.468(7)
N1A-C2A	1.469(7)
N1A-C3A	1.420(7)
N1A-C12A	1.364(7)
N2A-N3A	1.245(14)
N2A-C2A	1.470(7)
N2A-N3B	1.220(12)
N3A-N4A	1.116(17)
C1A-C2A	1.563(8)
C1A-C4A	1.502(7)
C1A-C9A	1.529(7)
C3A-C4A	1.399(8)
C3A-C8A	1.382(8)
C4A-C5A	1.379(8)
C5A-C6A	1.376(8)
C6A-C7A	1.399(9)
C7A-C8A	1.389(8)
C9A-C10A	1.529(8)
C10A-C11A	1.504(9)
C13A-C14A	1.488(8)
O1-C1	1.432(6)
O1-C11	1.371(7)
O2-C11	1.203(7)
O3-C12	1.214(7)
O4-C12	1.330(6)
O4-C13	1.447(7)
N1-C2	1.458(7)
N1-C3	1.416(7)
N1-C12	1.372(7)
N2-N3	1.239(7)
N2-C2	1.482(7)
N3-N4	1.124(7)
C1-C2	1.558(7)

C1-C4	1.502(8)
C1-C9	1.544(7)
C3-C4	1.389(8)
C3-C8	1.396(8)
C4-C5	1.371(8)
C5-C6	1.395(8)
C6-C7	1.382(9)
C7-C8	1.384(8)
C9-C10	1.525(8)
C10-C11	1.496(9)
C13-C14	1.501(9)
N3B-N4B	1.139(15)

C11A-O1A-C1A	110.8(4)
C12A-O4A-C13A	115.0(4)
C3A-N1A-C2A	110.3(4)
C12A-N1A-C2A	118.4(4)
C12A-N1A-C3A	131.3(5)
N3A-N2A-C2A	117.7(7)
N3B-N2A-C2A	111.4(7)
N4A-N3A-N2A	176.3(14)
O1A-C1A-C2A	110.6(4)
O1A-C1A-C4A	111.4(4)
O1A-C1A-C9A	105.4(4)
C4A-C1A-C2A	102.8(4)
C4A-C1A-C9A	114.8(5)
C9A-C1A-C2A	112.0(5)
N1A-C2A-N2A	108.1(4)
N1A-C2A-C1A	103.6(4)
N2A-C2A-C1A	112.2(5)
C4A-C3A-N1A	108.9(5)
C8A-C3A-N1A	129.6(5)
C8A-C3A-C4A	121.5(5)
C3A-C4A-C1A	110.3(5)
C5A-C4A-C1A	129.5(5)
C5A-C4A-C3A	120.1(5)
C6A-C5A-C4A	119.4(6)
C5A-C6A-C7A	120.3(5)
C8A-C7A-C6A	121.1(6)
C3A-C8A-C7A	117.7(5)

C10A-C9A-C1A	103.2(4)
C11A-C10A-C9A	103.3(5)
O1A-C11A-C10A	110.0(5)
O2A-C11A-O1A	120.6(6)
O2A-C11A-C10A	129.4(6)
O3A-C12A-O4A	125.3(5)
O3A-C12A-N1A	122.2(5)
O4A-C12A-N1A	112.5(5)
O4A-C13A-C14A	106.7(5)
C11-O1-C1	109.8(4)
C12-O4-C13	115.0(4)
C3-N1-C2	110.1(4)
C12-N1-C2	118.3(4)
C12-N1-C3	131.3(4)
N3-N2-C2	114.6(5)
N4-N3-N2	173.1(6)
O1-C1-C2	110.8(4)
O1-C1-C4	113.0(4)
O1-C1-C9	104.7(4)
C4-C1-C2	103.0(4)
C4-C1-C9	113.8(4)
C9-C1-C2	111.6(4)
N1-C2-N2	110.2(4)
N1-C2-C1	103.1(4)
N2-C2-C1	110.4(4)
C4-C3-N1	109.6(5)
C4-C3-C8	120.8(5)
C8-C3-N1	129.7(5)
C3-C4-C1	109.3(5)
C5-C4-C1	129.5(5)
C5-C4-C3	121.0(5)
C4-C5-C6	119.0(6)
C7-C6-C5	119.7(5)
C6-C7-C8	122.1(5)
C7-C8-C3	117.4(5)
C10-C9-C1	102.6(4)
C11-C10-C9	103.1(5)
O1-C11-C10	110.9(5)
O2-C11-O1	120.3(6)
O2-C11-C10	128.8(6)

O3-C12-O4	124.7(5)
O3-C12-N1	122.7(5)
O4-C12-N1	112.6(4)
O4-C13-C14	107.2(5)
N4B-N3B-N2A	170.9(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for *syn*-**5h**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1A	32(2)	32(2)	32(2)	-4(2)	-2(2)	-8(2)
O2A	54(3)	57(3)	45(3)	16(2)	-16(2)	-14(2)
O3A	36(2)	27(2)	42(2)	-6(2)	11(2)	-7(2)
O4A	31(2)	29(2)	28(2)	0(2)	0(2)	-6(2)
N1A	33(2)	21(2)	31(2)	-3(2)	6(2)	-3(2)
N2A	22(2)	28(3)	50(3)	-8(2)	-1(2)	-1(2)
N3A	24(5)	38(6)	56(7)	-8(6)	-12(5)	0(4)
N4A	57(8)	40(7)	104(12)	1(7)	-51(8)	0(6)
C1A	29(3)	21(3)	28(3)	2(2)	-2(2)	-6(2)
C2A	28(3)	18(3)	35(3)	0(2)	0(2)	-6(2)
C3A	24(3)	19(3)	32(3)	-4(2)	-5(2)	-2(2)
C4A	27(3)	24(3)	28(3)	-3(2)	-2(2)	-3(2)
C5A	28(3)	28(3)	35(3)	-3(2)	-2(2)	-1(2)
C6A	29(3)	31(3)	46(3)	-17(3)	-3(3)	8(2)
C7A	31(3)	28(3)	42(3)	-11(3)	-8(3)	-2(2)
C8A	26(3)	25(3)	35(3)	-4(2)	-5(2)	-5(2)
C9A	27(3)	35(3)	26(3)	-4(2)	-3(2)	-11(2)
C10A	30(3)	29(3)	37(3)	-4(2)	9(2)	-4(2)
C11A	28(3)	38(3)	40(3)	1(3)	3(3)	-3(3)
C12A	25(3)	25(3)	31(3)	-7(2)	1(2)	-6(2)
C13A	38(3)	38(3)	26(3)	-5(2)	5(2)	-8(3)
C14A	42(3)	40(3)	31(3)	-7(3)	0(3)	-4(3)
O1	36(2)	28(2)	30(2)	-5(2)	1(2)	-7(2)
O2	55(3)	54(3)	40(3)	-19(2)	1(2)	4(2)
O3	33(2)	25(2)	38(2)	-9(2)	-7(2)	-5(2)
O4	35(2)	31(2)	28(2)	-11(2)	-3(2)	-4(2)
N1	28(2)	22(2)	29(2)	-8(2)	-2(2)	-1(2)
N2	25(2)	24(2)	42(3)	-3(2)	1(2)	-2(2)
N3	22(2)	34(3)	46(3)	-10(2)	2(2)	-8(2)
N4	27(3)	41(3)	72(4)	1(3)	2(3)	-3(3)
C1	25(3)	29(3)	25(3)	-7(2)	4(2)	-6(2)
C2	23(2)	20(2)	30(3)	-6(2)	-1(2)	-7(2)

C3	18(2)	18(2)	35(3)	-1(2)	0(2)	-5(2)
C4	24(3)	24(3)	35(3)	-4(2)	4(2)	-5(2)
C5	32(3)	30(3)	38(3)	-8(2)	-4(2)	-7(2)
C6	29(3)	24(3)	45(3)	-4(3)	-10(3)	8(2)
C7	31(3)	20(3)	42(3)	-9(2)	3(2)	-4(2)
C8	27(3)	28(3)	31(3)	-12(2)	-2(2)	-3(2)
C9	26(3)	31(3)	30(3)	-4(2)	-2(2)	-12(2)
C10	41(3)	30(3)	35(3)	-10(2)	-6(3)	-5(3)
C11	41(3)	31(3)	29(3)	-6(2)	-8(3)	5(3)
C12	27(3)	22(3)	31(3)	-6(2)	3(2)	-6(2)
C13	45(4)	46(4)	25(3)	-1(3)	-7(3)	-11(3)
C14	56(4)	54(4)	29(3)	-10(3)	0(3)	-6(3)
N3B	27(5)	32(5)	56(7)	-12(5)	1(5)	-9(4)
N4B	41(7)	46(7)	119(12)	-38(7)	-17(7)	6(6)

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

	x	y	z	U(eq)
H2A	1034	4268	9518	33
H5A	4812	903	8231	37
H6A	5541	-1575	8664	43
H7A	4411	-2491	9692	40
H8A	2413	-959	10275	34
H9AA	3944	4274	9303	35
H9AB	5102	3493	8760	35
H10A	2686	6178	8544	39
H10B	4411	5736	8121	39
H13A	-1300	977	11463	41
H13B	212	1614	11731	41
H14A	486	-1399	11669	57
H14B	-65	-751	12322	57
H14C	1821	-714	12010	57
H2	6070	3874	5476	28
H5	9213	-606	6814	39
H6	9970	-2774	6361	41
H7	9166	-2814	5302	37
H8	7492	-769	4683	34
H9A	9938	2331	6298	34
H9B	9044	3484	5701	34
H10C	9173	4248	6859	41
H10D	7620	5013	6380	41
H13C	5838	2981	3238	46
H13D	4217	2192	3440	46
H14D	5774	1176	2573	70
H14E	5918	-36	3210	70
H14F	7489	787	2984	70

Table 6. Torsion angles [°] for *syn*-**5h**.

O1A-C1A-C2A-N1A	138.4(4)
O1A-C1A-C2A-N2A	22.1(6)
O1A-C1A-C4A-C3A	-133.1(5)
O1A-C1A-C4A-C5A	51.8(8)
O1A-C1A-C9A-C10A	26.4(5)
N1A-C3A-C4A-C1A	3.7(6)
N1A-C3A-C4A-C5A	179.4(5)
N1A-C3A-C8A-C7A	-179.8(5)
N3A-N2A-C2A-N1A	158.0(10)
N3A-N2A-C2A-C1A	-88.4(11)
C1A-O1A-C11A-O2A	-176.5(6)
C1A-O1A-C11A-C10A	2.4(6)
C1A-C4A-C5A-C6A	176.5(5)
C1A-C9A-C10A-C11A	-24.4(5)
C2A-N1A-C3A-C4A	10.0(6)
C2A-N1A-C3A-C8A	-168.4(5)
C2A-N1A-C12A-O3A	-0.4(8)
C2A-N1A-C12A-O4A	179.2(5)
C2A-C1A-C4A-C3A	-14.7(6)
C2A-C1A-C4A-C5A	170.2(6)
C2A-C1A-C9A-C10A	-93.9(5)
C3A-N1A-C2A-N2A	100.7(5)
C3A-N1A-C2A-C1A	-18.6(6)
C3A-N1A-C12A-O3A	-177.8(5)
C3A-N1A-C12A-O4A	1.8(8)
C3A-C4A-C5A-C6A	1.8(9)
C4A-C1A-C2A-N1A	19.4(5)
C4A-C1A-C2A-N2A	-97.0(5)
C4A-C1A-C9A-C10A	149.4(5)
C4A-C3A-C8A-C7A	1.9(8)
C4A-C5A-C6A-C7A	-1.5(9)
C5A-C6A-C7A-C8A	1.5(9)
C6A-C7A-C8A-C3A	-1.7(8)
C8A-C3A-C4A-C1A	-177.7(5)
C8A-C3A-C4A-C5A	-2.1(8)
C9A-C1A-C2A-N1A	-104.3(5)
C9A-C1A-C2A-N2A	139.3(5)
C9A-C1A-C4A-C3A	107.3(5)

C9A-C1A-C4A-C5A	-67.8(8)
C9A-C10A-C11A-O1A	14.6(6)
C9A-C10A-C11A-O2A	-166.6(6)
C11A-O1A-C1A-C2A	102.7(5)
C11A-O1A-C1A-C4A	-143.6(5)
C11A-O1A-C1A-C9A	-18.5(6)
C12A-O4A-C13A-C14A	-178.1(5)
C12A-N1A-C2A-N2A	-77.2(6)
C12A-N1A-C2A-C1A	163.5(5)
C12A-N1A-C3A-C4A	-172.5(6)
C12A-N1A-C3A-C8A	9.1(10)
C13A-O4A-C12A-O3A	1.7(8)
C13A-O4A-C12A-N1A	-177.9(5)
O1-C1-C2-N1	-142.8(4)
O1-C1-C2-N2	-25.1(6)
O1-C1-C4-C3	136.2(5)
O1-C1-C4-C5	-48.9(8)
O1-C1-C9-C10	-29.8(5)
N1-C3-C4-C1	-4.6(6)
N1-C3-C4-C5	180.0(5)
N1-C3-C8-C7	179.9(5)
N3-N2-C2-N1	-106.3(5)
N3-N2-C2-C1	140.5(5)
C1-O1-C11-O2	172.9(5)
C1-O1-C11-C10	-6.8(6)
C1-C4-C5-C6	-174.9(5)
C1-C9-C10-C11	25.2(6)
C2-N1-C3-C4	-10.6(6)
C2-N1-C3-C8	169.3(5)
C2-N1-C12-O3	1.1(8)
C2-N1-C12-O4	-179.7(4)
C2-C1-C4-C3	16.6(6)
C2-C1-C4-C5	-168.5(6)
C2-C1-C9-C10	90.1(5)
C3-N1-C2-N2	-97.5(5)
C3-N1-C2-C1	20.3(5)
C3-N1-C12-O3	174.2(5)
C3-N1-C12-O4	-6.6(8)
C3-C4-C5-C6	-0.6(9)
C4-C1-C2-N1	-21.7(5)

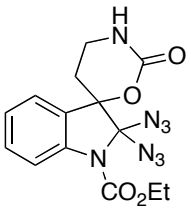
C4-C1-C2-N2	96.0(5)
C4-C1-C9-C10	-153.8(5)
C4-C3-C8-C7	-0.2(8)
C4-C5-C6-C7	1.3(9)
C5-C6-C7-C8	-1.5(9)
C6-C7-C8-C3	0.9(9)
C8-C3-C4-C1	175.5(5)
C8-C3-C4-C5	0.1(8)
C9-C1-C2-N1	100.9(5)
C9-C1-C2-N2	-141.5(4)
C9-C1-C4-C3	-104.5(5)
C9-C1-C4-C5	70.4(7)
C9-C10-C11-O1	-12.7(6)
C9-C10-C11-O2	167.6(6)
C11-O1-C1-C2	-97.2(5)
C11-O1-C1-C4	147.8(5)
C11-O1-C1-C9	23.3(6)
C12-O4-C13-C14	177.5(5)
C12-N1-C2-N2	77.0(6)
C12-N1-C2-C1	-165.2(4)
C12-N1-C3-C4	175.8(5)
C12-N1-C3-C8	-4.2(9)
C13-O4-C12-O3	0.1(8)
C13-O4-C12-N1	-179.0(5)
N3B-N2A-C2A-N1A	114.2(9)
N3B-N2A-C2A-C1A	-132.2(9)

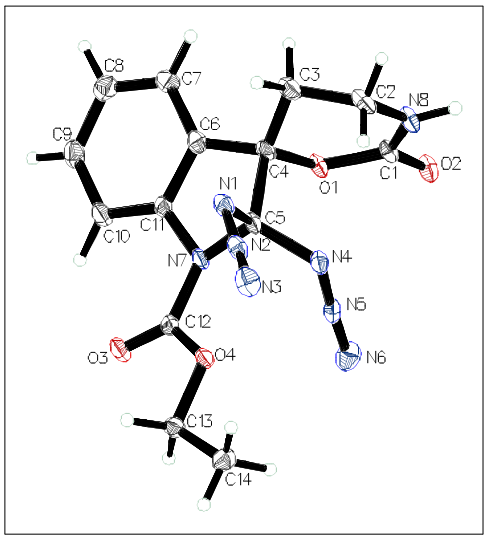
Symmetry transformations used to generate equivalent atoms:

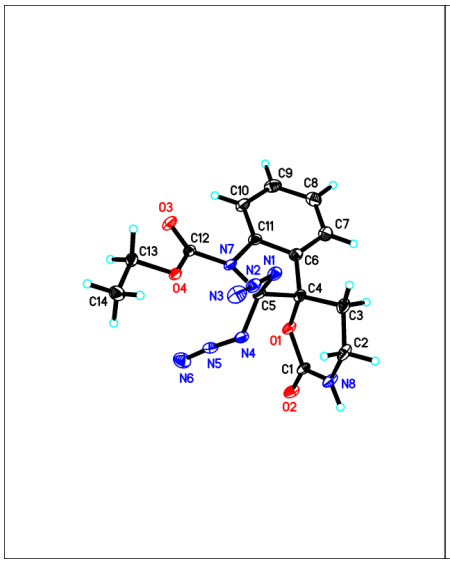
Table 7. Hydrogen bonds for *syn*-**5h** [Å and °].

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

The X-ray structure of compound **5i**







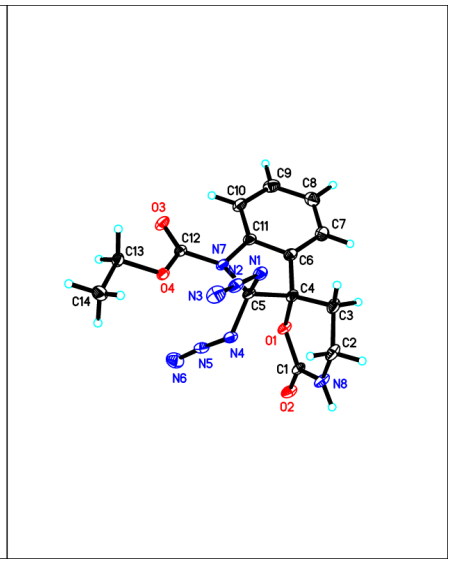


Table 1. Crystal data and structure refinement for **5i**.

Identification code	**5i**
Empirical formula	$C_{14}H_{14}N_8O_4$
Formula weight	358.33
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 13.410(3) A alpha = 90 deg. b = 6.5061(13) A beta = 94.61(3) deg. c = 18.689(4) A gamma = 90 deg.
Volume	1625.3(6) A^3
Z, Calculated density	4, 1.464 Mg/m^3
Absorption coefficient	0.112 mm^-1
F(000)	744
Crystal size	0.35 x 0.21 x 0.13 mm
Theta range for data collection	2.19 to 25.00 deg.
Limiting indices	-15<=h<=15, -7<=k<=7, -22<=l<=22
Reflections collected / unique	9426 / 2850 [R(int) = 0.0361]
Completeness to theta = 25.00	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7854
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2850 / 0 / 236
Goodness-of-fit on F^2	1.205
Final R indices [I>2sigma(I)]	R1 = 0.0534, wR2 = 0.1803
R indices (all data)	R1 = 0.0634, wR2 = 0.2237
Largest diff. peak and hole	0.515 and -0.470 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **5i**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	8735(1)	5990(3)	1462(1)	28(1)
O(2)	9602(1)	7008(3)	582(1)	34(1)
O(3)	5555(1)	8462(3)	2545(1)	33(1)
O(4)	5218(1)	6832(3)	1495(1)	26(1)
N(1)	6477(2)	2954(3)	1682(1)	28(1)
N(2)	5716(2)	2688(3)	1263(1)	30(1)
N(3)	5027(2)	2240(4)	913(1)	41(1)
N(4)	6885(2)	5404(3)	809(1)	27(1)
N(5)	6814(2)	7252(4)	668(1)	27(1)
N(6)	6719(2)	8876(4)	477(1)	45(1)
N(7)	6753(1)	6487(3)	2066(1)	25(1)
N(8)	9114(2)	3685(3)	576(1)	32(1)
C(1)	9169(2)	5565(4)	844(1)	26(1)
C(2)	8560(2)	1987(4)	867(2)	35(1)
C(3)	8531(2)	2294(4)	1663(2)	34(1)
C(4)	8157(2)	4430(4)	1819(1)	26(1)
C(5)	7020(2)	4869(4)	1568(1)	24(1)
C(6)	8211(2)	5094(4)	2589(1)	29(1)
C(7)	8928(2)	4647(5)	3147(2)	35(1)
C(8)	8854(2)	5560(5)	3805(2)	38(1)
C(9)	8089(2)	6958(5)	3897(2)	39(1)
C(10)	7363(2)	7399(4)	3353(1)	33(1)
C(11)	7424(2)	6394(4)	2699(1)	27(1)
C(12)	5800(2)	7350(4)	2079(1)	24(1)
C(13)	4192(2)	7610(4)	1482(1)	28(1)
C(14)	3690(2)	7104(5)	765(2)	39(1)

Table 3. Bond lengths [A] and angles [deg] for **5i**.

O(1)-C(1)	1.364(3)
O(1)-C(4)	1.469(3)
O(2)-C(1)	1.226(3)
O(3)-C(12)	1.197(3)
O(4)-C(12)	1.334(3)
O(4)-C(13)	1.465(3)
N(1)-N(2)	1.247(3)
N(1)-C(5)	1.468(3)
N(2)-N(3)	1.127(3)
N(4)-N(5)	1.232(3)
N(4)-C(5)	1.459(3)
N(5)-N(6)	1.119(3)
N(7)-C(12)	1.398(3)
N(7)-C(11)	1.429(3)
N(7)-C(5)	1.468(3)
N(8)-C(1)	1.321(3)
N(8)-C(2)	1.460(3)
N(8)-H(8)	0.8800
C(2)-C(3)	1.504(4)
C(2)-H(2B)	0.9900
C(2)-H(2A)	0.9900
C(3)-C(4)	1.514(4)
C(3)-H(3B)	0.9900
C(3)-H(3A)	0.9900
C(4)-C(6)	1.498(4)
C(4)-C(5)	1.584(3)
C(6)-C(11)	1.380(4)
C(6)-C(7)	1.391(4)
C(7)-C(8)	1.376(4)
C(7)-H(7)	0.9500
C(8)-C(9)	1.392(4)
C(8)-H(8A)	0.9500
C(9)-C(10)	1.380(4)
C(9)-H(9)	0.9500
C(10)-C(11)	1.396(4)
C(10)-H(10)	0.9500
C(13)-C(14)	1.487(4)
C(13)-H(13B)	0.9900
C(13)-H(13A)	0.9900
C(14)-H(14C)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14A)	0.9800

C(1)-O(1)-C(4)	121.41(18)
C(12)-O(4)-C(13)	114.36(18)
N(2)-N(1)-C(5)	114.7(2)
N(3)-N(2)-N(1)	172.6(3)
N(5)-N(4)-C(5)	116.21(19)
N(6)-N(5)-N(4)	173.5(3)
C(12)-N(7)-C(11)	121.21(19)
C(12)-N(7)-C(5)	124.75(19)
C(11)-N(7)-C(5)	108.90(19)
C(1)-N(8)-C(2)	125.1(2)
C(1)-N(8)-H(8)	117.5
C(2)-N(8)-H(8)	117.5
O(2)-C(1)-N(8)	124.9(2)
O(2)-C(1)-O(1)	115.5(2)
N(8)-C(1)-O(1)	119.6(2)
N(8)-C(2)-C(3)	108.8(2)
N(8)-C(2)-H(2B)	109.9
C(3)-C(2)-H(2B)	109.9
N(8)-C(2)-H(2A)	109.9
C(3)-C(2)-H(2A)	109.9
H(2B)-C(2)-H(2A)	108.3
C(2)-C(3)-C(4)	110.4(2)
C(2)-C(3)-H(3B)	109.6
C(4)-C(3)-H(3B)	109.6
C(2)-C(3)-H(3A)	109.6
C(4)-C(3)-H(3A)	109.6
H(3B)-C(3)-H(3A)	108.1
O(1)-C(4)-C(6)	104.46(19)
O(1)-C(4)-C(3)	110.57(19)
C(6)-C(4)-C(3)	117.3(2)
O(1)-C(4)-C(5)	105.78(18)
C(6)-C(4)-C(5)	101.72(19)
C(3)-C(4)-C(5)	115.8(2)
N(4)-C(5)-N(1)	108.63(19)
N(4)-C(5)-N(7)	115.3(2)
N(1)-C(5)-N(7)	111.54(19)
N(4)-C(5)-C(4)	111.84(19)
N(1)-C(5)-C(4)	106.36(19)
N(7)-C(5)-C(4)	102.74(18)
C(11)-C(6)-C(7)	120.4(2)
C(11)-C(6)-C(4)	109.9(2)
C(7)-C(6)-C(4)	129.6(2)
C(8)-C(7)-C(6)	119.0(3)
C(8)-C(7)-H(7)	120.5
C(6)-C(7)-H(7)	120.5
C(7)-C(8)-C(9)	120.0(3)

C(7)-C(8)-H(8A)	120.0
C(9)-C(8)-H(8A)	120.0
C(10)-C(9)-C(8)	121.8(3)
C(10)-C(9)-H(9)	119.1
C(8)-C(9)-H(9)	119.1
C(9)-C(10)-C(11)	117.4(2)
C(9)-C(10)-H(10)	121.3
C(11)-C(10)-H(10)	121.3
C(6)-C(11)-C(10)	121.1(2)
C(6)-C(11)-N(7)	109.9(2)
C(10)-C(11)-N(7)	128.9(2)
O(3)-C(12)-O(4)	124.8(2)
O(3)-C(12)-N(7)	124.1(2)
O(4)-C(12)-N(7)	111.0(2)
O(4)-C(13)-C(14)	107.1(2)
O(4)-C(13)-H(13B)	110.3
C(14)-C(13)-H(13B)	110.3
O(4)-C(13)-H(13A)	110.3
C(14)-C(13)-H(13A)	110.3
H(13B)-C(13)-H(13A)	108.5
C(13)-C(14)-H(14C)	109.5
C(13)-C(14)-H(14B)	109.5
H(14C)-C(14)-H(14B)	109.5
C(13)-C(14)-H(14A)	109.5
H(14C)-C(14)-H(14A)	109.5
H(14B)-C(14)-H(14A)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **5i**. 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
O(1)	24(1)	29(1)	32(1)	-5(1)	13(1)	-2(1)
O(2)	36(1)	31(1)	36(1)	-5(1)	18(1)	-6(1)
O(3)	25(1)	45(1)	30(1)	-13(1)	6(1)	5(1)
O(4)	21(1)	34(1)	22(1)	-3(1)	4(1)	4(1)
N(1)	23(1)	30(1)	33(1)	0(1)	9(1)	-3(1)
N(2)	26(1)	29(1)	36(1)	-3(1)	14(1)	-3(1)
N(3)	33(1)	46(2)	44(2)	-9(1)	6(1)	-10(1)
N(4)	31(1)	28(1)	23(1)	-2(1)	10(1)	0(1)
N(5)	28(1)	35(1)	21(1)	-1(1)	7(1)	-6(1)
N(6)	53(2)	39(2)	42(1)	9(1)	8(1)	-6(1)
N(7)	20(1)	37(1)	19(1)	-5(1)	5(1)	4(1)
N(8)	30(1)	29(1)	38(1)	-10(1)	18(1)	-3(1)
C(1)	19(1)	32(1)	29(1)	-4(1)	10(1)	-1(1)
C(2)	28(1)	26(1)	52(2)	-8(1)	19(1)	-1(1)
C(3)	26(1)	31(2)	47(2)	2(1)	14(1)	3(1)
C(4)	18(1)	30(1)	33(1)	3(1)	10(1)	1(1)
C(5)	22(1)	28(1)	24(1)	-1(1)	11(1)	1(1)
C(6)	22(1)	34(1)	30(1)	3(1)	8(1)	-2(1)
C(7)	19(1)	47(2)	39(2)	3(1)	3(1)	0(1)
C(8)	28(1)	50(2)	35(2)	8(1)	-2(1)	-4(1)
C(9)	33(2)	56(2)	28(1)	-7(1)	4(1)	-6(1)
C(10)	27(1)	43(2)	29(1)	-8(1)	6(1)	4(1)
C(11)	19(1)	39(1)	24(1)	2(1)	5(1)	-1(1)
C(12)	22(1)	27(1)	23(1)	0(1)	6(1)	1(1)
C(13)	22(1)	29(1)	33(1)	3(1)	5(1)	3(1)
C(14)	34(2)	53(2)	30(2)	7(1)	-1(1)	3(1)

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

	x	y	z	U(eq)
H(8)	9435	3437	193	38
H(2B)	8891	666	771	41
H(2A)	7871	1947	634	41
H(3B)	9210	2103	1902	41
H(3A)	8084	1257	1856	41
H(7)	9460	3725	3075	42
H(8A)	9325	5236	4195	46
H(9)	8067	7628	4347	46
H(10)	6842	8350	3422	39
H(13B)	4195	9116	1559	33
H(13A)	3835	6954	1865	33
H(14C)	4024	7832	392	59
H(14B)	2987	7527	747	59
H(14A)	3728	5619	683	59

Table 6. Torsion angles [deg] for **5i**.

C(5)-N(1)-N(2)-N(3)	169(2)
C(5)-N(4)-N(5)-N(6)	-167(2)
C(2)-N(8)-C(1)-O(2)	-177.0(3)
C(2)-N(8)-C(1)-O(1)	4.0(4)
C(4)-O(1)-C(1)-O(2)	177.5(2)
C(4)-O(1)-C(1)-N(8)	-3.4(3)
C(1)-N(8)-C(2)-C(3)	-29.2(3)
N(8)-C(2)-C(3)-C(4)	51.9(3)
C(1)-O(1)-C(4)-C(6)	155.4(2)
C(1)-O(1)-C(4)-C(3)	28.3(3)
C(1)-O(1)-C(4)-C(5)	-97.7(2)
C(2)-C(3)-C(4)-O(1)	-52.4(3)
C(2)-C(3)-C(4)-C(6)	-172.0(2)
C(2)-C(3)-C(4)-C(5)	67.8(3)
N(5)-N(4)-C(5)-N(1)	143.9(2)
N(5)-N(4)-C(5)-N(7)	17.9(3)
N(5)-N(4)-C(5)-C(4)	-99.0(2)
N(2)-N(1)-C(5)-N(4)	-30.4(3)
N(2)-N(1)-C(5)-N(7)	97.8(2)
N(2)-N(1)-C(5)-C(4)	-150.9(2)
C(12)-N(7)-C(5)-N(4)	60.6(3)
C(11)-N(7)-C(5)-N(4)	-144.7(2)
C(12)-N(7)-C(5)-N(1)	-63.9(3)
C(11)-N(7)-C(5)-N(1)	90.8(2)
C(12)-N(7)-C(5)-C(4)	-177.5(2)
C(11)-N(7)-C(5)-C(4)	-22.7(2)
O(1)-C(4)-C(5)-N(4)	40.6(3)
C(6)-C(4)-C(5)-N(4)	149.4(2)
C(3)-C(4)-C(5)-N(4)	-82.2(3)
O(1)-C(4)-C(5)-N(1)	159.02(18)
C(6)-C(4)-C(5)-N(1)	-92.1(2)
C(3)-C(4)-C(5)-N(1)	36.2(3)
O(1)-C(4)-C(5)-N(7)	-83.7(2)
C(6)-C(4)-C(5)-N(7)	25.2(2)
C(3)-C(4)-C(5)-N(7)	153.5(2)
O(1)-C(4)-C(6)-C(11)	89.8(2)
C(3)-C(4)-C(6)-C(11)	-147.4(2)
C(5)-C(4)-C(6)-C(11)	-20.1(3)
O(1)-C(4)-C(6)-C(7)	-87.5(3)
C(3)-C(4)-C(6)-C(7)	35.3(4)
C(5)-C(4)-C(6)-C(7)	162.6(3)
C(11)-C(6)-C(7)-C(8)	-1.6(4)
C(4)-C(6)-C(7)-C(8)	175.4(3)

C(6)-C(7)-C(8)-C(9)	-2.1(4)
C(7)-C(8)-C(9)-C(10)	3.1(4)
C(8)-C(9)-C(10)-C(11)	-0.3(4)
C(7)-C(6)-C(11)-C(10)	4.4(4)
C(4)-C(6)-C(11)-C(10)	-173.2(2)
C(7)-C(6)-C(11)-N(7)	-175.6(2)
C(4)-C(6)-C(11)-N(7)	6.8(3)
C(9)-C(10)-C(11)-C(6)	-3.4(4)
C(9)-C(10)-C(11)-N(7)	176.6(3)
C(12)-N(7)-C(11)-C(6)	166.8(2)
C(5)-N(7)-C(11)-C(6)	11.1(3)
C(12)-N(7)-C(11)-C(10)	-13.2(4)
C(5)-N(7)-C(11)-C(10)	-168.9(3)
C(13)-O(4)-C(12)-O(3)	-4.6(3)
C(13)-O(4)-C(12)-N(7)	177.66(19)
C(11)-N(7)-C(12)-O(3)	17.7(4)
C(5)-N(7)-C(12)-O(3)	169.5(2)
C(11)-N(7)-C(12)-O(4)	-164.5(2)
C(5)-N(7)-C(12)-O(4)	-12.7(3)
C(12)-O(4)-C(13)-C(14)	173.6(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **5i** [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(8)-H(8)...O(2)#1	0.88	2.04	2.908(3)	170.0

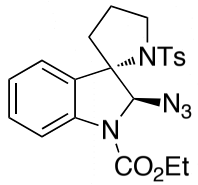
Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+1,-z

Table 2, entry 1

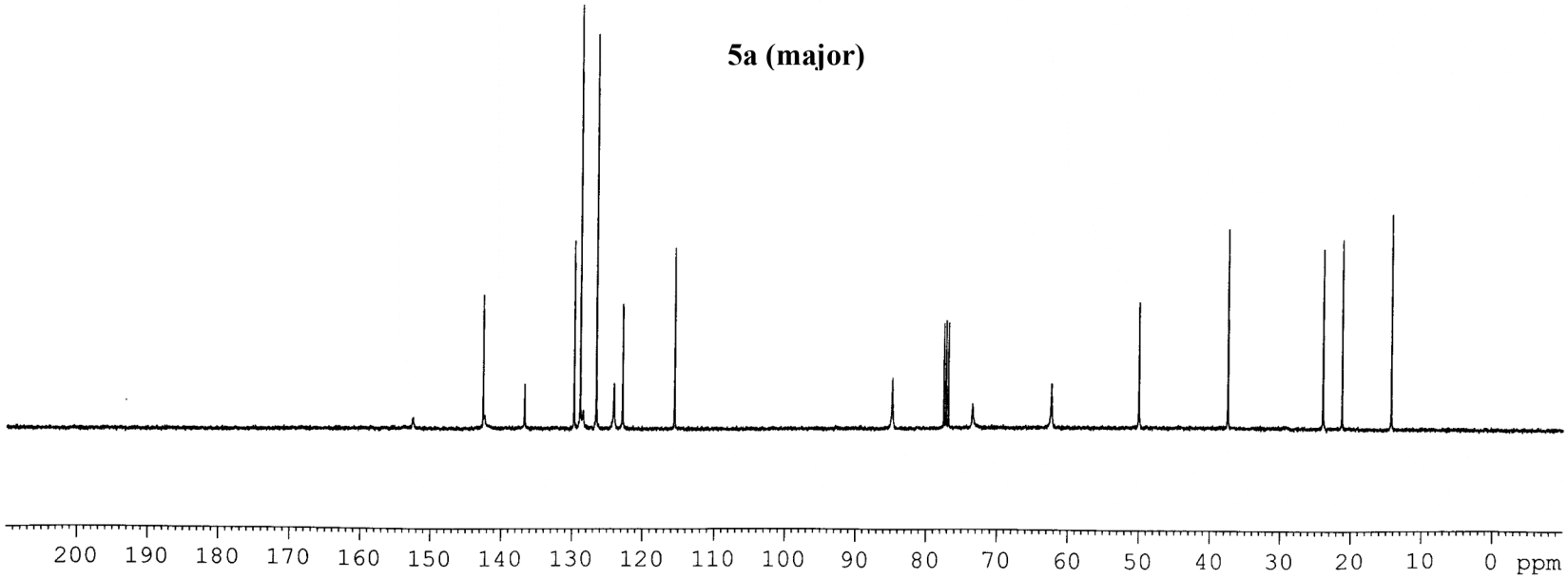
5a (major)

152.53
142.61
142.39
136.79
129.83
128.94
128.51
126.62
124.12
122.90
115.63
84.84
77.55
77.23
76.91
73.51
62.39
50.03
37.49
23.96
21.29
14.35

Table 2, entry 1



5a (major)



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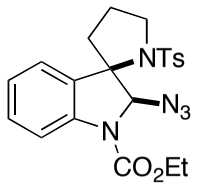
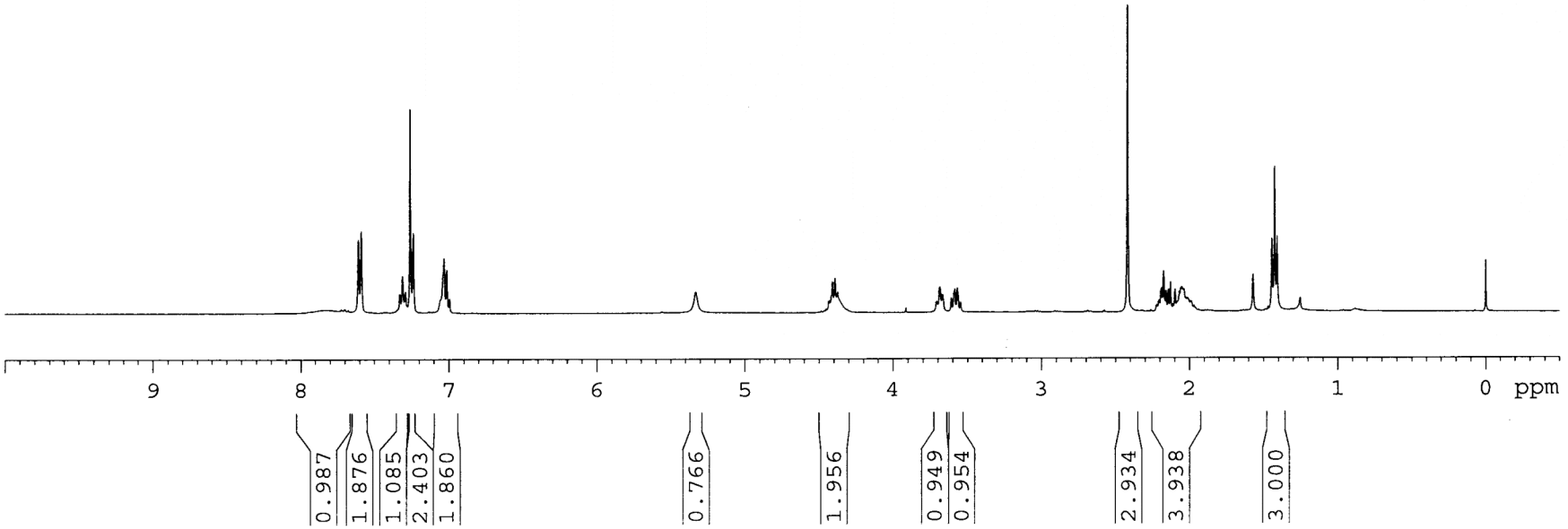


5a (minor)



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138.101
130.249
129.707
129.294
127.614
124.139
123.250
115.098
83.261
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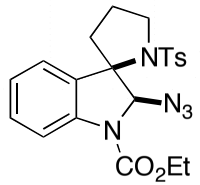
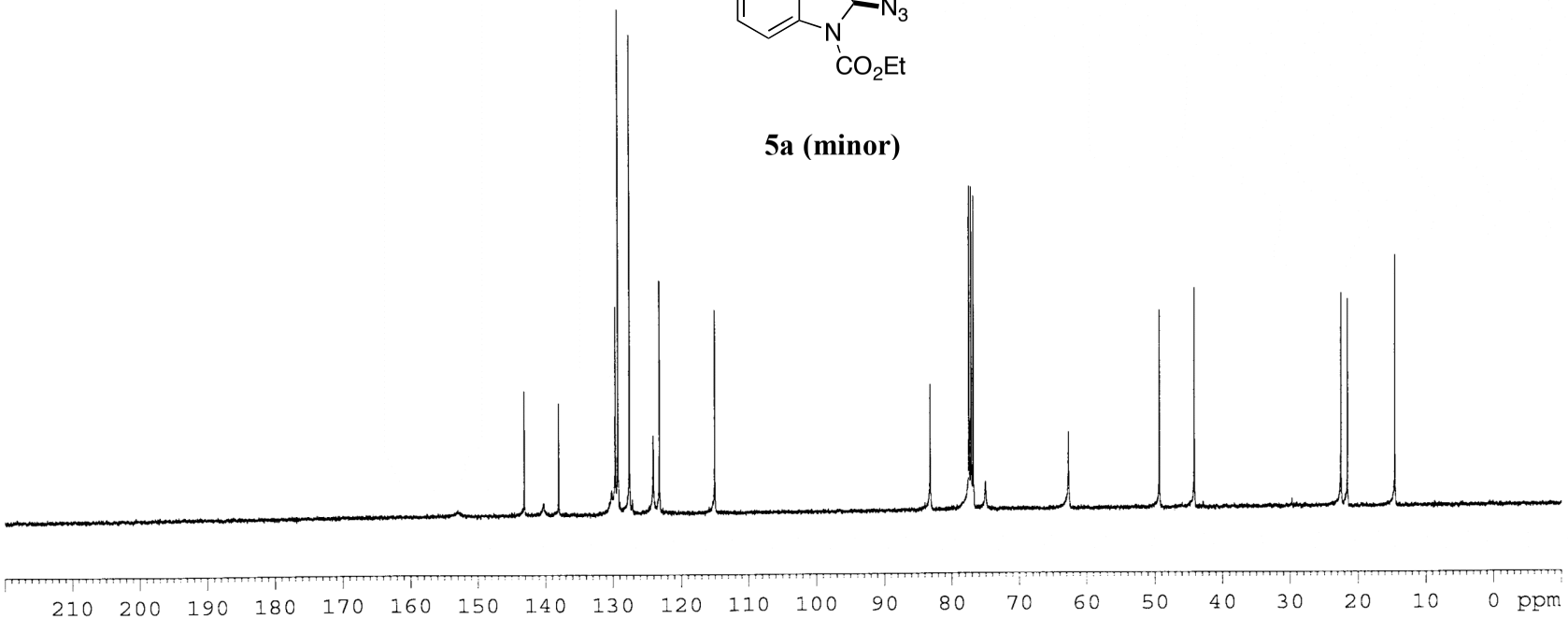
77.226
76.909
75.097
62.785
49.380
44.242
22.586
21.607
14.615

Table 2, entry 1



5a (minor)



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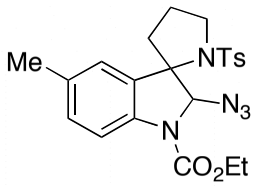
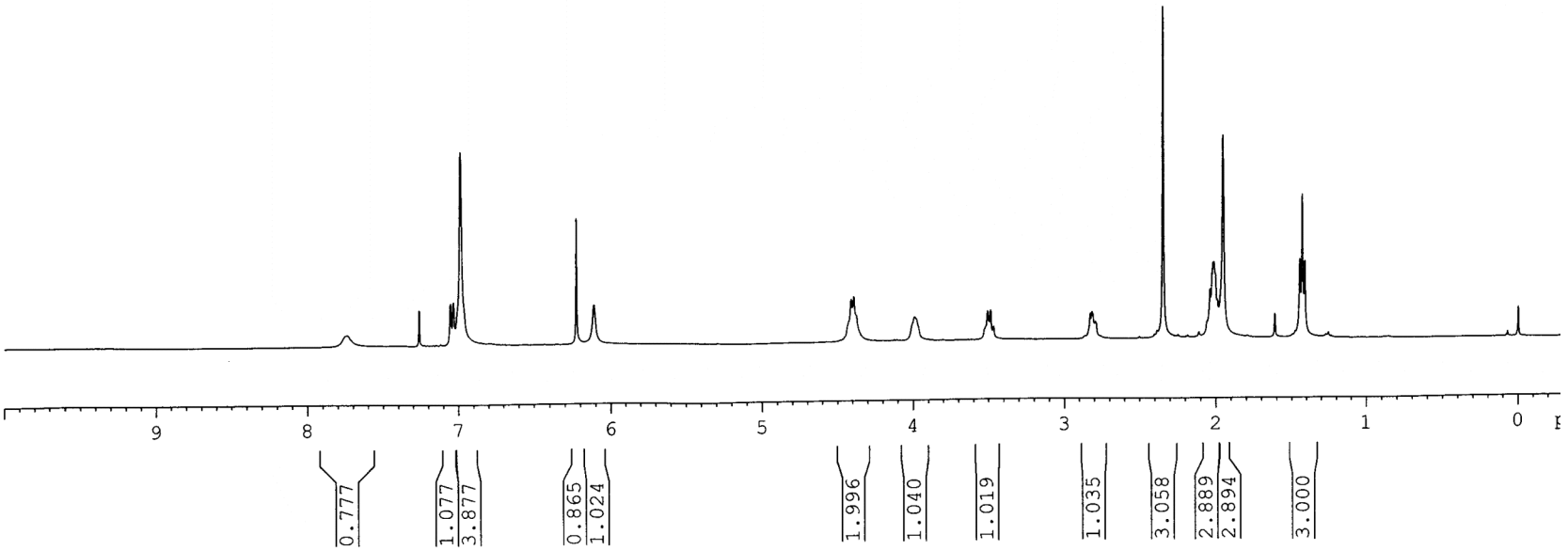
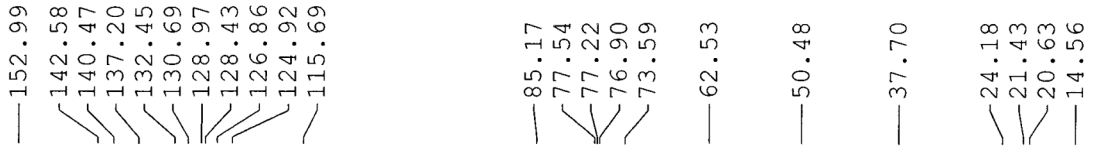


5b (major)





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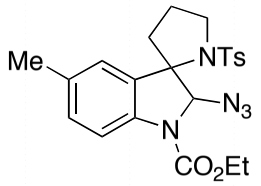
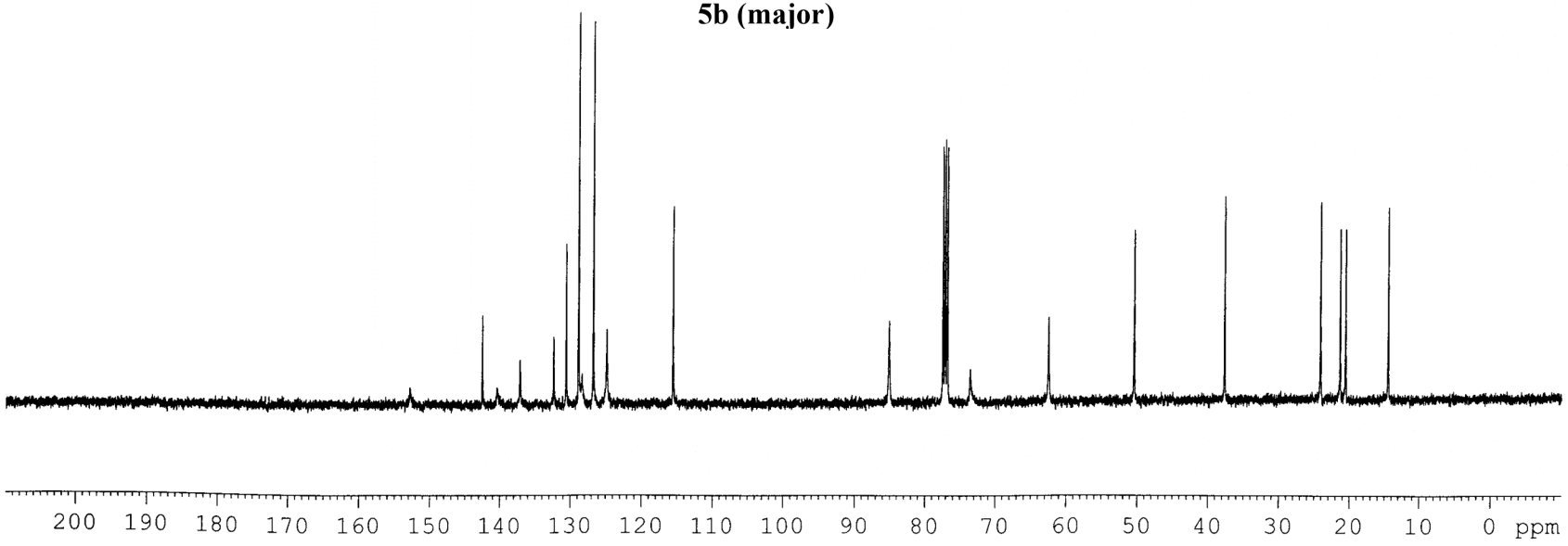


5b (major)



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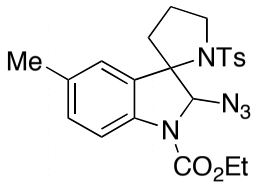
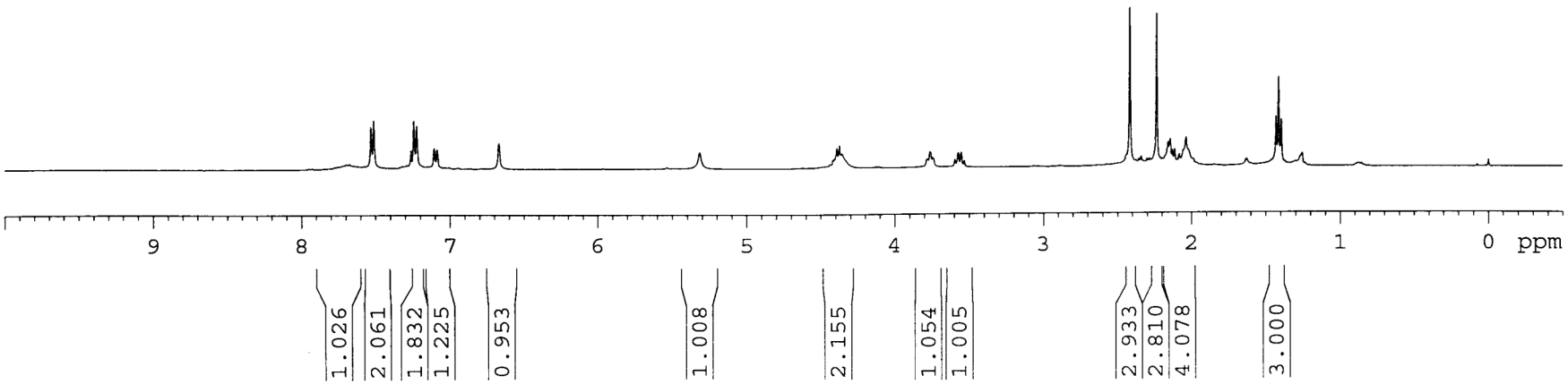


5b (minor)



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129.904
129.247
127.544
127.193
124.777
114.942
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77.547
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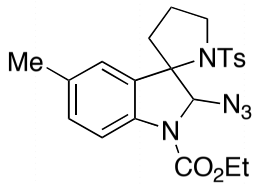
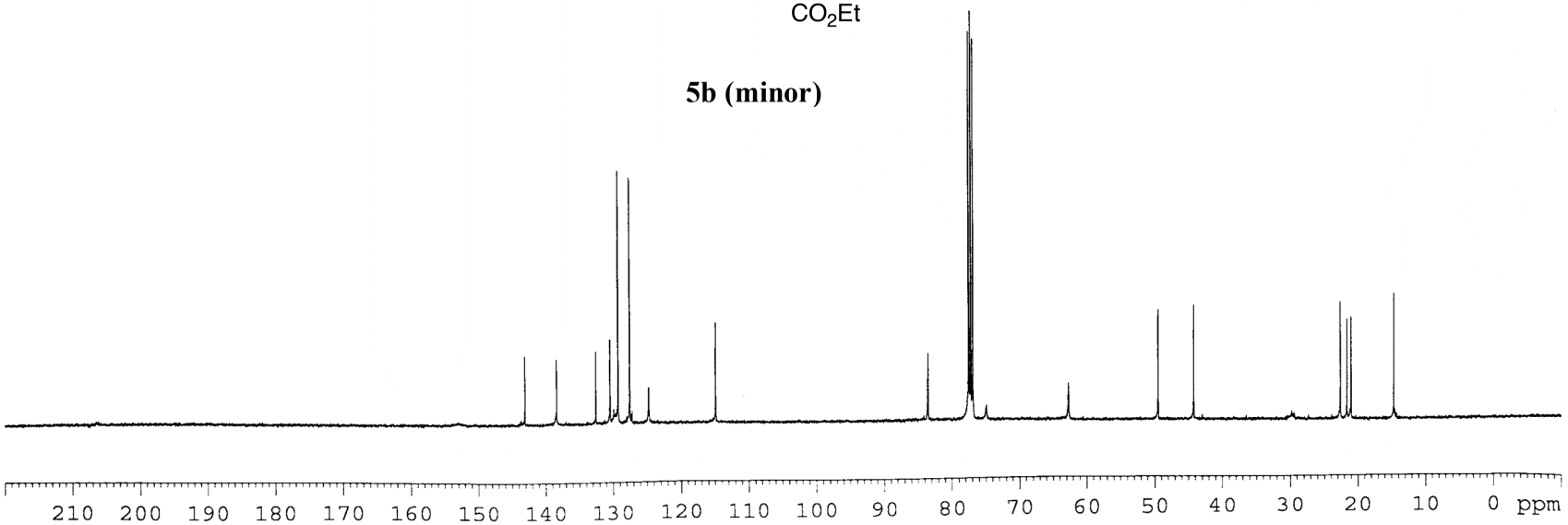
21.648
21.050
14.685

Table 2, entry 2



5b (minor)



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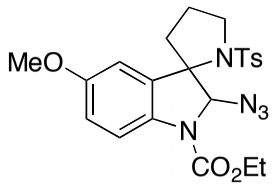
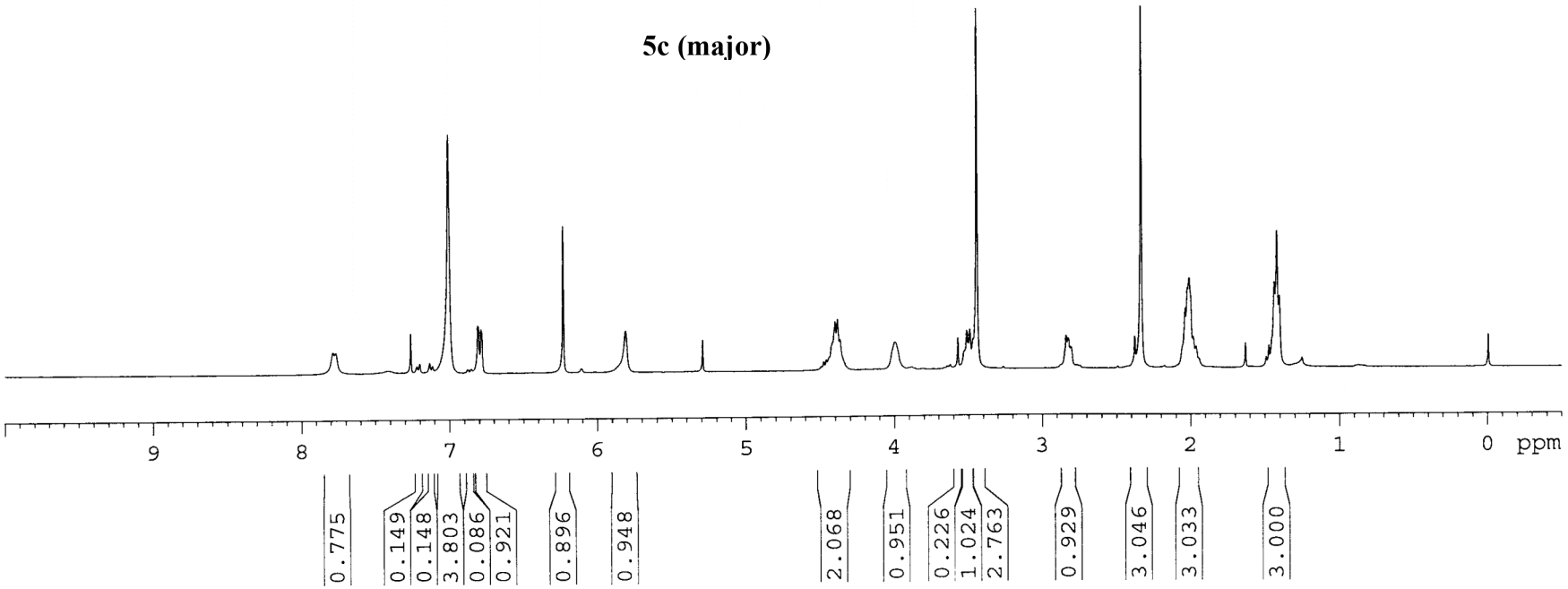


5c (major)



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136.350
129.383
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116.788
115.969
109.091
85.153
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77.229
76.910
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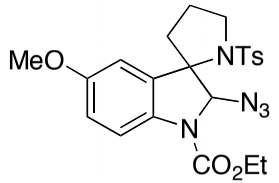
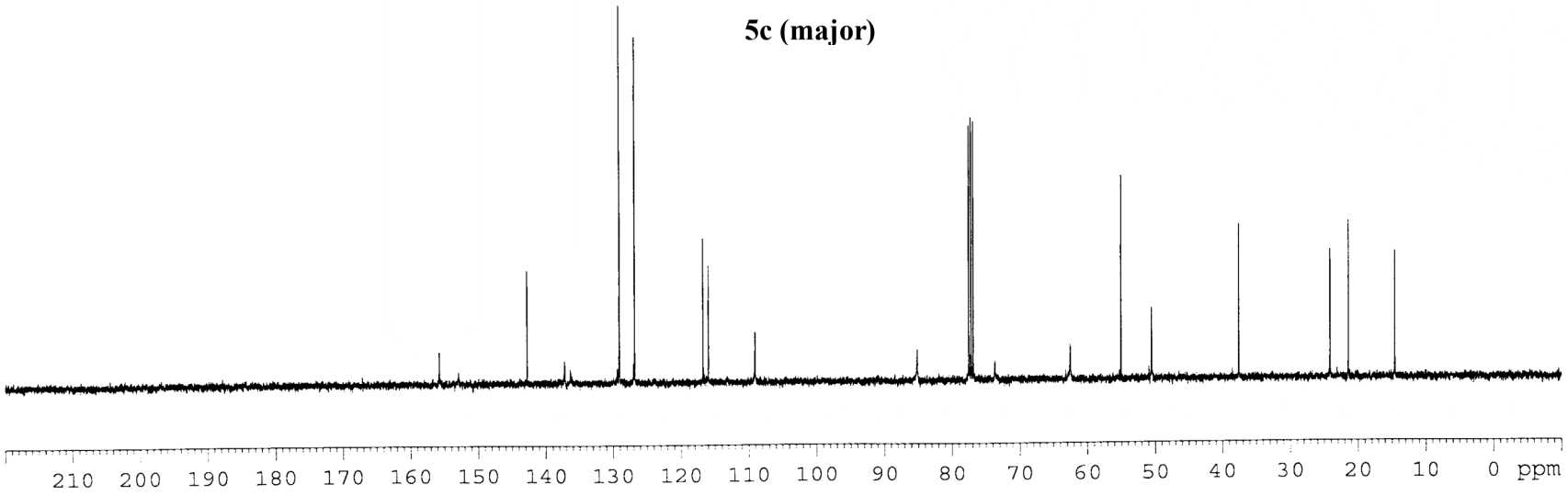
24.172
21.468
14.599

Table 2, entry 3



5c (major)



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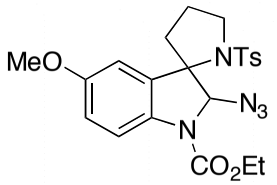
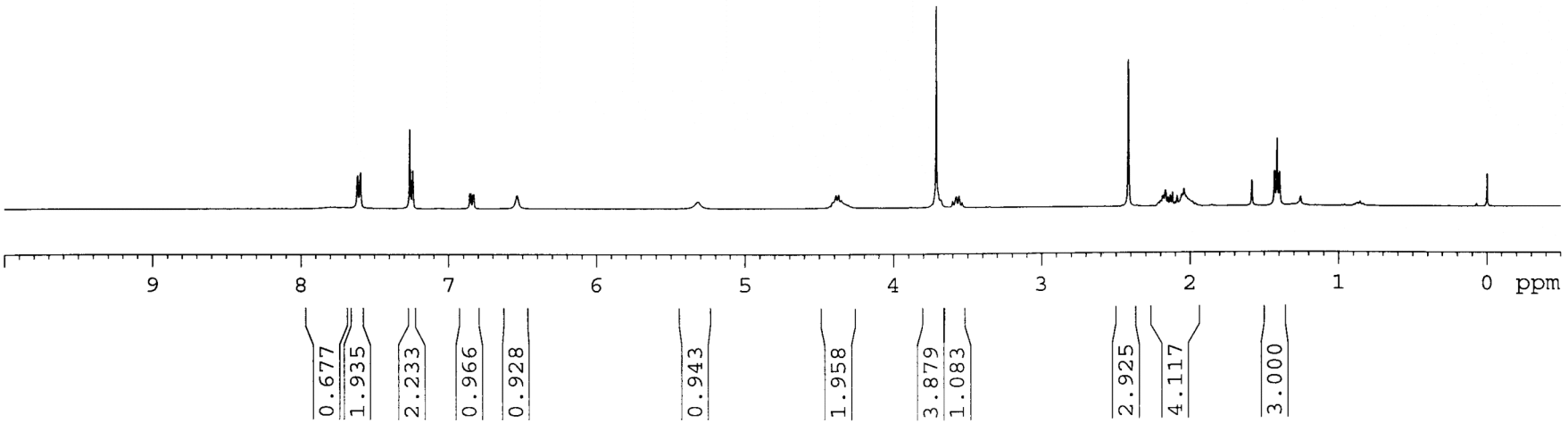
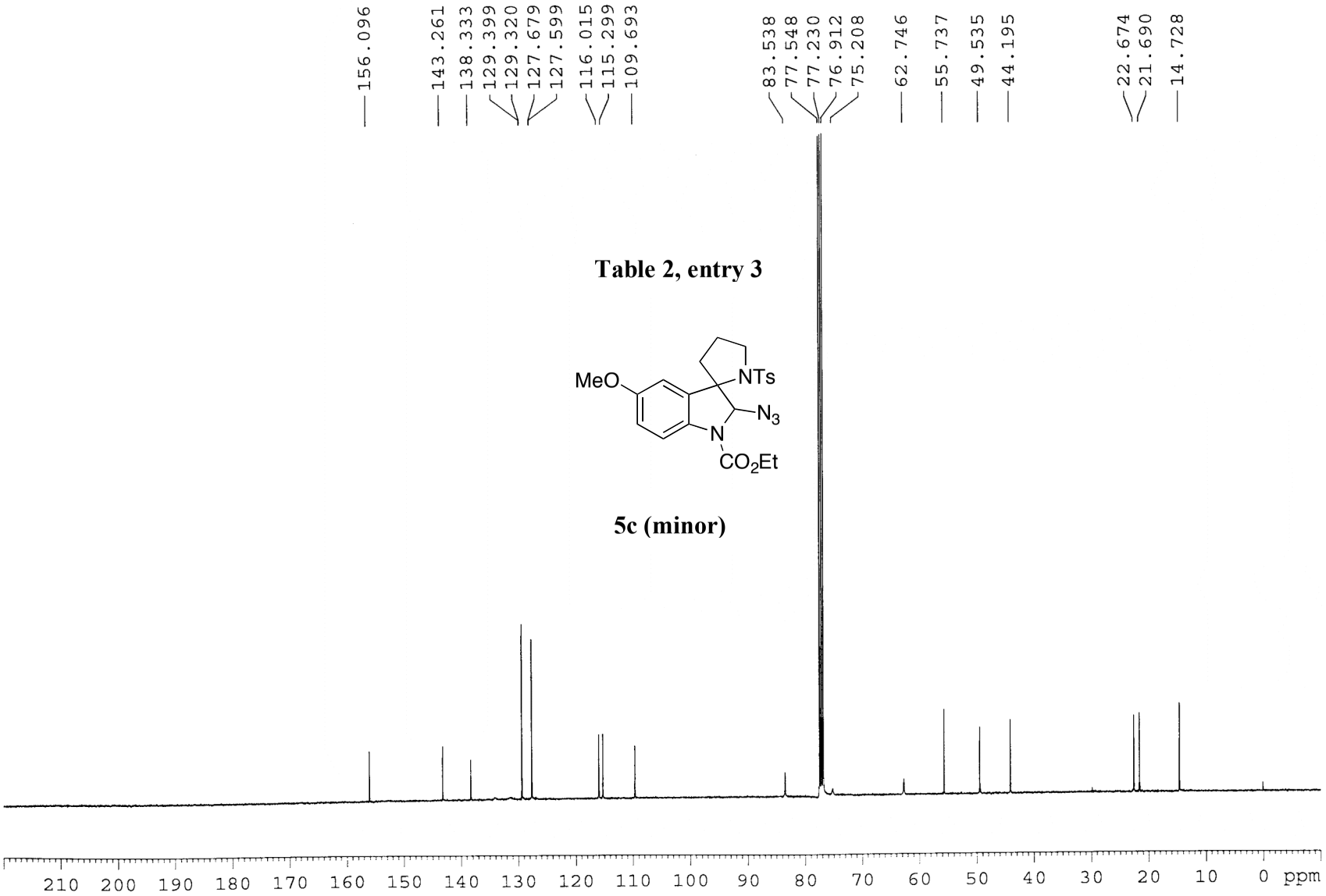


5c (minor)

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129.399
129.320
127.679
127.599
116.015
115.299
109.693
83.538
77.548
77.230
76.912
75.208
62.746
55.737
49.535
44.195
22.674
21.690
14.728
Table 2, entry 3
MeO
NTs
N3
N
CO2Et
5c (minor)
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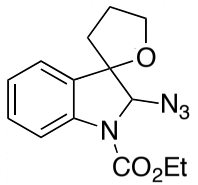
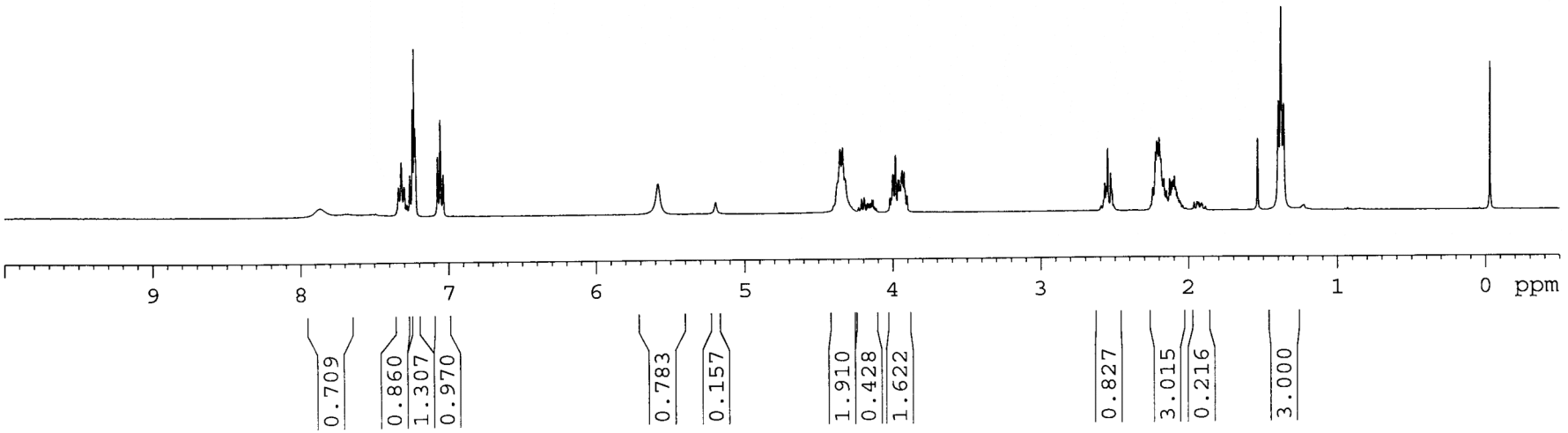
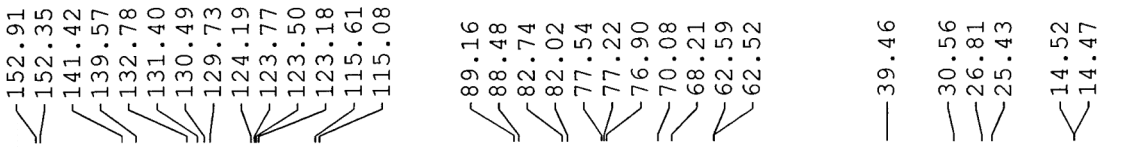
Table 2, entry 4



5d major and minor isomers





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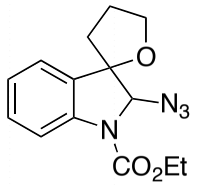
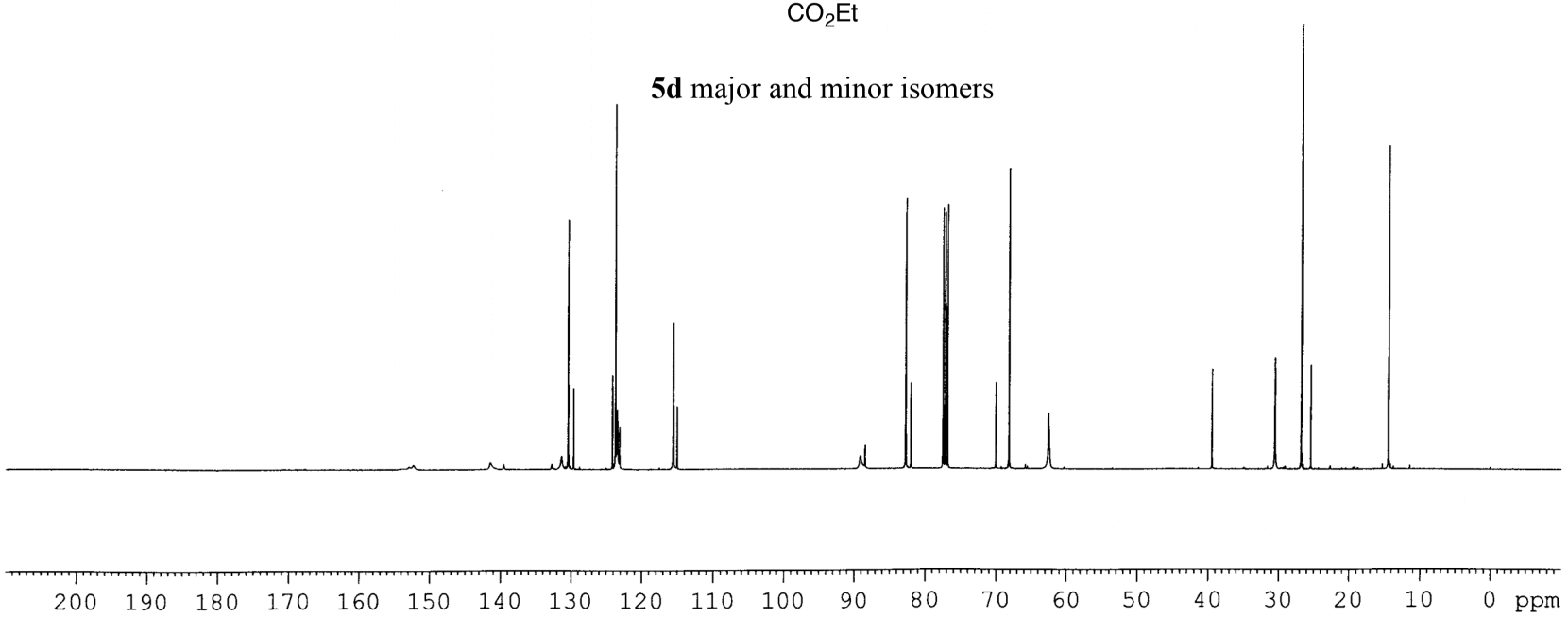


5d major and minor isomers



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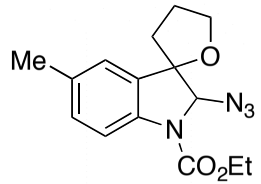
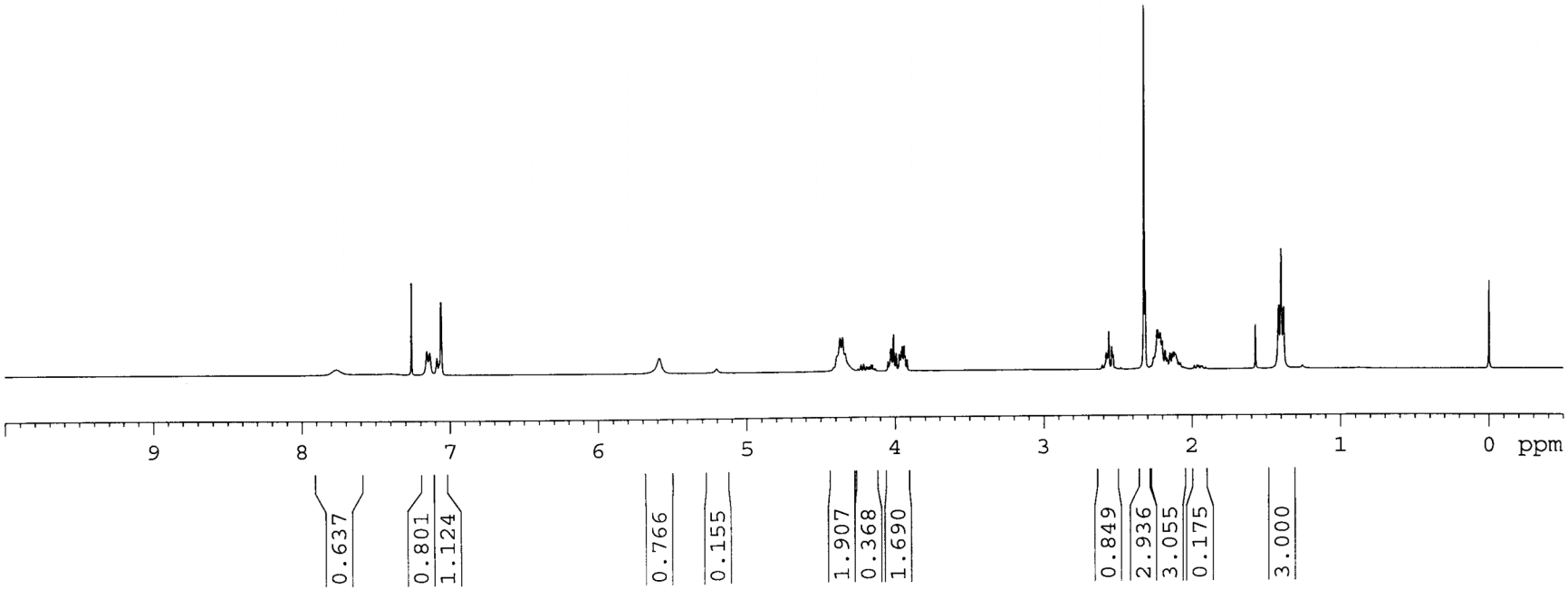
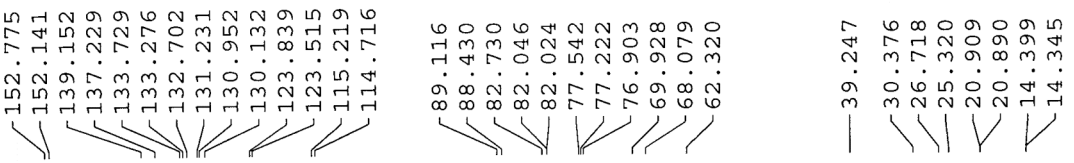


5e major and minor isomers





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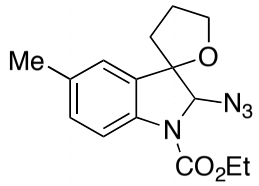
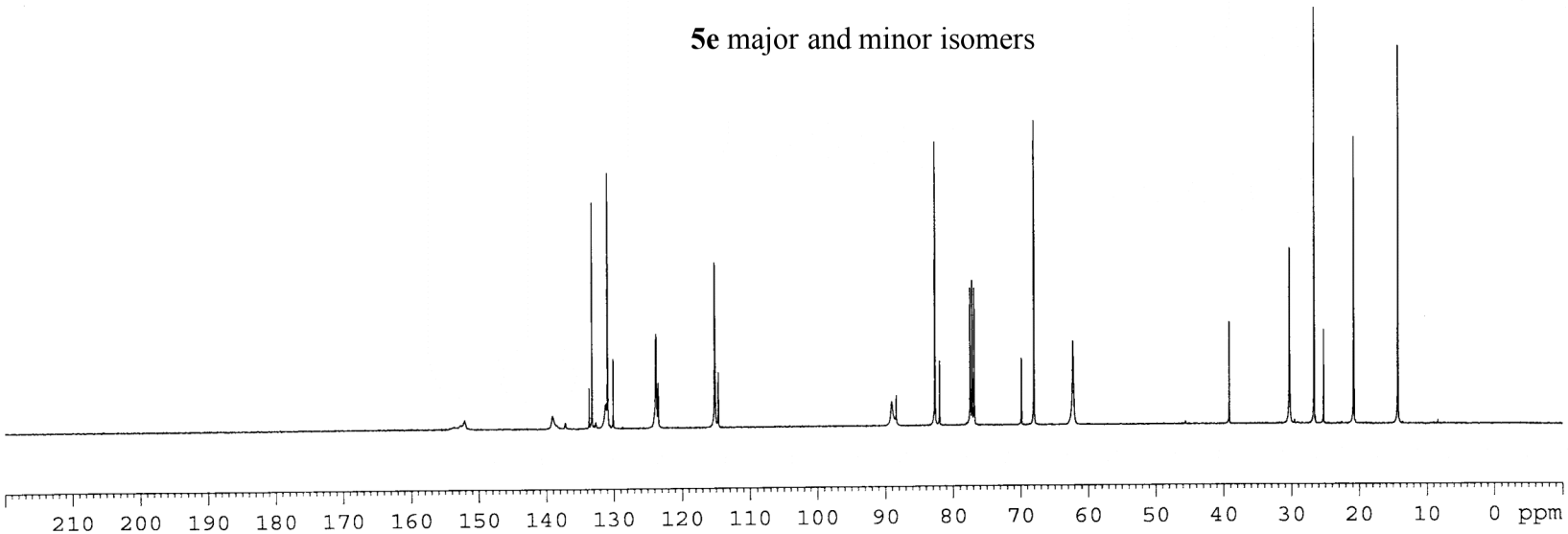


5e major and minor isomers



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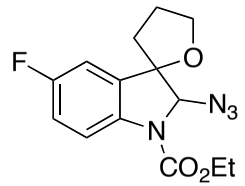
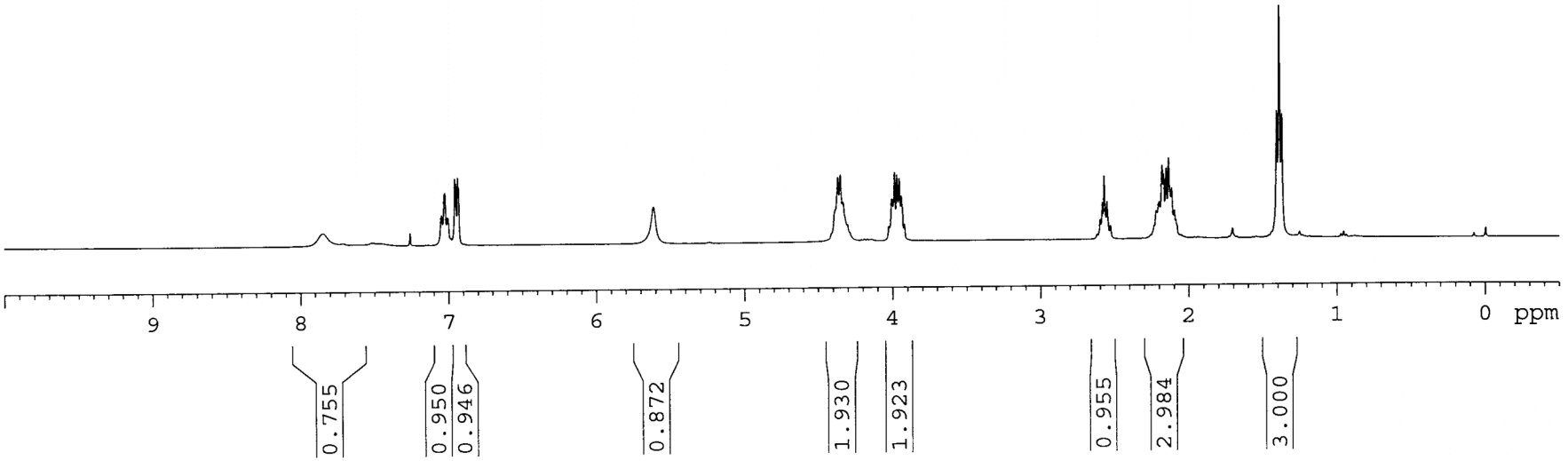
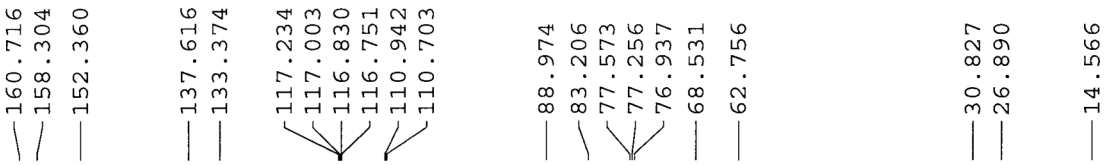


5f (major)





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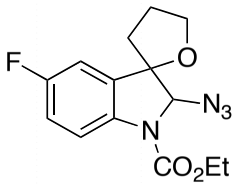
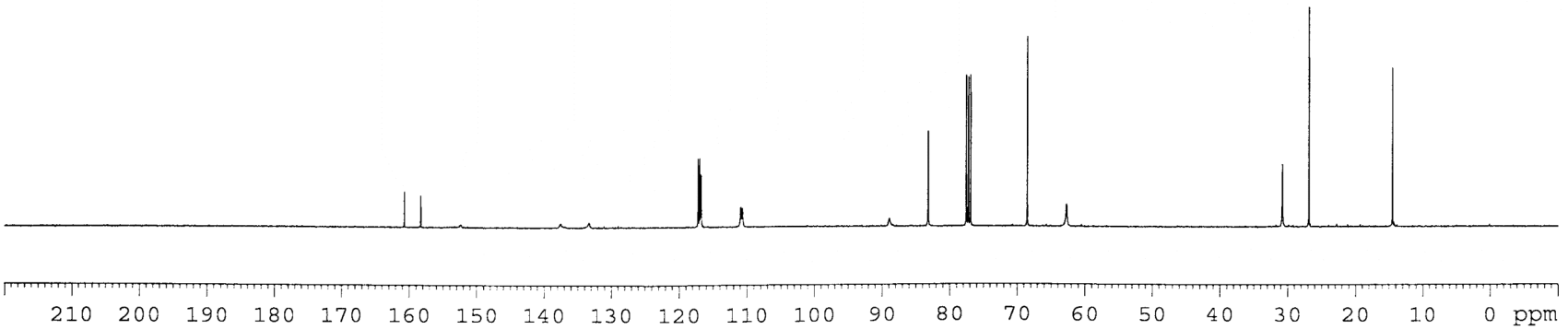


5f (major)



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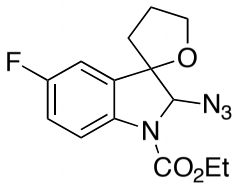
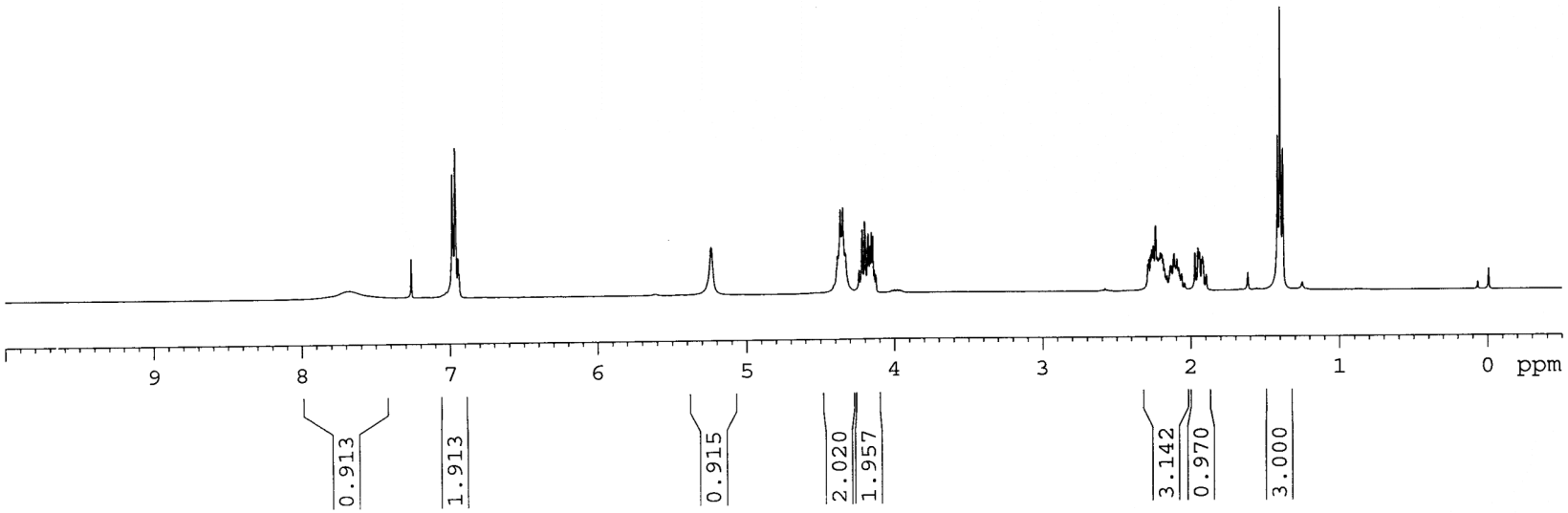
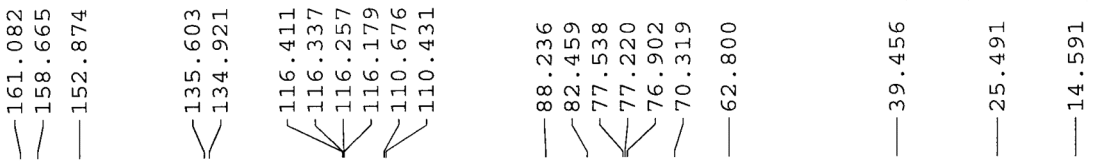


5f (minor)





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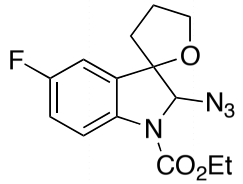
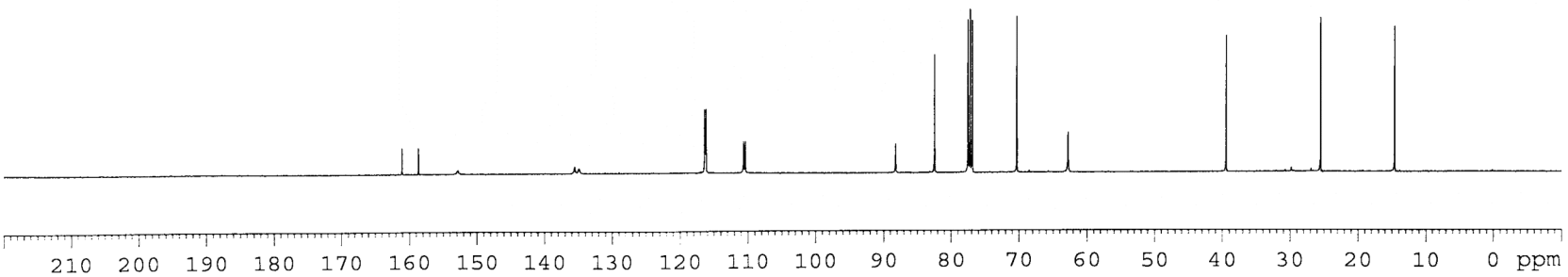


5f (minor)



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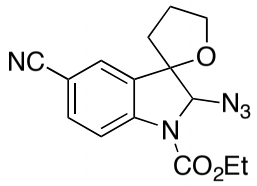
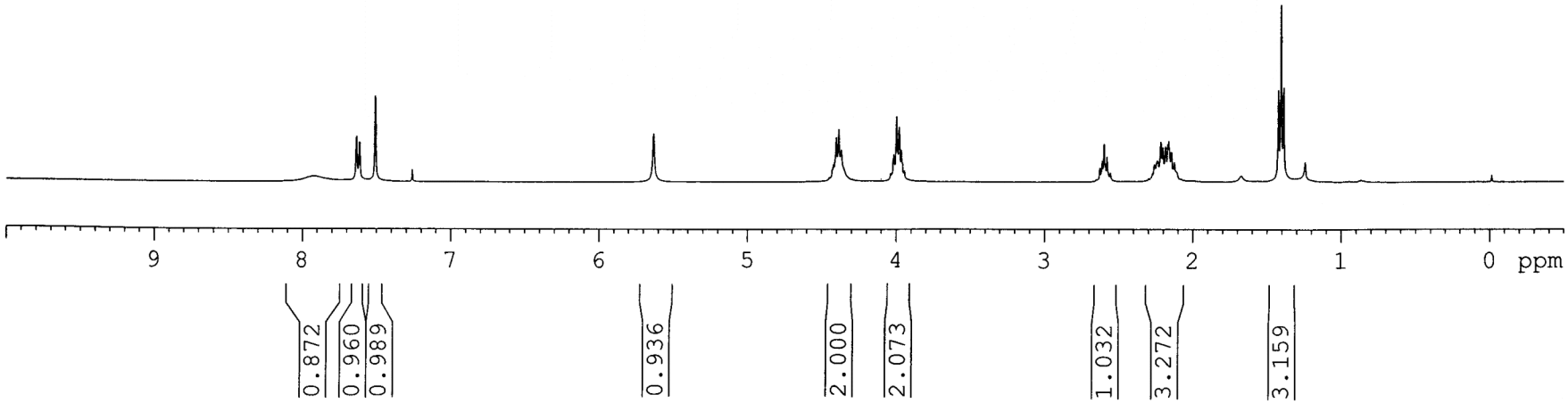


5g (major)



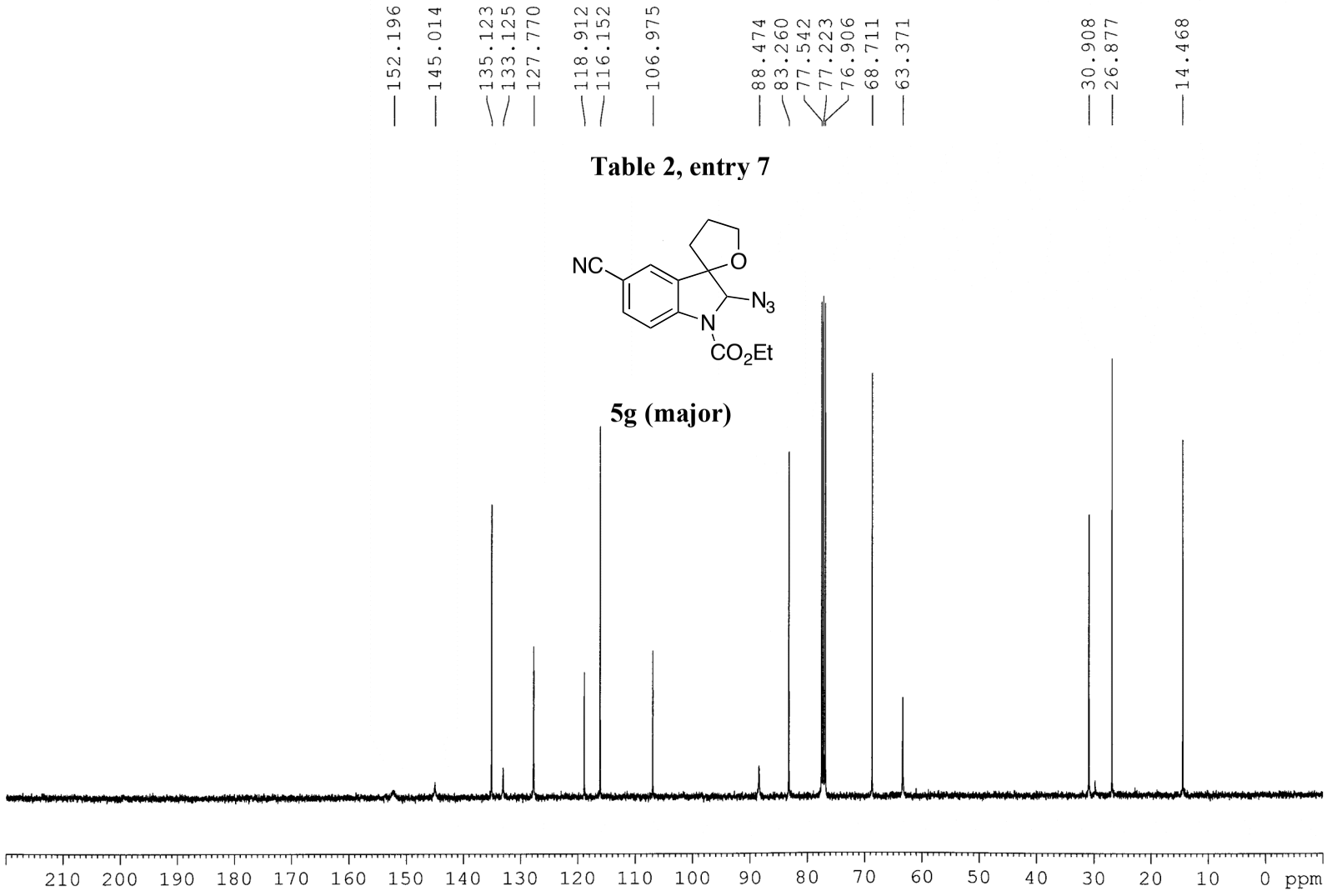
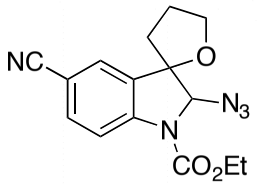


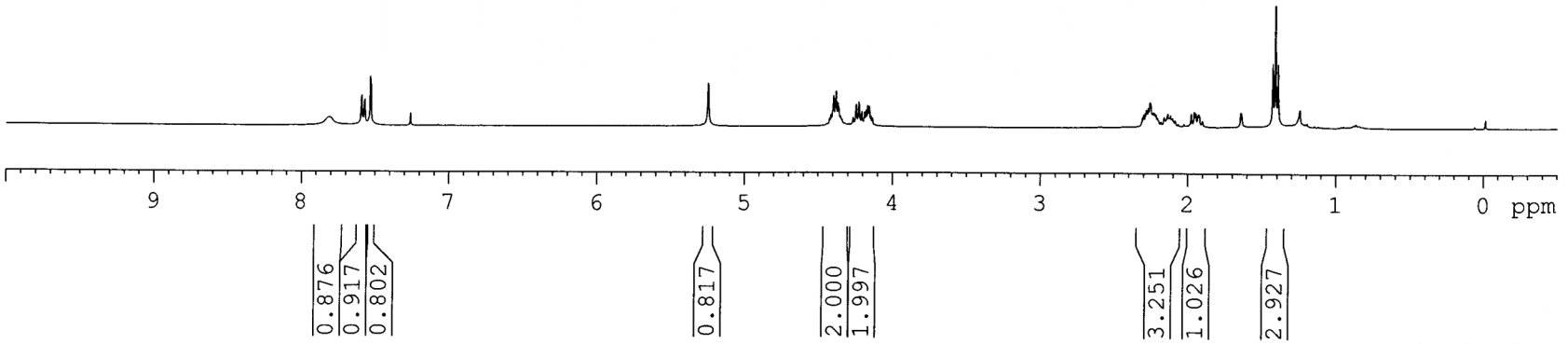
Table 2, entry 7

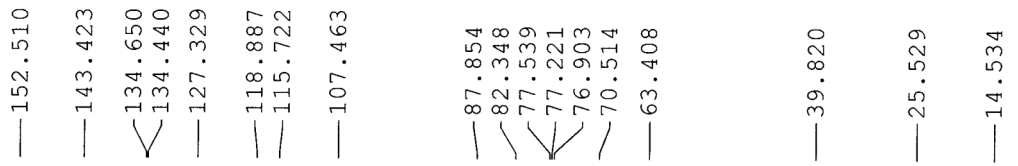
5g (major)

Table 2, entry 7



5g (minor)





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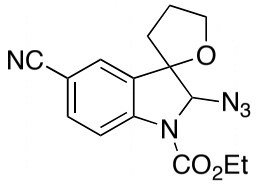
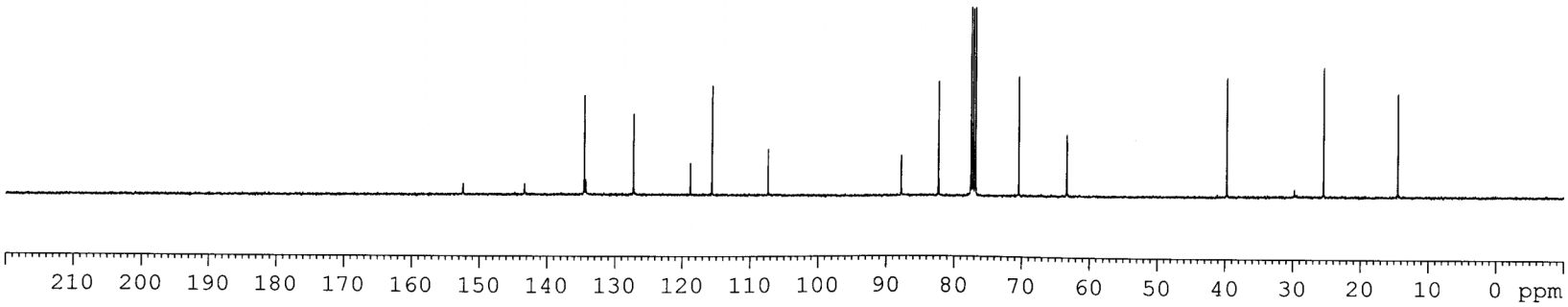


5g (minor)



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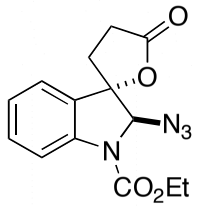
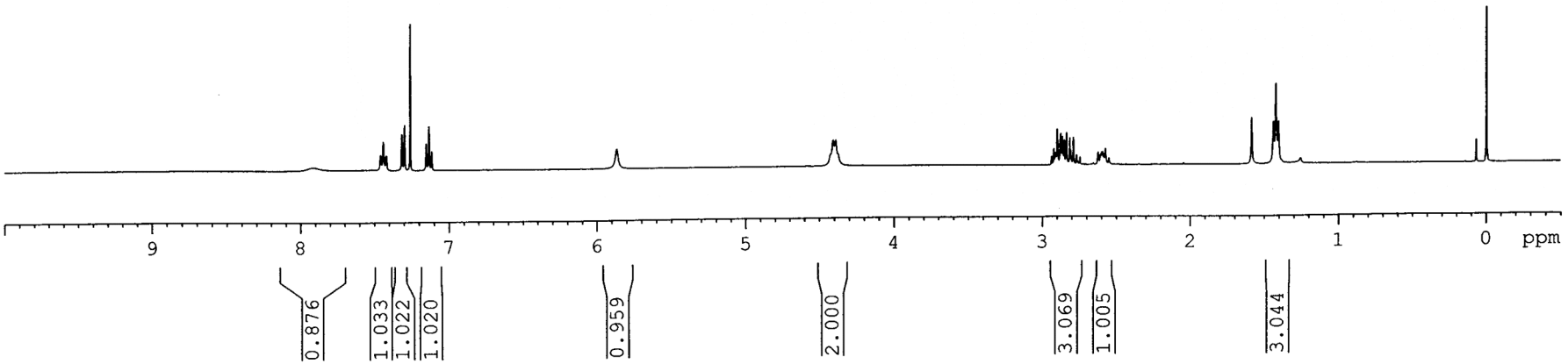


5h (major)



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132.030
128.157
124.188
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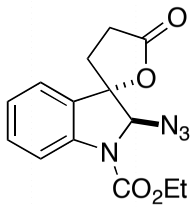
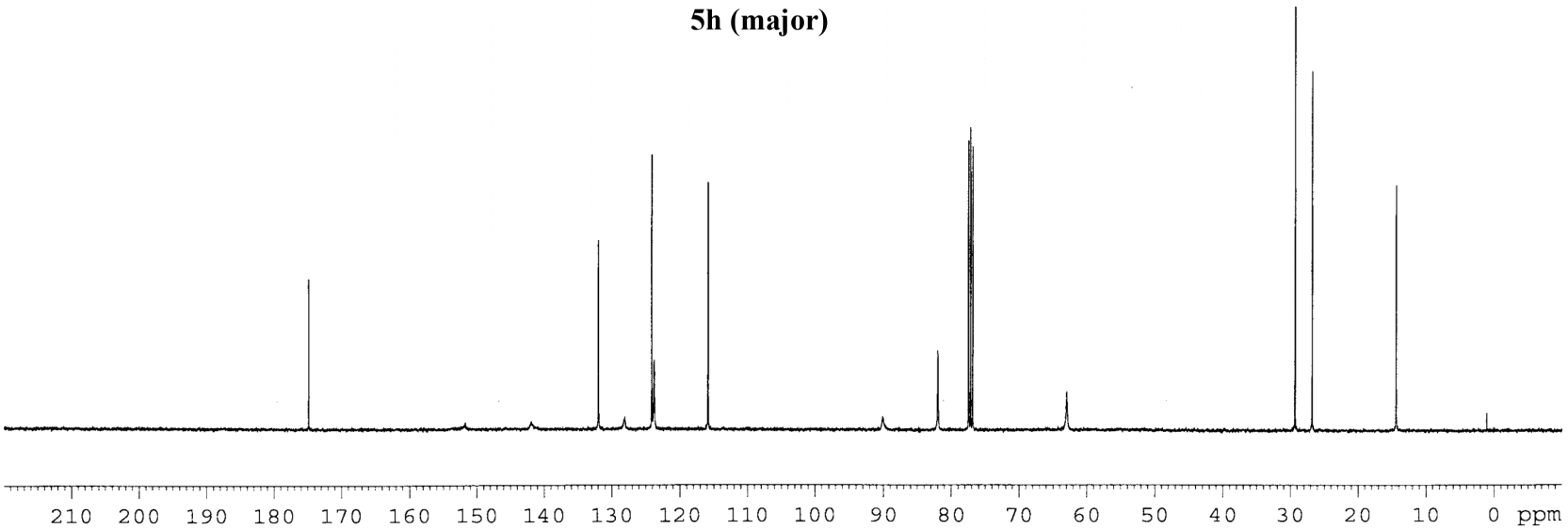
26.856
14.445

Table 2, entry 8



5h (major)



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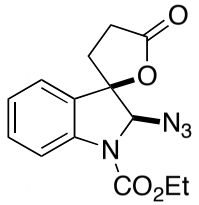
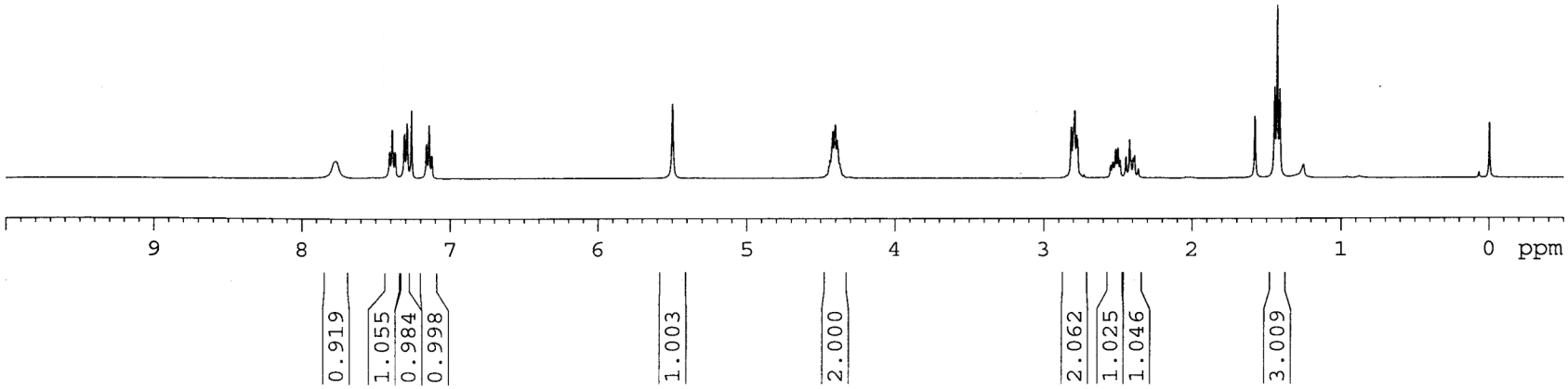
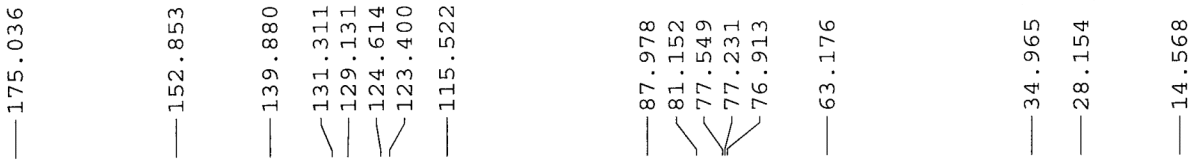


5h (minor)





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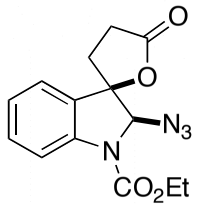
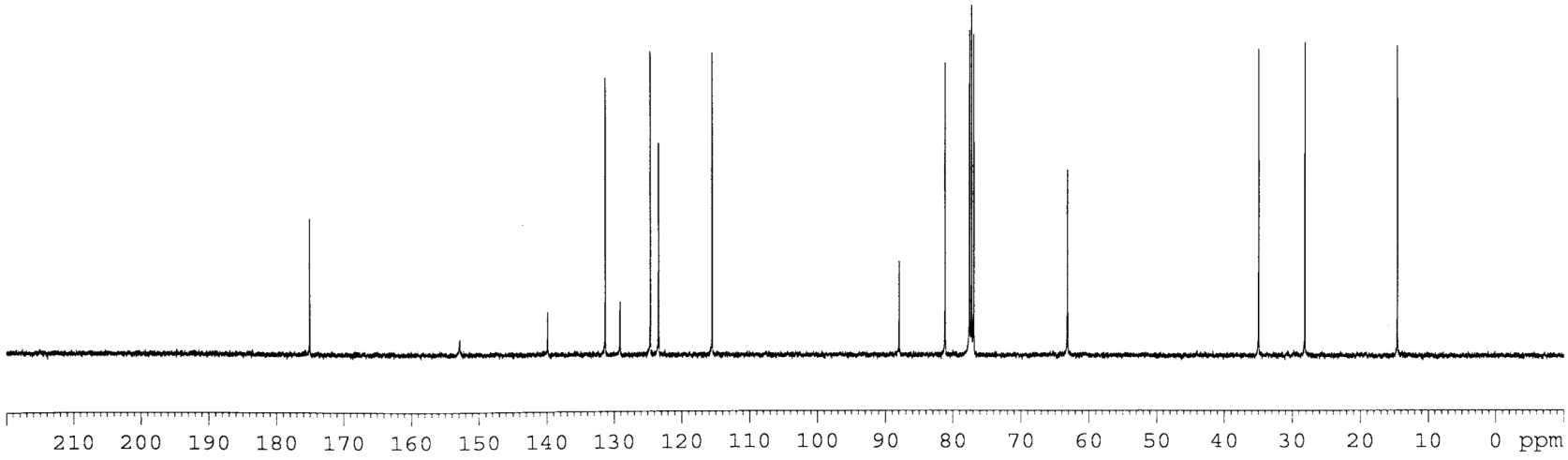


5h (minor)



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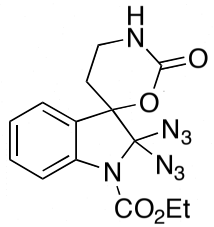
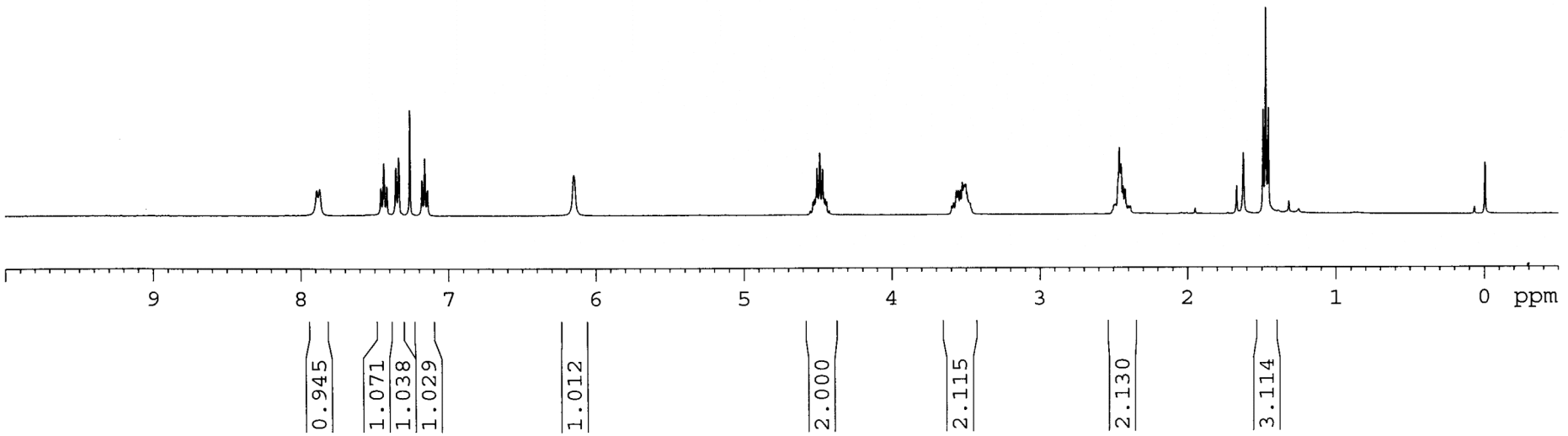


5i



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88.060
77.543
77.226
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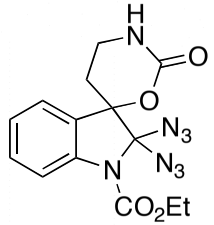
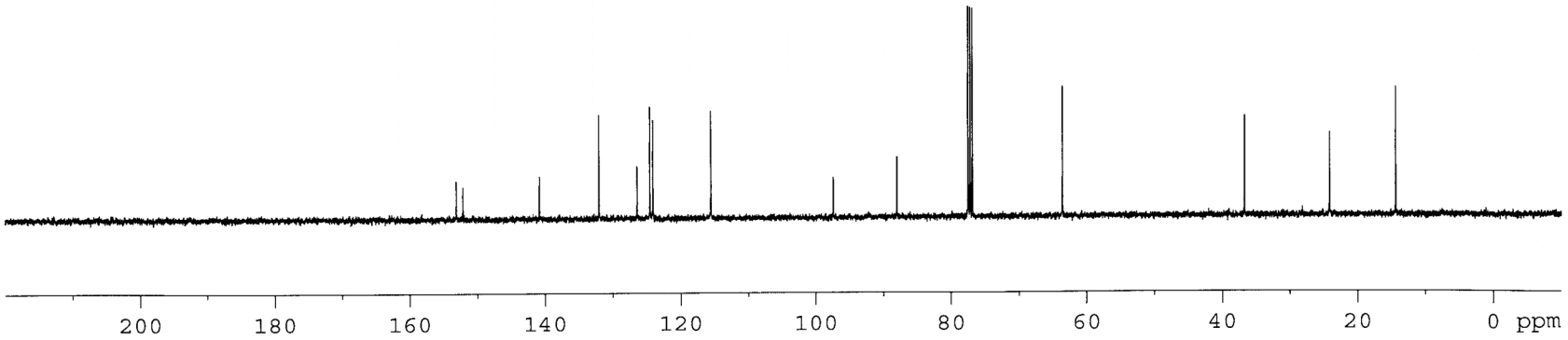
63.616
36.756
24.198
14.441

Table 2, entry 9



5i



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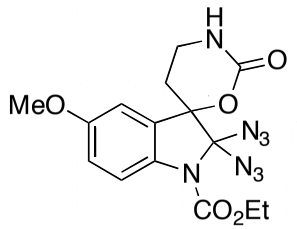
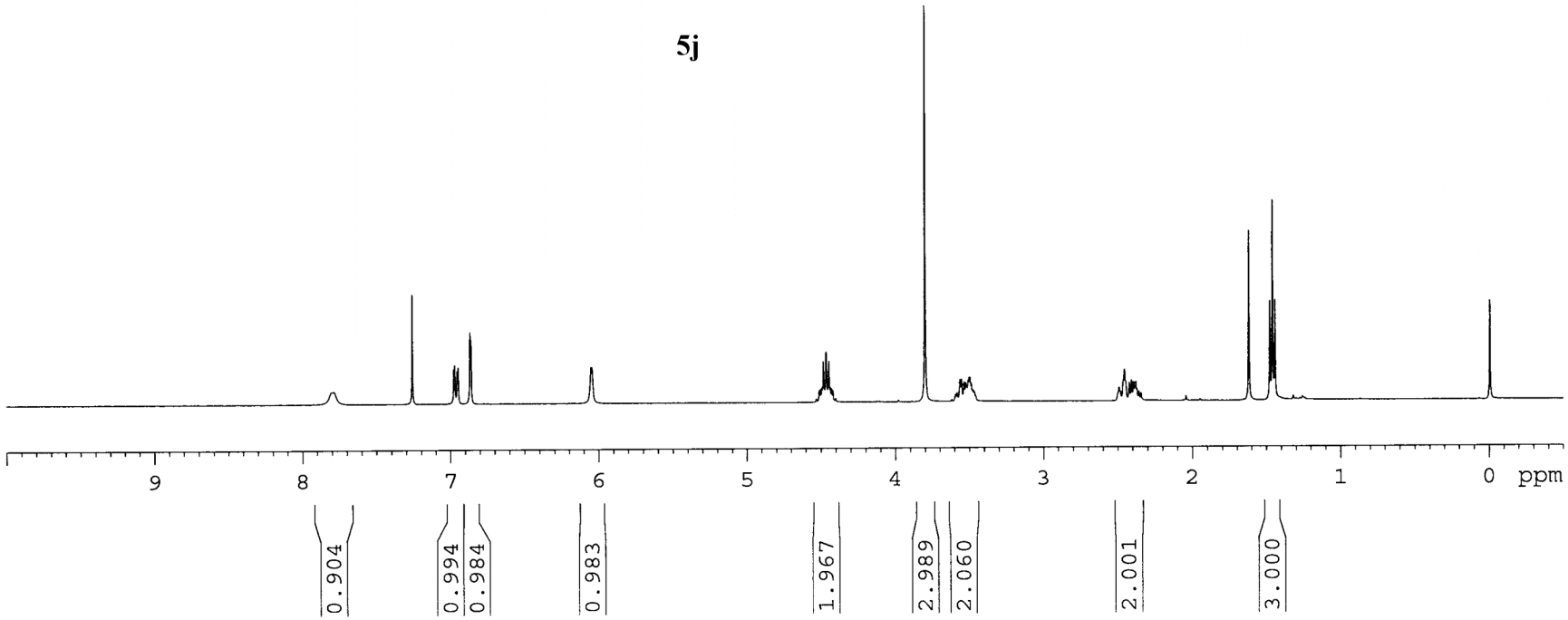
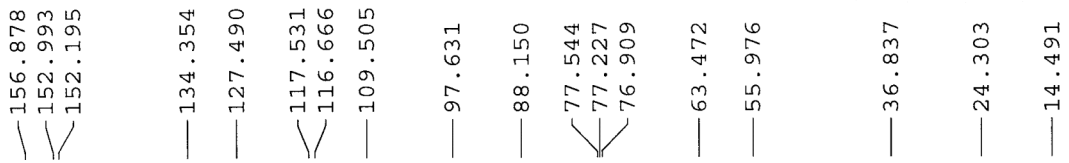


5j





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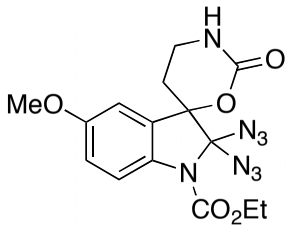

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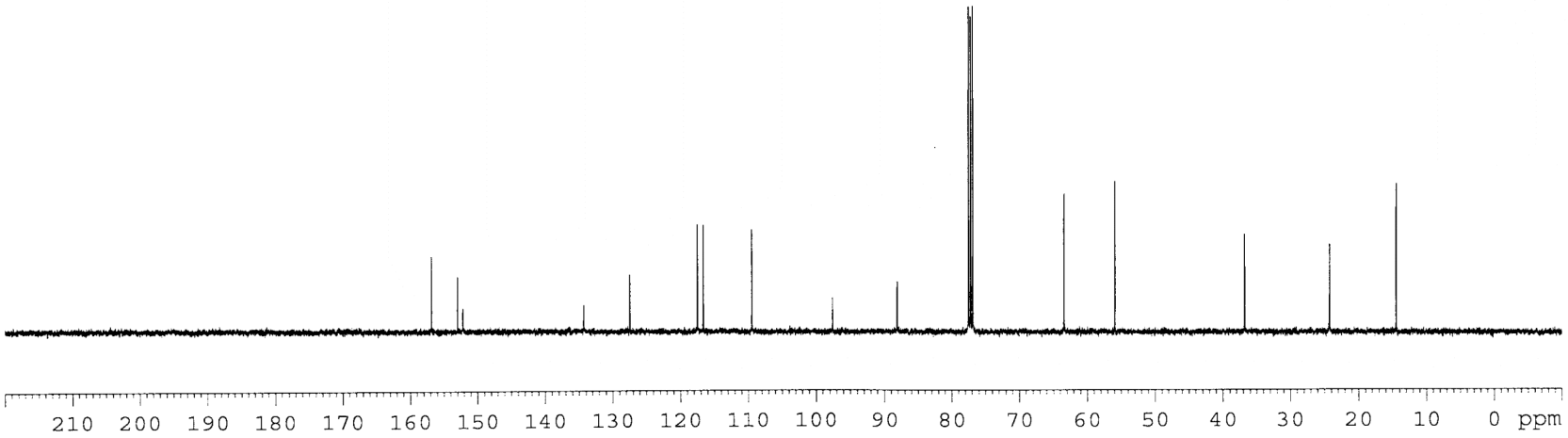

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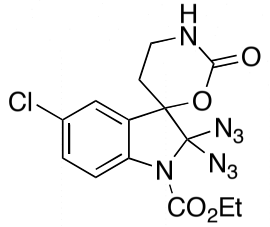
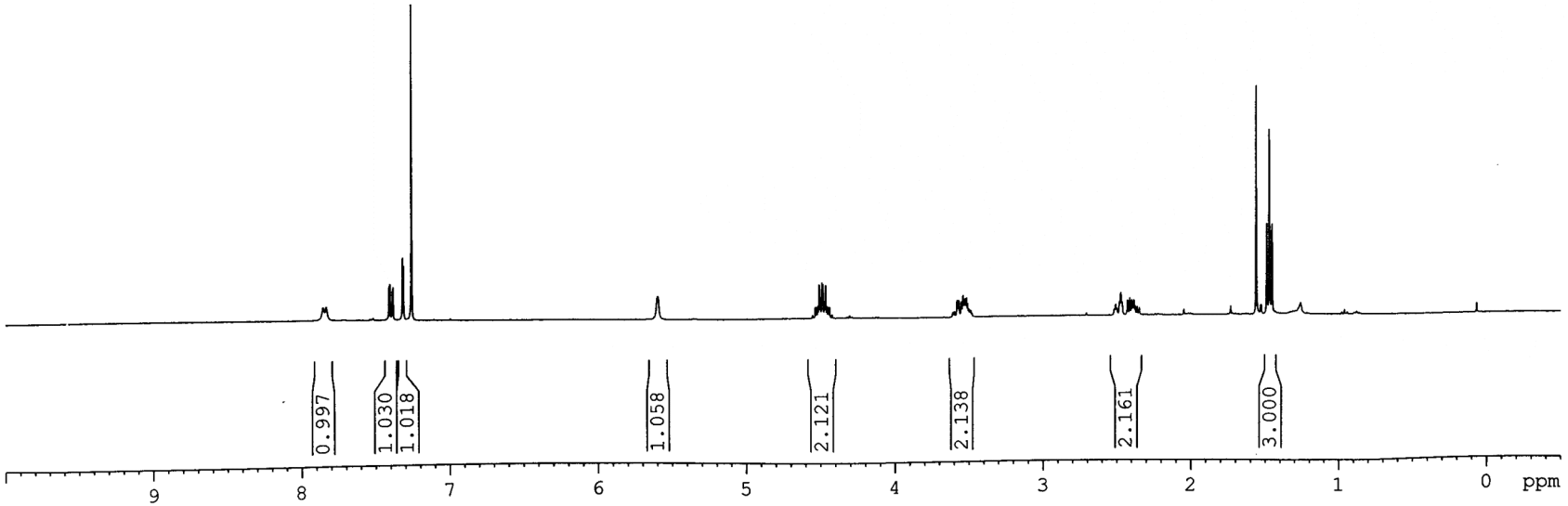
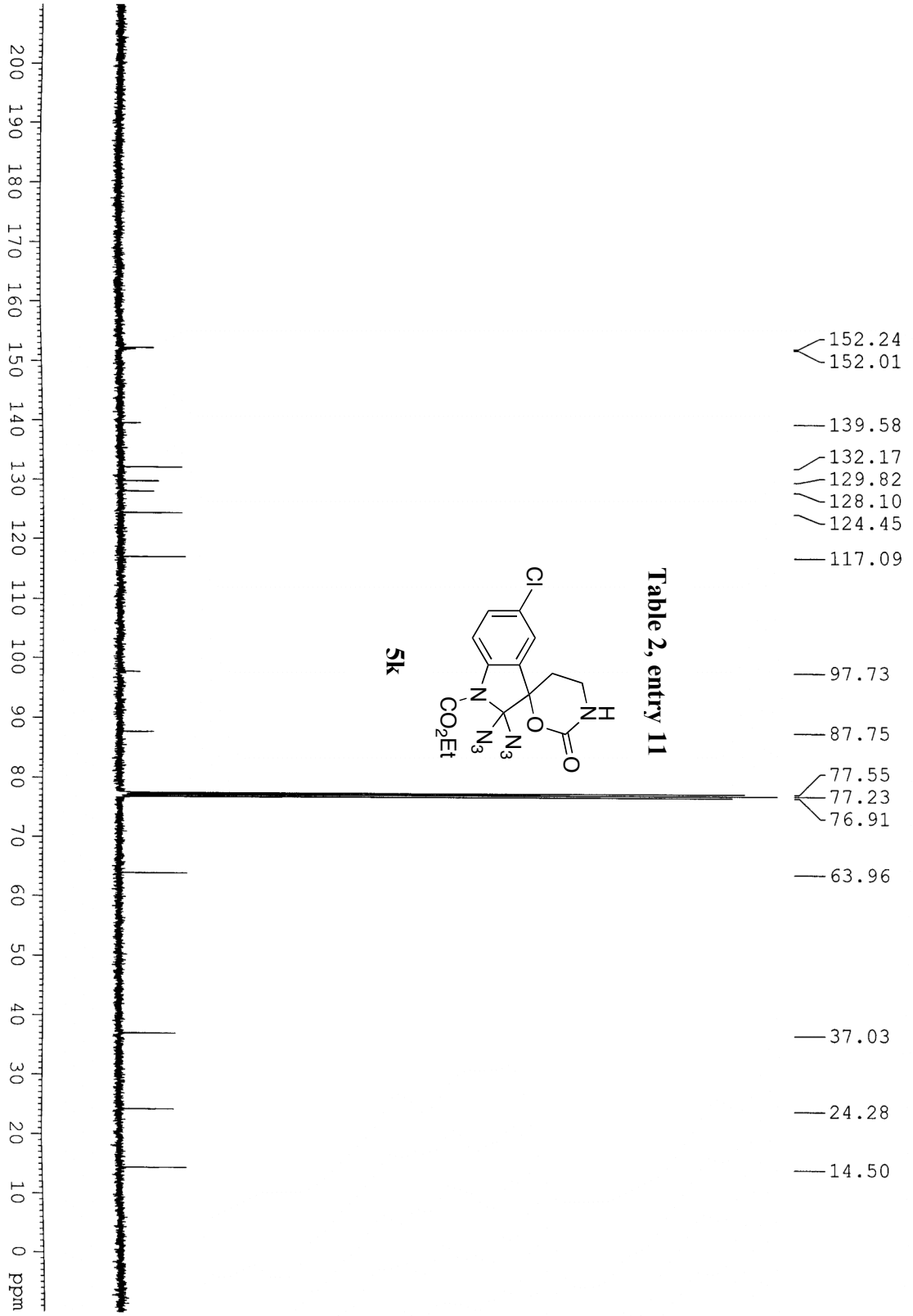


5k

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128.10
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117.09
97.73
87.75
77.55
77.23
76.91
63.96
37.03
24.28
14.50
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Table 2, entry 11
5k
Cl
N
H
O
O
N
N3
N3
CO2Et