Magnesium Halide-Catalyzed Anti-Aldol Reactions of Chiral *N*-Acylthiazolidinethiones

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Supporting Information

General Information. Acylation reactions were carried out under an atmosphere of nitrogen in flame-dried glassware with magnetic stirring. Aldol reactions were carried out under ambient atmosphere with a septum cap and magnetic stirring; no special precautions were taken to exclude water from the reaction vessel. THF and CH₂Cl₂ were purified by passage through a bed of activated alumina.¹ MeOH was distilled from Mg(OMe)₂. All other reaction solvents were used as received. Benzaldehyde and cinnamaldehyde were distilled from CaCl₂. All other aldehydes were purified by passage through a plug of flame-dried silica and alumina, except methacrolein, which was used as received from Aldrich Chemical Co. MgBr₂•OEt₂ was used as received from Aldrich Chemical Co. and stored in a glove box. Acid chlorides were used as received from Aldrich Chemical Co., except propionic acid chloride and butyric acid chloride, which were distilled from CaCl₂ prior to use. Triethylamine, Ethyldiisopropylamine, *N*-Methylmorpholine, and TMSCl were distilled from CaH₂. TESCl, imidazole, and TMSBr were used as received from Aldrich Chemical Co. Reagents were purified prior to use unless otherwise stated following the guidelines of Perrin and Armarego.² Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and ceric ammonium nitrate stain, followed by heating. Optical rotations were measured on a Jasco DIP-0181 digital polarimeter with a sodium lamp and are reported as follows: [α]^T_D (c = g/100 mL, solvent). Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. ¹H-NMR spectra were recorded on a Varian Inova-500 (500 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl₃ at 7.26 ppm). Data are reported as (ap = apparent, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad; coupling constant(s) in Hz; integration. Proton-decoupled ¹³C-NMR spectra were recorded on a Varian Mercury 400 (100 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl₃ at 77.0 ppm). Low- and high-resolution mass spectra were obtained on Jeol AX-505 or SX-102 spectrometers in the Harvard University Mass Spectrometry Laboratory.

General Procedure A: magnesium-catalyzed direct aldol reaction of *N*-acylthiazolidinethiones with aldehydes To a 50-mL round-bottom flask in a glove box was added MgBr₂•OEt₂ (0.50 mmol, 0.1 equiv.). The flask was fitted with a septum cap and removed to ambient atmosphere, where it was charged sequentially with acylthiazolidinethione (5.0 mmol, 1 equiv.), EtOAc (12.5 mL), aldehyde (5.5 mmol, 1.1 equiv.), Et₃N (10.0 mmol, 2.0 equiv.), and freshly distilled TMSCl (7.5 mmol, 1.5 equiv.). The cloudy, yellow reaction mixture was stirred 18-27 h until complete by TLC, then filtered directly through a plug of silica gel (5.0 cm x 5.0 cm) and eluted with Et₂O. The eluent was concentrated *in vacuo* and the yellow residue analyzed by ¹H NMR to determine diastereoselectivity. The unpurified mixture was dissolved in THF (100 mL) and treated with 1.0 N HCl (20 mL). After stirring 1 h, the mixture was diluted with Et₂O (200 mL) and water (200 mL). The layers were separated and the organic layer was extracted sequentially with saturated NaHCO₃ (200 mL) and brine (200 mL). The organic layer was dried over Na₂SO₄ for 1 h, then filtered through a cotton plug and concentrated *in vacuo*. Flash chromatography (10-20% EtOAc/hexanes) afforded the pure major diastereomer, except where otherwise noted.

(1) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.
(2) Perrin, D. D. and Armarego, W. L. *Purification of Laboratory Chemicals*; 3rd ed., Pergamon Press, Oxford. 1988.

(4E)-1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3S)-3-hydroxy-2-methyl-5-phenylpent-4-en-1-one (2d). The title compound was prepared according to General Procedure A and isolated as a yellow foam (87% yield): $[\alpha]_D^{25} = +282.9^\circ$ ($c = 1.40$, CH_2Cl_2); IR (film) 3429, 3060, 3027, 2979, 2933, 2876, 1952, 1884, 1812, 1692, 1603, 1494, 1454, 1360, 1319, 1293, 1264, 1191, 1168, 1138, 1055, 1027, 989, 902, 842, 739, 701, 660 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.47-7.22 (m, 10H, ArH), 6.67 (d, 1H, $J = 16$ Hz, $\text{PhCH}=\text{CHCH}(\text{OH})$), 6.23 (dd, 1H, $J = 6.3$, 16 Hz, $\text{PhCH}=\text{CHCH}(\text{OH})$), 5.22 (ap ddd, 1H, $J = 3.9$, 6.8, 10.7 Hz, NCHCH_2Ph), 4.59 (dq, 1H, $J = 6.8$, 6.8 Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$), 4.51 (dd, 1H, $J = 5.9$, 11.7 Hz, $\text{PhCH}=\text{CHCH}(\text{OH})$), 3.29-3.22 (m, 2H, NCHCH_2Ph and $\text{S}=\text{CSCH}_2$), 3.06 (dd, 1H, $J = 10.7$, 13.2 Hz, NCHCH_2Ph), 2.85 (ap d, 1H, $J = 11.2$ Hz, $\text{S}=\text{CSCH}_2$), 2.56 (d, 1H, $J = 5.4$ Hz, OH), 1.35 (d, 3H, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$); ^{13}C NMR (100 MHz, CDCl_3) 201.8, 178.0, 136.7, 136.4, 132.1, 129.9, 129.7, 129.1, 128.9, 128.3, 127.5, 126.7, 76.3, 69.1, 45.0, 36.9, 32.9, 14.9; HRMS (EI): Exact mass calcd for $\text{C}_{22}\text{H}_{23}\text{O}_2\text{NS}_2$ $[\text{M}]^+$, 397.1170. Found 397.1169.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-2-methyl-3-phenylpropan-1-one (2a). The title compound was prepared according to General Procedure A and isolated as a yellow foam (85% yield): $[\alpha]_D^{25} = +307.8^\circ$ ($c = 1.23$, CH_2Cl_2); IR (film) 3443, 2975, 1694, 1494, 1454, 1371, 1341, 1293, 1263, 1192, 1166, 1137, 1033, 742, 701, 610 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.40-7.20 (m, 10H, ArH), 5.22 (ap ddd, 1H, $J = 3.7$, 6.8, 12.5 Hz, NCHCH_2Ph), 4.88 (ap d, 1H, $J = 6.8$ Hz, $\text{PhCH}(\text{OH})$), 4.79 (dq, 1H, $J = 6.3$, 6.3 Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$), 3.20 (dd, 1H, $J = 3.9$, 13.2 Hz, NCHCH_2Ph), 3.03-2.91 (m, 2H, NCHCH_2Ph and $\text{S}=\text{CSCH}_2$), 2.88 (d, 1H, $J = 6.8$ Hz, OH), 2.74 (ap d, 1H, $J = 11.2$ Hz, $\text{S}=\text{CSCH}_2$), 1.26 (d, 3H, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$); ^{13}C NMR (100 MHz, CDCl_3) 201.9, 178.4, 142.4, 136.7, 129.7, 129.1, 128.8, 128.1, 127.5, 126.2, 69.0, 46.2, 36.8, 32.8, 15.0; HRMS (EI): Exact mass calcd for $\text{C}_{20}\text{H}_{21}\text{O}_2\text{NS}_2$ $[\text{M}]^+$, 371.1014. Found 371.1010.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-2-methyl-3-(4-methylphenyl)propan-1-one (2b). The title compound was prepared according to General Procedure A and isolated as yellow crystals (92% yield): mp: 119.5 to 121.3 $^\circ\text{C}$; $[\alpha]_D^{25} = +297.4^\circ$ ($c = 1.04$, CH_2Cl_2); IR (film) 3444, 3028, 2977, 1668, 1456, 1361, 1341, 1262, 1191, 1165, 1137, 1030, 816, 739, 702 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.40-7.13 (m, 9H, ArH), 4.98 (ap ddd, 1H, $J = 3.9$, 6.8, 10.8 Hz, NCHCH_2Ph), 4.84 (ap d, 1H, $J = 6.8$ Hz, $\text{CH}(\text{OH})$), 4.76 (dq, 1H, $J = 6.8$, 6.8 Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$), 3.21 (dd, 1H, $J = 3.9$, 13.2 Hz, NCHCH_2Ph), 3.03-2.91 (m, 2H, NCHCH_2Ph and $\text{S}=\text{CSCH}_2$), 2.77 (ap d, 1H, $J = 11.7$ Hz, $\text{S}=\text{CSCH}_2$), 2.71 (d, 1H, $J = 6.8$ Hz, OH), 2.32 (s, 3H, ArCH_3), 1.22 (d, 3H, $J = 6.8$ Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$); ^{13}C NMR (100 MHz, CDCl_3) 201.8, 178.4, 139.3, 137.9, 136.7, 129.7, 129.4, 129.1, 127.4, 126.2, 77.7, 69.1, 46.3, 36.8, 32.8, 21.3, 14.9; HRMS (EI): Exact mass calcd for $\text{C}_{21}\text{H}_{23}\text{O}_2\text{NS}_2$ $[\text{M}]^+$, 385.1170. Found 385.1177.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-3-(4-methoxyphenyl)-2-methylpropan-1-one (2c). The title compound was prepared according to General Procedure A and purified by crystallization from CH_2Cl_2 /hexanes (91% yield): mp: 134.3 to 137.4 $^\circ\text{C}$; $[\alpha]_D^{25} = +283.6^\circ$ ($c = 1.05$, CH_2Cl_2); IR (film) 3479, 2964, 1667, 1614, 1504, 1455, 1372, 1338, 1236, 1190, 1161, 1136, 1030, 820, 743 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.37-7.24 (m, 7H, ArH), 6.90-6.86 (m, 2H, ArH), 5.04 (ap ddd, 1H, $J = 3.9$, 6.6, 10.5 Hz, NCHCH_2Ph), 4.83 (ap d, 1H, $J = 6.3$ Hz, $\text{CH}(\text{OH})$), 4.75 (dq, 1H, $J = 6.8$, 6.8 Hz, $\text{CH}(\text{CH}_3)\text{C}=\text{O}$), 3.79 (s, 3H, OCH_3), 3.22 (dd, 1H, $J = 3.9$, 13.2 Hz, NCHCH_2Ph), 3.09 (dd, 1H, $J = 7.3$, 11.2 Hz, $\text{S}=\text{CSCH}_2$), 3.01 (dd, 1H, $J = 10.7$, 13.2 Hz, NCHCH_2Ph), 2.80 (ap d, 1H, $J =$

11.2 Hz, S=CSC₂H₅), 2.61 (d, 1H, *J* = 6.3 Hz, OH), 1.19 (d, 3H, *J* = 6.8 Hz, CH(CH₃)C=O); ¹³C NMR (100 MHz, CDCl₃) 201.8, 178.4, 159.6, 136.7, 134.4, 129.7, 129.1, 127.6, 127.4, 114.1, 77.5, 69.2, 55.6, 46.3, 36.9, 32.9, 15.0; HRMS (EI): Exact mass calcd for C₂₁H₂₃O₃NS₂ [M]⁺, 401.1120. Found 401.1118.

(4E)-1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-2,4-dimethyl-5-phenylpent-4-en-1-one (2e). The title compound was prepared according to General Procedure A and isolated as a yellow foam (90% yield): [α]_D²⁵ = +227.4° (*c* = 1.06, CH₂Cl₂); IR (film) 3447, 2975, 2874, 1698, 1493, 1454, 1371, 1342, 1292, 1261, 1192, 1165, 1137, 1035, 1006, 918, 844, 755 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 7.40-7.22 (m, 10H, ArH), 6.57 (s, 1H, PhCH=C), 5.24 (ap ddd, 1H, *J* = 3.9, 6.8, 10.8 Hz, NCHCH₂Ph), 4.76 (dq, 1H, *J* = 6.8, 6.8 Hz, CH(CH₃)C=O), 4.40 (ap d, 1H, *J* = 6.8 Hz, PhCH=C(CH₃)CH(OH)), 3.35-3.20 (m, 2H, NCHCH₂Ph and S=CSC₂H₅), 3.07 (dd, 1H, *J* = 10.7, 13.2 Hz, NCHCH₂Ph), 2.88 (ap d, 1H, *J* = 11.2 Hz, S=CSC₂H₅), 2.51 (d, 1H, *J* = 5.9 Hz, OH), 1.90 (s, 3H, PhCH=C(CH₃)), 1.30 (d, 3H, *J* = 6.8 Hz, CH(CH₃)C=O); ¹³C NMR (100 MHz, CDCl₃) 201.9, 178.2, 137.9, 137.2, 136.7, 129.7, 129.2, 129.1, 128.5, 128.0, 127.5, 127.1, 81.7, 69.2, 42.4, 36.9, 32.9, 15.2, 13.9; HRMS (CI, NH₃): Exact mass calcd for C₂₃H₂₅O₂NS₂ [M]⁺, 412.1405. Found 412.1401.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-2-methyl-3-(2-naphthyl)propan-1-one (2f). The title compound was prepared according to General Procedure A and purified by flash chromatography (15-50% Et₂O/pentane) to give a yellow foam (84% yield): [α]_D²⁵ = +238.1° (*c* = 1.04, CH₂Cl₂); IR (film) 3442, 2974, 1684, 1654, 1456, 1363, 1341, 1262, 1191, 1165, 1137, 1029, 779, 738, 702 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 8.15 d, 1H, *J* = 8.3 Hz, ArH), 7.90-7.80 (m, 2H, ArH), 7.61-7.23 (m, 9H, ArH), 5.69 (ap d, 1H, *J* = 6.8 Hz, ArCH(OH)), 5.20 (dq, 1H, *J* = 6.8, 6.8 Hz, CH(CH₃)C=O), 5.00 (ap ddd, 1H, *J* = 3.9, 6.8, 10.3 Hz, NCHCH₂Ph), 3.23 (dd, 1H, *J* = 3.4, 13.2 Hz, NCHCH₂Ph), 3.05-2.91 (m, 2H, NCHCH₂Ph and S=CSC₂H₅), 2.74 (ap d, 1H, *J* = 11.2 Hz, S=CSC₂H₅), 1.30 (d, 3H, *J* = 6.8 Hz, CH(CH₃)C=O); ¹³C NMR (100 MHz, CDCl₃) 201.6, 178.3, 137.8, 136.7, 134.0, 130.7, 129.7, 129.15, 129.11, 128.9, 127.4, 126.8, 126.2, 125.4, 124.0, 123.5, 75.1, 69.0, 45.6, 36.8, 32.8, 15.2; HRMS (EI): Exact mass calcd for C₂₄H₂₅O₂NS₂ [M]⁺, 421.1170. Found 421.1163.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-2,4-dimethylpent-4-en-1-one (2g). The title compound was prepared according to General Procedure A and isolated as yellow crystals (56% yield): mp: 111.1 to 114.0 °C; [α]_D²⁵ = +256.5° (*c* = 1.04, CH₂Cl₂); IR (film) 3496, 2974, 2935, 1698, 1495, 1455, 1361, 1341, 1929, 1263, 1192, 1167, 1137, 1065, 1031, 1010, 907, 740 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 7.37-7.25 (m, 5H, ArH), 5.22 (ap ddd, 1H, *J* = 3.9, 6.8, 10.8 Hz, NCHCH₂Ph), 4.97 (d, 2H, *J* = 38.6 Hz, CH₂=C(CH₃)CHOH), 4.58 (dq, 1H, *J* = 6.8, 6.8 Hz, CH(CH₃)C=O), 4.25 (ap d, 1H, *J* = 6.8 Hz, CH₂=C(CH₃)CH(OH)), 3.39 (dd, 1H, *J* = 6.8, 11.2 Hz, S=CSC₂H₅), 3.25 (dd, 1H, *J* = 3.9, 13.2 Hz, NCHCH₂Ph), 3.05 (dd, 1H, *J* = 10.7, 13.2 Hz, NCHCH₂Ph), 2.90 (ap d, 1H, *J* = 11.7 Hz, S=CSC₂H₅), 2.41 (d, 1H, *J* = 6.3 Hz, OH), 1.73 (s, 3H, CH₂=C(CH₃)), 1.22 (d, 3H, *J* = 7.3 Hz, CH(CH₃)C=O); ¹³C NMR (100 MHz, CDCl₃) 201.8, 178.3, 145.3, 136.7, 129.7, 129.1, 127.5, 113.9, 79.7, 69.2, 42.2, 36.9, 33.0, 17.8, 15.1; HRMS (CI, NH₃): Exact mass calcd for C₁₇H₂₁O₂NS₂ [M + NH₄]⁺, 353.1357. Found 353.1353.

2-((1R)hydroxyphenylmethyl)(2S)-1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]butan-1-one (3b). The title compound was prepared according to General Procedure A and isolated as yellow crystals (88% yield): mp: 107.5 to 110.0 °C; [α]_D²⁵ = +280.0° (*c* = 1.03, CH₂Cl₂); IR (film) 3500, 3027, 1964, 2930, 1662, 1494, 1455, 1362, 1340, 1262, 1191, 1164, 1136, 1036, 744, 700 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 7.41-7.22 (m, 10H, ArH), 5.10-4.95 (m, 2H, NCHCH₂Ph and CH(OH)), 4.83-4.77 (m, 1H, CH(CH₂CH₃)C=O), 3.22 (d, 1H, *J* = 7.8 Hz, OH), 3.14 (dd, 1H, *J* = 3.9, 13.2 Hz,

NCHCH₂Ph), 2.94 (dd, 1H, *J* = 10.7, 13.7 Hz, NCHCH₂Ph), 2.69-2.62 (m, 2H, S=CSCCH₂), 1.97-1.84 (m, 2H, CH(CH₂CH₃)C=O), 1.09 (t, 3H, *J* = 7.3 Hz, CH(CH₂CH₃)C=O); ¹³C NMR (100 MHz, CDCl₃) 202.2, 177.6, 142.9, 136.6, 129.7, 129.1, 128.7, 127.7, 127.5, 125.5, 74.2, 68.8, 51.5, 36.9, 32.5, 22.5, 11.5; HRMS (CI, NH₃): Exact mass calcd for C₂₁H₂₃O₂NS₂ [M]⁺, 385.1171. Found 385.1172.

2-((1R)hydroxyphenylmethyl)(2S)-1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]pent-4-en-1-one (3c).

The title compound was prepared according to General Procedure A and isolated as yellow crystals (91% yield): mp: 97.0 to 98.7 °C; [α]_D²⁵ = +243.0° (*c* = 1.08, CH₂Cl₂); IR (film) 3484, 3025, 1690, 1366, 1341, 1290, 1261, 1192, 1162, 1136, 1034, 916, 744, 700 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 7.40-7.18 (m, 10H, ArH), 5.99-5.81 (m, 1H, CH₂=CHCH₂), 5.21 (dd, 1H, *J* = 2.0, 17.1, CH₂=CHCH₂), 5.17-5.07 (m, 2H, CH₂=CHCH₂ and O=CCHCH₂CH=CH₂), 4.95 (dd, 1H, *J* = 5.4, 8.3 Hz, CH(OH)), 4.82 (ap ddd, 1H, *J* = 3.9, 5.9, 10.7, NCHCH₂Ph), 3.24 (d, 1H, *J* = 8.3 Hz, OH), 3.11 (dd, 1H, *J* = 3.4, 13.2 Hz, NCHCH₂Ph), 2.91 (dd, 1H, *J* = 10.7, 13.2 Hz, NCHCH₂Ph), 2.70-2.56 (m, 4H, S=CSCCH₂ and CH₂=CHCH₂), 1.09 (t, 3H, *J* = 7.3 Hz, CH(CH₂CH₃)C=O); ¹³C NMR (100 MHz, CDCl₃) 202.2, 176.9, 142.6, 136.6, 134.5, 129.6, 129.1, 128.7, 127.8, 127.5, 125.6, 118.4, 74.2, 68.8, 50.0, 36.9, 33.3, 32.4; HRMS (EI): Exact mass calcd for C₂₂H₂₃O₂NS₂ [M]⁺, 397.1170. Found 397.1178.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)](2S,3R)-3-hydroxy-3-phenyl-2-benzylpropan-1-one (3d).

The title compound was prepared according to General Procedure A and purified by flash chromatography (10% Et₂O/pentane) as a yellow foam (84% yield): [α]_D²⁵ = +190.1° (*c* = 1.00, CH₂Cl₂); IR (film) 3496, 3027, 2924, 1670, 1494, 1454, 1372, 1341, 1293, 1262, 1192, 1162, 1137, 1053, 1031, 817, 745, 700 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 7.50-7.08 (m, 15H, ArH), 5.23-5.16 (m, 1H, O=CCHCH₂Ph), 4.70 (dd, 1H, *J* = 4.4, 10.3, PhCH(OH)), 4.63 (ap ddd, 1H, *J* = 3.9, 6.8, 10.3, NCHCH₂Ph), 3.57 (d, 1H, *J* = 10.3 Hz, OH), 3.25 (dd, 1H, *J* = 4.9, 13.2 Hz, O=CCHCH₂Ph), 3.13 (dd, 1H, *J* = 10.7, 13.2 Hz, O=CCHCH₂Ph), 3.02 (dd, 1H, *J* = 3.9, 13.2 NCHCH₂Ph), 2.84 (dd, 1H, *J* = 10.3, 13.2 Hz, NCHCH₂Ph), 2.58 (ap d, 1H, *J* = 11.2 Hz, S=CSCCH₂), 2.48 (dd, 1H, *J* = 6.8, 11.2 Hz, S=CSCCH₂); ¹³C NMR (100 MHz, CDCl₃) 202.3, 177.4, 142.8, 138.2, 136.5, 129.8, 129.6, 129.1, 128.8, 128.6, 127.52, 127.48, 127.0, 125.1, 72.6, 68.7, 52.3, 36.7, 34.2, 32.6; HRMS (EI): Exact mass calcd for C₂₆H₂₅O₂NS₂ [M]⁺, 447.1327. Found 447.1325.

2-((1R)hydroxyphenylmethyl)(2S)-1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]-4-methyl-pentan-1-one (3e).

The title compound was prepared according to General Procedure A and isolated as a yellow foam (93% yield): [α]_D²⁵ = +234.9° (*c* = 1.26, CH₂Cl₂); IR (film) 3504, 2955, 2868, 1685, 1456, 1364, 1340, 1291, 1263, 1192, 1162, 1136, 1034, 743, 701 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) 7.35-7.18 (m, 10H, ArH), 5.20 (ap dd, 1H, *J* = 4.8, 7.3 Hz, O=CCHCH(OH)), 4.91 (dd, 1H, *J* = 4.8, 8.8 Hz, PhCH(OH)), 4.76 (m, 1H, NCHCH₂Ph), 3.24 (d, 1H, *J* = 9.2 Hz, OH), 3.08 (dd, 1H, *J* = 3.7, 13.2 Hz, NCHCH₂Ph), 2.88 (dd, 1H, *J* = 10.6, 13.2 Hz, NCHCH₂Ph), 2.61-2.50 (m, 2H, S=CSCCH₂), 1.95-1.83 (m, 1H, CH(CH₃)₂), 1.82-1.69 (m, 2H, CH₂CH(CH₃)₂), 0.97 (dd, 6H, *J* = 6.6, 9.9 Hz, CH(CH₃)₂); ¹³C NMR (100 MHz, CDCl₃) 202.3, 177.6, 142.8, 136.6, 129.6, 129.1, 128.7, 127.7, 127.4, 125.3, 74.6, 68.8, 48.4, 37.9, 36.8, 32.2, 25.9, 23.9, 22.3; HRMS (EI): Exact mass calcd for C₂₃H₂₇O₂NS₂ [M]⁺, 413.1483. Found 412.1488.

General Procedure B: Thiazolidinethione acylation. To an oven-dried round-bottom flask under nitrogen atmosphere with a magnetic stirbar was added (4S)-4-benzyl-1,3-thiazolidine-2-thione³ (11.9 mmol, 1 equiv.), CH₂Cl₂ (20 mL), and Et₃N (12.9 mmol, 1.1 equiv.). The mixture was cooled to 0 °C. The acid chloride

(12.5 mmol, 1.05 equiv.) was diluted in 5 mL CH_2Cl_2 and added dropwise over thirty minutes *via* addition funnel. The funnel was rinsed with 5 mL CH_2Cl_2 and the yellow reaction mixture was allowed to warm slowly to room temperature. After two hours, the reaction mixture was diluted with water. The layers were separated and the organic layer was extracted with brine. The organic layer was dried over Na_2SO_4 for 1 h, then filtered through a cotton plug and concentrated *in vacuo*. Flash chromatography (20% CH_2Cl_2 /hexanes) afforded the acylthiazolidinethione as a yellow, crystalline solid.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]butan-1-one (1b) The title compound was prepared according to General Procedure B and isolated as yellow crystals (90% yield): mp: 82.1 to 87.4 °C; $[\alpha]_{\text{D}}^{25} = +221.1^\circ$ ($c = 1.00$, CH_2Cl_2); IR (film) 3837, 3734, 3674, 2963, 1698, 1652, 1558, 1540, 1506, 1456, 1362, 1340, 1261, 1165, 1037, 746, 702 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.37-7.28 (m, 5H, ArH), 5.38 (ap ddd, 1H, $J = 3.9, 6.8, 10.8$ Hz, NCHCH₂Ph), 3.40-3.31 (m, 2H, O=CCH₂CH₂CH₃), 3.22 (dd, 1H, $J = 3.4, 13.2$ Hz, NCHCH₂Ph), 3.15-2.98 (m, 2H, NCHCH₂Ph and S=CSCCH₂), 2.88 (ap d, 1H, $J = 11.7$ Hz, S=CSCCH₂), 1.82-1.63 (m, 2H, O=CCH₂CH₂CH₃), 0.99 (t, 3H, $J = 7.8$ Hz, O=CCH₂CH₂CH₃); ^{13}C NMR (100 MHz, CDCl_3) 201.4, 174.3, 136.8, 129.7, 129.1, 127.4, 68.9, 40.6, 37.0, 32.1, 18.4, 13.9; HRMS (CI, NH_3): Exact mass calcd for $\text{C}_{14}\text{H}_{17}\text{ONS}_2$ $[\text{M}]^+$, 279.0752. Found 279.0754.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]pent-4-en-1-one (1c) The title compound was prepared according to General Procedure B and isolated as yellow crystals (76% yield): mp: 66.0 to 69.0 °C; $[\alpha]_{\text{D}}^{25} = +222.1^\circ$ ($c = 1.30$, CH_2Cl_2); IR (film) 3027, 2923, 1695, 1395, 1360, 1342, 1293, 1266, 1191, 1163, 1137, 1041, 916, 747, 702 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.37-7.25 (m, 5H, ArH), 5.92-5.82 (m, 1H, CH₂=CHCH₂), 5.37 (ap ddd, 1H, $J = 3.9, 7.1, 10.5$ Hz, NCHCH₂Ph), 5.09 (dd, 1H, $J = 1.5, 17.1$ Hz, CH₂=CHCH₂), 5.03 (dd, 1H, $J = 1.5, 10.3$ Hz, CH₂=CHCH₂), 3.49 (ddd, 1H, $J = 6.3, 8.3, 17.6$ Hz, O=CCH₂CH₂CH=CH₂), 3.38 (dd, 1H, $J = 7.3, 11.2$ Hz, S=CSCCH₂), 3.30-3.20 (m, 2H, O=CCH₂CH₂CH=CH₂ and NCHCH₂Ph), 3.05 (dd, 1H, $J = 10.7, 13.2$ Hz, NCHCH₂Ph), 2.89 (ap d, 1H, $J = 11.7$ Hz, S=CSCCH₂), 2.53-2.38 (m, 2H, CH₂=CHCH₂); ^{13}C NMR (100 MHz, CDCl_3) 201.4, 173.6, 137.0, 136.8, 129.7, 129.2, 127.5, 115.9, 68.8, 38.1, 37.0, 32.2, 28.9; HRMS (EI): Exact mass calcd for $\text{C}_{15}\text{H}_{17}\text{ONS}_2$ $[\text{M}]^+$, 291.0752. Found 291.0756.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]-3-phenylpropan-1-one (1d) The title compound was prepared according to General Procedure B and isolated as yellow crystals (76% yield): mp: 75.5 to 78.0 °C; $[\alpha]_{\text{D}}^{25} = +195.6^\circ$ ($c = 1.00$, CH_2Cl_2); IR (film) 3448, 3026, 2924, 1696, 1496, 1454, 1362, 1341, 1294, 1264, 1190, 1160, 1136, 1044, 747, 700 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.37-7.19 (m, 10H, ArH), 5.33 (ap ddd, 1H, $J = 3.9, 6.8, 10.8$ Hz, NCHCH₂Ph), 3.70 (ddd, 1H, $J = 5.9, 9.0, 17.3$ Hz, O=CCH₂CH₂Ph), 3.52 (ddd, 1H, $J = 6.8, 9.0, 17.3$ Hz, O=CCH₂CH₂Ph), 3.34 (dd, 1H, $J = 7.3, 11.2$ Hz, S=CSCCH₂), 3.22 (dd, 1H, $J = 3.9, 13.2$ Hz, NCHCH₂Ph), 3.11-2.95 (m, 3H, O=CCH₂CH₂Ph and NCHCH₂Ph), 2.87 (ap d, 1H, $J = 11.2$ Hz, S=CSCCH₂); ^{13}C NMR (100 MHz, CDCl_3) 201.4, 173.5, 140.7, 136.7, 129.7, 129.2, 128.75, 128.73, 127.5, 126.5, 68.8m 40.2, 27.0, 32.2, 31.0; HRMS (EI): Exact mass calcd for $\text{C}_{19}\text{H}_{19}\text{ONS}_2$ $[\text{M}]^+$, 341.0909. Found 341.0901.

1-[(4S)-4-benzyl-2-thioxo(1,3-thiazolidin-3-yl)]-4-methylpentan-1-one (1e) The title compound was prepared according to General Procedure B and isolated as yellow crystals (87% yield): mp: 55.4 °C; $[\alpha]_{\text{D}}^{25} = +198.8^\circ$ ($c = 1.21$, CH_2Cl_2); IR (film) 2955, 2868, 1698, 1455, 1365, 1341, 1292, 1256, 1190, 1162, 1138, 1041, 747 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.36-7.27 (m, 5H, ArH), 5.37 (ap ddd, 1H, $J = 3.9, 6.8, 10.8$ Hz, NCHCH₂Ph), 3.41-3.33 (m, 2H, O=CCH₂CH₂CH(CH₃)₂), 3.24-3.12 (m, 2H, NCHCH₂Ph and S=CSCCH₂), 3.05 (dd, 1H, $J = 10.7, 13.2$ Hz, NCHCH₂Ph), 2.88 (ap d, 1H, $J = 11.7$ Hz, S=CSCCH₂), 1.68-1.59 (m, 2H,

$\text{O}=\text{CCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2$, 1.58-1.48 (m, 2H, $\text{O}=\text{CCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2$), 0.93 (d, 6H, $J = 6.3$ Hz, $\text{O}=\text{CCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2$); ^{13}C NMR (100 MHz, CDCl_3) 201.4, 174.7, 136.8, 129.7, 129.1, 127.4, 68.9, 37.0, 36.8, 33.7, 32.1, 27.9, 22.7, 22.6; HRMS (CI, NH_3): Exact mass calcd for $\text{C}_{16}\text{H}_{21}\text{ONS}_2$ $[\text{M}]^+$, 307.1064. Found 307.1068.

Semiempirical Calculations

General Information Transition-structure optimizations were performed at the PM3 level using the Spartan Semiempirical Program 5.0 (Wavefunction Inc., Irvine, CA 92715) on a Silicon Graphics Impact 10000 (195 MHz, 128 M RAM) running IRIX 6.2. Calculations were performed without solvent using these parameters: optcycle = 2000, maxcycle = 2000, charge = 0, multiplicity = 0. Calculations converged (energy difference between cycles < 0.0005 kcal/mol) in 4 h cpu time.

Oxazolidinone boat conformer E To an octahedral magnesium atom were complexed two molecules of water in a cis relationship. Trans to one water was complexed a molecule of benzaldehyde; the exocyclic oxygen of the oxazolidinone auxiliary was complexed trans to the other water. The enolate oxygen was complexed to the magnesium such that the *Z*-enolate *re* face was oriented toward the aldehyde. The aldehyde was oriented such that its *si* face was open to attack. The six atoms of the pericyclic transition state were arranged in a boat geometry, and the forming carbon-carbon bond was constrained to 2.0 Å. A bromine atom was complexed to the magnesium trans to the enolate oxygen. The optimal geometry was determined in a two-step fashion. First, a PM3-level geometry optimization calculation was performed. Second, the resultant octahedral complex was re-optimized at the PM3 level as a transition structure. $H = -305.026$ kcal/mol.

Oxazolidinone chair conformer F To an octahedral magnesium atom were complexed two molecules of water in a cis relationship. Trans to one water was complexed a molecule of benzaldehyde; the exocyclic oxygen of the oxazolidinone auxiliary was complexed trans to the other water. The enolate oxygen was complexed to the magnesium such that the *Z*-enolate *re* face was oriented toward the aldehyde. The aldehyde was oriented such that its *re* face was open to attack. The six atoms of the pericyclic transition state were arranged in a chair geometry, and the forming carbon-carbon bond was constrained to 2.0 Å. A bromine atom was complexed to the magnesium trans to the enolate oxygen. The optimal geometry was determined in a two-step fashion. First, a PM3-level geometry optimization calculation was performed. Second, the resultant octahedral complex was re-optimized at the PM3 level as a transition structure. $H = -302.492$ kcal/mol.

Thiazolidinethione boat conformer G To an octahedral magnesium atom were complexed two molecules of water in a cis relationship. Trans to water A was complexed a molecule of benzaldehyde; trans to water B was complexed a third molecule of water (C). The enolate oxygen was complexed to the magnesium such that the *Z*-enolate *si* face was oriented toward the aldehyde. The aldehyde was oriented such that its *re* face was open to attack. The six atoms of the pericyclic transition state were arranged in a boat geometry, and the forming carbon-carbon bond was constrained to 2.0 Å. The exocyclic sulfur of the auxiliary was oriented directly away from the oxygen of the enolate. A bromine atom was complexed to the magnesium trans to the enolate oxygen. The optimal geometry was determined in a two-step fashion. First, a PM3-level

geometry optimization calculation was performed. Second, the resultant octahedral complex was re-optimized at the PM3 level as a transition structure. $H = -260.767$ kcal/mol.

Thiazolidinethione chair conformer H To an octahedral magnesium atom were complexed two molecules of water in a cis relationship. Trans to water A was complexed a molecule of benzaldehyde; trans to water B was complexed a third molecule of water (C). The enolate oxygen was complexed to the magnesium such that the *Z*-enolate *si* face was oriented toward the aldehyde. The aldehyde was oriented such that its *si* face was open to attack. The six atoms of the pericyclic transition state were arranged in a chair geometry, and the forming carbon-carbon bond was constrained to 2.0 Å. The exocyclic sulfur of the auxiliary was oriented directly away from the oxygen of the enolate. A bromine atom was complexed to the magnesium trans to the enolate oxygen. The optimal geometry was determined in a two-step fashion. First, a PM3-level geometry optimization calculation was performed. Second, the resultant octahedral complex was re-optimized at the PM3 level as a transition structure. $H = -257.943$ kcal/mol.

Stereochemical Proofs

Aldol adduct **2a** was transformed to its methyl ester by literature procedure,⁴ and its optical rotation ($[\alpha]_D^{25} = +52.1^\circ$ ($c = 1.09$, CH_2Cl_2)) compared to literature value ($[\alpha]_D^{25} = +53.1^\circ$ ($c = 1.05$, CHCl_3)).⁵ Relative stereochemistry was determined according to the method of Heathcock.⁶

Aldol adduct **2d** was transformed to its methyl ester by literature procedure,⁴ and its optical rotation ($[\alpha]_D^{25} = -15.1^\circ$ ($c = 2.22$, MeOH)) compared to literature value of its enantiomer ($[\alpha]_D^{25} = +22.0^\circ$ ($c = 0.785$, MeOH)).⁷ Relative stereochemistry was determined according to the method of Heathcock.⁶

Aldol adduct **2e** was transformed to its methyl ester by literature procedure,⁴ and its optical rotation measured ($[\alpha]_D^{25} = -62.6^\circ$ ($c = 1.56$, CH_2Cl_2)). Known oxazolidinone **A**⁸ was transformed to its methyl ester by literature procedure⁹, and the optical rotation ($[\alpha]_D^{25} = +64.9^\circ$ ($c = 0.35$, CH_2Cl_2)) compared. Relative stereochemistry was determined according to the method of Heathcock.⁶

Aldol adduct **2f** was transformed to its methyl ester by literature procedure,⁴ and its optical rotation measured ($[\alpha]_D^{25} = +59.9^\circ$ ($c = 1.47$, CH_2Cl_2)). Known oxazolidinone **B**⁷ was transformed to its methyl ester by literature procedure,⁹ and the optical rotation ($[\alpha]_D^{25} = -51.4^\circ$ ($c = 0.26$, CH_2Cl_2)) compared. Relative stereochemistry was determined according to the method of Heathcock.⁶

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Absolute stereochemistry for aldol adducts **2b**, **2c**, **2g**, **3b**, and **3c** was determined by X-ray crystallography:

X-ray crystallographic data for aldol adduct **2b**:

Table 1. Crystal data and structure refinement for **2b**.

Identification code	cwd75t	
Empirical formula	C ₂₁ H ₂₃ N O ₂ S ₂	
Formula weight	385.52	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 12.0820(7) Å	= 90°.
	b = 12.9214(7) Å	= 90.0090(10)°.
	c = 26.1938(14) Å	= 90°.
Volume	4089.3(4) Å ³	
Z	8	
Density (calculated)	1.252 Mg/m ³	
Absorption coefficient	0.275 mm ⁻¹	
F(000)	1632	
Crystal size	2.0 x 3.2 x 7.0 mm ³	
Theta range for data collection	1.55 to 27.74°.	
Index ranges	-15<=h<=15, -11<=k<=16, -20<=l<=34	
Reflections collected	25816	
Independent reflections	14300 [R(int) = 0.0565]	
Completeness to theta = 27.74°	90.2 %	
Absorption correction	None	

Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	14300 / 481 / 837
Goodness-of-fit on F ²	1.039
Final R indices [I>2sigma(I)]	R1 = 0.0754, wR2 = 0.2075
R indices (all data)	R1 = 0.0861, wR2 = 0.2193
Absolute structure parameter	0.01(11)
Largest diff. peak and hole	0.615 and -0.710 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	-3519(2)	4339(2)	-2575(1)	48(1)
S(2)	-4768(2)	4192(2)	-1623(1)	48(1)
O(1)	-6584(5)	2031(4)	-2668(2)	41(1)
O(2)	-4791(7)	723(6)	-2073(3)	76(2)
N(1)	-5170(5)	3123(5)	-2500(2)	31(1)
C(1)	-4583(7)	3804(6)	-2219(3)	36(2)
C(2)	-3824(6)	3566(6)	-3131(3)	34(1)
C(3)	-5027(6)	3268(6)	-3061(3)	30(1)
C(4)	-5811(5)	4111(6)	-3247(2)	28(1)
C(5)	-5871(3)	4179(3)	-3825(1)	27(1)
C(6)	-6520(3)	3486(3)	-4099(1)	33(1)
C(7)	-6575(3)	3554(4)	-4628(1)	42(1)
C(8)	-5980(4)	4316(4)	-4883(1)	41(1)
C(9)	-5330(3)	5009(3)	-4609(1)	41(1)
C(10)	-5276(3)	4941(3)	-4080(1)	32(1)
C(11)	-5953(7)	2402(6)	-2339(3)	34(1)
C(12)	-5994(8)	2012(7)	-1789(3)	43(2)
C(13)	-7105(8)	2294(7)	-1576(3)	46(2)
C(14)	-5827(10)	871(9)	-1815(4)	64(2)
C(15)	-5816(7)	325(7)	-1285(2)	86(2)
C(16)	-4862(6)	375(8)	-989(3)	109(3)
C(17)	-4828(6)	-112(9)	-516(3)	121(3)
C(18)	-5749(8)	-648(9)	-339(2)	122(3)
C(19)	-6702(7)	-698(8)	-635(3)	112(3)
C(20)	-6736(6)	-211(8)	-1108(3)	99(2)
C(21)	-5754(16)	-1323(19)	189(6)	159(5)
S(3)	-36(2)	-5707(2)	-1618(1)	54(1)
S(4)	1439(2)	-5759(2)	-2505(1)	48(1)
O(3)	-1789(5)	-7870(5)	-2665(2)	48(2)
O(4)	114(6)	-9098(5)	-2128(2)	55(2)
N(2)	-353(5)	-6807(5)	-2484(2)	34(1)
C(51)	242(7)	-6162(5)	-2190(3)	31(2)
C(52)	1207(6)	-6623(7)	-3036(3)	40(2)
C(53)	-48(6)	-6773(6)	-3035(3)	35(1)
C(54)	-666(5)	-5866(6)	-3305(3)	32(1)
C(55)	-350(3)	-5743(3)	-3853(1)	27(1)
C(56)	-828(3)	-6337(3)	-4237(1)	34(1)
C(57)	-465(4)	-6235(4)	-4738(1)	44(1)
C(58)	375(4)	-5540(4)	-4855(1)	50(2)
C(59)	853(3)	-4947(4)	-4471(2)	51(2)
C(60)	491(3)	-5048(4)	-3970(1)	40(1)
C(61)	-1136(7)	-7520(6)	-2327(3)	37(2)
C(62)	-1185(7)	-7932(7)	-1792(3)	41(1)
C(63)	-2271(9)	-7754(9)	-1555(4)	58(2)
C(64)	-886(8)	-9061(7)	-1843(3)	47(2)
C(65)	-783(6)	-9623(6)	-1336(2)	61(2)
C(66)	175(5)	-9517(6)	-1047(3)	78(2)
C(67)	271(6)	-10025(8)	-582(3)	102(2)
C(68)	-592(7)	-10640(8)	-405(3)	107(2)
C(69)	-1551(6)	-10746(7)	-694(3)	94(2)
C(70)	-1646(5)	-10238(7)	-1159(3)	80(2)
S(7)	2632(2)	-3387(2)	-3370(1)	52(1)
S(8)	3956(2)	-3462(2)	-2436(1)	56(1)
O(7)	894(6)	-1173(5)	-2302(2)	53(2)
O(8)	2829(5)	-63(5)	-2934(2)	47(1)
N(4)	2219(6)	-2297(5)	-2481(2)	33(1)

C(151)	2865(7)	-2983(8)	-2780(3)	46(2)
C(152)	3683(8)	-2589(9)	-1900(3)	53(2)
C(153)	2441(8)	-2408(7)	-1929(3)	46(2)
C(154)	1706(10)	-3273(8)	-1708(3)	61(2)
C(155)	1800(11)	-3421(7)	-1157(2)	108(2)
C(156)	2462(11)	-4209(7)	-964(3)	131(3)
C(157)	2559(11)	-4343(8)	-439(4)	144(3)
C(158)	1994(12)	-3690(9)	-108(2)	147(3)
C(159)	1332(10)	-2902(8)	-301(3)	141(3)
C(160)	1235(9)	-2768(7)	-825(4)	124(3)
C(161)	1443(7)	-1557(6)	-2618(3)	37(2)
C(162)	1392(7)	-1176(6)	-3186(3)	36(1)
C(163)	165(8)	-1291(9)	-3379(4)	57(2)
C(164)	1794(6)	-53(5)	-3194(2)	31(1)
C(165)	1856(4)	332(3)	-3735(1)	29(1)
C(166)	1123(3)	1069(3)	-3922(1)	38(1)
C(167)	1193(4)	1385(3)	-4429(1)	42(1)
C(168)	1996(4)	964(3)	-4747(1)	34(1)
C(169)	2729(3)	228(3)	-4559(1)	35(1)
C(170)	2659(3)	-88(3)	-4053(1)	32(1)
C(171)	2056(7)	1313(6)	-5306(2)	52(2)
S(5)	-1012(2)	-3456(2)	-2480(1)	48(1)
S(6)	-2421(3)	-3437(2)	-3396(1)	59(1)
O(5)	-4219(5)	-1355(5)	-2353(2)	51(2)
O(6)	-2468(6)	80(6)	-2863(2)	61(2)
N(3)	-2766(5)	-2352(5)	-2515(2)	33(1)
C(101)	-2135(7)	-2993(8)	-2825(3)	44(2)
C(102)	-1317(7)	-2583(8)	-1940(3)	43(2)
C(103)	-2555(8)	-2422(7)	-1968(3)	43(2)
C(104)	-3267(9)	-3276(8)	-1727(3)	58(2)
C(105)	-3148(8)	-3327(7)	-1164(2)	80(2)
C(106)	-3782(8)	-2691(6)	-852(3)	96(2)
C(107)	-3705(9)	-2779(8)	-324(3)	116(3)
C(108)	-2995(10)	-3504(8)	-109(2)	126(3)
C(109)	-2361(9)	-4141(7)	-421(3)	121(3)
C(110)	-2438(9)	-4052(7)	-949(3)	103(2)
C(111)	-3613(7)	-1633(6)	-2663(3)	36(2)
C(112)	-3598(6)	-1195(6)	-3211(3)	33(1)
C(113)	-4708(8)	-1478(7)	-3491(3)	49(2)
C(114)	-3448(6)	-22(5)	-3153(2)	31(1)
C(115)	-3391(3)	562(3)	-3651(1)	25(1)
C(116)	-2399(3)	677(3)	-3914(1)	30(1)
C(117)	-2368(3)	1248(3)	-4363(1)	34(1)
C(118)	-3328(4)	1704(3)	-4550(1)	35(1)
C(119)	-4320(3)	1589(3)	-4287(1)	36(1)
C(120)	-4352(3)	1018(3)	-3838(1)	29(1)
C(121)	-3308(8)	2344(6)	-5038(3)	55(2)
C(71)	-409(17)	-11119(19)	153(7)	160(5)

Table 3. Bond lengths [Å] and angles [°] for **2b**.

S(1)-C(1)	1.733(8)	C(12)-C(14)	1.489(14)
S(1)-C(2)	1.804(8)	C(12)-C(13)	1.498(12)
S(2)-C(1)	1.655(8)	C(14)-C(15)	1.556(11)
O(1)-C(11)	1.245(9)	C(15)-C(16)	1.3900
O(2)-C(14)	1.437(13)	C(15)-C(20)	1.3900
N(1)-C(1)	1.348(10)	C(16)-C(17)	1.3900
N(1)-C(11)	1.394(10)	C(17)-C(18)	1.3900
N(1)-C(3)	1.489(9)	C(18)-C(19)	1.3900
C(2)-C(3)	1.515(10)	C(18)-C(21)	1.635(16)
C(3)-C(4)	1.524(10)	C(19)-C(20)	1.3900
C(4)-C(5)	1.518(6)	S(3)-C(51)	1.646(7)
C(5)-C(6)	1.3900	S(4)-C(51)	1.746(7)
C(5)-C(10)	1.3900	S(4)-C(52)	1.804(9)
C(6)-C(7)	1.3900	O(3)-C(61)	1.269(10)
C(7)-C(8)	1.3900	O(4)-C(64)	1.421(11)
C(8)-C(9)	1.3900	N(2)-C(51)	1.343(10)
C(9)-C(10)	1.3900	N(2)-C(61)	1.383(10)
C(11)-C(12)	1.528(11)	N(2)-C(53)	1.491(9)

C(52)-C(53)	1.528(11)	C(115)-C(120)	1.3900
C(53)-C(54)	1.559(10)	C(116)-C(117)	1.3900
C(54)-C(55)	1.494(7)	C(117)-C(118)	1.3900
C(55)-C(56)	1.3900	C(118)-C(119)	1.3900
C(55)-C(60)	1.3900	C(118)-C(121)	1.523(7)
C(56)-C(57)	1.3900	C(119)-C(120)	1.3900
C(57)-C(58)	1.3900	C(1)-S(1)-C(2)	93.5(4)
C(58)-C(59)	1.3900	C(1)-N(1)-C(11)	128.8(6)
C(59)-C(60)	1.3900	C(1)-N(1)-C(3)	113.3(6)
C(61)-C(62)	1.500(11)	C(11)-N(1)-C(3)	117.5(6)
C(62)-C(63)	1.469(12)	N(1)-C(1)-S(2)	130.0(6)
C(62)-C(64)	1.509(13)	N(1)-C(1)-S(1)	110.9(5)
C(64)-C(65)	1.519(10)	S(2)-C(1)-S(1)	119.1(5)
C(65)-C(66)	1.3900	C(3)-C(2)-S(1)	103.8(5)
C(65)-C(70)	1.3900	N(1)-C(3)-C(2)	105.3(6)
C(66)-C(67)	1.3900	N(1)-C(3)-C(4)	109.5(5)
C(67)-C(68)	1.3900	C(2)-C(3)-C(4)	112.1(6)
C(68)-C(69)	1.3900	C(5)-C(4)-C(3)	113.0(5)
C(68)-C(71)	1.601(18)	C(6)-C(5)-C(10)	120.0
C(69)-C(70)	1.3900	C(6)-C(5)-C(4)	120.2(4)
S(7)-C(151)	1.655(9)	C(10)-C(5)-C(4)	119.8(4)
S(8)-C(151)	1.713(10)	C(7)-C(6)-C(5)	120.0
S(8)-C(152)	1.830(9)	C(8)-C(7)-C(6)	120.0
O(7)-C(161)	1.171(10)	C(7)-C(8)-C(9)	120.0
O(8)-C(164)	1.424(10)	C(8)-C(9)-C(10)	120.0
N(4)-C(161)	1.387(11)	C(9)-C(10)-C(5)	120.0
N(4)-C(151)	1.417(11)	O(1)-C(11)-N(1)	117.6(7)
N(4)-C(153)	1.477(10)	O(1)-C(11)-C(12)	120.4(7)
C(152)-C(153)	1.520(14)	N(1)-C(11)-C(12)	121.9(7)
C(153)-C(154)	1.540(14)	C(14)-C(12)-C(13)	112.3(8)
C(154)-C(155)	1.461(10)	C(14)-C(12)-C(11)	106.2(7)
C(155)-C(156)	1.3900	C(13)-C(12)-C(11)	107.4(7)
C(155)-C(160)	1.3900	O(2)-C(14)-C(12)	105.7(9)
C(156)-C(157)	1.3900	O(2)-C(14)-C(15)	110.6(8)
C(157)-C(158)	1.3900	C(12)-C(14)-C(15)	114.2(9)
C(158)-C(159)	1.3900	C(16)-C(15)-C(20)	120.0
C(159)-C(160)	1.3900	C(16)-C(15)-C(14)	118.9(7)
C(161)-C(162)	1.567(10)	C(20)-C(15)-C(14)	121.1(7)
C(162)-C(164)	1.531(10)	C(15)-C(16)-C(17)	120.0
C(162)-C(163)	1.573(12)	C(18)-C(17)-C(16)	120.0
C(164)-C(165)	1.503(7)	C(17)-C(18)-C(19)	120.0
C(165)-C(166)	1.3900	C(17)-C(18)-C(21)	123.5(9)
C(165)-C(170)	1.3900	C(19)-C(18)-C(21)	116.3(10)
C(166)-C(167)	1.3900	C(20)-C(19)-C(18)	120.0
C(167)-C(168)	1.3900	C(19)-C(20)-C(15)	120.0
C(168)-C(169)	1.3900	C(51)-S(4)-C(52)	92.9(4)
C(168)-C(171)	1.534(6)	C(51)-N(2)-C(61)	127.4(7)
C(169)-C(170)	1.3900	C(51)-N(2)-C(53)	113.9(6)
S(5)-C(101)	1.737(9)	C(61)-N(2)-C(53)	118.6(6)
S(5)-C(102)	1.845(9)	N(2)-C(51)-S(3)	129.5(6)
S(6)-C(101)	1.638(9)	N(2)-C(51)-S(4)	110.9(5)
O(5)-C(111)	1.151(10)	S(3)-C(51)-S(4)	119.5(5)
O(6)-C(114)	1.412(10)	C(53)-C(52)-S(4)	103.5(5)
N(3)-C(101)	1.388(11)	N(2)-C(53)-C(52)	104.5(6)
N(3)-C(111)	1.435(9)	N(2)-C(53)-C(54)	110.0(6)
N(3)-C(103)	1.459(10)	C(52)-C(53)-C(54)	112.3(7)
C(102)-C(103)	1.512(13)	C(55)-C(54)-C(53)	113.1(5)
C(103)-C(104)	1.535(13)	C(56)-C(55)-C(60)	120.0
C(104)-C(105)	1.482(10)	C(56)-C(55)-C(54)	122.0(4)
C(105)-C(106)	1.3900	C(60)-C(55)-C(54)	117.9(4)
C(105)-C(110)	1.3900	C(57)-C(56)-C(55)	120.0
C(106)-C(107)	1.3900	C(56)-C(57)-C(58)	120.0
C(107)-C(108)	1.3900	C(59)-C(58)-C(57)	120.0
C(108)-C(109)	1.3900	C(58)-C(59)-C(60)	120.0
C(109)-C(110)	1.3900	C(59)-C(60)-C(55)	120.0
C(111)-C(112)	1.543(10)	O(3)-C(61)-N(2)	117.0(7)
C(112)-C(114)	1.535(10)	O(3)-C(61)-C(62)	120.0(7)
C(112)-C(113)	1.571(11)	N(2)-C(61)-C(62)	122.8(7)
C(114)-C(115)	1.508(8)	C(63)-C(62)-C(61)	112.0(8)
C(115)-C(116)	1.3900	C(63)-C(62)-C(64)	113.7(8)

C(61)-C(62)-C(64)	104.5(7)	C(166)-C(167)-C(168)	120.0
O(4)-C(64)-C(62)	106.5(7)	C(167)-C(168)-C(169)	120.0
O(4)-C(64)-C(65)	112.0(7)	C(167)-C(168)-C(171)	119.4(4)
C(62)-C(64)-C(65)	113.9(8)	C(169)-C(168)-C(171)	120.6(4)
C(66)-C(65)-C(70)	120.0	C(170)-C(169)-C(168)	120.0
C(66)-C(65)-C(64)	119.8(5)	C(169)-C(170)-C(165)	120.0
C(70)-C(65)-C(64)	120.2(5)	C(101)-S(5)-C(102)	91.9(4)
C(67)-C(66)-C(65)	120.0	C(101)-N(3)-C(111)	128.3(7)
C(66)-C(67)-C(68)	120.0	C(101)-N(3)-C(103)	116.2(7)
C(69)-C(68)-C(67)	120.0	C(111)-N(3)-C(103)	115.5(6)
C(69)-C(68)-C(71)	125.0(9)	N(3)-C(101)-S(6)	128.8(7)
C(67)-C(68)-C(71)	114.9(9)	N(3)-C(101)-S(5)	109.2(6)
C(68)-C(69)-C(70)	120.0	S(6)-C(101)-S(5)	121.3(6)
C(69)-C(70)-C(65)	120.0	C(103)-C(102)-S(5)	104.2(6)
C(151)-S(8)-C(152)	92.4(4)	N(3)-C(103)-C(102)	103.2(6)
C(161)-N(4)-C(151)	131.3(7)	N(3)-C(103)-C(104)	110.5(7)
C(161)-N(4)-C(153)	116.3(6)	C(102)-C(103)-C(104)	115.9(8)
C(151)-N(4)-C(153)	112.4(7)	C(105)-C(104)-C(103)	112.7(8)
N(4)-C(151)-S(7)	128.3(7)	C(106)-C(105)-C(110)	120.0
N(4)-C(151)-S(8)	111.0(6)	C(106)-C(105)-C(104)	120.4(7)
S(7)-C(151)-S(8)	120.6(6)	C(110)-C(105)-C(104)	119.5(7)
C(153)-C(152)-S(8)	103.6(6)	C(105)-C(106)-C(107)	120.0
N(4)-C(153)-C(152)	104.0(7)	C(106)-C(107)-C(108)	120.0
N(4)-C(153)-C(154)	109.4(7)	C(109)-C(108)-C(107)	120.0
C(152)-C(153)-C(154)	116.1(9)	C(108)-C(109)-C(110)	120.0
C(155)-C(154)-C(153)	114.9(9)	C(109)-C(110)-C(105)	120.0
C(156)-C(155)-C(160)	120.0	O(5)-C(111)-N(3)	117.8(7)
C(156)-C(155)-C(154)	120.1(8)	O(5)-C(111)-C(112)	123.2(7)
C(160)-C(155)-C(154)	119.9(8)	N(3)-C(111)-C(112)	118.7(6)
C(155)-C(156)-C(157)	120.0	C(114)-C(112)-C(111)	105.8(6)
C(156)-C(157)-C(158)	120.0	C(114)-C(112)-C(113)	112.1(6)
C(157)-C(158)-C(159)	120.0	C(111)-C(112)-C(113)	109.8(7)
C(158)-C(159)-C(160)	120.0	O(6)-C(114)-C(115)	112.4(6)
C(159)-C(160)-C(155)	120.0	O(6)-C(114)-C(112)	104.1(6)
O(7)-C(161)-N(4)	119.5(7)	C(115)-C(114)-C(112)	114.5(5)
O(7)-C(161)-C(162)	121.0(8)	C(116)-C(115)-C(120)	120.0
N(4)-C(161)-C(162)	119.3(7)	C(116)-C(115)-C(114)	121.4(4)
C(164)-C(162)-C(161)	107.4(6)	C(120)-C(115)-C(114)	118.6(4)
C(164)-C(162)-C(163)	112.5(7)	C(115)-C(116)-C(117)	120.0
C(161)-C(162)-C(163)	108.2(7)	C(116)-C(117)-C(118)	120.0
O(8)-C(164)-C(165)	114.1(6)	C(119)-C(118)-C(117)	120.0
O(8)-C(164)-C(162)	105.2(6)	C(119)-C(118)-C(121)	119.2(5)
C(165)-C(164)-C(162)	110.1(5)	C(117)-C(118)-C(121)	120.8(5)
C(166)-C(165)-C(170)	120.0	C(120)-C(119)-C(118)	120.0
C(166)-C(165)-C(164)	121.9(4)	C(119)-C(120)-C(115)	120.0
C(170)-C(165)-C(164)	118.1(4)		
C(165)-C(166)-C(167)	120.0		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2b**. The anisotropic displacement factor exponent takes the form: $-2 \sum h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(1)	48(1)	52(1)	45(1)	-9(1)	5(1)	-18(1)
S(2)	63(1)	47(1)	35(1)	-11(1)	2(1)	-6(1)
O(1)	49(3)	32(3)	42(3)	5(2)	-11(2)	2(2)
O(2)	116(6)	46(4)	64(4)	17(3)	55(4)	22(4)
N(1)	29(3)	32(3)	31(3)	-5(2)	-2(2)	4(2)
C(1)	54(4)	15(3)	39(4)	4(3)	7(3)	-5(3)
C(2)	35(3)	36(3)	30(3)	-1(3)	9(2)	2(3)
C(3)	32(3)	28(3)	28(3)	1(2)	4(2)	2(2)
C(4)	28(2)	30(3)	26(2)	3(2)	3(2)	10(2)
C(5)	25(2)	34(2)	23(2)	4(2)	6(2)	13(2)
C(6)	26(2)	42(3)	31(2)	0(2)	2(2)	6(2)
C(7)	44(3)	51(3)	30(3)	0(2)	-7(2)	15(3)
C(8)	47(3)	54(3)	22(2)	9(2)	-5(2)	25(3)
C(9)	34(3)	61(3)	29(2)	17(2)	13(2)	13(3)
C(10)	25(2)	43(3)	29(2)	12(2)	3(2)	6(2)

C(11)	41(3)	21(3)	40(3)	-5(3)	-1(3)	-1(3)
C(12)	52(3)	36(3)	39(3)	3(3)	0(3)	-1(3)
C(13)	56(4)	38(4)	45(4)	-3(3)	9(3)	-5(4)
C(14)	76(4)	69(4)	47(3)	14(3)	12(3)	13(4)
C(15)	93(4)	106(4)	57(4)	31(3)	18(3)	35(4)
C(16)	103(5)	148(6)	77(4)	37(4)	25(4)	41(5)
C(17)	115(5)	166(6)	83(5)	39(5)	28(4)	45(5)
C(18)	129(5)	155(6)	82(5)	41(5)	33(4)	48(5)
C(19)	126(5)	136(6)	75(4)	32(5)	38(4)	36(5)
C(20)	113(5)	121(5)	64(4)	26(4)	31(4)	35(5)
C(21)	171(10)	194(11)	111(8)	51(9)	41(8)	57(9)
S(3)	70(1)	52(1)	40(1)	-8(1)	7(1)	-8(1)
S(4)	40(1)	61(1)	44(1)	3(1)	0(1)	-11(1)
O(3)	36(3)	46(3)	61(4)	18(3)	-9(3)	-8(3)
O(4)	68(4)	43(3)	55(4)	8(3)	28(3)	8(3)
N(2)	35(3)	27(3)	39(3)	-1(2)	5(3)	-1(3)
C(51)	46(4)	16(3)	31(3)	9(2)	9(3)	-2(3)
C(52)	37(3)	48(4)	36(3)	2(3)	4(3)	10(3)
C(53)	37(3)	31(3)	35(3)	3(2)	-1(2)	7(2)
C(54)	30(2)	30(3)	37(3)	6(2)	0(2)	4(2)
C(55)	23(2)	29(2)	31(2)	15(2)	-3(2)	-5(2)
C(56)	37(3)	36(3)	28(2)	4(2)	-6(2)	0(2)
C(57)	54(3)	47(3)	31(2)	6(2)	-12(2)	17(3)
C(58)	54(3)	68(4)	29(2)	16(3)	4(2)	13(3)
C(59)	45(3)	69(4)	39(3)	25(3)	2(2)	-13(3)
C(60)	32(2)	55(3)	34(3)	18(2)	-11(2)	-23(2)
C(61)	37(3)	35(3)	40(3)	1(3)	-1(3)	2(3)
C(62)	43(3)	39(3)	42(3)	12(3)	2(3)	2(3)
C(63)	63(5)	59(5)	52(4)	9(4)	17(4)	1(4)
C(64)	50(3)	48(3)	43(3)	11(3)	3(3)	-3(3)
C(65)	60(3)	63(4)	58(3)	14(3)	7(3)	-6(3)
C(66)	63(4)	91(5)	81(4)	29(4)	10(4)	-12(4)
C(67)	85(4)	121(5)	100(5)	33(4)	5(4)	-3(4)
C(68)	100(4)	120(5)	100(5)	32(4)	16(4)	0(4)
C(69)	95(4)	100(5)	87(4)	19(4)	23(4)	0(4)
C(70)	85(4)	85(4)	70(4)	10(4)	17(4)	-1(4)
S(7)	73(1)	39(1)	42(1)	-18(1)	-8(1)	5(1)
S(8)	43(1)	69(2)	56(1)	-19(1)	-3(1)	11(1)
O(7)	79(4)	45(3)	35(3)	2(3)	8(3)	12(3)
O(8)	64(4)	37(3)	40(3)	4(2)	-10(3)	-16(3)
N(4)	50(4)	27(3)	21(3)	5(2)	2(3)	-2(3)
C(151)	36(4)	55(5)	47(4)	-15(4)	14(3)	-24(4)
C(152)	51(4)	65(4)	44(4)	-12(3)	-2(3)	0(4)
C(153)	56(3)	47(3)	34(3)	-6(3)	2(3)	0(3)
C(154)	92(5)	44(3)	48(3)	-4(3)	1(3)	-6(4)
C(155)	199(6)	58(4)	68(4)	6(3)	2(5)	-7(4)
C(156)	227(7)	79(5)	86(5)	11(4)	-9(6)	-4(6)
C(157)	247(8)	87(5)	98(5)	25(4)	-8(6)	-5(6)
C(158)	250(8)	90(5)	102(5)	16(5)	-3(6)	-8(6)
C(159)	243(8)	81(5)	98(5)	4(4)	9(6)	-16(6)
C(160)	222(7)	69(5)	81(5)	1(4)	16(6)	-14(5)
C(161)	48(3)	31(3)	33(3)	0(3)	6(3)	-5(3)
C(162)	48(3)	32(3)	29(3)	2(2)	1(2)	-10(3)
C(163)	48(4)	62(5)	61(5)	12(4)	-7(4)	-11(4)
C(164)	48(3)	22(2)	23(2)	0(2)	-1(2)	-5(2)
C(165)	38(2)	24(2)	24(2)	1(2)	5(2)	1(2)
C(166)	56(3)	27(3)	32(3)	3(2)	-1(2)	13(2)
C(167)	63(3)	29(3)	34(3)	4(2)	-1(2)	4(3)
C(168)	62(3)	24(2)	15(2)	-3(2)	2(2)	-16(2)
C(169)	47(3)	35(3)	22(2)	-5(2)	13(2)	-10(2)
C(170)	44(3)	23(2)	30(2)	-1(2)	5(2)	0(2)
C(171)	96(5)	46(4)	13(2)	7(3)	-7(3)	-28(4)
S(5)	53(1)	52(1)	38(1)	0(1)	2(1)	19(1)
S(6)	92(2)	53(1)	31(1)	-13(1)	-8(1)	31(1)
O(5)	65(4)	49(4)	39(3)	9(3)	14(3)	22(3)
O(6)	79(4)	58(4)	47(3)	-1(3)	-20(3)	-26(4)
N(3)	42(3)	31(3)	25(3)	2(2)	1(3)	6(3)
C(101)	39(4)	58(5)	34(4)	4(3)	11(3)	0(4)
C(102)	46(4)	51(4)	33(3)	3(3)	0(3)	-3(3)
C(103)	53(3)	42(3)	33(3)	-6(3)	4(3)	0(3)

C(104)	79(4)	51(4)	45(3)	-9(3)	8(3)	-5(3)
C(105)	123(5)	61(4)	55(3)	-10(3)	16(4)	-9(4)
C(106)	147(6)	77(5)	63(4)	-16(4)	27(4)	-9(5)
C(107)	173(6)	95(5)	80(5)	-14(4)	25(5)	-8(5)
C(108)	187(7)	106(5)	85(5)	-2(4)	16(5)	-6(6)
C(109)	181(7)	99(5)	83(5)	9(4)	10(5)	-3(5)
C(110)	155(6)	85(5)	70(4)	2(4)	12(5)	-6(5)
C(111)	40(3)	30(3)	37(3)	10(3)	1(3)	12(3)
C(112)	39(3)	28(3)	30(2)	-5(2)	-1(2)	6(2)
C(113)	52(4)	45(4)	48(4)	10(3)	-15(3)	-9(4)
C(114)	45(3)	24(3)	25(2)	-16(2)	0(2)	5(2)
C(115)	43(2)	12(2)	19(2)	-7(2)	3(2)	5(2)
C(116)	49(3)	11(2)	30(2)	-10(2)	7(2)	0(2)
C(117)	53(3)	19(2)	28(2)	-14(2)	11(2)	-11(2)
C(118)	68(3)	13(2)	23(2)	-8(2)	8(2)	-6(2)
C(119)	61(3)	19(2)	28(2)	-1(2)	3(2)	16(2)
C(120)	49(3)	17(2)	21(2)	-7(2)	7(2)	9(2)
C(121)	98(6)	31(3)	35(3)	20(3)	5(4)	-2(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **2b**.

	x	y	z	U(eq)
H(2A)	-4706	99	-2137	113
H(2B)	-3721	3967	-3445	40
H(2C)	-3350	2951	-3144	40
H(3A)	-5182	2611	-3241	35
H(4A)	-6554	3974	-3113	34
H(4B)	-5566	4779	-3111	34
H(6A)	-6922	2971	-3926	40
H(7A)	-7014	3085	-4813	50
H(8A)	-6017	4362	-5241	50
H(9A)	-4928	5524	-4782	50
H(10A)	-4837	5410	-3895	39
H(12A)	-5398	2337	-1584	51
H(13A)	-7151	2071	-1223	69
H(13B)	-7679	1956	-1774	69
H(13C)	-7203	3038	-1594	69
H(14A)	-6425	566	-2025	77
H(16A)	-4240	737	-1109	131
H(17A)	-4183	-78	-316	145
H(19A)	-7325	-1060	-515	134
H(20A)	-7381	-245	-1308	119
H(21A)	-5037	-1267	354	238
H(21B)	-5903	-2043	110	238
H(21C)	-6323	-1060	416	238
H(4C)	301	-9710	-2170	83
H(52A)	1458	-6313	-3357	48
H(52B)	1591	-7283	-2985	48
H(53A)	-239	-7438	-3201	41
H(54A)	-1465	-5987	-3283	39
H(54B)	-504	-5220	-3123	39
H(56A)	-1396	-6806	-4157	40
H(57A)	-788	-6637	-4997	53
H(58A)	621	-5472	-5194	60
H(59A)	1422	-4477	-4551	61
H(60A)	814	-4647	-3711	48
H(62A)	-611	-7582	-1585	49
H(63A)	-2268	-8030	-1210	87
H(63B)	-2839	-8098	-1754	87
H(63C)	-2421	-7017	-1544	87
H(64A)	-1471	-9405	-2045	56
H(66A)	759	-9102	-1167	94
H(67A)	919	-9954	-386	122
H(69A)	-2134	-11162	-574	113
H(70A)	-2295	-10310	-1355	96
H(8B)	3005	538	-2857	71

H(15A)	3889	-2910	-1575	64
H(15B)	4091	-1938	-1939	64
H(15C)	2262	-1748	-1756	55
H(15D)	933	-3116	-1790	74
H(15E)	1896	-3924	-1878	74
H(15F)	2844	-4651	-1188	157
H(15G)	3007	-4876	-308	173
H(15H)	2060	-3781	247	177
H(15I)	950	-2460	-77	169
H(16B)	787	-2235	-956	148
H(16C)	1887	-1608	-3399	43
H(16D)	-46	-2015	-3372	85
H(16E)	109	-1029	-3725	85
H(16F)	-324	-899	-3157	85
H(16G)	1266	383	-3000	37
H(16H)	580	1353	-3707	46
H(16I)	697	1883	-4556	50
H(16J)	3272	-56	-4774	42
H(17B)	3155	-586	-3926	39
H(17C)	2657	953	-5476	77
H(17D)	2187	2053	-5320	77
H(17E)	1363	1153	-5476	77
H(6B)	-2326	703	-2822	92
H(10B)	-1105	-2901	-1615	52
H(10C)	-922	-1924	-1978	52
H(10D)	-2742	-1752	-1806	51
H(10E)	-4045	-3151	-1812	70
H(10F)	-3056	-3945	-1874	70
H(10G)	-4262	-2200	-998	115
H(10H)	-4134	-2349	-113	139
H(10I)	-2943	-3564	248	151
H(10J)	-1881	-4631	-276	146
H(11A)	-2009	-4483	-1160	124
H(11B)	-2964	-1489	-3401	39
H(11C)	-4772	-2224	-3518	73
H(11D)	-4708	-1176	-3830	73
H(11E)	-5329	-1209	-3297	73
H(11F)	-4073	254	-2951	37
H(11G)	-1750	369	-3787	36
H(11H)	-1697	1326	-4541	40
H(11I)	-4969	1897	-4414	43
H(12B)	-5022	941	-3660	35
H(12C)	-4044	2607	-5108	82
H(12D)	-3067	1913	-5320	82
H(12E)	-2799	2919	-4997	82
H(71A)	377	-11152	226	240
H(71B)	-722	-11810	165	240
H(71C)	-772	-10685	404	240

Table 6. Torsion angles [°] for **2b**.

C(11)-N(1)-C(1)-S(2)	-14.6(13)
C(3)-N(1)-C(1)-S(2)	158.1(6)
C(11)-N(1)-C(1)-S(1)	167.6(6)
C(3)-N(1)-C(1)-S(1)	-19.8(8)
C(2)-S(1)-C(1)-N(1)	-2.7(6)
C(2)-S(1)-C(1)-S(2)	179.2(5)
C(1)-S(1)-C(2)-C(3)	22.8(6)
C(1)-N(1)-C(3)-C(2)	37.0(8)
C(11)-N(1)-C(3)-C(2)	-149.5(7)
C(1)-N(1)-C(3)-C(4)	-83.6(8)
C(11)-N(1)-C(3)-C(4)	89.9(7)
S(1)-C(2)-C(3)-N(1)	-35.3(6)
S(1)-C(2)-C(3)-C(4)	83.6(6)
N(1)-C(3)-C(4)-C(5)	-169.8(5)
C(2)-C(3)-C(4)-C(5)	73.8(7)
C(3)-C(4)-C(5)-C(6)	79.8(6)
C(3)-C(4)-C(5)-C(10)	-100.4(6)

C(10)-C(5)-C(6)-C(7)	0.0
C(4)-C(5)-C(6)-C(7)	179.8(4)
C(5)-C(6)-C(7)-C(8)	0.0
C(6)-C(7)-C(8)-C(9)	0.0
C(7)-C(8)-C(9)-C(10)	0.0
C(8)-C(9)-C(10)-C(5)	0.0
C(6)-C(5)-C(10)-C(9)	0.0
C(4)-C(5)-C(10)-C(9)	-179.8(4)
C(1)-N(1)-C(11)-O(1)	162.8(8)
C(3)-N(1)-C(11)-O(1)	-9.5(10)
C(1)-N(1)-C(11)-C(12)	-21.6(12)
C(3)-N(1)-C(11)-C(12)	166.0(7)
O(1)-C(11)-C(12)-C(14)	55.3(11)
N(1)-C(11)-C(12)-C(14)	-120.1(9)
O(1)-C(11)-C(12)-C(13)	-65.0(9)
N(1)-C(11)-C(12)-C(13)	119.6(8)
C(13)-C(12)-C(14)-O(2)	174.0(8)
C(11)-C(12)-C(14)-O(2)	56.9(10)
C(13)-C(12)-C(14)-C(15)	-64.2(12)
C(11)-C(12)-C(14)-C(15)	178.8(8)
O(2)-C(14)-C(15)-C(16)	40.5(11)
C(12)-C(14)-C(15)-C(16)	-78.7(10)
O(2)-C(14)-C(15)-C(20)	-139.2(8)
C(12)-C(14)-C(15)-C(20)	101.7(10)
C(20)-C(15)-C(16)-C(17)	0.0
C(14)-C(15)-C(16)-C(17)	-179.6(8)
C(15)-C(16)-C(17)-C(18)	0.0
C(16)-C(17)-C(18)-C(19)	0.0
C(16)-C(17)-C(18)-C(21)	174.7(11)
C(17)-C(18)-C(19)-C(20)	0.0
C(21)-C(18)-C(19)-C(20)	-175.1(10)
C(18)-C(19)-C(20)-C(15)	0.0
C(16)-C(15)-C(20)-C(19)	0.0
C(14)-C(15)-C(20)-C(19)	179.6(9)
C(61)-N(2)-C(51)-S(3)	-24.1(12)
C(53)-N(2)-C(51)-S(3)	160.5(6)
C(61)-N(2)-C(51)-S(4)	158.4(6)
C(53)-N(2)-C(51)-S(4)	-17.0(8)
C(52)-S(4)-C(51)-N(2)	-6.5(6)
C(52)-S(4)-C(51)-S(3)	175.8(5)
C(51)-S(4)-C(52)-C(53)	26.1(6)
C(51)-N(2)-C(53)-C(52)	36.7(9)
C(61)-N(2)-C(53)-C(52)	-139.2(7)
C(51)-N(2)-C(53)-C(54)	-84.1(8)
C(61)-N(2)-C(53)-C(54)	100.1(8)
S(4)-C(52)-C(53)-N(2)	-37.5(7)
S(4)-C(52)-C(53)-C(54)	81.6(7)
N(2)-C(53)-C(54)-C(55)	176.6(6)
C(52)-C(53)-C(54)-C(55)	60.7(8)
C(53)-C(54)-C(55)-C(56)	82.9(6)
C(53)-C(54)-C(55)-C(60)	-93.7(6)
C(60)-C(55)-C(56)-C(57)	0.0
C(54)-C(55)-C(56)-C(57)	-176.5(5)
C(55)-C(56)-C(57)-C(58)	0.0
C(56)-C(57)-C(58)-C(59)	0.0
C(57)-C(58)-C(59)-C(60)	0.0
C(58)-C(59)-C(60)-C(55)	0.0
C(56)-C(55)-C(60)-C(59)	0.0
C(54)-C(55)-C(60)-C(59)	176.6(5)
C(51)-N(2)-C(61)-O(3)	163.3(7)
C(53)-N(2)-C(61)-O(3)	-21.4(10)
C(51)-N(2)-C(61)-C(62)	-20.8(12)
C(53)-N(2)-C(61)-C(62)	154.4(7)
O(3)-C(61)-C(62)-C(63)	-60.8(11)
N(2)-C(61)-C(62)-C(63)	123.5(9)
O(3)-C(61)-C(62)-C(64)	62.8(10)
N(2)-C(61)-C(62)-C(64)	-113.0(8)
C(63)-C(62)-C(64)-O(4)	173.5(8)
C(61)-C(62)-C(64)-O(4)	51.1(9)
C(63)-C(62)-C(64)-C(65)	-62.6(10)

C(61)-C(62)-C(64)-C(65)	175.0(7)
O(4)-C(64)-C(65)-C(66)	41.3(10)
C(62)-C(64)-C(65)-C(66)	-79.5(8)
O(4)-C(64)-C(65)-C(70)	-139.0(6)
C(62)-C(64)-C(65)-C(70)	100.1(8)
C(70)-C(65)-C(66)-C(67)	0.0
C(64)-C(65)-C(66)-C(67)	179.6(8)
C(65)-C(66)-C(67)-C(68)	0.0
C(66)-C(67)-C(68)-C(69)	0.0
C(66)-C(67)-C(68)-C(71)	-176.1(11)
C(67)-C(68)-C(69)-C(70)	0.0
C(71)-C(68)-C(69)-C(70)	175.7(12)
C(68)-C(69)-C(70)-C(65)	0.0
C(66)-C(65)-C(70)-C(69)	0.0
C(64)-C(65)-C(70)-C(69)	-179.6(8)
C(161)-N(4)-C(151)-S(7)	-21.4(13)
C(153)-N(4)-C(151)-S(7)	159.1(7)
C(161)-N(4)-C(151)-S(8)	162.7(7)
C(153)-N(4)-C(151)-S(8)	-16.8(8)
C(152)-S(8)-C(151)-N(4)	-8.0(7)
C(152)-S(8)-C(151)-S(7)	175.7(6)
C(151)-S(8)-C(152)-C(153)	28.8(8)
C(161)-N(4)-C(153)-C(152)	-141.3(8)
C(151)-N(4)-C(153)-C(152)	38.2(9)
C(161)-N(4)-C(153)-C(154)	94.1(9)
C(151)-N(4)-C(153)-C(154)	-86.4(9)
S(8)-C(152)-C(153)-N(4)	-40.7(9)
S(8)-C(152)-C(153)-C(154)	79.5(8)
N(4)-C(153)-C(154)-C(155)	-176.6(8)
C(152)-C(153)-C(154)-C(155)	66.0(12)
C(153)-C(154)-C(155)-C(156)	-99.1(10)
C(153)-C(154)-C(155)-C(160)	80.2(12)
C(160)-C(155)-C(156)-C(157)	0.0
C(154)-C(155)-C(156)-C(157)	179.4(11)
C(155)-C(156)-C(157)-C(158)	0.0
C(156)-C(157)-C(158)-C(159)	0.0
C(157)-C(158)-C(159)-C(160)	0.0
C(158)-C(159)-C(160)-C(155)	0.0
C(156)-C(155)-C(160)-C(159)	0.0
C(154)-C(155)-C(160)-C(159)	-179.4(11)
C(151)-N(4)-C(161)-O(7)	169.5(8)
C(153)-N(4)-C(161)-O(7)	-11.1(11)
C(151)-N(4)-C(161)-C(162)	-16.1(12)
C(153)-N(4)-C(161)-C(162)	163.3(7)
O(7)-C(161)-C(162)-C(164)	63.4(10)
N(4)-C(161)-C(162)-C(164)	-110.9(8)
O(7)-C(161)-C(162)-C(163)	-58.4(11)
N(4)-C(161)-C(162)-C(163)	127.3(8)
C(161)-C(162)-C(164)-O(8)	51.8(8)
C(163)-C(162)-C(164)-O(8)	170.8(6)
C(161)-C(162)-C(164)-C(165)	175.2(6)
C(163)-C(162)-C(164)-C(165)	-65.8(8)
O(8)-C(164)-C(165)-C(166)	-132.1(5)
C(162)-C(164)-C(165)-C(166)	109.9(6)
O(8)-C(164)-C(165)-C(170)	49.5(7)
C(162)-C(164)-C(165)-C(170)	-68.6(6)
C(170)-C(165)-C(166)-C(167)	0.0
C(164)-C(165)-C(166)-C(167)	-178.4(5)
C(165)-C(166)-C(167)-C(168)	0.0
C(166)-C(167)-C(168)-C(169)	0.0
C(166)-C(167)-C(168)-C(171)	179.4(5)
C(167)-C(168)-C(169)-C(170)	0.0
C(171)-C(168)-C(169)-C(170)	-179.4(5)
C(168)-C(169)-C(170)-C(165)	0.0
C(166)-C(165)-C(170)-C(169)	0.0
C(164)-C(165)-C(170)-C(169)	178.5(5)
C(111)-N(3)-C(101)-S(6)	-23.9(14)
C(103)-N(3)-C(101)-S(6)	155.3(7)
C(111)-N(3)-C(101)-S(5)	165.1(6)
C(103)-N(3)-C(101)-S(5)	-15.6(9)

C(102)-S(5)-C(101)-N(3)	-8.6(7)
C(102)-S(5)-C(101)-S(6)	179.7(6)
C(101)-S(5)-C(102)-C(103)	28.4(7)
C(101)-N(3)-C(103)-C(102)	36.9(10)
C(111)-N(3)-C(103)-C(102)	-143.8(8)
C(101)-N(3)-C(103)-C(104)	-87.6(9)
C(111)-N(3)-C(103)-C(104)	91.7(8)
S(5)-C(102)-C(103)-N(3)	-38.8(8)
S(5)-C(102)-C(103)-C(104)	82.1(7)
N(3)-C(103)-C(104)-C(105)	-175.6(7)
C(102)-C(103)-C(104)-C(105)	67.5(10)
C(103)-C(104)-C(105)-C(106)	84.9(10)
C(103)-C(104)-C(105)-C(110)	-98.2(9)
C(110)-C(105)-C(106)-C(107)	0.0
C(104)-C(105)-C(106)-C(107)	176.8(9)
C(105)-C(106)-C(107)-C(108)	0.0
C(106)-C(107)-C(108)-C(109)	0.0
C(107)-C(108)-C(109)-C(110)	0.0
C(108)-C(109)-C(110)-C(105)	0.0
C(106)-C(105)-C(110)-C(109)	0.0
C(104)-C(105)-C(110)-C(109)	-176.9(9)
C(101)-N(3)-C(111)-O(5)	161.9(9)
C(103)-N(3)-C(111)-O(5)	-17.3(11)
C(101)-N(3)-C(111)-C(112)	-23.9(12)
C(103)-N(3)-C(111)-C(112)	156.9(7)
O(5)-C(111)-C(112)-C(114)	55.8(11)
N(3)-C(111)-C(112)-C(114)	-118.1(7)
O(5)-C(111)-C(112)-C(113)	-65.4(11)
N(3)-C(111)-C(112)-C(113)	120.7(8)
C(111)-C(112)-C(114)-O(6)	55.9(7)
C(113)-C(112)-C(114)-O(6)	175.6(6)
C(111)-C(112)-C(114)-C(115)	179.0(6)
C(113)-C(112)-C(114)-C(115)	-61.3(8)
O(6)-C(114)-C(115)-C(116)	32.7(6)
C(112)-C(114)-C(115)-C(116)	-85.8(6)
O(6)-C(114)-C(115)-C(120)	-145.3(5)
C(112)-C(114)-C(115)-C(120)	96.2(6)
C(120)-C(115)-C(116)-C(117)	0.0
C(114)-C(115)-C(116)-C(117)	-178.0(4)
C(115)-C(116)-C(117)-C(118)	0.0
C(116)-C(117)-C(118)-C(119)	0.0
C(116)-C(117)-C(118)-C(121)	179.2(5)
C(117)-C(118)-C(119)-C(120)	0.0
C(121)-C(118)-C(119)-C(120)	-179.2(5)
C(118)-C(119)-C(120)-C(115)	0.0
C(116)-C(115)-C(120)-C(119)	0.0
C(114)-C(115)-C(120)-C(119)	178.0(4)

X-ray crystallographic data for aldol adduct **2c**:

Table 1. Crystal data and structure refinement for **2c**.

Identification code	cwd73t	
Empirical formula	C ₂₁ H ₂₃ N O ₃ S ₂	
Formula weight	401.52	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 17.929(2) Å	= 90°.
	b = 6.0614(8) Å	= 90.845(3)°.
	c = 19.058(3) Å	= 90°.
Volume	2070.9(5) Å ³	
Z	4	
Density (calculated)	1.288 Mg/m ³	
Absorption coefficient	0.278 mm ⁻¹	
F(000)	848	
Crystal size	0.5 x 0.5 x 3.8 mm ³	
Theta range for data collection	1.07 to 28.34°.	
Index ranges	-23<=h<=21, -8<=k<=6, -25<=l<=24	
Reflections collected	13893	
Independent reflections	8279 [R(int) = 0.0775]	
Completeness to theta = 28.34°	96.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	8279 / 1 / 487	
Goodness-of-fit on F ²	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0849, wR2 = 0.1639	
R indices (all data)	R1 = 0.1327, wR2 = 0.1870	
Absolute structure parameter	0.09(12)	
Largest diff. peak and hole	0.696 and -0.338 e.Å ⁻³	

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for **2c**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(51)	3257(1)	1555(3)	2906(1)	41(1)
S(2)	11308(1)	9616(3)	2592(1)	39(1)
S(52)	4344(1)	4271(4)	2166(1)	46(1)
S(1)	12290(1)	7119(3)	1671(1)	41(1)
N(51)	3869(2)	4922(8)	3506(2)	25(1)
N(1)	11570(2)	10496(8)	1203(2)	26(1)
O(3)	6849(2)	11252(8)	2178(2)	42(1)
O(51)	4137(2)	7746(8)	4209(2)	41(1)
C(61)	4370(3)	6517(11)	3761(3)	32(1)
C(15)	8914(3)	11569(10)	1214(2)	27(1)
C(19)	7718(3)	13260(12)	1467(3)	40(2)
C(53)	3176(3)	4509(11)	3912(3)	29(1)
C(3)	12169(3)	10140(9)	691(3)	25(1)
C(12)	10296(3)	12130(11)	1426(3)	29(1)
C(1)	11656(3)	9248(10)	1811(3)	32(1)
C(11)	11016(3)	12075(11)	1036(3)	34(1)
C(5)	13391(3)	11710(10)	260(3)	31(1)
C(64)	5645(3)	6080(11)	4189(3)	35(2)
O(1)	11138(2)	13281(7)	549(2)	41(1)
C(4)	12836(3)	11662(11)	847(3)	34(1)
C(62)	5167(3)	6558(11)	3524(3)	31(1)
C(14)	9672(3)	11650(12)	887(3)	37(2)
C(56)	1788(3)	7171(11)	4661(3)	37(2)
C(54)	2561(3)	6086(11)	3628(3)	36(2)
C(51)	3875(3)	3733(11)	2881(3)	32(1)
C(18)	7522(3)	11470(11)	1851(3)	33(1)
C(65)	6472(3)	5996(10)	4023(3)	29(1)
O(2)	9868(2)	9601(10)	580(2)	52(1)
O(52)	5377(2)	4010(9)	4427(2)	49(1)
C(16)	8689(3)	9768(12)	1606(3)	37(2)

C(60)	1268(3)	4701(12)	3825(3)	42(2)
C(70)	6916(3)	7844(12)	4142(3)	39(2)
C(52)	3000(3)	2113(11)	3800(3)	38(2)
C(55)	1863(3)	5999(10)	4057(3)	31(1)
O(53)	8691(2)	5925(10)	3401(2)	60(2)
C(20)	8415(3)	13298(11)	1155(3)	37(2)
C(66)	6801(3)	4198(11)	3707(3)	38(2)
C(69)	7674(3)	7840(12)	3954(3)	42(2)
C(68)	7969(3)	6085(13)	3637(3)	40(2)
C(67)	7537(3)	4229(13)	3517(3)	46(2)
C(59)	610(3)	4653(13)	4209(3)	46(2)
C(57)	1129(3)	7099(13)	5040(4)	50(2)
C(58)	549(3)	5856(13)	4810(3)	45(2)
C(17)	8007(3)	9693(11)	1931(3)	36(1)
C(6)	14059(4)	10634(12)	314(3)	47(2)
C(10)	13228(3)	12841(11)	-356(3)	40(2)
C(13)	10203(3)	14323(13)	1793(3)	48(2)
C(2)	12360(4)	7712(11)	742(3)	38(2)
C(9)	13721(4)	12870(12)	-889(3)	46(2)
C(71)	9146(4)	7816(16)	3476(4)	67(3)
C(63)	5342(3)	8759(12)	3185(3)	46(2)
C(7)	14567(4)	10679(13)	-232(4)	56(2)
C(8)	14394(4)	11804(13)	-841(3)	48(2)
C(21)	6291(4)	12873(14)	2024(4)	60(2)

Table 3. Bond lengths [Å] and angles [°] for **2c**.

S(51)-C(51)	1.724(6)	C(65)-C(66)	1.381(8)
S(51)-C(52)	1.803(6)	C(65)-C(70)	1.391(8)
S(2)-C(1)	1.639(6)	C(16)-C(17)	1.379(7)
S(52)-C(51)	1.645(5)	C(60)-C(55)	1.392(8)
S(1)-C(1)	1.742(6)	C(60)-C(59)	1.399(8)
S(1)-C(2)	1.812(6)	C(70)-C(69)	1.410(8)
N(51)-C(51)	1.392(7)	O(53)-C(68)	1.381(7)
N(51)-C(61)	1.403(7)	O(53)-C(71)	1.413(10)
N(51)-C(53)	1.494(6)	C(66)-C(67)	1.374(8)
N(1)-C(1)	1.390(7)	C(69)-C(68)	1.336(9)
N(1)-C(11)	1.413(7)	C(68)-C(67)	1.383(10)
N(1)-C(3)	1.478(7)	C(59)-C(58)	1.362(9)
O(3)-C(18)	1.373(6)	C(57)-C(58)	1.352(9)
O(3)-C(21)	1.429(8)	C(6)-C(7)	1.393(9)
O(51)-C(61)	1.212(6)	C(10)-C(9)	1.357(8)
C(61)-C(62)	1.504(7)	C(9)-C(8)	1.371(9)
C(15)-C(20)	1.382(8)	C(7)-C(8)	1.377(10)
C(15)-C(16)	1.386(8)	C(51)-S(51)-C(52)	93.2(3)
C(15)-C(14)	1.504(7)	C(1)-S(1)-C(2)	93.3(3)
C(19)-C(18)	1.358(9)	C(51)-N(51)-C(61)	129.8(4)
C(19)-C(20)	1.390(8)	C(51)-N(51)-C(53)	111.9(4)
C(53)-C(52)	1.501(9)	C(61)-N(51)-C(53)	118.0(4)
C(53)-C(54)	1.551(8)	C(1)-N(1)-C(11)	128.8(5)
C(3)-C(2)	1.514(8)	C(1)-N(1)-C(3)	113.5(4)
C(3)-C(4)	1.536(7)	C(11)-N(1)-C(3)	117.7(4)
C(12)-C(11)	1.499(8)	C(18)-O(3)-C(21)	117.3(5)
C(12)-C(13)	1.512(10)	O(51)-C(61)-N(51)	116.2(5)
C(12)-C(14)	1.535(7)	O(51)-C(61)-C(62)	122.7(5)
C(11)-O(1)	1.204(7)	N(51)-C(61)-C(62)	120.9(5)
C(5)-C(6)	1.366(8)	C(20)-C(15)-C(16)	116.6(5)
C(5)-C(10)	1.386(8)	C(20)-C(15)-C(14)	122.0(6)
C(5)-C(4)	1.509(7)	C(16)-C(15)-C(14)	121.4(5)
C(64)-O(52)	1.420(8)	C(18)-C(19)-C(20)	119.0(6)
C(64)-C(65)	1.522(7)	N(51)-C(53)-C(52)	105.3(4)
C(64)-C(62)	1.548(7)	N(51)-C(53)-C(54)	108.0(4)
C(62)-C(63)	1.517(9)	C(52)-C(53)-C(54)	113.5(5)
C(14)-O(2)	1.419(8)	N(1)-C(3)-C(2)	105.3(4)
C(56)-C(55)	1.360(8)	N(1)-C(3)-C(4)	110.8(4)
C(56)-C(57)	1.393(8)	C(2)-C(3)-C(4)	113.4(5)
C(54)-C(55)	1.506(8)	C(11)-C(12)-C(13)	110.5(5)
C(18)-C(17)	1.392(8)	C(11)-C(12)-C(14)	106.7(4)
		C(13)-C(12)-C(14)	113.1(5)
		N(1)-C(1)-S(2)	130.1(5)

N(1)-C(1)-S(1)	109.9(4)	C(70)-C(65)-C(64)	119.7(6)
S(2)-C(1)-S(1)	119.9(3)	C(17)-C(16)-C(15)	122.4(6)
O(1)-C(11)-N(1)	116.7(5)	C(55)-C(60)-C(59)	119.5(6)
O(1)-C(11)-C(12)	122.5(5)	C(65)-C(70)-C(69)	120.6(6)
N(1)-C(11)-C(12)	120.7(5)	C(53)-C(52)-S(51)	105.1(4)
C(6)-C(5)-C(10)	118.3(5)	C(56)-C(55)-C(60)	118.6(5)
C(6)-C(5)-C(4)	121.4(6)	C(56)-C(55)-C(54)	122.3(5)
C(10)-C(5)-C(4)	120.3(5)	C(60)-C(55)-C(54)	119.0(6)
O(52)-C(64)-C(65)	111.9(5)	C(68)-O(53)-C(71)	116.9(6)
O(52)-C(64)-C(62)	103.9(5)	C(15)-C(20)-C(19)	122.5(6)
C(65)-C(64)-C(62)	111.5(4)	C(67)-C(66)-C(65)	121.5(6)
C(5)-C(4)-C(3)	112.8(4)	C(68)-C(69)-C(70)	120.4(6)
C(61)-C(62)-C(63)	110.2(5)	C(69)-C(68)-O(53)	125.6(6)
C(61)-C(62)-C(64)	105.5(4)	C(69)-C(68)-C(67)	119.9(6)
C(63)-C(62)-C(64)	113.4(5)	O(53)-C(68)-C(67)	114.5(6)
O(2)-C(14)-C(15)	111.9(5)	C(66)-C(67)-C(68)	120.3(7)
O(2)-C(14)-C(12)	105.0(5)	C(58)-C(59)-C(60)	120.5(6)
C(15)-C(14)-C(12)	112.5(4)	C(58)-C(57)-C(56)	120.2(6)
C(55)-C(56)-C(57)	121.1(6)	C(57)-C(58)-C(59)	120.0(6)
C(55)-C(54)-C(53)	112.4(5)	C(16)-C(17)-C(18)	118.8(6)
N(51)-C(51)-S(52)	128.1(5)	C(5)-C(6)-C(7)	121.0(6)
N(51)-C(51)-S(51)	111.1(4)	C(9)-C(10)-C(5)	120.6(6)
S(52)-C(51)-S(51)	120.8(4)	C(3)-C(2)-S(1)	103.7(4)
C(19)-C(18)-O(3)	123.9(6)	C(10)-C(9)-C(8)	121.8(6)
C(19)-C(18)-C(17)	120.7(5)	C(8)-C(7)-C(6)	119.9(6)
O(3)-C(18)-C(17)	115.3(6)	C(9)-C(8)-C(7)	118.4(6)
C(66)-C(65)-C(70)	117.3(5)		
C(66)-C(65)-C(64)	122.7(5)		

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

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(51)	43(1)	38(1)	42(1)	-14(1)	6(1)	-11(1)
S(2)	43(1)	48(1)	25(1)	4(1)	-4(1)	6(1)
S(52)	33(1)	79(1)	25(1)	-6(1)	5(1)	-13(1)
S(1)	59(1)	28(1)	37(1)	4(1)	0(1)	12(1)
N(51)	20(2)	30(3)	25(2)	-1(2)	-4(2)	1(2)
N(1)	24(2)	30(3)	23(2)	3(2)	-2(2)	-2(2)
O(3)	32(2)	53(3)	42(2)	3(2)	2(2)	10(2)
O(51)	41(2)	46(3)	36(2)	-13(2)	-3(2)	0(2)
C(61)	35(3)	39(4)	20(3)	-7(3)	-4(2)	3(3)
C(15)	26(3)	38(4)	18(2)	0(3)	-8(2)	-1(3)
C(19)	27(3)	46(5)	46(4)	8(3)	-4(3)	5(3)
C(53)	29(3)	36(4)	22(3)	-7(3)	8(2)	-6(3)
C(3)	29(3)	20(3)	26(3)	1(2)	-6(2)	-3(2)
C(12)	24(3)	34(4)	28(3)	9(3)	-3(2)	1(3)
C(1)	37(3)	23(3)	35(3)	-3(3)	-15(2)	-7(3)
C(11)	30(3)	35(4)	36(3)	1(3)	-16(2)	-1(3)
C(5)	20(3)	27(4)	46(3)	-12(3)	2(2)	1(2)
C(64)	25(3)	44(4)	36(3)	-1(3)	-3(2)	-1(3)
O(1)	27(2)	51(3)	44(2)	23(2)	-4(2)	5(2)
C(4)	37(3)	33(4)	30(3)	-8(3)	-4(2)	-3(3)
C(62)	29(3)	38(4)	25(3)	-4(3)	0(2)	1(3)
C(14)	27(3)	56(5)	28(3)	0(3)	-1(2)	-2(3)
C(56)	26(3)	41(4)	45(4)	2(3)	-2(3)	1(3)
C(54)	40(3)	33(4)	36(3)	9(3)	2(3)	1(3)
C(51)	16(3)	48(4)	30(3)	7(3)	-1(2)	-1(3)
C(18)	27(3)	44(4)	27(3)	-1(3)	3(2)	-1(3)
C(65)	31(3)	33(4)	22(3)	1(3)	-5(2)	1(3)
O(2)	30(2)	78(4)	46(2)	-30(3)	-1(2)	-1(3)
O(52)	41(2)	62(3)	43(2)	27(2)	-12(2)	-18(2)
C(16)	29(3)	38(4)	44(3)	2(3)	-8(3)	13(3)
C(60)	43(3)	42(4)	39(3)	-2(3)	2(3)	-7(3)
C(70)	34(3)	47(4)	35(3)	-6(3)	-9(3)	-2(3)

C(52)	38(3)	33(4)	43(3)	5(3)	6(3)	2(3)
C(55)	28(3)	36(4)	31(3)	9(3)	-1(2)	3(3)
O(53)	29(2)	90(4)	59(3)	-12(3)	3(2)	6(3)
C(20)	28(3)	49(4)	32(3)	15(3)	-5(3)	3(3)
C(66)	34(3)	33(4)	47(3)	-5(3)	-10(3)	-1(3)
C(69)	31(3)	48(5)	47(4)	-7(3)	-4(3)	-11(3)
C(68)	30(3)	58(5)	32(3)	-2(3)	-8(3)	5(3)
C(67)	44(4)	47(5)	47(4)	-7(4)	-3(3)	11(4)
C(59)	26(3)	58(5)	53(4)	6(4)	2(3)	-10(3)
C(57)	38(4)	57(5)	54(4)	-9(4)	3(3)	9(4)
C(58)	28(3)	63(5)	44(4)	-5(4)	17(3)	-2(3)
C(17)	37(3)	30(4)	41(3)	12(3)	1(3)	0(3)
C(6)	44(4)	49(5)	47(4)	11(3)	7(3)	9(3)
C(10)	34(3)	42(4)	42(4)	1(3)	-3(3)	5(3)
C(13)	35(3)	52(5)	57(4)	-9(4)	-2(3)	-7(4)
C(2)	42(4)	37(4)	34(3)	-9(3)	7(3)	-2(3)
C(9)	57(4)	47(5)	35(3)	9(3)	-2(3)	-7(4)
C(71)	28(4)	107(8)	66(5)	6(5)	-12(3)	-13(4)
C(63)	33(3)	46(5)	59(4)	24(3)	1(3)	1(3)
C(7)	42(4)	49(5)	78(5)	1(4)	4(4)	14(4)
C(8)	50(4)	50(5)	45(4)	-13(4)	13(3)	-8(4)
C(21)	48(4)	69(6)	63(5)	1(4)	3(4)	20(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **2c**.

	x	y	z	U(eq)
H(19A)	7388	14455	1412	48
H(53A)	3271	4797	4417	35
H(3A)	11973	10460	213	30
H(12A)	10302	10942	1782	34
H(64A)	5554	7226	4548	42
H(4A)	12653	13162	929	40
H(4B)	13089	11162	1278	40
H(62A)	5243	5363	3178	37
H(14A)	9673	12809	521	45
H(56A)	2186	8045	4825	45
H(54A)	2753	7599	3629	43
H(54B)	2437	5687	3142	43
H(2A)	9563	9292	263	77
H(52A)	5544	3765	4828	73
H(16A)	9012	8554	1651	45
H(60A)	1309	3863	3412	50
H(70A)	6710	9107	4349	47
H(52B)	2467	1831	3867	46
H(52C)	3288	1189	4127	46
H(20A)	8551	14548	895	44
H(66A)	6515	2924	3621	45
H(69A)	7974	9078	4053	50
H(67A)	7748	2983	3303	55
H(59A)	206	3785	4052	55
H(57A)	1088	7917	5456	60
H(58A)	104	5822	5064	54
H(17A)	7873	8464	2202	43
H(6A)	14178	9851	726	56
H(10A)	12772	13595	-404	47
H(13A)	10602	14518	2136	72
H(13B)	9726	14353	2027	72
H(13C)	10220	15505	1450	72
H(2B)	12865	7428	576	45
H(2C)	12005	6819	468	45
H(9A)	13597	13641	-1303	55
H(71A)	9636	7512	3290	100
H(71B)	9193	8198	3969	100
H(71C)	8921	9037	3221	100
H(63A)	5036	8941	2765	69
H(63B)	5864	8804	3063	69

H(63C)	5236	9941	3512	69
H(7A)	15026	9943	-185	68
H(8A)	14730	11840	-1215	58
H(21A)	5843	12538	2283	90
H(21B)	6177	12863	1525	90
H(21C)	6474	14320	2160	90

Table 6. Torsion angles [°] for **2c**.

C(51)-N(51)-C(61)-O(51)	159.4(5)
C(53)-N(51)-C(61)-O(51)	-14.4(8)
C(51)-N(51)-C(61)-C(62)	-25.0(9)
C(53)-N(51)-C(61)-C(62)	161.2(5)
C(51)-N(51)-C(53)-C(52)	36.8(6)
C(61)-N(51)-C(53)-C(52)	-148.4(5)
C(51)-N(51)-C(53)-C(54)	-84.8(6)
C(61)-N(51)-C(53)-C(54)	90.0(6)
C(1)-N(1)-C(3)-C(2)	36.6(6)
C(11)-N(1)-C(3)-C(2)	-145.9(5)
C(1)-N(1)-C(3)-C(4)	-86.4(6)
C(11)-N(1)-C(3)-C(4)	91.1(6)
C(11)-N(1)-C(1)-S(2)	-18.4(9)
C(3)-N(1)-C(1)-S(2)	158.8(4)
C(11)-N(1)-C(1)-S(1)	166.2(5)
C(3)-N(1)-C(1)-S(1)	-16.7(5)
C(2)-S(1)-C(1)-N(1)	-6.5(4)
C(2)-S(1)-C(1)-S(2)	177.5(4)
C(1)-N(1)-C(11)-O(1)	164.6(5)
C(3)-N(1)-C(11)-O(1)	-12.4(8)
C(1)-N(1)-C(11)-C(12)	-18.7(9)
C(3)-N(1)-C(11)-C(12)	164.3(5)
C(13)-C(12)-C(11)-O(1)	-64.2(7)
C(14)-C(12)-C(11)-O(1)	59.1(8)
C(13)-C(12)-C(11)-N(1)	119.3(6)
C(14)-C(12)-C(11)-N(1)	-117.4(6)
C(6)-C(5)-C(4)-C(3)	-105.1(7)
C(10)-C(5)-C(4)-C(3)	74.2(8)
N(1)-C(3)-C(4)-C(5)	-168.1(5)
C(2)-C(3)-C(4)-C(5)	73.7(6)
O(51)-C(61)-C(62)-C(63)	-63.5(7)
N(51)-C(61)-C(62)-C(63)	121.2(6)
O(51)-C(61)-C(62)-C(64)	59.3(8)
N(51)-C(61)-C(62)-C(64)	-116.0(6)
O(52)-C(64)-C(62)-C(61)	57.5(6)
C(65)-C(64)-C(62)-C(61)	178.2(5)
O(52)-C(64)-C(62)-C(63)	178.2(5)
C(65)-C(64)-C(62)-C(63)	-61.1(7)
C(20)-C(15)-C(14)-O(2)	-139.3(5)
C(16)-C(15)-C(14)-O(2)	42.3(7)
C(20)-C(15)-C(14)-C(12)	102.7(6)
C(16)-C(15)-C(14)-C(12)	-75.7(7)
C(11)-C(12)-C(14)-O(2)	55.9(6)
C(13)-C(12)-C(14)-O(2)	177.6(5)
C(11)-C(12)-C(14)-C(15)	177.8(6)
C(13)-C(12)-C(14)-C(15)	-60.4(7)
N(51)-C(53)-C(54)-C(55)	-173.3(5)
C(52)-C(53)-C(54)-C(55)	70.4(6)
C(61)-N(51)-C(51)-S(52)	-17.2(9)
C(53)-N(51)-C(51)-S(52)	156.8(5)
C(61)-N(51)-C(51)-S(51)	166.4(5)
C(53)-N(51)-C(51)-S(51)	-19.5(6)
C(52)-S(51)-C(51)-N(51)	-2.8(4)
C(52)-S(51)-C(51)-S(52)	-179.4(4)
C(20)-C(19)-C(18)-O(3)	-179.7(5)
C(20)-C(19)-C(18)-C(17)	-0.2(9)
C(21)-O(3)-C(18)-C(19)	8.7(8)
C(21)-O(3)-C(18)-C(17)	-170.8(6)
O(52)-C(64)-C(65)-C(66)	38.5(7)
C(62)-C(64)-C(65)-C(66)	-77.3(7)
O(52)-C(64)-C(65)-C(70)	-146.2(5)
C(62)-C(64)-C(65)-C(70)	97.9(6)
C(20)-C(15)-C(16)-C(17)	-0.8(8)
C(14)-C(15)-C(16)-C(17)	177.6(5)
C(66)-C(65)-C(70)-C(69)	-0.9(8)
C(64)-C(65)-C(70)-C(69)	-176.4(5)
N(51)-C(53)-C(52)-S(51)	-36.1(5)
C(54)-C(53)-C(52)-S(51)	81.8(5)

C(51)-S(51)-C(52)-C(53)	23.3(4)
C(57)-C(56)-C(55)-C(60)	-0.5(10)
C(57)-C(56)-C(55)-C(54)	178.6(6)
C(59)-C(60)-C(55)-C(56)	0.7(9)
C(59)-C(60)-C(55)-C(54)	-178.3(6)
C(53)-C(54)-C(55)-C(56)	82.8(7)
C(53)-C(54)-C(55)-C(60)	-98.2(6)
C(16)-C(15)-C(20)-C(19)	-0.3(9)
C(14)-C(15)-C(20)-C(19)	-178.7(5)
C(18)-C(19)-C(20)-C(15)	0.8(9)
C(70)-C(65)-C(66)-C(67)	-0.2(8)
C(64)-C(65)-C(66)-C(67)	175.2(5)
C(65)-C(70)-C(69)-C(68)	2.3(9)
C(70)-C(69)-C(68)-O(53)	176.9(6)
C(70)-C(69)-C(68)-C(67)	-2.5(9)
C(71)-O(53)-C(68)-C(69)	-2.8(9)
C(71)-O(53)-C(68)-C(67)	176.7(6)
C(65)-C(66)-C(67)-C(68)	0.0(9)
C(69)-C(68)-C(67)-C(66)	1.4(9)
O(53)-C(68)-C(67)-C(66)	-178.1(5)
C(55)-C(60)-C(59)-C(58)	-0.3(10)
C(55)-C(56)-C(57)-C(58)	-0.2(11)
C(56)-C(57)-C(58)-C(59)	0.6(11)
C(60)-C(59)-C(58)-C(57)	-0.3(11)
C(15)-C(16)-C(17)-C(18)	1.4(9)
C(19)-C(18)-C(17)-C(16)	-0.8(9)
O(3)-C(18)-C(17)-C(16)	178.7(5)
C(10)-C(5)-C(6)-C(7)	0.4(10)
C(4)-C(5)-C(6)-C(7)	179.7(6)
C(6)-C(5)-C(10)-C(9)	0.1(10)
C(4)-C(5)-C(10)-C(9)	-179.3(6)
N(1)-C(3)-C(2)-S(1)	-37.8(5)
C(4)-C(3)-C(2)-S(1)	83.6(5)
C(1)-S(1)-C(2)-C(3)	26.1(4)
C(5)-C(10)-C(9)-C(8)	-0.3(11)
C(5)-C(6)-C(7)-C(8)	-0.6(11)
C(10)-C(9)-C(8)-C(7)	0.1(11)
C(6)-C(7)-C(8)-C(9)	0.4(11)

X-ray crystallographic data for aldol adduct **2g**: Table 1. Crystal data and structure refinement for **2g**.

Identification code	cwd71t
Empirical formula	C17 H21 N O2 S2
Formula weight	335.47
Temperature	213(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic

Space group	P2(1)	
Unit cell dimensions	a = 11.9782(15) Å	= 90°.
	b = 6.0259(7) Å	= 91.015(2)°.
	c = 12.4618(15) Å	= 90°.
Volume	899.34(19) Å ³	
Z	2	
Density (calculated)	1.239 Mg/m ³	
Absorption coefficient	0.302 mm ⁻¹	
F(000)	356	
Crystal size	0.8 x 2.0 x 2.3 mm ³	
Theta range for data collection	1.63 to 27.66°.	
Index ranges	-15<=h<=15, -7<=k<=3, -13<=l<=15	
Reflections collected	5692	
Independent reflections	2752 [R(int) = 0.0421]	
Completeness to theta = 27.66°	90.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2752 / 1 / 199	
Goodness-of-fit on F ²	0.995	
Final R indices [I>2sigma(I)]	R1 = 0.0342, wR2 = 0.0808	
R indices (all data)	R1 = 0.0415, wR2 = 0.0846	
Absolute structure parameter	0.03(8)	
Largest diff. peak and hole	0.229 and -0.201 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2g**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	3066(1)	910(1)	3953(1)	46(1)
N(1)	4930(2)	1598(3)	2770(2)	29(1)
S(2)	5077(1)	-1857(1)	3955(1)	43(1)
O(1)	5274(1)	4450(3)	1665(1)	42(1)
O(2)	3968(2)	709(4)	388(1)	52(1)
C(3)	6145(2)	1117(4)	2821(2)	31(1)
C(11)	4573(2)	3227(5)	2038(2)	32(1)
C(1)	4308(2)	389(5)	3482(2)	32(1)
C(12)	3368(2)	3329(5)	1657(2)	32(1)
C(2)	6227(2)	-1333(4)	3063(2)	39(1)
C(13)	2875(2)	5579(5)	1888(2)	46(1)
C(10)	8442(2)	3941(5)	2764(2)	49(1)
C(14)	3380(2)	2762(4)	452(2)	35(1)
C(5)	7981(2)	2609(4)	3534(2)	36(1)
C(4)	6729(2)	2561(4)	3675(2)	35(1)
C(15)	2206(2)	2578(5)	-19(2)	40(1)
C(6)	8681(2)	1339(6)	4167(2)	56(1)
C(7)	9826(2)	1373(8)	4009(3)	76(1)
C(17)	1828(2)	4077(6)	-690(2)	56(1)
C(9)	9581(2)	4025(7)	2619(3)	67(1)
C(16)	1526(3)	638(7)	328(3)	68(1)
C(8)	10275(2)	2728(8)	3245(3)	73(1)

Table 3. Bond lengths [Å] and angles [°] for **2g**.

S(1)-C(1)	1.639(2)	C(12)-C(13)	1.508(4)
N(1)-C(1)	1.378(3)	C(12)-C(14)	1.541(3)
N(1)-C(11)	1.401(3)	C(10)-C(5)	1.374(4)
N(1)-C(3)	1.483(3)	C(10)-C(9)	1.380(4)
S(2)-C(1)	1.734(3)	C(14)-C(15)	1.519(3)
S(2)-C(2)	1.812(2)	C(5)-C(6)	1.374(4)
O(1)-C(11)	1.216(3)	C(5)-C(4)	1.513(3)
O(2)-C(14)	1.426(3)	C(15)-C(17)	1.307(4)
C(3)-C(2)	1.510(4)	C(15)-C(16)	1.493(4)
C(3)-C(4)	1.535(3)	C(6)-C(7)	1.389(4)
C(11)-C(12)	1.513(3)	C(7)-C(8)	1.372(5)

C(9)-C(8)	1.374(5)	C(11)-C(12)-C(14)	105.73(18)
C(1)-N(1)-C(11)	128.81(19)	C(3)-C(2)-S(2)	104.22(17)
C(1)-N(1)-C(3)	114.21(18)	C(5)-C(10)-C(9)	121.5(3)
C(11)-N(1)-C(3)	116.96(18)	O(2)-C(14)-C(15)	111.7(2)
C(1)-S(2)-C(2)	93.48(12)	O(2)-C(14)-C(12)	105.10(19)
N(1)-C(3)-C(2)	105.1(2)	C(15)-C(14)-C(12)	111.61(19)
N(1)-C(3)-C(4)	110.72(19)	C(6)-C(5)-C(10)	118.5(2)
C(2)-C(3)-C(4)	112.9(2)	C(6)-C(5)-C(4)	121.3(2)
O(1)-C(11)-N(1)	117.9(2)	C(10)-C(5)-C(4)	120.2(2)
O(1)-C(11)-C(12)	121.1(2)	C(5)-C(4)-C(3)	111.78(19)
N(1)-C(11)-C(12)	120.8(2)	C(17)-C(15)-C(16)	122.8(3)
N(1)-C(1)-S(1)	129.4(2)	C(17)-C(15)-C(14)	120.3(3)
N(1)-C(1)-S(2)	109.95(16)	C(16)-C(15)-C(14)	116.8(2)
S(1)-C(1)-S(2)	120.51(15)	C(5)-C(6)-C(7)	120.3(3)
C(13)-C(12)-C(11)	110.5(2)	C(8)-C(7)-C(6)	120.5(3)
C(13)-C(12)-C(14)	113.3(2)	C(8)-C(9)-C(10)	119.6(3)
		C(7)-C(8)-C(9)	119.4(3)

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

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(1)	29(1)	72(1)	36(1)	18(1)	8(1)	7(1)
N(1)	24(1)	36(1)	27(1)	2(1)	1(1)	-1(1)
S(2)	38(1)	41(1)	51(1)	14(1)	6(1)	4(1)
O(1)	39(1)	49(1)	39(1)	12(1)	3(1)	-10(1)
O(2)	56(1)	64(1)	36(1)	-9(1)	1(1)	23(1)
C(3)	26(1)	36(1)	31(1)	-3(1)	4(1)	2(1)
C(11)	34(1)	36(1)	25(1)	1(1)	1(1)	-2(1)
C(1)	30(1)	41(2)	25(1)	1(1)	-3(1)	-1(1)
C(12)	30(1)	40(1)	25(1)	3(1)	0(1)	-1(1)
C(2)	32(1)	40(2)	46(2)	-5(1)	6(1)	2(1)
C(13)	46(2)	47(2)	44(2)	-4(1)	-3(1)	7(1)
C(10)	32(1)	59(2)	56(2)	8(2)	-3(1)	-2(1)
C(14)	34(1)	43(2)	26(1)	4(1)	2(1)	4(1)
C(5)	28(1)	43(2)	37(1)	-6(1)	-2(1)	1(1)
C(4)	29(1)	38(2)	38(1)	-8(1)	1(1)	1(1)
C(15)	40(1)	54(2)	27(1)	-2(1)	-1(1)	5(1)
C(6)	40(2)	72(2)	55(2)	17(2)	-2(1)	5(2)
C(7)	40(2)	106(3)	81(2)	21(2)	-16(2)	18(2)
C(17)	43(2)	84(2)	42(2)	12(2)	-5(1)	9(2)
C(9)	37(2)	95(3)	68(2)	21(2)	2(1)	-7(2)
C(16)	62(2)	77(3)	63(2)	3(2)	-18(2)	-24(2)
C(8)	30(1)	114(4)	76(2)	13(2)	3(1)	4(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2g**.

	x	y	z	U(eq)
H(2A)	4154	487	-242	78
H(3A)	6474	1416	2112	37
H(12A)	2935	2182	2038	38
H(2B)	6943	-1689	3415	47
H(2C)	6149	-2214	2405	47
H(13A)	2879	5830	2657	69
H(13B)	2114	5638	1611	69
H(13C)	3316	6717	1544	69
H(10A)	7971	4816	2326	59
H(14A)	3796	3924	64	41
H(4A)	6436	4078	3629	42

H(4B)	6563	1981	4389	42
H(6A)	8384	443	4709	67
H(7A)	10296	460	4429	91
H(17A)	1099	3961	-977	68
H(17B)	2286	5272	-887	68
H(9A)	9880	4964	2094	80
H(16A)	798	691	-25	102
H(16B)	1436	688	1099	102
H(16C)	1902	-727	134	102
H(8A)	11052	2770	3150	88

Table 6. Torsion angles [°] for **2g**.

C(1)-N(1)-C(3)-C(2)	35.3(3)
C(11)-N(1)-C(3)-C(2)	-146.2(2)
C(1)-N(1)-C(3)-C(4)	-86.9(3)
C(11)-N(1)-C(3)-C(4)	91.6(2)
C(1)-N(1)-C(11)-O(1)	163.1(2)
C(3)-N(1)-C(11)-O(1)	-15.2(3)
C(1)-N(1)-C(11)-C(12)	-21.9(4)
C(3)-N(1)-C(11)-C(12)	159.8(2)
C(11)-N(1)-C(1)-S(1)	-19.5(4)
C(3)-N(1)-C(1)-S(1)	158.79(19)
C(11)-N(1)-C(1)-S(2)	165.1(2)
C(3)-N(1)-C(1)-S(2)	-16.6(3)
C(2)-S(2)-C(1)-N(1)	-5.72(19)
C(2)-S(2)-C(1)-S(1)	178.45(16)
O(1)-C(11)-C(12)-C(13)	-61.9(3)
N(1)-C(11)-C(12)-C(13)	123.3(3)
O(1)-C(11)-C(12)-C(14)	61.2(3)
N(1)-C(11)-C(12)-C(14)	-113.6(2)
N(1)-C(3)-C(2)-S(2)	-35.8(2)
C(4)-C(3)-C(2)-S(2)	84.9(2)
C(1)-S(2)-C(2)-C(3)	24.80(19)
C(13)-C(12)-C(14)-O(2)	174.4(2)
C(11)-C(12)-C(14)-O(2)	53.1(3)
C(13)-C(12)-C(14)-C(15)	-64.4(3)
C(11)-C(12)-C(14)-C(15)	174.4(2)
C(9)-C(10)-C(5)-C(6)	-0.1(5)
C(9)-C(10)-C(5)-C(4)	179.8(3)
C(6)-C(5)-C(4)-C(3)	-101.3(3)
C(10)-C(5)-C(4)-C(3)	78.8(3)
N(1)-C(3)-C(4)-C(5)	-163.53(19)
C(2)-C(3)-C(4)-C(5)	79.0(3)
O(2)-C(14)-C(15)-C(17)	-132.6(3)
C(12)-C(14)-C(15)-C(17)	110.1(3)
O(2)-C(14)-C(15)-C(16)	47.8(3)
C(12)-C(14)-C(15)-C(16)	-69.6(3)
C(10)-C(5)-C(6)-C(7)	-1.5(5)
C(4)-C(5)-C(6)-C(7)	178.7(3)
C(5)-C(6)-C(7)-C(8)	2.2(6)
C(5)-C(10)-C(9)-C(8)	0.9(5)
C(6)-C(7)-C(8)-C(9)	-1.3(6)
C(10)-C(9)-C(8)-C(7)	-0.2(6)

X-ray crystallographic data for aldol adduct **3b**: Table 1. Crystal data and structure refinement for **3b**.

Identification code	cwd79t
Empirical formula	C ₂₁ H ₂₃ N O ₂ S ₂
Formula weight	385.52
Temperature	213(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)
Unit cell dimensions	a = 11.754(4) Å = 90°. b = 7.320(3) Å = 97.474(7)°. c = 12.160(4) Å = 90°.
Volume	1037.4(7) Å ³
Z	2
Density (calculated)	1.234 Mg/m ³
Absorption coefficient	0.271 mm ⁻¹
F(000)	408
Crystal size	0.6 x 1.4 x 1.8 mm ³
Theta range for data collection	1.69 to 28.28°
Index ranges	-15<=h<=12, -9<=k<=5, -15<=l<=15
Reflections collected	6904
Independent reflections	3813 [R(int) = 0.0726]
Completeness to theta = 28.28°	97.7 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3813 / 1 / 235
Goodness-of-fit on F ²	1.174
Final R indices [I>2sigma(I)]	R1 = 0.0964, wR2 = 0.1936
R indices (all data)	R1 = 0.1265, wR2 = 0.2075
Absolute structure parameter	0.11(17)
Largest diff. peak and hole	0.514 and -0.343 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for **3b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	1139(1)	4593(3)	6882(1)	47(1)
S(2)	661(1)	7057(3)	4994(1)	47(1)
N(1)	2243(3)	4642(8)	5022(3)	32(1)
O(1)	3545(4)	2677(8)	4546(3)	61(2)

C(5)	1208(5)	4633(11)	1894(4)	35(1)
C(2)	1585(5)	7207(12)	3920(5)	42(2)
O(2)	4449(4)	6152(8)	6005(4)	63(2)
C(6)	2104(5)	4311(10)	1284(5)	42(2)
C(10)	239(6)	5529(11)	1395(5)	47(2)
C(3)	2070(4)	5311(9)	3863(4)	31(1)
C(16)	5347(4)	4855(10)	7722(4)	37(2)
C(15)	4810(4)	4516(12)	6552(5)	46(2)
C(11)	3156(5)	3436(11)	5309(5)	43(2)
C(4)	1300(5)	4022(10)	3090(4)	37(2)
C(12)	3729(5)	3264(9)	6491(5)	33(1)
C(21)	6440(5)	4138(11)	8109(6)	46(2)
C(7)	2021(7)	4924(12)	211(6)	63(2)
C(1)	1459(4)	5295(10)	5660(5)	35(2)
C(18)	5312(6)	6219(12)	9507(6)	56(2)
C(13)	3972(7)	1291(12)	6789(6)	59(2)
C(17)	4812(5)	5877(11)	8449(6)	46(2)
C(20)	6942(6)	4493(13)	9172(6)	57(2)
C(9)	170(8)	6123(14)	317(6)	64(2)
C(8)	1074(8)	5851(12)	-266(6)	64(2)
C(14)	2932(8)	108(13)	6768(8)	83(3)
C(19)	6396(7)	5540(13)	9876(6)	60(2)

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

S(1)-C(1)	1.662(6)	C(10)-C(5)-C(4)	120.9(5)
S(2)-C(1)	1.732(7)	C(6)-C(5)-C(4)	120.1(6)
S(2)-C(2)	1.806(6)	C(3)-C(2)-S(2)	104.1(5)
N(1)-C(1)	1.365(7)	C(7)-C(6)-C(5)	119.3(7)
N(1)-C(11)	1.399(8)	C(9)-C(10)-C(5)	120.8(7)
N(1)-C(3)	1.481(7)	N(1)-C(3)-C(2)	105.4(4)
O(1)-C(11)	1.220(7)	N(1)-C(3)-C(4)	112.1(5)
C(5)-C(10)	1.384(9)	C(2)-C(3)-C(4)	113.4(5)
C(5)-C(6)	1.385(8)	C(17)-C(16)-C(21)	117.5(5)
C(5)-C(4)	1.511(8)	C(17)-C(16)-C(15)	122.0(5)
C(2)-C(3)	1.506(10)	C(21)-C(16)-C(15)	120.5(5)
O(2)-C(15)	1.408(9)	O(2)-C(15)-C(16)	111.8(7)
C(6)-C(7)	1.370(10)	O(2)-C(15)-C(12)	106.5(4)
C(10)-C(9)	1.374(10)	C(16)-C(15)-C(12)	112.4(5)
C(3)-C(4)	1.540(8)	O(1)-C(11)-N(1)	116.7(5)
C(16)-C(17)	1.372(9)	O(1)-C(11)-C(12)	121.4(6)
C(16)-C(21)	1.411(8)	N(1)-C(11)-C(12)	121.5(5)
C(16)-C(15)	1.500(8)	C(5)-C(4)-C(3)	111.7(5)
C(15)-C(12)	1.560(9)	C(13)-C(12)-C(11)	110.8(6)
C(11)-C(12)	1.511(8)	C(13)-C(12)-C(15)	115.1(5)
C(12)-C(13)	1.507(10)	C(11)-C(12)-C(15)	104.8(5)
C(21)-C(20)	1.375(10)	C(20)-C(21)-C(16)	120.3(6)
C(7)-C(8)	1.366(12)	C(8)-C(7)-C(6)	121.3(7)
C(18)-C(17)	1.366(10)	N(1)-C(1)-S(1)	130.4(5)
C(18)-C(19)	1.387(11)	N(1)-C(1)-S(2)	111.1(4)
C(13)-C(14)	1.496(11)	S(1)-C(1)-S(2)	118.5(3)
C(20)-C(19)	1.368(11)	C(17)-C(18)-C(19)	120.2(7)
C(9)-C(8)	1.367(13)	C(14)-C(13)-C(12)	115.0(6)
C(1)-S(2)-C(2)	92.5(3)	C(18)-C(17)-C(16)	122.0(6)
C(1)-N(1)-C(11)	129.2(5)	C(19)-C(20)-C(21)	121.0(6)
C(1)-N(1)-C(3)	113.9(5)	C(8)-C(9)-C(10)	119.7(8)
C(11)-N(1)-C(3)	116.8(4)	C(7)-C(8)-C(9)	119.8(7)
C(10)-C(5)-C(6)	119.0(6)	C(20)-C(19)-C(18)	119.0(6)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3b**. The anisotropic displacement factor exponent takes the form: $-2 \sum h^2 a^* 2U^{11} + \dots + 2 \sum h k a^* b^* U^{12}$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(1)	35(1)	79(2)	26(1)	-1(1)	9(1)	-2(1)
S(2)	41(1)	59(1)	42(1)	-5(1)	13(1)	16(1)

N(1)	30(2)	40(3)	26(2)	-2(3)	5(2)	3(3)
O(1)	57(3)	88(5)	36(2)	-7(3)	4(2)	40(3)
C(5)	41(3)	35(4)	30(3)	-8(3)	3(2)	-9(3)
C(2)	39(3)	60(5)	29(3)	-5(4)	6(2)	2(4)
O(2)	42(2)	89(5)	57(3)	38(3)	6(2)	-12(3)
C(6)	51(3)	34(4)	43(3)	-4(3)	12(3)	5(3)
C(10)	46(4)	50(5)	43(4)	-9(3)	-6(3)	-3(3)
C(3)	29(3)	39(4)	25(3)	-3(3)	8(2)	3(3)
C(16)	35(3)	40(4)	34(3)	0(3)	5(2)	0(3)
C(15)	25(3)	70(5)	41(3)	6(4)	4(2)	9(4)
C(11)	40(3)	63(5)	25(3)	-8(3)	3(2)	3(3)
C(4)	42(3)	39(4)	28(3)	1(3)	2(2)	-2(3)
C(12)	35(3)	32(4)	31(3)	4(3)	-4(2)	6(3)
C(21)	34(3)	44(5)	58(4)	-2(4)	1(3)	1(3)
C(7)	97(5)	51(6)	49(4)	-12(4)	36(4)	-10(5)
C(1)	20(2)	50(5)	33(3)	-14(3)	-1(2)	-4(3)
C(18)	59(4)	51(5)	59(4)	-14(4)	11(3)	0(4)
C(13)	63(4)	60(6)	48(4)	-3(4)	-12(3)	10(4)
C(17)	38(3)	50(5)	51(4)	3(4)	7(3)	0(3)
C(20)	48(3)	51(5)	68(4)	3(5)	-10(3)	9(4)
C(9)	76(5)	71(7)	39(4)	-7(4)	-19(4)	4(5)
C(8)	126(7)	33(5)	29(4)	8(3)	-4(4)	-9(5)
C(14)	90(6)	37(6)	111(7)	12(5)	-25(5)	-20(5)
C(19)	77(5)	60(6)	38(4)	3(4)	-13(4)	-19(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **3b**.

	x	y	z	U(eq)
H(2A)	1147	7555	3210	51
H(2B)	2197	8103	4116	51
H(2C)	5013	6692	5816	94
H(6A)	2761	3679	1601	50
H(10A)	-379	5733	1799	57
H(3A)	2828	5397	3593	37
H(15A)	5384	3920	6142	55
H(4A)	1617	2783	3155	44
H(4B)	532	3989	3321	44
H(12A)	3207	3764	6993	40
H(21A)	6827	3415	7637	55
H(7A)	2628	4703	-204	76
H(18A)	4920	6918	9987	67
H(13A)	4462	783	6271	70
H(13B)	4404	1239	7534	70
H(17A)	4079	6355	8213	55
H(20A)	7672	4010	9420	69
H(9A)	-497	6713	-18	77
H(8A)	1045	6300	-994	77
H(14A)	3165	-1136	6959	125
H(14B)	2501	129	6032	125
H(14C)	2454	563	7301	125
H(19A)	6751	5794	10598	72

Table 6. Torsion angles [$^\circ$] for **3b**.

C(1)-S(2)-C(2)-C(3)	27.3(4)
C(10)-C(5)-C(6)-C(7)	1.6(11)
C(4)-C(5)-C(6)-C(7)	-178.0(7)
C(6)-C(5)-C(10)-C(9)	-1.2(12)
C(4)-C(5)-C(10)-C(9)	178.4(7)
C(1)-N(1)-C(3)-C(2)	32.3(7)
C(11)-N(1)-C(3)-C(2)	-148.5(6)
C(1)-N(1)-C(3)-C(4)	-91.5(6)
C(11)-N(1)-C(3)-C(4)	87.7(6)
S(2)-C(2)-C(3)-N(1)	-36.4(5)
S(2)-C(2)-C(3)-C(4)	86.6(5)

C(17)-C(16)-C(15)-O(2)	51.4(8)
C(21)-C(16)-C(15)-O(2)	-127.8(6)
C(17)-C(16)-C(15)-C(12)	-68.3(9)
C(21)-C(16)-C(15)-C(12)	112.5(7)
C(1)-N(1)-C(11)-O(1)	161.0(7)
C(3)-N(1)-C(11)-O(1)	-18.1(9)
C(1)-N(1)-C(11)-C(12)	-26.6(11)
C(3)-N(1)-C(11)-C(12)	154.4(6)
C(10)-C(5)-C(4)-C(3)	-102.5(7)
C(6)-C(5)-C(4)-C(3)	77.0(8)
N(1)-C(3)-C(4)-C(5)	-175.3(5)
C(2)-C(3)-C(4)-C(5)	65.4(6)
O(1)-C(11)-C(12)-C(13)	-49.7(9)
N(1)-C(11)-C(12)-C(13)	138.2(7)
O(1)-C(11)-C(12)-C(15)	75.1(9)
N(1)-C(11)-C(12)-C(15)	-97.0(7)
O(2)-C(15)-C(12)-C(13)	166.4(6)
C(16)-C(15)-C(12)-C(13)	-70.9(8)
O(2)-C(15)-C(12)-C(11)	44.4(7)
C(16)-C(15)-C(12)-C(11)	167.1(6)
C(17)-C(16)-C(21)-C(20)	-1.0(11)
C(15)-C(16)-C(21)-C(20)	178.2(7)
C(5)-C(6)-C(7)-C(8)	0.2(12)
C(11)-N(1)-C(1)-S(1)	-14.3(10)
C(3)-N(1)-C(1)-S(1)	164.8(5)
C(11)-N(1)-C(1)-S(2)	169.3(6)
C(3)-N(1)-C(1)-S(2)	-11.6(6)
C(2)-S(2)-C(1)-N(1)	-10.0(5)
C(2)-S(2)-C(1)-S(1)	173.1(4)
C(11)-C(12)-C(13)-C(14)	-63.4(9)
C(15)-C(12)-C(13)-C(14)	177.9(6)
C(19)-C(18)-C(17)-C(16)	0.4(12)
C(21)-C(16)-C(17)-C(18)	0.8(11)
C(15)-C(16)-C(17)-C(18)	-178.5(7)
C(16)-C(21)-C(20)-C(19)	0.1(13)
C(5)-C(10)-C(9)-C(8)	-1.0(13)
C(6)-C(7)-C(8)-C(9)	-2.4(13)
C(10)-C(9)-C(8)-C(7)	2.7(13)
C(21)-C(20)-C(19)-C(18)	1.1(13)
C(17)-C(18)-C(19)-C(20)	-1.4(13)

X-ray crystallographic data for aldol adduct **3c**:

Table 1. Crystal data and structure refinement for **10**.

Identification code	cwd81t	
Empirical formula	C ₂₂ H ₂₃ N O ₂ S ₂	
Formula weight	397.53	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 11.568(2) Å	= 90°.
	b = 8.7811(15) Å	= 118.786(3)°.
	c = 11.759(2) Å	= 90°.
Volume	1046.9(3) Å ³	
Z	2	
Density (calculated)	1.261 Mg/m ³	
Absorption coefficient	0.271 mm ⁻¹	
F(000)	420	
Crystal size	1.4 x 1.8 x 6.8 mm ³	
Theta range for data collection	1.98 to 28.29°.	
Index ranges	-14<=h<=15, -10<=k<=11, -14<=l<=15	
Reflections collected	7498	
Independent reflections	4988 [R(int) = 0.0481]	
Completeness to theta = 28.29°	99.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4988 / 1 / 244	
Goodness-of-fit on F ²	0.881	
Final R indices [I>2sigma(I)]	R1 = 0.0427, wR2 = 0.0898	
R indices (all data)	R1 = 0.0546, wR2 = 0.0949	
Absolute structure parameter	-0.01(6)	
Largest diff. peak and hole	0.323 and -0.177 e.Å ⁻³	

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for **3c**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	-4700(1)	-7720(1)	-4274(1)	55(1)
S(2)	-2884(1)	-5241(1)	-2769(1)	57(1)
N(1)	-3642(2)	-7237(2)	-1680(2)	36(1)
O(2)	-5665(2)	-5839(2)	-1231(2)	56(1)
O(1)	-3903(2)	-8812(2)	-323(1)	48(1)
C(11)	-4425(2)	-8202(2)	-1373(2)	36(1)
C(16)	-6452(2)	-7174(2)	-1560(2)	40(1)
C(12)	-5906(2)	-8227(2)	-2248(2)	37(1)
C(17)	-7903(2)	-6849(3)	-2422(2)	43(1)
C(3)	-2461(2)	-6567(2)	-589(2)	39(1)
C(1)	-3832(2)	-6838(2)	-2890(2)	41(1)
C(4)	-1263(2)	-7603(3)	-208(2)	45(1)
C(5)	-70(2)	-7107(2)	1025(2)	45(1)
C(18)	-8856(2)	-7712(3)	-2332(2)	55(1)
C(13)	-6465(2)	-9850(3)	-2397(3)	49(1)
C(14)	-5931(2)	-10969(3)	-2976(3)	49(1)
C(2)	-2333(2)	-5005(2)	-1067(3)	52(1)
C(6)	1012(2)	-6440(3)	1008(3)	62(1)
C(10)	-15(3)	-7350(3)	2206(3)	68(1)
C(22)	-8305(3)	-5702(3)	-3331(3)	64(1)
C(7)	2092(3)	-6016(3)	2157(5)	87(1)
C(19)	-10189(3)	-7447(4)	-3155(3)	73(1)
C(9)	1061(4)	-6917(4)	3347(3)	97(1)
C(15)	-5498(3)	-12317(3)	-2504(3)	67(1)
C(20)	-10566(3)	-6315(5)	-4053(3)	84(1)
C(8)	2129(4)	-6245(4)	3306(5)	101(2)
C(21)	-9637(3)	-5430(4)	-4155(3)	83(1)

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

S(1)-C(1)	1.638(2)	O(1)-C(11)-C(12)	122.20(18)
S(2)-C(1)	1.744(2)	N(1)-C(11)-C(12)	119.40(17)
S(2)-C(2)	1.796(3)	O(2)-C(16)-C(17)	112.70(18)
N(1)-C(1)	1.377(3)	O(2)-C(16)-C(12)	105.45(15)
N(1)-C(11)	1.408(3)	C(17)-C(16)-C(12)	111.24(17)
N(1)-C(3)	1.473(3)	C(11)-C(12)-C(13)	111.46(18)
O(2)-C(16)	1.419(3)	C(11)-C(12)-C(16)	104.39(16)
O(1)-C(11)	1.208(2)	C(13)-C(12)-C(16)	110.97(17)
C(11)-C(12)	1.516(3)	C(22)-C(17)-C(18)	118.5(2)
C(16)-C(17)	1.513(3)	C(22)-C(17)-C(16)	120.6(2)
C(16)-C(12)	1.549(3)	C(18)-C(17)-C(16)	120.9(2)
C(12)-C(13)	1.540(3)	N(1)-C(3)-C(2)	104.65(18)
C(17)-C(22)	1.376(4)	N(1)-C(3)-C(4)	110.26(16)
C(17)-C(18)	1.382(3)	C(2)-C(3)-C(4)	114.15(18)
C(3)-C(2)	1.517(3)	N(1)-C(1)-S(1)	128.54(17)
C(3)-C(4)	1.533(3)	N(1)-C(1)-S(2)	109.85(16)
C(4)-C(5)	1.507(3)	S(1)-C(1)-S(2)	121.46(13)
C(5)-C(10)	1.375(4)	C(5)-C(4)-C(3)	112.94(17)
C(5)-C(6)	1.391(3)	C(10)-C(5)-C(6)	118.4(2)
C(18)-C(19)	1.390(3)	C(10)-C(5)-C(4)	120.2(2)
C(13)-C(14)	1.488(3)	C(6)-C(5)-C(4)	121.4(2)
C(14)-C(15)	1.301(4)	C(17)-C(18)-C(19)	120.8(3)
C(6)-C(7)	1.378(4)	C(14)-C(13)-C(12)	114.80(19)
C(10)-C(9)	1.373(4)	C(15)-C(14)-C(13)	124.4(3)
C(22)-C(21)	1.391(4)	C(3)-C(2)-S(2)	105.28(16)
C(7)-C(8)	1.346(6)	C(7)-C(6)-C(5)	119.8(3)
C(19)-C(20)	1.360(5)	C(9)-C(10)-C(5)	121.6(3)
C(9)-C(8)	1.391(6)	C(17)-C(22)-C(21)	120.9(3)
C(20)-C(21)	1.377(5)	C(8)-C(7)-C(6)	121.3(3)
C(1)-S(2)-C(2)	93.15(10)	C(20)-C(19)-C(18)	119.8(3)
C(1)-N(1)-C(11)	128.02(17)	C(10)-C(9)-C(8)	119.1(4)
C(1)-N(1)-C(3)	114.70(17)	C(19)-C(20)-C(21)	120.5(3)
C(11)-N(1)-C(3)	117.27(17)	C(7)-C(8)-C(9)	119.9(3)
O(1)-C(11)-N(1)	117.54(18)	C(20)-C(21)-C(22)	119.5(3)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3c**. The anisotropic displacement factor exponent takes the form: $-2 \text{ h}^2 \text{ a}^* \text{ U}^{11} + \dots + 2 \text{ h k a}^* \text{ b}^* \text{ U}^{12}$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(1)	60(1)	71(1)	38(1)	4(1)	26(1)	7(1)
S(2)	64(1)	47(1)	78(1)	16(1)	47(1)	3(1)
N(1)	34(1)	36(1)	38(1)	1(1)	18(1)	0(1)
O(2)	54(1)	50(1)	76(1)	-26(1)	39(1)	-11(1)
O(1)	42(1)	56(1)	42(1)	11(1)	16(1)	-8(1)
C(11)	35(1)	37(1)	33(1)	-1(1)	16(1)	-2(1)
C(16)	40(1)	44(1)	40(1)	-5(1)	23(1)	-3(1)
C(12)	33(1)	41(1)	38(1)	-6(1)	18(1)	-2(1)
C(17)	43(1)	49(1)	43(1)	-7(1)	26(1)	5(1)
C(3)	35(1)	35(1)	47(1)	-6(1)	21(1)	-3(1)
C(1)	40(1)	40(1)	51(1)	11(1)	29(1)	15(1)
C(4)	39(1)	35(1)	60(1)	-4(1)	23(1)	2(1)
C(5)	33(1)	32(1)	64(2)	0(1)	18(1)	4(1)
C(18)	46(1)	67(2)	63(2)	0(1)	35(1)	5(1)
C(13)	36(1)	50(1)	59(2)	-12(1)	22(1)	-8(1)
C(14)	42(1)	44(1)	59(2)	-10(1)	23(1)	-6(1)
C(2)	43(1)	33(1)	82(2)	-3(1)	33(1)	-2(1)
C(6)	40(1)	38(1)	104(2)	8(1)	31(2)	2(1)
C(10)	54(2)	78(2)	63(2)	0(2)	20(1)	0(1)
C(22)	61(2)	67(2)	68(2)	10(1)	35(1)	10(1)
C(7)	38(2)	44(2)	151(4)	11(2)	22(2)	-1(1)
C(19)	45(2)	103(2)	80(2)	-17(2)	38(1)	-5(2)
C(9)	91(3)	94(2)	62(2)	-2(2)	2(2)	12(2)
C(15)	71(2)	53(2)	77(2)	0(1)	37(2)	6(1)

C(20)	47(2)	130(3)	68(2)	1(2)	24(1)	29(2)
C(8)	57(2)	56(2)	116(3)	-6(2)	-18(2)	5(2)
C(21)	72(2)	104(2)	76(2)	25(2)	36(2)	35(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **3c**.

	x	y	z	U(eq)
H(2A)	-5840	-5288	-761	85
H(16A)	-6328	-7674	-754	48
H(12A)	-6129	-7806	-3110	45
H(3A)	-2616	-6449	164	46
H(4A)	-1496	-8645	-99	54
H(4B)	-1041	-7610	-914	54
H(18A)	-8598	-8488	-1706	66
H(13A)	-6280	-10221	-1540	58
H(13B)	-7425	-9805	-2940	58
H(14A)	-5905	-10682	-3734	58
H(2B)	-1415	-4657	-613	62
H(2C)	-2884	-4261	-927	62
H(6A)	1006	-6278	215	75
H(10A)	-730	-7824	2232	82
H(22A)	-7670	-5095	-3395	76
H(7A)	2818	-5559	2138	105
H(19A)	-10828	-8048	-3091	88
H(9A)	1075	-7073	4144	116
H(15A)	-5510	-12639	-1747	80
H(15B)	-5173	-12968	-2919	80
H(20A)	-11468	-6135	-4609	100
H(8A)	2875	-5950	4079	121
H(21A)	-9902	-4649	-4777	100

Table 6. Torsion angles [$^\circ$] for **3c**.

C(1)-N(1)-C(11)-O(1)	155.87(19)
C(3)-N(1)-C(11)-O(1)	-23.5(3)
C(1)-N(1)-C(11)-C(12)	-34.5(3)
C(3)-N(1)-C(11)-C(12)	146.17(17)
O(1)-C(11)-C(12)-C(13)	-49.3(3)
N(1)-C(11)-C(12)-C(13)	141.5(2)
O(1)-C(11)-C(12)-C(16)	70.5(3)
N(1)-C(11)-C(12)-C(16)	-98.6(2)
O(2)-C(16)-C(12)-C(11)	47.6(2)
C(17)-C(16)-C(12)-C(11)	170.03(17)
O(2)-C(16)-C(12)-C(13)	167.74(18)
C(17)-C(16)-C(12)-C(13)	-69.8(2)
O(2)-C(16)-C(17)-C(22)	34.1(3)
C(12)-C(16)-C(17)-C(22)	-84.1(3)
O(2)-C(16)-C(17)-C(18)	-147.0(2)
C(12)-C(16)-C(17)-C(18)	94.8(2)
C(1)-N(1)-C(3)-C(2)	34.6(2)
C(11)-N(1)-C(3)-C(2)	-146.01(18)
C(1)-N(1)-C(3)-C(4)	-88.6(2)
C(11)-N(1)-C(3)-C(4)	90.8(2)
C(11)-N(1)-C(1)-S(1)	-20.8(3)
C(3)-N(1)-C(1)-S(1)	158.54(15)
C(11)-N(1)-C(1)-S(2)	163.63(16)
C(3)-N(1)-C(1)-S(2)	-17.0(2)
C(2)-S(2)-C(1)-N(1)	-4.94(15)
C(2)-S(2)-C(1)-S(1)	179.13(14)
N(1)-C(3)-C(4)-C(5)	-171.36(18)
C(2)-C(3)-C(4)-C(5)	71.2(3)
C(3)-C(4)-C(5)-C(10)	74.5(3)
C(3)-C(4)-C(5)-C(6)	-107.5(2)
C(22)-C(17)-C(18)-C(19)	1.1(4)
C(16)-C(17)-C(18)-C(19)	-177.8(2)

C(11)-C(12)-C(13)-C(14)	-60.5(3)
C(16)-C(12)-C(13)-C(14)	-176.4(2)
C(12)-C(13)-C(14)-C(15)	132.1(3)
N(1)-C(3)-C(2)-S(2)	-34.90(18)
C(4)-C(3)-C(2)-S(2)	85.7(2)
C(1)-S(2)-C(2)-C(3)	23.75(15)
C(10)-C(5)-C(6)-C(7)	-1.2(3)
C(4)-C(5)-C(6)-C(7)	-179.3(2)
C(6)-C(5)-C(10)-C(9)	1.6(4)
C(4)-C(5)-C(10)-C(9)	179.7(3)
C(18)-C(17)-C(22)-C(21)	-1.0(4)
C(16)-C(17)-C(22)-C(21)	177.9(3)
C(5)-C(6)-C(7)-C(8)	0.4(4)
C(17)-C(18)-C(19)-C(20)	-0.7(4)
C(5)-C(10)-C(9)-C(8)	-1.3(5)
C(18)-C(19)-C(20)-C(21)	0.1(5)
C(6)-C(7)-C(8)-C(9)	-0.1(5)
C(10)-C(9)-C(8)-C(7)	0.5(5)
C(19)-C(20)-C(21)-C(22)	0.0(5)
C(17)-C(22)-C(21)-C(20)	0.4(5)