Correspondence between molecular functionality and crystal structures. Supramolecular chemistry of a family of homologated aminophenols

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Supporting Information
(123 pages)

(i) Synthetic procedures and relevant literature.

(ii) Structure refinement parameters.

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(viii) Simulated powder X-ray diagrams of selected compounds.

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For the structures **1** through **5, 1a, 2a, 2b, 2c, 3a, 2d, 1b** and **6a.**

(i) Synthetic procedures for the compounds in this study

General Methods. IR spectra were recorded on a Jasco-FT-IR model 5300 or a Perkin-Elmer model 1310 spectrometer. ^{1}H NMR (200 MHz) was recorded in DMSO-d_6 on a Bruker-AC-200 spectrometer. All reactions were carried out using standard techniques and general literature procedures. Work up means drying of the combined organic extracts with $MgSO_4$, filtration, and concentration of the crude residue in vacuo. All compounds were purified by column chromatography using silica gel (100-200 mesh) with EtOAc–hexane. Yields reported here are for column separated products. All reagents and solvents were dried and distilled prior to use.

4-(4-aminobenzyl)phenol (1).[1] was prepared in 20 % yield according to the published procedure, mp 147 °C. IR (cm^{-1}): 3381, 3310, 3015. ^{1}H NMR : δ 3.61 (s, 2H), 4.81 (s, 2H), 6.46 (d, J 8, 2H), 6.66 (d, J 8, 2H), 6.80 (d, J 8, 2H), 6.96 (d, J 8, 2H) and 9.12 (s, 1H).

4-(4-aminophenethyl)phenol (2). *trans*-4-nitro-4'-methoxystilbene was prepared according to the Wittig reaction.[2a] To this compound (510 mg, 2 mmol) 15 mL of EtOH and $N_2H_4.H_2O$ (0.38 mL, 8 mmol) was added and stirred. Pd/C was slowly added and the reaction mixture refluxed at 70 °C for 3 h. The reaction mixture was filtered off to remove Pd/C, then the filtrate evaporated to yield 320 mg (71 %) of 4-amino-4'-methoxydiphenylethane.[2b] To a degassed solution of this compound (227 mg, 1 mmol) in dry CH_2Cl_2 (15 mL), a solution of BBr_3 in CH_2Cl_2 was added dropwise and the mixture was stirred under N_2 at –78 °C for 3 h and stirring was continued overnight. The mixture

was diluted with CH_2Cl_2 (10 mL), and washed with H_2O. Workup afforded 100 mg (42 %) of **2**[2c] with mp 222 °C. IR (cm^{-1}): 3370, 3310, ^1H NMR: 2.70 (s, 4H), 4.8 (s, 2H), 6.57 (d, J 8, 2H), 6.67 (d, J 8, 2H), 6.90 (d, J 8, 2H), 7.00 (d, J 8, 2H), 9.15 (s, 1H).

4-[3-(4-aminophenyl)propyl]phenol (3). 4'-Hydroxy-4-nitrochalcone was prepared in 78 % yield by the Claisen-Schmidt condensation[3a] of 4-hydroxyacetophenone (1.49 g, 11 mmol) and 4-nitrobenzaldehyde (1.51 g, 10 mmol) in aqueous NaOH (1.14 g, 28 mmol) by stirring at room temperature. This chalcone (1.85 g, 7 mmol) was reduced to furnish 1.25 g (67 %) of 4'-hydroxy-4-hydroxylamino-1,3-diphenyl propane-1-one by refluxing overnight with Et_3N (15 mL), HCO_2H (1.7 mL, 45 mmol) and Pd/C (144 mg, 0.14 mmol).[3b] This product (1.2 g, 4.7 mmol) was further reduced using CF_3COOH (25 mL) and $NaBH_4$ (1.02 g, 27.5 mmol) at 0-5°C for 30 min under N_2 and then for an additional 30 h at room temperature,[3c] to yield 180 mg (17 %) of compound **3** with mp 142 °C, IR (cm^{-1}): 3372, 3308, ^1H NMR: 1.75 (m, 2H), 2.43 (m, 4H), 4.84 (s, 2H), 6.05 (d, J 8, 2H), 6.70 (d, J 8, 2H), 6.85 (d, J 8, 2H), 7.00 (d, J 8, 2H), 9.10 (s, 1H).

4-[4-(4-aminophenyl)butyl]phenol (4). Wittig reaction[2a] between *p*-nitrobenzyltriphenylphosphonium bromide and 4-methoxycinnamaldehyde gave 1-(4-methoxyphenyl)-4-(4'-nitrophenyl)-1,3-butadiene in 51 % yield. As described in **2**, this compound was reduced to give 1-(4-methoxyphenyl)-4-(4'-aminophenyl)butane in 70 % yield. Methoxy deprotection afforded **4** in 40 % yield, mp 185 °C. IR (cm^{-1}): 3366, 3290.^1H NMR: 1.4 (s, 4H), 2.4 (m, 4H), 4.8 (s, 2H), 6.42 (d, J 8, 2H), 6.64 (d, J 8, 2H), 6.82 (d, J 8, 2H), 6.92 (d, J 8, 2H), 9.07 (s, 1H).

4-[5-(4-aminophenyl)pentyl]phenol (5). 4-hydroxybenzylideneacetone was prepared in 62 % yield by the condensation of 4-hydroxybenzaldehyde and acetone[4a]. Pure 4-hydroxybenzylideneacetone was further condensed with 4-nitrobenzaldehyde to give (81 %) 4-hydroxy-4'-nitrodibenzylideneacetone. This compound (885 mg, 3 mmol) was subjected to hydrogenation with PtO_2 (56.25 mg, 3 mmol)/H_2 in MeOH/THF (1:1) solution for 15 h[4b] to give 150 mg (18 %) of 1-(4-hydroxyphenyl)-5(4'-aminophenyl)pentane-3-one. This was further reduced with Zn–Hg, HCl (g), conc. HCl as described[4c] (Na_2CO_3 was used for the neutralisation) to get 62 % of **5**, mp 106 °C. IR (cm^{-1}): 3375, 3304, 2924,1616, ^{1}H NMR: 1.22 (m, 2H), 1.47 (m, 4H), 2.4 (m, 4H), 4.75 (m, 4H), 6.45 (d, J 8, 2H), 6.65 (d, J 8, 2H), 6.80 (d, J 8, 2H), 6.92 (d, J 8, 2H), 9.1 (s, 1H).

4-(4-aminophenylsulfamyl)phenol (1a). To a solution of 4-hydroxythiophenol (151 mg, 1.2 mmol) and anhy. K_2CO_3 in dry CH_3CN (20 mL), 4-nitroiodobenzene (300 mg, 1.2 mmol) was added under N_2 atmosphere and refluxed for 12 h. The workup yielded 208 mg (70 %) of 4-hydroxy-4'-nitrodiphenylsulfide.[5a] To this nitrothioether (250 mg, 1.01 mmol) in 10 mL of EtOH, $N_2H_4.H_2O$ (0.26 mL, 5.4 mmol) and Pd/C were added and stirred for 2 h at room temperature.[5b] The reaction mixture was filtered off and evaporated to yield 153 mg (70 %) of **1a** with mp 147-148 °C. IR (cm^{-1}): 3371, 3308. ^{1}H NMR: 5.30 (s, 2H), 6.52 (d, J 8, 2H), 6.70 (d, J 8, 2H), 7.10 (m, 4H), 9.45 (s, H).

4-(4-aminobenzylsulfamyl)phenol (2a). As in **1a** the thiolation reaction of 4-hydroxythiophenol with 4-nitrobenzyl bromide gave 62 % of 4-nitrobenzyl-4'-

hydroxyphenylthioether. To a solution of the nitrothioether (250 mg, 0.957 mmol) in dry EtOAc, $SnCl_2.2H_2O$ (1.07 g, 4.78 mmol) was added and refluxed for 1.5 h. The mixture was cooled, neutralised with $NaHCO_3$, and worked up to give 152 mg (69 %) of **2a**[6] with mp 211-212 °C. IR (cm^{-1}): 3360, 3287. ^{1}HNMR: 4.0 (s, 2H), 5.0 (s, 2H) ,6.52 (d, J 8, 2H), 6.70 (d, J 8, 2H), 6.94 (d, J 8, 2H),7.1(d, 2H), 9.42 (s, 1H).

4-(4-aminophenylsulfamylmethyl)phenol (2b). 4-Hydroxybenzaldehyde (800 mg, 6.55 mmol) was subjected to phenolic protection using *t*-butylchlorodiphenylsilane (5.40 g, 19.65 mmol) and imidazole (668 mg, 9.82 mmol) in dry DMF in N_2 atmosphere at 0°C for 3 h, followed by workup and purification to obtain 1.89 g (80%) of the silylated compound.[7a] Then the silyl protected benzaldehyde (1 g, 2.77 mmol) was reduced using $NaBH_4$ (157 mg, 4.16 mmol) to yield 623 mg (62 %) of the corresponding alcohol. This benzyl alcohol 500 mg, (1.37 mmol) was converted to the silyl protected benzyl bromide with PBr_3 0.64 mL (0.685 mmol) in dry Et_2O (20 mL) and was stirred at 0 °C for 1 h. The reaction mixture was subjected to workup and immediately added dropwise to a refluxing solution of 4-hydroxythiophenol (172 mg, 1.37 mmol) and anhy. K_2CO_3 in dry CH_3CN. The resultant mixture was refluxed for 12 h to yield 712 mg (55 %) of silyl protected aminophenol. Then the compound (500 mg, 1.06 mmol) was deprotected using TBAF[7b] (0.306 mL, 1.06 mmol) in THF to produce100 mg (60 %) of **2b** with mp 205-207 °C. IR (cm^{-1}): 3369, 3295, ^{1}H NMR: 3.85 (s, 2H), 5.24 (s, 2H), 6.47 (d, J 8, 2H), 6.70 (d, J 8, 2H), 7.00 (m, 4H), 9.30 (s, 1H).

4-(4-aminophenyldisulfamyl)phenol (2c). 2,2'-Dithiobis(benzothiazole) (BTS–SBT) was prepared by the standard procedure of dimerization of 2-mercaptobenzothiazole in I_2 and EtOH. To a solution of 4-hydroxythiophenol (152 mg, 1.21 mmol) (P–HBSH) in 20 mL of $CHCl_3$, a stirred suspension of 2,2'-diithiobis(benzothiazol) disulfide (800 mg, 1.20 152 mmol) in 100 mL of $CHCl_3$ was added. Stirring was continued at room temperature for 24 h to give 275 mg (50 %) of unsymmetrical 2-benzothiazolyl 4-hydroxyphenyl disulfide (P–HBS–SBT). Using the preceding procedure, the reaction of P–HBS–SBT (200 mg, 0.438 mmol) with 66 mg (0.530 mmol) of 4-aminothiophenol yielded, after work up, 79 mg (60 %), mp 113-115 °C of **2c**[8]. IR (cm^{-1}): 3370, 3305, 750, ^1H NMR: 5.35 (s, 2H), 6.48 (d, J 8, 2H), 6.75 (d, J 8, 2H), 7.05 (d, J 8, 2H), 7.20 (d, J 8, 2H), 9.40 (s, 1H).

4-[2-(4-aminophenylsulfamyl)ethyl]phenol (3a). Treatment of 4-nitrophenylethyl bromide with 4-hydroxythiophenol as in the case of **1a** yielded (55 %) 4-nitrophenylethyl-4'-hydroxyphenylsulphide. This was reduced with $SnCl_2.2H_2O$ as in **2a** to get **3a** in 65 % yield with mp 116-118 °C. IR (cm^{-1}): 3370, 3305, ^1H NMR: 2.54 (t, 2H), 3.34 (t, 2H), 6.15 (d, J 8, 2H), 6.72 (d, J 8, 2H), 6.88 (d, J 8, 2H), 7.15 (d, J 8, 2H), 9.50 (s, 1H).

4-[(E)-2-(4-aminophenyl)-1-ethenyl]phenol (2d). To 4-nitrophenylacetic acid (1.81 g, 10 mmol), piperidine (850 mg, 10 mmol) was added dropwise, then 4-hydroxybenzaldehyde (1.22 g, 10 mmol) and the mixture heated at 100 °C under vacuo for 1h, then for an additional 2 h at 150 °C. This mixture was taken in 30 % NaOH

solution and heated on a water bath, then neutralised with conc. HCl, to give a yellow precipitate (90 %) of 4,4'-hydroxynitrostilbene.[9] This was reduced with $SnCl_2.2H_2O$, as in **2a**, to produce **2d** in 17 % yield, mp 270 °C. IR (cm^{-1}): 3356, 3292, 3015. ^1H NMR: 5.18 (s, 2H), 6.52 (d, J 8, 2H), 6.75 (d, J 8, 2H), 6.81 (s, 2H), 7.2 (d, J 8, 2H), 7.32 (d, J 8, 2H), 9.40 (s, 1H).

4-(4-aminophenoxy)phenol (1b). To a degassed solution of hydroquinone (270 mg, 2.5 mmol) in DMSO, KOH (56 mg, 10 mmol), 4-chloronitrobenzene (392 mg, 2.5 mmol) was added, and refluxed for 16 h.[10] Workup yielded 400 mg (70 %) of 4,4'-hydroxynitrodiphenylether. To this (250 mg, 1.08 mmol) were added 10 mL of EtOH, $N_2H_4.H_2O$ (0.26 ml, 5.4 mmol), Pd/C and stirred at room temperature for 1 h. The reaction mixture was filtered off and evaporated to yield 152 mg (70 %) of **1b**, mp 157 °C. IR (cm^{-1}): 3368, 3304, 1874. H^1 NMR: 4.85(s, 2H), 6.5(d, J 8, 2H), 6.7(m, 6H), 9.00(s, 1H).

4-(3-aminophenyl)phenol (6a). A solution of 4-methoxyboronic acid (200 mg, 1.31 mmol), 3-nitrobromobenzene (246 mg, 1.22 mmol), $Pd(OAc)_2$ (3 % mol), DAB-Cy (3 % mol) and Cs_2CO_3 (2 equiv) in dioxane (3 mL) was refluxed for 4 h under inert conditions as in the modified Suzuki-Miyaura cross-coupling.[11] Workup afforded 241 mg (80 %) of 4-methoxy-3'-nitrobiphenyl. To this compound (250 mg, 1.09 mmol) were added 10 mL of EtOH and $N_2H_4.H_2O$ (0.35 ml, 8.89 mmol) and stirred for 2 h at room temperature as for 1b. The reaction mixture was filtered off and evaporated to yield 152 mg (70 %) of 4,3'-methoxyaminobiphenyl. Methoxy deprotection as in **2** gave 50 mg (40 %) of **6a**, mp

183 °C. IR (cm^{-1}): 3398, 3317, 3202. ^{1}H NMR: 5.1 (s, 2H), 6.49 (d, 1H), 6.66 (d, 1H), 6.72 (s, 1H), 6.78 (d, 2H), 7.03 (t, 1H), 7.34 (d, 2H), 9.43 (s, 1H).

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(ii) Structure Refinement Parameters: Atomic co-ordinates, Bond lengths and angles, and Anisotropic displacement parameters

For structures **1**(X and N) – **5, 1a, 2a, 2b, 2c, 3a, 2d, 1b and 6a**

Tabl e 1. Crystal data and structure refinement for **1N.**

Identification code	1_Neutron	
Empirical formula	C13 H13 N O	
Formula weight	199.25	
Temperature	12(2) K	
Wavelength	0.5-5.0 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 5.9180(12) Å	α= 90°.
	b = 19.213(4) Å	β= 101.25(3)°.
	c = 9.6510(19) Å	γ = 90°.
Volume	1076.3(4) $Å^3$	
Z	4	
Density (calculated)	1.230 Mg/m^3	
Absorption coefficient	1.870, at 1 Angstrom mm^{-1}	
F(000)	21.27	
Crystal size	1.5 x 1.5 x 1.3 mm^3	
Theta range for data collection	1.56 to 24.72°.	
Index ranges	0<=h<=17, 0<=k<=51, -29<=l<=24	
Reflections collected	6172	
Independent reflections	2403 [R(int) = 0.064]	
Completeness to theta = 24.72°	5.7 %	
Absorption correction	Empirical	
Max. and min. transmission	0.82 and 0.62	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2403 / 0 / 253	
Goodness-of-fit on F^2	1.081	
Final R indices [I>2sigma(I)]	R1 = 0.0791, wR2 = 0.2230	
R indices (all data)	R1 = 0.0793, wR2 = 0.2231	
Extinction coefficient	0.044	
Largest diff. peak and hole	3.387 and -2.930 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1N.** U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	12754(8)	518(3)	633(4)	10(1)
C(1)	11360(5)	830(2)	1393(3)	7(1)
C(2)	10112(6)	463(2)	2240(3)	7(1)
C(3)	8633(6)	821(2)	2950(3)	7(1)
C(4)	8409(6)	1544(2)	2864(3)	7(1)
C(5)	9693(6)	1901(2)	2039(3)	7(1)
C(6)	11166(6)	1550(2)	1310(3)	7(1)
C(7)	6759(6)	1922(2)	3592(3)	8(1)
C(8)	6615(5)	1650(2)	5028(3)	5(1)
C(9)	4641(5)	1328(2)	5286(3)	6(1)
C(10)	4514(5)	1080(2)	6620(3)	6(1)
C(11)	6393(5)	1149(2)	7737(3)	6(1)
C(12)	8386(5)	1469(2)	7476(3)	6(1)
C(13)	8488(5)	1712(2)	6148(3)	6(1)
N(1)	6331(4)	869(2)	9071(2)	9(1)

Table 3. Bond lengths [Å] and angles [°] for **1N.**

O(1)-C(1)	1.347(5)
O(1)-H(1)	0.969(15)
C(1)-C(6)	1.388(6)
C(1)-C(2)	1.395(5)
C(2)-C(3)	1.394(5)
C(2)-H(2)	1.060(12)
C(3)-C(4)	1.396(6)
C(3)-H(3)	1.078(9)
C(4)-C(5)	1.385(5)
C(4)-C(7)	1.497(5)
C(5)-C(6)	1.396(5)
C(5)-H(5)	1.081(14)
C(6)-H(6)	1.080(9)
C(7)-C(8)	1.499(4)
C(7)-H(7A)	1.115(13)
C(7)-H(7B)	1.087(9)
C(8)-C(9)	1.387(5)
C(8)-C(13)	1.394(4)
C(9)-C(10)	1.388(4)
C(9)-H(9)	1.075(9)
C(10)-C(11)	1.396(5)
C(10)-H(10)	1.079(10)
C(11)-C(12)	1.396(5)
C(11)-N(1)	1.403(4)
C(12)-C(13)	1.376(4)
C(12)-H(12)	1.073(8)
C(13)-H(13)	1.070(9)
N(1)-H(1A)	1.009(8)
N(1)-H(1B)	1.001(10)
C(1)-O(1)-H(1)	113.0(7)
O(1)-C(1)-C(6)	117.7(4)
O(1)-C(1)-C(2)	122.9(4)
C(6)-C(1)-C(2)	119.4(3)
C(3)-C(2)-C(1)	119.6(4)
C(3)-C(2)-H(2)	120.4(6)
C(1)-C(2)-H(2)	120.0(6)
C(2)-C(3)-C(4)	121.6(3)
C(2)-C(3)-H(3)	118.4(8)
C(4)-C(3)-H(3)	120.0(7)
C(5)-C(4)-C(3)	118.0(3)
C(5)-C(4)-C(7)	120.8(4)
C(3)-C(4)-C(7)	121.2(3)
C(4)-C(5)-C(6)	121.2(4)
C(4)-C(5)-H(5)	120.6(6)
C(6)-C(5)-H(5)	118.2(6)
C(1)-C(6)-C(5)	120.3(3)
C(1)-C(6)-H(6)	119.5(7)
C(5)-C(6)-H(6)	120.3(7)
C(4)-C(7)-C(8)	115.2(3)
C(4)-C(7)-H(7A)	107.5(5)
C(8)-C(7)-H(7A)	109.4(6)
C(4)-C(7)-H(7B)	110.4(6)
C(8)-C(7)-H(7B)	108.8(5)

H(7A)-C(7)-H(7B)	105.2(10)
C(9)-C(8)-C(13)	117.9(3)
C(9)-C(8)-C(7)	121.6(3)
C(13)-C(8)-C(7)	120.5(3)
C(8)-C(9)-C(10)	121.3(3)
C(8)-C(9)-H(9)	119.3(6)
C(10)-C(9)-H(9)	119.4(6)
C(9)-C(10)-C(11)	120.3(3)
C(9)-C(10)-H(10)	120.5(6)
C(11)-C(10)-H(10)	119.2(6)
C(10)-C(11)-C(12)	118.4(3)
C(10)-C(11)-N(1)	120.9(3)
C(12)-C(11)-N(1)	120.6(3)
C(13)-C(12)-C(11)	120.6(3)
C(13)-C(12)-H(12)	121.0(6)
C(11)-C(12)-H(12)	118.3(6)
C(12)-C(13)-C(8)	121.4(3)
C(12)-C(13)-H(13)	119.6(6)
C(8)-C(13)-H(13)	119.0(6)
C(11)-N(1)-H(1A)	116.1(6)
C(11)-N(1)-H(1B)	113.0(6)
H(1A)-N(1)-H(1B)	109.1(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1N.** The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	12(2)	8(2)	12(1)	1(2)	8(1)	0(2)
H(1)	20(4)	24(5)	26(4)	-2(4)	9(3)	-2(4)
C(1)	6(1)	8(2)	6(1)	0(1)	2(1)	0(1)
C(2)	11(1)	5(1)	7(1)	0(1)	4(1)	0(1)
C(3)	8(1)	7(2)	8(1)	1(1)	4(1)	0(1)
C(4)	7(1)	9(2)	5(1)	0(1)	2(1)	1(1)
C(5)	10(1)	4(1)	6(1)	2(1)	3(1)	0(1)
C(6)	9(1)	7(2)	6(1)	-1(1)	3(1)	-1(1)
H(2)	34(5)	11(4)	31(4)	-3(4)	17(4)	7(4)
H(3)	27(4)	19(5)	27(4)	0(4)	16(3)	-10(4)
H(5)	31(5)	16(5)	33(4)	-2(4)	13(4)	6(4)
H(6)	28(4)	20(5)	20(3)	2(4)	13(3)	-8(4)
C(7)	7(1)	10(2)	6(1)	-1(1)	2(1)	2(1)
H(7A)	29(4)	15(4)	25(3)	1(4)	14(3)	0(4)
H(7B)	16(3)	30(6)	15(2)	4(4)	1(2)	4(4)
C(8)	5(1)	6(1)	4(1)	-1(1)	0(1)	-1(1)
C(9)	5(1)	7(2)	5(1)	1(1)	0(1)	-1(1)
C(10)	4(1)	8(2)	7(1)	0(1)	1(1)	-1(1)
C(11)	6(1)	8(2)	6(1)	-3(1)	2(1)	-3(1)
C(12)	5(1)	8(2)	6(1)	2(1)	1(1)	0(1)
C(13)	6(1)	5(1)	5(1)	2(1)	1(1)	0(1)
H(9)	15(3)	39(8)	15(2)	3(4)	-2(2)	-8(4)
H(10)	14(3)	37(6)	21(3)	7(4)	4(2)	-7(4)
H(12)	18(3)	36(6)	13(2)	5(4)	-7(2)	-11(4)
H(13)	9(3)	35(7)	32(4)	16(5)	6(3)	-4(3)
N(1)	7(1)	12(1)	7(1)	2(1)	3(1)	2(1)
H(1A)	16(3)	24(5)	24(3)	9(4)	9(2)	3(4)
H(1B)	26(4)	36(7)	14(2)	3(4)	0(2)	-17(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **1N.**

	x	y	z	U(eq)
H(1)	12905(17)	22(7)	806(11)	23(2)
H(2)	10260(20)	-86(6)	2316(12)	24(2)
H(3)	7683(17)	528(6)	3598(10)	23(2)
H(5)	9590(20)	2461(7)	1954(12)	26(2)
H(6)	12169(18)	1836(6)	679(9)	21(2)
H(7A)	7290(18)	2479(6)	3677(10)	22(2)
H(7B)	5043(15)	1921(7)	2937(8)	21(2)
H(9)	3185(15)	1266(7)	4435(9)	24(2)
H(10)	2981(15)	820(7)	6801(9)	24(2)
H(12)	9839(15)	1514(7)	8332(8)	24(2)
H(13)	10031(14)	1955(7)	5967(11)	25(2)
H(1A)	4764(15)	839(7)	9332(10)	21(2)
H(1B)	7425(17)	1104(7)	9854(9)	26(2)

Table 6. Torsion angles [°] for **1N.**

O(1)-C(1)-C(2)-C(3)	-177.4(4)
C(6)-C(1)-C(2)-C(3)	2.1(5)
C(1)-C(2)-C(3)-C(4)	-1.7(5)
C(2)-C(3)-C(4)-C(5)	0.5(5)
C(2)-C(3)-C(4)-C(7)	178.0(3)
C(3)-C(4)-C(5)-C(6)	0.2(5)
C(7)-C(4)-C(5)-C(6)	-177.3(3)
O(1)-C(1)-C(6)-C(5)	178.0(4)
C(2)-C(1)-C(6)-C(5)	-1.5(5)
C(4)-C(5)-C(6)-C(1)	0.4(5)
C(5)-C(4)-C(7)-C(8)	-143.3(4)
C(3)-C(4)-C(7)-C(8)	39.3(5)
C(4)-C(7)-C(8)-C(9)	-112.5(4)
C(4)-C(7)-C(8)-C(13)	67.2(5)
C(13)-C(8)-C(9)-C(10)	0.6(6)
C(7)-C(8)-C(9)-C(10)	-179.7(4)
C(8)-C(9)-C(10)-C(11)	-0.1(6)
C(9)-C(10)-C(11)-C(12)	-0.3(6)
C(9)-C(10)-C(11)-N(1)	-176.7(4)
C(10)-C(11)-C(12)-C(13)	0.2(6)
N(1)-C(11)-C(12)-C(13)	176.6(4)
C(11)-C(12)-C(13)-C(8)	0.3(6)
C(9)-C(8)-C(13)-C(12)	-0.7(6)
C(7)-C(8)-C(13)-C(12)	179.6(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Crystal data and structure refinement for **1X.**

Identification code	1_x-ray	
Empirical formula	C13 H13 N O	
Formula weight	199.24	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 5.9820(3) Å	α= 90°.
	b = 19.3670(9) Å	β= 100.860(2)°.
	c = 9.7390(4) Å	γ = 90°.
Volume	1108.09(9) $Å^3$	
Z	4	
Density (calculated)	1.194 Mg/m^3	
Absorption coefficient	0.076 mm^{-1}	
F(000)	424	
Crystal size	0.22 x 0.10 x 0.04 mm^3	
Theta range for data collection	2.10 to 27.48°.	
Index ranges	-7<=h<=7, -25<=k<=25, -12<=l<=12	
Reflections collected	12178	
Independent reflections	2535 [R(int) = 0.0482]	
Completeness to theta = 27.48°	100.0 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2535 / 0 / 188	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.0449, wR2 = 0.1049	
R indices (all data)	R1 = 0.0599, wR2 = 0.1131	
Extinction coefficient	.	
Largest diff. peak and hole	0.266 and -0.211 $e.Å^{-3}$	

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1X.** U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O	7765(2)	524(1)	641(1)	33(1)
N	1353(2)	867(1)	9084(1)	26(1)
C(1)	6363(2)	834(1)	1412(2)	24(1)
C(2)	5121(3)	467(1)	2251(2)	24(1)
C(3)	3665(3)	818(1)	2967(2)	23(1)
C(4)	3420(2)	1535(1)	2877(1)	21(1)
C(5)	4707(3)	1894(1)	2054(2)	24(1)
C(6)	6159(3)	1549(1)	1326(2)	24(1)
C(7)	1775(3)	1922(1)	3617(2)	26(1)
C(8)	1399(2)	1147(1)	7745(2)	22(1)
C(9)	3380(2)	1463(1)	7484(2)	23(1)
C(10)	3480(2)	1708(1)	6159(2)	22(1)
C(11)	1631(2)	1645(1)	5051(2)	21(1)
C(12)	-328(3)	1323(1)	5318(2)	24(1)
C(13)	-451(3)	1075(1)	6648(2)	25(1)

Table 9. Bond lengths [Å] and angles [°] for **1X.**

O-C(1)	1.3662(17)
O-H(1)	0.92(2)
N-C(11)	1.4177(19)
N-H(1A)	0.90(2)
N-H(1B)	0.92(2)
C(1)-C(6)	1.391(2)
C(1)-C(2)	1.398(2)
C(2)-C(3)	1.392(2)
C(2)-H(2)	0.949(19)
C(3)-C(4)	1.397(2)
C(3)-H(3)	0.981(17)
C(4)-C(5)	1.397(2)
C(4)-C(7)	1.522(2)
C(5)-C(6)	1.392(2)
(C5)-H(5)	0.977(18)
C(6)-H(6)	0.962(18)
C(7)-C(8)	1.515(2)
C(7)-H(7B)	0.977(19)
C(7)-H(7A)	1.02(2)
C(11)-C(10)	1.393(2)
C(11)-C(12)	1.399(2)
C(12)-C(13)	1.387(2)
C(12)-H(12)	0.979(17)
C(13)-C(8)	1.397(2)
C(13)-H(13)	0.975(17)
C(8)-C(9)	1.394(2)
C(9)-C(10)	1.396(2)
C(9)-H(9)	0.942(19)
C(10)-H(10)	0.974(19
C(1)-O-H(1)	114.1(14)
C(11)-N-H(1A)	116.4(12)
C(11)-N-H(1B)	114.1(13)
H(1A)-N-H(1B)	108.6(17)
O-C(1)-C(6)	117.41(13)
O-C(1)-C(2)	123.13(13)
C(6)-C(1)-C(2)	119.45(13)
C(3)-C(2)-C(1)	119.71(13)
C(3)-C(2)-H(2)	121.2(11)
C(1)-C(2)-H(2)	119.1(11)
C(2)-C(3)-C(4)	121.50(13)
C(2)-C(3)-H(3)	118.0(10)
C(4)-C(3)-H(3)	120.5(10)
C(3)-C(4)-C(5)	117.94(13)
C(3)-C(4)-C(7)	121.97(13)
C(5)-C(4)-C(7)	120.07(13)
C(6)-C(5)-C(4)	121.11(13)
C(6)-C(5)-H(5)	120.0(10)
C(4)-C(5)-H(5)	118.9(10)
C(1)-C(6)-C(5)	120.26(13)
C(1)-C(6)-H(6)	118.5(10)
C(5)-C(6)-H(6)	121.2(10)
C(8)-C(7)-C(4)	114.57(12)
C(8)-C(7)-H(7B)	106.7(11)

C(4)-C(7)-H(7B)	109.8(11)
C(8)-C(7)-H(7A)	109.4(10)
C(4)-C(7)-H(7A)	108.4(10)
H(7B)-C(7)-H(7A)	107.9(15)
C(10)-C(11)-C(12)	118.83(13)
C(10)-C(11)-N	121.13(14)
C(12)-C(11)-N	119.91(14)
C(13)-C(12)-C(11)	120.34(14)
C(13)-C(12)-H(12)	120.7(10)
C(11)-C(12)-H(12)	119.0(10)
C(12)-C(13)-C(8)	121.46(13)
C(12)-C(13)-H(13)	120.0(10)
C(8)-C(13)-H(13)	118.5(10)
C(9)-C(8)-C(13)	117.79(13)
C(9)-C(8)-C(7)	121.77(14)
C(13)-C(8)-C(7)	120.45(13)
C(8)-C(9)-C(10)	121.32(14)
C(8)-C(9)-H(9)	119.7(11)
C(10)-C(9)-H(9)	118.9(11)
C(11)-C(10)-C(9)	120.26(14)
C(11)-C(10)-H(10)	118.2(11)
C(9)-C(10)-H(10)	121.5(11)

Symmetry transformations used to generate equivalent atoms:

Table 10. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1X.** The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O	43(1)	24(1)	38(1)	2(1)	26(1)	2(1)
N	29(1)	27(1)	26(1)	3(1)	13(1)	0(1)
C(1)	28(1)	24(1)	21(1)	-1(1)	8(1)	0(1)
C(2)	31(1)	17(1)	25(1)	1(1)	9(1)	-1(1)
C(3)	28(1)	22(1)	22(1)	2(1)	10(1)	-1(1)
C(4)	24(1)	22(1)	17(1)	-1(1)	2(1)	2(1)
C(5)	33(1)	17(1)	21(1)	0(1)	3(1)	-1(1)
C(6)	31(1)	22(1)	22(1)	2(1)	8(1)	-5(1)
C(7)	31(1)	25(1)	23(1)	2(1)	6(1)	8(1)
C(11)	27(1)	16(1)	25(1)	0(1)	11(1)	3(1)
C(12)	22(1)	22(1)	24(1)	0(1)	4(1)	-1(1)
C(13)	21(1)	20(1)	27(1)	0(1)	8(1)	0(1)
C(8)	26(1)	17(1)	22(1)	-2(1)	8(1)	6(1)
C(9)	23(1)	22(1)	27(1)	-4(1)	2(1)	3(1)
C(10)	21(1)	21(1)	34(1)	-2(1)	9(1)	-1(1)

Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **1X.**

	x	y	z	U(eq)
H(1)	7930(40)	55(13)	780(20)	56(6)
HA	-20(30)	830(10)	9322(19)	35(5)
HB	2320(40)	1081(11)	9800(20)	43(6)
H(2)	5270(30)	-21(10)	2305(19)	34(5)
H(3)	2770(30)	545(9)	3520(17)	26(4)
H(5)	4600(30)	2398(9)	2010(17)	25(4)
H(6)	7070(30)	1796(9)	774(18)	28(4)
H(7B)	240(30)	1899(9)	3058(19)	35(5)
H(7A)	2250(30)	2429(11)	3699(18)	36(5)
H(12)	4700(30)	1503(9)	8248(18)	26(4)
H(13)	4870(30)	1927(8)	5982(17)	23(4)
H(9)	-1600(30)	1272(9)	4591(19)	32(5)
H(10)	-1820(30)	849(10)	6836(19)	35(5)

Table 12. Torsion angles [°] for **1X.**

O-C(1)-C(2)-C(3)	-177.73(14)
C(6)-C(1)-C(2)-C(3)	1.2(2)
C(1)-C(2)-C(3)-C(4)	-0.3(2)
C(2)-C(3)-C(4)-C(5)	-0.8(2)
C(2)-C(3)-C(4)-C(7)	177.92(14)
C(3)-C(4)-C(5)-C(6)	1.2(2)
C(7)-C(4)-C(5)-C(6)	-177.59(14)
O-C(1)-C(6)-C(5)	178.14(13)
C(2)-C(1)-C(6)-C(5)	-0.8(2)
C(4)-C(5)-C(6)-C(1)	-0.4(2)
C(3)-C(4)-C(7)-C(8)	38.0(2)
C(5)-C(4)-C(7)-C(8)	-143.26(14)
C(10)-C(11)-C(12)-C(13)	0.8(2)
N-C(11)-C(12)-C(13)	176.77(13)
C(11)-C(12)-C(13)-C(8)	-0.3(2)
C(12)-C(13)-C(8)-C(9)	-0.3(2)
C(12)-C(13)-C(8)-C(7)	179.40(13)
C(4)-C(7)-C(8)-C(9)	-112.08(16)
C(4)-C(7)-C(8)-C(13)	68.25(18)
C(13)-C(8)-C(9)-C(10)	0.3(2)
C(7)-C(8)-C(9)-C(10)	-179.36(13)
C(12)-C(11)-C(10)-C(9)	-0.8(2)
N-C(11)-C(10)-C(9)	-176.68(13)
C(8)-C(9)-C(10)-C(11)	0.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 13. Crystal data and structure refinement for **2.**

Identification code	2	
Empirical formula	C14 H15 N O	
Formula weight	213.27	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 13.682(3) Å	α= 90°.
	b = 5.2619(11) Å	β= 107.28(3)°.
	c = 8.1916(16) Å	γ = 90°.
Volume	563.1(2) $Å^3$	
Z	2	
Density (calculated)	1.258 Mg/m^3	
Absorption coefficient	0.079 mm^{-1}	
F(000)	228	
Crystal size	0.50 x 0.35 x 0.10 mm^3	
Theta range for data collection	1.56 to 30.32°.	
Index ranges	-18<=h<=15, -7<=k<=7, -10<=l<=11	
Reflections collected	6709	
Independent reflections	2509 [R(int) = 0.0322]	
Completeness to theta = 30.32°	92.1 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2509 / 2 / 205	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0405, wR2 = 0.1087	
R indices (all data)	R1 = 0.0448, wR2 = 0.1137	
Absolute structure parameter	-0.7(13)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.370 and -0.186 e.$Å^{-3}$	

Table 14. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2.** U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8204(1)	1649(2)	14343(2)	22(1)
C(1)	7180(1)	2022(3)	13471(2)	17(1)
C(2)	6686(1)	4128(3)	13910(2)	19(1)
C(3)	5653(1)	4556(3)	13057(2)	19(1)
C(4)	5096(1)	2910(3)	11762(2)	17(1)
C(5)	5610(1)	825(3)	11345(2)	19(1)
C(6)	6637(1)	382(3)	12179(2)	18(1)
C(7)	3971(1)	3340(3)	10895(2)	19(1)
C(8)	3288(1)	1866(3)	11771(2)	20(1)
C(9)	2156(1)	2150(3)	10851(2)	17(1)
C(10)	1580(1)	4136(3)	11234(2)	19(1)
C(11)	537(1)	4398(3)	10362(2)	18(1)
C(12)	52(1)	2658(3)	9084(2)	17(1)
C(13)	622(1)	674(3)	8690(2)	19(1)
C(14)	1658(1)	441(3)	9564(2)	19(1)
N(1)	-1025(1)	2801(3)	8266(2)	20(1)

Table 15. Bond lengths [Å] and angles [°] for **2**.

O(1)-C(1)	1.385(2)
O(1)-H(1)	0.80(3)
C(1)-C(6)	1.396(2)
C(1)-C(2)	1.399(2)
C(2)-C(3)	1.396(3)
C(2)-H(2)	1.00(2)
C(3)-C(4)	1.405(2)
C(3)-H(3)	0.96(2)
C(4)-C(5)	1.400(2)
C(4)-C(7)	1.508(2)
C(5)-C(6)	1.387(2)
C(5)-H(5)	1.01(3)
C(6)-H(6)	0.97(2)
C(7)-C(8)	1.5452(19)
C(7)-H(8A)	0.99(3)
C(7)-H(8B)	1.01(2)
C(8)-C(9)	1.516(2)
C(8)-H(7A)	1.00(2)
C(8)-H(7B)	0.97(3)
C(9)-C(10)	1.399(2)
C(9)-C(14)	1.399(2)
C(10)-C(11)	1.400(2)
C(10)-H(10)	0.97(2)
C(11)-C(12)	1.401(2)
C(11)-H(11)	0.99(2)
C(12)-C(13)	1.397(2)
C(12)-N(1)	1.428(2)
C(13)-C(14)	1.390(3)
C(13)-H(13)	0.98(3)
C(14)-H(14)	0.99(3)
N(1)-H(1A)	0.97(3)
N(1)-H(1B)	0.91(2)
C(1)-O(1)-H(1)	111(2)

O(1)-C(1)-C(6)	122.17(15)
O(1)-C(1)-C(2)	118.14(15)
C(6)-C(1)-C(2)	119.68(15)
C(3)-C(2)-C(1)	119.56(15)
C(3)-C(2)-H(2)	120.7(13)
C(1)-C(2)-H(2)	119.7(13)
C(2)-C(3)-C(4)	121.32(16)
C(2)-C(3)-H(3)	120.0(16)
C(4)-C(3)-H(3)	118.7(16)
C(5)-C(4)-C(3)	117.90(15)
C(5)-C(4)-C(7)	121.27(15)
C(3)-C(4)-C(7)	120.80(16)
C(6)-C(5)-C(4)	121.37(16)
C(6)-C(5)-H(5)	121.7(15)
C(4)-C(5)-H(5)	116.9(15)
C(5)-C(6)-C(1)	120.17(15)
C(5)-C(6)-H(6)	120.9(16)
C(1)-C(6)-H(6)	118.9(16)
C(4)-C(7)-C(8)	112.31(12)
C(4)-C(7)-H(8A)	111.4(15)
C(8)-C(7)-H(8A)	109.4(14)
C(4)-C(7)-H(8B)	108.7(14)
C(8)-C(7)-H(8B)	105.5(13)
H(8A)-C(7)-H(8B)	109(2)
C(9)-C(8)-C(7)	112.93(12)
C(9)-C(8)-H(7A)	111.6(12)
C(7)-C(8)-H(7A)	106.2(12)
C(9)-C(8)-H(7B)	110.8(15)
C(7)-C(8)-H(7B)	110.2(15)
H(7A)-C(8)-H(7B)	105(2)
C(10)-C(9)-C(14)	117.92(15)
C(10)-C(9)-C(8)	121.52(15)
C(14)-C(9)-C(8)	120.56(15)
C(9)-C(10)-C(11)	121.05(15)
C(9)-C(10)-H(10)	122.1(15)
C(11)-C(10)-H(10)	116.9(15)

C(10)-C(11)-C(12)	120.13(15)
C(10)-C(11)-H(11)	120.7(14)
C(12)-C(11)-H(11)	119.1(14)
C(13)-C(12)-C(11)	119.19(15)
C(13)-C(12)-N(1)	120.01(15)
C(11)-C(12)-N(1)	120.68(15)
C(14)-C(13)-C(12)	120.06(15)
C(14)-C(13)-H(13)	119.4(16)
C(12)-C(13)-H(13)	120.5(16)
C(13)-C(14)-C(9)	121.66(16)
C(13)-C(14)-H(14)	119.0(15)
C(9)-C(14)-H(14)	119.3(15)
C(12)-N(1)-H(1A)	108.2(19)
C(12)-N(1)-H(1B)	111.6(17)
H(1A)-N(1)-H(1B)	114(3)

Symmetry transformations used to generate equivalent atoms:

Table 16. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	15(1)	24(1)	23(1)	-5(1)	1(1)	0(1)
C(1)	16(1)	19(1)	15(1)	3(1)	5(1)	0(1)
C(2)	18(1)	20(1)	16(1)	-2(1)	3(1)	-2(1)
C(3)	19(1)	17(1)	21(1)	0(1)	8(1)	2(1)
C(4)	15(1)	20(1)	17(1)	2(1)	4(1)	-1(1)
C(5)	18(1)	20(1)	17(1)	0(1)	4(1)	0(1)
C(6)	19(1)	18(1)	17(1)	-1(1)	5(1)	0(1)
C(7)	15(1)	23(1)	19(1)	6(1)	4(1)	3(1)
C(8)	16(1)	24(1)	20(1)	3(1)	4(1)	0(1)
C(9)	16(1)	19(1)	17(1)	3(1)	6(1)	0(1)
C(10)	18(1)	18(1)	19(1)	-2(1)	2(1)	-2(1)
C(11)	16(1)	19(1)	21(1)	0(1)	5(1)	2(1)
C(12)	15(1)	19(1)	15(1)	1(1)	3(1)	-2(1)
C(13)	19(1)	20(1)	19(1)	-3(1)	6(1)	-1(1)
C(14)	18(1)	20(1)	19(1)	-2(1)	6(1)	2(1)
N(1)	14(1)	25(1)	21(1)	0(1)	3(1)	1(1)

Table 17. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **2.**

	x	y	z	U(eq)
H(1)	8410(20)	320(50)	14090(40)	31(7)
H(2)	7083(18)	5350(40)	14800(30)	22(6)
H(3)	5317(19)	6040(40)	13320(30)	23(6)
H(5)	5200(20)	-330(50)	10400(30)	31(6)
H(6)	6980(20)	-1100(50)	11920(30)	30(6)
H(7A)	3465(16)	2520(40)	12970(30)	20(5)
H(7B)	3482(19)	90(50)	11890(30)	30(6)
H(8A)	3781(19)	2870(40)	9670(30)	27(6)
H(8B)	3811(19)	5190(50)	11000(30)	26(6)
H(10)	1878(19)	5390(40)	12110(30)	26(6)
H(11)	137(19)	5850(40)	10610(30)	34(7)
H(13)	300(20)	-530(40)	7770(30)	32(6)
H(14)	2050(20)	-1010(50)	9290(40)	35(7)
H(1A)	-1270(20)	4400(50)	8590(40)	46(8)
H(1B)	-1180(20)	2570(50)	7120(30)	39(6)

Table 18. Torsion angles [°] for **2.**

O(1)-C(1)-C(2)-C(3)	-179.93(15)
C(6)-C(1)-C(2)-C(3)	0.4(2)
C(1)-C(2)-C(3)-C(4)	0.0(3)
C(2)-C(3)-C(4)-C(5)	-0.3(3)
C(2)-C(3)-C(4)-C(7)	177.74(15)
C(3)-C(4)-C(5)-C(6)	0.1(3)
C(7)-C(4)-C(5)-C(6)	-177.95(16)
C(4)-C(5)-C(6)-C(1)	0.4(3)
O(1)-C(1)-C(6)-C(5)	179.72(15)
C(2)-C(1)-C(6)-C(5)	-0.7(3)
C(5)-C(4)-C(7)-C(8)	85.66(19)
C(3)-C(4)-C(7)-C(8)	-92.31(18)
C(4)-C(7)-C(8)-C(9)	-176.50(17)
C(7)-C(8)-C(9)-C(10)	-88.87(18)
C(7)-C(8)-C(9)-C(14)	90.08(18)
C(14)-C(9)-C(10)-C(11)	0.2(2)
C(8)-C(9)-C(10)-C(11)	179.21(16)
C(9)-C(10)-C(11)-C(12)	0.1(3)
C(10)-C(11)-C(12)-C(13)	-0.3(3)
C(10)-C(11)-C(12)-N(1)	175.71(15)
C(11)-C(12)-C(13)-C(14)	0.2(3)
N(1)-C(12)-C(13)-C(14)	-175.89(15)
C(12)-C(13)-C(14)-C(9)	0.2(3)
C(10)-C(9)-C(14)-C(13)	-0.4(3)
C(8)-C(9)-C(14)-C(13)	-179.38(16)

Symmetry transformations used to generate equivalent atoms:

Table 19. Crystal data and structure refinement for **3**.

Identification code	3	
Empirical formula	C15 H17 N O	
Formula weight	227.30	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pca2(1)	
Unit cell dimensions	a = 23.9370(7) Å	α= 90°.
	b = 6.2160(2) Å	β= 90°.
	c = 8.3970(3) Å	γ = 90°.
Volume	1249.41(7) $Å^3$	
Z	4	
Density (calculated)	1.208 Mg/m^3	
Absorption coefficient	0.075 mm^{-1}	
F(000)	488	
Crystal size	0.52 x 0.25 x 0.07 mm^3	
Theta range for data collection	1.70 to 28.27°.	
Index ranges	-31<=h<=31, -5<=k<=8, -11<=l<=10	
Reflections collected	8492	
Independent reflections	3069 [R(int) = 0.0365]	
Completeness to theta = 28.27°	100.0 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3069 / 1 / 223	
Goodness-of-fit on F^2	1.016	
Final R indices [I>2sigma(I)]	R1 = 0.0389, wR2 = 0.0856	
R indices (all data)	R1 = 0.0510, wR2 = 0.0918	
Absolute structure parameter	0.3(15)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.205 and -0.159 e.$Å^{-3}$	

Table 20. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3091(1)	6252(2)	8976(2)	30(1)
C(1)	3602(1)	5501(3)	8474(2)	25(1)
C(2)	3776(1)	3403(3)	8759(2)	26(1)
C(3)	4287(1)	2707(3)	8160(2)	**25(1)**
C(4)	4634(1)	4049(3)	7282(2)	23(1)
C(5)	4457(1)	6161(3)	7039(2)	25(1)
C(6)	3942(1)	6890(3)	7620(2)	26(1)
C(7)	5171(1)	3205(3)	6551(2)	27(1)
C(8)	5562(1)	2066(3)	7723(2)	24(1)
C(9)	6078(1)	1120(3)	6910(2)	32(1)
C(10)	6451(1)	-143(3)	8018(2)	26(1)
C(11)	6298(1)	-2195(3)	8552(2)	27(1)
C(12)	6636(1)	-3346(3)	9589(2)	26(1)
C(13)	7136(1)	-2478(3)	10137(2)	24(1)
C(14)	7295(1)	-434(3)	9621(2)	26(1)
C(15)	6952(1)	704(3)	8573(2)	28(1)
N(1)	7502(1)	-3713(3)	11117(2)	27(1)

Table 21. Bond lengths [Å] and angles [°] for **3**.

O(1)-C(1)	1.3756(19)
O(1)-H(1)	0.91(2)
C(1)-C(6)	1.387(2)
C(1)-C(2)	1.390(2)
C(2)-C(3)	1.390(2)
C(2)-H(2)	0.943(19)
C(3)-C(4)	1.390(2)
C(3)-H(3)	0.93(2)
C(4)-C(5)	1.396(2)
C(4)-C(7)	1.516(2)
C(5)-C(6)	1.399(2)
C(5)-H(5)	0.95(2)
C(6)-H(6)	0.960(18)
C(7)-C(8)	1.532(2)
C(7)-H(7A)	0.97(2)
C(7)-H(7B)	0.98(2)
C(8)-C(9)	1.529(2)
C(8)-H(8A)	0.95(2)
C(8)-H(8B)	0.99(2)
C(9)-C(10)	1.510(2)
C(9)-H(9A)	0.98(2)
C(9)-H(9B)	0.99(3)
C(10)-C(15)	1.391(2)
C(10)-C(11)	1.400(2)
C(11)-C(12)	1.387(2)
C(11)-H(11)	0.942(19)
C(12)-C(13)	1.393(2)
C(12)-H(12)	0.96(2)
C(13)-C(14)	1.395(2)
C(13)-N(1)	1.425(2)
C(14)-C(15)	1.395(3)
C(14)-H(14)	0.96(2)
C(15)-H(15)	0.95(2)
N(1)-H(1A)	0.85(2)
N(1)-H(1B)	0.90(2)
C(1)-O(1)-H(1)	112.4(14)
O(1)-C(1)-C(6)	118.05(15)
O(1)-C(1)-C(2)	122.21(15)
C(6)-C(1)-C(2)	119.72(15)
C(3)-C(2)-C(1)	119.59(16)
C(3)-C(2)-H(2)	119.6(11)
C(1)-C(2)-H(2)	120.8(11)
C(4)-C(3)-C(2)	122.12(16)
C(4)-C(3)-H(3)	120.4(13)
C(2)-C(3)-H(3)	117.4(13)
C(3)-C(4)-C(5)	117.34(15)
C(3)-C(4)-C(7)	120.95(15)
C(5)-C(4)-C(7)	121.64(15)
C(4)-C(5)-C(6)	121.47(15)
C(4)-C(5)-H(5)	118.0(12)
C(6)-C(5)-H(5)	120.5(12)
C(1)-C(6)-C(5)	119.73(15)
C(1)-C(6)-H(6)	120.2(11)

C(5)-C(6)-H(6)	120.0(11)
C(4)-C(7)-C(8)	114.63(14)
C(4)-C(7)-H(7A)	109.0(13)
C(8)-C(7)-H(7A)	109.5(13)
C(4)-C(7)-H(7B)	108.2(13)
C(8)-C(7)-H(7B)	109.3(12)
H(7A)-C(7)-H(7B)	105.9(18)
C(9)-C(8)-C(7)	112.60(14)
C(9)-C(8)-H(8A)	110.3(11)
C(7)-C(8)-H(8A)	110.0(12)
C(9)-C(8)-H(8B)	112.0(12)
C(7)-C(8)-H(8B)	108.4(12)
H(8A)-C(8)-H(8B)	103.2(16)
C(10)-C(9)-C(8)	113.74(14)
C(10)-C(9)-H(9A)	110.1(13)
C(8)-C(9)-H(9A)	109.2(13)
C(10)-C(9)-H(9B)	111.4(14)
C(8)-C(9)-H(9B)	107.0(14)
H(9A)-C(9)-H(9B)	105(2)
C(15)-C(10)-C(11)	117.58(15)
C(15)-C(10)-C(9)	121.31(16)
C(11)-C(10)-C(9)	121.10(16)
C(12)-C(11)-C(10)	121.27(15)
C(12)-C(11)-H(11)	120.2(12)
C(10)-C(11)-H(11)	118.4(12)
C(11)-C(12)-C(13)	120.61(16)
C(11)-C(12)-H(12)	119.8(11)
C(13)-C(12)-H(12)	119.4(11)
C(12)-C(13)-C(14)	118.91(15)
C(12)-C(13)-N(1)	120.72(16)
C(14)-C(13)-N(1)	120.21(15)
C(13)-C(14)-C(15)	119.92(16)
C(13)-C(14)-H(14)	120.1(13)
C(15)-C(14)-H(14)	120.0(13)
C(10)-C(15)-C(14)	121.72(16)
C(10)-C(15)-H(15)	119.6(11)
C(14)-C(15)-H(15)	118.7(11)
C(13)-N(1)-H(1A)	111.8(16)
C(13)-N(1)-H(1B)	111.3(12)
H(1A)-N(1)-H(1B)	113(2)

Symmetry transformations used to generate equivalent atoms:

Table 22. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3.** The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	23(1)	31(1)	38(1)	-2(1)	2(1)	4(1)
C(1)	21(1)	28(1)	24(1)	-5(1)	-4(1)	0(1)
C(2)	23(1)	27(1)	29(1)	-1(1)	0(1)	-2(1)
C(3)	24(1)	21(1)	30(1)	0(1)	1(1)	0(1)
C(4)	21(1)	27(1)	21(1)	-1(1)	-2(1)	1(1)
C(5)	26(1)	28(1)	22(1)	0(1)	-5(1)	-3(1)
C(6)	29(1)	21(1)	28(1)	-2(1)	-8(1)	3(1)
C(7)	24(1)	33(1)	23(1)	3(1)	3(1)	4(1)
C(8)	23(1)	29(1)	21(1)	0(1)	1(1)	1(1)
C(9)	29(1)	43(1)	23(1)	4(1)	4(1)	8(1)
C(10)	24(1)	34(1)	21(1)	1(1)	5(1)	5(1)
C(11)	19(1)	37(1)	25(1)	-2(1)	2(1)	-4(1)
C(12)	24(1)	27(1)	25(1)	3(1)	4(1)	-4(1)
C(13)	22(1)	30(1)	21(1)	-1(1)	4(1)	2(1)
C(14)	21(1)	29(1)	29(1)	0(1)	0(1)	-4(1)
C(15)	28(1)	25(1)	30(1)	2(1)	6(1)	-1(1)
N(1)	23(1)	33(1)	25(1)	6(1)	-1(1)	-2(1)

Table 23. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **3**.

	x	y	z	U(eq)
H(1)	2923(9)	5350(40)	9670(30)	40(6)
H(2)	3551(8)	2440(30)	9340(20)	26(5)
H(3)	4383(8)	1270(30)	8330(30)	35(5)
H(5)	4698(8)	7110(30)	6480(20)	27(5)
H(6)	3824(8)	8340(30)	7420(20)	27(5)
H(7A)	5367(9)	4390(40)	6050(30)	41(6)
H(7B)	5072(9)	2210(30)	5690(30)	35(5)
H(8A)	5670(8)	3030(30)	8550(20)	29(5)
H(8B)	5345(8)	960(30)	8300(20)	37(6)
H(9A)	6290(9)	2280(40)	6400(30)	47(6)
H(9B)	5943(10)	220(40)	6020(30)	54(7)
H(11)	5949(8)	-2750(30)	8240(20)	32(5)
H(12)	6513(8)	-4720(30)	9990(20)	28(5)
H(14)	7643(9)	170(30)	9960(30)	36(6)
H(15)	7066(7)	2090(30)	8240(20)	28(5)
H(1A)	7706(9)	-2920(40)	11690(30)	46(7)
H(1B)	7314(8)	-4710(30)	11670(20)	29(5)

Table 24. Torsion angles [°] for **3**.

O(1)-C(1)-C(2)-C(3)	177.09(15)
C(6)-C(1)-C(2)-C(3)	-1.2(2)
C(1)-C(2)-C(3)-C(4)	0.3(3)
C(2)-C(3)-C(4)-C(5)	1.0(2)
C(2)-C(3)-C(4)-C(7)	-175.94(16)
C(3)-C(4)-C(5)-C(6)	-1.6(2)
C(7)-C(4)-C(5)-C(6)	175.38(15)
O(1)-C(1)-C(6)-C(5)	-177.69(15)
C(2)-C(1)-C(6)-C(5)	0.7(2)
C(4)-C(5)-C(6)-C(1)	0.7(2)
C(3)-C(4)-C(7)-C(8)	-53.2(2)
C(5)-C(4)-C(7)-C(8)	129.92(17)
C(4)-C(7)-C(8)-C(9)	176.32(15)
C(7)-C(8)-C(9)-C(10)	-176.04(16)
C(8)-C(9)-C(10)-C(15)	-104.29(19)
C(8)-C(9)-C(10)-C(11)	74.4(2)
C(15)-C(10)-C(11)-C(12)	-0.3(2)
C(9)-C(10)-C(11)-C(12)	-178.98(16)
C(10)-C(11)-C(12)-C(13)	0.4(2)
C(11)-C(12)-C(13)-C(14)	-0.3(2)
C(11)-C(12)-C(13)-N(1)	-175.72(15)
C(12)-C(13)-C(14)-C(15)	0.0(2)
N(1)-C(13)-C(14)-C(15)	175.50(16)
C(11)-C(10)-C(15)-C(14)	0.0(3)
C(9)-C(10)-C(15)-C(14)	178.74(15)
C(13)-C(14)-C(15)-C(10)	0.1(3)

Symmetry transformations used to generate equivalent atoms:

Table 25. Crystal data and structure refinement for **4**.

Identification code	4	
Empirical formula	C16 H19 N O	
Formula weight	241.32	
Temperature	105(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 15.7888(16) Å	α= 90°.
	b = 5.2088(6) Å	β= 100.912(5)°.
	c = 8.3399(8) Å	γ = 90°.
Volume	673.48(12) $Å^3$	
Z	2	
Density (calculated)	1.190 Mg/m^3	
Absorption coefficient	0.074 mm^{-1}	
F(000)	260	
Crystal size	0.40 x 0.20 x 0.20 mm^3	
Theta range for data collection	2.63 to 27.54°.	
Index ranges	-20<=h<=19, -6<=k<=5, -10<=l<=10	
Reflections collected	4048	
Independent reflections	2816 [R(int) = 0.0546]	
Completeness to theta = 27.54°	99.7 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2816 / 2 / 240	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.0418, wR2 = 0.0921	
R indices (all data)	R1 = 0.0615, wR2 = 0.1038	
Absolute structure parameter	0.1(19)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.200 and -0.181 $e.Å^{-3}$	

Table 26. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **4**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	2503(1)	2900(4)	3231(3)	26(1)
O(1)	11862(1)	1547(4)	6491(2)	27(1)
C(1)	10994(2)	1972(5)	6492(3)	21(1)
C(2)	10516(2)	372(5)	7324(3)	22(1)
C(3)	9652(2)	871(5)	7286(3)	23(1)
C(4)	9237(2)	2979(5)	6437(3)	22(1)
C(5)	9728(2)	4570(6)	5608(4)	25(1)
C(6)	10599(2)	4087(5)	5631(3)	22(1)
C(7)	8284(2)	3506(6)	6369(3)	26(1)
C(8)	7691(2)	2193(6)	4928(4)	24(1)
C(9)	6739(2)	2942(5)	4786(4)	25(1)
C(10)	6151(2)	1651(6)	3318(4)	27(1)
C(11)	3418(2)	2711(5)	3313(3)	20(1)
C(12)	3883(2)	705(5)	4188(3)	24(1)
C(13)	4753(2)	414(5)	4168(3)	23(1)
C(14)	5193(2)	2067(5)	3295(3)	22(1)
C(15)	4728(2)	4079(5)	2431(3)	24(1)
C(16)	3843(2)	4405(5)	2438(3)	23(1)

Table 27. Bond lengths [Å] and angles [°] for **4**.

N(1)-C(11)	1.438(3)
N(1)-H(1A)	0.99(3)
N(1)-H(1B)	0.90(3)
O(1)-C(1)	1.388(3)
O(1)-H(1)	0.88(6)
C(1)-C(2)	1.394(4)
C(1)-C(6)	1.397(4)
C(2)-C(3)	1.383(4)
C(2)-H(2)	1.01(4)
C(3)-C(4)	1.400(4)
C(3)-H(3)	0.97(3)
C(4)-C(5)	1.404(4)
C(4)-C(7)	1.520(4)
C(5)-C(6)	1.394(4)
C(5)-H(5)	0.91(3)
C(6)-H(6)	1.06(3)
C(7)-C(8)	1.537(4)
C(7)-H(7A)	1.04(3)
C(7)-H(7B)	1.03(3)
C(8)-C(9)	1.535(2)
C(8)-H(8A)	1.00(4)
C(8)-H(8B)	1.01(3)
C(9)-C(10)	1.544(4)
C(9)-H(9A)	1.00(3)
C(9)-H(9B)	0.97(3)
C(10)-C(14)	1.524(4)
C(10)-H(10A)	0.98(3)
C(10)-H(10B)	0.98(3)
C(11)-C(16)	1.396(4)
C(11)-C(12)	1.400(4)
C(12)-C(13)	1.385(4)
C(12)-H(12)	0.92(3)
C(13)-C(14)	1.396(4)
C(13)-H(13)	0.98(3)
C(14)-C(15)	1.399(4)
C(15)-C(16)	1.408(4)
C(15)-H(15)	1.00(3)
C(16)-H(16)	0.97(3)
C(11)-N(1)-H(1A)	107.9(16)
C(11)-N(1)-H(1B)	111.0(17)
H(1A)-N(1)-H(1B)	117(2)
C(1)-O(1)-H(1)	111(4)
O(1)-C(1)-C(2)	122.2(2)
O(1)-C(1)-C(6)	118.1(2)
C(2)-C(1)-C(6)	119.7(3)
C(3)-C(2)-C(1)	120.1(2)
C(3)-C(2)-H(2)	121(2)
C(1)-C(2)-H(2)	119(2)
C(2)-C(3)-C(4)	121.6(2)
C(2)-C(3)-H(3)	121.0(18)
C(4)-C(3)-H(3)	117.3(18)
C(3)-C(4)-C(5)	117.5(3)
C(3)-C(4)-C(7)	121.8(3)

C(5)-C(4)-C(7)	120.7(3)
C(6)-C(5)-C(4)	121.5(3)
C(6)-C(5)-H(5)	118.3(19)
C(4)-C(5)-H(5)	120.1(19)
C(5)-C(6)-C(1)	119.5(3)
C(5)-C(6)-H(6)	120.3(14)
C(1)-C(6)-H(6)	120.2(14)
C(4)-C(7)-C(8)	113.6(2)
C(4)-C(7)-H(7A)	111.6(17)
C(8)-C(7)-H(7A)	108.9(17)
C(4)-C(7)-H(7B)	106.3(14)
C(8)-C(7)-H(7B)	106.7(14)
H(7A)-C(7)-H(7B)	110(2)
C(9)-C(8)-C(7)	112.91(18)
C(9)-C(8)-H(8A)	110.9(18)
C(7)-C(8)-H(8A)	114.1(18)
C(9)-C(8)-H(8B)	109.2(17)
C(7)-C(8)-H(8B)	107.3(17)
H(8A)-C(8)-H(8B)	102(2)
C(8)-C(9)-C(10)	112.57(19)
C(8)-C(9)-H(9A)	108.5(15)
C(10)-C(9)-H(9A)	106.1(14)
C(8)-C(9)-H(9B)	109.6(17)
C(10)-C(9)-H(9B)	111.3(17)
H(9A)-C(9)-H(9B)	109(2)
C(14)-C(10)-C(9)	113.2(2)
C(14)-C(10)-H(10A)	110.9(17)
C(9)-C(10)-H(10A)	113.9(17)
C(14)-C(10)-H(10B)	108.0(17)
C(9)-C(10)-H(10B)	107.3(16)
H(10A)-C(10)-H(10B)	103(2)
C(16)-C(11)-C(12)	119.2(2)
C(16)-C(11)-N(1)	120.7(3)
C(12)-C(11)-N(1)	119.9(2)
C(13)-C(12)-C(11)	119.9(3)
C(13)-C(12)-H(12)	118.2(18)
C(11)-C(12)-H(12)	121.9(19)
C(12)-C(13)-C(14)	122.1(3)
C(12)-C(13)-H(13)	119.5(19)
C(14)-C(13)-H(13)	118.2(19)
C(13)-C(14)-C(15)	117.8(3)
C(13)-C(14)-C(10)	119.8(2)
C(15)-C(14)-C(10)	122.4(3)
C(14)-C(15)-C(16)	120.9(3)
C(14)-C(15)-H(15)	122.1(16)
C(16)-C(15)-H(15)	117.0(16)
C(11)-C(16)-C(15)	120.1(2)
C(11)-C(16)-H(16)	120.7(17)
C(15)-C(16)-H(16)	119.1(17)

Symmetry transformations used to generate equivalent atoms:

Table 28. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **4**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	15(1)	33(1)	29(1)	1(1)	3(1)	1(1)
O(1)	18(1)	32(1)	32(1)	4(1)	5(1)	-1(1)
C(1)	17(1)	27(1)	19(1)	-2(1)	-1(1)	2(1)
C(2)	23(1)	23(1)	19(1)	3(1)	2(1)	-1(1)
C(3)	20(1)	26(1)	24(2)	1(1)	6(1)	-2(1)
C(4)	18(1)	27(1)	19(2)	-6(1)	2(1)	0(1)
C(5)	26(2)	25(1)	22(2)	-1(1)	1(1)	4(1)
C(6)	23(1)	26(1)	20(1)	-1(1)	5(1)	-2(1)
C(7)	20(1)	32(1)	24(2)	-6(1)	4(1)	4(1)
C(8)	20(1)	27(1)	25(2)	-2(1)	4(1)	2(1)
C(9)	17(1)	31(2)	25(2)	0(1)	4(1)	1(1)
C(10)	18(1)	35(2)	28(2)	-4(1)	4(1)	0(1)
C(11)	16(1)	26(1)	19(1)	-5(1)	4(1)	-5(1)
C(12)	23(1)	24(1)	24(2)	2(1)	3(1)	-1(1)
C(13)	21(1)	26(1)	20(1)	1(1)	0(1)	3(1)
C(14)	18(1)	26(1)	23(2)	-6(1)	2(1)	0(1)
C(15)	24(1)	24(1)	23(2)	-1(1)	5(1)	-4(1)
C(16)	17(1)	25(1)	25(1)	-2(1)	1(1)	3(1)

Table 29. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **4**.

	x	y	z	U(eq)
H(1A)	2308(17)	4550(50)	2710(30)	20(6)
H(1B)	2365(17)	2580(50)	4210(30)	34(7)
H(1)	12010(40)	-20(120)	6810(70)	140(20)
H(2)	10800(20)	-1170(60)	7930(40)	40(9)
H(3)	9300(20)	-290(60)	7800(40)	32(8)
H(5)	9481(19)	5960(60)	5040(40)	26(8)
H(6)	10954(17)	5290(50)	4970(30)	16(7)
H(7A)	8090(20)	2970(50)	7450(40)	31(8)
H(7B)	8202(16)	5450(50)	6200(30)	18(6)
H(8A)	7760(20)	290(70)	4900(40)	43(10)
H(8B)	7895(19)	2730(50)	3900(40)	27(8)
H(9A)	6683(17)	4840(50)	4590(30)	18(7)
H(9B)	6550(19)	2550(50)	5810(40)	23(7)
H(10A)	6275(18)	-170(60)	3190(40)	32(8)
H(10B)	6288(19)	2420(50)	2320(40)	19(7)
H(12)	3624(19)	-480(60)	4750(40)	32(8)
H(13)	5060(20)	-1060(60)	4720(40)	33(8)
H(15)	4997(19)	5310(50)	1760(40)	22(7)
H(16)	3548(19)	5880(50)	1900(40)	21(7)

Table 30. Torsion angles [°] for **4**.

O(1)-C(1)-C(2)-C(3)	-179.9(2)
C(6)-C(1)-C(2)-C(3)	0.4(4)
C(1)-C(2)-C(3)-C(4)	-0.6(4)
C(2)-C(3)-C(4)-C(5)	0.5(4)
C(2)-C(3)-C(4)-C(7)	178.9(3)
C(3)-C(4)-C(5)-C(6)	-0.3(4)
C(7)-C(4)-C(5)-C(6)	-178.7(2)
C(4)-C(5)-C(6)-C(1)	0.2(4)
O(1)-C(1)-C(6)-C(5)	-179.9(3)
C(2)-C(1)-C(6)-C(5)	-0.2(4)
C(3)-C(4)-C(7)-C(8)	-89.5(3)
C(5)-C(4)-C(7)-C(8)	88.9(3)
C(4)-C(7)-C(8)-C(9)	-174.8(2)
C(7)-C(8)-C(9)-C(10)	179.0(3)
C(8)-C(9)-C(10)-C(14)	171.9(2)
C(16)-C(11)-C(12)-C(13)	-0.3(4)
N(1)-C(11)-C(12)-C(13)	175.3(3)
C(11)-C(12)-C(13)-C(14)	-0.1(4)
C(12)-C(13)-C(14)-C(15)	0.5(4)
C(12)-C(13)-C(14)-C(10)	-179.9(3)
C(9)-C(10)-C(14)-C(13)	-85.9(3)
C(9)-C(10)-C(14)-C(15)	93.6(3)
C(13)-C(14)-C(15)-C(16)	-0.4(4)
C(10)-C(14)-C(15)-C(16)	-180.0(3)
C(12)-C(11)-C(16)-C(15)	0.4(4)
N(1)-C(11)-C(16)-C(15)	-175.2(2)
C(14)-C(15)-C(16)-C(11)	0.0(4)

Symmetry transformations used to generate equivalent atoms:

Table 31. Crystal data and structure refinement for **5**.

Identification code	5	
Empirical formula	C17 H21 N O	
Formula weight	255.35	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 14.9554(9) Å	α= 90°.
	b = 11.2370(8) Å	β= 90.893(3)°.
	c = 8.6841(6) Å	γ = 90°.
Volume	1459.22(17) $Å^3$	
Z	4	
Density (calculated)	1.162 Mg/m^3	
Absorption coefficient	0.071 mm^{-1}	
F(000)	552	
Crystal size	0.60 x 0.55 x 0.30 mm^3	
Theta range for data collection	2.27 to 27.55°.	
Index ranges	-19<=h<=19, -11<=k<=14, -11<=l<=11	
Reflections collected	9991	
Independent reflections	5476 [R(int) = 0.0215]	
Completeness to theta = 27.55°	99.6 %	
Absorption correction	Psi-scan	
Max. and min. transmission	0.99477 and 0.89622	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5476 / 2 / 367	
Goodness-of-fit on F^2	1.033	
Final R indices [I>2sigma(I)]	R1 = 0.0355, wR2 = 0.0885	
R indices (all data)	R1 = 0.0418, wR2 = 0.0938	
Absolute structure parameter	-0.2(11)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.252 and -0.160 $e.Å^{-3}$	

Table 32. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	18714(1)	6720(1)	636(2)	38(1)
C(1)	17914(1)	6397(2)	-47(2)	30(1)
C(2)	17506(1)	5312(2)	257(2)	29(1)
C(3)	16713(1)	5019(2)	-500(2)	30(1)
C(4)	16299(1)	5791(2)	-1547(2)	30(1)
C(5)	16721(1)	6871(2)	-1837(2)	37(1)
C(6)	17522(1)	7174(2)	-1107(2)	36(1)
C(7)	15441(1)	5428(2)	-2362(2)	35(1)
C(8)	14582(1)	5618(2)	-1442(2)	31(1)
C(9)	14380(1)	6928(2)	-1123(2)	28(1)
C(10)	13428(1)	7113(2)	-545(2)	27(1)
C(11)	13162(1)	8426(2)	-443(2)	26(1)
C(12)	12212(1)	8621(1)	90(2)	24(1)
C(13)	11499(1)	7944(2)	-504(2)	28(1)
C(14)	10630(1)	8094(2)	28(2)	29(1)
C(15)	10452(1)	8917(1)	1171(2)	26(1)
C(16)	11146(1)	9633(2)	1735(2)	29(1)
C(17)	12011(1)	9476(1)	1189(2)	28(1)
N(1)	9560(1)	9069(1)	1715(2)	32(1)
O(21)	28910(1)	1044(1)	181(2)	37(1)
C(21)	28013(1)	1060(2)	-189(2)	29(1)
C(22)	27414(1)	219(2)	371(2)	31(1)
C(23)	26524(1)	257(2)	-101(2)	33(1)
C(24)	26206(1)	1121(2)	-1127(2)	29(1)
C(25)	26814(1)	1964(2)	-1646(2)	33(1)
C(26)	27703(1)	1945(2)	-1177(2)	33(1)
C(27)	25235(1)	1162(2)	-1624(2)	35(1)
C(28)	24639(1)	1813(2)	-486(2)	30(1)
C(29)	23683(1)	1956(2)	-1076(2)	33(1)
C(30)	23051(1)	2622(2)	-14(2)	28(1)
C(31)	22137(1)	2800(2)	-777(2)	44(1)
C(32)	21444(1)	3419(2)	188(2)	30(1)
C(33)	20646(1)	2855(2)	556(2)	31(1)
C(34)	20000(1)	3415(2)	1428(2)	29(1)
C(35)	20143(1)	4564(2)	1972(2)	26(1)
C(36)	20930(1)	5153(2)	1584(2)	29(1)
C(37)	21568(1)	4583(2)	715(2)	31(1)
N(21)	19474(1)	5186(2)	2814(2)	34(1)

Table 33. Bond lengths [Å] and angles [°] for **5**.

O(1)-C(1)	1.375(2)
O(1)-H(1)	0.85(3)
C(1)-C(2)	1.391(2)
C(1)-C(6)	1.392(3)
C(2)-C(3)	1.386(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.396(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.392(3)
C(4)-C(7)	1.512(3)
C(5)-C(6)	1.388(3)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
C(7)-C(8)	1.539(3)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(9)	1.529(2)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.530(2)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-C(11)	1.532(2)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.517(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(17)	1.390(2)
C(12)-C(13)	1.402(2)
C(13)-C(14)	1.395(2)
C(13)-H(13)	0.9500
C(14)-C(15)	1.386(2)
C(14)-H(14)	0.9500
C(15)-C(16)	1.395(2)

C(15)-N(1)	1.432(2)
C(16)-C(17)	1.397(2)
C(16)-H(16)	0.9500
C(17)-H(17)	0.9500
N(1)-H(1B)	0.92(3)
N(1)-H(1A)	0.90(3)
O(21)-C(21)	1.374(2)
O(21)-H(21)	0.89(3)
C(21)-C(26)	1.389(3)
C(21)-C(22)	1.395(2)
C(22)-C(23)	1.388(3)
C(22)-H(22)	0.9500
C(23)-C(24)	1.396(3)
C(23)-H(23)	0.9500
C(24)-C(25)	1.393(3)
C(24)-C(27)	1.509(2)
C(25)-C(26)	1.385(3)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500
C(27)-C(28)	1.527(2)
C(27)-H(27A)	0.9900
C(27)-H(27B)	0.9900
C(28)-C(29)	1.520(2)
C(28)-H(28A)	0.9900
C(28)-H(28B)	0.9900
C(29)-C(30)	1.527(2)
C(29)-H(29A)	0.9900
C(29)-H(29B)	0.9900
C(30)-C(31)	1.523(3)
C(30)-H(30A)	0.9900
C(30)-H(30B)	0.9900
C(31)-C(32)	1.512(3)
C(31)-H(31A)	0.9900
C(31)-H(31B)	0.9900
C(32)-C(33)	1.392(3)
C(32)-C(37)	1.397(3)
C(33)-C(34)	1.388(3)

C(33)-H(33)	0.9500
C(34)-C(35)	1.390(2)
C(34)-H(34)	0.9500
C(35)-C(36)	1.397(2)
C(35)-N(21)	1.430(2)
C(36)-C(37)	1.383(3)
C(36)-H(36)	0.9500
C(37)-H(37)	0.9500
N(21)-H(21A)	0.90(3)
N(21)-H(21B)	0.86(3)

C(1)-O(1)-H(1)	111.9(19)
O(1)-C(1)-C(2)	122.08(16)
O(1)-C(1)-C(6)	118.31(17)
C(2)-C(1)-C(6)	119.58(17)
C(3)-C(2)-C(1)	119.57(16)
C(3)-C(2)-H(2)	120.2
C(1)-C(2)-H(2)	120.2
C(2)-C(3)-C(4)	121.91(17)
C(2)-C(3)-H(3)	119.0
C(4)-C(3)-H(3)	119.0
C(5)-C(4)-C(3)	117.50(17)
C(5)-C(4)-C(7)	122.36(17)
C(3)-C(4)-C(7)	120.11(17)
C(6)-C(5)-C(4)	121.47(17)
C(6)-C(5)-H(5)	119.3
C(4)-C(5)-H(5)	119.3
C(5)-C(6)-C(1)	119.96(17)
C(5)-C(6)-H(6)	120.0
C(1)-C(6)-H(6)	120.0
C(4)-C(7)-C(8)	115.39(15)
C(4)-C(7)-H(7A)	108.4
C(8)-C(7)-H(7A)	108.4
C(4)-C(7)-H(7B)	108.4
C(8)-C(7)-H(7B)	108.4
H(7A)-C(7)-H(7B)	107.5
C(9)-C(8)-C(7)	113.38(15)

C(9)-C(8)-H(8A)	108.9
C(7)-C(8)-H(8A)	108.9
C(9)-C(8)-H(8B)	108.9
C(7)-C(8)-H(8B)	108.9
H(8A)-C(8)-H(8B)	107.7
C(8)-C(9)-C(10)	112.19(14)
C(8)-C(9)-H(9A)	109.2
C(10)-C(9)-H(9A)	109.2
C(8)-C(9)-H(9B)	109.2
C(10)-C(9)-H(9B)	109.2
H(9A)-C(9)-H(9B)	107.9
C(9)-C(10)-C(11)	113.20(14)
C(9)-C(10)-H(10A)	108.9
C(11)-C(10)-H(10A)	108.9
C(9)-C(10)-H(10B)	108.9
C(11)-C(10)-H(10B)	108.9
H(10A)-C(10)-H(10B)	107.8
C(12)-C(11)-C(10)	113.73(13)
C(12)-C(11)-H(11A)	108.8
C(10)-C(11)-H(11A)	108.8
C(12)-C(11)-H(11B)	108.8
C(10)-C(11)-H(11B)	108.8
H(11A)-C(11)-H(11B)	107.7
C(17)-C(12)-C(13)	117.18(16)
C(17)-C(12)-C(11)	121.48(15)
C(13)-C(12)-C(11)	121.34(15)
C(14)-C(13)-C(12)	121.29(16)
C(14)-C(13)-H(13)	119.4
C(12)-C(13)-H(13)	119.4
C(15)-C(14)-C(13)	120.51(15)
C(15)-C(14)-H(14)	119.7
C(13)-C(14)-H(14)	119.7
C(14)-C(15)-C(16)	119.11(16)
C(14)-C(15)-N(1)	120.41(15)
C(16)-C(15)-N(1)	120.41(16)
C(15)-C(16)-C(17)	119.70(15)
C(15)-C(16)-H(16)	120.1

C(17)-C(16)-H(16)	120.1
C(12)-C(17)-C(16)	122.09(15)
C(12)-C(17)-H(17)	119.0
C(16)-C(17)-H(17)	119.0
C(15)-N(1)-H(1B)	111.5(16)
C(15)-N(1)-H(1A)	113.1(17)
H(1B)-N(1)-H(1A)	110(2)
C(21)-O(21)-H(21)	109.6(19)
O(21)-C(21)-C(26)	117.94(16)
O(21)-C(21)-C(22)	122.66(16)
C(26)-C(21)-C(22)	119.40(17)
C(23)-C(22)-C(21)	119.65(16)
C(23)-C(22)-H(22)	120.2
C(21)-C(22)-H(22)	120.2
C(22)-C(23)-C(24)	121.68(17)
C(22)-C(23)-H(23)	119.2
C(24)-C(23)-H(23)	119.2
C(25)-C(24)-C(23)	117.51(17)
C(25)-C(24)-C(27)	121.14(17)
C(23)-C(24)-C(27)	121.33(17)
C(26)-C(25)-C(24)	121.57(17)
C(26)-C(25)-H(25)	119.2
C(24)-C(25)-H(25)	119.2
C(25)-C(26)-C(21)	120.14(17)
C(25)-C(26)-H(26)	119.9
C(21)-C(26)-H(26)	119.9
C(24)-C(27)-C(28)	113.48(15)
C(24)-C(27)-H(27A)	108.9
C(28)-C(27)-H(27A)	108.9
C(24)-C(27)-H(27B)	108.9
C(28)-C(27)-H(27B)	108.9
H(27A)-C(27)-H(27B)	107.7
C(29)-C(28)-C(27)	112.82(14)
C(29)-C(28)-H(28A)	109.0
C(27)-C(28)-H(28A)	109.0
C(29)-C(28)-H(28B)	109.0
C(27)-C(28)-H(28B)	109.0

H(28A)-C(28)-H(28B)	107.8
C(28)-C(29)-C(30)	115.87(14)
C(28)-C(29)-H(29A)	108.3
C(30)-C(29)-H(29A)	108.3
C(28)-C(29)-H(29B)	108.3
C(30)-C(29)-H(29B)	108.3
H(29A)-C(29)-H(29B)	107.4
C(31)-C(30)-C(29)	111.20(15)
C(31)-C(30)-H(30A)	109.4
C(29)-C(30)-H(30A)	109.4
C(31)-C(30)-H(30B)	109.4
C(29)-C(30)-H(30B)	109.4
H(30A)-C(30)-H(30B)	108.0
C(32)-C(31)-C(30)	115.96(16)
C(32)-C(31)-H(31A)	108.3
C(30)-C(31)-H(31A)	108.3
C(32)-C(31)-H(31B)	108.3
C(30)-C(31)-H(31B)	108.3
H(31A)-C(31)-H(31B)	107.4
C(33)-C(32)-C(37)	117.40(17)
C(33)-C(32)-C(31)	121.04(18)
C(37)-C(32)-C(31)	121.55(19)
C(34)-C(33)-C(32)	121.72(17)
C(34)-C(33)-H(33)	119.1
C(32)-C(33)-H(33)	119.1
C(33)-C(34)-C(35)	120.14(17)
C(33)-C(34)-H(34)	119.9
C(35)-C(34)-H(34)	119.9
C(34)-C(35)-C(36)	118.89(16)
C(34)-C(35)-N(21)	121.56(16)
C(36)-C(35)-N(21)	119.37(16)
C(37)-C(36)-C(35)	120.24(16)
C(37)-C(36)-H(36)	119.9
C(35)-C(36)-H(36)	119.9
C(36)-C(37)-C(32)	121.57(17)
C(36)-C(37)-H(37)	119.2
C(32)-C(37)-H(37)	119.2

C(35)-N(21)-H(21A)	111.1(16)
C(35)-N(21)-H(21B)	105.0(18)
H(21A)-N(21)-H(21B)	110(2)

Symmetry transformations used to generate equivalent atoms:

Table 34. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	24(1)	36(1)	52(1)	-1(1)	-1(1)	-3(1)
C(1)	22(1)	31(1)	36(1)	-6(1)	6(1)	4(1)
C(2)	24(1)	32(1)	32(1)	3(1)	4(1)	4(1)
C(3)	25(1)	32(1)	33(1)	-1(1)	6(1)	0(1)
C(4)	26(1)	37(1)	27(1)	-6(1)	3(1)	5(1)
C(5)	41(1)	32(1)	36(1)	0(1)	0(1)	9(1)
C(6)	35(1)	28(1)	44(1)	1(1)	5(1)	0(1)
C(7)	31(1)	41(1)	33(1)	-8(1)	-3(1)	7(1)
C(8)	27(1)	36(1)	31(1)	-1(1)	-4(1)	3(1)
C(9)	24(1)	33(1)	27(1)	3(1)	0(1)	3(1)
C(10)	23(1)	31(1)	26(1)	2(1)	0(1)	1(1)
C(11)	23(1)	30(1)	26(1)	2(1)	-1(1)	-1(1)
C(12)	23(1)	25(1)	24(1)	3(1)	-3(1)	0(1)
C(13)	27(1)	28(1)	28(1)	-4(1)	-2(1)	-1(1)
C(14)	23(1)	30(1)	34(1)	-3(1)	-4(1)	-4(1)
C(15)	22(1)	26(1)	30(1)	5(1)	-1(1)	2(1)
C(16)	28(1)	25(1)	33(1)	-6(1)	-5(1)	4(1)
C(17)	23(1)	25(1)	34(1)	-1(1)	-7(1)	-1(1)
N(1)	23(1)	34(1)	40(1)	-3(1)	1(1)	2(1)
O(21)	22(1)	33(1)	56(1)	6(1)	0(1)	-3(1)
C(21)	22(1)	27(1)	38(1)	-3(1)	4(1)	2(1)
C(22)	24(1)	29(1)	40(1)	6(1)	0(1)	2(1)
C(23)	24(1)	35(1)	40(1)	4(1)	3(1)	-2(1)
C(24)	24(1)	34(1)	30(1)	-6(1)	2(1)	8(1)
C(25)	35(1)	30(1)	34(1)	3(1)	5(1)	9(1)
C(26)	32(1)	25(1)	44(1)	4(1)	9(1)	1(1)
C(27)	28(1)	47(1)	31(1)	-8(1)	-2(1)	9(1)
C(28)	23(1)	38(1)	29(1)	-4(1)	-2(1)	7(1)
C(29)	27(1)	42(1)	29(1)	-5(1)	-5(1)	10(1)
C(30)	23(1)	34(1)	29(1)	-1(1)	-3(1)	5(1)
C(31)	31(1)	64(1)	36(1)	-14(1)	-10(1)	19(1)
C(32)	25(1)	40(1)	26(1)	-5(1)	-6(1)	8(1)
C(33)	31(1)	26(1)	35(1)	-2(1)	-11(1)	3(1)
C(34)	25(1)	32(1)	29(1)	5(1)	-3(1)	-5(1)
C(35)	22(1)	34(1)	21(1)	2(1)	-1(1)	-1(1)
C(36)	27(1)	29(1)	30(1)	-3(1)	-2(1)	-4(1)
C(37)	21(1)	39(1)	34(1)	0(1)	1(1)	-5(1)
N(21)	27(1)	47(1)	28(1)	-6(1)	5(1)	-3(1)

Table 35. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5**.

	x	y	z	U(eq)
H(1)	18880(19)	6220(20)	1320(30)	59(8)
H(2)	17769	4775	978	35
H(3)	16444	4270	-299	36
H(5)	16456	7411	-2551	44
H(6)	17801	7912	-1330	43
H(7A)	15484	4574	-2634	42
H(7B)	15388	5882	-3336	42
H(8A)	14072	5265	-2022	37
H(8B)	14639	5190	-449	37
H(9A)	14459	7392	-2080	34
H(9B)	14811	7232	-343	34
H(10A)	13378	6746	486	32
H(10B)	13004	6698	-1248	32
H(11A)	13578	8835	280	31
H(11B)	13229	8797	-1469	31
H(13)	11609	7371	-1283	33
H(14)	10157	7628	-398	35
H(16)	11029	10224	2487	35
H(17)	12478	9970	1581	33
H(1B)	9215(18)	8400(20)	1530(30)	51(7)
H(1A)	9542(17)	9270(20)	2720(30)	44(6)
H(21)	29030(19)	430(20)	790(30)	56(8)
H(22)	27615	-377	1072	37
H(23)	26120	-320	284	39
H(25)	26614	2565	-2339	40
H(26)	28102	2540	-1531	40
H(27A)	25012	338	-1753	42
H(27B)	25187	1562	-2638	42
H(28A)	24635	1366	496	36
H(28B)	24895	2610	-274	36
H(29A)	23432	1154	-1274	39
H(29B)	23697	2380	-2074	39
H(30A)	22982	2166	952	34
H(30B)	23312	3406	252	34
H(31A)	22218	3268	-1731	53
H(31B)	21900	2011	-1082	53
H(33)	20542	2067	201	37
H(34)	19459	3012	1653	35
H(36)	21027	5948	1917	35
H(37)	22103	4992	471	38
H(21A)	19722(17)	5620(20)	3580(30)	45(6)
H(21B)	19140(20)	4640(20)	3190(30)	55(8)

Table 36. Torsion angles [°] for **5**.

O(1)-C(1)-C(2)-C(3)	-178.14(17)
C(6)-C(1)-C(2)-C(3)	0.0(3)
C(1)-C(2)-C(3)-C(4)	-1.1(3)
C(2)-C(3)-C(4)-C(5)	1.3(3)
C(2)-C(3)-C(4)-C(7)	179.16(17)
C(3)-C(4)-C(5)-C(6)	-0.4(3)
C(7)-C(4)-C(5)-C(6)	-178.23(17)
C(4)-C(5)-C(6)-C(1)	-0.6(3)
O(1)-C(1)-C(6)-C(5)	179.06(17)
C(2)-C(1)-C(6)-C(5)	0.9(3)
C(5)-C(4)-C(7)-C(8)	-99.7(2)
C(3)-C(4)-C(7)-C(8)	82.5(2)
C(4)-C(7)-C(8)-C(9)	65.3(2)
C(7)-C(8)-C(9)-C(10)	166.85(14)
C(8)-C(9)-C(10)-C(11)	-171.56(15)
C(9)-C(10)-C(11)-C(12)	178.54(14)
C(10)-C(11)-C(12)-C(17)	135.31(16)
C(10)-C(11)-C(12)-C(13)	-44.6(2)
C(17)-C(12)-C(13)-C(14)	-2.3(2)
C(11)-C(12)-C(13)-C(14)	177.65(16)
C(12)-C(13)-C(14)-C(15)	-0.4(3)
C(13)-C(14)-C(15)-C(16)	2.9(3)
C(13)-C(14)-C(15)-N(1)	-179.99(16)
C(14)-C(15)-C(16)-C(17)	-2.7(2)
N(1)-C(15)-C(16)-C(17)	-179.76(15)
C(13)-C(12)-C(17)-C(16)	2.5(2)
C(11)-C(12)-C(17)-C(16)	-177.40(15)
C(15)-C(16)-C(17)-C(12)	-0.1(3)
O(21)-C(21)-C(22)-C(23)	-177.28(18)
C(26)-C(21)-C(22)-C(23)	1.9(3)
C(21)-C(22)-C(23)-C(24)	-0.1(3)
C(22)-C(23)-C(24)-C(25)	-1.1(3)
C(22)-C(23)-C(24)-C(27)	-179.46(17)
C(23)-C(24)-C(25)-C(26)	0.5(3)

C(27)-C(24)-C(25)-C(26)	178.89(17)
C(24)-C(25)-C(26)-C(21)	1.3(3)
O(21)-C(21)-C(26)-C(25)	176.75(17)
C(22)-C(21)-C(26)-C(25)	-2.5(3)
C(25)-C(24)-C(27)-C(28)	-95.0(2)
C(23)-C(24)-C(27)-C(28)	83.3(2)
C(24)-C(27)-C(28)-C(29)	174.19(17)
C(27)-C(28)-C(29)-C(30)	-178.78(17)
C(28)-C(29)-C(30)-C(31)	175.30(17)
C(29)-C(30)-C(31)-C(32)	178.18(18)
C(30)-C(31)-C(32)-C(33)	-119.6(2)
C(30)-C(31)-C(32)-C(37)	61.8(3)
C(37)-C(32)-C(33)-C(34)	-0.8(3)
C(31)-C(32)-C(33)-C(34)	-179.36(17)
C(32)-C(33)-C(34)-C(35)	-0.6(3)
C(33)-C(34)-C(35)-C(36)	2.1(2)
C(33)-C(34)-C(35)-N(21)	177.11(16)
C(34)-C(35)-C(36)-C(37)	-2.2(3)
N(21)-C(35)-C(36)-C(37)	-177.30(16)
C(35)-C(36)-C(37)-C(32)	0.8(3)
C(33)-C(32)-C(37)-C(36)	0.7(3)
C(31)-C(32)-C(37)-C(36)	179.28(17)

Symmetry transformations used to generate equivalent atoms:

Table 37. Crystal data and structure refinement for **1a**.

Identification code	1a	
Empirical formula	C12 H11 N O S	
Formula weight	217.28	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 9.8597(3) Å	α= 90°.
	b = 10.0879(3) Å	β= 102.8090(10)°.
	c = 21.8081(7) Å	γ = 90°.
Volume	2115.13(11) $Å^3$	
Z	8	
Density (calculated)	1.365 Mg/m^3	
Absorption coefficient	0.276 mm^{-1}	
F(000)	912	
Crystal size	0.24 x 0.16 x 0.04 mm^3	
Theta range for data collection	1.92 to 26.90°.	
Index ranges	-12<=h<=12, -12<=k<=12, -27<=l<=27	
Reflections collected	20994	
Independent reflections	4561 [R(int) = 0.0763]	
Completeness to theta = 26.90°	99.9 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4561 / 0 / 295	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0437, wR2 = 0.1170	
R indices (all data)	R1 = 0.0611, wR2 = 0.1275	
Extinction coefficient	.	
Largest diff. peak and hole	0.556 and -0.327 $e.Å^{-3}$	

Table 38. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	3667(1)	4338(1)	1526(1)	26(1)
O(1)	1389(2)	8774(1)	-170(1)	31(1)
N(1)	3685(2)	6137(2)	4157(1)	30(1)
C(1)	8648(2)	4593(2)	2239(1)	29(1)
C(2)	7423(2)	4970(2)	2413(1)	35(1)
C(3)	9903(2)	4713(2)	2675(1)	32(1)
C(4)	9928(2)	5174(2)	3278(1)	32(1)
C(5)	8716(2)	5569(2)	3449(1)	30(1)
C(6)	7453(2)	5474(2)	3002(1)	35(1)
C(7)	7956(2)	5525(2)	1035(1)	26(1)
C(8)	7053(2)	5422(2)	450(1)	29(1)
C(9)	6559(2)	6562(2)	113(1)	30(1)
C(10)	6952(2)	7795(2)	367(1)	28(1)
C(11)	7873(2)	7901(2)	947(1)	32(1)
C(12)	8370(2)	6771(2)	1278(1)	32(1)
S(2)	8595(1)	4054(1)	1457(1)	31(1)
O(2)	6405(2)	8911(1)	40(1)	35(1)
N(2)	8723(2)	5976(2)	4069(1)	36(1)
C(13)	993(2)	6736(2)	348(1)	26(1)
C(14)	1542(2)	5726(2)	761(1)	26(1)
C(15)	1862(2)	7737(2)	218(1)	23(1)
C(16)	2957(2)	5701(2)	1043(1)	24(1)
C(17)	3818(2)	6705(2)	913(1)	25(1)
C(18)	3268(2)	7725(2)	506(1)	26(1)
C(19)	3562(2)	6255(2)	2443(1)	27(1)
C(20)	3570(2)	6639(2)	3055(1)	27(1)
C(21)	3676(2)	5712(2)	3531(1)	25(1)
C(22)	3822(2)	4381(2)	3392(1)	27(1)
C(23)	3822(2)	3994(2)	2781(1)	25(1)
C(24)	3671(2)	4924(2)	2297(1)	22(1)

Table 39. Bond lengths [Å] and angles [°] for **1a**.

S(1)-C(16)	1.7779(18)
S(1)-C(24)	1.7815(19)
O(1)-C(15)	1.361(2)
O(1)-H(1A)	0.96(3)
N(1)-C(21)	1.430(2)
N(1)-H(1B)	0.88(2)
N(1)-H(1C)	0.80(2)
C(1)-C(3)	1.389(3)
C(1)-C(2)	1.397(3)
C(1)-S(2)	1.781(2)
C(2)-C(6)	1.374(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.389(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.387(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.405(3)
C(5)-N(2)	1.411(3)
C(6)-H(6)	0.9500
C(7)-C(8)	1.388(3)
C(7)-C(12)	1.390(3)
C(7)-S(2)	1.7852(19)
C(8)-C(9)	1.394(3)
C(8)-H(8)	0.9500
C(9)-C(10)	1.382(3)
C(9)-H(9)	0.9500
C(10)-O(2)	1.378(2)
C(10)-C(11)	1.388(3)
C(11)-C(12)	1.379(3)
C(11)-H(11)	0.9500
C(12)-H(12)	0.9500
O(2)-H(2A)	0.97(3)
N(2)-H(2B)	0.95(3)
N(2)-H(2C)	0.84(3)

C(13)-C(14)	1.388(3)
C(13)-C(15)	1.394(3)
C(13)-H(13)	0.9500
C(14)-C(16)	1.394(3)
C(14)-H(14)	0.9500
C(15)-C(18)	1.389(2)
C(16)-C(17)	1.392(3)
C(17)-C(18)	1.388(3)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500
C(19)-C(20)	1.386(3)
C(19)-C(24)	1.390(3)
C(19)-H(19)	0.9500
C(20)-C(21)	1.384(3)
C(20)-H(20)	0.9500
C(21)-C(22)	1.391(3)
C(22)-C(23)	1.388(3)
C(22)-H(22)	0.9500
C(23)-C(24)	1.394(2)
C(23)-H(23)	0.9500

C(16)-S(1)-C(24)	102.76(8)
C(15)-O(1)-H(1A)	115.9(17)
C(21)-N(1)-H(1B)	113.1(14)
C(21)-N(1)-H(1C)	116.9(17)
H(1B)-N(1)-H(1C)	108(2)
C(3)-C(1)-C(2)	118.92(18)
C(3)-C(1)-S(2)	121.02(16)
C(2)-C(1)-S(2)	119.98(15)
C(6)-C(2)-C(1)	120.89(18)
C(6)-C(2)-H(2)	119.6
C(1)-C(2)-H(2)	119.6
C(4)-C(3)-C(1)	120.18(18)
C(4)-C(3)-H(3)	119.9
C(1)-C(3)-H(3)	119.9
C(5)-C(4)-C(3)	121.01(18)

C(5)-C(4)-H(4)	119.5
C(3)-C(4)-H(4)	119.5
C(4)-C(5)-C(6)	118.57(18)
C(4)-C(5)-N(2)	121.33(18)
C(6)-C(5)-N(2)	119.96(18)
C(2)-C(6)-C(5)	120.35(19)
C(2)-C(6)-H(6)	119.8
C(5)-C(6)-H(6)	119.8
C(8)-C(7)-C(12)	119.47(18)
C(8)-C(7)-S(2)	119.54(14)
C(12)-C(7)-S(2)	120.97(15)
C(7)-C(8)-C(9)	120.13(18)
C(7)-C(8)-H(8)	119.9
C(9)-C(8)-H(8)	119.9
C(10)-C(9)-C(8)	119.72(17)
C(10)-C(9)-H(9)	120.1
C(8)-C(9)-H(9)	120.1
O(2)-C(10)-C(9)	118.95(17)
O(2)-C(10)-C(11)	120.76(17)
C(9)-C(10)-C(11)	120.29(18)
C(12)-C(11)-C(10)	119.85(18)
C(12)-C(11)-H(11)	120.1
C(10)-C(11)-H(11)	120.1
C(11)-C(12)-C(7)	120.50(18)
C(11)-C(12)-H(12)	119.8
C(7)-C(12)-H(12)	119.8
C(1)-S(2)-C(7)	99.93(9)
C(10)-O(2)-H(2A)	112.3(19)
C(5)-N(2)-H(2B)	111.0(17)
C(5)-N(2)-H(2C)	108(2)
H(2B)-N(2)-H(2C)	113(3)
C(14)-C(13)-C(15)	119.68(16)
C(14)-C(13)-H(13)	120.2
C(15)-C(13)-H(13)	120.2
C(13)-C(14)-C(16)	120.53(17)
C(13)-C(14)-H(14)	119.7

C(16)-C(14)-H(14)	119.7
O(1)-C(15)-C(18)	117.25(16)
O(1)-C(15)-C(13)	122.81(16)
C(18)-C(15)-C(13)	119.90(16)
C(17)-C(16)-C(14)	119.52(17)
C(17)-C(16)-S(1)	120.39(14)
C(14)-C(16)-S(1)	119.98(14)
C(18)-C(17)-C(16)	120.04(17)
C(18)-C(17)-H(17)	120.0
C(16)-C(17)-H(17)	120.0
C(17)-C(18)-C(15)	120.32(17)
C(17)-C(18)-H(18)	119.8
C(15)-C(18)-H(18)	119.8
C(20)-C(19)-C(24)	120.42(17)
C(20)-C(19)-H(19)	119.8
C(24)-C(19)-H(19)	119.8
C(21)-C(20)-C(19)	121.06(18)
C(21)-C(20)-H(20)	119.5
C(19)-C(20)-H(20)	119.5
C(20)-C(21)-C(22)	118.86(17)
C(20)-C(21)-N(1)	119.84(18)
C(22)-C(21)-N(1)	121.26(18)
C(23)-C(22)-C(21)	120.16(17)
C(23)-C(22)-H(22)	119.9
C(21)-C(22)-H(22)	119.9
C(22)-C(23)-C(24)	120.95(17)
C(22)-C(23)-H(23)	119.5
C(24)-C(23)-H(23)	119.5
C(19)-C(24)-C(23)	118.50(17)
C(19)-C(24)-S(1)	123.55(14)
C(23)-C(24)-S(1)	117.93(14)

Symmetry transformations used to generate equivalent atoms:

Table 40. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	31(1)	25(1)	21(1)	1(1)	4(1)	2(1)
O(1)	30(1)	29(1)	32(1)	8(1)	1(1)	0(1)
N(1)	29(1)	38(1)	23(1)	-1(1)	6(1)	-3(1)
C(1)	31(1)	31(1)	25(1)	4(1)	4(1)	-1(1)
C(2)	26(1)	49(1)	28(1)	2(1)	1(1)	-2(1)
C(3)	24(1)	36(1)	35(1)	1(1)	5(1)	4(1)
C(4)	23(1)	38(1)	33(1)	1(1)	0(1)	1(1)
C(5)	29(1)	34(1)	25(1)	2(1)	3(1)	-1(1)
C(6)	24(1)	50(1)	31(1)	4(1)	4(1)	1(1)
C(7)	27(1)	29(1)	24(1)	-1(1)	7(1)	1(1)
C(8)	34(1)	28(1)	25(1)	-6(1)	7(1)	0(1)
C(9)	33(1)	37(1)	21(1)	-2(1)	4(1)	5(1)
C(10)	27(1)	29(1)	29(1)	3(1)	10(1)	3(1)
C(11)	33(1)	29(1)	33(1)	-3(1)	4(1)	-3(1)
C(12)	30(1)	32(1)	30(1)	0(1)	-1(1)	-2(1)
S(2)	38(1)	28(1)	27(1)	0(1)	5(1)	2(1)
O(2)	39(1)	30(1)	34(1)	5(1)	2(1)	4(1)
N(2)	33(1)	46(1)	27(1)	-6(1)	3(1)	1(1)
C(13)	23(1)	29(1)	24(1)	-3(1)	2(1)	0(1)
C(14)	26(1)	28(1)	23(1)	-1(1)	6(1)	-4(1)
C(15)	28(1)	24(1)	17(1)	-1(1)	3(1)	1(1)
C(16)	28(1)	27(1)	16(1)	0(1)	3(1)	1(1)
C(17)	23(1)	31(1)	20(1)	-1(1)	3(1)	-1(1)
C(18)	28(1)	27(1)	22(1)	-1(1)	6(1)	-5(1)
C(19)	31(1)	24(1)	25(1)	5(1)	4(1)	3(1)
C(20)	28(1)	26(1)	27(1)	-1(1)	3(1)	1(1)
C(21)	18(1)	34(1)	20(1)	-1(1)	0(1)	-2(1)
C(22)	25(1)	31(1)	23(1)	6(1)	1(1)	-1(1)
C(23)	24(1)	24(1)	27(1)	2(1)	2(1)	0(1)
C(24)	19(1)	27(1)	19(1)	1(1)	0(1)	0(1)

Table 41. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **1a**.

	x	y	z	U(eq)
H(1A)	430(30)	8730(30)	-384(13)	78(9)
H(1B)	3220(20)	6880(20)	4170(10)	31(6)
H(1C)	3440(20)	5610(20)	4382(12)	43(7)
H(2)	6557	4878	2121	42
H(3)	10748	4478	2561	38
H(4)	10788	5220	3577	39
H(6)	6614	5759	3107	42
H(8)	6771	4574	279	35
H(9)	5954	6492	-291	37
H(11)	8161	8749	1116	39
H(12)	9001	6845	1675	38
H(2A)	6480(30)	9690(30)	307(15)	105(12)
H(2B)	9640(30)	6210(30)	4289(14)	73(9)
H(2C)	8150(30)	6600(30)	4046(15)	87(11)
H(13)	29	6745	155	31
H(14)	949	5047	853	31
H(17)	4784	6692	1102	30
H(18)	3856	8418	424	31
H(19)	3481	6906	2122	32
H(20)	3501	7553	3148	33
H(22)	3922	3735	3716	32
H(23)	3927	3083	2691	30

Table 42. Torsion angles [°] for **1a**.

C(3)-C(1)-C(2)-C(6)	1.1(3)
S(2)-C(1)-C(2)-C(6)	-175.56(17)
C(2)-C(1)-C(3)-C(4)	1.3(3)
S(2)-C(1)-C(3)-C(4)	177.98(16)
C(1)-C(3)-C(4)-C(5)	-2.4(3)
C(3)-C(4)-C(5)-C(6)	1.0(3)
C(3)-C(4)-C(5)-N(2)	176.78(19)
C(1)-C(2)-C(6)-C(5)	-2.5(3)
C(4)-C(5)-C(6)-C(2)	1.5(3)
N(2)-C(5)-C(6)-C(2)	-174.4(2)
C(12)-C(7)-C(8)-C(9)	-0.5(3)
S(2)-C(7)-C(8)-C(9)	-179.30(15)
C(7)-C(8)-C(9)-C(10)	-1.1(3)
C(8)-C(9)-C(10)-O(2)	-177.14(17)
C(8)-C(9)-C(10)-C(11)	2.2(3)
O(2)-C(10)-C(11)-C(12)	177.68(18)
C(9)-C(10)-C(11)-C(12)	-1.7(3)
C(10)-C(11)-C(12)-C(7)	0.0(3)
C(8)-C(7)-C(12)-C(11)	1.0(3)
S(2)-C(7)-C(12)-C(11)	179.85(16)
C(3)-C(1)-S(2)-C(7)	-114.57(17)
C(2)-C(1)-S(2)-C(7)	62.04(18)
C(8)-C(7)-S(2)-C(1)	-144.17(16)
C(12)-C(7)-S(2)-C(1)	37.02(18)
C(15)-C(13)-C(14)-C(16)	-0.4(3)
C(14)-C(13)-C(15)-O(1)	-178.29(16)
C(14)-C(13)-C(15)-C(18)	-0.6(3)
C(13)-C(14)-C(16)-C(17)	0.6(3)
C(13)-C(14)-C(16)-S(1)	-175.40(14)
C(24)-S(1)-C(16)-C(17)	87.74(16)
C(24)-S(1)-C(16)-C(14)	-96.28(16)
C(14)-C(16)-C(17)-C(18)	0.2(3)
S(1)-C(16)-C(17)-C(18)	176.21(14)
C(16)-C(17)-C(18)-C(15)	-1.2(3)

O(1)-C(15)-C(18)-C(17)	179.24(16)
C(13)-C(15)-C(18)-C(17)	1.4(3)
C(24)-C(19)-C(20)-C(21)	-0.5(3)
C(19)-C(20)-C(21)-C(22)	2.1(3)
C(19)-C(20)-C(21)-N(1)	179.89(16)
C(20)-C(21)-C(22)-C(23)	-1.7(3)
N(1)-C(21)-C(22)-C(23)	-179.47(16)
C(21)-C(22)-C(23)-C(24)	-0.3(3)
C(20)-C(19)-C(24)-C(23)	-1.5(3)
C(20)-C(19)-C(24)-S(1)	179.89(14)
C(22)-C(23)-C(24)-C(19)	1.9(3)
C(22)-C(23)-C(24)-S(1)	-179.43(14)
C(16)-S(1)-C(24)-C(19)	-17.87(17)
C(16)-S(1)-C(24)-C(23)	163.56(14)

Symmetry transformations used to generate equivalent atoms:

Table 43. Crystal data and structure refinement for **2a**.

Identification code	2a	
Empirical formula	C13 H13 N O S	
Formula weight	231.30	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 13.844(3) Å	α= 90°.
	b = 5.1626(10) Å	β= 107.22(3)°.
	c = 8.2485(16) Å	γ = 90°.
Volume	563.07(21) $Å^3$	
Z	2	
Density (calculated)	1.364 Mg/m^3	
Absorption coefficient	2.353 mm^{-1}	
F(000)	244	
Crystal size	0.40 x 0.10 x 0.05 mm^3	
Theta range for data collection	3.34 to 74.96°.	
Index ranges	-17<=h<=17, -6<=k<=6, -6<=l<=10	
Reflections collected	3842	
Independent reflections	1935 [R(int) = 0.0449]	
Completeness to theta = 74.96°	89.4 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1935 / 2 / 158	
Goodness-of-fit on F^2	1.095	
Final R indices [I>2sigma(I)]	R1 = 0.0435, wR2 = 0.1170	
R indices (all data)	R1 = 0.0497, wR2 = 0.1259	
Absolute structure parameter	-0.02(3)	
Extinction coefficient	0.0069(11)	
Largest diff. peak and hole	0.312 and -0.318 e.$Å^{-3}$	

Table 44. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	5064(1)	1351(2)	4410(1)	29(1)
N(1)	9998(2)	2184(7)	6593(5)	28(1)
O(1)	784(2)	3402(5)	505(3)	28(1)
C(1)	1773(3)	2984(7)	1412(5)	22(1)
C(2)	2273(3)	867(7)	1014(5)	24(1)
C(3)	3274(3)	392(7)	1913(5)	25(1)
C(4)	3800(3)	2018(7)	3225(5)	23(1)
C(5)	3288(3)	4130(7)	3632(5)	24(1)
C(6)	2287(3)	4636(7)	2737(5)	24(1)
C(7)	5749(3)	3265(7)	3247(5)	26(1)
C(8)	6862(3)	2931(7)	4119(4)	22(1)
C(9)	7419(3)	916(7)	3719(5)	25(1)
C(11)	8932(3)	2362(7)	5807(5)	22(1)
C(12)	8387(3)	4365(7)	6239(5)	26(1)
C(10)	8434(3)	623(7)	4541(5)	23(1)
C(13)	7371(3)	4648(7)	5402(5)	24(1)

Table 45. Bond lengths [Å] and angles [°] for **2a**.

S(1)-C(4)	1.768(4)
S(1)-C(7)	1.827(4)
N(1)-C(11)	1.429(4)
N(1)-H(1B)	0.92(5)
N(1)-H(1A)	0.85(5)
O(1)-C(1)	1.370(4)
O(1)-H(1)	0.98(7)
C(1)-C(2)	1.384(5)
C(1)-C(6)	1.402(5)
C(2)-C(3)	1.386(5)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.392(5)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.394(5)
C(5)-C(6)	1.388(5)
C(5)-H(5A)	0.9500
C(6)-H(6A)	0.9500
C(7)-C(8)	1.504(5)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(9)	1.391(5)
C(8)-C(13)	1.400(5)
C(9)-C(10)	1.376(5)
C(9)-H(9A)	0.9500
C(11)-C(12)	1.387(5)
C(11)-C(10)	1.395(5)
C(12)-C(13)	1.379(5)
C(12)-H(12A)	0.9500
C(10)-H(10A)	0.9500
C(13)-H(13A)	0.9500
C(4)-S(1)-C(7)	100.80(17)
C(11)-N(1)-H(1B)	112(3)
C(11)-N(1)-H(1A)	110(3)

H(1B)-N(1)-H(1A)	108(4)
C(1)-O(1)-H(1)	112(4)
O(1)-C(1)-C(2)	118.9(3)
O(1)-C(1)-C(6)	121.6(3)
C(2)-C(1)-C(6)	119.4(3)
C(1)-C(2)-C(3)	120.2(3)
C(1)-C(2)-H(2A)	119.9
C(3)-C(2)-H(2A)	119.9
C(2)-C(3)-C(4)	121.2(3)
C(2)-C(3)-H(3A)	119.4
C(4)-C(3)-H(3A)	119.4
C(3)-C(4)-C(5)	118.2(3)
C(3)-C(4)-S(1)	121.1(3)
C(5)-C(4)-S(1)	120.6(3)
C(6)-C(5)-C(4)	121.2(3)
C(6)-C(5)-H(5A)	119.4
C(4)-C(5)-H(5A)	119.4
C(5)-C(6)-C(1)	119.7(4)
C(5)-C(6)-H(6A)	120.1
C(1)-C(6)-H(6A)	120.1
C(8)-C(7)-S(1)	107.7(3)
C(8)-C(7)-H(7A)	110.2
S(1)-C(7)-H(7A)	110.2
C(8)-C(7)-H(7B)	110.2
S(1)-C(7)-H(7B)	110.2
H(7A)-C(7)-H(7B)	108.5
C(9)-C(8)-C(13)	117.5(3)
C(9)-C(8)-C(7)	122.2(3)
C(13)-C(8)-C(7)	120.3(3)
C(10)-C(9)-C(8)	121.5(3)
C(10)-C(9)-H(9A)	119.2
C(8)-C(9)-H(9A)	119.2
C(12)-C(11)-C(10)	119.1(3)
C(12)-C(11)-N(1)	119.9(3)
C(10)-C(11)-N(1)	120.9(3)
C(13)-C(12)-C(11)	120.2(3)

C(13)-C(12)-H(12A)	119.9
C(11)-C(12)-H(12A)	119.9
C(9)-C(10)-C(11)	120.3(3)
C(9)-C(10)-H(10A)	119.9
C(11)-C(10)-H(10A)	119.9
C(12)-C(13)-C(8)	121.5(4)
C(12)-C(13)-H(13A)	119.3
C(8)-C(13)-H(13A)	119.3

Symmetry transformations used to generate equivalent atoms:

Table 46. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	24(1)	32(1)	28(1)	9(1)	5(1)	3(1)
N(1)	28(2)	27(2)	26(2)	0(1)	3(1)	1(1)
O(1)	24(1)	30(1)	26(2)	-4(1)	3(1)	2(1)
C(1)	19(2)	24(2)	21(2)	1(1)	5(1)	0(1)
C(2)	21(2)	26(2)	25(2)	-2(2)	7(2)	-2(2)
C(3)	34(2)	24(2)	21(2)	3(2)	16(2)	6(2)
C(4)	24(2)	28(2)	20(2)	8(2)	7(2)	-1(2)
C(5)	24(2)	24(2)	20(2)	0(2)	2(2)	2(2)
C(6)	28(2)	23(2)	20(2)	0(2)	3(2)	-1(2)
C(7)	29(2)	28(2)	20(2)	4(2)	7(2)	-3(2)
C(8)	23(2)	23(2)	20(2)	2(1)	5(2)	-4(1)
C(9)	27(2)	22(2)	24(2)	-2(1)	4(2)	-4(1)
C(11)	18(2)	23(2)	22(2)	2(1)	2(1)	-3(1)
C(12)	29(2)	23(2)	23(2)	-4(2)	0(2)	-1(2)
C(10)	26(2)	20(2)	24(2)	-2(1)	8(2)	1(1)
C(13)	24(2)	23(2)	23(2)	-2(2)	5(2)	-1(2)

Table 47. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **2a**.

	x	y	z	U(eq)
H(1B)	10170(30)	2480(90)	7740(60)	34(12)
H(1A)	10210(40)	680(100)	6440(60)	42(14)
H(1)	520(50)	4970(140)	880(80)	73(18)
H(2A)	1929	-264	122	28
H(3A)	3608	-1070	1629	30
H(5A)	3631	5242	4537	29
H(6A)	1952	6095	3021	29
H(7A)	5581	2661	2057	31
H(7B)	5560	5115	3244	31
H(9A)	7091	-284	2860	30
H(12A)	8714	5544	7113	32
H(10A)	8797	-770	4244	28
H(13A)	7009	6039	5702	28

Table 48. Torsion angles [°] for **2a**.

O(1)-C(1)-C(2)-C(3)	-179.9(4)
C(6)-C(1)-C(2)-C(3)	-0.3(6)
C(1)-C(2)-C(3)-C(4)	-0.1(6)
C(2)-C(3)-C(4)-C(5)	0.9(6)
C(2)-C(3)-C(4)-S(1)	178.1(3)
C(7)-S(1)-C(4)-C(3)	93.7(3)
C(7)-S(1)-C(4)-C(5)	-89.2(3)
C(3)-C(4)-C(5)-C(6)	-1.2(6)
S(1)-C(4)-C(5)-C(6)	-178.4(3)
C(4)-C(5)-C(6)-C(1)	0.8(6)
O(1)-C(1)-C(6)-C(5)	179.5(4)
C(2)-C(1)-C(6)-C(5)	0.0(6)
C(4)-S(1)-C(7)-C(8)	179.4(3)
S(1)-C(7)-C(8)-C(9)	87.8(4)
S(1)-C(7)-C(8)-C(13)	-91.1(4)
C(13)-C(8)-C(9)-C(10)	-0.5(6)
C(7)-C(8)-C(9)-C(10)	-179.4(4)
C(10)-C(11)-C(12)-C(13)	-1.1(6)
N(1)-C(11)-C(12)-C(13)	175.6(4)
C(8)-C(9)-C(10)-C(11)	0.0(6)
C(12)-C(11)-C(10)-C(9)	0.8(6)
N(1)-C(11)-C(10)-C(9)	-175.9(3)
C(11)-C(12)-C(13)-C(8)	0.6(6)
C(9)-C(8)-C(13)-C(12)	0.1(6)
C(7)-C(8)-C(13)-C(12)	179.1(4)

Symmetry transformations used to generate equivalent atoms:

Table 49. Crystal data and structure refinement for **2b**.

Identification code	2b	
Empirical formula	C13 H13 N O S	
Formula weight	231.30	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 13.7422(12) Å	α= 90°.
	b = 5.1725(4) Å	β= 105.548(4)°.
	c = 8.3055(7) Å	γ = 90°.
Volume	568.76(8) $Å^3$	
Z	2	
Density (calculated)	1.351 Mg/m^3	
Absorption coefficient	0.261 mm^{-1}	
F(000)	244	
Crystal size	0.25 x 0.08 x 0.04 mm^3	
Theta range for data collection	1.54 to 27.51°.	
Index ranges	-17<=h<=14, -6<=k<=6, -8<=l<=10	
Reflections collected	3913	
Independent reflections	1847 [R(int) = 0.0510]	
Completeness to theta = 27.51°	99.7 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1847 / 2 / 157	
Goodness-of-fit on F^2	1.021	
Final R indices [I>2sigma(I)]	R1 = 0.0402, wR2 = 0.0841	
R indices (all data)	R1 = 0.0533, wR2 = 0.0882	
Absolute structure parameter	0.12(11)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.269 and -0.225 $e.Å^{-3}$	

Table 50. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 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)
O(1)	11257(2)	1672(5)	7094(3)	23(1)
C(1)	10239(2)	2061(6)	6877(4)	18(1)
C(2)	9792(3)	4181(6)	5934(4)	21(1)
C(3)	8772(3)	4627(6)	5695(5)	21(1)
C(4)	8165(2)	2995(6)	6368(4)	17(1)
C(5)	8634(2)	887(6)	7307(4)	20(1)
C(6)	9658(2)	416(6)	7586(4)	18(1)
C(7)	7053(2)	3479(7)	6071(4)	23(1)
S(1)	6388(1)	1582(2)	4232(1)	23(1)
C(8)	5101(2)	2088(6)	4223(4)	18(1)
C(9)	4534(3)	4103(6)	3315(4)	19(1)
C(10)	3524(2)	4385(6)	3250(4)	20(1)
C(11)	3053(2)	2651(6)	4099(4)	16(1)
C(12)	3625(2)	674(6)	5026(4)	20(1)
C(13)	4630(2)	386(6)	5070(4)	19(1)
N(1)	2006(2)	2821(6)	3939(4)	22(1)

Table 51. Bond lengths [Å] and angles [°] for **2b**.

O(1)-C(1)	1.376(4)
O(1)-H(1)	0.80(4)
C(1)-C(2)	1.391(4)
C(1)-C(6)	1.400(4)
C(2)-C(3)	1.382(5)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.404(5)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.394(4)
C(4)-C(7)	1.503(4)
C(5)-C(6)	1.386(4)
C(5)-H(5A)	0.9500
C(6)-H(6A)	0.9500
C(7)-S(1)	1.839(3)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
S(1)-C(8)	1.786(3)
C(8)-C(13)	1.389(5)
C(8)-C(9)	1.395(4)
C(9)-C(10)	1.383(5)
C(9)-H(9A)	0.9500
C(10)-C(11)	1.402(4)
C(10)-H(10A)	0.9500
C(11)-C(12)	1.390(4)
C(11)-N(1)	1.412(4)
C(12)-C(13)	1.380(4)
C(12)-H(12A)	0.9500
C(13)-H(13A)	0.9500
N(1)-H(1B)	0.87(4)
N(1)-H(1A)	0.94(4)
C(1)-O(1)-H(1)	112(3)
O(1)-C(1)-C(2)	117.9(3)
O(1)-C(1)-C(6)	122.0(3)
C(2)-C(1)-C(6)	120.1(3)
C(3)-C(2)-C(1)	119.3(3)
C(3)-C(2)-H(2A)	120.3
C(1)-C(2)-H(2A)	120.3
C(2)-C(3)-C(4)	122.0(3)
C(2)-C(3)-H(3A)	119.0
C(4)-C(3)-H(3A)	119.0
C(5)-C(4)-C(3)	117.3(3)
C(5)-C(4)-C(7)	121.3(3)
C(3)-C(4)-C(7)	121.4(3)
C(6)-C(5)-C(4)	121.8(3)
C(6)-C(5)-H(5A)	119.1
C(4)-C(5)-H(5A)	119.1
C(5)-C(6)-C(1)	119.4(3)
C(5)-C(6)-H(6A)	120.3
C(1)-C(6)-H(6A)	120.3
C(4)-C(7)-S(1)	107.7(2)
C(4)-C(7)-H(7A)	110.2
S(1)-C(7)-H(7A)	110.2
C(4)-C(7)-H(7B)	110.2

S(1)-C(7)-H(7B)	110.2
H(7A)-C(7)-H(7B)	108.5
C(8)-S(1)-C(7)	101.33(14)
C(13)-C(8)-C(9)	118.6(3)
C(13)-C(8)-S(1)	120.0(2)
C(9)-C(8)-S(1)	121.4(2)
C(10)-C(9)-C(8)	120.6(3)
C(10)-C(9)-H(9A)	119.7
C(8)-C(9)-H(9A)	119.7
C(9)-C(10)-C(11)	120.5(3)
C(9)-C(10)-H(10A)	119.8
C(11)-C(10)-H(10A)	119.8
C(12)-C(11)-C(10)	118.7(3)
C(12)-C(11)-N(1)	120.6(3)
C(10)-C(11)-N(1)	120.6(3)
C(13)-C(12)-C(11)	120.5(3)
C(13)-C(12)-H(12A)	119.8
C(11)-C(12)-H(12A)	119.8
C(12)-C(13)-C(8)	121.1(3)
C(12)-C(13)-H(13A)	119.4
C(8)-C(13)-H(13A)	119.4
C(11)-N(1)-H(1B)	113(2)
C(11)-N(1)-H(1A)	116(2)
H(1B)-N(1)-H(1A)	106(3)

Symmetry transformations used to generate equivalent atoms:

Table 52. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	12(1)	27(1)	29(1)	6(1)	5(1)	0(1)
C(1)	13(2)	23(2)	17(2)	-2(1)	3(1)	-1(1)
C(2)	21(2)	19(2)	23(2)	0(1)	6(1)	-3(1)
C(3)	23(2)	19(2)	20(2)	2(2)	2(1)	0(2)
C(4)	16(2)	20(2)	17(2)	-5(1)	6(1)	1(1)
C(5)	20(2)	23(2)	20(2)	-3(1)	9(1)	-6(1)
C(6)	20(2)	18(2)	17(2)	1(1)	5(1)	0(1)
C(7)	19(2)	28(2)	24(2)	-5(2)	8(1)	0(2)
S(1)	14(1)	32(1)	23(1)	-7(1)	6(1)	1(1)
C(8)	10(2)	27(2)	16(2)	-6(1)	3(1)	3(1)
C(9)	22(2)	16(2)	21(2)	-1(1)	8(2)	-5(1)
C(10)	18(2)	20(2)	22(2)	4(1)	2(2)	3(1)
C(11)	15(2)	17(2)	16(2)	-5(1)	4(1)	-2(1)
C(12)	19(2)	19(2)	21(2)	-1(1)	6(1)	-2(1)
C(13)	19(2)	23(2)	16(2)	1(1)	3(1)	4(1)
N(1)	16(1)	27(2)	24(2)	2(1)	5(1)	1(1)

Table 53. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **2b**.

	x	y	z	U(eq)
H(1)	11440(30)	340(90)	7560(50)	49(14)
H(2A)	10185	5310	5459	25
H(3A)	8472	6084	5055	25
H(5A)	8240	-259	7768	24
H(6A)	9964	-1010	8253	22
H(7A)	6904	5343	5870	28
H(7B)	6830	2945	7061	28
H(9A)	4845	5292	2736	23
H(10A)	3146	5764	2626	24
H(12A)	3322	-488	5634	24
H(13A)	5007	-998	5689	23
H(1B)	1750(30)	4310(70)	3560(50)	25(10)
H(1A)	1800(30)	2510(60)	4910(40)	20(9)

Table 54. Torsion angles [°] for **2b**.

O(1)-C(1)-C(2)-C(3)	-179.9(3)
C(6)-C(1)-C(2)-C(3)	-0.6(5)
C(1)-C(2)-C(3)-C(4)	-0.3(5)
C(2)-C(3)-C(4)-C(5)	0.3(5)
C(2)-C(3)-C(4)-C(7)	-178.9(3)
C(3)-C(4)-C(5)-C(6)	0.6(4)
C(7)-C(4)-C(5)-C(6)	179.9(3)
C(4)-C(5)-C(6)-C(1)	-1.5(5)
O(1)-C(1)-C(6)-C(5)	-179.2(3)
C(2)-C(1)-C(6)-C(5)	1.5(4)
C(5)-C(4)-C(7)-S(1)	-85.9(3)
C(3)-C(4)-C(7)-S(1)	93.3(3)
C(4)-C(7)-S(1)-C(8)	173.5(3)
C(7)-S(1)-C(8)-C(13)	-91.0(3)
C(7)-S(1)-C(8)-C(9)	92.0(3)
C(13)-C(8)-C(9)-C(10)	-0.5(5)
S(1)-C(8)-C(9)-C(10)	176.6(3)
C(8)-C(9)-C(10)-C(11)	0.1(5)
C(9)-C(10)-C(11)-C(12)	1.0(5)
C(9)-C(10)-C(11)-N(1)	-175.4(3)
C(10)-C(11)-C(12)-C(13)	-1.8(4)
N(1)-C(11)-C(12)-C(13)	174.7(3)
C(11)-C(12)-C(13)-C(8)	1.4(5)
C(9)-C(8)-C(13)-C(12)	-0.3(5)
S(1)-C(8)-C(13)-C(12)	-177.4(2)

Symmetry transformations used to generate equivalent atoms:

Table 55. Crystal data and structure refinement for **2c**.

Identification code	2c	
Empirical formula	C12 H11 N O S2	
Formula weight	249.34	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 10.4321(12) Å	α= 90°.
	b = 8.1179(11) Å	β= 109.633(6)°.
	c = 14.791(2) Å	γ = 90°.
Volume	1179.8(3) $Å^3$	
Z	4	
Density (calculated)	1.404 Mg/m^3	
Absorption coefficient	0.428 mm^{-1}	
F(000)	520	
Crystal size	0.55 x 0.50 x 0.10 mm^3	
Theta range for data collection	2.07 to 28.32°.	
Index ranges	-12<=h<=13, -7<=k<=10, -19<=l<=19	
Reflections collected	8343	
Independent reflections	2926 [R(int) = 0.0278]	
Completeness to theta = 28.32°	99.6 %	
Absorption correction	Psi-scan	
Max. and min. transmission	0.90251 and 0.76269	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2926 / 0 / 157	
Goodness-of-fit on F^2	1.078	
Final R indices [I>2sigma(I)]	R1 = 0.0348, wR2 = 0.0836	
R indices (all data)	R1 = 0.0417, wR2 = 0.0871	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.381 and -0.221 $e.Å^{-3}$	

Table 56. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2c**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	681(1)	-5936(1)	-1851(1)	27(1)
C(1)	1004(2)	-4928(2)	-1068(1)	21(1)
C(2)	2213(2)	-5240(2)	-304(1)	25(1)
C(3)	2566(2)	-4276(2)	520(1)	25(1)
C(4)	1724(2)	-2978(2)	596(1)	22(1)
C(5)	507(2)	-2685(2)	-162(1)	21(1)
C(6)	149(1)	-3641(2)	-991(1)	21(1)
S(1)	2155(1)	-1739(1)	1653(1)	28(1)
S(2)	2842(1)	410(1)	1242(1)	27(1)
N(1)	8715(1)	-411(2)	1572(1)	23(1)
C(11)	7318(2)	-264(2)	1485(1)	21(1)
C(12)	6577(2)	1139(2)	1068(1)	23(1)
C(13)	5220(2)	1294(2)	1003(1)	24(1)
C(14)	4592(2)	69(2)	1375(1)	21(1)
C(15)	5337(2)	-1320(2)	1808(1)	23(1)
C(16)	6691(2)	-1488(2)	1854(1)	23(1)

Table 57. Bond lengths [Å] and angles [°] for **2c**.

O(1)-C(1)	1.3651(19)
O(1)-H(1)	0.85(2)
C(1)-C(6)	1.403(2)
C(1)-C(2)	1.405(2)
C(2)-C(3)	1.390(2)
C(2)-H(2)	0.9500
C(3)-C(4)	1.401(2)
C(3)-H(3)	0.9500
C(4)-C(5)	1.403(2)
C(4)-S(1)	1.7845(16)
C(5)-C(6)	1.391(2)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
S(1)-S(2)	2.0540(6)
S(2)-C(14)	1.7910(15)
N(1)-C(11)	1.4242(19)
N(1)-H(1A)	0.82(2)
N(1)-H(1B)	0.80(2)
C(11)-C(16)	1.398(2)
C(11)-C(12)	1.398(2)
C(12)-C(13)	1.392(2)
C(12)-H(12)	0.9500
C(13)-C(14)	1.401(2)
C(13)-H(13)	0.9500
C(14)-C(15)	1.397(2)
C(15)-C(16)	1.398(2)
C(15)-H(15)	0.9500
C(16)-H(16A)	0.9500
C(1)-O(1)-H(1)	110.8(14)
O(1)-C(1)-C(6)	122.24(13)
O(1)-C(1)-C(2)	118.26(14)
C(6)-C(1)-C(2)	119.46(14)
C(3)-C(2)-C(1)	120.41(14)
C(3)-C(2)-H(2)	119.8
C(1)-C(2)-H(2)	119.8

C(2)-C(3)-C(4)	120.35(14)
C(2)-C(3)-H(3)	119.8
C(4)-C(3)-H(3)	119.8
C(3)-C(4)-C(5)	119.08(14)
C(3)-C(4)-S(1)	120.95(12)
C(5)-C(4)-S(1)	119.94(12)
C(6)-C(5)-C(4)	120.88(14)
C(6)-C(5)-H(5)	119.6
C(4)-C(5)-H(5)	119.6
C(5)-C(6)-C(1)	119.80(13)
C(5)-C(6)-H(6)	120.1
C(1)-C(6)-H(6)	120.1
C(4)-S(1)-S(2)	103.30(5)
C(14)-S(2)-S(1)	106.28(5)
C(11)-N(1)-H(1A)	109.7(17)
C(11)-N(1)-H(1B)	111.5(15)
H(1A)-N(1)-H(1B)	112(2)
C(16)-C(11)-C(12)	119.28(14)
C(16)-C(11)-N(1)	120.31(14)
C(12)-C(11)-N(1)	120.34(14)
C(13)-C(12)-C(11)	120.21(14)
C(13)-C(12)-H(12)	119.9
C(11)-C(12)-H(12)	119.9
C(12)-C(13)-C(14)	120.48(14)
C(12)-C(13)-H(13)	119.8
C(14)-C(13)-H(13)	119.8
C(15)-C(14)-C(13)	119.48(14)
C(15)-C(14)-S(2)	124.34(12)
C(13)-C(14)-S(2)	116.17(12)
C(14)-C(15)-C(16)	119.86(14)
C(14)-C(15)-H(15)	120.1
C(16)-C(15)-H(15)	120.1
C(15)-C(16)-C(11)	120.66(14)
C(15)-C(16)-H(16A)	119.7
C(11)-C(16)-H(16A)	119.7

Symmetry transformations used to generate equivalent atoms:

Table 58. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2c**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	26(1)	21(1)	29(1)	-1(1)	3(1)	5(1)
C(1)	21(1)	17(1)	26(1)	3(1)	8(1)	-2(1)
C(2)	21(1)	21(1)	33(1)	4(1)	7(1)	4(1)
C(3)	18(1)	27(1)	27(1)	7(1)	4(1)	0(1)
C(4)	18(1)	26(1)	23(1)	1(1)	8(1)	-4(1)
C(5)	18(1)	22(1)	26(1)	3(1)	9(1)	1(1)
C(6)	17(1)	21(1)	25(1)	4(1)	5(1)	0(1)
S(1)	21(1)	43(1)	22(1)	-4(1)	9(1)	-4(1)
S(2)	19(1)	28(1)	32(1)	-8(1)	6(1)	2(1)
N(1)	19(1)	25(1)	25(1)	4(1)	8(1)	-1(1)
C(11)	19(1)	23(1)	18(1)	-2(1)	5(1)	-3(1)
C(12)	26(1)	19(1)	23(1)	1(1)	7(1)	-4(1)
C(13)	26(1)	20(1)	24(1)	0(1)	4(1)	2(1)
C(14)	18(1)	24(1)	19(1)	-4(1)	4(1)	0(1)
C(15)	21(1)	25(1)	23(1)	3(1)	7(1)	-3(1)
C(16)	20(1)	22(1)	24(1)	5(1)	5(1)	1(1)

Table 59. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **2c**.

	x	y	z	U(eq)
H(1)	60(20)	-5510(30)	-2322(16)	36(6)
H(2)	2792	-6114	-351	30
H(3)	3383	-4499	1034	30
H(5)	-80	-1824	-109	26
H(6)	-672	-3422	-1503	25
H(1A)	8850(20)	80(30)	1130(17)	48(7)
H(1B)	8940(20)	-1350(30)	1591(14)	28(5)
H(12)	7000	1989	829	28
H(13)	4716	2238	704	29
H(15)	4924	-2150	2071	28
H(16A)	7190	-2443	2140	27

Table 60. Torsion angles [°] for **2c**.

O(1)-C(1)-C(2)-C(3)	-178.17(14)
C(6)-C(1)-C(2)-C(3)	-0.4(2)
C(1)-C(2)-C(3)-C(4)	-0.3(2)
C(2)-C(3)-C(4)-C(5)	1.3(2)
C(2)-C(3)-C(4)-S(1)	179.50(12)
C(3)-C(4)-C(5)-C(6)	-1.5(2)
S(1)-C(4)-C(5)-C(6)	-179.77(11)
C(4)-C(5)-C(6)-C(1)	0.8(2)
O(1)-C(1)-C(6)-C(5)	177.83(13)
C(2)-C(1)-C(6)-C(5)	0.2(2)
C(3)-C(4)-S(1)-S(2)	104.33(12)
C(5)-C(4)-S(1)-S(2)	-77.46(12)
C(4)-S(1)-S(2)-C(14)	-83.34(7)
C(16)-C(11)-C(12)-C(13)	-1.5(2)
N(1)-C(11)-C(12)-C(13)	-178.66(14)
C(11)-C(12)-C(13)-C(14)	1.6(2)
C(12)-C(13)-C(14)-C(15)	-0.4(2)
C(12)-C(13)-C(14)-S(2)	-179.81(12)
S(1)-S(2)-C(14)-C(15)	-8.33(14)
S(1)-S(2)-C(14)-C(13)	171.10(10)
C(13)-C(14)-C(15)-C(16)	-1.0(2)
S(2)-C(14)-C(15)-C(16)	178.45(12)
C(14)-C(15)-C(16)-C(11)	1.1(2)
C(12)-C(11)-C(16)-C(15)	0.1(2)
N(1)-C(11)-C(16)-C(15)	177.33(14)

Symmetry transformations used to generate equivalent atoms:

Table 61. Crystal data and structure refinement for **3a**.

Identification code	3a	
Empirical formula	C14 H15 N O S	
Formula weight	245.33	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 12.6341(9) Å	α= 90°.
	b = 5.8636(4) Å	β= 90.351(3)°.
	c = 8.5671(5) Å	γ = 90°.
Volume	634.65(7) $Å^3$	
Z	2	
Density (calculated)	1.284 Mg/m^3	
Absorption coefficient	0.238 mm^{-1}	
F(000)	260	
Crystal size	0.35 x 0.25 x 0.20 mm^3	
Theta range for data collection	1.61 to 30.08°.	
Index ranges	-17<=h<=17, -6<=k<=7, -11<=l<=11	
Reflections collected	4751	
Independent reflections	3138 [R(int) = 0.0256]	
Completeness to theta = 30.08°	92.6 %	
Absorption correction	Psi-scan	
Max. and min. transmission	0.93286 and 0.88891	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3138 / 2 / 166	
Goodness-of-fit on F^2	1.055	
Final R indices [I>2sigma(I)]	R1 = 0.0322, wR2 = 0.0822	
R indices (all data)	R1 = 0.0337, wR2 = 0.0835	
Absolute structure parameter	0.04(6)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.304 and -0.177 e.$Å^{-3}$	

Table 62. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **3a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8085(1)	7481(2)	1880(2)	24(1)
C(1)	7044(1)	7568(3)	2291(2)	19(1)
C(2)	6345(1)	9240(3)	1732(2)	20(1)
C(3)	5301(1)	9265(3)	2265(2)	21(1)
C(4)	4946(1)	7645(3)	3339(2)	18(1)
C(5)	5650(1)	5968(3)	3871(2)	22(1)
C(6)	6691(1)	5930(3)	3354(2)	23(1)
S(1)	3635(1)	7765(1)	4081(1)	19(1)
C(7)	2959(1)	5733(4)	2813(2)	28(1)
C(8)	1893(1)	5115(3)	3513(2)	22(1)
C(9)	1192(1)	3671(3)	2470(2)	16(1)
C(10)	200(1)	4484(3)	1989(2)	17(1)
C(11)	-465(1)	3211(3)	1018(2)	17(1)
C(12)	-142(1)	1062(3)	489(2)	16(1)
C(13)	839(1)	201(3)	974(2)	16(1)
C(14)	1496(1)	1493(3)	1954(2)	17(1)
N(1)	-844(1)	-267(3)	-449(2)	19(1)

Table 63. Bond lengths [Å] and angles [°] for **3a**.

O(1)-C(1)	1.365(2)
O(1)-H(1)	0.85(3)
C(1)-C(6)	1.399(2)
C(1)-C(2)	1.402(2)
C(2)-C(3)	1.398(2)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.398(2)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.401(2)
C(4)-S(1)	1.7787(17)
C(5)-C(6)	1.390(2)
C(5)-H(5A)	0.9500
C(6)-H(6A)	0.9500
S(1)-C(7)	1.8218(18)
C(7)-C(8)	1.521(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(9)	1.514(2)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.400(2)
C(9)-C(14)	1.405(2)
C(10)-C(11)	1.395(2)
C(10)-H(10A)	0.9500
C(11)-C(12)	1.401(2)
C(11)-H(11A)	0.9500
C(12)-C(13)	1.400(2)
C(12)-N(1)	1.425(2)
C(13)-C(14)	1.400(2)
C(13)-H(13A)	0.9500
C(14)-H(14A)	0.9500
N(1)-H(1A)	0.82(3)
N(1)-H(1B)	0.90(3)
C(1)-O(1)-H(1)	111(2)
O(1)-C(1)-C(6)	117.01(15)
O(1)-C(1)-C(2)	122.96(15)
C(6)-C(1)-C(2)	120.01(16)
C(3)-C(2)-C(1)	119.28(15)
C(3)-C(2)-H(2A)	120.4
C(1)-C(2)-H(2A)	120.4
C(2)-C(3)-C(4)	121.02(15)
C(2)-C(3)-H(3A)	119.5
C(4)-C(3)-H(3A)	119.5
C(3)-C(4)-C(5)	118.98(16)
C(3)-C(4)-S(1)	120.79(13)
C(5)-C(4)-S(1)	120.18(13)
C(6)-C(5)-C(4)	120.56(15)
C(6)-C(5)-H(5A)	119.7
C(4)-C(5)-H(5A)	119.7
C(5)-C(6)-C(1)	120.14(16)
C(5)-C(6)-H(6A)	119.9
C(1)-C(6)-H(6A)	119.9
C(4)-S(1)-C(7)	101.26(8)

C(8)-C(7)-S(1)	109.47(12)
C(8)-C(7)-H(7A)	109.8
S(1)-C(7)-H(7A)	109.8
C(8)-C(7)-H(7B)	109.8
S(1)-C(7)-H(7B)	109.8
H(7A)-C(7)-H(7B)	108.2
C(9)-C(8)-C(7)	114.62(13)
C(9)-C(8)-H(8A)	108.6
C(7)-C(8)-H(8A)	108.6
C(9)-C(8)-H(8B)	108.6
C(7)-C(8)-H(8B)	108.6
H(8A)-C(8)-H(8B)	107.6
C(10)-C(9)-C(14)	117.58(14)
C(10)-C(9)-C(8)	120.17(14)
C(14)-C(9)-C(8)	122.25(15)
C(11)-C(10)-C(9)	121.82(14)
C(11)-C(10)-H(10A)	119.1
C(9)-C(10)-H(10A)	119.1
C(10)-C(11)-C(12)	119.96(15)
C(10)-C(11)-H(11A)	120.0
C(12)-C(11)-H(11A)	120.0
C(13)-C(12)-C(11)	119.15(14)
C(13)-C(12)-N(1)	121.16(14)
C(11)-C(12)-N(1)	119.56(14)
C(14)-C(13)-C(12)	120.23(14)
C(14)-C(13)-H(13A)	119.9
C(12)-C(13)-H(13A)	119.9
C(13)-C(14)-C(9)	121.23(14)
C(13)-C(14)-H(14A)	119.4
C(9)-C(14)-H(14A)	119.4
C(12)-N(1)-H(1A)	111.0(16)
C(12)-N(1)-H(1B)	114.7(16)
H(1A)-N(1)-H(1B)	105(2)

Symmetry transformations used to generate equivalent atoms:

Table 64. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	15(1)	26(1)	30(1)	4(1)	4(1)	2(1)
C(1)	16(1)	19(1)	21(1)	-2(1)	1(1)	-1(1)
C(2)	16(1)	21(1)	24(1)	5(1)	2(1)	1(1)
C(3)	16(1)	25(1)	22(1)	4(1)	0(1)	3(1)
C(4)	13(1)	23(1)	19(1)	-1(1)	-1(1)	-3(1)
C(5)	21(1)	23(1)	23(1)	6(1)	1(1)	-3(1)
C(6)	17(1)	21(1)	32(1)	5(1)	0(1)	2(1)
S(1)	13(1)	25(1)	18(1)	-4(1)	1(1)	-4(1)
C(7)	15(1)	42(1)	26(1)	-16(1)	6(1)	-11(1)
C(8)	20(1)	29(1)	16(1)	-5(1)	2(1)	-8(1)
C(9)	16(1)	19(1)	12(1)	-1(1)	2(1)	-5(1)
C(10)	19(1)	15(1)	18(1)	-1(1)	4(1)	-1(1)
C(11)	16(1)	19(1)	17(1)	2(1)	2(1)	1(1)
C(12)	17(1)	17(1)	13(1)	0(1)	2(1)	-2(1)
C(13)	18(1)	15(1)	16(1)	-1(1)	3(1)	0(1)
C(14)	14(1)	20(1)	16(1)	3(1)	2(1)	2(1)
N(1)	18(1)	24(1)	17(1)	-5(1)	-1(1)	-1(1)

Table 65. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **3a**.

	x	y	z	U(eq)
H(1)	8200(30)	8350(60)	1110(40)	53(8)
H(2A)	6578	10343	998	24
H(3A)	4827	10401	1893	25
H(5A)	5415	4848	4591	26
H(6A)	7164	4788	3724	28
H(7A)	2852	6411	1765	33
H(7B)	3395	4342	2697	33
H(8A)	2018	4282	4503	26
H(8B)	1512	6540	3769	26
H(10A)	-26	5944	2334	21
H(11A)	-1135	3801	716	20
H(13A)	1061	-1266	637	19
H(14A)	2159	887	2277	20
H(1A)	-1145(18)	530(40)	-1110(30)	24(6)
H(1B)	-531(19)	-1380(40)	-1000(30)	31(6)

Table 66. Torsion angles [°] for **3a**.

O(1)-C(1)-C(2)-C(3)	-177.19(17)
C(6)-C(1)-C(2)-C(3)	0.9(3)
C(1)-C(2)-C(3)-C(4)	-0.4(3)
C(2)-C(3)-C(4)-C(5)	-0.5(3)
C(2)-C(3)-C(4)-S(1)	177.11(14)
C(3)-C(4)-C(5)-C(6)	0.7(3)
S(1)-C(4)-C(5)-C(6)	-176.87(14)
C(4)-C(5)-C(6)-C(1)	-0.2(3)
O(1)-C(1)-C(6)-C(5)	177.55(16)
C(2)-C(1)-C(6)-C(5)	-0.7(3)
C(3)-C(4)-S(1)-C(7)	97.08(16)
C(5)-C(4)-S(1)-C(7)	-85.38(16)
C(4)-S(1)-C(7)-C(8)	165.34(14)
S(1)-C(7)-C(8)-C(9)	172.53(13)
C(7)-C(8)-C(9)-C(10)	-119.39(18)
C(7)-C(8)-C(9)-C(14)	60.9(2)
C(14)-C(9)-C(10)-C(11)	-0.8(2)
C(8)-C(9)-C(10)-C(11)	179.47(14)
C(9)-C(10)-C(11)-C(12)	-0.4(2)
C(10)-C(11)-C(12)-C(13)	1.4(2)
C(10)-C(11)-C(12)-N(1)	177.37(14)
C(11)-C(12)-C(13)-C(14)	-1.1(2)
N(1)-C(12)-C(13)-C(14)	-177.02(13)
C(12)-C(13)-C(14)-C(9)	-0.2(2)
C(10)-C(9)-C(14)-C(13)	1.1(2)
C(8)-C(9)-C(14)-C(13)	-179.19(14)

Symmetry transformations used to generate equivalent atoms:

Table 67. Crystal data and structure refinement for **2d**.

Identification code	2d	
Empirical formula	C14 H13 N O	
Formula weight	211.25	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pc	
Unit cell dimensions	a = 12.951(8) Å	α= 90°.
	b = 5.226(3) Å	β= 98.12(3)°.
	c = 8.046(3) Å	γ = 90°.
Volume	539.2(5) $Å^3$	
Z	2	
Density (calculated)	1.301 Mg/m^3	
Absorption coefficient	0.082 mm^{-1}	
F(000)	224	
Crystal size	0.3 x 0.26 x 0.10 mm^3	
Theta range for data collection	1.59 to 27.00°.	
Index ranges	-8<=h<=16, -5<=k<=6, -10<=l<=8	
Reflections collected	3112	
Independent reflections	1601 [R(int) = 0.0367]	
Completeness to theta = 27.00°	99.0 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1601 / 2 / 197	
Goodness-of-fit on F^2	1.045	
Final R indices [I>2sigma(I)]	R1 = 0.0392, wR2 = 0.0996	
R indices (all data)	R1 = 0.0554, wR2 = 0.1122	
Absolute structure parameter	7(2)	
Extinction coefficient	.	
Largest diff. peak and hole	0.194 and -0.177 e.$Å^{-3}$	

Table 68. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2d**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(2)	-1386(2)	4428(6)	645(4)	23(1)
C(13)	4666(3)	367(6)	3341(4)	24(1)
C(11)	4640(3)	4144(7)	5032(4)	22(1)
O(1)	6222(2)	1762(4)	5167(3)	28(1)
C(10)	3590(3)	4477(6)	4398(4)	23(1)
C(8)	1967(2)	3231(6)	2591(4)	24(1)
C(7)	1385(3)	1912(6)	1380(4)	23(1)
N(1)	-2958(2)	2756(6)	-1047(3)	26(1)
C(6)	-1274(3)	682(6)	-1041(4)	25(1)
C(5)	-217(3)	516(6)	-433(4)	24(1)
C(4)	282(2)	2251(5)	737(4)	21(1)
C(9)	3066(2)	2788(5)	3230(4)	22(1)
C(14)	3635(3)	710(6)	2713(4)	24(1)
C(3)	-334(3)	4246(6)	1253(4)	23(1)
C(12)	5174(2)	2071(6)	4511(4)	23(1)
C(1)	-1871(2)	2651(6)	-501(4)	21(1)

Table 69. Bond lengths [Å] and angles [°] for **2d**.

C(2)-C(3)	1.385(5)
C(2)-C(1)	1.394(4)
C(2)-H(2)	0.98(4)
C(13)-C(14)	1.371(5)
C(13)-C(12)	1.392(4)
C(13)-H(13)	1.03(4)
C(11)-C(12)	1.382(4)
C(11)-C(10)	1.394(5)
C(11)-H(11)	0.91(4)
O(1)-C(12)	1.395(4)
O(1)-H(1B)	0.94(4)
C(10)-C(9)	1.394(5)
C(10)-H(10)	1.00(3)
C(8)-C(7)	1.337(3)
C(8)-C(9)	1.461(5)
C(8)-H(8)	0.99(3)
C(7)-C(4)	1.459(5)
C(7)-H(7)	0.88(3)
N(1)-C(1)	1.415(4)
N(1)-H(1A)	0.86(4)
N(1)-H(1C)	0.95(3)
C(6)-C(5)	1.390(5)
C(6)-C(1)	1.392(4)
C(6)-H(6)	1.11(4)
C(5)-C(4)	1.399(5)
C(5)-H(5)	1.02(3)
C(4)-C(3)	1.411(4)
C(9)-C(14)	1.408(4)
C(14)-H(14)	1.10(4)
C(3)-H(3)	0.96(3)
C(3)-C(2)-C(1)	121.1(3)
C(3)-C(2)-H(2)	122(2)
C(1)-C(2)-H(2)	117(2)

C(14)-C(13)-C(12)	120.6(3)
C(14)-C(13)-H(13)	121(2)
C(12)-C(13)-H(13)	118(2)
C(12)-C(11)-C(10)	119.4(3)
C(12)-C(11)-H(11)	117(2)
C(10)-C(11)-H(11)	123(2)
C(12)-O(1)-H(1B)	110(3)
C(9)-C(10)-C(11)	121.6(3)
C(9)-C(10)-H(10)	121.4(19)
C(11)-C(10)-H(10)	116.8(19)
C(7)-C(8)-C(9)	126.6(2)
C(7)-C(8)-H(8)	118(2)
C(9)-C(8)-H(8)	115(2)
C(8)-C(7)-C(4)	128.2(3)
C(8)-C(7)-H(7)	121(3)
C(4)-C(7)-H(7)	111(3)
C(1)-N(1)-H(1A)	115(3)
C(1)-N(1)-H(1C)	113(2)
H(1A)-N(1)-H(1C)	107(4)
C(5)-C(6)-C(1)	119.9(3)
C(5)-C(6)-H(6)	124(2)
C(1)-C(6)-H(6)	116(2)
C(6)-C(5)-C(4)	122.4(3)
C(6)-C(5)-H(5)	123.7(18)
C(4)-C(5)-H(5)	113.9(18)
C(5)-C(4)-C(3)	116.7(3)
C(5)-C(4)-C(7)	119.8(3)
C(3)-C(4)-C(7)	123.5(3)
C(10)-C(9)-C(14)	117.5(3)
C(10)-C(9)-C(8)	119.6(3)
C(14)-C(9)-C(8)	122.8(3)
C(13)-C(14)-C(9)	121.0(3)
C(13)-C(14)-H(14)	117.9(19)
C(9)-C(14)-H(14)	121.0(19)
C(2)-C(3)-C(4)	121.1(3)
C(2)-C(3)-H(3)	121.9(19)

C(4)-C(3)-H(3)	117.0(19)
C(11)-C(12)-C(13)	119.9(3)
C(11)-C(12)-O(1)	118.5(3)
C(13)-C(12)-O(1)	121.6(3)
C(6)-C(1)-C(2)	118.8(3)
C(6)-C(1)-N(1)	120.2(3)
C(2)-C(1)-N(1)	120.9(3)

Symmetry transformations used to generate equivalent atoms:

Table 70. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2d**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(2)	19(2)	24(2)	28(2)	2(1)	4(2)	4(1)
C(13)	24(2)	22(2)	24(2)	-3(1)	3(2)	-1(1)
C(11)	20(2)	24(2)	21(2)	-3(1)	0(2)	-6(1)
O(1)	18(1)	33(1)	33(1)	-5(1)	-1(1)	0(1)
C(10)	21(2)	26(2)	23(2)	1(1)	4(2)	3(1)
C(8)	24(2)	25(2)	23(2)	2(1)	8(2)	3(1)
C(7)	18(2)	26(2)	25(2)	-2(1)	2(2)	-1(1)
N(1)	19(2)	32(2)	27(2)	-1(1)	0(1)	3(1)
C(6)	27(2)	24(2)	24(2)	-2(1)	4(2)	0(1)
C(5)	26(2)	21(2)	24(2)	-1(1)	3(2)	1(1)
C(4)	19(2)	23(2)	22(2)	4(1)	3(2)	0(1)
C(9)	19(2)	22(2)	23(2)	2(1)	1(2)	-4(1)
C(14)	22(2)	25(2)	24(2)	0(1)	3(2)	1(1)
C(3)	24(2)	20(2)	24(2)	-3(1)	2(2)	-4(1)
C(12)	20(2)	27(2)	21(2)	4(1)	4(2)	1(1)
C(1)	17(2)	25(2)	21(2)	3(1)	0(2)	-4(1)

Table 71. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **2d**.

	x	y	z	U(eq)
H(8)	1650(30)	4740(70)	3070(50)	34(10)
H(10)	3240(20)	6060(50)	4760(40)	15(8)
H(11)	5010(30)	5260(60)	5760(50)	22(8)
H(5)	270(20)	-840(60)	-810(40)	19(8)
H(14)	3280(30)	-630(60)	1750(50)	32(10)
H(7)	1640(30)	620(70)	880(50)	33(10)
H(2)	-1830(30)	5760(60)	1010(50)	31(10)
H(3)	10(30)	5480(60)	2010(40)	21(9)
H(13)	5090(30)	-1130(70)	2960(50)	48(12)
H(6)	-1690(30)	-680(70)	-1950(50)	50(11)
H(1A)	-3250(30)	4200(80)	-880(60)	60(14)
H(1B)	6500(30)	360(80)	4650(60)	42(11)
H(1C)	-3140(30)	2340(70)	-2200(40)	48(10)

Table 72. Torsion angles [°] for **2d**.

C(12)-C(11)-C(10)-C(9)	-0.9(5)
C(9)-C(8)-C(7)-C(4)	178.3(3)
C(1)-C(6)-C(5)-C(4)	0.9(5)
C(6)-C(5)-C(4)-C(3)	-1.5(4)
C(6)-C(5)-C(4)-C(7)	177.5(3)
C(8)-C(7)-C(4)-C(5)	-174.0(3)
C(8)-C(7)-C(4)-C(3)	5.0(5)
C(11)-C(10)-C(9)-C(14)	0.3(4)
C(11)-C(10)-C(9)-C(8)	-179.5(3)
C(7)-C(8)-C(9)-C(10)	174.4(3)
C(7)-C(8)-C(9)-C(14)	-5.4(4)
C(12)-C(13)-C(14)-C(9)	0.1(5)
C(10)-C(9)-C(14)-C(13)	0.1(4)
C(8)-C(9)-C(14)-C(13)	179.9(3)
C(1)-C(2)-C(3)-C(4)	-0.2(5)
C(5)-C(4)-C(3)-C(2)	1.2(4)
C(7)-C(4)-C(3)-C(2)	-177.7(3)
C(10)-C(11)-C(12)-C(13)	1.1(4)
C(10)-C(11)-C(12)-O(1)	-179.6(3)
C(14)-C(13)-C(12)-C(11)	-0.7(4)
C(14)-C(13)-C(12)-O(1)	-180.0(3)
C(5)-C(6)-C(1)-C(2)	0.2(4)
C(5)-C(6)-C(1)-N(1)	-176.7(3)
C(3)-C(2)-C(1)-C(6)	-0.5(4)
C(3)-C(2)-C(1)-N(1)	176.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 73. Crystal data and structure refinement for **1b**.

Identification code	1b	
Empirical formula	C12 H11 N O2	
Formula weight	201.22	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 22.4911(11) Å	α= 90°.
	b = 5.4647(2) Å	β= 95.674(2)°.
	c = 8.0466(4) Å	γ = 90°.
Volume	984.14(8) $Å^3$	
Z	4	
Density (calculated)	1.358 Mg/m^3	
Absorption coefficient	0.093 mm^{-1}	
F(000)	424	
Crystal size	0.4 x 0.4 x 0.1 mm^3	
Theta range for data collection	1.82 to 27.49°.	
Index ranges	-29<=h<=29, -7<=k<=7, -10<=l<=10	
Reflections collected	5187	
Independent reflections	2069 [R(int) = 0.0380]	
Completeness to theta = 27.49°	99.6 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2069 / 2 / 180	
Goodness-of-fit on F^2	1.047	
Final R indices [I>2sigma(I)]	R1 = 0.0329, wR2 = 0.0863	
R indices (all data)	R1 = 0.0332, wR2 = 0.0866	
Absolute structure parameter	0.1(9)	
Largest diff. peak and hole	0.188 and -0.203 $e.Å^{-3}$	

Table 74. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	9272(1)	7725(2)	-984(1)	27(1)
O(2)	7020(1)	7173(2)	1213(1)	25(1)
N(1)	11584(1)	7713(3)	2306(2)	23(1)
C(11)	8028(1)	5530(3)	1287(2)	19(1)
C(12)	7599(1)	7297(2)	788(2)	18(1)
C(9)	8324(1)	9473(3)	-668(2)	20(1)
C(3)	10571(1)	9393(3)	1954(2)	21(1)
C(10)	7750(1)	9291(3)	-159(2)	20(1)
C(8)	8601(1)	5699(3)	777(2)	19(1)
C(5)	10210(1)	5756(3)	-350(2)	21(1)
C(4)	9989(1)	9387(3)	1168(2)	21(1)
C(6)	9818(1)	7591(2)	2(2)	18(1)
C(2)	10792(1)	5766(3)	445(2)	22(1)
C(7)	8740(1)	7654(3)	-233(2)	18(1)
C(1)	10977(1)	7596(3)	1577(2)	18(1)

Table75. Bond lengths [Å] and angles [°] for **1b**.

O(1)-C(7)	1.3940(14)
O(1)-C(6)	1.3964(17)
O(2)-C(12)	1.3814(15)
O(2)-H(12)	0.87(3)
N(1)-C(1)	1.4346(16)
N(1)-H(1A)	0.91(2)
N(1)-H(1B)	0.92(2)
C(11)-C(12)	1.3947(19)
C(11)-C(8)	1.3953(17)
C(11)-H(11)	0.96(2)
C(12)-C(10)	1.3909(19)
C(9)-C(7)	1.386(2)
C(9)-C(10)	1.3946(18)
C(9)-H(9)	0.98(2)
C(3)-C(1)	1.3942(18)
C(3)-C(4)	1.3951(18)
C(3)-H(3)	0.93(2)
C(10)-H(10)	0.97(2)
C(8)-C(7)	1.3956(19)
C(8)-H(8)	0.98(2)
C(5)-C(6)	1.383(2)
C(5)-C(2)	1.399(2)
C(5)-H(5)	0.95(3)
C(4)-C(6)	1.386(2)
C(4)-H(4)	0.95(2)
C(2)-C(1)	1.3883(19)
C(2)-H(2)	1.02(2)
C(7)-O(1)-C(6)	119.83(10)
C(12)-O(2)-H(12)	109.1(17)
C(1)-N(1)-H(1A)	110.6(12)
C(1)-N(1)-H(1B)	109.0(14)
H(1A)-N(1)-H(1B)	111(2)
C(12)-C(11)-C(8)	120.14(12)

C(12)-C(11)-H(11)	120.4(12)
C(8)-C(11)-H(11)	119.5(12)
O(2)-C(12)-C(10)	117.49(12)
O(2)-C(12)-C(11)	122.44(12)
C(10)-C(12)-C(11)	120.06(12)
C(7)-C(9)-C(10)	119.65(12)
C(7)-C(9)-H(9)	121.8(12)
C(10)-C(9)-H(9)	118.5(12)
C(1)-C(3)-C(4)	120.27(12)
C(1)-C(3)-H(3)	120.4(14)
C(4)-C(3)-H(3)	119.3(14)
C(12)-C(10)-C(9)	120.00(12)
C(12)-C(10)-H(10)	119.0(12)
C(9)-C(10)-H(10)	120.9(12)
C(11)-C(8)-C(7)	119.17(12)
C(11)-C(8)-H(8)	123.1(12)
C(7)-C(8)-H(8)	117.7(12)
C(6)-C(5)-C(2)	119.37(13)
C(6)-C(5)-H(5)	118.6(14)
C(2)-C(5)-H(5)	122.0(14)
C(6)-C(4)-C(3)	119.60(12)
C(6)-C(4)-H(4)	120.4(13)
C(3)-C(4)-H(4)	120.0(13)
C(5)-C(6)-C(4)	120.78(13)
C(5)-C(6)-O(1)	117.69(13)
C(4)-C(6)-O(1)	121.17(13)
C(1)-C(2)-C(5)	120.53(12)
C(1)-C(2)-H(2)	120.0(13)
C(5)-C(2)-H(2)	119.4(13)
C(9)-C(7)-O(1)	117.21(12)
C(9)-C(7)-C(8)	120.85(13)
O(1)-C(7)-C(8)	121.60(12)
C(2)-C(1)-C(3)	119.40(12)
C(2)-C(1)-N(1)	120.76(12)
C(3)-C(1)-N(1)	119.80(12)

Symmetry transformations used to generate equivalent atoms:

Table 76. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	14(1)	49(1)	18(1)	2(1)	2(1)	-2(1)
O(2)	14(1)	29(1)	31(1)	5(1)	4(1)	3(1)
N(1)	14(1)	34(1)	22(1)	4(1)	1(1)	1(1)
C(11)	17(1)	19(1)	20(1)	2(1)	0(1)	0(1)
C(12)	13(1)	21(1)	19(1)	-2(1)	1(1)	0(1)
C(9)	19(1)	19(1)	20(1)	2(1)	0(1)	-4(1)
C(3)	20(1)	21(1)	21(1)	-4(1)	2(1)	-2(1)
C(10)	18(1)	17(1)	23(1)	0(1)	-3(1)	2(1)
C(8)	16(1)	21(1)	21(1)	1(1)	-1(1)	4(1)
C(5)	22(1)	20(1)	22(1)	-3(1)	2(1)	-3(1)
C(4)	18(1)	23(1)	24(1)	-3(1)	5(1)	5(1)
C(6)	13(1)	25(1)	18(1)	3(1)	3(1)	-2(1)
C(2)	20(1)	22(1)	25(1)	0(1)	5(1)	5(1)
C(7)	12(1)	25(1)	16(1)	-2(1)	-1(1)	-3(1)
C(1)	13(1)	23(1)	17(1)	5(1)	2(1)	-1(1)

Table 77. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **1b**.

	x	y	z	U(eq)
H(3)	10685(11)	10610(40)	2740(30)	31(5)
H(11)	7933(9)	4200(40)	2000(30)	28(5)
H(10)	7449(9)	10510(40)	-480(30)	28(5)
H(4)	9717(9)	10630(40)	1410(30)	34(5)
H(9)	8413(9)	10850(40)	-1380(30)	31(5)
H(8)	8915(10)	4500(40)	1090(20)	34(5)
H(5)	10076(10)	4540(50)	-1140(30)	42(6)
H(2)	11075(10)	4380(40)	220(30)	40(6)
H(1A)	11599(9)	8040(40)	3420(30)	27(5)
H(1B)	11771(10)	6250(40)	2120(30)	41(6)
H(12)	6953(11)	5710(50)	1590(30)	47(7)

Table 78. Torsion angles [°] for **1b**.

C(8)-C(11)-C(12)-O(2)	177.83(12)
C(8)-C(11)-C(12)-C(10)	-2.8(2)
O(2)-C(12)-C(10)-C(9)	-178.01(12)
C(11)-C(12)-C(10)-C(9)	2.58(19)
C(7)-C(9)-C(10)-C(12)	0.33(18)
C(12)-C(11)-C(8)-C(7)	0.08(19)
C(1)-C(3)-C(4)-C(6)	-0.31(19)
C(2)-C(5)-C(6)-C(4)	-2.1(2)
C(2)-C(5)-C(6)-O(1)	171.01(12)
C(3)-C(4)-C(6)-C(5)	2.2(2)
C(3)-C(4)-C(6)-O(1)	-170.60(12)
C(7)-O(1)-C(6)-C(5)	124.00(14)
C(7)-O(1)-C(6)-C(4)	-62.95(18)
C(6)-C(5)-C(2)-C(1)	0.0(2)
C(10)-C(9)-C(7)-O(1)	170.32(12)
C(10)-C(9)-C(7)-C(8)	-3.1(2)
C(6)-O(1)-C(7)-C(9)	129.17(14)
C(6)-O(1)-C(7)-C(8)	-57.48(18)
C(11)-C(8)-C(7)-C(9)	2.87(19)
C(11)-C(8)-C(7)-O(1)	-170.24(12)
C(5)-C(2)-C(1)-C(3)	1.89(19)
C(5)-C(2)-C(1)-N(1)	-175.60(13)
C(4)-C(3)-C(1)-C(2)	-1.73(19)
C(4)-C(3)-C(1)-N(1)	175.79(12)

Symmetry transformations used to generate equivalent atoms:

Table 79. Crystal data and structure refinement for **6a**.

Identification code	6a	
Empirical formula	C12 H11 N O	
Formula weight	185.22	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pca2(1)	
Unit cell dimensions	a = 19.2352(8) Å	α= 90°.
	b = 6.0039(3) Å	β= 90°.
	c = 8.1627(4) Å	γ = 90°.
Volume	942.68(8) $Å^3$	
Z	4	
Density (calculated)	1.305 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	392	
Crystal size	0.40 x 0.14 x 0.04 mm^3	
Theta range for data collection	2.12 to 27.11°.	
Index ranges	-24<=h<=24, -7<=k<=7, -10<=l<=10	
Reflections collected	9004	
Independent reflections	2083 [R(int) = 0.0639]	
Completeness to theta = 27.11°	100.0 %	
Absorption correction	None	
Max. and min. transmission	. and .	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2083 / 1 / 139	
Goodness-of-fit on F^2	1.033	
Final R indices [I>2sigma(I)]	R1 = 0.0378, wR2 = 0.0930	
R indices (all data)	R1 = 0.0519, wR2 = 0.1000	
Absolute structure parameter	0.3(17)	
Extinction coefficient	not refined	
Largest diff. peak and hole	0.211 and -0.159 e.$Å^{-3}$	

Table 80. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **6a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3192(1)	10099(2)	1999(2)	30(1)
N(1)	7619(1)	12374(3)	4411(2)	30(1)
C(1)	3849(1)	10934(3)	2158(2)	25(1)
C(2)	4038(1)	12713(3)	1190(2)	32(1)
C(3)	4704(1)	13589(3)	1286(2)	31(1)
C(4)	5200(1)	12740(3)	2364(2)	25(1)
C(5)	4989(1)	10969(3)	3349(3)	35(1)
C(6)	4329(1)	10049(3)	3253(3)	33(1)
C(7)	5911(1)	13718(3)	2485(2)	26(1)
C(8)	6435(1)	12624(3)	3363(2)	26(1)
C(9)	7092(1)	13567(3)	3554(2)	27(1)
C(10)	7241(1)	15617(3)	2838(2)	32(1)
C(11)	6733(1)	16689(3)	1943(3)	33(1)
C(12)	6073(1)	15769(3)	1766(2)	32(1)

Table 81. Bond lengths [Å] and angles [°] for **6a**.

O(1)-C(1)	1.3647(19)
O(1)-H(1)	0.91(2)
N(1)-C(9)	1.425(2)
N(1)-H(1A)	0.88(2)
N(1)-H(1B)	0.94(2)
C(1)-C(2)	1.378(2)
C(1)-C(6)	1.391(2)
C(2)-C(3)	1.386(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.395(2)
C(3)-H(3)	0.9500
C(4)-C(5)	1.393(2)
C(4)-C(7)	1.491(2)
C(5)-C(6)	1.385(2)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
C(7)-C(12)	1.400(2)
C(7)-C(8)	1.402(2)
C(8)-C(9)	1.393(2)
C(8)-H(8)	0.9500
C(9)-C(10)	1.392(2)
C(10)-C(11)	1.381(3)
C(10)-H(10)	0.9500
C(11)-C(12)	1.391(2)
C(11)-H(11)	0.9500
C(12)-H(12)	0.9500
C(1)-O(1)-H(1)	113.5(12)
C(9)-N(1)-H(1A)	114.2(17)
C(9)-N(1)-H(1B)	111.7(13)
H(1A)-N(1)-H(1B)	110(2)
O(1)-C(1)-C(2)	118.36(16)
O(1)-C(1)-C(6)	122.41(16)
C(2)-C(1)-C(6)	119.23(16)

C(1)-C(2)-C(3)	120.41(17)
C(1)-C(2)-H(2)	119.8
C(3)-C(2)-H(2)	119.8
C(2)-C(3)-C(4)	121.94(18)
C(2)-C(3)-H(3)	119.0
C(4)-C(3)-H(3)	119.0
C(5)-C(4)-C(3)	116.30(17)
C(5)-C(4)-C(7)	122.00(15)
C(3)-C(4)-C(7)	121.68(16)
C(6)-C(5)-C(4)	122.60(17)
C(6)-C(5)-H(5)	118.7
C(4)-C(5)-H(5)	118.7
C(5)-C(6)-C(1)	119.50(17)
C(5)-C(6)-H(6)	120.2
C(1)-C(6)-H(6)	120.2
C(12)-C(7)-C(8)	117.78(16)
C(12)-C(7)-C(4)	121.57(16)
C(8)-C(7)-C(4)	120.64(15)
C(9)-C(8)-C(7)	121.34(17)
C(9)-C(8)-H(8)	119.3
C(7)-C(8)-H(8)	119.3
C(10)-C(9)-C(8)	119.95(17)
C(10)-C(9)-N(1)	120.25(16)
C(8)-C(9)-N(1)	119.72(17)
C(11)-C(10)-C(9)	119.23(17)
C(11)-C(10)-H(10)	120.4
C(9)-C(10)-H(10)	120.4
C(10)-C(11)-C(12)	121.08(17)
C(10)-C(11)-H(11)	119.5
C(12)-C(11)-H(11)	119.5
C(11)-C(12)-C(7)	120.60(17)
C(11)-C(12)-H(12)	119.7
C(7)-C(12)-H(12)	119.7

Symmetry transformations used to generate equivalent atoms:

Table 82. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **6a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	22(1)	38(1)	30(1)	4(1)	-3(1)	-4(1)
N(1)	22(1)	38(1)	30(1)	-1(1)	-1(1)	-3(1)
C(1)	21(1)	29(1)	25(1)	-5(1)	1(1)	1(1)
C(2)	26(1)	37(1)	32(1)	8(1)	-5(1)	1(1)
C(3)	30(1)	32(1)	32(1)	6(1)	0(1)	-1(1)
C(4)	22(1)	27(1)	26(1)	-2(1)	2(1)	3(1)
C(5)	26(1)	38(1)	40(1)	10(1)	-8(1)	-2(1)
C(6)	26(1)	34(1)	38(1)	9(1)	-2(1)	-2(1)
C(7)	24(1)	28(1)	27(1)	-5(1)	1(1)	1(1)
C(8)	24(1)	29(1)	23(1)	-3(1)	3(1)	-1(1)
C(9)	25(1)	32(1)	25(1)	-7(1)	2(1)	3(1)
C(10)	25(1)	35(1)	35(1)	-6(1)	7(1)	-8(1)
C(11)	32(1)	29(1)	38(1)	1(1)	5(1)	-4(1)
C(12)	28(1)	31(1)	35(1)	3(1)	1(1)	0(1)

Table 83. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **6a**.

	x	y	z	U(eq)
H(1)	3066(9)	9170(30)	2840(30)	35(6)
H(1A)	7459(14)	11430(30)	5150(30)	57(8)
H(1B)	7946(11)	13340(40)	4890(30)	40(6)
H(2)	3711	13343	452	38
H(3)	4825	14805	598	38
H(5)	5309	10370	4116	42
H(6)	4206	8824	3930	39
H(8)	6341	11211	3838	31
H(10)	7688	16270	2965	38
H(11)	6835	18077	1439	39
H(12)	5729	16541	1151	38

Table 84. Torsion angles [°] for **6a**.

O(1)-C(1)-C(2)-C(3)	-178.76(17)
C(6)-C(1)-C(2)-C(3)	1.0(3)
C(1)-C(2)-C(3)-C(4)	-0.8(3)
C(2)-C(3)-C(4)-C(5)	-0.5(3)
C(2)-C(3)-C(4)-C(7)	-178.79(17)
C(3)-C(4)-C(5)-C(6)	1.5(3)
C(7)-C(4)-C(5)-C(6)	179.79(17)
C(4)-C(5)-C(6)-C(1)	-1.2(3)
O(1)-C(1)-C(6)-C(5)	179.72(17)
C(2)-C(1)-C(6)-C(5)	-0.1(3)
C(5)-C(4)-C(7)-C(12)	-165.93(18)
C(3)-C(4)-C(7)-C(12)	12.3(3)
C(5)-C(4)-C(7)-C(8)	12.5(3)
C(3)-C(4)-C(7)-C(8)	-169.27(17)
C(12)-C(7)-C(8)-C(9)	1.6(3)
C(4)-C(7)-C(8)-C(9)	-176.86(15)
C(7)-C(8)-C(9)-C(10)	-1.2(2)
C(7)-C(8)-C(9)-N(1)	-178.00(17)
C(8)-C(9)-C(10)-C(11)	-0.1(3)
N(1)-C(9)-C(10)-C(11)	176.68(17)
C(9)-C(10)-C(11)-C(12)	0.9(3)
C(10)-C(11)-C(12)-C(7)	-0.5(3)
C(8)-C(7)-C(12)-C(11)	-0.8(3)
C(4)-C(7)-C(12)-C(11)	177.71(16)

Symmetry transformations used to generate equivalent atoms:

(iii) Table 85. Geometrical Parameters of Hydrogen bonds for the compounds in this study

Compound	H-bond	*d* (Å)[a]	*D* (Å)	*θ* (deg)
1 -Neutron	O–H···N	1.76	2.723(7)	167.8
	N–H···O	1.99	2.908(5)	150.2
	N–H···π	2.39	3.334	155.5
	C–H···π	2.57	3.536	147.6
	C–H···π	2.66	3.727	173.1
1 -X-ray	O–H···N	1.77	2.7485(18)	170.5
	N–H···O	1.99	2.9281(17)	152.2
	N–H···π	2.43	3.377	156.0
	C–H···π	2.63	3.582	145.9
	C–H···π	2.72	3.797	172.1
2	O–H···N	1.84	2.815(2)	173.3
	N–H···O	2.13	3.130(2)	169.7
	N–H···O	2.30	3.312(2)	177.3
3	O–H···N	1.79	2.776(2)	172.5
	N–H···O	2.26	3.205(2)	154.3
	N–H···π	2.38	3.354	161.2
	C–H···O	2.53	3.268 (2)	124.5
	C–H···O	2.59	3.564(2)	149.3
4	O–H···N	1.88	2.818(3)	159.0
	N–H···O	2.15	3.155(3)	172.3
	N–H···O	2.29	3.307(3)	174.5
5	O–H···N	1.80	2.758(2)	163.1
	O–H···N	1.82	2.788(2)	169.0
	N–H···O	2.09	3.067(2)	162.7
	N–H···O	2.28	3.181(2)	146.9
	N–H···O	2.57	3.461(2)	146.4
	N–H···π	2.60	3.542	154.2
	C–H···O	2.47	3.305(2)	132.9
	C–H···O	2.60	3.298(2)	121.0
	C–H···O	2.62	3.312(2)	120.5
	C–H···O	2.67	3.510(2)	133.8

Table 85. (contd)

Compound	H-bond	*d* (Å)[a]	*D* (Å)	*θ* (deg)
1a	O–H···N	1.77	2.745(2)	171.5
	O–H···N	1.91	2.871(2)	164.6
	N–H···O	2.06	3.001(2)	153.7
	N–H···O	2.31	3.261(2)	156.9
	N–H···S	2.91	3.875(2)	158.9
	N–H···S	3.15	4.016(2)	150.2
	C–H···S	2.78	3.833(2)	163.7
	C–H···S	2.83	3.646(2)	131.9
	C–H···S	2.90	3.7428(19)	134.4
	C–H···S	2.96	3.9422(19)	150.6
	C–H···π	2.56	3.329	126.5
	C–H···O	2.77	3.634(2)	136.4
2a	O–H···N	1.80	2.786(4)	178.1
	N–H···O	2.15	3.148(5)	172.1
	N–H···O	2.30	3.299(4)	169.6
	C–H···S	2.86	3.849(4)	152.3
	C–H···S	3.02	3.774(4)	127.2
2b	O–H···N	1.84	2.822(4)	173.3
	N–H···O	2.11	3.119(4)	177.1
	N–H···O	2.28	3.268(4)	166.9
	C–H···S	2.97	3.787(3)	132.8
	C–H···S	3.03	3.984(3)	146.5
	C–H···S	3.06	4.045(3)	151.8
2c	O–H···N	1.78	2.7623(19)	176.6
	N–H···O	2.04	3.031(2)	166.7
	N–H···π	2.58	3.530	156.4
	C–H···O	2.64	3.449(2)	130.4
	C–H···S	2.88	3.6853(17)	131.3
	C–H···S	2.93	3.1300	169.4
	C–H···π	2.56	3.629	167.5
3a	O–H···N	1.86	2.754(2)	149.7
	N–H···O	2.11	3.114(2)	174.1
	N–H···π	2.51	3.382	143.7
	C–H···O	2.51	3.199(2)	120.7
	C–H···O	2.51	3.192(2)	120.3
	C–H···S	2.91	3.9167(17)	154.9

C–H···S	2.92	3.8520(18)	144.1
C–H···π	2.67	3.426	126.5

Table 85. (contd)

Compound	H-bond	*d* (Å)[a]	*D* (Å)	*θ* (deg)
2d	O–H···N	1.84	2.819(4)	170.1
	N–H···O	2.12	3.125(4)	170.6
	N–H···O	2.26	3.253(4)	165.9
1b	O–H···N	1.84	2.8008(19)	163.8
	N–H···O	2.26	3.1991(16)	153.3
	N–H···O	2.34	3.3271(19)	164.7
	C–H···π	2.68	3.565	138.5
	C–H···π	2.71	3.612	139.5
	C–H···π	2.74	3.631	139.0
	C–H···π	2.77	3.654	138.5
6a	O–H···N	1.73	2.701(2)	166.0
	N–H···O	2.05	3.017(2)	160.6
	N–H···π	2.51	3.359	141.8
	C–H···O	2.43	3.230(2)	129.9
	C–H···π	2.52	3.494	149.1
	C–H···π	2.80	3.736	144.5

[a] O–H, N–H and C–H distances are neutron normalized to 0.983, 1.009 and 1.083.

Table 86. The geometry of the N_1–H_{1b}···π hydrogen bonds in Compound 1(X and N)

	X-ray			Neutron		
H-bond	***d* (Å)**[a]	***D* (Å)**	**θ (deg)**	***d* (Å)**	***D* (Å)**	**θ (deg)**
N_1–H_{1b}···π	2.43	3.377	156.0	2.39	3.334	155.5
H_{1b}···C_1	2.59	3.401	136.4	2.56	3.359	136.5
H_{1b}···C_2	2.84	3.546	127.3	2.81	3.507	126.8
H_{1b}···C_3	3.02	3.776	132.8	2.98	3.726	131.8
H_{1b}···C_4	2.99	3.889	148.3	2.97	3.852	147.1
H_{1b}···C_5	2.76	3.764	170.0	2.74	3.724	168.9
H_{1b}···C_6	2.56	3.523	159.0	2.53	3.486	159.9
N_1–H_{1b}···π (C_5–C_6)	2.57	3.578	173.7	2.54	3.539	174.7

[a] N–H distance is neutron normalized to1.009.

Table 87. The geometry of the C_9–H_9···π hydrogen bonds in Compound 1(X and N)

	X-ray			Neutron		
H-bond	***d* (Å)**[a]	***D* (Å)**	**θ (deg)**	***d* (Å)**	***D* (Å)**	**θ (deg)**
C_9–H_9···π	2.72	3.797	172.1	2.65	3.727	173.1
H_9···C_1	3.10	4.056	147.6	3.04	3.995	148.1
H_9···C_2	3.00	4.003	154.0	2.94	3.939	153.9
H_9···C_3	2.96	4.001	160.0	2.92	3.943	158.8
H_9···C_4	3.04	4.057	156.6	2.98	3.985	155.2
H_9···C_5	3.09	4.073	150.0	3.03	4.003	149.5
H_9···C_6	3.13	4.080	146.0	3.06	4.008	146.3

[a] C–H distance is neutron normalized to1.083.

Table 88. The geometry of the C_5–H_5···π hydrogen bonds in Compound 1(X and N)

	X-ray			Neutron		
H-bond	***d* (Å)**[a]	***D* (Å)**	**θ (deg)**	***d* (Å)**	***D* (Å)**	**θ (deg)**
C_5–H_5···π	2.63	3.582	145.9	2.57	3.536	147.6
H_5···C_8	3.01	3.739	125.2	2.94	3.698	127.1
H_5···C_9	2.89	3.842	146.6	2.83	3.798	148.8
H_5···C_{10}	2.87	3.951	174.0	2.82	3.901	176.6

$H_5\cdots C_{11}$	2.95	3.950	152.3	2.91	3.903	151.6
$H_5\cdots C_{12}$	3.06	3.844	129.5	3.01	3.795	129.7
$H_5\cdots C_{13}$	3.07	3.732	119.5	3.02	3.693	120.7
$C_5–H_5\cdots\pi$ ($C_9–C_{10}$)	2.79	3.834	160.5	2.74	3.786	162.9

[a] C–H distance is neutron normalized to1.083.

(iv) Details of CSD searches

A CSD search on C-S-C:

A search of the CSD (Version 5.23, April 2002) for error free C-S-C organic acyclic structures, non-ionic, non polymeric structures with R<0.10. Disordered, 'no co-ordinates present' structures were excluded from the search. Of the 589 entries in the subset, 21 hits with corresponding C-C-C were found. Of which however, we found only 8 pairs of isostructural. (Isostructural C-S-C and C-C-C pairs respectively: DAPHSD, CEHCOH; HPTBZC, GUHKUP; LMETON02, LNLEUC10; MDTHAC, PIMELA04; MDTPRA, AZELAC03; TGLYCL/01, GLURAC03/04; ZAGKAT, ZAGJUM; ZZZPZE01, PIMELA). (Non-isostructural pairs: BIRFEN, ZZZEMS01/02/03; BODPEP, SUBRAC05; CUZZAY, CUZYEB; DABMOI, DABKUM; GALCEB, GALCAX; LIQWAJ, BITVUV; LIQWIR, PENTAN01; NTPXMB10, CBYMBZ; PHMESF, BOKCEJ; VUKPOG, JEMTIE; WANWOX, WANXOY; WANWEN, WANWUD; WANWIR, WANXEO.

A CSD search on Benzene-thiophene exchange:

A search of the CSD (Version 5.23, April 2002) for error free thiophene only organic, non-ionic, non polymeric structures with R<0.10. 'No co-ordinates present' structures were excluded from the search. Of the 8 entries in the subset, 2 hits with the

corresponding benzene structure were found and they are isostructural (VEVJUB, FACROQ; WOZVEX, WOZWAT).

A CSD search on C-S-S-C torsion angle:

A search of the CSD (Version 5.23, April 2002) for error free C-S-S-C organic, aromatic, acyclic, non-ionic, non polymeric structures with R<0.10. Disordered, 'no co-ordinates present' structures were excluded from the search. Of the 51 hits with torsion angle of approximately 90° were found.

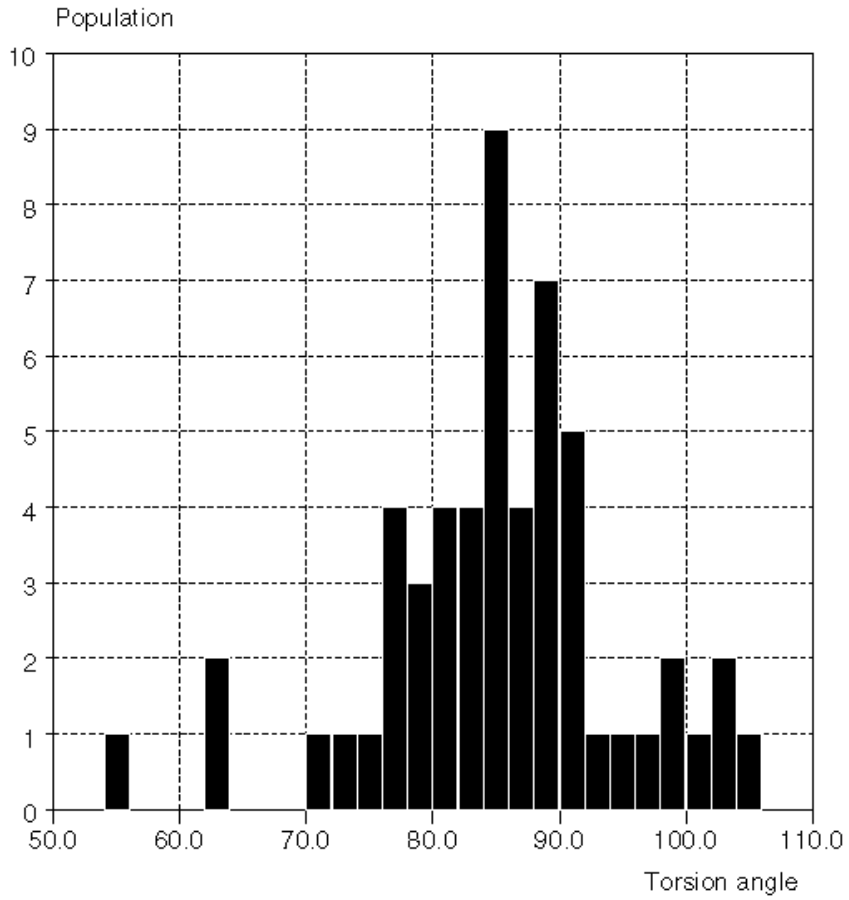


Figure 1. C-S-S-C torsion angle

A CSD search on C–O–C and C–C–C:

A search of the CSD (Version 5.23, April 2002) for error free Ar–O–Ar organic, aromatic, acyclic, non-ionic, non polymeric structures with R<0.075. Disordered, 'no co-ordinates present' structures were excluded from the search. Of the 418 entries in the subset, 4 hits with corresponding C–O–C were found. Of which however, only one pair is isostructural CAKGOK, GUHKUP. (Non-isostructural pairs: NPXMBZ10, CBYMBZ; CUCVER, CEHCOH; XILQOY, XILRAL).

CSD search on C–O–C angle:

A search of the CSD (Version 5.23, November 2002) for error free C-O-C organic, aromatic, acyclic, non-ionic, non polymeric structures with R<0.10. Disordered, 'no co-ordinates present' structures were excluded from the search. Of the 168 entries in the subset the highest population of C-O-C angle is in between 118 - 120°.

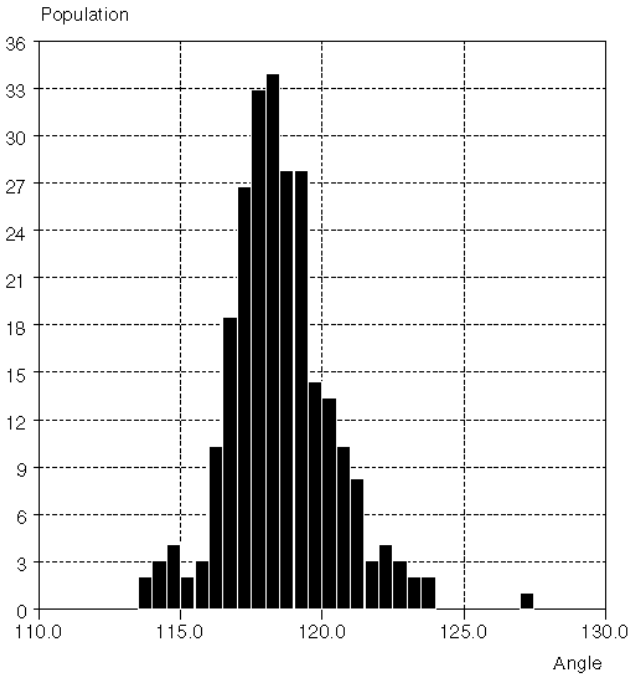
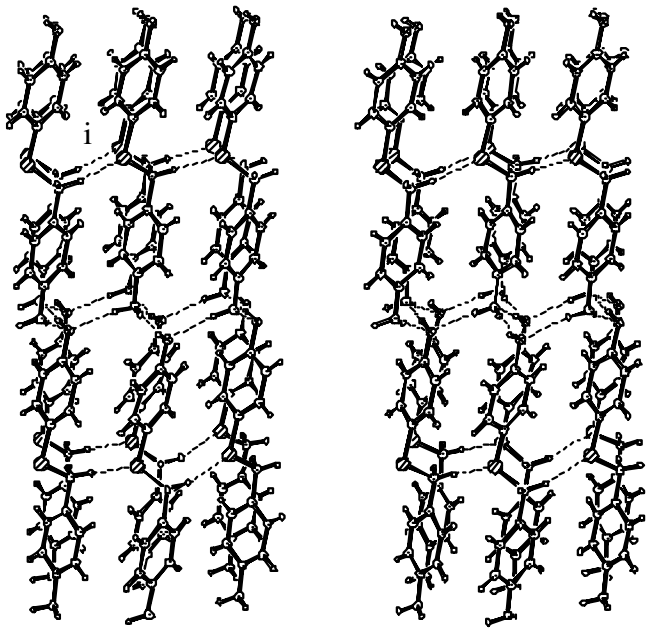


Figure 2. C–O–C angle

(v) Additional stereoviews of the crystal structures

a



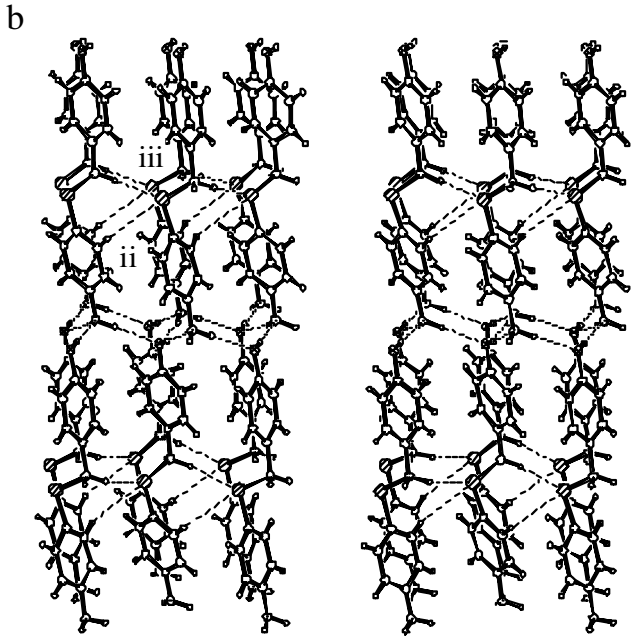


Figure 3. (a) Stereoview of the overall crystal packing in **2a**. C–H···S (i) (2.86Å, 152.3°) interaction, (b) in **2b.** C–H···S (ii) (2.97Å, 132.8°), (iii) (3.03Å, 146.5°).

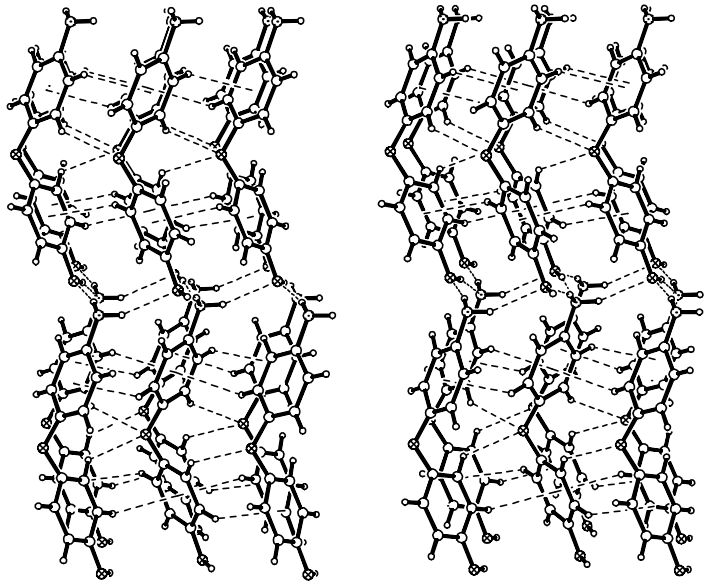


Figure 4. Stereoview of the overall crystal packing in **1b.** Notice the bifurcated C–H···O and also C–H···π interactions.

(vi) Lattice parameters of selected compounds

Table 89. Lattice parameters of 2, 2a and 2b in *Pc* space group

compound	*a* [Å]	*b* [Å]	*c* [Å]	*α* [°]	*β* [°]	*γ* [°]
2	13.682(3)	5.2619(11)	8.1916(2)	90	107.28(3)	90
2a	13.844(3)	5.1626(10)	8.2485(16)	90	107.22(3)	90
2b	13.7422(12)	5.1725(4)	8.3055(7)	90	105.548(4)	90

Unit cell similarity Index **(Π) =** 0.00031 for **2b** in relation to **2a**.
Unit cell similarity Index **(Π) =** 0.0047 for **2** in relation to **2b**.
(where **zero** indicates an exact match).

Table 90. Lattice parameters of 2d in *Pc* space group: (Compare with **2**)

compound	*a* [Å]	*B* [Å]	*c* [Å]	*α* [°]	*β* [°]	*γ* [°]
2d	12.951(8)	5.226(3)	8.046(3)	90	98.12(3)	90

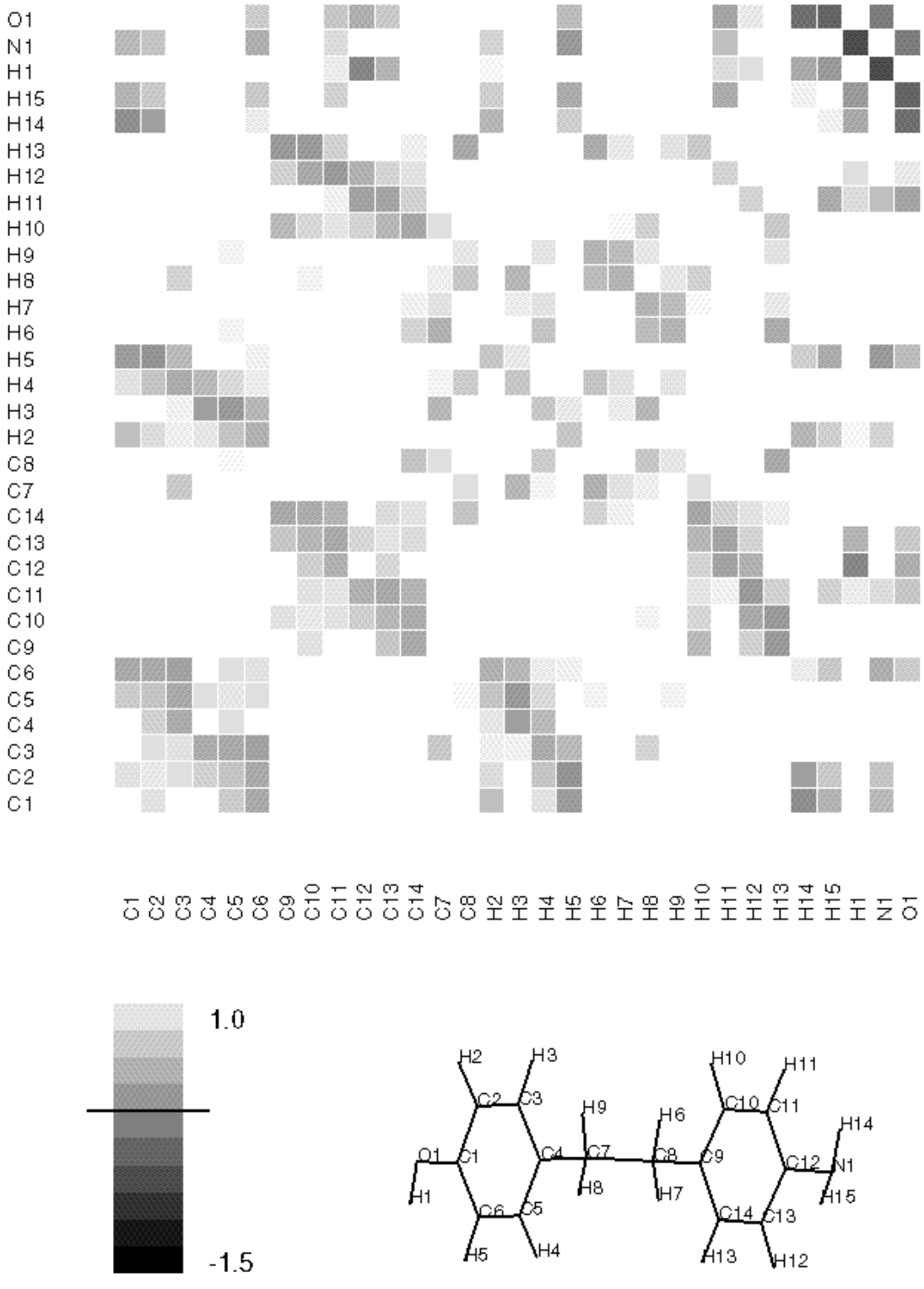
The value of **Π** for **2** in relation to **2d** is 0.027

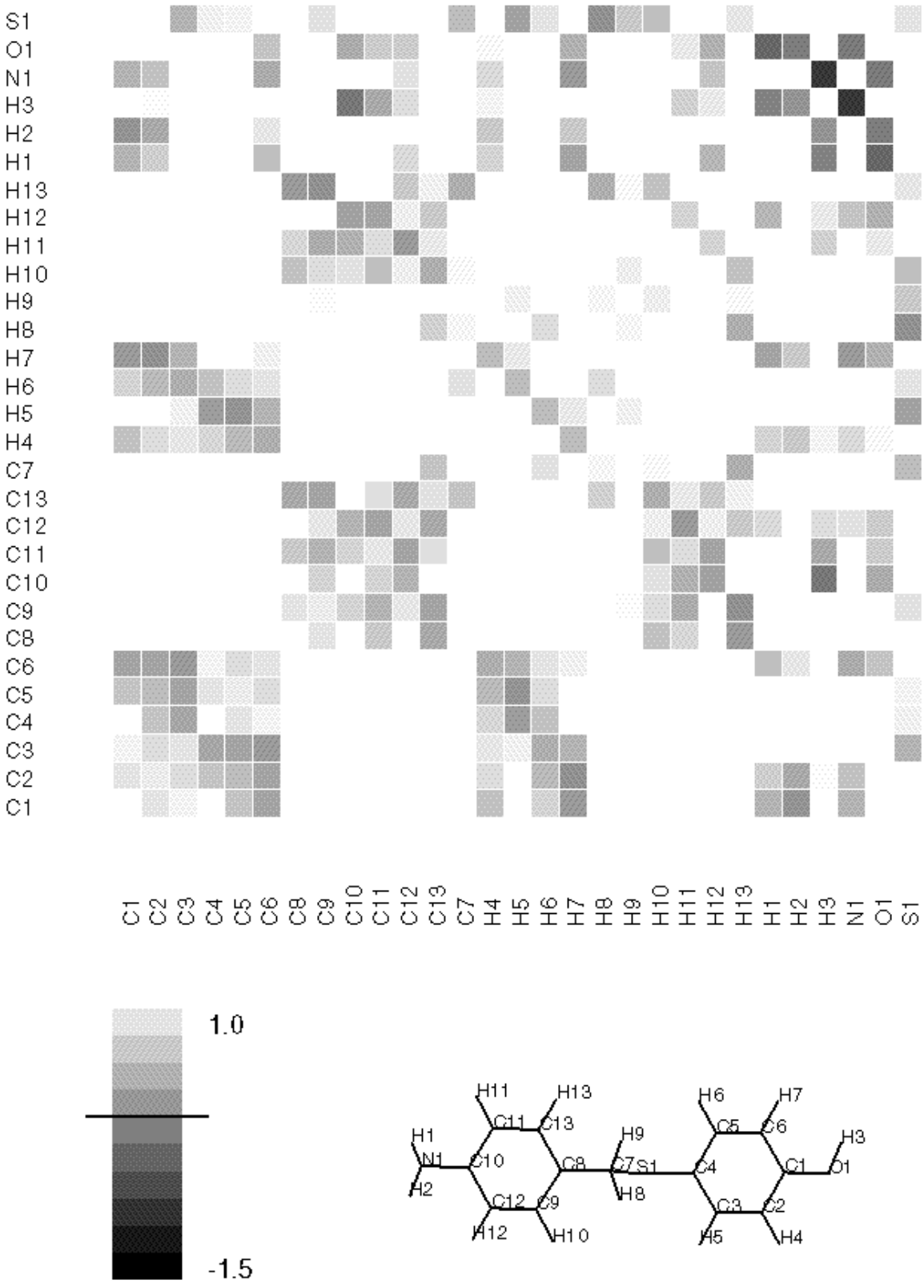
Table 91. Lattice parameters of 3AP**, 3 and 6a in *Pca*2_1 space group**

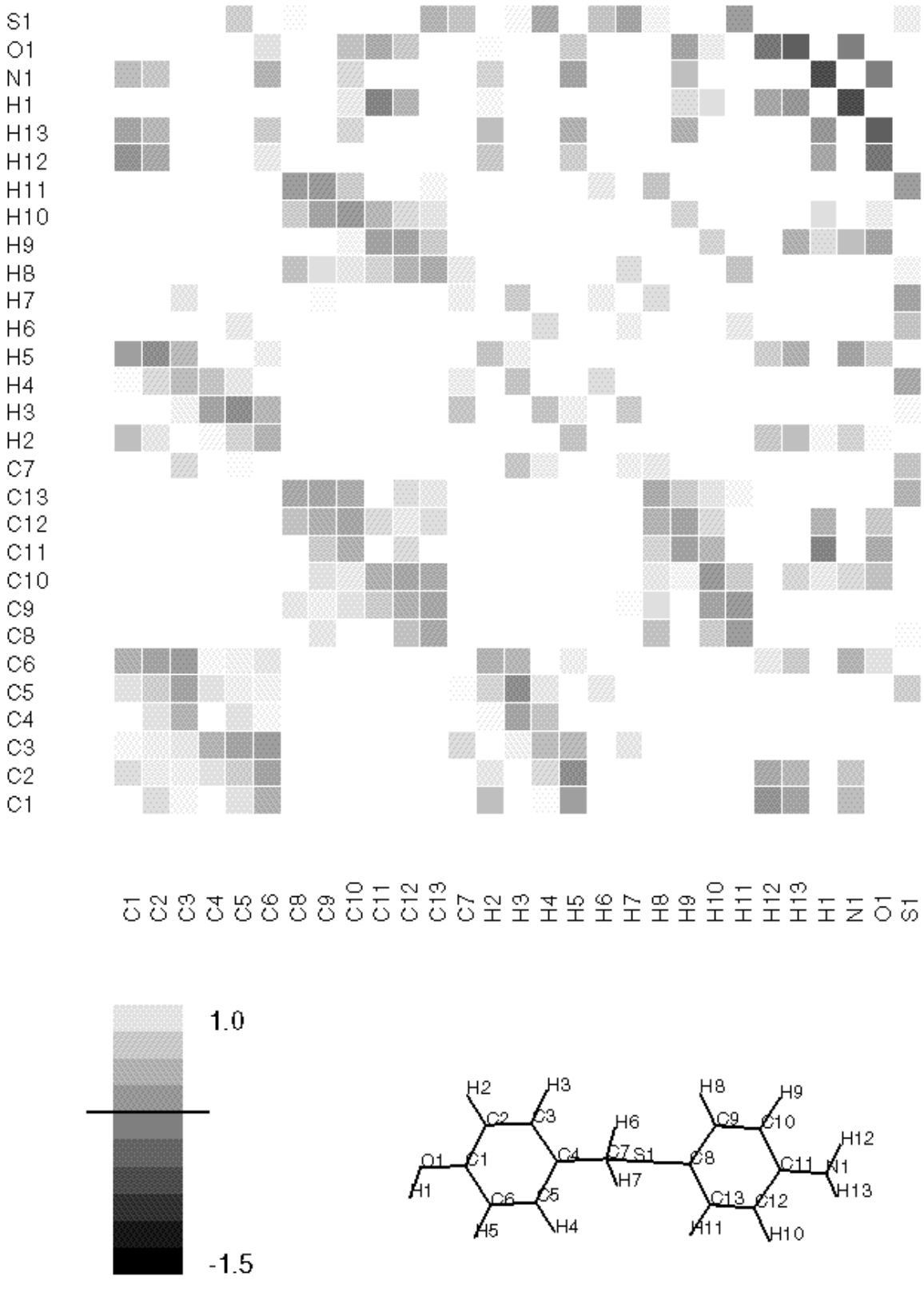
compound	*a* [Å]	*B* [Å]	*c* [Å]	*α* [°]	*β* [°]	*γ* [°]
3AP	11.226	6.101	8.282	90	90	90
3	23.9370(7)	6.2160(2)	8.3970(3)	90	90	90
6a	19.2352(8)	6.0039(3)	8.1627(4)	90	90	90

The value of **Π** for **6a** in relation to **3** is 0.15

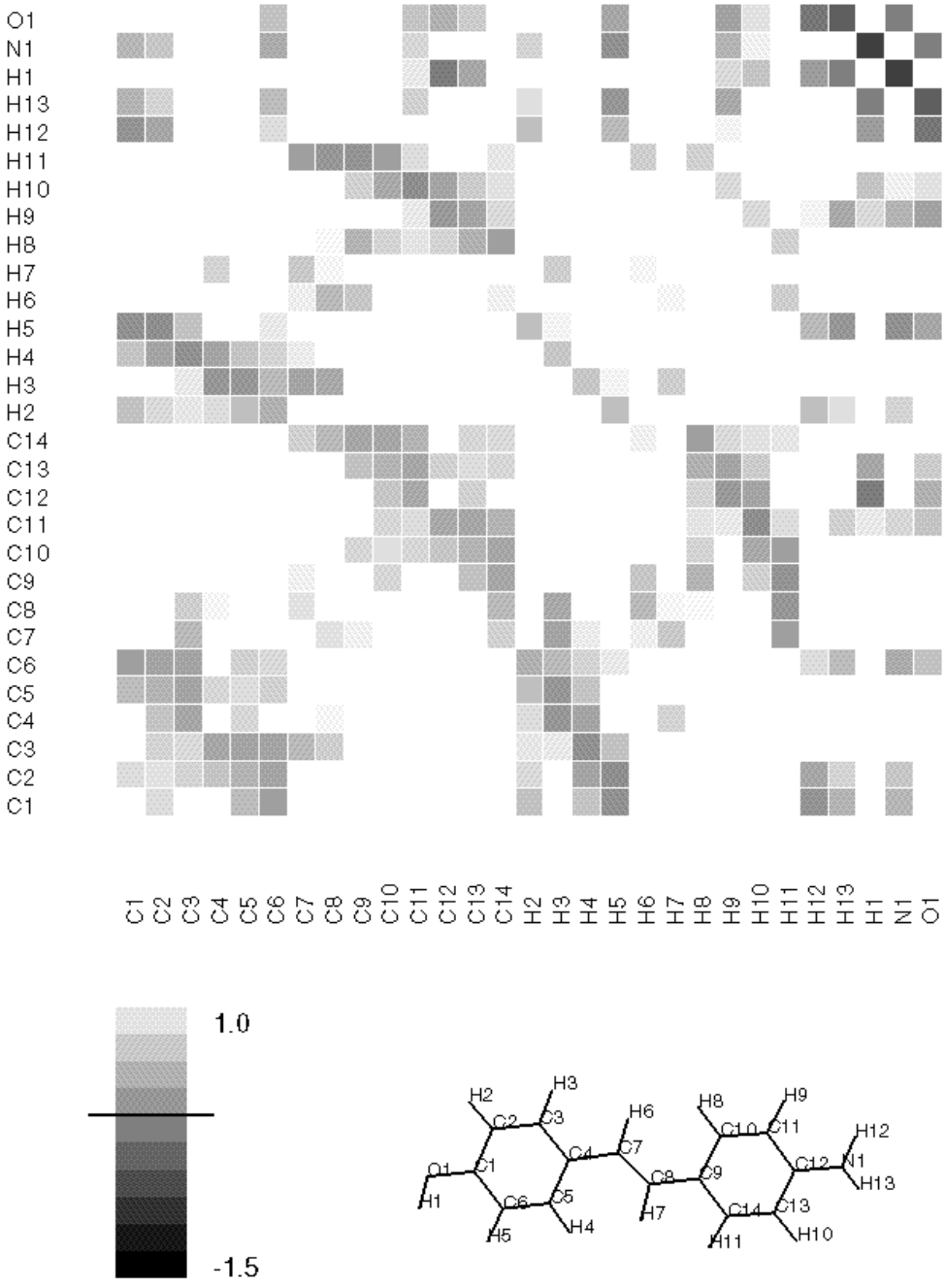
(vii) NIPMAT plots (For details of these plots see *Acc. Chem. Res*. **1996**, *29*, 441).
NIPMAT plots of **2, 2a** and **2b** shows the near identity of the packing:


O1
N1
H1
H15
H14
H13
H12
H11
H10
H9
H8
H7
H6
H5
H4
H3
H2
C8
C7
C14
C13
C12
C11
C10
C9
C6
C5
C4
C3
C2
C1
C1 C2 C3 C4 C5 C6 C9 C10 C11 C12 C13 C14 C7 C8 H2 H3 H4 H5 H6 H7 H8 H9 H10 H11 H12 H13 H14 H15 H1 N1 O1
1.0
-1.5
H2 H3 H10 H11
C2 C3 H9 H6 C10 C11 H14
O1 C1 C4 C7 C8 C9 C12 N1
H1 H8 H7 H15
C6 C5 C14 C13
H5 H4 H13 H12

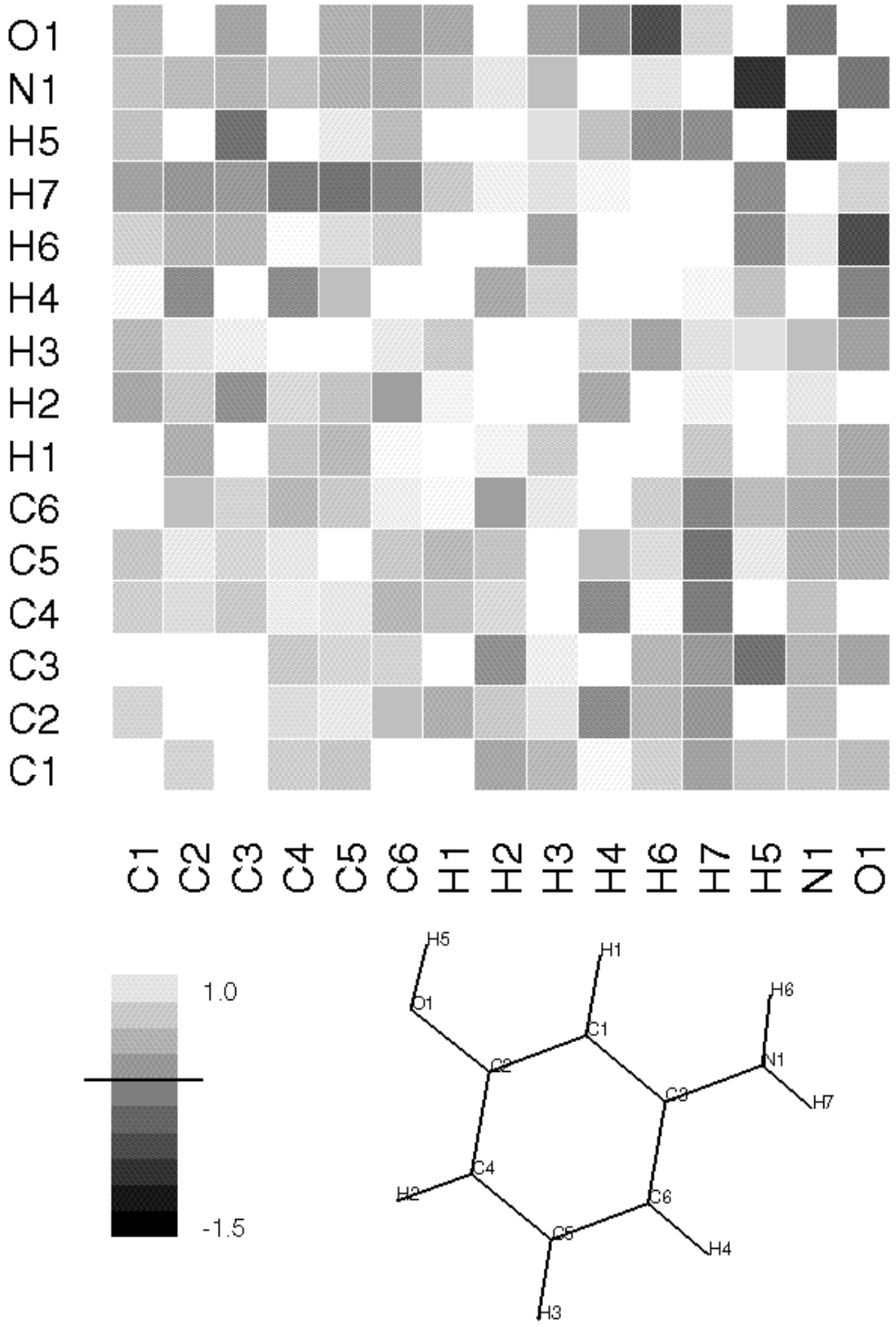

S1
O1
N1
H3
H2
H1
H13
H12
H11
H10
H9
H8
H7
H6
H5
H4
C7
C13
C12
C11
C10
C9
C8
C6
C5
C4
C3
C2
C1
C1 C2 C3 C4 C5 C6 C8 C9 C10 C11 C12 C13 C7 H4 H5 H6 H7 H8 H9 H10 H11 H12 H13 H1 H2 H3 N1 O1 S1
1.0
-1.5
H11 H13 H6 H7
C11 C13 C5 C6
H1 H9 H3
N1 C10 C8 C7 S1 C4 C1 O1
H2 H8
C12 C9 C3 C2
H12 H10 H5 H4

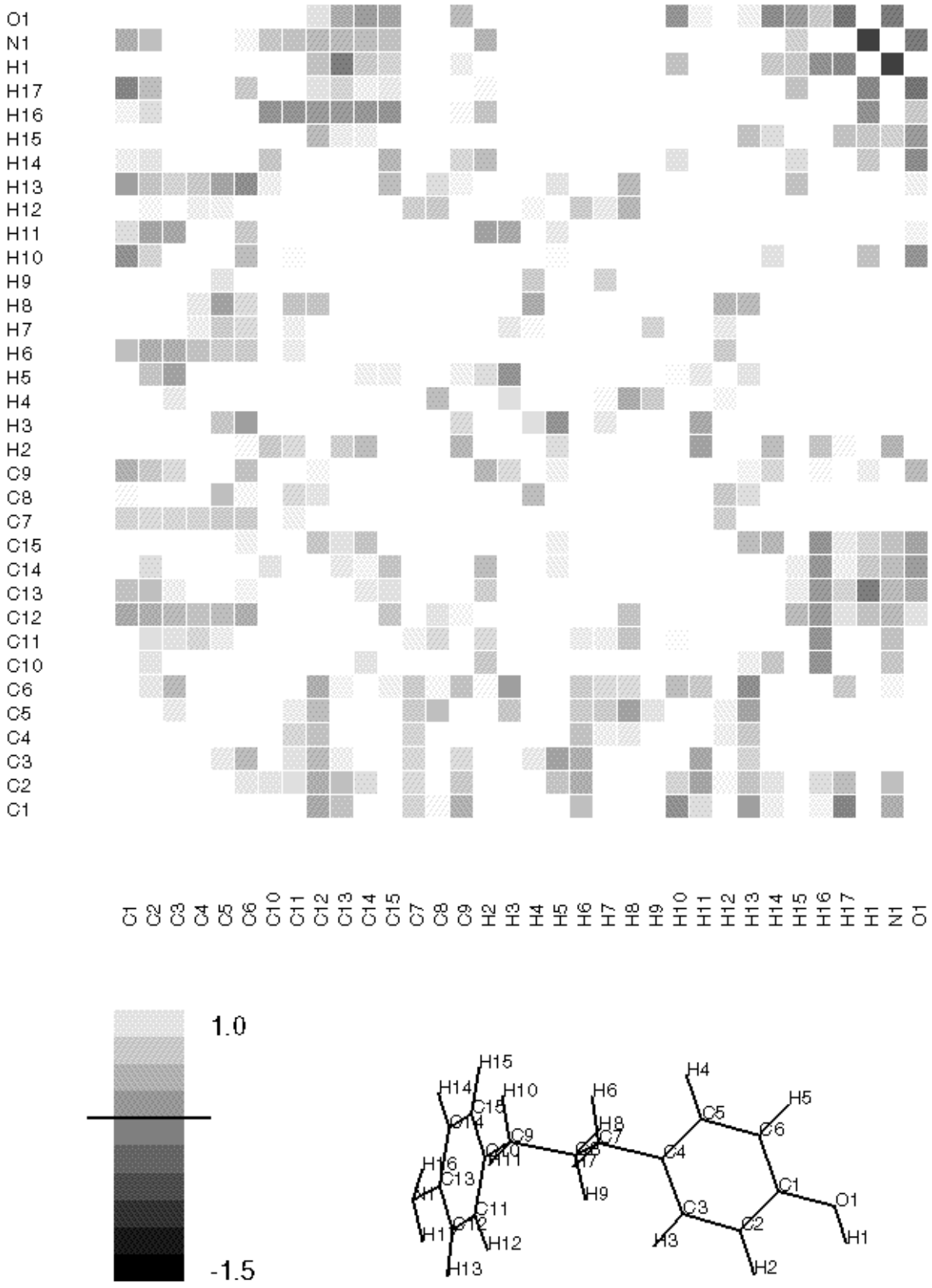

S1
O1
N1
H1
H13
H12
H11
H10
H9
H8
H7
H6
H5
H4
H3
H2
C7
C13
C12
C11
C10
C9
C8
C6
C5
C4
C3
C2
C1
C1 C2 C3 C4 C5 C6 C8 C9 C10 C11 C12 C13 C7 H2 H3 H4 H5 H6 H7 H8 H9 H10 H11 H12 H13 H1 N1 O1 S1
1.0
-1.5
H2 H3 H8 H9
C2 C3 C9 C10
H6
O1 C1 C4 C7 S1 C8 C11 N1 H12
H1 H7 H13
C6 C5 C13 C12
H5 H4 H11 H10

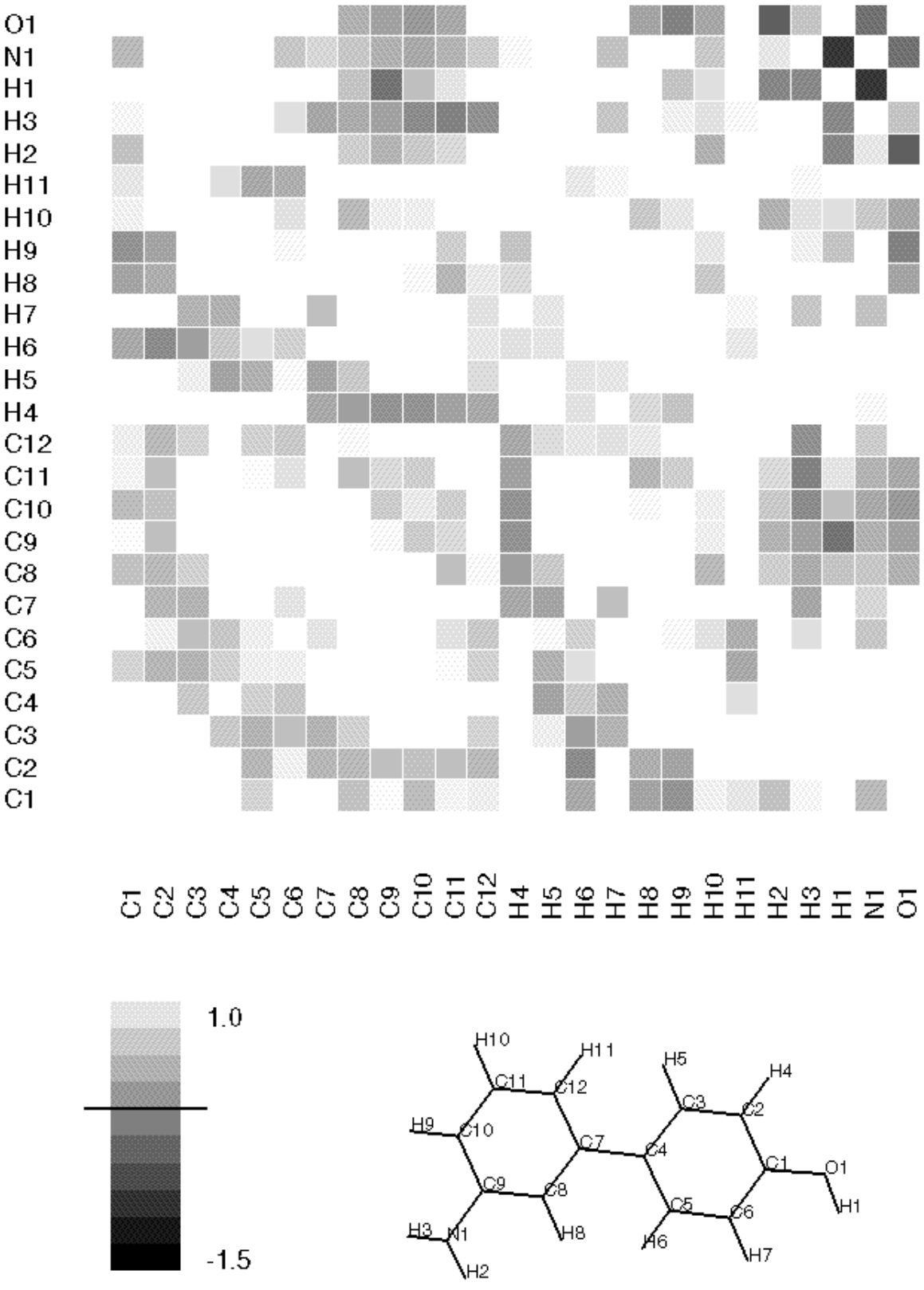
Nipmat plot of **2d**: Compare with compound **2**, shows the near identity of the packing.



NIPMAT plots of 3AP, **3** and **6a** shows the near identity of the packing:

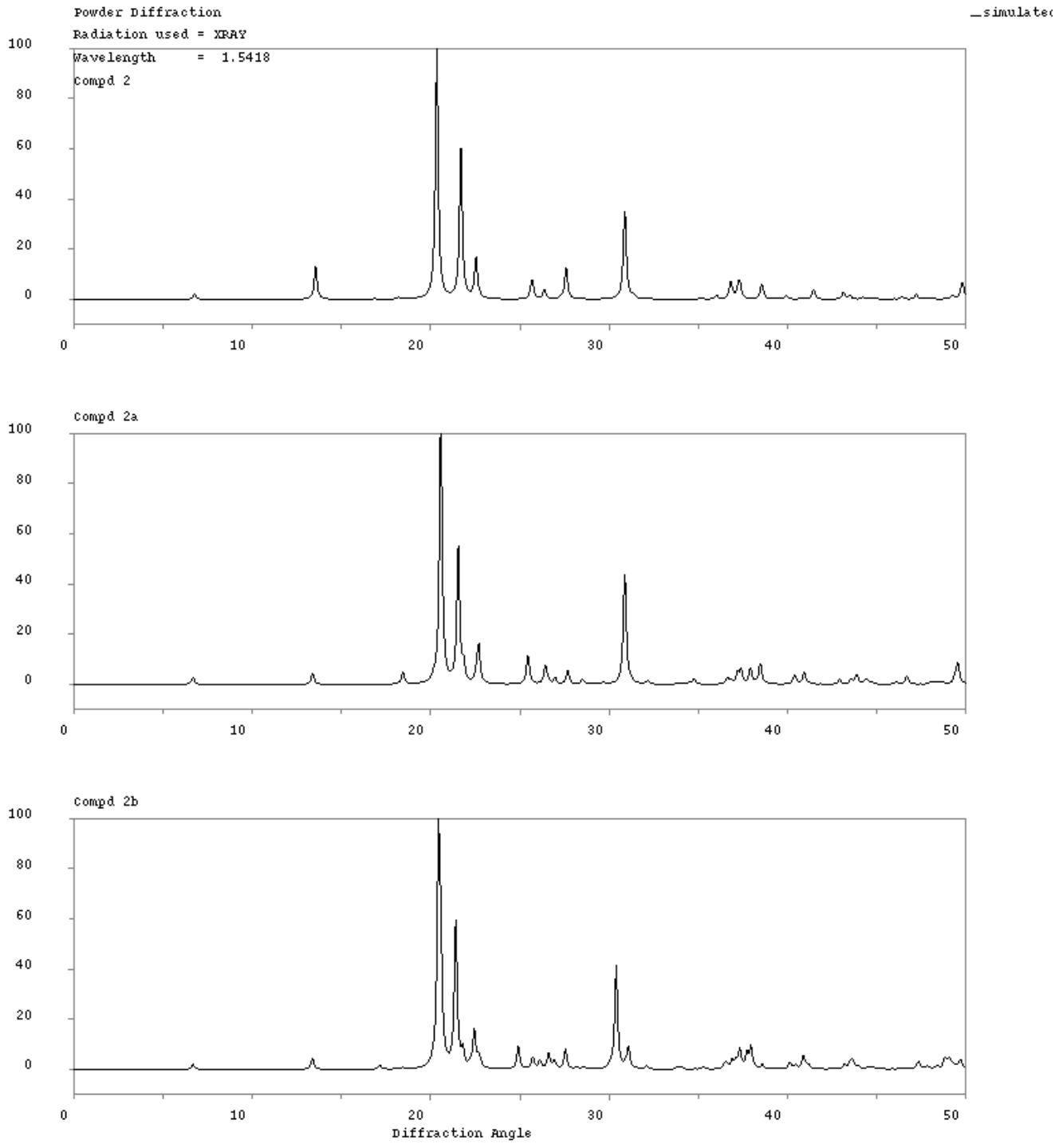



O1
N1
H1
H17
H16
H15
H14
H13
H12
H11
H10
H9
H8
H7
H6
H5
H4
H3
H2
C9
C8
C7
C15
C14
C13
C12
C11
C10
C6
C5
C4
C3
C2
C1
C1 C2 C3 C4 C5 C6 C10 C11 C12 C13 C14 C15 C7 C8 C9 H2 H3 H4 H5 H6 H7 H8 H9 H10 H11 H12 H13 H14 H15 H16 H17 H1 N1 O1
1.0
-1.5
H15
H14
H10
H6
H4
H5
C15
C5
C14
H8
C9
C7
C6
C10
H11
C8
C4
H16
C13
H7
C1
H1
H9
O1
C11
C3
C12
C2
H1
H12
H3
H13
H2

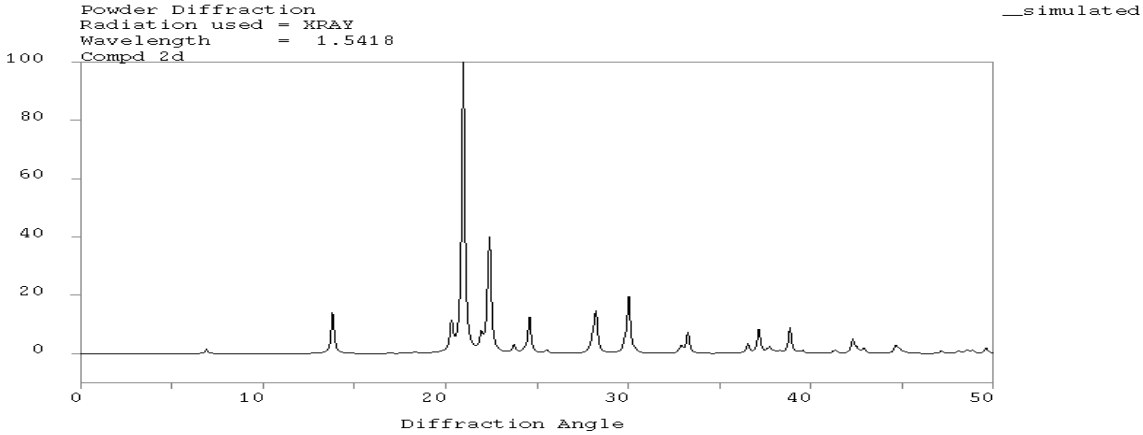

O1
N1
H1
H3
H2
H11
H10
H9
H8
H7
H6
H5
H4
C12
C11
C10
C9
C8
C7
C6
C5
C4
C3
C2
C1
C1 C2 C3 C4 C5 C6 C7 C8 C9 C10 C11 C12 H4 H5 H6 H7 H8 H9 H10 H11 H2 H3 H1 N1 O1
1.0
-1.5
H10
H11
H5
H4
C11
C12
C3
C2
H9
C10
C7
C4
C1
O1
C9
C8
C5
C6
H1
H3
N1
H8
H6
H7
H2

(viii) Simulated powder X-ray diagrams of selected compounds

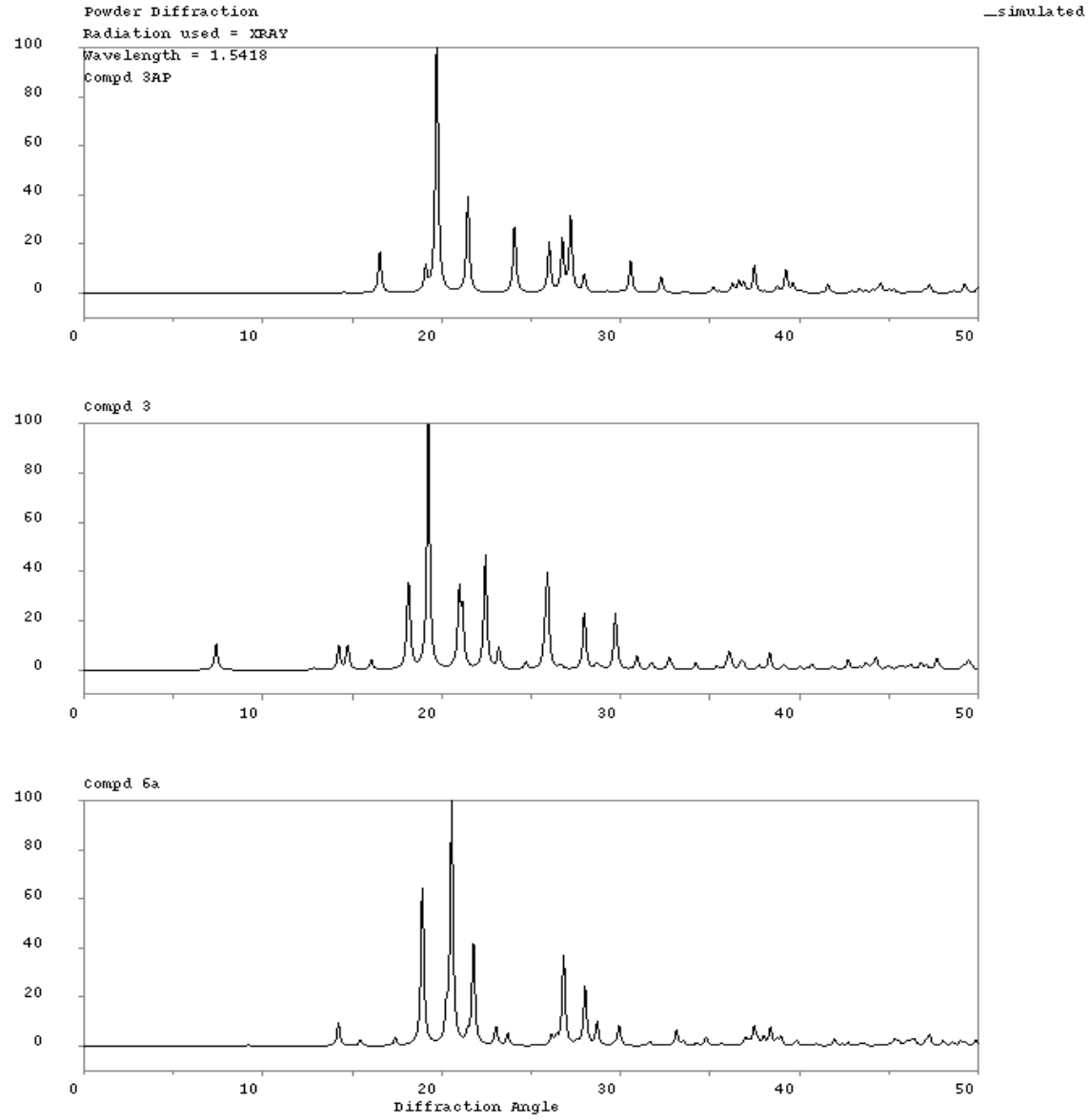
Simulated powder plots of **2, 2a** and **2b** further confirms the near identity of packing:



Simulated powder plot of **2d**: (Compare with compound **2**)



Simulated powder plots of 3AP, **3** and **6a** further confirms the near identity of packing:



(ix) Mulliken charges of compound 1, 1a and 1b at RHF/6-31G* using Spartan program

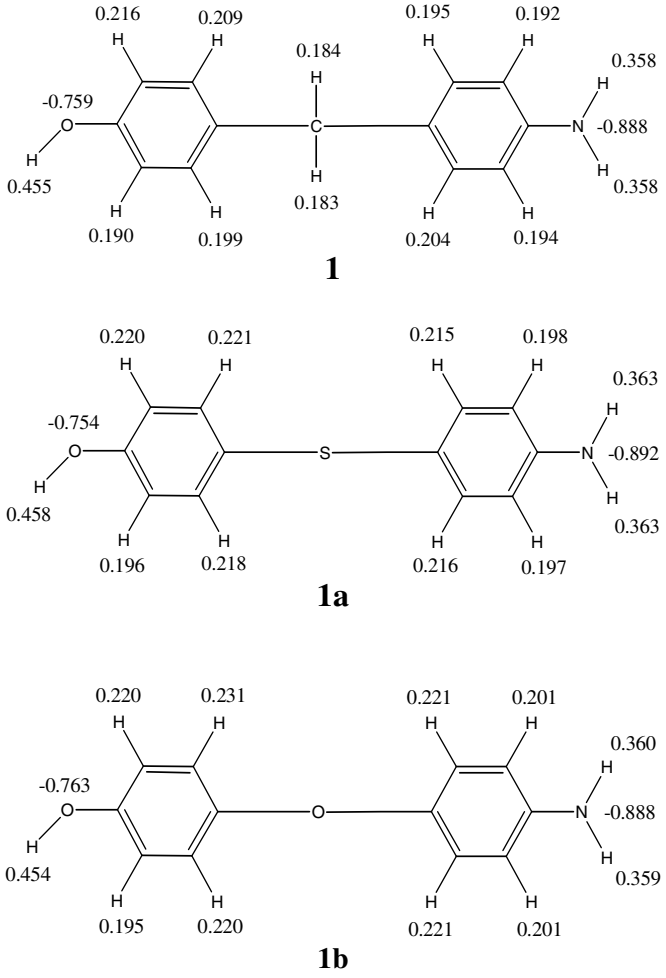


Figure5. Note that in **1b**, the phenyl hydrogens adjacent to linker O-atom are activated.