Supporting information

Switching and tuning organic solid state luminescence via a supramolecular approach

INDEX	

Experimental Details:

Synthesis and Characterization:

Triphenylamine**,** cyanoacetic acid, $POCl_3$, anhydrous dimethyl formamide (99.8%), pyridine, pyrrolidine, piperidine and morpholine were obtained from Aldrich and used as received.

2-cyano-3(4-(diphenylamino)phenyl)acrylic acid (CDPA). A 70 mL acetonitrile solution of 4-diphenylaminobenzaldehyde (1.0 g, 3.66 mmol), cyanoacetic acid (0.34 g, 4.0 mmol), and piperidine (0.62 g, 7.32 mmol) was refluxed for 4 h under nitrogen atmosphere. Solvent removal by rotary evaporator followed by solvent extraction (CH_2Cl_2 and aq HCl (0.1 M)) yielded the product as a dark purple solid, (1.05 g, 85%): Mp 213-214 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.90 (d, 2H), 7.39 (t, 4H), 7.23 (m, 6H), 6.98 (d, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.5, 155.0, 153.2, 145.1, 132.7, 129.6, 126.3, 125.5, 122.8, 118.2, 116.9, 95.5.

General procedure for 1 – 4 preparation: A mixture of CDPA (100 mg, 0.29 mmol) and amine (0.3 mmol) was dissolved in acetonitrile (15 ml) and left to stand at room temperature. Crystals of 1 form after near complete evaporation of solvent and 2 – 4 form in a day. Above 90 % yield was obtained for 1 – 4.

Details of UV-Visible and luminescence studies:

Absorption and luminescence spectra were recorded using Perking Elmer Lambda 1050 and Horiba Jobin Yvon Fluorolog instruments. Solid state luminescence was measured by spreading the powdered samples on a glass plate. To compare the intensity of the solid state luminescence with CDPA in CH_2Cl_2 solution, transparent KBr pellets of CDPA, 1-3 were prepared and the concentration of the compounds in solution as well as in solid matrix were adjusted to keep the optical density (OD) around 0.5. KBr pellets of these samples show similar luminescence λ_{max} as their pure solid samples. However pellet of 4 shows different luminescence λ_{max} and hence we have omitted from the Figure S4. Switching of luminescence was carried out by spreading the powdered CDPA on a glass plate and placing it upside down on top of a 50 ml beaker containing a few drops of amine. To convert back to CDPA, the amine exposed samples were immersed in 0.1 M HCl solution for about 2 h at room temperature.

Details of powder X-ray diffraction (PXRD), single crystals and thermogravimetric studies:

PXRD measurements were recorded using Siemens diffraktometer-D500 at room temperature.

Single crystals were carefully chosen after they were viewed through a polarizing microscope. The crystals were glued to a thin glass fiber using an adhesive (cyano acrylate) and mounted on a diffractometer equipped with an APEX CCD area detector. The data collection was carried out at 150K and no extraordinary methods were employed, except that the crystals were smeared in NIH immersion oil to protect them from ambient laboratory conditions. The intensity data were processed using Bruker's suite of data processing programs (SAINT), and absorption corrections were applied using SADABS.[1] The structure solution of all the complexes was carried out by direct methods, and refinements were performed by full-matrix least-squares on F^2 using the SHELXTL-PLUS[2] suite of programs. All the structures converged to good *R* factors. All the non-hydrogen atoms were refined anisotropically, and the hydrogen atoms were fixed on calculated position using appropriate HFIX options in Shelxtl and were refined isotropically. Intermolecular interactions were computed using the PLATON program.[3]

TGA was carried out using Perkin-Elmer Pyris 1 TG analyzer and the experiments were carried out in an inert atmosphere with a heating rate of 10 ^{o}C/min. It is noted that even after drying 3 for very long time (24h) under vacuum TGA shows the presence of some CH_3CN molecule in the lattice. Despite that the dried sample 3 exhibits different luminescence. To keep the uniformity, the same batch of dried sample 3 was used for luminescence, PXRD and TGA measurements.

References:

(1) Sheldrick, G. M. SADABS, Area Detector Correction. 2002. Madison, WI, Siemens Industrial Automation, Inc.

(2) (a) Sheldrick, G. M. SAINT Area Detector Integration Software. 1998. Madison, WI, Siemens Industrial Automation, Inc. (b) Sheldrick, G. M. SHELX97 programs for crystal Strucutre Analysis. (97-2). 1998. Institüt für Anorganische Chemie der Universität. (c) Sheldrick, G. M. XPREP. (V5.1). 1997. Madison, WI, Bruker Analytical X-Ray Systems.

(3) Speck, A. L. PLATON. *Acta Crystallogr., Sect. A*, 1990, *A46*, C34.

Supporting Figures:

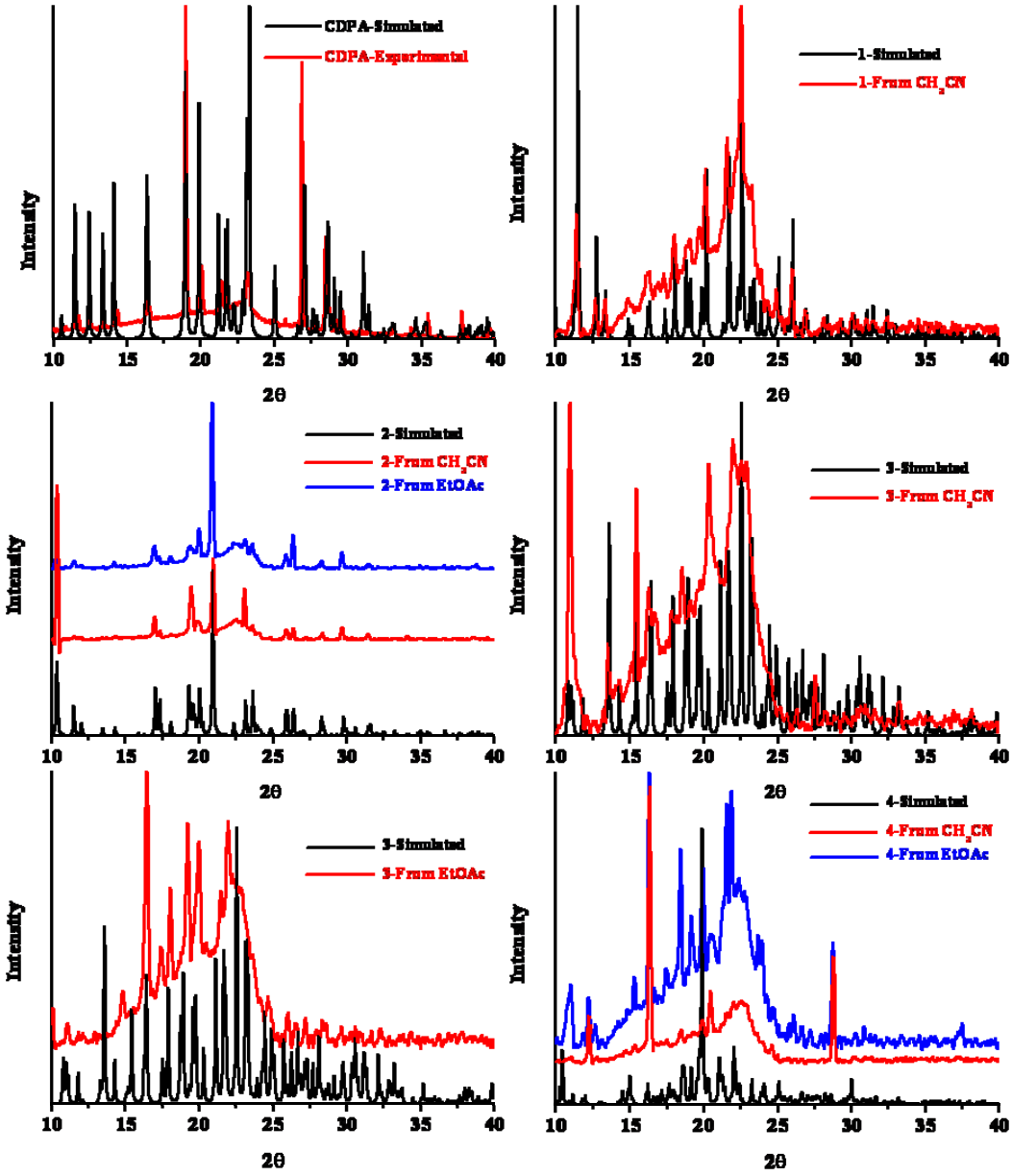


Figure S1: Simulated (from single crystal) and experimental PXRD pattern of CDPA and 1-4.

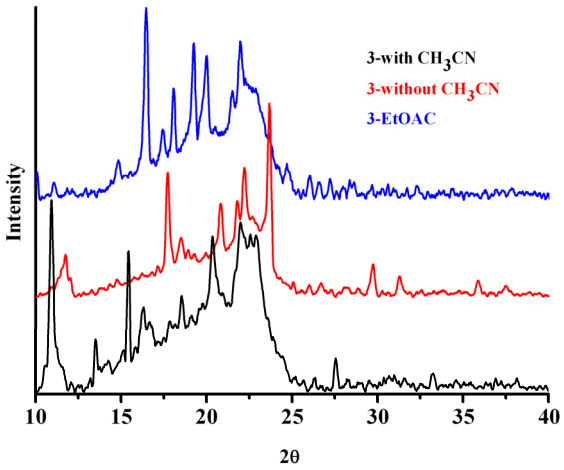


Figure S2: Solvent dependence of the PXRD patterns of **3**

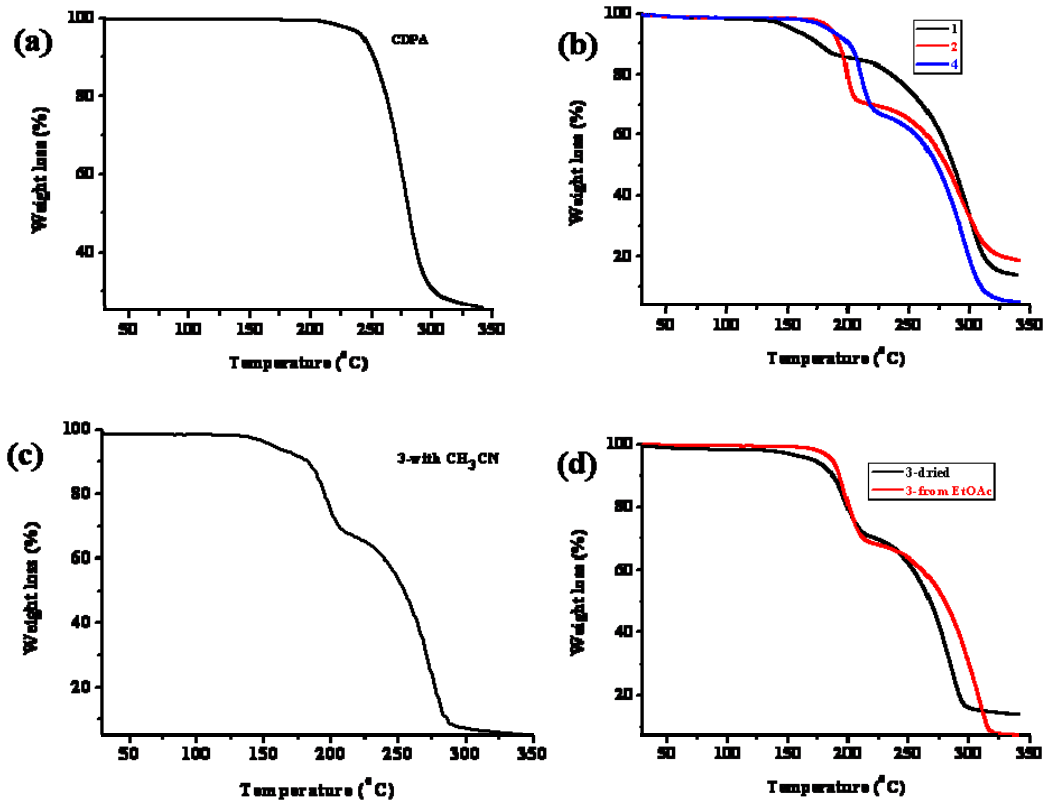


Figure S3: Thermogravimetric analysis of (a) CDPA, (b) 1,2 and 4, (c) 3 with CH_3CN and (d) 3 dried and obtained from EtOAC.

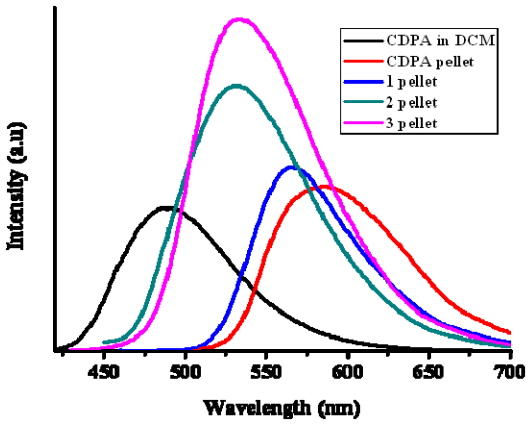


Figure S4: Luminescence spectra of CDPA in DCM and KBr pellets of CDPA and 1-3.

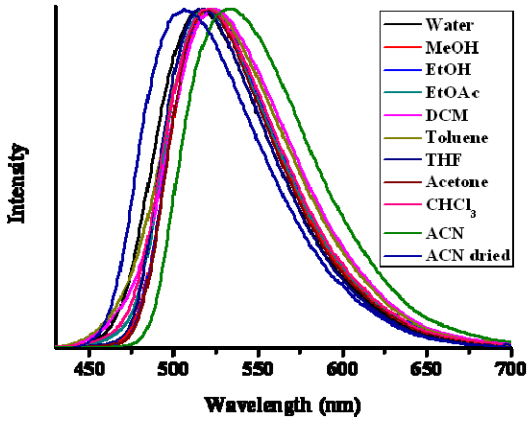


Figure S5: Solvent dependent solid state luminescence spectra of 3.

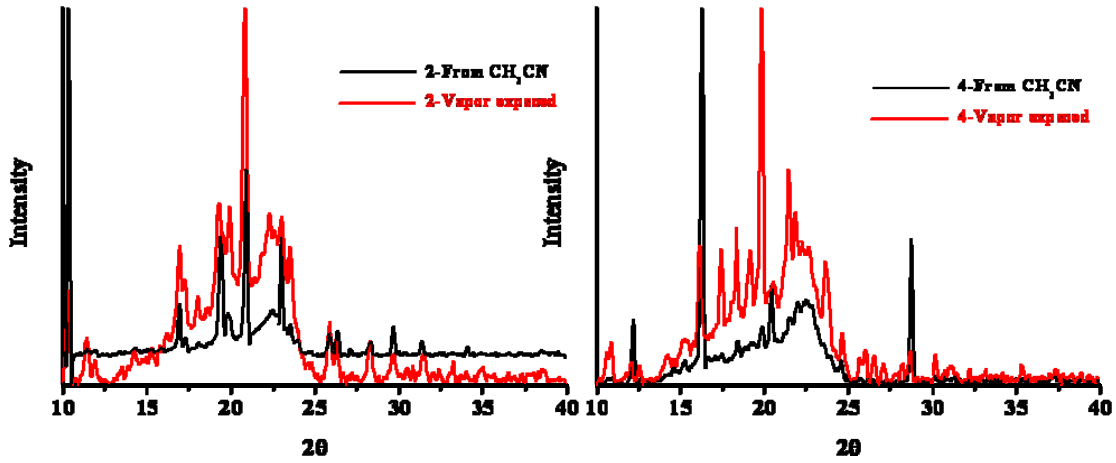


Figure S6: PXRD pattern of 2 and 4 prepared from CH_3CN and after exposure to pyrrolidine (2) and morpholine (4) vapor.

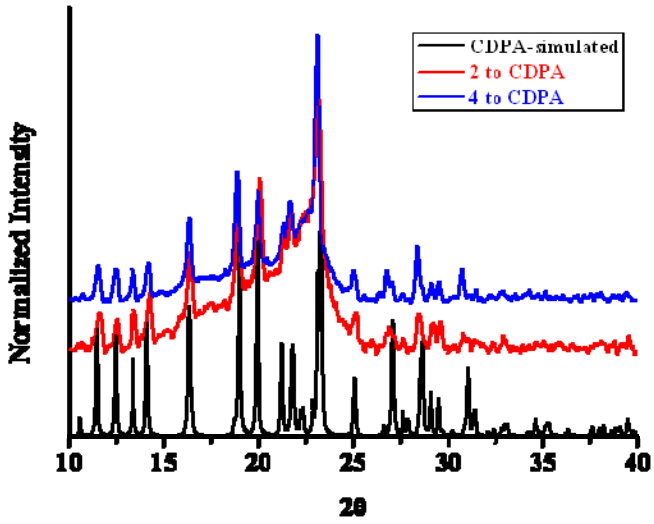


Figure S7: Simulated PXRD pattern of CDPA and amine exposed samples of 2 and 4 after immersion in 0.1 M HCl solution.

H-bond distances and angles in CDPA and 1-4.

CDPA

O(1A)	--H(1A)	..N(3)	1.95	2.7515	158
C(4)	--H(4)	..O(1B)	2.56	3.4870	164
C(10)	--H(10)	..O(1B)	2.95	3.7837	148
C(13)	--H(13)	..O(1A)	2.93	3.6744	137
C(13)	--H(13)	..N(3)	2.67	3.5963	164
C(19)	--H(19)	..O(1A)	2.89	3.5436	127
C(20)	--H(20)	..O(1B)	2.81	3.6227	144
C(20)	--H(20)	..O(1B)	2.57	3.2838	132

1

N(25)	--H(25)	..O(1B)	2.08	3.0139	167
C(15)	--H(15)	..O(1B)	2.64	3.2940	126
C(19)	--H(19)	..O(1B)	2.84	3.6247	140
C(24)	--H(24)	..N(3)	2.65	3.4187	138
C(26)	--H(26)	..N(3)	2.64	3.2496	123

2

C(8)	--H(8)	..O(1A)	3.04	3.6787	126
C(16)	--H(16)	..O(1B)	2.51	3.3925	154
C(27)	--H(27A)	..O(1A)	1.86	2.7744	152
C(27)	--H(27A)	..O(1B)	2.23	3.0502	140
C(27)	--H(27B)	..O(1A)	1.92	2.7837	144
C(27)	--H(27B)	..N(3)	2.79	3.4649	126
C(28)	--H(28A)	..N(3)	2.81	3.5282	130

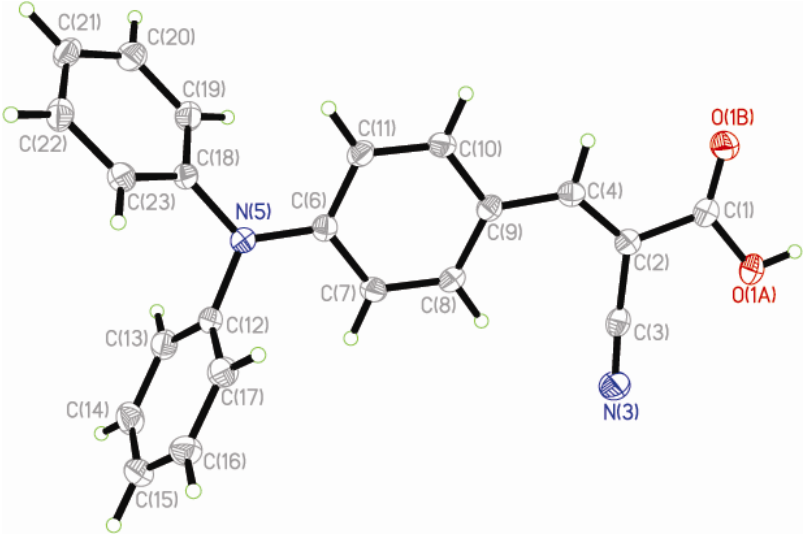
3

O(1A)	--H(1A)	..N(24)	2.15	2.9460	158
N(24)	--H(24)	..O(1A)	2.40	2.9460	120
N(24)	--H(24)	..O(1A)	2.19	2.7611	122
C(10)	--H(10)	..N(33)	2.86	3.6575	143
C(14)	--H(14)	..O(1B)	2.86	3.6135	137
C(15)	--H(15)	..N(33)	2.89	3.7069	145
C(16)	--H(16)	..N(3)	2.68	3.4636	140
C(22)	--H(22)	..N(3)	2.71	3.3404	125
C(26)	--H(26A)	..N(3)	2.58	3.4994	154
C(27)	--H(27B)	..O(1B)	2.64	3.4604	140
C(28)	--H(28A)	..N(30)	2.96	3.6668	129
C(28)	--H(28A)	..N(3)	2.93	3.7563	142
C(28)	--H(28B)	..N(33)	2.73	3.5287	137
C(29)	--H(29A)	..N(30)	2.62	3.5795	163
C(29)	--H(29B)	..N(33)	2.78	3.6449	147
C(32)	--H(32B)	..O(1A)	2.49	3.3195	142
C(32)	--H(32B)	..N(3)	2.82	3.6110	138

4

N1A	--H24A	..O14A	1.68	2.6955	169
N1A	--H24B	..O13A	2.46	3.0998	123
N1A	--H24B	..O13B	1.80	2.7681	169
N1B	--H24C	..O11A	1.78	2.6796	169
N1B	--H24D	..O12B	1.82	2.8684	173
N1C	--H24E	..O13A	1.84	2.6893	164
N1C	--H24F	..O14A	2.34	3.0340	123
N1C	--H24F	..O14B	1.76	2.7850	167
N1D	--H24G	..O11A	2.46	3.0676	122
N1D	--H24G	..O11B	1.84	2.7807	173
N1D	--H24H	..O12A	1.79	2.7221	159
C3B	--H3BB	..O12B	2.83	3.5354	129
C5B	--H5BB	..N32	2.93	3.5307	120
C5B	--H5BA	..O12B	2.97	3.6606	128
C6B	--H6BB	..N31	2.86	3.7502	149
C6B	--H6BA	..N31	2.66	3.4462	137
C3D	--H3DA	..O11B	2.82	3.5038	127
C5D	--H5DB	..O11B	2.98	3.6469	126
C5D	--H5DB	..O12B	2.99	3.6299	123
C5D	--H5DA	..N31	2.87	3.4769	121
C6D	--H6DB	..N32	2.65	3.4494	138
C6D	--H6DA	..O11A	2.88	3.7247	143
C6D	--H6DA	..O12A	2.79	3.6163	141
C6D	--H6DA	..N1B	2.98	3.8306	145
C2A	--H2AB	..N33	2.95	3.6198	126
C2A	--H2AB	..N34	2.76	3.6004	143
C2A	--H2AA	..N34	2.84	3.5776	131
C5A	--H5AA	..O13B	2.83	3.5065	127
C2C	--H2CB	..O13B	2.27	3.2481	170
C2C	--H2CA	..N33	2.68	3.4825	139
C3C	--H3CB	..N34	2.79	3.4678	126
C3C	--H3CA	..O14B	2.96	3.6311	126
C5C	--H5CB	..O14B	2.72	3.4252	129
C73	--H73	..O4A	2.89	3.4775	121
C74	--H74	..O27D	2.65	3.3248	128
C83	--H83	..O4A	2.79	3.4374	126
C112	--H112	..O4B	2.62	3.2572	125
C143	--H143	..N31	2.78	3.5224	136
C143	--H143	..O4D	2.80	3.5833	140
C144	--H144	..O14B	2.87	3.7757	159
C163	--H163	..O13B	2.92	3.5641	126
C164	--H164	..O14B	2.68	3.4092	134
C172	--H172	..O4B	2.54	3.4620	163
C191	--H191	..O12B	2.78	3.5206	135
C194	--H194	..O27D	2.48	3.4112	167
C201	--H201	..O11B	2.58	3.3842	143
C202	--H202	..O12B	2.68	3.5452	152
C221	--H221	..N33	2.84	3.6695	146

Crystal data and structure refinement for **CDPA**.



Identification code	CDPA (CCDC No: 737400)	
Empirical formula	C22 H16 N2 O2	
Formula weight	340.37	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 13.568(1) Å	α= 90°.
	b = 9.459(1) Å	β= 104.167(2)°.
	c = 13.642(1) Å	γ = 90°.
Volume	1697.6(3) $Å^3$	
Z	4	
Density (calculated)	1.332 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	712	
Crystal size	0.23 x 0.15 x 0.12 mm^3	
Theta range for data collection	1.90 to 25.00°.	
Index ranges	-15<=h<=16, -10<=k<=11, -16<=l<=13	
Reflections collected	9595	
Independent reflections	2985 [R(int) = 0.0248]	
Completeness to theta = 25.00°	99.8 %	
Absorption correction	Multi scan	

Max. and min. transmission	0.9897 and 0.9804
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2985 / 65 / 235
Goodness-of-fit on F^2	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0330, wR2 = 0.0893
R indices (all data)	R1 = 0.0369, wR2 = 0.0923
Largest diff. peak and hole	0.171 and -0.170 e.Å^{-3}

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

	x	y	z	U(eq)
O(1A)	12198(1)	8257(1)	6976(1)	29(1)
O(1B)	11234(1)	9493(1)	5684(1)	29(1)
N(3)	11327(1)	5293(1)	7435(1)	32(1)
N(5)	6393(1)	3546(1)	4614(1)	23(1)
C(1)	11368(1)	8479(1)	6239(1)	22(1)
C(2)	10628(1)	7309(1)	6189(1)	21(1)
C(3)	10993(1)	6164(1)	6867(1)	23(1)
C(4)	9698(1)	7367(1)	5535(1)	21(1)
C(6)	7200(1)	4481(1)	4827(1)	19(1)
C(7)	8115(1)	4087(1)	5505(1)	22(1)
C(8)	8928(1)	4992(1)	5739(1)	21(1)
C(9)	8880(1)	6358(1)	5321(1)	20(1)
C(10)	7963(1)	6738(1)	4636(1)	22(1)
C(11)	7146(1)	5833(1)	4391(1)	22(1)
C(12)	6345(1)	2373(1)	5276(1)	22(1)
C(13)	6355(1)	1008(1)	4919(1)	26(1)
C(14)	6263(1)	-121(1)	5540(1)	30(1)
C(15)	6144(1)	113(1)	6502(1)	29(1)
C(16)	6130(1)	1478(1)	6853(1)	28(1)
C(17)	6234(1)	2611(1)	6244(1)	25(1)
C(18)	5527(1)	3724(1)	3781(1)	21(1)
C(19)	5638(1)	3928(1)	2804(1)	26(1)
C(20)	4779(1)	4073(1)	2014(1)	29(1)

C(21)	3816(1)	3989(1)	2189(1)	27(1)
C(22)	3711(1)	3750(1)	3158(1)	26(1)
C(23)	4564(1)	3632(1)	3954(1)	23(1)

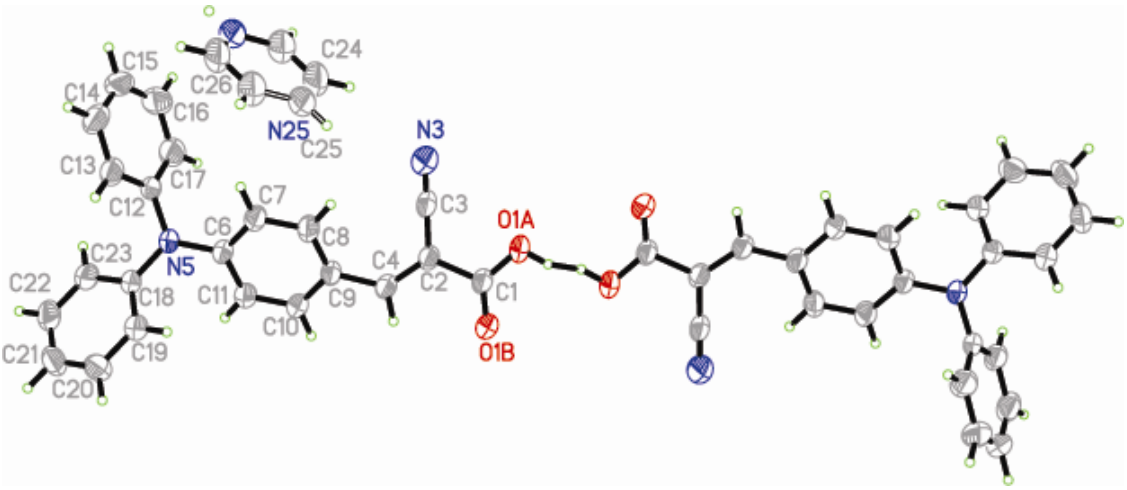
Table 3. Bond lengths [Å] and angles [°] for CDPA.

O(1A)-C(1)	1.3298(14)	C(6)-N(5)-C(18)	123.12(10)
O(1B)-C(1)	1.2069(15)	C(6)-N(5)-C(12)	121.04(9)
N(3)-C(3)	1.1459(16)	C(18)-N(5)-C(12)	115.69(9)
N(5)-C(6)	1.3814(15)	O(1B)-C(1)-O(1A)	124.67(11)
N(5)-C(18)	1.4304(14)	O(1B)-C(1)-C(2)	124.58(10)
N(5)-C(12)	1.4420(15)	O(1A)-C(1)-C(2)	110.75(10)
C(1)-C(2)	1.4848(16)	C(4)-C(2)-C(3)	125.08(11)
C(2)-C(4)	1.3567(16)	C(4)-C(2)-C(1)	120.60(10)
C(2)-C(3)	1.4313(17)	C(3)-C(2)-C(1)	114.32(10)
C(4)-C(9)	1.4393(16)	N(3)-C(3)-C(2)	176.38(13)
C(6)-C(11)	1.4048(17)	C(2)-C(4)-C(9)	130.80(11)
C(6)-C(7)	1.4060(16)	N(5)-C(6)-C(11)	122.51(10)
C(7)-C(8)	1.3704(16)	N(5)-C(6)-C(7)	119.72(10)
C(8)-C(9)	1.4068(16)	C(11)-C(6)-C(7)	117.77(10)
C(9)-C(10)	1.4075(16)	C(8)-C(7)-C(6)	121.24(11)
C(10)-C(11)	1.3766(17)	C(7)-C(8)-C(9)	121.64(10)
C(12)-C(13)	1.3814(17)	C(8)-C(9)-C(10)	116.73(10)
C(12)-C(17)	1.3844(17)	C(8)-C(9)-C(4)	124.77(10)
C(13)-C(14)	1.3880(18)	C(10)-C(9)-C(4)	118.50(10)
C(14)-C(15)	1.3783(19)	C(11)-C(10)-C(9)	122.05(11)
C(15)-C(16)	1.3794(19)	C(10)-C(11)-C(6)	120.56(10)
C(16)-C(17)	1.3839(18)	C(13)-C(12)-C(17)	120.10(11)
C(18)-C(23)	1.3856(17)	C(13)-C(12)-N(5)	119.52(11)
C(18)-C(19)	1.3912(17)	C(17)-C(12)-N(5)	120.28(10)
C(19)-C(20)	1.3868(17)	C(12)-C(13)-C(14)	119.58(11)
C(20)-C(21)	1.3869(18)	C(15)-C(14)-C(13)	120.47(12)
C(21)-C(22)	1.3828(19)	C(14)-C(15)-C(16)	119.69(11)

C(22)-C(23)	1.3833(17)	C(15)-C(16)-C(17)	120.33(12)
		C(16)-C(17)-C(12)	119.82(11)
		C(23)-C(18)-C(19)	119.92(11)
		C(23)-C(18)-N(5)	118.93(10)
		C(19)-C(18)-N(5)	121.10(10)
		C(20)-C(19)-C(18)	119.41(12)
		C(19)-C(20)-C(21)	120.58(12)
		C(22)-C(21)-C(20)	119.68(11)
		C(21)-C(22)-C(23)	120.11(12)
		C(22)-C(23)-C(18)	120.27(11)

Symmetry transformations used to generate equivalent atoms:

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



Identification code	1 (CCDC No: 737402)
Empirical formula	$2(C_{22}H_{16}N_2O_2)$, C_5H_5N
Formula weight	759.84
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic

Space group	P 21/c	
Unit cell dimensions	a = 7.877(1) Å	α= 90°.
	b = 9.424(1) Å	β= 93.023(3)°.
	c = 26.835(3) Å	γ = 90°.
Volume	1989.3(4) $Å^3$	
Z	2	
Density (calculated)	1.269 Mg/m^3	
Absorption coefficient	0.082 mm^{-1}	
F(000)	796	
Crystal size	0.19 x 0.13 x 0.09 mm^3	
Theta range for data collection	2.29 to 25.02°.	
Index ranges	-9<=h<=9, -11<=k<=8, -31<=l<=31	
Reflections collected	11236	
Independent reflections	3497 [R(int) = 0.0307]	
Completeness to theta = 25.02°	99.3 %	
Absorption correction	Multi scan	
Max. and min. transmission	0.9927 and 0.9846	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3497 / 0 / 262	
Goodness-of-fit on F^2	1.065	
Final R indices [I>2sigma(I)]	R1 = 0.0553, wR2 = 0.1404	
R indices (all data)	R1 = 0.0706, wR2 = 0.1533	
Largest diff. peak and hole	0.463 and -0.223 $e.Å^{-3}$	

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

	x	y	z	U(eq)
O(1A)	-3572(2)	9465(2)	4912(1)	54(1)
O(1B)	-3300(2)	11056(2)	4309(1)	71(1)
N(3)	-210(3)	7261(3)	5024(1)	69(1)
N(5)	5793(2)	7812(2)	3229(1)	47(1)
C(1)	-2804(3)	10037(2)	4555(1)	44(1)
C(2)	-1146(2)	9330(2)	4450(1)	38(1)
C(3)	-628(3)	8178(2)	4769(1)	45(1)

C(4)	-248(3)	9768(2)	4065(1)	42(1)
C(6)	4301(3)	8285(2)	3438(1)	40(1)
C(7)	3911(3)	7868(2)	3915(1)	46(1)
C(8)	2459(3)	8333(2)	4125(1)	46(1)
C(9)	1324(3)	9246(2)	3873(1)	40(1)
C(10)	1733(3)	9663(2)	3394(1)	47(1)
C(11)	3164(3)	9184(2)	3177(1)	49(1)
C(12)	6528(3)	6497(2)	3391(1)	39(1)
C(13)	8168(3)	6440(2)	3594(1)	47(1)
C(14)	8839(3)	5174(3)	3769(1)	59(1)
C(15)	7891(4)	3961(3)	3741(1)	68(1)
C(16)	6268(4)	4006(3)	3535(1)	71(1)
C(17)	5586(3)	5261(3)	3356(1)	57(1)
C(18)	6724(3)	8711(2)	2917(1)	42(1)
C(19)	6819(3)	10153(2)	2998(1)	46(1)
C(20)	7746(3)	11011(3)	2693(1)	54(1)
C(21)	8616(3)	10432(3)	2315(1)	61(1)
C(22)	8552(3)	8999(3)	2237(1)	63(1)
C(23)	7603(3)	8130(3)	2530(1)	54(1)
N(25)	4313(4)	6226(4)	5195(1)	98(1)
C(24)	3293(5)	5129(5)	4989(2)	112(2)
C(25)	4313(4)	6226(4)	5195(1)	98(1)
C(26)	6004(4)	6051(4)	5199(1)	102(1)

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

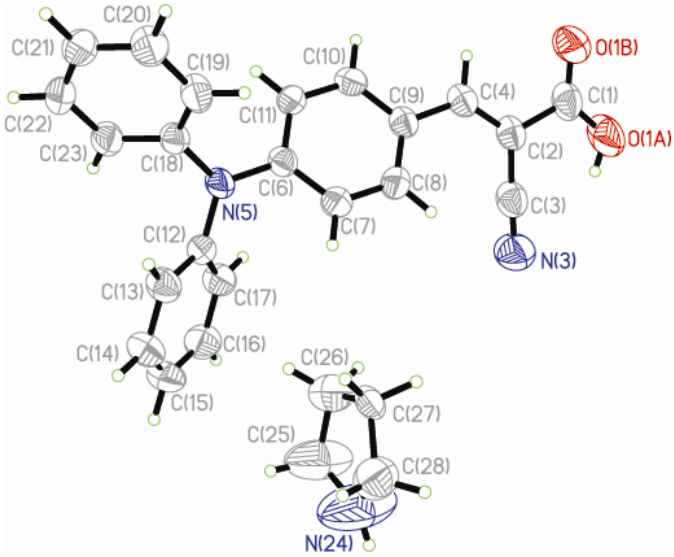
O(1A)-C(1)	1.278(3)	C(6)-N(5)-C(18)	121.07(17)
O(1B)-C(1)	1.218(3)	C(6)-N(5)-C(12)	119.28(17)
N(3)-C(3)	1.139(3)	C(18)-N(5)-C(12)	118.95(16)
N(5)-C(6)	1.402(3)	O(1B)-C(1)-O(1A)	126.02(19)
N(5)-C(18)	1.421(3)	O(1B)-C(1)-C(2)	120.21(19)
N(5)-C(12)	1.427(3)	O(1A)-C(1)-C(2)	113.77(19)
C(1)-C(2)	1.506(3)	C(4)-C(2)-C(3)	123.17(18)
C(2)-C(4)	1.349(3)	C(4)-C(2)-C(1)	120.25(19)

C(2)-C(3)	1.429(3)	C(3)-C(2)-C(1)	116.55(18)
C(4)-C(9)	1.452(3)	N(3)-C(3)-C(2)	179.8(3)
C(6)-C(7)	1.388(3)	C(2)-C(4)-C(9)	131.4(2)
C(6)-C(11)	1.395(3)	C(7)-C(6)-C(11)	117.91(19)
C(7)-C(8)	1.374(3)	C(7)-C(6)-N(5)	120.68(19)
C(8)-C(9)	1.391(3)	C(11)-C(6)-N(5)	121.41(19)
C(9)-C(10)	1.396(3)	C(8)-C(7)-C(6)	121.2(2)
C(10)-C(11)	1.372(3)	C(7)-C(8)-C(9)	121.6(2)
C(12)-C(13)	1.377(3)	C(8)-C(9)-C(10)	116.64(18)
C(12)-C(17)	1.381(3)	C(8)-C(9)-C(4)	125.07(19)
C(13)-C(14)	1.377(3)	C(10)-C(9)-C(4)	118.28(19)
C(14)-C(15)	1.365(4)	C(11)-C(10)-C(9)	122.2(2)
C(15)-C(16)	1.367(4)	C(10)-C(11)-C(6)	120.4(2)
C(16)-C(17)	1.376(4)	C(13)-C(12)-C(17)	118.9(2)
C(18)-C(19)	1.377(3)	C(13)-C(12)-N(5)	120.8(2)
C(18)-C(23)	1.390(3)	C(17)-C(12)-N(5)	120.2(2)
C(19)-C(20)	1.386(3)	C(12)-C(13)-C(14)	120.3(2)
C(20)-C(21)	1.367(4)	C(15)-C(14)-C(13)	120.5(2)
C(21)-C(22)	1.366(4)	C(14)-C(15)-C(16)	119.6(2)
C(22)-C(23)	1.383(3)	C(15)-C(16)-C(17)	120.5(3)
N(25)-C(26)	1.342(4)	C(16)-C(17)-C(12)	120.2(2)
N(25)-C(24)	1.405(6)	C(19)-C(18)-C(23)	118.9(2)
C(24)-C(26)#1	1.351(6)	C(19)-C(18)-N(5)	121.33(19)
C(26)-C(24)#1	1.351(6)	C(23)-C(18)-N(5)	119.8(2)
		C(18)-C(19)-C(20)	120.5(2)
		C(21)-C(20)-C(19)	120.4(2)
		C(22)-C(21)-C(20)	119.5(2)
		C(21)-C(22)-C(23)	120.9(2)
		C(22)-C(23)-C(18)	119.8(2)
		C(26)-N(25)-C(24)	117.3(3)
		C(26)#1-C(24)-N(25)	121.0(3)
		N(25)-C(26)-C(24)#1	121.7(4)

Symmetry transformations used to generate equivalent atoms:

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

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



Identification code	2 (CCDC No: 737399)	
Empirical formula	$C_{22}H_{16}N_2O_2$, C_4H_9N	
Formula weight	411.49	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 9.461(1) Å	α= 90°.
	b = 25.424(2) Å	β= 125.704(5)°.
	c = 11.161(1) Å	γ = 90°.
Volume	2180.0(4) $Å^3$	
Z	4	
Density (calculated)	1.254 Mg/m^3	
Absorption coefficient	0.080 mm^{-1}	
F(000)	872	
Crystal size	0.22 x 0.14 x 0.11 mm^3	
Theta range for data collection	1.60 to 25.03°.	
Index ranges	-11<=h<=11, -30<=k<=22, -13<=l<=12	
Reflections collected	12692	

Independent reflections	3855 [R(int) = 0.0402]
Completeness to theta = 25.03°	99.8 %
Absorption correction	Multi scan
Max. and min. transmission	0.9912 and 0.9825
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3855 / 0 / 280
Goodness-of-fit on F^2	1.049
Final R indices [I>2sigma(I)]	R1 = 0.0611, wR2 = 0.1660
R indices (all data)	R1 = 0.0904, wR2 = 0.1832
Largest diff. peak and hole	0.329 and -0.372 e.Å^{-3}

Table 2. 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(1A)	1814(3)	4695(1)	10359(3)	96(1)
O(1B)	3283(3)	4088(1)	12036(3)	85(1)
N(3)	2889(4)	4804(1)	8023(3)	85(1)
N(5)	9220(3)	3112(1)	8965(2)	47(1)
C(1)	2924(4)	4334(1)	10952(3)	61(1)
C(2)	3868(3)	4215(1)	10265(3)	49(1)
C(3)	3368(4)	4538(1)	9025(3)	58(1)
C(4)	5023(3)	3822(1)	10757(3)	47(1)
C(6)	8217(3)	3295(1)	9442(2)	40(1)
C(7)	7603(3)	3807(1)	9167(3)	48(1)
C(8)	6560(3)	3982(1)	9565(3)	50(1)
C(9)	6111(3)	3656(1)	10297(3)	42(1)
C(10)	6808(3)	3149(1)	10633(3)	45(1)
C(11)	7816(3)	2966(1)	10198(3)	43(1)
C(12)	8882(3)	3294(1)	7619(3)	41(1)
C(13)	10231(3)	3408(1)	7512(3)	51(1)
C(14)	9878(4)	3578(1)	6197(4)	65(1)
C(15)	8204(4)	3642(1)	4979(3)	67(1)
C(16)	6858(4)	3536(1)	5069(3)	60(1)
C(17)	7187(3)	3355(1)	6377(3)	50(1)

C(18)	10670(3)	2776(1)	9890(3)	42(1)
C(19)	11780(3)	2867(1)	11384(3)	51(1)
C(20)	13187(3)	2540(1)	12272(3)	59(1)
C(21)	13517(4)	2131(1)	11671(3)	59(1)
C(22)	12452(4)	2048(1)	10183(3)	58(1)
C(23)	11021(3)	2360(1)	9292(3)	51(1)
N(24)	7907(9)	5553(2)	5424(4)	192(3)
C(25)	7468(8)	5027(2)	5292(5)	134(2)
C(26)	8099(5)	4830(1)	6758(4)	85(1)
C(27)	9283(3)	5238(1)	7821(3)	46(1)
C(28)	9408(5)	5659(1)	6982(4)	78(1)

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

O(1A)-C(1)	1.253(4)	C(1)-O(1A)-H(1A)	109.5
O(1A)-H(1A)	0.8201	C(6)-N(5)-C(12)	120.45(19)
O(1B)-C(1)	1.221(4)	C(6)-N(5)-C(18)	120.01(19)
N(3)-C(3)	1.150(4)	C(12)-N(5)-C(18)	119.34(19)
N(5)-C(6)	1.411(3)	O(1B)-C(1)-O(1A)	124.1(3)
N(5)-C(12)	1.419(3)	O(1B)-C(1)-C(2)	119.8(3)
N(5)-C(18)	1.422(3)	O(1A)-C(1)-C(2)	116.1(3)
C(1)-C(2)	1.509(4)	C(4)-C(2)-C(3)	123.7(2)
C(2)-C(4)	1.340(4)	C(4)-C(2)-C(1)	121.6(3)
C(2)-C(3)	1.431(4)	C(3)-C(2)-C(1)	114.6(2)
C(4)-C(9)	1.455(3)	N(3)-C(3)-C(2)	176.4(3)
C(4)-H(4)	0.9500	C(2)-C(4)-C(9)	130.3(3)
C(6)-C(7)	1.385(4)	C(2)-C(4)-H(4)	114.8
C(6)-C(11)	1.390(3)	C(9)-C(4)-H(4)	114.8
C(7)-C(8)	1.372(3)	C(7)-C(6)-C(11)	118.2(2)
C(7)-H(7)	0.9500	C(7)-C(6)-N(5)	120.9(2)
C(8)-C(9)	1.393(3)	C(11)-C(6)-N(5)	120.9(2)
C(8)-H(8)	0.9500	C(8)-C(7)-C(6)	121.4(2)
C(9)-C(10)	1.396(4)	C(8)-C(7)-H(7)	119.3
C(10)-C(11)	1.379(3)	C(6)-C(7)-H(7)	119.3
C(10)-H(10)	0.9500	C(7)-C(8)-C(9)	121.3(2)

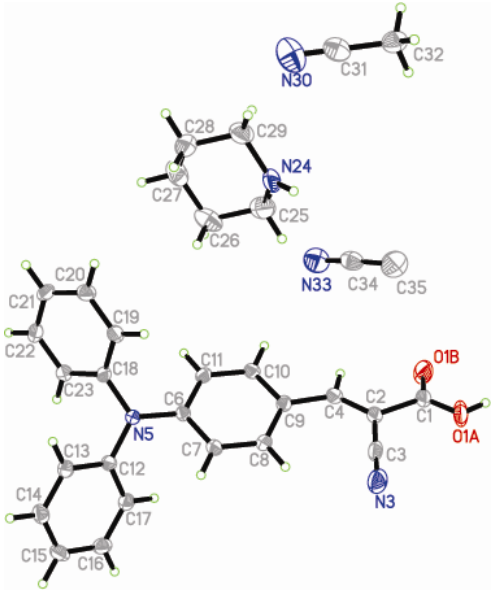
C(11)-H(11)	0.9500	C(7)-C(8)-H(8)	119.3
C(12)-C(13)	1.380(3)	C(9)-C(8)-H(8)	119.3
C(12)-C(17)	1.386(3)	C(8)-C(9)-C(10)	116.7(2)
C(13)-C(14)	1.371(4)	C(8)-C(9)-C(4)	123.7(2)
C(13)-H(13)	0.9500	C(10)-C(9)-C(4)	119.5(2)
C(14)-C(15)	1.367(4)	C(11)-C(10)-C(9)	122.1(2)
C(14)-H(14)	0.9500	C(11)-C(10)-H(10)	118.9
C(15)-C(16)	1.361(4)	C(9)-C(10)-H(10)	118.9
C(15)-H(15)	0.9500	C(10)-C(11)-C(6)	120.1(2)
C(16)-C(17)	1.382(4)	C(10)-C(11)-H(11)	120.0
C(16)-H(16)	0.9500	C(6)-C(11)-H(11)	120.0
C(17)-H(17)	0.9500	C(13)-C(12)-C(17)	118.6(2)
C(18)-C(19)	1.376(3)	C(13)-C(12)-N(5)	120.8(2)
C(18)-C(23)	1.390(3)	C(17)-C(12)-N(5)	120.6(2)
C(19)-C(20)	1.379(4)	C(14)-C(13)-C(12)	119.9(3)
C(19)-H(19)	0.9500	C(14)-C(13)-H(13)	120.0
C(20)-C(21)	1.369(4)	C(12)-C(13)-H(13)	120.0
C(20)-H(20)	0.9500	C(15)-C(14)-C(13)	121.2(3)
C(21)-C(22)	1.366(4)	C(15)-C(14)-H(14)	119.4
C(21)-H(21)	0.9500	C(13)-C(14)-H(14)	119.4
C(22)-C(23)	1.371(4)	C(16)-C(15)-C(14)	119.6(3)
C(22)-H(22)	0.9500	C(16)-C(15)-H(15)	120.2
C(23)-H(23)	0.9500	C(14)-C(15)-H(15)	120.2
N(24)-C(25)	1.384(6)	C(15)-C(16)-C(17)	120.0(3)
N(24)-C(28)	1.494(5)	C(15)-C(16)-H(16)	120.0
N(24)-H(24)	0.8601	C(17)-C(16)-H(16)	120.0
C(25)-C(26)	1.464(5)	C(16)-C(17)-C(12)	120.6(3)
C(25)-H(25A)	0.9900	C(16)-C(17)-H(17)	119.7
C(25)-H(25B)	0.9900	C(12)-C(17)-H(17)	119.7
C(26)-C(27)	1.479(4)	C(19)-C(18)-C(23)	118.8(2)
C(26)-H(26A)	0.9900	C(19)-C(18)-N(5)	120.7(2)
C(26)-H(26B)	0.9900	C(23)-C(18)-N(5)	120.5(2)
C(27)-C(28)	1.471(4)	C(18)-C(19)-C(20)	120.3(2)
C(27)-H(27A)	0.9900	C(18)-C(19)-H(19)	119.8
C(27)-H(27B)	0.9900	C(20)-C(19)-H(19)	119.8

C(28)-H(28A)	0.9900	C(21)-C(20)-C(19)	120.3(2)
C(28)-H(28B)	0.9900	C(21)-C(20)-H(20)	119.8
		C(19)-C(20)-H(20)	119.8
		C(22)-C(21)-C(20)	119.8(2)
		C(22)-C(21)-H(21)	120.1
		C(20)-C(21)-H(21)	120.1
		C(21)-C(22)-C(23)	120.5(3)
		C(21)-C(22)-H(22)	119.8
		C(23)-C(22)-H(22)	119.8
		C(22)-C(23)-C(18)	120.3(2)
		C(22)-C(23)-H(23)	119.9
		C(18)-C(23)-H(23)	119.9
		C(25)-N(24)-C(28)	108.7(4)
		C(25)-N(24)-H(24)	125.5
		C(28)-N(24)-H(24)	125.8
		N(24)-C(25)-C(26)	108.1(4)
		N(24)-C(25)-H(25A)	110.1
		C(26)-C(25)-H(25A)	110.1
		N(24)-C(25)-H(25B)	110.1
		C(26)-C(25)-H(25B)	110.1
		H(25A)-C(25)-H(25B)	108.4
		C(25)-C(26)-C(27)	106.3(3)
		C(25)-C(26)-H(26A)	110.5
		C(27)-C(26)-H(26A)	110.5
		C(25)-C(26)-H(26B)	110.5
		C(27)-C(26)-H(26B)	110.5
		H(26A)-C(26)-H(26B)	108.7
		C(28)-C(27)-C(26)	107.7(2)
		C(28)-C(27)-H(27A)	110.2
		C(26)-C(27)-H(27A)	110.2
		C(28)-C(27)-H(27B)	110.2
		C(26)-C(27)-H(27B)	110.2
		H(27A)-C(27)-H(27B)	108.5
		C(27)-C(28)-N(24)	103.6(3)
		C(27)-C(28)-H(28A)	111.0

	N(24)-C(28)-H(28A)	111.0
	C(27)-C(28)-H(28B)	111.0
	N(24)-C(28)-H(28B)	111.0
	H(28A)-C(28)-H(28B)	109.0

Symmetry transformations used to generate equivalent atoms:

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



Identification code	3 (CCDC No: 737403)	
Empirical formula	$2(C_{22}H_{16}N_2O_2)$, $2(C_5H_{11}N)$, $3(C_2H_3N)$	
Formula weight	971.17	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbcn	
Unit cell dimensions	a = 32.166(2) Å	α= 90°.
	b = 9.900(7) Å	β= 90°.
	c = 16.407(1) Å	γ = 90°.

Volume	5225.0(4) $Å^3$
Z	4
Density (calculated)	1.235 Mg/m^3
Absorption coefficient	0.079 mm^{-1}
F(000)	2060
Crystal size	0.23 x 0.16 x 0.11 mm^3
Theta range for data collection	1.27 to 25.18°.
Index ranges	-38<=h<=38, -11<=k<=11, -19<=l<=19
Reflections collected	53187
Independent reflections	4686 [R(int) = 0.0287]
Completeness to theta = 25.18°	99.9 %
Absorption correction	Multi scan
Max. and min. transmission	0.9913 and 0.9820
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4686 / 0 / 340
Goodness-of-fit on F^2	1.036
Final R indices [I>2sigma(I)]	R1 = 0.0490, wR2 = 0.1397
R indices (all data)	R1 = 0.0563, wR2 = 0.1513
Largest diff. peak and hole	0.501 and -0.525 $e.Å^{-3}$

Table 2. 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(1A)	5557(1)	5601(2)	9679(1)	47(1)
O(1B)	5524(1)	7786(2)	9360(1)	68(1)
N(3)	6420(1)	4144(2)	8870(1)	38(1)
N(5)	7490(1)	8194(2)	5986(1)	22(1)
C(1)	5682(1)	6651(3)	9318(1)	39(1)
C(2)	6068(1)	6479(2)	8788(1)	26(1)
C(3)	6266(1)	5191(2)	8822(1)	27(1)
C(4)	6194(1)	7495(2)	8303(1)	25(1)
C(6)	7178(1)	7993(2)	6570(1)	19(1)
C(7)	7188(1)	6900(2)	7114(1)	23(1)
C(8)	6875(1)	6715(2)	7680(1)	23(1)

C(9)	6535(1)	7605(2)	7729(1)	20(1)
C(10)	6532(1)	8697(2)	7185(1)	21(1)
C(11)	6844(1)	8896(2)	6620(1)	21(1)
C(12)	7908(1)	7772(2)	6124(1)	20(1)
C(13)	8145(1)	7260(2)	5482(1)	22(1)
C(14)	8555(1)	6894(2)	5606(1)	26(1)
C(15)	8735(1)	7014(2)	6371(1)	29(1)
C(16)	8501(1)	7512(2)	7009(1)	28(1)
C(17)	8090(1)	7902(2)	6891(1)	24(1)
C(18)	7391(1)	8833(2)	5228(1)	20(1)
C(19)	7040(1)	8448(2)	4794(1)	24(1)
C(20)	6952(1)	9045(2)	4051(1)	27(1)
C(21)	7213(1)	10019(2)	3730(1)	27(1)
C(22)	7562(1)	10406(2)	4161(1)	25(1)
C(23)	7650(1)	9825(2)	4913(1)	22(1)
N(24)	5185(1)	6911(2)	4627(1)	46(1)
C(25)	5405(1)	7968(3)	5090(1)	54(1)
C(26)	5790(1)	8442(2)	4638(2)	43(1)
C(27)	5679(1)	8890(2)	3783(2)	46(1)
C(28)	5451(1)	7782(2)	3337(1)	42(1)
C(29)	5070(1)	7349(2)	3797(1)	42(1)
N(30)	4384(1)	4842(2)	2983(2)	60(1)
C(31)	4112(1)	4655(2)	3420(1)	38(1)
C(32)	3768(1)	4424(2)	3966(1)	36(1)
N(33)	5436(1)	9566(4)	6901(3)	50(1)
C(34)	5227(1)	8759(5)	7171(2)	36(1)
C(35)	5000	7720(4)	7500	58(1)

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

O(1A)-C(1)	1.262(3)	C(6)-N(5)-C(12)	121.75(14)
O(1B)-C(1)	1.235(3)	C(6)-N(5)-C(18)	119.80(14)
N(3)-C(3)	1.150(3)	C(12)-N(5)-C(18)	118.45(13)
N(5)-C(6)	1.402(2)	O(1B)-C(1)-O(1A)	126.31(19)
N(5)-C(12)	1.424(2)	O(1B)-C(1)-C(2)	117.90(19)

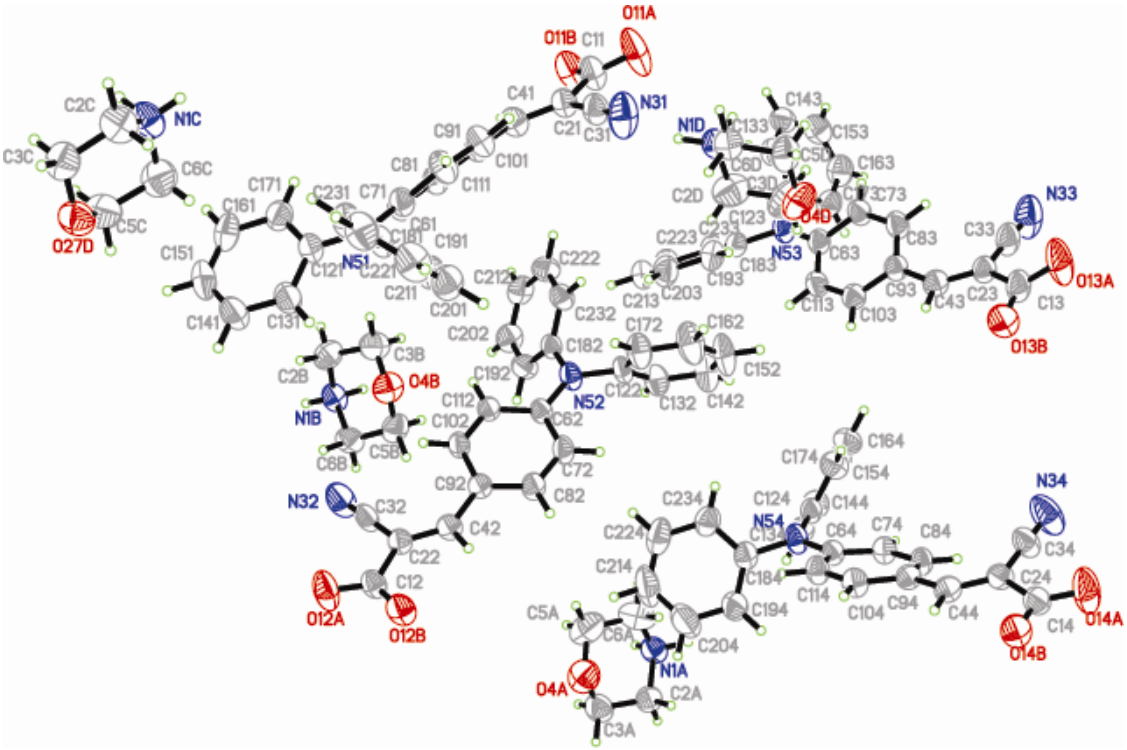
N(5)-C(18)	1.432(2)	O(1A)-C(1)-C(2)	115.8(2)
C(1)-C(2)	1.527(3)	C(4)-C(2)-C(3)	123.98(16)
C(2)-C(4)	1.344(3)	C(4)-C(2)-C(1)	119.84(18)
C(2)-C(3)	1.427(3)	C(3)-C(2)-C(1)	116.13(17)
C(4)-C(9)	1.452(2)	N(3)-C(3)-C(2)	178.0(2)
C(6)-C(11)	1.401(2)	C(2)-C(4)-C(9)	131.82(17)
C(6)-C(7)	1.403(2)	C(11)-C(6)-N(5)	119.98(15)
C(7)-C(8)	1.381(2)	C(11)-C(6)-C(7)	118.22(15)
C(8)-C(9)	1.407(2)	N(5)-C(6)-C(7)	121.80(15)
C(9)-C(10)	1.402(2)	C(8)-C(7)-C(6)	120.91(16)
C(10)-C(11)	1.380(2)	C(7)-C(8)-C(9)	121.37(16)
C(12)-C(13)	1.395(2)	C(10)-C(9)-C(8)	116.98(15)
C(12)-C(17)	1.395(2)	C(10)-C(9)-C(4)	117.68(15)
C(13)-C(14)	1.384(3)	C(8)-C(9)-C(4)	125.34(15)
C(14)-C(15)	1.387(3)	C(11)-C(10)-C(9)	122.13(16)
C(15)-C(16)	1.381(3)	C(10)-C(11)-C(6)	120.39(15)
C(16)-C(17)	1.389(3)	C(13)-C(12)-C(17)	118.94(16)
C(18)-C(23)	1.388(2)	C(13)-C(12)-N(5)	120.16(15)
C(18)-C(19)	1.390(2)	C(17)-C(12)-N(5)	120.87(15)
C(19)-C(20)	1.385(3)	C(14)-C(13)-C(12)	120.35(17)
C(20)-C(21)	1.384(3)	C(13)-C(14)-C(15)	120.58(17)
C(21)-C(22)	1.381(3)	C(16)-C(15)-C(14)	119.27(17)
C(22)-C(23)	1.389(2)	C(15)-C(16)-C(17)	120.77(18)
N(24)-C(25)	1.473(4)	C(16)-C(17)-C(12)	120.08(17)
N(24)-C(29)	1.477(3)	C(23)-C(18)-C(19)	119.42(16)
C(25)-C(26)	1.515(3)	C(23)-C(18)-N(5)	120.26(15)
C(26)-C(27)	1.515(4)	C(19)-C(18)-N(5)	120.31(15)
C(27)-C(28)	1.508(3)	C(20)-C(19)-C(18)	120.10(17)
C(28)-C(29)	1.501(3)	C(21)-C(20)-C(19)	120.47(17)
N(30)-C(31)	1.145(3)	C(22)-C(21)-C(20)	119.54(16)
C(31)-C(32)	1.444(3)	C(21)-C(22)-C(23)	120.37(17)
N(33)-C(34)	1.135(6)	C(18)-C(23)-C(22)	120.08(16)
C(34)-C(35)	1.371(6)	C(25)-N(24)-C(29)	112.81(18)
C(34)-C(34)#1	1.814(9)	N(24)-C(25)-C(26)	111.08(18)
C(35)-C(34)#1	1.371(6)	C(27)-C(26)-C(25)	110.60(18)

	C(28)-C(27)-C(26)	110.50(18)
	C(29)-C(28)-C(27)	111.19(19)
	N(24)-C(29)-C(28)	109.96(17)
	N(30)-C(31)-C(32)	179.5(3)
	N(33)-C(34)-C(35)	175.6(4)
	N(33)-C(34)-C(34)#1	135.2(3)
	C(35)-C(34)-C(34)#1	48.6(2)
	C(34)-C(35)-C(34)#1	82.8(4)

Symmetry transformations used to generate equivalent atoms:

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

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



Identification code	4 (CCDC No: 737401)
Empirical formula	C26 H25 N3 O3
Formula weight	427.49

Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 11.231(1) Å	α= 99.435(1)°.
	b = 16.368(1) Å	β= 102.219(1)°.
	c = 26.031(2) Å	γ = 99.471(2)°.
Volume	4514.0(6) $Å^3$	
Z	8	
Density (calculated)	1.258 Mg/m^3	
Absorption coefficient	0.083 mm^{-1}	
F(000)	1808	
Crystal size	0.23 x 0.14 x 0.09 mm^3	
Theta range for data collection	0.82 to 25.04°.	
Index ranges	-13<=h<=13, -19<=k<=19, -30<=l<=30	
Reflections collected	49086	
Independent reflections	15976 [R(int) = 0.0505]	
Completeness to theta = 25.04°	99.8 %	
Absorption correction	Multi scan	
Max. and min. transmission	0.9925 and 0.9811	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	15976 / 0 / 1185	
Goodness-of-fit on F^2	1.015	
Final R indices [I>2sigma(I)]	R1 = 0.0580, wR2 = 0.1282	
R indices (all data)	R1 = 0.1035, wR2 = 0.1494	
Largest diff. peak and hole	0.307 and -0.200 $e.Å^{-3}$	

Table 2. 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)
O(13A)	4573(2)	-780(2)	9139(1)	105(1)
O(13B)	5706(2)	321(1)	8961(1)	73(1)
N(33)	1735(2)	-419(2)	9210(1)	103(1)
N(53)	-365(2)	2428(1)	7672(1)	52(1)
C(13)	4712(2)	-80(2)	9009(1)	61(1)
C(23)	3562(2)	276(2)	8870(1)	49(1)
C(33)	2526(3)	-110(2)	9047(1)	65(1)
C(43)	3540(2)	904(2)	8600(1)	50(1)
C(63)	611(2)	2054(1)	7892(1)	46(1)
C(73)	342(2)	1249(2)	8004(1)	54(1)
C(83)	1270(2)	880(2)	8241(1)	54(1)
C(93)	2518(2)	1278(2)	8365(1)	46(1)
C(103)	2782(2)	2066(2)	8233(1)	49(1)
C(113)	1861(2)	2453(2)	8012(1)	49(1)
C(123)	-1602(2)	2091(2)	7709(1)	50(1)
C(133)	-2523(2)	1731(2)	7247(1)	54(1)
C(143)	-3711(2)	1406(2)	7278(1)	59(1)
C(153)	-3972(3)	1411(2)	7770(1)	69(1)
C(163)	-3060(3)	1774(2)	8231(1)	70(1)
C(173)	-1875(2)	2123(2)	8204(1)	61(1)
C(183)	-231(2)	3073(2)	7374(1)	47(1)
C(193)	624(2)	3134(2)	7061(1)	58(1)
C(203)	724(3)	3766(2)	6776(1)	70(1)
C(213)	-35(3)	4339(2)	6787(1)	73(1)
C(223)	-891(3)	4284(2)	7089(1)	67(1)
C(233)	-991(2)	3660(2)	7385(1)	56(1)
O(12A)	9854(2)	8411(1)	4872(1)	84(1)
O(12B)	10862(2)	7750(1)	5430(1)	61(1)
N(32)	6786(2)	8087(2)	4834(1)	83(1)
N(52)	4526(2)	4975(1)	6110(1)	48(1)
C(12)	9896(2)	7939(2)	5198(1)	48(1)

C(22)	8671(2)	7545(1)	5300(1)	41(1)
C(32)	7611(2)	7841(2)	5040(1)	53(1)
C(42)	8592(2)	6950(1)	5594(1)	43(1)
C(62)	5519(2)	5443(1)	5970(1)	40(1)
C(72)	6742(2)	5512(2)	6252(1)	48(1)
C(82)	7713(2)	5993(1)	6119(1)	47(1)
C(92)	7523(2)	6436(1)	5706(1)	40(1)
C(102)	6294(2)	6334(1)	5414(1)	42(1)
C(112)	5322(2)	5848(1)	5536(1)	41(1)
C(122)	4718(2)	4387(1)	6452(1)	44(1)
C(132)	4422(2)	4518(2)	6939(1)	57(1)
C(142)	4563(3)	3944(2)	7268(1)	75(1)
C(152)	4996(3)	3240(2)	7107(1)	84(1)
C(162)	5284(3)	3100(2)	6616(1)	82(1)
C(172)	5147(2)	3676(2)	6288(1)	63(1)
C(182)	3269(2)	5068(1)	5933(1)	40(1)
C(192)	2959(2)	5853(2)	5977(1)	52(1)
C(202)	1729(2)	5917(2)	5827(1)	57(1)
C(212)	809(2)	5211(2)	5638(1)	57(1)
C(222)	1111(2)	4430(2)	5584(1)	57(1)
C(232)	2340(2)	4360(2)	5734(1)	49(1)
O(11A)	1538(2)	-603(1)	4213(1)	98(1)
O(11B)	711(2)	464(1)	4002(1)	74(1)
N(31)	4451(2)	-427(2)	4092(1)	81(1)
N(51)	6546(2)	2450(1)	2671(1)	51(1)
C(11)	1559(2)	64(2)	4046(1)	57(1)
C(21)	2700(2)	385(2)	3858(1)	46(1)
C(31)	3680(2)	-73(2)	3974(1)	52(1)
C(41)	2775(2)	1038(2)	3609(1)	49(1)
C(61)	5588(2)	2103(1)	2885(1)	44(1)
C(71)	4753(2)	2566(1)	3053(1)	45(1)
C(81)	3847(2)	2215(1)	3279(1)	45(1)
C(91)	3736(2)	1385(1)	3361(1)	44(1)
C(101)	4555(2)	919(2)	3180(1)	51(1)
C(111)	5445(2)	1262(2)	2946(1)	52(1)
C(121)	6484(2)	3135(1)	2401(1)	45(1)

C(131)	7562(2)	3714(2)	2439(1)	57(1)
C(141)	7522(3)	4356(2)	2158(1)	66(1)
C(151)	6434(3)	4440(2)	1844(1)	65(1)
C(161)	5365(3)	3876(2)	1811(1)	62(1)
C(171)	5382(2)	3224(2)	2083(1)	52(1)
C(181)	7608(2)	2063(1)	2679(1)	47(1)
C(191)	8342(2)	1996(2)	3162(1)	53(1)
C(201)	9349(2)	1616(2)	3171(1)	61(1)
C(211)	9642(3)	1318(2)	2700(1)	66(1)
C(221)	8932(3)	1396(2)	2221(1)	64(1)
C(231)	7904(2)	1757(2)	2209(1)	57(1)
O(14A)	13297(2)	1702(2)	10124(1)	101(1)
O(14B)	14199(2)	2482(1)	9655(1)	63(1)
N(34)	10319(2)	1891(2)	10043(1)	92(1)
N(54)	8392(2)	5051(1)	8813(1)	51(1)
C(14)	13306(2)	2213(2)	9828(1)	52(1)
C(24)	12134(2)	2562(1)	9692(1)	44(1)
C(34)	11122(2)	2199(2)	9890(1)	57(1)
C(44)	12092(2)	3179(1)	9414(1)	45(1)
C(64)	9333(2)	4620(1)	8980(1)	42(1)
C(74)	9336(2)	4219(1)	9411(1)	44(1)
C(84)	10216(2)	3754(1)	9556(1)	44(1)
C(94)	11151(2)	3675(1)	9285(1)	42(1)
C(104)	11183(2)	4124(2)	8877(1)	48(1)
C(114)	10292(2)	4579(2)	8721(1)	47(1)
C(124)	7263(2)	4920(2)	8985(1)	43(1)
C(134)	6908(2)	5599(2)	9252(1)	48(1)
C(144)	5827(2)	5487(2)	9424(1)	57(1)
C(154)	5099(2)	4698(2)	9331(1)	62(1)
C(164)	5442(2)	4018(2)	9067(1)	63(1)
C(174)	6519(2)	4123(2)	8889(1)	58(1)
C(184)	8546(2)	5693(2)	8509(1)	47(1)
C(194)	9511(2)	6383(2)	8691(1)	72(1)
C(204)	9596(3)	7005(2)	8398(2)	103(1)
C(214)	8728(3)	6949(2)	7931(2)	101(1)
C(224)	7758(3)	6275(2)	7755(1)	76(1)

C(234)	7669(2)	5644(2)	8040(1)	57(1)
O(4B)	3687(2)	6914(1)	4815(1)	65(1)
N(1B)	2356(2)	8218(2)	4715(1)	54(1)
C(2B)	2046(2)	7435(2)	4299(1)	66(1)
C(3B)	2411(2)	6721(2)	4550(1)	67(1)
C(5B)	3962(2)	7642(2)	5224(1)	60(1)
C(6B)	3690(2)	8394(2)	5001(1)	56(1)
O(4D)	3101(2)	442(2)	6637(1)	84(1)
N(1D)	1205(2)	644(2)	5792(1)	54(1)
C(2D)	1637(3)	1268(2)	6300(1)	86(1)
C(3D)	2127(3)	841(2)	6745(1)	98(1)
C(5D)	2647(3)	-178(2)	6161(1)	67(1)
C(6D)	2199(2)	199(2)	5690(1)	55(1)
O(4A)	8725(2)	9604(2)	8439(1)	84(1)
N(1A)	7385(2)	9300(2)	9219(1)	57(1)
C(2A)	8670(2)	9799(2)	9369(1)	58(1)
C(3A)	8922(3)	10206(2)	8920(1)	70(1)
C(5A)	7472(3)	9155(2)	8285(1)	94(1)
C(6A)	7153(3)	8699(2)	8706(1)	89(1)
O(27D)	7986(2)	3268(1)	238(1)	67(1)
N(1C)	6103(2)	1953(2)	307(1)	60(1)
C(2C)	7072(2)	1786(2)	22(1)	63(1)
C(3C)	7508(2)	2551(2)	-182(1)	64(1)
C(5C)	7042(3)	3446(2)	496(1)	71(1)
C(6C)	6570(3)	2724(2)	733(1)	73(1)

Table 3. Bond lengths [Å] and angles [°] for 4.

O(13A)-C(13)	1.243(3)	C(63)-N(53)-C(183)	124.12(19)
O(13B)-C(13)	1.239(3)	C(63)-N(53)-C(123)	118.44(18)
N(33)-C(33)	1.143(3)	C(183)-N(53)-C(123)	117.21(18)
N(53)-C(63)	1.405(3)	O(13B)-C(13)-O(13A)	125.1(2)
N(53)-C(183)	1.415(3)	O(13B)-C(13)-C(23)	118.3(2)
N(53)-C(123)	1.438(3)	O(13A)-C(13)-C(23)	116.6(2)

C(13)-C(23)	1.503(3)	C(43)-C(23)-C(33)	124.1(2)
C(23)-C(43)	1.339(3)	C(43)-C(23)-C(13)	121.1(2)
C(23)-C(33)	1.429(4)	C(33)-C(23)-C(13)	114.9(2)
C(43)-C(93)	1.457(3)	N(33)-C(33)-C(23)	176.9(3)
C(63)-C(113)	1.393(3)	C(23)-C(43)-C(93)	131.2(2)
C(63)-C(73)	1.395(3)	C(113)-C(63)-C(73)	117.1(2)
C(73)-C(83)	1.375(3)	C(113)-C(63)-N(53)	123.1(2)
C(83)-C(93)	1.390(3)	C(73)-C(63)-N(53)	119.8(2)
C(93)-C(103)	1.390(3)	C(83)-C(73)-C(63)	121.2(2)
C(103)-C(113)	1.373(3)	C(73)-C(83)-C(93)	121.8(2)
C(123)-C(133)	1.379(3)	C(103)-C(93)-C(83)	116.7(2)
C(123)-C(173)	1.380(3)	C(103)-C(93)-C(43)	119.3(2)
C(133)-C(143)	1.377(3)	C(83)-C(93)-C(43)	123.9(2)
C(143)-C(153)	1.372(4)	C(113)-C(103)-C(93)	122.1(2)
C(153)-C(163)	1.373(4)	C(103)-C(113)-C(63)	121.1(2)
C(163)-C(173)	1.382(4)	C(133)-C(123)-C(173)	119.8(2)
C(183)-C(193)	1.386(3)	C(133)-C(123)-N(53)	119.8(2)
C(183)-C(233)	1.387(3)	C(173)-C(123)-N(53)	120.5(2)
C(193)-C(203)	1.372(3)	C(143)-C(133)-C(123)	120.3(3)
C(203)-C(213)	1.368(4)	C(153)-C(143)-C(133)	120.0(3)
C(213)-C(223)	1.367(4)	C(143)-C(153)-C(163)	119.9(3)
C(223)-C(233)	1.379(3)	C(153)-C(163)-C(173)	120.5(3)
O(12A)-C(12)	1.236(3)	C(123)-C(173)-C(163)	119.6(3)
O(12B)-C(12)	1.238(3)	C(193)-C(183)-C(233)	118.2(2)
N(32)-C(32)	1.136(3)	C(193)-C(183)-N(53)	122.4(2)
N(52)-C(62)	1.394(3)	C(233)-C(183)-N(53)	119.4(2)
N(52)-C(122)	1.426(3)	C(203)-C(193)-C(183)	120.7(3)
N(52)-C(182)	1.431(3)	C(213)-C(203)-C(193)	120.5(3)
C(12)-C(22)	1.516(3)	C(223)-C(213)-C(203)	119.5(3)
C(22)-C(42)	1.338(3)	C(213)-C(223)-C(233)	120.6(3)
C(22)-C(32)	1.437(3)	C(223)-C(233)-C(183)	120.3(3)
C(42)-C(92)	1.459(3)	C(62)-N(52)-C(122)	121.36(17)
C(62)-C(72)	1.391(3)	C(62)-N(52)-C(182)	122.56(17)
C(62)-C(112)	1.397(3)	C(122)-N(52)-C(182)	116.07(17)
C(72)-C(82)	1.375(3)	O(12A)-C(12)-O(12B)	124.2(2)

C(82)-C(92)	1.393(3)	O(12A)-C(12)-C(22)	117.2(2)
C(92)-C(102)	1.395(3)	O(12B)-C(12)-C(22)	118.5(2)
C(102)-C(112)	1.368(3)	C(42)-C(22)-C(32)	123.4(2)
C(122)-C(132)	1.370(3)	C(42)-C(22)-C(12)	122.0(2)
C(122)-C(172)	1.371(3)	C(32)-C(22)-C(12)	114.57(19)
C(132)-C(142)	1.378(3)	N(32)-C(32)-C(22)	178.9(3)
C(142)-C(152)	1.361(4)	C(22)-C(42)-C(92)	131.6(2)
C(152)-C(162)	1.376(4)	C(72)-C(62)-N(52)	121.24(19)
C(162)-C(172)	1.376(4)	C(72)-C(62)-C(112)	117.6(2)
C(182)-C(232)	1.369(3)	N(52)-C(62)-C(112)	121.17(19)
C(182)-C(192)	1.379(3)	C(82)-C(72)-C(62)	120.8(2)
C(192)-C(202)	1.380(3)	C(72)-C(82)-C(92)	122.1(2)
C(202)-C(212)	1.360(4)	C(82)-C(92)-C(102)	116.3(2)
C(212)-C(222)	1.368(3)	C(82)-C(92)-C(42)	119.32(19)
C(222)-C(232)	1.382(3)	C(102)-C(92)-C(42)	124.4(2)
O(11A)-C(11)	1.238(3)	C(112)-C(102)-C(92)	122.2(2)
O(11B)-C(11)	1.238(3)	C(102)-C(112)-C(62)	120.9(2)
N(31)-C(31)	1.136(3)	C(132)-C(122)-C(172)	119.7(2)
N(51)-C(61)	1.396(3)	C(132)-C(122)-N(52)	119.6(2)
N(51)-C(121)	1.421(3)	C(172)-C(122)-N(52)	120.6(2)
N(51)-C(181)	1.437(3)	C(122)-C(132)-C(142)	120.6(2)
C(11)-C(21)	1.512(3)	C(152)-C(142)-C(132)	119.6(3)
C(21)-C(41)	1.338(3)	C(142)-C(152)-C(162)	120.1(3)
C(21)-C(31)	1.438(3)	C(152)-C(162)-C(172)	120.1(3)
C(41)-C(91)	1.455(3)	C(122)-C(172)-C(162)	119.8(3)
C(61)-C(71)	1.394(3)	C(232)-C(182)-C(192)	118.9(2)
C(61)-C(111)	1.397(3)	C(232)-C(182)-N(52)	119.4(2)
C(71)-C(81)	1.373(3)	C(192)-C(182)-N(52)	121.7(2)
C(81)-C(91)	1.399(3)	C(182)-C(192)-C(202)	120.1(2)
C(91)-C(101)	1.395(3)	C(212)-C(202)-C(192)	120.7(2)
C(101)-C(111)	1.369(3)	C(202)-C(212)-C(222)	119.6(2)
C(121)-C(171)	1.380(3)	C(212)-C(222)-C(232)	120.1(2)
C(121)-C(131)	1.384(3)	C(182)-C(232)-C(222)	120.6(2)
C(131)-C(141)	1.375(3)	C(61)-N(51)-C(121)	123.08(18)
C(141)-C(151)	1.360(4)	C(61)-N(51)-C(181)	119.44(17)

C(151)-C(161)	1.366(4)	C(121)-N(51)-C(181)	117.28(18)
C(161)-C(171)	1.375(3)	O(11A)-C(11)-O(11B)	125.5(2)
C(181)-C(231)	1.374(3)	O(11A)-C(11)-C(21)	116.3(2)
C(181)-C(191)	1.381(3)	O(11B)-C(11)-C(21)	118.1(2)
C(191)-C(201)	1.376(3)	C(41)-C(21)-C(31)	123.9(2)
C(201)-C(211)	1.372(4)	C(41)-C(21)-C(11)	122.2(2)
C(211)-C(221)	1.368(4)	C(31)-C(21)-C(11)	113.9(2)
C(221)-C(231)	1.378(3)	N(31)-C(31)-C(21)	176.5(3)
O(14A)-C(14)	1.227(3)	C(21)-C(41)-C(91)	130.0(2)
O(14B)-C(14)	1.229(3)	C(71)-C(61)-N(51)	122.6(2)
N(34)-C(34)	1.140(3)	C(71)-C(61)-C(111)	117.3(2)
N(54)-C(64)	1.399(3)	N(51)-C(61)-C(111)	120.1(2)
N(54)-C(184)	1.426(3)	C(81)-C(71)-C(61)	121.1(2)
N(54)-C(124)	1.427(3)	C(71)-C(81)-C(91)	121.7(2)
C(14)-C(24)	1.516(3)	C(101)-C(91)-C(81)	116.7(2)
C(24)-C(44)	1.337(3)	C(101)-C(91)-C(41)	123.2(2)
C(24)-C(34)	1.426(3)	C(81)-C(91)-C(41)	120.1(2)
C(44)-C(94)	1.453(3)	C(111)-C(101)-C(91)	121.7(2)
C(64)-C(74)	1.389(3)	C(101)-C(111)-C(61)	121.3(2)
C(64)-C(114)	1.392(3)	C(171)-C(121)-C(131)	118.6(2)
C(74)-C(84)	1.372(3)	C(171)-C(121)-N(51)	121.8(2)
C(84)-C(94)	1.395(3)	C(131)-C(121)-N(51)	119.6(2)
C(94)-C(104)	1.391(3)	C(141)-C(131)-C(121)	120.0(2)
C(104)-C(114)	1.376(3)	C(151)-C(141)-C(131)	121.3(3)
C(124)-C(134)	1.374(3)	C(141)-C(151)-C(161)	118.9(2)
C(124)-C(174)	1.381(3)	C(151)-C(161)-C(171)	121.0(3)
C(134)-C(144)	1.376(3)	C(161)-C(171)-C(121)	120.2(2)
C(144)-C(154)	1.364(4)	C(231)-C(181)-C(191)	119.3(2)
C(154)-C(164)	1.364(4)	C(231)-C(181)-N(51)	120.5(2)
C(164)-C(174)	1.380(3)	C(191)-C(181)-N(51)	120.2(2)
C(184)-C(194)	1.370(3)	C(201)-C(191)-C(181)	120.3(2)
C(184)-C(234)	1.378(3)	C(211)-C(201)-C(191)	119.9(3)
C(194)-C(204)	1.370(4)	C(221)-C(211)-C(201)	120.1(3)
C(204)-C(214)	1.369(5)	C(211)-C(221)-C(231)	120.2(3)
C(214)-C(224)	1.357(4)	C(181)-C(231)-C(221)	120.1(2)

C(224)-C(234)	1.370(4)	C(64)-N(54)-C(184)	122.57(18)
O(4B)-C(5B)	1.405(3)	C(64)-N(54)-C(124)	120.64(18)
O(4B)-C(3B)	1.411(3)	C(184)-N(54)-C(124)	116.54(17)
N(1B)-C(2B)	1.475(3)	O(14A)-C(14)-O(14B)	125.1(2)
N(1B)-C(6B)	1.482(3)	O(14A)-C(14)-C(24)	116.3(2)
C(2B)-C(3B)	1.501(3)	O(14B)-C(14)-C(24)	118.6(2)
C(5B)-C(6B)	1.492(3)	C(44)-C(24)-C(34)	123.7(2)
O(4D)-C(5D)	1.405(3)	C(44)-C(24)-C(14)	120.9(2)
O(4D)-C(3D)	1.417(3)	C(34)-C(24)-C(14)	115.5(2)
N(1D)-C(2D)	1.465(3)	N(34)-C(34)-C(24)	178.3(3)
N(1D)-C(6D)	1.474(3)	C(24)-C(44)-C(94)	131.3(2)
C(2D)-C(3D)	1.504(4)	C(74)-C(64)-C(114)	118.1(2)
C(5D)-C(6D)	1.490(3)	C(74)-C(64)-N(54)	120.8(2)
O(4A)-C(3A)	1.412(3)	C(114)-C(64)-N(54)	121.1(2)
O(4A)-C(5A)	1.415(4)	C(84)-C(74)-C(64)	120.9(2)
N(1A)-C(6A)	1.469(4)	C(74)-C(84)-C(94)	121.7(2)
N(1A)-C(2A)	1.474(3)	C(104)-C(94)-C(84)	116.7(2)
C(2A)-C(3A)	1.491(3)	C(104)-C(94)-C(44)	119.2(2)
C(5A)-C(6A)	1.495(4)	C(84)-C(94)-C(44)	124.1(2)
O(27D)-C(3C)	1.408(3)	C(114)-C(104)-C(94)	122.1(2)
O(27D)-C(5C)	1.414(3)	C(104)-C(114)-C(64)	120.4(2)
N(1C)-C(6C)	1.475(3)	C(134)-C(124)-C(174)	119.2(2)
N(1C)-C(2C)	1.478(3)	C(134)-C(124)-N(54)	119.4(2)
C(2C)-C(3C)	1.488(4)	C(174)-C(124)-N(54)	121.4(2)
C(5C)-C(6C)	1.492(4)	C(124)-C(134)-C(144)	120.4(2)
		C(154)-C(144)-C(134)	120.1(2)
		C(144)-C(154)-C(164)	120.1(2)
		C(154)-C(164)-C(174)	120.3(3)
		C(164)-C(174)-C(124)	119.8(2)
		C(194)-C(184)-C(234)	119.5(2)
		C(194)-C(184)-N(54)	121.0(2)
		C(234)-C(184)-N(54)	119.3(2)
		C(204)-C(194)-C(184)	119.3(3)
		C(214)-C(204)-C(194)	121.0(3)
		C(224)-C(214)-C(204)	119.8(3)

	C(214)-C(224)-C(234)	119.9(3)
	C(224)-C(234)-C(184)	120.5(3)
	C(5B)-O(4B)-C(3B)	110.57(19)
	C(2B)-N(1B)-C(6B)	110.3(2)
	N(1B)-C(2B)-C(3B)	109.5(2)
	O(4B)-C(3B)-C(2B)	111.1(2)
	O(4B)-C(5B)-C(6B)	111.2(2)
	N(1B)-C(6B)-C(5B)	108.8(2)
	C(5D)-O(4D)-C(3D)	109.3(2)
	C(2D)-N(1D)-C(6D)	110.7(2)
	N(1D)-C(2D)-C(3D)	109.4(2)
	O(4D)-C(3D)-C(2D)	111.2(3)
	O(4D)-C(5D)-C(6D)	111.7(2)
	N(1D)-C(6D)-C(5D)	109.1(2)
	C(3A)-O(4A)-C(5A)	109.7(2)
	C(6A)-N(1A)-C(2A)	110.6(2)
	N(1A)-C(2A)-C(3A)	109.9(2)
	O(4A)-C(3A)-C(2A)	111.8(2)
	O(4A)-C(5A)-C(6A)	111.5(3)
	N(1A)-C(6A)-C(5A)	110.0(3)
	C(3C)-O(27D)-C(5C)	109.8(2)
	C(6C)-N(1C)-C(2C)	110.7(2)
	N(1C)-C(2C)-C(3C)	108.8(2)
	O(27D)-C(3C)-C(2C)	111.6(2)
	O(27D)-C(5C)-C(6C)	111.3(2)
	N(1C)-C(6C)-C(5C)	109.3(2)

Symmetry transformations used to generate equivalent atoms: