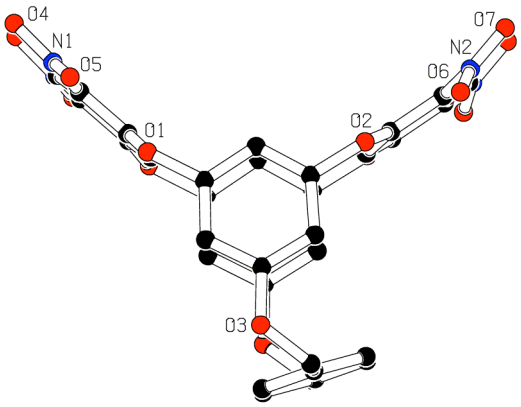
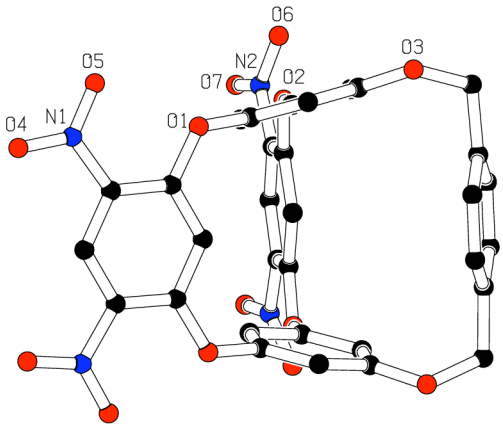
X-Ray Structure Determination, $C_{32}H_{18}N_4O_{14}$·unknown solvate (host 2)

X-ray diffraction intensity data from a pale yellow plate crystal were measured at 150(1) K on a Bruker SMART APEX diffractometer (Mo Kα radiation, λ = 0.71073 Å).[1] Raw area detector data frame integration was performed with SAINT+.[1] Final unit cell parameters were determined by least-squares refinement of 3703 strong reflections from the data set. Direct methods structure solution, difference Fourier calculations and full-matrix least-squares refinement against F^2 were performed with SHELXTL.[2]

Systematic absences were consistent with the space groups $P2_1$ and $P2_1/m$; intensity statistics indicated a centric structure. The space group $P2_1/m$ was determined by the solution and refinement of the data. The asymmetric unit consists of half of a $C_{32}H_{18}N_4O_{14}$ molecule located on a crystallographic mirror plane, and two independent regions of disordered solvent molecules. Several attempts to model the solvent disorder indicated these to be dichloromethane molecules, but no reasonable disorder model could be achieved. The program SQUEEZE (PLATON)[3] was therefore used to removed the contribution of the disordered species from the structure factor calculations. The program calculated a solvent-accessible void volume of 506.6 $Å^3$ per unit cell, corresponding to 156 electrons per cell. Note that the d_{calc}, F(000), and M.W. refer to known unit cell contents only. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in geometrically idealized positions and included as riding atoms. Nitro group atoms N2, O6, and O7 show elongated displacement ellipsoids, likely because of minor positional disorder of the molecule across the mirror plane. Trial refinements in $P2_1$ to remove the imposed mirror symmetry gave no improvement and resulted in an unstable refinement. The largest residual electron density peaks are in the vicinity of this nitro group.

(1) SMART Version 5.630, SAINT+ Version 6.45. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 2003.
(2) Sheldrick, G. M. SHELXTL Version 6.14; Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 2000.
(3) PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, Spek, A. L. 1998.





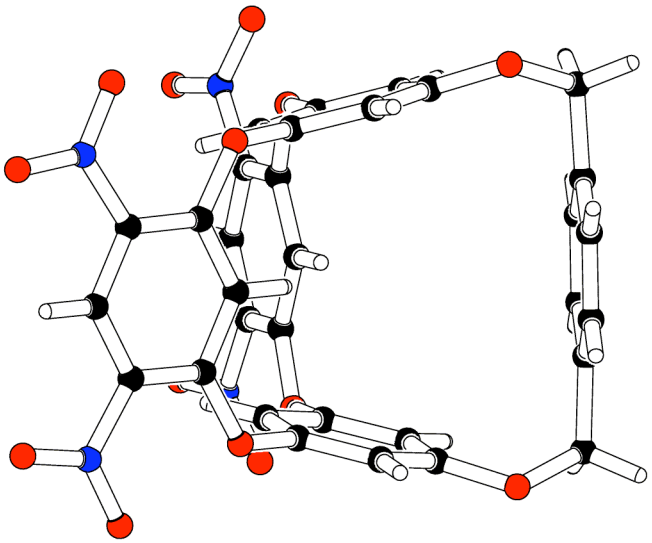


Table 1. Crystal data and structure refinement for **2**.

Identification code	**2**	
Empirical formula	C32 H18 N4 O14	
Formula weight	682.50	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/m$	
Unit cell dimensions	a = 8.8759(8) Å	α= 90°.
	b = 12.7053(12) Å	β= 103.925(2)°.
	c = 16.6618(15) Å	γ = 90°.
Volume	1823.7(3) $Å^3$	
Z	2	
Density (calculated)	1.243 Mg/m^3	
Absorption coefficient	0.100 mm^{-1}	
F(000)	700	
Crystal size	0.38 x 0.20 x 0.05 mm^3	
Theta range for data collection	1.26 to 22.54°.	
Index ranges	-9<=h<=9, -13<=k<=13, -17<=l<=17	
Reflections collected	15369	
Independent reflections	2533 [R(int) = 0.0705]	
Completeness to theta = 22.54°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2533 / 0 / 232	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.0480, wR2 = 0.1206	
R indices (all data)	R1 = 0.0648, wR2 = 0.1269	
Largest diff. peak and hole	0.519 and -0.361 $e.Å^{-3}$	

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)
C(1)	4182(3)	572(2)	1331(2)	33(1)
C(2)	3570(3)	716(2)	2012(2)	37(1)
C(3)	4577(3)	582(2)	2774(2)	39(1)
C(4)	6120(3)	318(2)	2875(2)	39(1)
C(5)	6677(3)	181(2)	2177(2)	36(1)
C(6)	5704(3)	311(2)	1392(2)	35(1)
C(7)	2533(3)	1546(2)	245(2)	30(1)
C(8)	1245(3)	1555(2)	-438(2)	28(1)
C(9)	630(4)	2500	-762(2)	29(1)
C(10)	3145(4)	2500	569(2)	30(1)
C(11)	3774(3)	1550(3)	3824(2)	44(1)
C(12)	3140(4)	1555(3)	4513(2)	57(1)
C(13)	2828(5)	2500	4845(3)	66(2)
C(14)	4111(4)	2500	3497(2)	44(1)
C(15)	9353(3)	218(2)	2909(2)	45(1)
C(16)	9428(3)	1397(2)	2943(2)	38(1)
C(17)	9278(3)	1957(2)	3639(2)	46(1)
C(18)	9576(3)	1957(2)	2248(2)	38(1)
O(1)	3181(2)	615(1)	536(1)	39(1)
O(2)	3999(2)	610(2)	3492(1)	49(1)
O(3)	8163(2)	-131(2)	2204(1)	41(1)
N(1)	457(3)	603(2)	-815(1)	33(1)
O(4)	-718(2)	716(2)	-1369(1)	44(1)
O(5)	998(2)	-257(2)	-562(1)	39(1)
N(2)	2685(4)	595(4)	4879(2)	74(1)
O(6)	3297(5)	-257(3)	4769(2)	124(1)
O(7)	1818(3)	663(3)	5324(2)	112(1)

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

C(1)-C(6)	1.371(4)
C(1)-C(2)	1.383(4)
C(1)-O(1)	1.407(3)
C(2)-C(3)	1.375(4)
C(2)-H(2)	0.9500
C(3)-C(4)	1.380(4)
C(3)-O(2)	1.412(3)
C(4)-C(5)	1.380(4)
C(4)-H(4)	0.9500
C(5)-O(3)	1.367(3)
C(5)-C(6)	1.393(4)
C(6)-H(6)	0.9500
C(7)-O(1)	1.353(3)
C(7)-C(10)	1.384(3)
C(7)-C(8)	1.406(4)
C(8)-C(9)	1.375(3)
C(8)-N(1)	1.461(3)
C(9)-C(8)#1	1.375(3)
C(9)-H(9)	0.9500
C(10)-C(7)#1	1.384(3)
C(10)-H(10)	0.9500
C(11)-O(2)	1.352(4)
C(11)-C(14)	1.386(4)
C(11)-C(12)	1.395(4)
C(12)-C(13)	1.377(4)
C(12)-N(2)	1.463(5)
C(13)-C(12)#1	1.377(4)
C(13)-H(13)	0.9500
C(14)-C(11)#1	1.386(4)
C(14)-H(14)	0.9500
C(15)-O(3)	1.447(3)
C(15)-C(16)	1.501(4)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900

C(16)-C(18)	1.391(4)
C(16)-C(17)	1.395(4)
C(17)-C(17)#1	1.379(6)
C(17)-H(17)	0.9500
C(18)-C(18)#1	1.381(6)
C(18)-H(18)	0.9500
N(1)-O(4)	1.224(3)
N(1)-O(5)	1.226(3)
N(2)-O(7)	1.193(4)
N(2)-O(6)	1.244(5)

C(6)-C(1)-C(2)	123.1(3)
C(6)-C(1)-O(1)	117.8(2)
C(2)-C(1)-O(1)	118.9(2)
C(3)-C(2)-C(1)	116.4(3)
C(3)-C(2)-H(2)	121.8
C(1)-C(2)-H(2)	121.8
C(2)-C(3)-C(4)	123.2(3)
C(2)-C(3)-O(2)	119.5(3)
C(4)-C(3)-O(2)	117.0(2)
C(5)-C(4)-C(3)	118.3(3)
C(5)-C(4)-H(4)	120.8
C(3)-C(4)-H(4)	120.8
O(3)-C(5)-C(4)	123.2(2)
O(3)-C(5)-C(6)	116.2(2)
C(4)-C(5)-C(6)	120.6(3)
C(1)-C(6)-C(5)	118.4(3)
C(1)-C(6)-H(6)	120.8
C(5)-C(6)-H(6)	120.8
O(1)-C(7)-C(10)	122.2(2)
O(1)-C(7)-C(8)	119.3(2)
C(10)-C(7)-C(8)	118.4(2)
C(9)-C(8)-C(7)	119.6(2)
C(9)-C(8)-N(1)	116.8(2)
C(7)-C(8)-N(1)	123.6(2)
C(8)#1-C(9)-C(8)	121.8(3)

C(8)#1-C(9)-H(9)	119.1
C(8)-C(9)-H(9)	119.1
C(7)-C(10)-C(7)#1	122.3(3)
C(7)-C(10)-H(10)	118.8
C(7)#1-C(10)-H(10)	118.8
O(2)-C(11)-C(14)	122.8(3)
O(2)-C(11)-C(12)	118.0(3)
C(14)-C(11)-C(12)	119.2(3)
C(13)-C(12)-C(11)	119.6(4)
C(13)-C(12)-N(2)	117.2(3)
C(11)-C(12)-N(2)	123.1(4)
C(12)#1-C(13)-C(12)	121.3(4)
C(12)#1-C(13)-H(13)	119.3
C(12)-C(13)-H(13)	119.3
C(11)#1-C(14)-C(11)	121.0(4)
C(11)#1-C(14)-H(14)	119.5
C(11)-C(14)-H(14)	119.5
O(3)-C(15)-C(16)	110.7(2)
O(3)-C(15)-H(15A)	109.5
C(16)-C(15)-H(15A)	109.5
O(3)-C(15)-H(15B)	109.5
C(16)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	108.1
C(18)-C(16)-C(17)	118.6(3)
C(18)-C(16)-C(15)	119.4(3)
C(17)-C(16)-C(15)	121.9(3)
C(17)#1-C(17)-C(16)	120.68(17)
C(17)#1-C(17)-H(17)	119.7
C(16)-C(17)-H(17)	119.7
C(18)#1-C(18)-C(16)	120.72(17)
C(18)#1-C(18)-H(18)	119.6
C(16)-C(18)-H(18)	119.6
C(7)-O(1)-C(1)	119.27(19)
C(11)-O(2)-C(3)	119.2(2)
C(5)-O(3)-C(15)	116.6(2)
O(4)-N(1)-O(5)	123.8(2)

O(4)-N(1)-C(8)	117.4(2)
O(5)-N(1)-C(8)	118.9(2)
O(7)-N(2)-O(6)	121.6(4)
O(7)-N(2)-C(12)	118.8(4)
O(6)-N(2)-C(12)	119.4(3)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. 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}
C(1)	38(2)	20(1)	38(2)	2(1)	1(1)	-2(1)
C(2)	32(2)	32(2)	43(2)	5(1)	5(1)	-3(1)
C(3)	39(2)	40(2)	38(2)	11(1)	12(1)	-6(1)
C(4)	34(2)	43(2)	37(2)	14(1)	4(1)	-2(1)
C(5)	31(2)	32(2)	43(2)	12(1)	7(1)	0(1)
C(6)	40(2)	26(2)	39(2)	1(1)	8(1)	1(1)
C(7)	30(2)	26(2)	33(2)	0(1)	10(1)	2(1)
C(8)	29(2)	28(2)	28(2)	-3(1)	7(1)	-2(1)
C(9)	24(2)	37(2)	26(2)	0	5(2)	0
C(10)	27(2)	30(2)	31(2)	0	2(2)	0
C(11)	25(2)	70(2)	35(2)	8(2)	5(1)	1(2)
C(12)	36(2)	102(3)	33(2)	10(2)	10(2)	-8(2)
C(13)	32(3)	135(6)	30(3)	0	8(2)	0
C(14)	31(2)	68(3)	35(2)	0	13(2)	0
C(15)	35(2)	53(2)	44(2)	9(2)	6(2)	5(2)
C(16)	24(2)	51(2)	36(2)	7(2)	3(1)	3(1)
C(17)	38(2)	64(2)	36(2)	9(1)	6(1)	-3(1)
C(18)	31(2)	47(2)	36(2)	0(1)	10(1)	2(1)
O(1)	45(1)	24(1)	41(1)	-1(1)	-3(1)	5(1)
O(2)	45(1)	63(2)	42(1)	16(1)	15(1)	-5(1)
O(3)	31(1)	45(1)	46(1)	6(1)	7(1)	5(1)
N(1)	34(1)	34(2)	34(1)	-3(1)	10(1)	-3(1)
O(4)	34(1)	42(1)	49(1)	-7(1)	-3(1)	-8(1)
O(5)	47(1)	27(1)	45(1)	-2(1)	11(1)	-3(1)
N(2)	50(2)	125(3)	51(2)	32(2)	19(2)	-2(2)
O(6)	189(4)	115(3)	92(2)	17(2)	80(3)	-36(3)
O(7)	72(2)	187(3)	90(2)	87(2)	48(2)	32(2)

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

	x	y	z	U(eq)
H(2)	2512	897	1957	44
H(4)	6781	234	3411	47
H(6)	6086	222	910	42
H(9)	-243	2500	-1222	35
H(10)	4015	2500	1031	36
H(13)	2388	2500	5311	79
H(14)	4579	2500	3041	53
H(15A)	10371	-67	2870	54
H(15B)	9124	-55	3424	54
H(17)	9175	1587	4119	56
H(18)	9678	1587	1769	45

Table 6. Torsion angles [°] for 2.

C(6)-C(1)-C(2)-C(3)	-0.1(4)
O(1)-C(1)-C(2)-C(3)	175.0(2)
C(1)-C(2)-C(3)-C(4)	0.0(4)
C(1)-C(2)-C(3)-O(2)	-173.5(2)
C(2)-C(3)-C(4)-C(5)	-0.1(4)
O(2)-C(3)-C(4)-C(5)	173.5(2)
C(3)-C(4)-C(5)-O(3)	-176.7(2)
C(3)-C(4)-C(5)-C(6)	0.3(4)
C(2)-C(1)-C(6)-C(5)	0.3(4)
O(1)-C(1)-C(6)-C(5)	-174.8(2)
O(3)-C(5)-C(6)-C(1)	176.8(2)
C(4)-C(5)-C(6)-C(1)	-0.4(4)
O(1)-C(7)-C(8)-C(9)	-176.8(2)
C(10)-C(7)-C(8)-C(9)	-0.1(4)
O(1)-C(7)-C(8)-N(1)	5.8(4)
C(10)-C(7)-C(8)-N(1)	-177.5(3)
C(7)-C(8)-C(9)-C(8)#1	0.2(5)
N(1)-C(8)-C(9)-C(8)#1	177.8(2)
O(1)-C(7)-C(10)-C(7)#1	176.5(2)
C(8)-C(7)-C(10)-C(7)#1	-0.1(5)
O(2)-C(11)-C(12)-C(13)	-177.0(3)
C(14)-C(11)-C(12)-C(13)	1.4(5)
O(2)-C(11)-C(12)-N(2)	-1.1(4)
C(14)-C(11)-C(12)-N(2)	177.3(3)
C(11)-C(12)-C(13)-C(12)#1	-0.5(6)
N(2)-C(12)-C(13)-C(12)#1	-176.6(3)
O(2)-C(11)-C(14)-C(11)#1	176.0(2)
C(12)-C(11)-C(14)-C(11)#1	-2.4(6)
O(3)-C(15)-C(16)-C(18)	-52.5(3)
O(3)-C(15)-C(16)-C(17)	124.4(3)
C(18)-C(16)-C(17)-C(17)#1	0.0(3)
C(15)-C(16)-C(17)-C(17)#1	-176.93(18)
C(17)-C(16)-C(18)-C(18)#1	0.0(3)
C(15)-C(16)-C(18)-C(18)#1	177.01(17)

C(10)-C(7)-O(1)-C(1)	20.0(4)
C(8)-C(7)-O(1)-C(1)	-163.4(2)
C(6)-C(1)-O(1)-C(7)	-115.8(3)
C(2)-C(1)-O(1)-C(7)	68.9(3)
C(14)-C(11)-O(2)-C(3)	-1.1(4)
C(12)-C(11)-O(2)-C(3)	177.3(2)
C(2)-C(3)-O(2)-C(11)	-82.6(3)
C(4)-C(3)-O(2)-C(11)	103.6(3)
C(4)-C(5)-O(3)-C(15)	-32.9(4)
C(6)-C(5)-O(3)-C(15)	150.0(2)
C(16)-C(15)-O(3)-C(5)	-60.4(3)
C(9)-C(8)-N(1)-O(4)	-2.5(3)
C(7)-C(8)-N(1)-O(4)	175.0(2)
C(9)-C(8)-N(1)-O(5)	177.6(2)
C(7)-C(8)-N(1)-O(5)	-4.9(4)
C(13)-C(12)-N(2)-O(7)	14.3(5)
C(11)-C(12)-N(2)-O(7)	-161.6(3)
C(13)-C(12)-N(2)-O(6)	-160.4(4)
C(11)-C(12)-N(2)-O(6)	23.6(5)

Symmetry transformations used to generate equivalent atoms:

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

X-Ray Structure Determination, $C_{38}H_{24}N_4O_{12}$·unknown solvate (host 3)

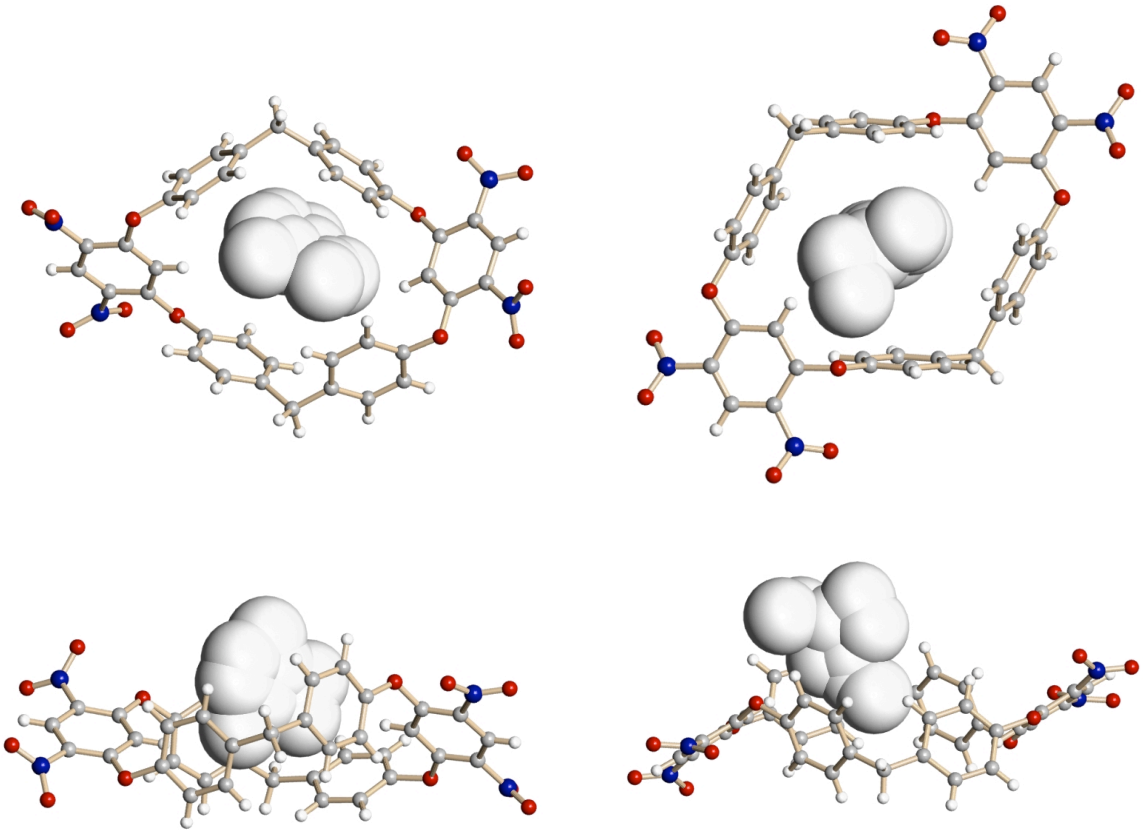
X-ray diffraction intensity data from a colorless blocklike crystal were measured at 150(1) K on a Bruker SMART APEX diffractometer (Mo Kα radiation, λ = 0.71073 Å).[1] Raw area detector data frame integration was performed with SAINT+.[1] Final unit cell parameters were determined by least-squares refinement of 8678 strong reflections from the data set. Direct methods structure solution, difference Fourier calculations and full-matrix least-squares refinement against F^2 were performed with SHELXTL.[2]

The compound crystallizes in the triclinic system. The space group $P\bar{1}$ was assumed and confirmed by the successful solution and refinement of the structure. The asymmetric unit consists of one and a half independent $C_{38}H_{24}N_4O_{12}$ molecules and unidentified solvent species. The half-molecule is located on a crystallographic inversion center. There are two independent but closely separated regions of disordered electron density present in the asymmetric unit. From unsuccessful modeling attempts, each region likely contains acetone molecules disordered over at least three sites. Ultimately no reasonable disorder model could be obtained. The program Squeeze was therefore used to remove the contribution of these diffusely scattering species from the structure factor calculations.[3] The disordered solvent species occupy a volume of 502.8 $Å^3$ per unit cell, or 17.8% of the total cell volume. Note that the final reported FW, F(000), and d_{calc} correspond to crystallographically identifiable species only. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in geometrically idealized positions and included as riding atoms.

(1) SMART Version 5.625, SAINT+ Version 6.45. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 2001.

(2) SHELXTL Version 6.14; Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 2000.

(3) PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, Spek, A. L. 1998.



Location of the unknown solvent species (large white spheres). Molecule 1 (acentric, N1-N4) on left, molecule 2 (centrosymmetric, N5-N6) on right.

Table 1. Crystal data and structure refinement for **3**.

Identification code	**3**	
Empirical formula	C38 H24 N4 O12	
Formula weight	728.61	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 12.1115(4) Å	α= 79.9180(10)°.
	b = 14.0678(4) Å	β= 86.4530(10)°.
	c = 17.4915(5) Å	γ = 74.7700(10)°.
Volume	2830.77(15) $Å^3$	
Z	3	
Density (calculated)	1.282 Mg/m^3	
Absorption coefficient	0.098 mm^{-1}	
F(000)	1128	
Crystal size	0.44 x 0.20 x 0.10 mm^3	
Theta range for data collection	1.18 to 25.03°.	
Index ranges	-14<=h<=14, -16<=k<=16, -20<=l<=20	
Reflections collected	30721	
Independent reflections	10008 [R(int) = 0.0412]	
Completeness to theta = 25.03°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10008 / 0 / 730	
Goodness-of-fit on F^2	0.911	
Final R indices [I>2sigma(I)]	R1 = 0.0377, wR2 = 0.0782	
R indices (all data)	R1 = 0.0530, wR2 = 0.0827	
Largest diff. peak and hole	0.189 and -0.166 $e.Å^{-3}$	

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)
C(1)	10209(1)	-2214(1)	2482(1)	25(1)
C(2)	9949(1)	-1559(1)	3019(1)	26(1)
C(3)	9399(1)	-563(1)	2766(1)	28(1)
C(4)	9076(1)	-231(1)	2001(1)	27(1)
C(5)	9295(1)	-920(1)	1490(1)	28(1)
C(6)	9870(1)	-1898(1)	1721(1)	28(1)
C(7)	9478(1)	-1547(1)	4359(1)	28(1)
C(8)	8341(1)	-1532(1)	4351(1)	36(1)
C(9)	7645(1)	-1244(1)	4967(1)	36(1)
C(10)	8061(1)	-977(1)	5595(1)	30(1)
C(11)	9211(2)	-1002(1)	5583(1)	34(1)
C(12)	9926(1)	-1282(1)	4966(1)	33(1)
C(13)	7270(2)	-666(1)	6267(1)	36(1)
C(14)	6711(1)	442(1)	6162(1)	30(1)
C(15)	5615(1)	842(1)	5869(1)	36(1)
C(16)	5122(1)	1860(1)	5752(1)	36(1)
C(17)	5727(1)	2492(1)	5928(1)	29(1)
C(18)	6810(1)	2123(1)	6231(1)	32(1)
C(19)	7287(1)	1102(1)	6339(1)	33(1)
C(20)	5696(1)	4171(1)	6003(1)	28(1)
C(21)	5374(1)	4533(1)	6697(1)	29(1)
C(22)	5836(1)	5251(1)	6898(1)	31(1)
C(23)	6632(1)	5594(1)	6417(1)	30(1)
C(24)	6981(1)	5240(1)	5719(1)	31(1)
C(25)	6484(1)	4537(1)	5515(1)	30(1)
C(26)	8240(1)	5166(1)	4626(1)	33(1)
C(27)	7894(1)	5722(1)	3917(1)	37(1)
C(28)	8371(1)	5357(1)	3251(1)	35(1)
C(29)	9176(1)	4447(1)	3292(1)	29(1)
C(30)	9510(2)	3907(1)	4018(1)	38(1)
C(31)	9045(2)	4263(1)	4690(1)	40(1)

C(32)	9715(1)	4076(1)	2558(1)	34(1)
C(33)	9407(1)	3161(1)	2379(1)	28(1)
C(34)	10243(1)	2291(1)	2315(1)	31(1)
C(35)	9981(1)	1466(1)	2113(1)	31(1)
C(36)	8861(1)	1512(1)	1989(1)	27(1)
C(37)	8003(1)	2363(1)	2048(1)	30(1)
C(38)	8282(1)	3180(1)	2238(1)	32(1)
C(39)	5968(1)	8584(1)	1869(1)	30(1)
C(40)	6295(1)	8042(1)	1259(1)	29(1)
C(41)	5450(1)	7955(1)	797(1)	31(1)
C(42)	4314(1)	8398(1)	931(1)	30(1)
C(43)	4018(1)	8967(1)	1524(1)	32(1)
C(44)	4845(1)	9065(1)	1988(1)	34(1)
C(45)	7751(1)	6935(1)	643(1)	30(1)
C(46)	7633(1)	5991(1)	917(1)	35(1)
C(47)	7978(1)	5261(1)	453(1)	33(1)
C(48)	8445(1)	5464(1)	-282(1)	28(1)
C(49)	8561(1)	6425(1)	-535(1)	36(1)
C(50)	8214(1)	7171(1)	-80(1)	37(1)
C(51)	8855(1)	4677(1)	-801(1)	33(1)
C(52)	8210(1)	3875(1)	-706(1)	29(1)
C(53)	8441(1)	3065(1)	-108(1)	31(1)
C(54)	7854(1)	2332(1)	-26(1)	30(1)
C(55)	7029(1)	2406(1)	-554(1)	28(1)
C(56)	6781(1)	3192(1)	-1160(1)	35(1)
C(57)	7375(1)	3923(1)	-1229(1)	34(1)
N(1)	10779(1)	-3271(1)	2711(1)	30(1)
N(2)	4549(1)	4185(1)	7239(1)	37(1)
N(3)	7129(1)	6324(1)	6678(1)	43(1)
N(4)	8859(1)	-640(1)	696(1)	39(1)
N(5)	6789(1)	8625(1)	2432(1)	35(1)
N(6)	2835(1)	9493(1)	1669(1)	43(1)
O(1)	10244(1)	-1916(1)	3776(1)	32(1)
O(2)	5185(1)	3511(1)	5761(1)	34(1)
O(3)	7805(1)	5583(1)	5294(1)	42(1)
O(4)	8530(1)	737(1)	1731(1)	34(1)

O(5)	10481(1)	-3875(1)	2399(1)	37(1)
O(6)	11533(1)	-3498(1)	3195(1)	47(1)
O(7)	4419(1)	3351(1)	7246(1)	50(1)
O(8)	4033(1)	4748(1)	7675(1)	59(1)
O(9)	7322(1)	6216(1)	7368(1)	53(1)
O(10)	7302(2)	7000(1)	6202(1)	91(1)
O(11)	7874(1)	-130(1)	610(1)	56(1)
O(12)	9477(1)	-977(1)	177(1)	54(1)
O(13)	7432(1)	7666(1)	1128(1)	35(1)
O(14)	6486(1)	1627(1)	-422(1)	35(1)
O(15)	6604(1)	9392(1)	2714(1)	57(1)
O(16)	7576(1)	7891(1)	2615(1)	42(1)
O(17)	2078(1)	9090(1)	1611(1)	58(1)
O(18)	2678(1)	10323(1)	1855(1)	59(1)

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

C(1)-C(6)	1.378(2)
C(1)-C(2)	1.394(2)
C(1)-N(1)	1.4614(19)
C(2)-O(1)	1.3651(17)
C(2)-C(3)	1.389(2)
C(3)-C(4)	1.383(2)
C(3)-H(3)	0.9500
C(4)-O(4)	1.3613(17)
C(4)-C(5)	1.396(2)
C(5)-C(6)	1.371(2)
C(5)-N(4)	1.468(2)
C(6)-H(6)	0.9500
C(7)-C(12)	1.370(2)
C(7)-C(8)	1.374(2)
C(7)-O(1)	1.4099(17)
C(8)-C(9)	1.377(2)
C(8)-H(8)	0.9500
C(9)-C(10)	1.385(2)
C(9)-H(9)	0.9500
C(10)-C(11)	1.382(2)
C(10)-C(13)	1.521(2)
C(11)-C(12)	1.387(2)
C(11)-H(11)	0.9500
C(12)-H(12)	0.9500
C(13)-C(14)	1.510(2)
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(14)-C(19)	1.382(2)
C(14)-C(15)	1.390(2)
C(15)-C(16)	1.383(2)
C(15)-H(15)	0.9500
C(16)-C(17)	1.374(2)
C(16)-H(16)	0.9500
C(17)-C(18)	1.379(2)

C(17)-O(2)	1.3987(17)
C(18)-C(19)	1.385(2)
C(18)-H(18)	0.9500
C(19)-H(19)	0.9500
C(20)-O(2)	1.3774(18)
C(20)-C(25)	1.381(2)
C(20)-C(21)	1.391(2)
C(21)-C(22)	1.380(2)
C(21)-N(2)	1.454(2)
C(22)-C(23)	1.368(2)
C(22)-H(22)	0.9500
C(23)-C(24)	1.398(2)
C(23)-N(3)	1.465(2)
C(24)-O(3)	1.3483(18)
C(24)-C(25)	1.390(2)
C(25)-H(25)	0.9500
C(26)-C(27)	1.370(2)
C(26)-C(31)	1.375(2)
C(26)-O(3)	1.4101(18)
C(27)-C(28)	1.387(2)
C(27)-H(27)	0.9500
C(28)-C(29)	1.383(2)
C(28)-H(28)	0.9500
C(29)-C(30)	1.384(2)
C(29)-C(32)	1.517(2)
C(30)-C(31)	1.388(2)
C(30)-H(30)	0.9500
C(31)-H(31)	0.9500
C(32)-C(33)	1.516(2)
C(32)-H(32A)	0.9900
C(32)-H(32B)	0.9900
C(33)-C(34)	1.385(2)
C(33)-C(38)	1.392(2)
C(34)-C(35)	1.387(2)
C(34)-H(34)	0.9500
C(35)-C(36)	1.370(2)

C(35)-H(35)	0.9500
C(36)-C(37)	1.379(2)
C(36)-O(4)	1.4072(17)
C(37)-C(38)	1.377(2)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
C(39)-C(44)	1.372(2)
C(39)-C(40)	1.397(2)
C(39)-N(5)	1.461(2)
C(40)-O(13)	1.3600(17)
C(40)-C(41)	1.385(2)
C(41)-C(42)	1.377(2)
C(41)-H(41)	0.9500
C(42)-O(14)#1	1.3674(18)
C(42)-C(43)	1.393(2)
C(43)-C(44)	1.375(2)
C(43)-N(6)	1.459(2)
C(44)-H(44)	0.9500
C(45)-C(46)	1.372(2)
C(45)-C(50)	1.374(2)
C(45)-O(13)	1.4086(17)
C(46)-C(47)	1.384(2)
C(46)-H(46)	0.9500
C(47)-C(48)	1.386(2)
C(47)-H(47)	0.9500
C(48)-C(49)	1.387(2)
C(48)-C(51)	1.517(2)
C(49)-C(50)	1.389(2)
C(49)-H(49)	0.9500
C(50)-H(50)	0.9500
C(51)-C(52)	1.514(2)
C(51)-H(51A)	0.9900
C(51)-H(51B)	0.9900
C(52)-C(57)	1.386(2)
C(52)-C(53)	1.386(2)
C(53)-C(54)	1.383(2)

C(53)-H(53)	0.9500
C(54)-C(55)	1.374(2)
C(54)-H(54)	0.9500
C(55)-C(56)	1.375(2)
C(55)-O(14)	1.3987(18)
C(56)-C(57)	1.387(2)
C(56)-H(56)	0.9500
C(57)-H(57)	0.9500
N(1)-O(5)	1.2240(15)
N(1)-O(6)	1.2254(16)
N(2)-O(8)	1.2216(17)
N(2)-O(7)	1.2224(17)
N(3)-O(10)	1.2035(18)
N(3)-O(9)	1.2204(18)
N(4)-O(12)	1.2206(17)
N(4)-O(11)	1.2223(17)
N(5)-O(16)	1.2175(16)
N(5)-O(15)	1.2257(16)
N(6)-O(17)	1.2159(18)
N(6)-O(18)	1.2306(18)
O(14)-C(42)#1	1.3674(18)

C(6)-C(1)-C(2)	120.90(14)
C(6)-C(1)-N(1)	117.34(13)
C(2)-C(1)-N(1)	121.62(13)
O(1)-C(2)-C(3)	122.09(13)
O(1)-C(2)-C(1)	118.99(13)
C(3)-C(2)-C(1)	118.93(14)
C(4)-C(3)-C(2)	120.77(14)
C(4)-C(3)-H(3)	119.6
C(2)-C(3)-H(3)	119.6
O(4)-C(4)-C(3)	122.69(13)
O(4)-C(4)-C(5)	118.68(14)
C(3)-C(4)-C(5)	118.61(14)
C(6)-C(5)-C(4)	121.47(14)
C(6)-C(5)-N(4)	117.17(13)

C(4)-C(5)-N(4)	121.25(14)
C(5)-C(6)-C(1)	119.17(14)
C(5)-C(6)-H(6)	120.4
C(1)-C(6)-H(6)	120.4
C(12)-C(7)-C(8)	121.19(15)
C(12)-C(7)-O(1)	117.61(14)
C(8)-C(7)-O(1)	120.99(14)
C(7)-C(8)-C(9)	118.92(15)
C(7)-C(8)-H(8)	120.5
C(9)-C(8)-H(8)	120.5
C(8)-C(9)-C(10)	121.90(16)
C(8)-C(9)-H(9)	119.0
C(10)-C(9)-H(9)	119.0
C(11)-C(10)-C(9)	117.52(15)
C(11)-C(10)-C(13)	121.91(14)
C(9)-C(10)-C(13)	120.56(15)
C(10)-C(11)-C(12)	121.57(15)
C(10)-C(11)-H(11)	119.2
C(12)-C(11)-H(11)	119.2
C(7)-C(12)-C(11)	118.89(15)
C(7)-C(12)-H(12)	120.6
C(11)-C(12)-H(12)	120.6
C(14)-C(13)-C(10)	113.40(13)
C(14)-C(13)-H(13A)	108.9
C(10)-C(13)-H(13A)	108.9
C(14)-C(13)-H(13B)	108.9
C(10)-C(13)-H(13B)	108.9
H(13A)-C(13)-H(13B)	107.7
C(19)-C(14)-C(15)	117.28(14)
C(19)-C(14)-C(13)	120.87(15)
C(15)-C(14)-C(13)	121.83(14)
C(16)-C(15)-C(14)	121.50(15)
C(16)-C(15)-H(15)	119.3
C(14)-C(15)-H(15)	119.3
C(17)-C(16)-C(15)	119.41(15)
C(17)-C(16)-H(16)	120.3

C(15)-C(16)-H(16)	120.3
C(16)-C(17)-C(18)	120.91(15)
C(16)-C(17)-O(2)	115.65(14)
C(18)-C(17)-O(2)	123.42(14)
C(17)-C(18)-C(19)	118.55(15)
C(17)-C(18)-H(18)	120.7
C(19)-C(18)-H(18)	120.7
C(14)-C(19)-C(18)	122.34(15)
C(14)-C(19)-H(19)	118.8
C(18)-C(19)-H(19)	118.8
O(2)-C(20)-C(25)	118.71(14)
O(2)-C(20)-C(21)	121.62(14)
C(25)-C(20)-C(21)	119.49(14)
C(22)-C(21)-C(20)	120.50(14)
C(22)-C(21)-N(2)	116.89(14)
C(20)-C(21)-N(2)	122.62(14)
C(23)-C(22)-C(21)	119.48(14)
C(23)-C(22)-H(22)	120.3
C(21)-C(22)-H(22)	120.3
C(22)-C(23)-C(24)	121.46(15)
C(22)-C(23)-N(3)	117.00(14)
C(24)-C(23)-N(3)	121.51(14)
O(3)-C(24)-C(25)	123.86(14)
O(3)-C(24)-C(23)	117.85(14)
C(25)-C(24)-C(23)	118.27(14)
C(20)-C(25)-C(24)	120.77(14)
C(20)-C(25)-H(25)	119.6
C(24)-C(25)-H(25)	119.6
C(27)-C(26)-C(31)	121.64(15)
C(27)-C(26)-O(3)	117.73(15)
C(31)-C(26)-O(3)	120.47(15)
C(26)-C(27)-C(28)	118.74(16)
C(26)-C(27)-H(27)	120.6
C(28)-C(27)-H(27)	120.6
C(29)-C(28)-C(27)	121.34(16)
C(29)-C(28)-H(28)	119.3

C(27)-C(28)-H(28)	119.3
C(28)-C(29)-C(30)	118.39(15)
C(28)-C(29)-C(32)	120.58(14)
C(30)-C(29)-C(32)	120.97(15)
C(29)-C(30)-C(31)	121.06(16)
C(29)-C(30)-H(30)	119.5
C(31)-C(30)-H(30)	119.5
C(26)-C(31)-C(30)	118.82(16)
C(26)-C(31)-H(31)	120.6
C(30)-C(31)-H(31)	120.6
C(33)-C(32)-C(29)	115.65(13)
C(33)-C(32)-H(32A)	108.4
C(29)-C(32)-H(32A)	108.4
C(33)-C(32)-H(32B)	108.4
C(29)-C(32)-H(32B)	108.4
H(32A)-C(32)-H(32B)	107.4
C(34)-C(33)-C(38)	117.59(14)
C(34)-C(33)-C(32)	121.06(14)
C(38)-C(33)-C(32)	121.28(14)
C(33)-C(34)-C(35)	121.72(15)
C(33)-C(34)-H(34)	119.1
C(35)-C(34)-H(34)	119.1
C(36)-C(35)-C(34)	118.76(15)
C(36)-C(35)-H(35)	120.6
C(34)-C(35)-H(35)	120.6
C(35)-C(36)-C(37)	121.32(14)
C(35)-C(36)-O(4)	122.41(14)
C(37)-C(36)-O(4)	116.04(13)
C(38)-C(37)-C(36)	119.05(15)
C(38)-C(37)-H(37)	120.5
C(36)-C(37)-H(37)	120.5
C(37)-C(38)-C(33)	121.54(15)
C(37)-C(38)-H(38)	119.2
C(33)-C(38)-H(38)	119.2
C(44)-C(39)-C(40)	121.22(14)
C(44)-C(39)-N(5)	116.80(13)

C(40)-C(39)-N(5)	121.91(14)
O(13)-C(40)-C(41)	123.36(14)
O(13)-C(40)-C(39)	118.09(14)
C(41)-C(40)-C(39)	118.48(14)
C(42)-C(41)-C(40)	120.85(14)
C(42)-C(41)-H(41)	119.6
C(40)-C(41)-H(41)	119.6
O(14)#1-C(42)-C(41)	119.23(14)
O(14)#1-C(42)-C(43)	120.92(14)
C(41)-C(42)-C(43)	119.37(14)
C(44)-C(43)-C(42)	120.61(14)
C(44)-C(43)-N(6)	117.59(14)
C(42)-C(43)-N(6)	121.79(14)
C(39)-C(44)-C(43)	119.36(14)
C(39)-C(44)-H(44)	120.3
C(43)-C(44)-H(44)	120.3
C(46)-C(45)-C(50)	121.58(14)
C(46)-C(45)-O(13)	119.01(14)
C(50)-C(45)-O(13)	119.36(14)
C(45)-C(46)-C(47)	119.32(15)
C(45)-C(46)-H(46)	120.3
C(47)-C(46)-H(46)	120.3
C(46)-C(47)-C(48)	121.21(15)
C(46)-C(47)-H(47)	119.4
C(48)-C(47)-H(47)	119.4
C(47)-C(48)-C(49)	117.70(14)
C(47)-C(48)-C(51)	122.65(14)
C(49)-C(48)-C(51)	119.63(14)
C(48)-C(49)-C(50)	122.04(15)
C(48)-C(49)-H(49)	119.0
C(50)-C(49)-H(49)	119.0
C(45)-C(50)-C(49)	118.15(15)
C(45)-C(50)-H(50)	120.9
C(49)-C(50)-H(50)	120.9
C(52)-C(51)-C(48)	115.46(13)
C(52)-C(51)-H(51A)	108.4

C(48)-C(51)-H(51A)	108.4
C(52)-C(51)-H(51B)	108.4
C(48)-C(51)-H(51B)	108.4
H(51A)-C(51)-H(51B)	107.5
C(57)-C(52)-C(53)	117.76(14)
C(57)-C(52)-C(51)	120.52(14)
C(53)-C(52)-C(51)	121.71(14)
C(54)-C(53)-C(52)	121.54(14)
C(54)-C(53)-H(53)	119.2
C(52)-C(53)-H(53)	119.2
C(55)-C(54)-C(53)	119.08(15)
C(55)-C(54)-H(54)	120.5
C(53)-C(54)-H(54)	120.5
C(54)-C(55)-C(56)	121.21(15)
C(54)-C(55)-O(14)	114.72(13)
C(56)-C(55)-O(14)	124.07(14)
C(55)-C(56)-C(57)	118.79(15)
C(55)-C(56)-H(56)	120.6
C(57)-C(56)-H(56)	120.6
C(52)-C(57)-C(56)	121.62(15)
C(52)-C(57)-H(57)	119.2
C(56)-C(57)-H(57)	119.2
O(5)-N(1)-O(6)	124.02(13)
O(5)-N(1)-C(1)	117.81(13)
O(6)-N(1)-C(1)	118.17(13)
O(8)-N(2)-O(7)	123.48(14)
O(8)-N(2)-C(21)	117.35(14)
O(7)-N(2)-C(21)	119.15(14)
O(10)-N(3)-O(9)	124.09(16)
O(10)-N(3)-C(23)	118.14(16)
O(9)-N(3)-C(23)	117.76(14)
O(12)-N(4)-O(11)	124.61(15)
O(12)-N(4)-C(5)	117.99(15)
O(11)-N(4)-C(5)	117.27(14)
O(16)-N(5)-O(15)	124.40(14)
O(16)-N(5)-C(39)	118.63(13)

O(15)-N(5)-C(39)	116.90(14)
O(17)-N(6)-O(18)	124.38(15)
O(17)-N(6)-C(43)	118.97(15)
O(18)-N(6)-C(43)	116.64(14)
C(2)-O(1)-C(7)	118.12(11)
C(20)-O(2)-C(17)	117.98(11)
C(24)-O(3)-C(26)	118.48(12)
C(4)-O(4)-C(36)	119.56(11)
C(40)-O(13)-C(45)	117.57(12)
C(42)#1-O(14)-C(55)	119.69(12)

Symmetry transformations used to generate equivalent atoms:

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

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	23(1)	21(1)	31(1)	-6(1)	2(1)	-6(1)
C(2)	25(1)	29(1)	26(1)	-4(1)	0(1)	-9(1)
C(3)	31(1)	25(1)	30(1)	-10(1)	0(1)	-6(1)
C(4)	28(1)	22(1)	32(1)	-4(1)	-3(1)	-5(1)
C(5)	32(1)	27(1)	25(1)	-4(1)	-3(1)	-10(1)
C(6)	29(1)	27(1)	29(1)	-10(1)	4(1)	-9(1)
C(7)	34(1)	21(1)	26(1)	-5(1)	0(1)	-3(1)
C(8)	40(1)	42(1)	29(1)	-12(1)	-4(1)	-12(1)
C(9)	33(1)	40(1)	35(1)	-9(1)	0(1)	-10(1)
C(10)	40(1)	19(1)	29(1)	-4(1)	2(1)	-7(1)
C(11)	44(1)	32(1)	29(1)	-10(1)	-6(1)	-11(1)
C(12)	33(1)	33(1)	34(1)	-9(1)	-3(1)	-8(1)
C(13)	47(1)	30(1)	32(1)	-8(1)	4(1)	-11(1)
C(14)	37(1)	28(1)	26(1)	-8(1)	6(1)	-9(1)
C(15)	39(1)	32(1)	44(1)	-14(1)	2(1)	-16(1)
C(16)	29(1)	38(1)	43(1)	-13(1)	-2(1)	-9(1)
C(17)	30(1)	26(1)	30(1)	-7(1)	4(1)	-7(1)
C(18)	31(1)	28(1)	40(1)	-10(1)	-2(1)	-10(1)
C(19)	28(1)	33(1)	37(1)	-9(1)	-2(1)	-5(1)
C(20)	27(1)	22(1)	32(1)	-4(1)	-4(1)	-2(1)
C(21)	25(1)	24(1)	33(1)	-1(1)	2(1)	-2(1)
C(22)	34(1)	27(1)	27(1)	-5(1)	3(1)	-1(1)
C(23)	36(1)	20(1)	32(1)	-6(1)	3(1)	-5(1)
C(24)	35(1)	23(1)	32(1)	-3(1)	6(1)	-6(1)
C(25)	34(1)	27(1)	28(1)	-6(1)	3(1)	-5(1)
C(26)	39(1)	35(1)	32(1)	-13(1)	11(1)	-19(1)
C(27)	36(1)	32(1)	42(1)	-11(1)	4(1)	-6(1)
C(28)	39(1)	35(1)	32(1)	-6(1)	0(1)	-11(1)
C(29)	33(1)	28(1)	32(1)	-7(1)	5(1)	-16(1)
C(30)	44(1)	26(1)	40(1)	-6(1)	5(1)	-6(1)
C(31)	55(1)	33(1)	30(1)	-2(1)	3(1)	-14(1)

C(32)	36(1)	35(1)	34(1)	-10(1)	7(1)	-14(1)
C(33)	33(1)	28(1)	21(1)	-5(1)	6(1)	-9(1)
C(34)	27(1)	32(1)	32(1)	-1(1)	0(1)	-8(1)
C(35)	30(1)	23(1)	37(1)	-5(1)	2(1)	0(1)
C(36)	35(1)	23(1)	23(1)	-5(1)	0(1)	-6(1)
C(37)	29(1)	29(1)	33(1)	-7(1)	-1(1)	-7(1)
C(38)	32(1)	26(1)	35(1)	-9(1)	5(1)	-3(1)
C(39)	28(1)	33(1)	32(1)	-12(1)	0(1)	-11(1)
C(40)	26(1)	27(1)	34(1)	-7(1)	3(1)	-7(1)
C(41)	34(1)	30(1)	32(1)	-11(1)	1(1)	-9(1)
C(42)	30(1)	31(1)	31(1)	-4(1)	-5(1)	-11(1)
C(43)	23(1)	36(1)	37(1)	-8(1)	3(1)	-5(1)
C(44)	30(1)	38(1)	34(1)	-15(1)	4(1)	-7(1)
C(45)	27(1)	34(1)	32(1)	-16(1)	2(1)	-7(1)
C(46)	37(1)	41(1)	26(1)	-9(1)	8(1)	-12(1)
C(47)	40(1)	30(1)	31(1)	-7(1)	5(1)	-13(1)
C(48)	26(1)	32(1)	27(1)	-9(1)	2(1)	-9(1)
C(49)	43(1)	39(1)	29(1)	-9(1)	10(1)	-15(1)
C(50)	44(1)	30(1)	41(1)	-8(1)	5(1)	-14(1)
C(51)	34(1)	37(1)	31(1)	-12(1)	9(1)	-13(1)
C(52)	27(1)	32(1)	29(1)	-15(1)	6(1)	-6(1)
C(53)	29(1)	34(1)	32(1)	-11(1)	-5(1)	-5(1)
C(54)	34(1)	28(1)	29(1)	-6(1)	-6(1)	-4(1)
C(55)	26(1)	29(1)	31(1)	-8(1)	0(1)	-7(1)
C(56)	34(1)	41(1)	32(1)	-3(1)	-11(1)	-12(1)
C(57)	38(1)	32(1)	31(1)	-3(1)	-2(1)	-8(1)
N(1)	33(1)	27(1)	30(1)	-8(1)	3(1)	-6(1)
N(2)	31(1)	37(1)	38(1)	-3(1)	4(1)	-7(1)
N(3)	55(1)	34(1)	45(1)	-17(1)	21(1)	-16(1)
N(4)	54(1)	26(1)	35(1)	-7(1)	-11(1)	-5(1)
N(5)	27(1)	43(1)	37(1)	-17(1)	1(1)	-8(1)
N(6)	29(1)	54(1)	46(1)	-17(1)	0(1)	-6(1)
O(1)	35(1)	31(1)	26(1)	-7(1)	-2(1)	1(1)
O(2)	29(1)	27(1)	44(1)	-6(1)	-4(1)	-6(1)
O(3)	55(1)	41(1)	39(1)	-18(1)	20(1)	-27(1)
O(4)	43(1)	21(1)	36(1)	-7(1)	-13(1)	-3(1)

O(5)	50(1)	26(1)	41(1)	-12(1)	2(1)	-13(1)
O(6)	50(1)	33(1)	52(1)	-12(1)	-21(1)	6(1)
O(7)	54(1)	37(1)	58(1)	-2(1)	14(1)	-18(1)
O(8)	59(1)	63(1)	61(1)	-26(1)	34(1)	-23(1)
O(9)	54(1)	62(1)	53(1)	-25(1)	0(1)	-21(1)
O(10)	183(2)	56(1)	60(1)	-25(1)	45(1)	-75(1)
O(11)	63(1)	42(1)	57(1)	-14(1)	-30(1)	8(1)
O(12)	76(1)	53(1)	33(1)	-15(1)	0(1)	-9(1)
O(13)	25(1)	41(1)	46(1)	-25(1)	4(1)	-8(1)
O(14)	36(1)	35(1)	36(1)	0(1)	-11(1)	-14(1)
O(15)	47(1)	62(1)	71(1)	-45(1)	-8(1)	-6(1)
O(16)	32(1)	48(1)	44(1)	-13(1)	-5(1)	-5(1)
O(17)	30(1)	72(1)	79(1)	-27(1)	4(1)	-17(1)
O(18)	38(1)	61(1)	81(1)	-40(1)	0(1)	2(1)

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

	x	y	z	U(eq)
H(3)	9243	-106	3124	34
H(6)	10032	-2353	1362	33
H(8)	8039	-1718	3927	43
H(9)	6856	-1227	4960	43
H(11)	9518	-822	6008	41
H(12)	10713	-1290	4965	40
H(13A)	6666	-1031	6327	43
H(13B)	7714	-862	6752	43
H(15)	5194	407	5748	43
H(16)	4372	2120	5551	43
H(18)	7220	2561	6362	38
H(19)	8036	845	6542	39
H(22)	5603	5506	7367	37
H(25)	6688	4304	5035	36
H(27)	7338	6345	3882	44
H(28)	8141	5739	2757	42
H(30)	10065	3283	4056	45
H(31)	9280	3889	5186	48
H(32A)	9488	4624	2113	41
H(32B)	10557	3921	2599	41
H(34)	11017	2259	2412	37
H(35)	10568	880	2062	38
H(37)	7230	2386	1959	36
H(38)	7694	3770	2273	38
H(41)	5657	7584	382	37
H(44)	4640	9461	2387	40
H(46)	7318	5840	1421	41
H(47)	7894	4607	641	39
H(49)	8888	6578	-1034	43
H(50)	8295	7826	-264	45

H(51A)	8801	5016	-1348	39
H(51B)	9673	4351	-696	39
H(53)	9017	3012	253	37
H(54)	8017	1785	390	37
H(56)	6214	3234	-1524	42
H(57)	7205	4470	-1644	41

Table 6. Torsion angles [°] for **3**.

C(6)-C(1)-C(2)-O(1)	176.33(13)
N(1)-C(1)-C(2)-O(1)	0.6(2)
C(6)-C(1)-C(2)-C(3)	-3.9(2)
N(1)-C(1)-C(2)-C(3)	-179.56(13)
O(1)-C(2)-C(3)-C(4)	-177.85(14)
C(1)-C(2)-C(3)-C(4)	2.4(2)
C(2)-C(3)-C(4)-O(4)	179.58(14)
C(2)-C(3)-C(4)-C(5)	1.2(2)
O(4)-C(4)-C(5)-C(6)	178.17(14)
C(3)-C(4)-C(5)-C(6)	-3.4(2)
O(4)-C(4)-C(5)-N(4)	-5.6(2)
C(3)-C(4)-C(5)-N(4)	172.89(14)
C(4)-C(5)-C(6)-C(1)	1.9(2)
N(4)-C(5)-C(6)-C(1)	-174.50(14)
C(2)-C(1)-C(6)-C(5)	1.8(2)
N(1)-C(1)-C(6)-C(5)	177.64(13)
C(12)-C(7)-C(8)-C(9)	0.1(2)
O(1)-C(7)-C(8)-C(9)	174.62(14)
C(7)-C(8)-C(9)-C(10)	-0.6(2)
C(8)-C(9)-C(10)-C(11)	0.5(2)
C(8)-C(9)-C(10)-C(13)	-179.78(15)
C(9)-C(10)-C(11)-C(12)	0.0(2)
C(13)-C(10)-C(11)-C(12)	-179.67(14)
C(8)-C(7)-C(12)-C(11)	0.5(2)
O(1)-C(7)-C(12)-C(11)	-174.27(13)
C(10)-C(11)-C(12)-C(7)	-0.5(2)
C(11)-C(10)-C(13)-C(14)	89.92(19)
C(9)-C(10)-C(13)-C(14)	-89.75(18)
C(10)-C(13)-C(14)-C(19)	-81.97(19)
C(10)-C(13)-C(14)-C(15)	96.69(18)
C(19)-C(14)-C(15)-C(16)	0.7(2)
C(13)-C(14)-C(15)-C(16)	-177.98(15)
C(14)-C(15)-C(16)-C(17)	-0.1(2)
C(15)-C(16)-C(17)-C(18)	-1.0(2)

C(15)-C(16)-C(17)-O(2)	177.41(14)
C(16)-C(17)-C(18)-C(19)	1.4(2)
O(2)-C(17)-C(18)-C(19)	-176.90(14)
C(15)-C(14)-C(19)-C(18)	-0.3(2)
C(13)-C(14)-C(19)-C(18)	178.39(14)
C(17)-C(18)-C(19)-C(14)	-0.7(2)
O(2)-C(20)-C(21)-C(22)	-174.96(13)
C(25)-C(20)-C(21)-C(22)	0.0(2)
O(2)-C(20)-C(21)-N(2)	4.9(2)
C(25)-C(20)-C(21)-N(2)	179.90(14)
C(20)-C(21)-C(22)-C(23)	-1.2(2)
N(2)-C(21)-C(22)-C(23)	178.88(13)
C(21)-C(22)-C(23)-C(24)	0.7(2)
C(21)-C(22)-C(23)-N(3)	-177.33(14)
C(22)-C(23)-C(24)-O(3)	-177.19(14)
N(3)-C(23)-C(24)-O(3)	0.7(2)
C(22)-C(23)-C(24)-C(25)	1.1(2)
N(3)-C(23)-C(24)-C(25)	179.01(14)
O(2)-C(20)-C(25)-C(24)	176.92(13)
C(21)-C(20)-C(25)-C(24)	1.8(2)
O(3)-C(24)-C(25)-C(20)	175.85(14)
C(23)-C(24)-C(25)-C(20)	-2.3(2)
C(31)-C(26)-C(27)-C(28)	0.1(2)
O(3)-C(26)-C(27)-C(28)	175.59(14)
C(26)-C(27)-C(28)-C(29)	0.5(2)
C(27)-C(28)-C(29)-C(30)	-0.7(2)
C(27)-C(28)-C(29)-C(32)	-178.09(14)
C(28)-C(29)-C(30)-C(31)	0.4(2)
C(32)-C(29)-C(30)-C(31)	177.77(15)
C(27)-C(26)-C(31)-C(30)	-0.4(2)
O(3)-C(26)-C(31)-C(30)	-175.76(14)
C(29)-C(30)-C(31)-C(26)	0.1(3)
C(28)-C(29)-C(32)-C(33)	-112.67(17)
C(30)-C(29)-C(32)-C(33)	70.0(2)
C(29)-C(32)-C(33)-C(34)	-120.64(16)
C(29)-C(32)-C(33)-C(38)	62.4(2)

C(38)-C(33)-C(34)-C(35)	0.4(2)
C(32)-C(33)-C(34)-C(35)	-176.71(14)
C(33)-C(34)-C(35)-C(36)	-1.3(2)
C(34)-C(35)-C(36)-C(37)	1.2(2)
C(34)-C(35)-C(36)-O(4)	175.43(13)
C(35)-C(36)-C(37)-C(38)	-0.2(2)
O(4)-C(36)-C(37)-C(38)	-174.84(13)
C(36)-C(37)-C(38)-C(33)	-0.7(2)
C(34)-C(33)-C(38)-C(37)	0.6(2)
C(32)-C(33)-C(38)-C(37)	177.67(14)
C(44)-C(39)-C(40)-O(13)	173.74(14)
N(5)-C(39)-C(40)-O(13)	-9.6(2)
C(44)-C(39)-C(40)-C(41)	-3.2(2)
N(5)-C(39)-C(40)-C(41)	173.44(14)
O(13)-C(40)-C(41)-C(42)	-176.34(14)
C(39)-C(40)-C(41)-C(42)	0.5(2)
C(40)-C(41)-C(42)-O(14)#1	174.16(14)
C(40)-C(41)-C(42)-C(43)	2.0(2)
O(14)#1-C(42)-C(43)-C(44)	-173.84(14)
C(41)-C(42)-C(43)-C(44)	-1.8(2)
O(14)#1-C(42)-C(43)-N(6)	5.0(2)
C(41)-C(42)-C(43)-N(6)	177.02(15)
C(40)-C(39)-C(44)-C(43)	3.4(2)
N(5)-C(39)-C(44)-C(43)	-173.39(15)
C(42)-C(43)-C(44)-C(39)	-0.9(2)
N(6)-C(43)-C(44)-C(39)	-179.77(15)
C(50)-C(45)-C(46)-C(47)	-0.7(2)
O(13)-C(45)-C(46)-C(47)	-177.99(14)
C(45)-C(46)-C(47)-C(48)	0.3(2)
C(46)-C(47)-C(48)-C(49)	0.4(2)
C(46)-C(47)-C(48)-C(51)	178.71(15)
C(47)-C(48)-C(49)-C(50)	-0.8(2)
C(51)-C(48)-C(49)-C(50)	-179.16(15)
C(46)-C(45)-C(50)-C(49)	0.3(2)
O(13)-C(45)-C(50)-C(49)	177.59(14)
C(48)-C(49)-C(50)-C(45)	0.5(3)

C(47)-C(48)-C(51)-C(52)	31.5(2)
C(49)-C(48)-C(51)-C(52)	-150.24(15)
C(48)-C(51)-C(52)-C(57)	100.25(17)
C(48)-C(51)-C(52)-C(53)	-80.82(19)
C(57)-C(52)-C(53)-C(54)	-0.8(2)
C(51)-C(52)-C(53)-C(54)	-179.74(14)
C(52)-C(53)-C(54)-C(55)	0.6(2)
C(53)-C(54)-C(55)-C(56)	0.0(2)
C(53)-C(54)-C(55)-O(14)	179.79(13)
C(54)-C(55)-C(56)-C(57)	-0.5(2)
O(14)-C(55)-C(56)-C(57)	179.77(14)
C(53)-C(52)-C(57)-C(56)	0.3(2)
C(51)-C(52)-C(57)-C(56)	179.27(14)
C(55)-C(56)-C(57)-C(52)	0.3(2)
C(6)-C(1)-N(1)-O(5)	-33.43(19)
C(2)-C(1)-N(1)-O(5)	142.41(14)
C(6)-C(1)-N(1)-O(6)	145.76(14)
C(2)-C(1)-N(1)-O(6)	-38.4(2)
C(22)-C(21)-N(2)-O(8)	24.3(2)
C(20)-C(21)-N(2)-O(8)	-155.53(15)
C(22)-C(21)-N(2)-O(7)	-154.47(14)
C(20)-C(21)-N(2)-O(7)	25.7(2)
C(22)-C(23)-N(3)-O(10)	-140.99(17)
C(24)-C(23)-N(3)-O(10)	41.0(2)
C(22)-C(23)-N(3)-O(9)	37.7(2)
C(24)-C(23)-N(3)-O(9)	-140.32(16)
C(6)-C(5)-N(4)-O(12)	-42.4(2)
C(4)-C(5)-N(4)-O(12)	141.23(15)
C(6)-C(5)-N(4)-O(11)	133.67(15)
C(4)-C(5)-N(4)-O(11)	-42.7(2)
C(44)-C(39)-N(5)-O(16)	142.81(15)
C(40)-C(39)-N(5)-O(16)	-34.0(2)
C(44)-C(39)-N(5)-O(15)	-34.1(2)
C(40)-C(39)-N(5)-O(15)	149.09(16)
C(44)-C(43)-N(6)-O(17)	-141.97(16)
C(42)-C(43)-N(6)-O(17)	39.2(2)

C(44)-C(43)-N(6)-O(18)	37.1(2)
C(42)-C(43)-N(6)-O(18)	-141.81(17)
C(3)-C(2)-O(1)-C(7)	38.50(19)
C(1)-C(2)-O(1)-C(7)	-141.71(14)
C(12)-C(7)-O(1)-C(2)	-134.74(14)
C(8)-C(7)-O(1)-C(2)	50.53(19)
C(25)-C(20)-O(2)-C(17)	88.94(17)
C(21)-C(20)-O(2)-C(17)	-96.02(17)
C(16)-C(17)-O(2)-C(20)	172.99(13)
C(18)-C(17)-O(2)-C(20)	-8.7(2)
C(25)-C(24)-O(3)-C(26)	-3.9(2)
C(23)-C(24)-O(3)-C(26)	174.30(14)
C(27)-C(26)-O(3)-C(24)	104.51(17)
C(31)-C(26)-O(3)-C(24)	-80.0(2)
C(3)-C(4)-O(4)-C(36)	39.7(2)
C(5)-C(4)-O(4)-C(36)	-141.88(14)
C(35)-C(36)-O(4)-C(4)	38.2(2)
C(37)-C(36)-O(4)-C(4)	-147.27(14)
C(41)-C(40)-O(13)-C(45)	-17.7(2)
C(39)-C(40)-O(13)-C(45)	165.46(14)
C(46)-C(45)-O(13)-C(40)	-72.66(19)
C(50)-C(45)-O(13)-C(40)	109.99(17)
C(54)-C(55)-O(14)-C(42)#1	-177.20(14)
C(56)-C(55)-O(14)-C(42)#1	2.5(2)

Symmetry transformations used to generate equivalent atoms:

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