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The pseudosymmetric crystal structure of 2,4,6,8,10,12-hexanitrohexaazaisowurtzitane dimethyl carbonate (2/3), $C_{21}H_{30}N_{24}O_{33}$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.50 \times 0.46 \times 0.23$ mm
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.17 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	28.3° , $>99\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	31127, 10583, 0.034
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 8856
$N(\text{param})_{\text{refined}}$:	709
Programs:	Bruker [1, 2], SHELX [3]

Abstract

$C_{21}H_{30}N_{24}O_{33}$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 12.6527(16) \text{ \AA}$, $b = 16.395(2) \text{ \AA}$, $c = 20.606(3) \text{ \AA}$, $V = 4274.5(9) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.0367$, $wR_{\text{ref}}(F^2) = 0.0907$, $T = 200(2) \text{ K}$.

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The asymmetric unit of the title crystal structure is shown in the figure (The central carbon atoms of dimethylcarbonate and the atoms in the wurtzite core are labelled). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

2,4,6,8,10,12-hexanitrohexaazaisowurtzitane CL20 is easily available by a literature known synthesis [4]. In a typical reaction 0.1 g CL20 was dissolved in 5 mL concentrated dimethyl carbonate. Evaporation at room temperature yielded colourless crystals of $(dpma)_2 2(\text{CH}_3\text{OCOOCH}_3)_3$.

Experimental details

The methyl groups were idealized and refined using rigid groups allowed to rotate about N–C bond (APEX2 option of the SHELXL-2014 program). The coordinates of the H-atoms were refined with the C–H distance restrained to $0.98 \text{ \AA}$. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{\text{eq}}(\text{C})$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{\text{eq}}(\text{C}, \text{N})$. The structural fit with centrosymmetric space group $Pbca$ is 87%. As the structure can be refined

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.67513(17)	0.11226(14)	−0.04947(11)	0.0176(5)
H1	0.6170	0.1213	−0.0816	0.021*
C2	0.71257(18)	0.10781(14)	0.06793(11)	0.0180(5)
H2	0.6778	0.1127	0.1113	0.022*
C3	0.88556(19)	0.12269(14)	0.02720(11)	0.0186(5)
H3	0.9569	0.1456	0.0373	0.022*
C4	0.86085(18)	0.12678(15)	−0.04829(11)	0.0186(5)
H4	0.9209	0.1521	−0.0726	0.022*
C5	0.72426(18)	0.02451(14)	−0.05742(11)	0.0163(5)
H5	0.6887	−0.0067	−0.0929	0.020*
C6	0.76366(18)	0.02093(14)	0.05954(11)	0.0165(5)
H6	0.7534	−0.0129	0.0995	0.020*
C7	0.16906(18)	0.88948(14)	−0.04092(11)	0.0176(5)
H7	0.1095	0.8825	−0.0723	0.021*
C8	0.35371(18)	0.86825(14)	−0.04170(11)	0.0184(5)
H8	0.4118	0.8408	−0.0663	0.022*
C9	0.37974(19)	0.87187(15)	0.03383(11)	0.0194(5)
H9	0.4498	0.8466	0.0439	0.023*
C10	0.20928(18)	0.89493(14)	0.07624(11)	0.0183(5)
H10	0.1757	0.8921	0.1201	0.022*
C11	0.26522(18)	0.97982(14)	0.06586(11)	0.0165(5)
H11	0.2591	1.0149	0.1054	0.020*
C12	0.22264(18)	0.97472(14)	−0.05036(11)	0.0163(5)
H12	0.1879	1.0062	−0.0860	0.020*
C13	0.4492(3)	0.6330(2)	0.24227(18)	0.0594(10)
H13A	0.3803	0.6586	0.2346	0.089*
H13B	0.5022	0.6580	0.2138	0.089*
H13C	0.4699	0.6410	0.2877	0.089*
C14	0.3681(2)	0.50758(18)	0.26046(13)	0.0377(7)
C15	0.3010(3)	0.37583(19)	0.27823(15)	0.0499(9)
H15A	0.3177	0.3781	0.3246	0.075*
H15B	0.3095	0.3198	0.2626	0.075*
H15C	0.2278	0.3934	0.2714	0.075*
C16	0.7686(3)	0.60670(18)	0.27272(14)	0.0407(7)
H16A	0.7123	0.6168	0.3044	0.061*
H16B	0.7924	0.6587	0.2543	0.061*
H16C	0.8280	0.5796	0.2943	0.061*
C17	0.6951(2)	0.48224(17)	0.24002(12)	0.0298(6)
C18	0.6158(3)	0.36378(18)	0.19977(15)	0.0418(7)
H18A	0.6671	0.3289	0.2222	0.063*
H18B	0.5961	0.3386	0.1584	0.063*
H18C	0.5527	0.3702	0.2269	0.063*
C19	0.4624(3)	0.1336(2)	0.76764(17)	0.0488(8)
H19A	0.4813	0.1492	0.8121	0.073*
H19B	0.3952	0.1593	0.7557	0.073*
H19C	0.5179	0.1517	0.7378	0.073*
C20	0.5343(2)	0.00383(17)	0.78435(13)	0.0324(6)
C21	0.5933(3)	−0.1312(2)	0.79420(17)	0.0515(9)
H21A	0.6596	−0.1184	0.7718	0.077*
H21B	0.5711	−0.1867	0.7830	0.077*
H21C	0.6038	−0.1272	0.8412	0.077*
N1	0.76299(15)	0.17019(12)	−0.06041(9)	0.0182(4)
N2	0.76032(17)	0.21102(12)	−0.12215(10)	0.0228(4)
N3	0.63625(15)	0.12045(12)	0.01645(10)	0.0192(4)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
N4	0.56919(16)	0.18908(13)	0.02691(11)	0.0244(5)
N5	0.80259(15)	0.16525(12)	0.06283(9)	0.0197(4)
N6	0.83572(19)	0.19763(13)	0.12476(10)	0.0281(5)
N7	0.83347(15)	0.04729(12)	−0.07550(9)	0.0176(4)
N8	0.91015(16)	−0.01199(15)	−0.07923(10)	0.0258(5)
N9	0.71715(16)	−0.01795(12)	0.00381(9)	0.0177(4)
N10	0.71156(18)	−0.10363(13)	0.00353(10)	0.0260(5)
N11	0.87663(15)	0.03741(12)	0.04816(9)	0.0180(4)
N12	0.94883(17)	0.01129(14)	0.09679(10)	0.0254(5)
N13	0.13075(15)	0.88337(12)	0.02540(10)	0.0190(4)
N14	0.06187(16)	0.81587(13)	0.03667(11)	0.0260(5)
N15	0.25249(15)	0.82830(12)	−0.05325(9)	0.0187(4)
N16	0.24530(18)	0.79141(13)	−0.11655(10)	0.0247(5)
N17	0.29435(16)	0.83362(12)	0.07026(9)	0.0201(4)
N18	0.32585(19)	0.79562(13)	0.12969(10)	0.0282(5)
N19	0.21885(16)	1.01884(11)	0.01018(9)	0.0171(4)
N20	0.20948(18)	1.10386(13)	0.00988(11)	0.0270(5)
N21	0.33004(15)	0.94896(12)	−0.06905(9)	0.0176(4)
N22	0.40889(16)	1.00570(14)	−0.07196(10)	0.0258(5)
N23	0.37591(15)	0.95743(12)	0.05306(9)	0.0177(4)
N24	0.45569(16)	0.98858(14)	0.09337(10)	0.0254(5)
O1	0.84458(15)	0.23413(12)	−0.14328(9)	0.0328(4)
O2	0.67422(14)	0.22262(12)	−0.14531(9)	0.0326(4)
O3	0.52551(16)	0.21602(12)	−0.02126(10)	0.0410(5)
O4	0.55785(15)	0.21182(11)	0.08219(9)	0.0321(4)
O5	0.76596(17)	0.21543(13)	0.16197(9)	0.0405(5)
O6	0.92974(16)	0.20657(15)	0.13298(10)	0.0442(5)
O7	1.00103(13)	0.01225(14)	−0.07588(10)	0.0364(5)
O8	0.88062(17)	−0.08185(12)	−0.08696(10)	0.0386(5)
O9	0.67274(17)	−0.13544(11)	−0.04388(10)	0.0373(5)
O10	0.7424(2)	−0.13729(11)	0.05235(10)	0.0442(6)
O11	1.03522(14)	0.04300(13)	0.09654(9)	0.0343(5)
O12	0.91996(17)	−0.04454(14)	0.13097(11)	0.0484(6)
O13	0.05004(16)	0.79488(13)	0.09236(10)	0.0392(5)
O14	0.01861(17)	0.78776(12)	−0.01076(10)	0.0413(5)
O15	0.15714(15)	0.78373(12)	−0.13831(10)	0.0363(5)
O16	0.32714(15)	0.76718(12)	−0.14031(9)	0.0338(5)
O17	0.25428(17)	0.77779(12)	0.16647(9)	0.0369(5)
O18	0.41859(17)	0.78036(15)	0.13639(10)	0.0468(6)
O19	0.2355(2)	1.13853(11)	0.05933(10)	0.0441(6)
O20	0.17145(18)	1.13437(11)	−0.03828(10)	0.0391(5)
O21	0.38337(17)	1.07637(12)	−0.07984(10)	0.0383(5)
O22	0.49939(13)	0.97899(15)	−0.06786(10)	0.0383(5)
O23	0.43954(16)	1.05537(12)	0.11665(9)	0.0364(5)
O24	0.53618(15)	0.94879(14)	0.09710(10)	0.0391(5)
O25	0.4423(2)	0.54612(16)	0.22842(12)	0.0632(8)
O26	0.30677(16)	0.53597(13)	0.29828(9)	0.0402(5)
O27	0.3716(2)	0.42919(14)	0.24302(10)	0.0571(7)
O28	0.72880(17)	0.55502(12)	0.22136(9)	0.0378(5)
O29	0.69442(16)	0.45652(11)	0.29486(8)	0.0360(5)
O30	0.66216(17)	0.44240(12)	0.18785(8)	0.0382(5)
O31	0.45195(17)	0.04644(14)	0.76383(11)	0.0460(5)
O32	0.61348(15)	0.03081(13)	0.80771(9)	0.0395(5)
O33	0.51323(18)	−0.07451(13)	0.77449(12)	0.0515(6)

without any serious disorder the non-centrosymmetric space group $P2_12_12_1$ has been chosen.

Comment

Pseudosymmetry is a problem, which may be present during the course of many crystal structure determinations [5]. A typical problem is the appearance of additional and weak superstructure reflections which necessitate a larger super lattice for a correct description of the true structure. Another problem is that the true space group is sometimes obscured by the fact that some extinction rules are slightly disrupted by weak reflections. It is obvious that some classes of compounds are predestined for this effect [6]. This contribution is part of our ongoing interest in the hydrogen bonding of CL-20 (2,4,6,8,10,12-hexanitrohexaazaisowurtzitane) and its derivatives [7, 8].

The asymmetric unit of the title structure consists of two CL20 molecules and three dimethyl carbonates. Simple CL20 solvate structures may be a class of compounds which feature pseudosymmetry problems. All molecules are connected *via* charge supported non-classical $CH \cdots O$ hydrogen bonds.

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