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Ethylenediammonium Bis(monohydrogen oxalate) Monohydrate and Two Modifications of Trimethylenediammonium Bis(monohydrogen oxalate) Monohydrate

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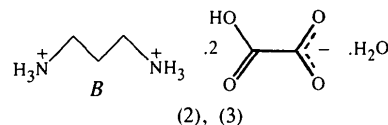
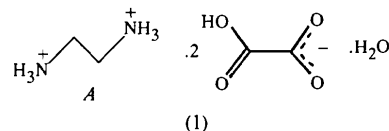
Abstract

Essential features of the crystal structures of $[\text{NH}_3(\text{CH}_2)_n\text{NH}_3]^{2+} \cdot 2(\text{HOOC}\text{COO})^- \cdot \text{H}_2\text{O}$ ($0 < n < 6$) are preserved through changes of conformation and space group. In each of the title structures, ethylenediammonium bis(monohydrogen oxalate) monohydrate, $\text{C}_2\text{H}_{10}\text{N}_2^{2+} \cdot 2\text{C}_2\text{HO}_4^- \cdot \text{H}_2\text{O}$, and two modifications of trimethylenediammonium bis(monohydrogen oxalate) monohydrate, $\text{C}_3\text{H}_{12}\text{N}_2^{2+} \cdot 2\text{C}_2\text{HO}_4^- \cdot \text{H}_2\text{O}$, the cation and the water molecule occupy special positions. Linear hydrogen-bonded $(\text{Hoxalate})_n$ chains are parallel to and connected to hydrogen-bonded spirals in which the water molecules link anions and cations. Unique to each structure are additional hydrogen bonds, perpendicular to these chains, which connect cations and anions into three-dimensional arrays.

Comment

The hydrogen oxalate ion is an important species in solution. The close proximity of the COOH groups in oxalic acid leads to a wide separation of the $\text{p}K_a$ values (1.37 and 3.81; McAuley & Nancollas, 1960) compared with, for example, succinic acid, $(\text{HOOC}-\text{CH}_2)_2$, for which the $\text{p}K_a$ values are 4.0 and 5.21 (Yasada *et al.*, 1960). There are many reports of the hydrogen oxalate ion in crystals. The ion is usually near-planar and connected into chains by short (2.49–2.57 Å) hydrogen bonds, with O—H typically 0.88 (3) and H···O 1.68 (3) Å (*e.g.* Küppers, 1973).

In the present work, the title salts, ethylenediammonium bis(monohydrogen oxalate) monohydrate, (1), and two modifications of trimethylenediammonium bis(monohydrogen oxalate) monohydrate, (2) and (3), were the only crystalline products obtained from aqueous mixtures of oxalic acid and ethylenediamine, or trimethylenediamine, regardless of the ratio of the components. Trimethylenediamine samples contained (2) and



(3) as morphologically different forms in a ratio of approximately 1:4. The same stoichiometry [cation²⁺·2(Hoxalate)·H₂O] is known for the tetramethylenediammonium (Babu, Weakley & Murthy, 1998) and hexamethylenediammonium (Vijayalakshmi & Srinivasan, 1983) salts. Although some of these crystals are monoclinic and others orthorhombic, the three-dimensional hydrogen-bonded networks are very similar. The water molecule and the cation lie on special positions, making the N atoms equivalent. The atomic numbering schemes for the ethylenediammonium cation, the trimethylenediammonium cation and the anion are shown in Fig. 1, while details of the symmetries and conformations are given in Table 2.

Packing diagrams are shown in Figs. 2, 3 and 4 for (1), (2) and (3), respectively. In each case, the hydrogen oxalate anions are connected into chains (perpendicular to the page in each figure) by O15—H151···O12' hydrogen bonds. Tetrahedral hydrogen bonding of the water molecule (O21) connects these anions and the cations into two symmetry-related spirals linked at the water molecules. Fig. 5 shows these spirals with the repeat unit [N1—H11···O21—H211···O11···H12'—N1'], in which N1···O21, O21···O11 and O11···N1' distances average 2.77 (1), 2.72 (1) and 2.83 (1) Å, respectively. The angles at the H atoms are in the range 157–175°. (Symmetry codes for the individual structures are given in the tables.)

Unique to each structure are the symmetry relationships between the anions in the chain and the interaction of the H13 atom (bonded to N1 but not used in the interactions described above) with O atoms of the anion to give two possible but less satisfactory hydrogen bonds, with N1···O distances in the range 2.8–3.2 Å and angles at hydrogen in the range 129–151°.

The same stoichiometry is also found in the much studied compound $(NH_4)_2 \cdot 2(\text{Hoxalate}) \cdot H_2O$, in which the two N atoms are not symmetry related. Both the monoclinic (Keller *et al.*, 1982) and orthorhombic (Küppers, 1973) forms of this compound have distorted versions of the pattern described above, associated with disordered H-atom sites. There is no evidence of H-atom disorder in compounds (1), (2) or (3).

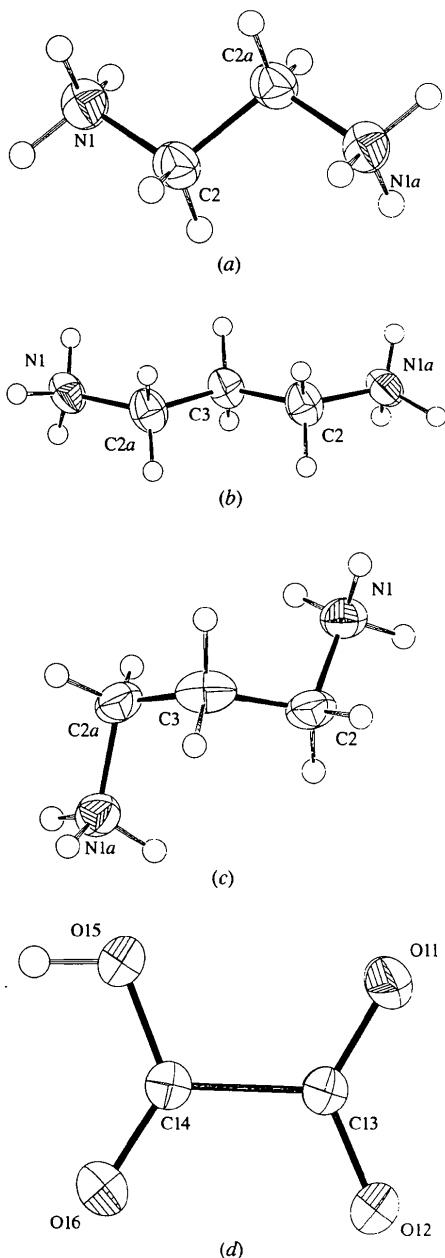


Fig. 1. Components of the title structures, showing (a) the ethylenediammonium ion in (1), (b) the trimethylenediammonium ion in (2), (c) the trimethylenediammonium ion in (3) and (d) the hydrogen oxalate ion in (1). Displacement ellipsoids are at 50% probability.

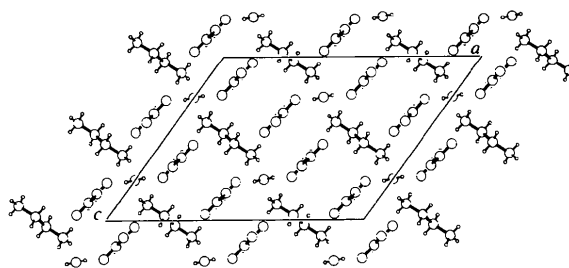


Fig. 2. Packing in (1), seen down the *b* axis.

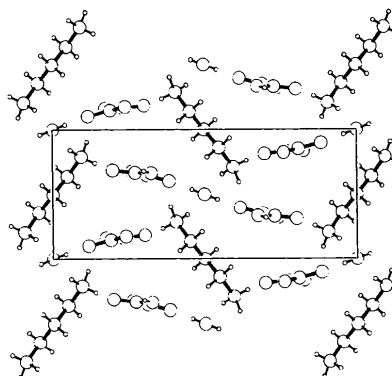


Fig. 3. Packing in (2), seen down the *c* axis.

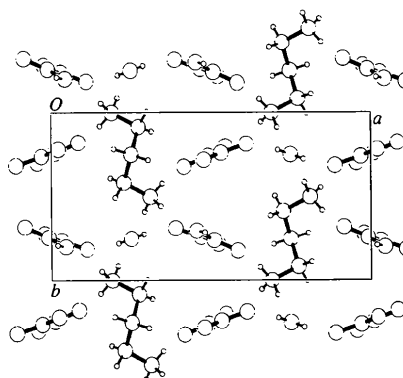


Fig. 4. Packing in (3), seen down the *c* axis.

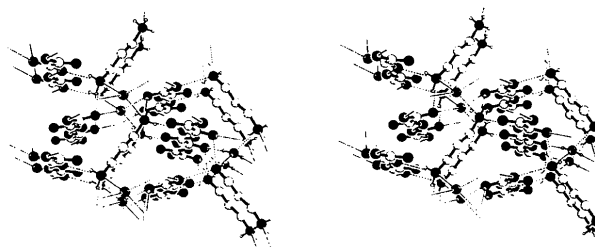
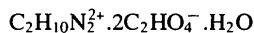


Fig. 5. Hydrogen-bonding network in (2), viewed close to the *c* axis.

Experimental

Aqueous solutions of the appropriate amine (1 mmol) and oxalic acid (2 mmol) were mixed and allowed to crystallize at room temperature. This and other amine-to-acid ratios gave only (1), (2) and (3) as crystalline materials. The trimethylenediamine sample contained (2) and (3) as morphologically different forms in a ratio of approximately 1:4.

Compound (1)*Crystal data* $M_r = 258.20$

Monoclinic

 $C2/c$ $a = 17.9713 (12) \text{ \AA}$ $b = 5.6693 (9) \text{ \AA}$ $c = 13.9522 (11) \text{ \AA}$ $\beta = 126.8700 (6)^\circ$ $V = 1137.2 (2) \text{ \AA}^3$ $Z = 4$ $D_x = 1.508 \text{ Mg m}^{-3}$ D_m not measured*Data collection*Enraf-Nonius CAD-4
diffractometer ω - 2θ scans

Absorption correction: none

1364 measured reflections

1307 independent reflections

930 reflections with

 $I > 2\sigma(I)$ *Refinement*Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.045$ $wR(F^2) = 0.120$ $S = 1.064$

1307 reflections

99 parameters

Only coordinates of H atoms
refinedMo $K\alpha$ radiation $\lambda = 0.71069 \text{ \AA}$ Cell parameters from 25
reflections $\theta = 14.0\text{--}15.0^\circ$ $\mu = 0.144 \text{ mm}^{-1}$ $T = 295 (2) \text{ K}$

Prism

 $0.40 \times 0.17 \times 0.07 \text{ mm}$

Colourless

 $R_{int} = 0.007$ $\theta_{max} = 27.46^\circ$ $h = -23 \rightarrow 18$ $k = 0 \rightarrow 7$ $l = 0 \rightarrow 18$

3 standard reflections

frequency: 60 min

intensity decay: 2%

 $w = 1/[\sigma^2(F_o^2) + (0.0829P)^2$
 $+ 0.6648P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{max} = 0.012$ $\Delta\rho_{max} = 0.339 \text{ e \AA}^{-3}$ $\Delta\rho_{min} = -0.491 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for
Crystallography (Vol. C)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (1)
$$U_{eq} = (1/3)\sum_i\sum_j U^{ij} a_i^* a_j^*$$

	x	y	z	U_{eq}
N1	0.11983 (10)	0.2468 (3)	0.40310 (14)	0.0336 (4)
C2	0.21540 (13)	0.1510 (3)	0.46988 (18)	0.0334 (4)
O11	-0.10157 (11)	-0.5658 (2)	0.76515 (13)	0.0486 (4)
O12	-0.11481 (12)	-0.7198 (2)	0.90017 (14)	0.0533 (5)
C13	-0.11119 (12)	-0.5517 (3)	0.84555 (16)	0.0319 (4)
C14	-0.11860 (12)	-0.3050 (3)	0.88605 (16)	0.0316 (4)
O15	-0.11019 (13)	-0.1354 (2)	0.83004 (15)	0.0528 (5)
O16	-0.12942 (12)	-0.2814 (2)	0.96233 (15)	0.0526 (4)
O21	0	0.1021 (3)	3/4	0.0345 (4)
H11	0.0765 (15)	0.118 (5)	0.368 (2)	0.050
H12	0.1095 (15)	0.355 (5)	0.346 (2)	0.050
H13	0.1119 (16)	0.312 (4)	0.454 (2)	0.050
H21	0.2214 (15)	0.073 (5)	0.414 (2)	0.050

H22	0.2248 (16)	0.049 (5)	0.525 (2)	0.050
H151	-0.1096 (19)	0.000 (6)	0.859 (3)	0.079
H211	-0.0302 (16)	0.195 (5)	0.761 (2)	0.052

Table 2. Symmetries and conformations in (1), (2) and (3)

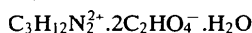
	Space group	Water site	Cation site	Cation conformation
(1)	$C2/c$	2 (4e)	$\bar{1}$ (4d)	anti
(2)	$P2_12_12$	2 (2a)	2 (2b)	anti-anti
(3)	$Pccn$	2 (4d)	2 (4c)	gauche-gauche

Table 3. Selected geometric parameters (\AA , $^\circ$) for (1)

N1—C2	1.481 (2)	C13—C14	1.544 (2)
C2—C2'	1.503 (4)	C14—O16	1.199 (2)
O11—C13	1.236 (2)	C14—O15	1.304 (2)
O12—C13	1.246 (2)		
N1—C2—C2'	109.63 (19)	O16—C14—O15	126.07 (16)
O11—C13—O12	126.34 (16)	O16—C14—C13	121.44 (15)
O11—C13—C14	118.73 (15)	O15—C14—C13	112.47 (14)
O12—C13—C14	114.92 (14)		
O12—C13—C14—O15	177.06 (18)	N1—C2—C2'—N1'	180.0

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.Table 4. Hydrogen-bonding geometry (\AA , $^\circ$) for (1)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H11 \cdots O21 ⁱ	0.96 (3)	1.86 (3)	2.756 (2)	155 (2)
N1—H12 \cdots O11 ⁱ	0.93 (3)	1.90 (3)	2.822 (2)	174 (2)
N1—H13 \cdots O12 ⁱⁱ	0.88 (3)	2.01 (3)	2.806 (2)	149 (2)
N1—H13 \cdots O16 ⁱⁱ	0.88 (3)	2.52 (2)	3.212 (2)	136 (2)
O15—H151 \cdots O12 ⁱⁱⁱ	0.86 (3)	1.71 (3)	2.5717 (19)	174 (3)
O21—H211 \cdots O11 ⁱⁱⁱ	0.83 (2)	1.89 (2)	2.7176 (18)	171 (2)

Symmetry codes: (i) $-x, -y, 1 - z$; (ii) $-x, 1 + y, \frac{3}{2} - z$; (iii) $x, 1 + y, z$.**Compound (2)***Crystal data* $M_r = 272.22$

Orthorhombic

 $P2_12_12$ $a = 6.7954 (8) \text{ \AA}$ $b = 16.1594 (13) \text{ \AA}$ $c = 5.6307 (10) \text{ \AA}$ $V = 618.30 (14) \text{ \AA}^3$ $Z = 2$ $D_x = 1.462 \text{ Mg m}^{-3}$ D_m not measuredMo $K\alpha$ radiation $\lambda = 0.71073 \text{ \AA}$ Cell parameters from 25
reflections $\theta = 14.0\text{--}15.0^\circ$ $\mu = 0.137 \text{ mm}^{-1}$ $T = 295 (2) \text{ K}$

Prism

 $0.50 \times 0.13 \times 0.09 \text{ mm}$

Colourless

*Data collection*Enraf-Nonius CAD-4
diffractometer ω - 2θ scans

Absorption correction: none

1076 measured reflections

1076 independent reflections

872 reflections with

 $I > 2\sigma(I)$ $\theta_{max} = 29.95^\circ$ $h = 0 \rightarrow 9$ $k = 0 \rightarrow 22$ $l = 0 \rightarrow 7$

3 standard reflections

frequency: 90 min

intensity decay: 3%

*Refinement*Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.036$ $wR(F^2) = 0.102$ $S = 1.005$

1076 reflections

107 parameters

 $w = 1/[\sigma^2(F_o^2) + (0.0679P)^2$
 $+ 0.0273P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{max} = 0.054$ $\Delta\rho_{max} = 0.176 \text{ e \AA}^{-3}$ $\Delta\rho_{min} = -0.247 \text{ e \AA}^{-3}$

Only coordinates of H atoms refined
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2067 measured reflections
1765 independent reflections
1365 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

$\theta_{\text{max}} = 29.97^\circ$
 $h = 0 \rightarrow 20$
 $k = 0 \rightarrow 10$
 $l = 0 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: none

Table 5. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
N1	0.2898 (3)	0.59389 (9)	0.9077 (3)	0.0387 (4)
C2	0.1393 (3)	0.54775 (12)	1.0447 (4)	0.0408 (4)
C3	0	1/2	0.8895 (5)	0.0361 (5)
O11	0.1033 (2)	0.88326 (7)	0.7549 (2)	0.0399 (3)
O12	0.1766 (3)	0.76015 (8)	0.9027 (2)	0.0545 (5)
C13	0.1481 (3)	0.80909 (10)	0.7363 (3)	0.0300 (3)
C14	0.1674 (3)	0.77075 (9)	0.4858 (3)	0.0306 (3)
O15	0.1640 (3)	0.82503 (8)	0.3152 (2)	0.0449 (4)
O16	0.1865 (3)	0.69750 (8)	0.4597 (2)	0.0550 (5)
O21	0	0	0.4260 (3)	0.0432 (5)
H11	0.343 (4)	0.5580 (16)	0.802 (5)	0.058
H12	0.405 (4)	0.6123 (16)	1.026 (4)	0.058
H13	0.239 (4)	0.6383 (17)	0.855 (5)	0.058
H21	0.212 (4)	0.5091 (16)	1.153 (5)	0.061
H22	0.066 (4)	0.5830 (17)	1.155 (5)	0.061
H31	0.073 (4)	0.4635 (12)	0.774 (5)	0.054
H151	0.179 (5)	0.7995 (17)	0.179 (5)	0.067
H211	-0.038 (4)	0.0418 (15)	0.515 (4)	0.065

Table 6. Selected geometric parameters (\AA , $^\circ$) for (2)

N1—C2	1.482 (2)	O12—C13	1.242 (2)
C2—C3	1.502 (2)	C13—C14	1.546 (2)
C3—C2'	1.502 (2)	C14—O16	1.200 (2)
O11—C13	1.2411 (19)	C14—O15	1.3009 (19)
N1—C2—C3	112.99 (17)	O11—C13—C14	119.01 (14)
C2—C3—C2'	108.8 (2)	O16—C14—O15	125.22 (15)
O12—C13—O11	126.12 (15)	O16—C14—C13	121.08 (14)
O12—C13—C14	114.85 (13)	O15—C14—C13	113.69 (13)
N1—C2—C3—C2'	177.1 (2)	O11—C13—C14—O15	10.3 (2)

Symmetry code: (i) $-x, 1-y, z$.

Table 7. Hydrogen-bonding geometry (\AA , $^\circ$) for (2)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H11...O21 ⁱ	0.91 (3)	1.91 (3)	2.806 (2)	167 (2)
N1—H12...O11 ⁱⁱ	1.07 (3)	1.83 (3)	2.878 (2)	166 (2)
N1—H13...O12	0.85 (3)	2.03 (3)	2.795 (2)	149 (3)
N1—H13...O16	0.85 (3)	2.45 (3)	3.108 (2)	135 (2)
O15—H151...O12 ⁱⁱⁱ	0.88 (3)	1.68 (3)	2.5498 (17)	171 (3)
O21—H211...O11 ^{iv}	0.88 (3)	1.87 (3)	2.7352 (17)	168 (2)
O21—H211...O15 ^v	0.88 (3)	2.58 (2)	3.1024 (14)	119 (2)

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$; (iii) $x, y, z - 1$; (iv) $-x, 1 - y, z$.

Compound (3)

Crystal data

C₃H₁₂N₂²⁺·2C₂HO₄⁻·H₂O $M_r = 272.22$

Orthorhombic

Pccn

 $a = 14.6727$ (19) \AA $b = 7.3876$ (6) \AA $c = 11.3348$ (18) \AA $V = 1228.6$ (3) \AA^3 $Z = 4$ $D_x = 1.472$ Mg m⁻³ D_m not measuredMo $K\alpha$ radiation $\lambda = 0.71079$ \AA

Cell parameters from 25 reflections

 $\theta = 14.1$ – 14.7° $\mu = 0.137$ mm⁻¹ $T = 295$ (2) K

Tablet

 $0.38 \times 0.36 \times 0.31$ mm

Colourless

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.108$ $S = 0.982$

1765 reflections

107 parameters

Only coordinates of H atoms refined

 $w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.1199P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.018$ $\Delta\rho_{\text{max}} = 0.273$ e \AA^{-3} $\Delta\rho_{\text{min}} = -0.226$ e \AA^{-3}

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)Table 8. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (3)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
N1	0.18992 (6)	0.00060 (13)	0.32780 (9)	0.0354 (2)
C2	0.27332 (7)	0.08496 (17)	0.27719 (11)	0.0406 (3)
C3	1/4	1/4	0.20436 (15)	0.0429 (4)
O11	0.61126 (5)	0.18659 (13)	0.35920 (6)	0.0415 (2)
O12	0.47795 (5)	0.28122 (13)	0.28783 (6)	0.0415 (2)
C13	0.53119 (6)	0.23961 (14)	0.36914 (8)	0.0284 (2)
C14	0.49086 (7)	0.25865 (14)	0.49465 (8)	0.0289 (2)
O15	0.54467 (5)	0.20205 (12)	0.57816 (7)	0.0406 (2)
O16	0.41587 (5)	0.32150 (15)	0.50988 (7)	0.0505 (3)
O21	3/4	1/4	0.00906 (11)	0.0440 (3)
H11	0.2075 (11)	-0.088 (2)	0.3860 (14)	0.053
H12	0.1572 (11)	-0.055 (2)	0.2702 (13)	0.053
H13	0.1527 (11)	0.081 (2)	0.3620 (12)	0.053
H21	0.3145 (12)	0.112 (2)	0.3433 (14)	0.061
H22	0.3002 (12)	-0.005 (2)	0.2254 (15)	0.061
H31	0.1958 (12)	0.217 (2)	0.1523 (15)	0.064
H151	0.5194 (12)	0.211 (2)	0.6486 (16)	0.061
H211	0.7099 (12)	0.277 (3)	-0.0391 (16)	0.066

Table 9. Selected geometric parameters (\AA , $^\circ$) for (3)

N1—C2	1.4883 (15)	O12—C13	1.2467 (12)
C2—C3	1.5117 (16)	C13—C14	1.5473 (13)
C3—C2'	1.5117 (16)	C14—O16	1.2067 (12)
O11—C13	1.2435 (12)	C14—O15	1.3016 (12)
N1—C2—C3	111.23 (8)	O12—C13—C14	114.69 (8)
C2'—C3—C2	113.80 (14)	O16—C14—O15	124.94 (9)
O11—C13—O12	127.07 (9)	O16—C14—C13	121.02 (9)
O11—C13—C14	118.24 (8)	O15—C14—C13	114.04 (8)
N1—C2—C3—C2'	76.09 (8)	O11—C13—C14—O15	-5.14 (15)

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, z$.Table 10. Hydrogen-bonding geometry (\AA , $^\circ$) for (3)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H11...O21 ⁱ	0.962 (16)	1.801 (17)	2.7611 (13)	175.2 (14)
N1—H12...O11 ⁱ	0.910 (16)	1.883 (16)	2.7816 (12)	169.1 (14)
N1—H13...O16 ⁱⁱ	0.893 (16)	2.084 (15)	2.8976 (13)	150.9 (13)
N1—H13...O12 ⁱⁱⁱ	0.893 (16)	2.329 (16)	2.9783 (13)	129.5 (11)
O15—H151...O12 ⁱⁱⁱ	0.883 (18)	1.692 (19)	2.5733 (11)	175.5 (17)
O21—H211...O11 ^{iv}	0.828 (17)	1.869 (17)	2.6923 (11)	172.7 (18)

Symmetry codes: (i) $x - \frac{1}{2}, -y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} - y, z$; (iii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

For all compounds, data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1996). Program(s) used to solve structures: *SHELXS97* (Sheldrick, 1997) for (1) and (2); *SHELXS* (Sheldrick, 1990) for (3). For all compounds, program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLUTON92* (Spek, 1992a) and *PLATON92* (Spek, 1992b)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1061). Services for accessing these data are described at the back of the journal.

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Twisted Structure of a Substituted 2-Chloro-3-(4-chloro-2-methyl-1,3-oxazol-5-yl)-1H-indole

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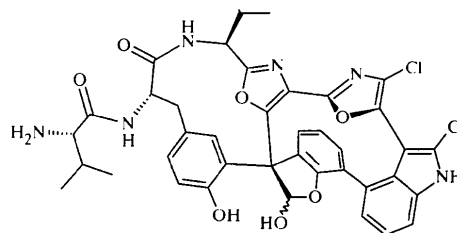
Abstract

2,2,2-Trichloroethyl 2-chloro-3-(4-chloro-2-methyl-1,3-oxazol-5-yl)-1H-indole-1-carboxylate, $C_{15}H_9Cl_5N_2O_3$, the first synthetic example of an oxazolyindole with a

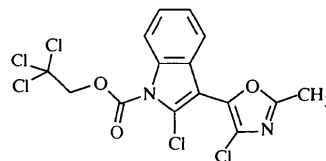
Cl atom at the *ortho* position of each ring, exhibits a dihedral angle of $45.6(1)^\circ$ between the heteroaromatic ring systems.

Comment

The natural product diazonamide A, isolated from the marine ascidian *Diazona chinensis*, exhibits interesting biological activity and possesses a unique structure (see Scheme below). Four adjacent heterocyclic rings, viz one oxazole, one chlorooxazole, one chloroindole and one benzofuran, are fixed in one macrocyclic ring of a bicyclic framework in such a way that any appreciable overlap of the conjugated heterocycles is prevented; e.g. the chlorooxazole and the chloroindole ring subtend an interplanar angle of 60° (Lindquist *et al.*, 1991). In order to elucidate to what extent this torsion is attributed to the macrocyclic arrangement of the four rings or the repulsive effect of the two *ortho*-chloro substituents, we focused our interest, with respect to the total synthesis of diazonamide A, on 2-chloro-3-(4-chloro-2-methyl-1,3-oxazol-5-yl)-1H-indole-1-carboxylic acid 2,2,2-trichloroethyl ester, (1), the first synthetic example of an oxazolyindole with one *ortho*-Cl atom on each heterocyclic ring.



Diazonamide A



(1)

Generally, X-ray structure analyses of molecules consisting of two five-membered heterocyclic rings connected to each other by a single bond and each bearing a substituent *ortho* to that bond are rare (Secondo *et al.*, 1996). The crystal structure determination of the new oxazolyindole (1) (Fig. 1) reveals that both heterocyclic rings are planar and that the molecular dimensions are similar to those of other oxazoles (Ambats & Marsh, 1965) and indoles (Chadwick, 1984). The two heterocyclic rings of (1), however, are twisted with respect to each other by a dihedral angle of $45.6(1)^\circ$. The distance