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Dimeric Aqua(3,5-dinitrobenzoato)(triethylene glycol)calcium(II) 3,5-Dinitrobenzoate Hydrate

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Abstract

The title dimeric compound, bis(μ -3,5-dinitrobenzoato-*O*:*O'*)bis[(triethylene glycol-*O*:*O'*,*O''*,*O'''*)-calcium(II)] bis(3,5-dinitrobenzoate) dihydrate, {[Ca(C₇H₃N₂O₆)(C₆H₁₄O₄)(H₂O)](C₇H₃N₂O₆)·H₂O}₂, has seven-coordinate Ca centres with distorted pentagonal bipyramidal geometry. Four O atoms in the coordination sphere belong to the triethylene glycol ligand, one belongs to the water molecule and the two remaining O atoms are from two different symmetry-related carboxylate groups belonging to two symmetry-related 3,5-dinitrobenzoate anions. The second dinitrobenzoate anion in the asymmetric unit does not participate in coordination, but is involved in hydrogen bonding to the hydroxy groups of the triethylene glycol ligand.

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Comment

In complexes of 3,5-dinitrobenzoate (DNB) with alkali and alkaline earth metal cations, the carboxylate group of DNB exhibits a variety of coordination modes. It can act as a monodentate (Hundal, Hundal, Kapoor & Poonia, 1991), an unsymmetrical bridging bidentate (Ward, Popov & Poonia, 1984) and a chelating bidentate ligand (Cradwick & Poonia, 1977). The DNB ligand is also capable of forming a homoconjugate of the type [DNB·H-DNB][−], which can act as a bidentate ligand to form complexes called acid salts (Tiekink, Hundal, Hundal, Kapoor & Poonia, 1990). The behaviour of DNB[−] as a counteranion is the result of a balance of various factors such as the charge on the cation and the steric crowding around it, operative solvent–cation interactions, flexibility of the neutral ligand and its role as a double-action species. As a net result, DNB[−] complexes can be either ion paired or charge separated. Our ongoing studies of [M(anion)(ligand)(solvent)] systems (where *M* is an alkaline earth cation) aim at distinguishing between the chemistry of Ca, Sr and Ba through crystallographic examination of the related complexes. The title compound, (I), is the first compound to be investigated in a series of complexes having triethylene glycol (TRIEG) as a neutral ligand and DNB as counteranion.

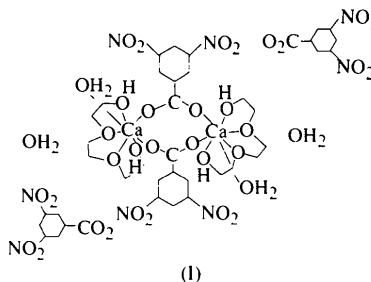


Fig. 1 is an ORTEP drawing (Johnson, 1965) of the title complex. Of the two DNB residues in the asymmetric unit, only one, *i.e.* DNBA, is coordinated to Ca²⁺, while DNBB is involved only in hydrogen bonding. Ca²⁺ is in a distorted pentagonal bipyramidal environment where all the four O atoms of TRIEG (O7, O8, O9 and O10) and atom O1A of DNBA form a pentagon and the water O11 atom and the carboxy O2A atom of the symmetry-related DNBA molecule occupy the apical positions. The carboxylate group of DNBA thus acts as a symmetrical bridge between two centrosymmetric Ca²⁺ ions. The Ca···Ca distance of 5.663(2) Å in the dimer is very long in comparison to that of 4.401(1) Å in the structure of [Sr(DNB)₂(tetraethylene glycol)]·H₂O (Kanters, Harder & Poonia, 1987). In the latter complex, the cluster is much more tightly packed because the bridging groups of DNB are triply coordinated to the Sr ion, whereas in the former the carboxylate groups are bidentate only. The two DNB anions of the title complex differ from

each other in terms of the twists of the carboxylate and the nitro groups with respect to the phenyl rings. In DNBA, the carboxylate and nitro groups make dihedral angles of 7.8 (5), 2.4 (17) and 4.3 (14)° with the phenyl ring, while these angles are 10.2 (7), 9.2 (12) and 17.5 (5)° for DNBB.

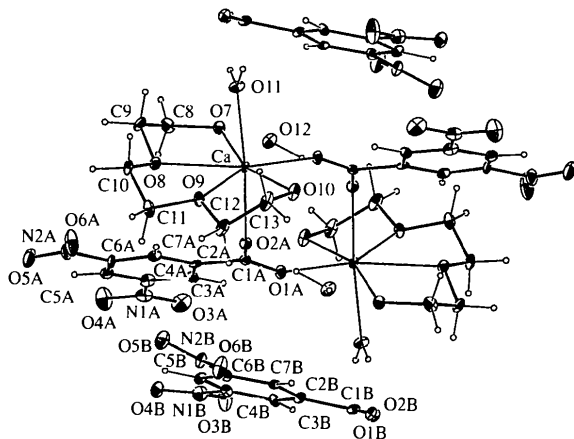


Fig. 1. ORTEP drawing (Johnson, 1965) of the final X-ray model showing the atomic labelling. The non-H atoms are shown with displacement ellipsoids drawn at the 30% probability level. H atoms have not been labelled for clarity.

The TRIEG ligand adopts a crown-ether-like conformation, as in the complex $[\text{Ca}(\text{TRIEG})_2]\text{Cl}_2 \cdot 4\text{H}_2\text{O}$ (Rogers & Bond, 1992), rather than a helical conformation. The O—C—C—O torsion angles (Table 2) alternate \pm gauche ($\pm 60^\circ$) and the C—O—C—C angles are all anti (180°), as in the common D_{3d} form of 18-crown-6. The Ca—O_{ether} and Ca—O_{hydroxy} distances observed in the title compound are comparable to those observed in the complex $[\text{Ca}(\text{TRIEG})_2]\text{Cl}_2 \cdot 4\text{H}_2\text{O}$.

The terminal TRIEG group, two water molecules, one carboxylate O atom of DNBA and both the carboxylate O atoms of DNBB are involved in extensive hydrogen bonding (Table 3). The uncoordinated water molecule donates one H atom to the carboxylate O2Aⁱⁱ atom of DNBA and another H atom to the carboxylate O1Bⁱ atom of DNBB (see Table 3). The O12 atom of this water molecule acts as a hydrogen-bond acceptor forming a hydrogen bond with the terminal hydroxy group O7 of the TRIEG ligand. The second terminal hydroxy group (O10) of TRIEG acts as a hydrogen-bond donor to the carboxylate O1B^{iv} atom of DNBB. The coordinated water molecule acts as a double donor to the carboxylate O2B atom of DNBB, thus giving hydrogen bonds to atoms O2Bⁱⁱ and O2Bⁱⁱⁱ. The water molecules and the TRIEG ligand serve as a link between the centrosymmetric cluster and the uncoordinated DNBB anion. DNBA is coordinated to the two centrosymmetrically related cations, while DNBB is effectively charge separated from the cation by the

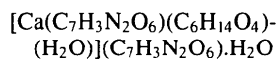
TRIEG molecule. The TRIEG ligand interacts with the DNBB molecule through its hydroxy H atom.

Comparing the title complex to $[\text{Ca}(\text{TRIEG})_2]\text{Cl}_2 \cdot 4\text{H}_2\text{O}$, it can be seen that the latter yields a charge-separated complex with the Cl⁻ ion being drawn out of the coordination sphere; TRIEG hydrogen bonds with the water O atom as well as the Cl⁻ ions. Neutral ligands such as TEG, TRIEG, *etc.*, have a polar H atom and generally coordinate to the cation and simultaneously hydrogen bond to the counteranion. This decreases the nucleophilic character of the anion resulting in charge-separated complexes. It appears that O—H...O anion bonding in the present complex is not strong enough to give rise to a completely charge-separated complex.

Experimental

The title compound was synthesized by the 1:1 reaction of calcium bis(3,5-dinitrobenzoate) and triethylene glycol in aqueous ethanol. The crystals were grown at room temperature by slow evaporation. The density D_m was measured by flotation in water/KI.

Crystal data



$M_r = 648.49$

Triclinic

$P\bar{1}$

$a = 13.939(7) \text{ \AA}$

$b = 12.210(8) \text{ \AA}$

$c = 9.298(3) \text{ \AA}$

$\alpha = 106.62(3)^\circ$

$\beta = 88.96(2)^\circ$

$\gamma = 114.32(5)^\circ$

$V = 1372.8(14) \text{ \AA}^3$

$Z = 2$

$D_x = 1.564 \text{ Mg m}^{-3}$

$D_m = 1.582 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 40 reflections

$\theta = 5\text{--}15^\circ$

$\mu = 0.308 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Prism

$0.3 \times 0.2 \times 0.1 \text{ mm}$

Pale yellow

Data collection

Philips PW1100 diffractometer

ω scans

Absorption correction:

none

4864 measured reflections

4358 independent reflections

1915 observed reflections

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0444$

$\theta_{\text{max}} = 25^\circ$

$h = -24 \rightarrow 24$

$k = -17 \rightarrow 17$

$l = 0 \rightarrow 16$

2 standard reflections

frequency: 90 min

intensity decay: 1%

Refinement

Refinement on F

$R = 0.065$

$wR = 0.058$

$S = 1.174$

$(\Delta/\sigma)_{\text{max}} = 0.003$

$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.08 \text{ e \AA}^{-3}$

Extinction correction: none

1915 reflections
388 parameters
H-atom parameters not refined
Weighting scheme: PESOS
(Martinez-Ripoll & Cano, 1975)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

O7—Ca—O8	65.5 (4)	O8—Ca—O1A'	145.2 (4)
O7—Ca—O9	129.8 (4)	O7—Ca—O1A'	80.6 (4)
O7—Ca—O10	162.1 (5)	O3A—N1A—O4A	125.1 (20)
O7—Ca—O11	78.8 (4)	O5A—N2A—O6A	126.9 (20)
O8—Ca—O9	64.4 (4)	O1A—C1A—O2A	126.5 (15)
O8—Ca—O10	130.4 (4)	O3B—N1B—O4B	127.5 (20)
O8—Ca—O11	84.1 (4)	O5B—N2B—O6B	124.1 (17)
O9—Ca—O10	66.3 (4)	O1B—C1B—O2B	127.6 (15)
O9—Ca—O11	92.9 (4)		

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U _{eq}
Ca	0.5873 (2)	0.76512 (3)	0.1338 (3)	0.028 (1)
O1A	0.5789 (8)	0.4022 (9)	-0.0654 (12)	0.048 (3)
O2A	0.6445 (8)	0.6121 (9)	-0.0053 (11)	0.044 (3)
O3A	0.7470 (14)	0.2169 (13)	0.1931 (18)	0.099 (6)
O4A	0.8960 (13)	0.3363 (15)	0.3352 (19)	0.014 (6)
O5A	1.0588 (12)	0.7766 (14)	0.4091 (16)	0.100 (5)
O6A	0.9859 (12)	0.8629 (13)	0.303 (2)	0.136 (8)
N1A	0.8192 (15)	0.3184 (17)	0.2529 (19)	0.071 (5)
N2A	0.9877 (13)	0.7760 (17)	0.333 (2)	0.083 (5)
C1A	0.6445 (11)	0.5096 (14)	0.0016 (15)	0.036 (4)
C2A	0.7365 (12)	0.5258 (11)	0.1066 (14)	0.031 (4)
C3A	0.7359 (11)	0.4183 (13)	0.1333 (17)	0.040 (4)
C4A	0.8183 (14)	0.4340 (15)	0.2282 (17)	0.049 (5)
C5A	0.9004 (13)	0.5447 (16)	0.2936 (17)	0.050 (4)
C6A	0.8998 (12)	0.6492 (15)	0.2597 (17)	0.050 (4)
C7A	0.8185 (13)	0.6427 (13)	0.1705 (18)	0.050 (4)
O1B	0.5738 (8)	0.3180 (9)	-0.6538 (10)	0.043 (3)
O2B	0.5381 (8)	0.1491 (9)	-0.5767 (11)	0.044 (3)
O3B	0.7770 (12)	0.1857 (15)	-0.1767 (19)	0.111 (6)
O4B	0.9147 (11)	0.3587 (14)	-0.0732 (13)	0.078 (4)
O5B	0.9217 (12)	0.7422 (13)	-0.1420 (17)	0.092 (4)
O6B	0.8520 (12)	0.7260 (12)	-0.3570 (17)	0.098 (5)
N1B	0.8342 (14)	0.2947 (17)	-0.1579 (17)	0.067 (5)
N2B	0.8680 (11)	0.6801 (13)	-0.2652 (16)	0.060 (4)
C1B	0.5881 (12)	0.2610 (13)	-0.5683 (15)	0.037 (4)
C2B	0.6805 (12)	0.3427 (14)	-0.4437 (15)	0.039 (4)
C3B	0.7158 (12)	0.2874 (14)	-0.3575 (17)	0.043 (4)
C4B	0.7983 (12)	0.3568 (14)	-0.2482 (17)	0.042 (4)
C5B	0.8532 (12)	0.4861 (14)	-0.2133 (15)	0.042 (4)
C6B	0.8156 (11)	0.5387 (13)	-0.2973 (18)	0.044 (4)
C7B	0.7320 (11)	0.4744 (13)	-0.4114 (15)	0.033 (3)
O7	0.5732 (8)	0.6929 (9)	0.3495 (11)	0.049 (3)
O8	0.7551 (8)	0.8709 (8)	0.3151 (11)	0.043 (3)
O9	0.7327 (8)	0.9312 (10)	0.0717 (11)	0.049 (3)
O10	0.5423 (9)	0.8157 (9)	-0.0843 (12)	0.059 (3)
O11	0.5356 (9)	0.9095 (9)	0.3091 (12)	0.051 (3)
O12	0.4563 (9)	0.4544 (9)	0.3090 (12)	0.058 (3)
C8	0.6640 (14)	0.7373 (19)	0.457 (2)	0.065 (6)
C9	0.7367 (13)	0.8715 (15)	0.4644 (16)	0.054 (4)
C10	0.8273 (12)	0.9932 (14)	0.3064 (18)	0.053 (4)
C11	0.8375 (13)	0.9819 (16)	0.145 (2)	0.065 (6)
C12	0.7264 (16)	0.9415 (16)	-0.0728 (17)	0.066 (6)
C13	0.6200 (16)	0.9318 (15)	-0.1058 (19)	0.067 (5)

Table 2. Selected geometric parameters (Å, °)

Ca—O2A	2.369 (12)	O3A—N1A	1.207 (19)
Ca—O7	2.387 (12)	O4A—N1A	1.23 (3)
Ca—O8	2.528 (10)	O5A—N2A	1.22 (3)
Ca—O9	2.407 (10)	O6A—N2A	1.18 (3)
Ca—O10	2.443 (13)	O1B—C1B	1.26 (2)
Ca—O11	2.375 (12)	O2B—C1B	1.231 (17)
Ca—O1A	2.315 (8)	O3B—N1B	1.20 (2)
O1A—C1A	1.229 (14)	O4B—N1B	1.20 (2)
O2A—C1A	1.27 (2)	O5B—N2B	1.231 (19)
O2A—Ca—O7	91.9 (4)	O10—Ca—O11	93.8 (4)
O2A—Ca—O8	90.0 (4)	O2A—Ca—O1A'	83.4 (4)
O2A—Ca—O9	91.5 (4)	O11—Ca—O1A'	97.1 (4)
O2A—Ca—O10	95.8 (4)	O10—Ca—O1A'	84.3 (4)
O2A—Ca—O11	170.4 (4)	O9—Ca—O1A'	149.4 (4)

O7—C8—C9—O8	-52.4 (18)	C9—O8—C10—C11	-178.1 (14)
O8—C10—C11—O9	54.3 (17)	C10—C11—O9—C12	165.2 (14)
O9—C12—C13—O10	-54.6 (18)	C11—O9—C12—C13	-158.5 (15)
C8—C9—O8—C10	-177.7 (14)		

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

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O12—H122W...O1B'	1.02	2.25	2.86	116
O12—H121W...O2A''	1.08	1.84	2.91	169
O11—H111W...O2B''	0.94	1.95	2.84	157
O11—H112W...O2B'''	0.91	1.99	2.79	145
O7...O12			2.60	
O10...O1B''			2.62	

Symmetry codes: (i) x, y, 1 + z; (ii) 1 - x, 1 - y, -z; (iii) x, 1 + y, 1 + z; (iv) 1 - x, 1 - y, -1 - z.

The weighting scheme used was $w = K/(a + b|F_o|)^2$: $0 \leq F_o \leq 7.0$, $a = 1.48$, $b = 0.17$; $7.0 < F_o \leq 16.8$, $a = 3.40$, $b = -0.15$; $16.8 < F_o$, $a = 0.89$, $b = 0.05$. All H atoms were fixed geometrically except for those attached to the water molecules, which were located in a difference Fourier map. H atoms attached to the terminal O atoms of the triethylene glycol ligand could not be located.

Data collection: Philips PW1100 diffractometer software. Cell refinement: *LSUCRE* (Appleman, 1995). Data reduction: *XRAY80* (Stewart, 1976). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *XRAY80*. Molecular graphics: *ORTEP* (Johnson, 1965) in *Xtal3.2* (Hall, Flack & Stewart, 1992). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1013). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(triethanolamine)calcium 3,5-Dinitrobenzoate

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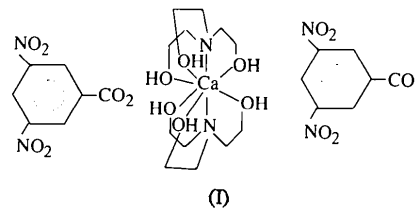
Abstract

In the title complex, $[\text{Ca}(\text{C}_6\text{H}_3\text{NO}_3)_2](\text{C}_7\text{H}_3\text{N}_2\text{O}_6)_2$, the Ca^{2+} ion is sandwiched between two triethanolamine ligands (IUPAC nomenclature: 2,2',2''-nitrilotriethanol), each of them coordinating through all four potential donor sites. The coordination polyhedron is distorted cubic. The 3,5-dinitrobenzoate counteranions do not interact directly with the cation but are hydrogen bonded to the hydroxy groups of the triethanolamine ligands.

Comment

In the complexes of *s*-block metal ions having a general formula $M(\text{anion})(\text{ligand})(\text{solvent})$, where the ligand is a neutral molecule having O or N as donor atom, the structure depends on whether or not the ligand has a polar or polarizable H atom (Poonia & Bajaj, 1979; Fenton, 1987). Important ligands in the

latter category are 1,10-phenanthroline, 2,2'-bipyridine, *etc.* These ligands only interact with the cation and do not interact with the counteranion through hydrogen bonding. The neutral ligands possessing polar or polarizable H atoms include hydroxy compounds and amines. Important examples include glycols, ethylenediamine, triethanolamine, *etc.* These ligands usually interact with the cation and are hydrogen bonded with the counteranion through polar H atoms. In the complexes of such ligands with *s*-block metals, the counteranion can interact with the complexed cation or can be excluded from the coordination sphere around the cation to give complexes where the counteranion is not directly interacting with the cation. The counteranion of the title compound is 3,5-dinitrobenzoate (DNB), which exhibits a wide range of coordination modes. In Ca^{2+} complexes, DNB has been found to act as a bridging bidentate, chelating bidentate and bridging tridentate ligand (Hundal, Martinez-Ripoll, Hundal & Poonia, 1995; Cradwick & Poonia, 1977; Kanters, Harder & Poonia, 1987). The structure of the title compound, (I), has been solved to determine the behaviour of the neutral ligand triethanolamine (TEA) and to determine the nature of the interaction of the counteranion 3,5-dinitrobenzoate with the cation and to compare the behaviour of the counteranion with that of 2,4-dinitrophenolate (DNP). Thermogravimetric analysis of the compound revealed that the solvent was not present in the final crystalline phase.



The crystal structure is shown in Fig. 1. The Ca atom is eightfold coordinated by two triethanolamine ligands. The TEA ligands use all four potential donor atoms, *i.e.* three O and one N atom. The coordination around Ca can be classified as distorted cubic, as can be seen from Fig. 2. The Ca—O distances are in the range 2.393 (7)–2.454 (8) Å and the Ca—N distances are 2.708 (5) and 2.730 (5) Å. The Ca—N bond distances are slightly longer than that observed in $\text{Ca}(\text{TEA})(\text{DNP})_2$ [2.600 (7) Å; Hundal *et al.*, 1995]. In the other complexes of TEA with NaI (Voegelé, Fischer & Weiss, 1974a), Sr(nitrate)₂ (Voegelé, Fischer & Weiss, 1974b), Ba(acetate)₂ (Voegelé, Thierry & Weiss, 1974), Ba(DNP)₂ (Kanters, Smeets, Venkatasubramanian & Poonia, 1984), and Ca(DNP)₂ (Hundal *et al.*, 1995), the TEA ligand normally has an extended conformation, due to the presence of at least three almost planar C—N—C—C fragments, and a *gauche* conformation of the terminal OH group with respect to the

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