CRYSTAL STRUCTURE OF *catena*-[AQUABIS(3,5-DINITROBENZOATO)CALCIUM(II)]

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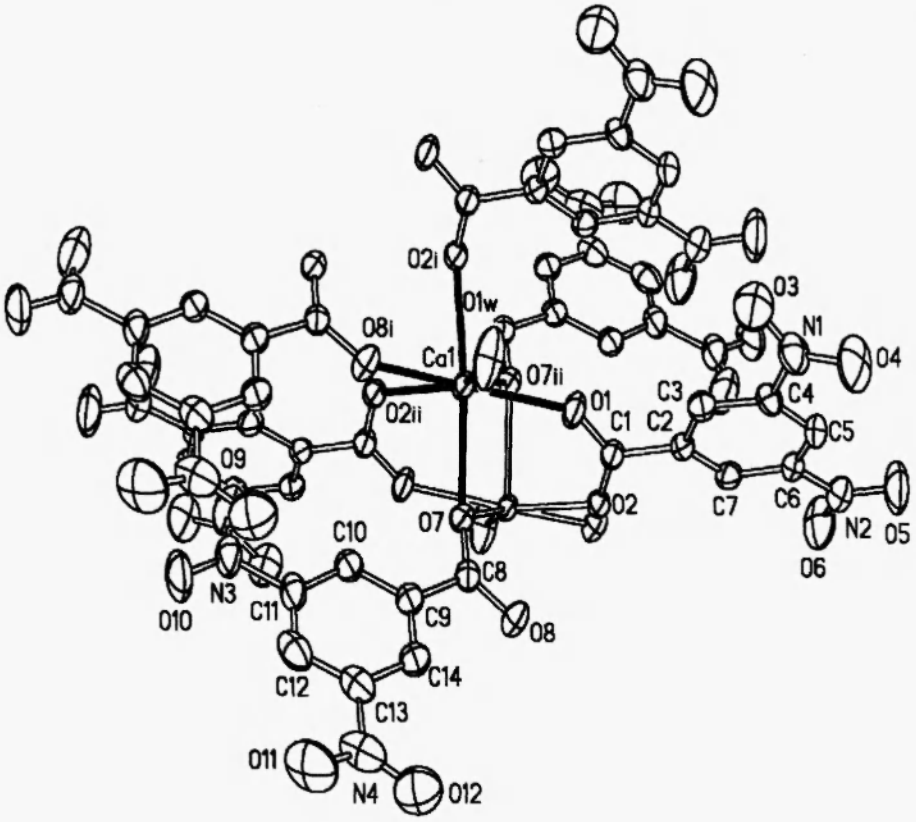

Figure 1. *ORTEP* plot illustrating the pentagonal bipyramidal geometry of the calcium atom in the title compound. Selected bond distances and angles: Ca1-O1 2.381(2), Ca1-O2^{i} 2.368(2), Ca1-O2^{ii} 2.485(2), Ca1-O7 2.373(2), Ca1-O7^{ii} 2.498(2), Ca1-O8^{i} 2.377(2), Ca1-O1*w* 2.377(3) Å; O1-Ca1-O2^{i} 105.5(1), O1-Ca1-O2^{ii} 145.9(1), O1-Ca1-O7 81.4(1), O1-Ca1-O7^{ii} 74.1(1), O1-Ca1-O8^{i} 141.5(1), O1-Ca1-O1*w* 72.5(1), O2^{i}-Ca1-O2^{ii} 84.0(1), O2^{i}-Ca1-O7 162.9(1), O2^{i}-Ca1-O7^{ii} 81.3(1), O2^{i}-Ca1-O8^{i} 82.2(1), O2^{i}-Ca1-O1*w* 89.2(1), O2^{ii}-Ca1-O7 81.9(1), O2^{ii}-Ca1-O7^{ii} 75.3(1), O2^{ii}-Ca1-O8^{i} 71.4(1), O2^{ii}-Ca1-O1*w* 141.2(1), O7-Ca1-O7^{ii} 85.8(1), O7-Ca1-O8^{i} 102.2(1), O7-Ca1-O1*w* 107.8(1), O7^{ii}-Ca1-O8^{i} 144.0(1), O7^{ii}-Ca1-O1*w* 141.3(1), O8^{i}-Ca1-O1*w* 69.9(1)°. Symmetry/translational code: *i* = *x*, 1 + *y*, *z*; *ii* = 0.5 – *x*, 1.5 – *y*, 1 – *z*.

Comment

The calcium atom in the title compound is bonded to six 3,5-dinitrobenzoato groups through the carboxyl oxygen atoms; the compound adopts a linear chain structure that propagates by two-fold screw axial translations. The pentagonal bipyramidal geometry is completed by the water ligand, which forms two relatively weak hydrogen bonds within the chain. The 3,5-dinitrobenzolate anion bridges three calcium atoms in a μ-*O*,*O*,*O*' manner. In an earlier study on the cadmium complex, the 3,5-dinitrobenzoato entity was found to chelate to the metal atom in the tetra(3,5-dinitrobenzoato)cadmate dianion, whose negative charge is balanced by two sodium atoms [1].

Experimental

Ammonium hydroxide was added dropwise to a mixture of calcium nitrate tetrahydrate (1 mmol, 0.24 g), 3,5-dinitrobenzoic acid (2 mmol, 0.42 g) and water (10 cm^3). When all had dissolved, the solution was filtered; slow evaporation of the solution gave colorless needles after two weeks. Calculated (Found) for $C_{14}H_8CaN_4O_{13}$: C 35.04 (35.01), H 1.80 (1.68), N 11.76% (11.66%). IR (KBr disc, cm^{-1}): 3659s, 3565m,

3136w, 3110m, 3097w, 1620vs, 1584s, 1555s, 1530s, 1460m, 1388s, 1346vs, 1193w, 1089w, 1075w, 927w, 913w, 799m, 725s, 716s, 538w.

Table 1. Crystal data for calcium bis(3,5-dinitrobenzoate) hydrate

Empirical formula	$C_{14}H_8CaN_4O_{13}$	Formula weight	480.32
Space group	*C*2/*c*	Diffractometer	Siemens CCD
Unit cell dimensions	*a* 25.768(4)	Volume, Å^3	3634(1)
	b 5.4660(8) β 114.271(3) °	*Z*	8
	c 28.304(4) Å	D_{calc}, g cm^{-3}	1.756
μ (Mo-*K*α), mm^{-1}	0.431	*F*(000)	1952
θ range, °	1.8 – 23.3	Temperature, °C	25
Reflections collected	7531	Independent reflections	2604 (R_{int} 0.053)
Reflections with $I>2\sigma(I)$	1753	No. parameters refined	297
R (all data)	0.077	Final *R* [$I>2\sigma(I)$]	0.040
wR (all data)	0.093	*wR* [$I>2\sigma(I)$]	0.081
w	$[\sigma^2+(0.0483P)^2]^{-1}$	Goodness-of-fit on F^2	0.95
Diff. hole and peak, *e*Å^{-3}	-0.25 – 0.26	CCDC deposition no.	166889
Programs	*SHELX*-97, *ORTEP* [2,3]		

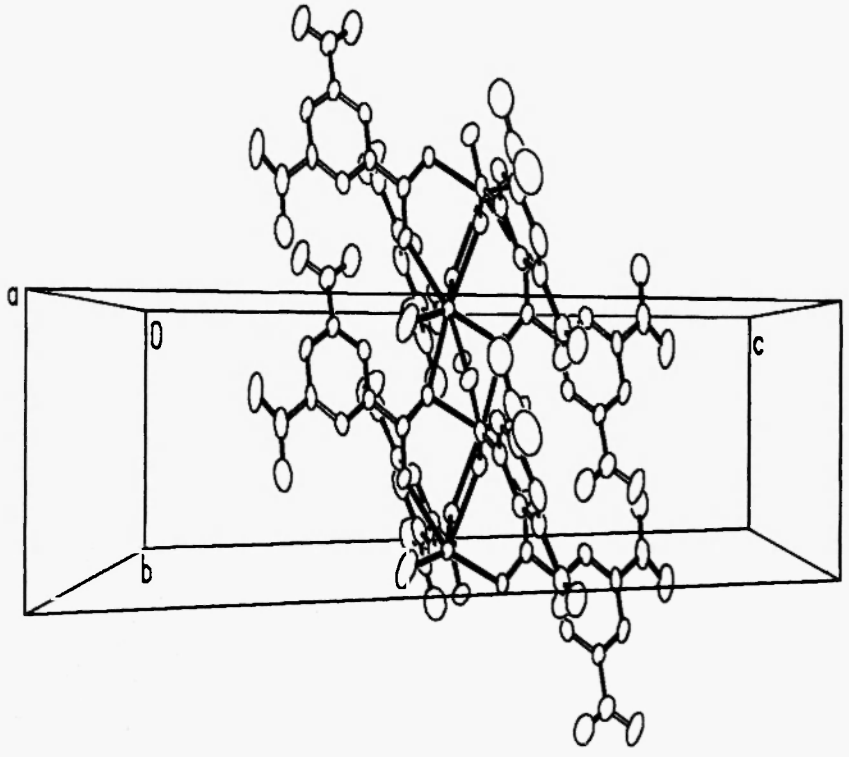

Figure 2. Plot of the polymeric structure projected on the *a*–*c* plane.

Acknowledgments
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