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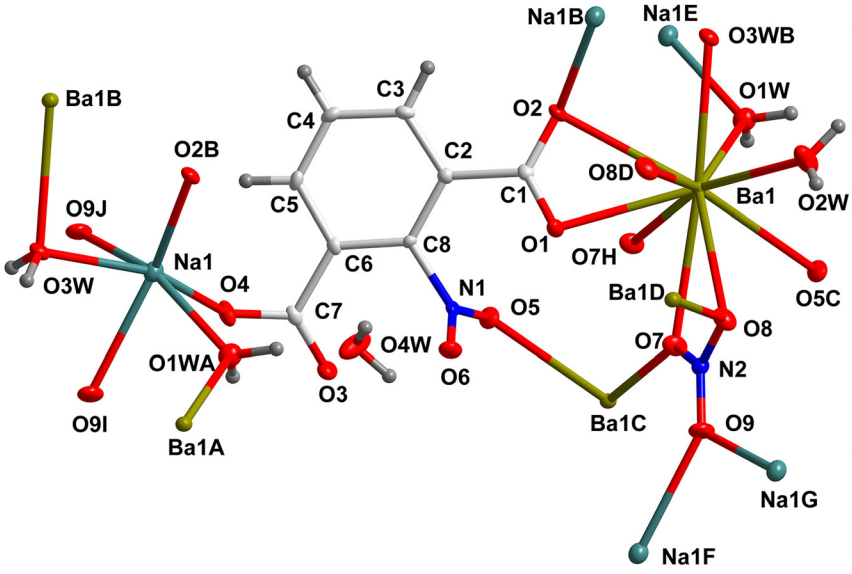
Crystal structure of poly[di-μ$_2$-aqua-aqua-nitrato-κ^2*O,O′*-(μ$_3$-2-nitroisophthalato-κ^4*O,O′:O″:O‴*) barium(II)natrium(II)] monohydrate, $C_8H_{11}BaN_2NaO_{13}$



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Abstract

$C_8H_{11}BaN_2NaO_{13}$, triclinic, $P\bar{1}$ (no. 2), a = 7.4892(6) Å, b = 9.9140(9) Å, c = 11.3274(7) Å, α = 106.813(7)°, β = 98.508(6)°, γ = 107.024(7)°, V = 744.53(11) Å^3, Z = 2, $R_{gt}(F)$ = 0.0316, $wR_{ref}(F^2)$ = 0.0663, T = 292.6(6) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.26 × 0.16 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.77 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	27.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5469, 3208, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2905
$N(param)_{refined}$:	248
Programs:	CrysAlis^{PRO} [1], Olex2 [2], Shelx [3, 4]

Source of materials

A mixture of 0.0261 g $Ba(NO_3)_2$ (0.10 mmol), 0.0211 g 2-nitrobenzene-1,3-dicarboxylic acid (0.10 mmol), 0.008 g NaOH (0.20 mmol) was added to 15 mL water and stirred for 15 min, then filtered. The clear solution evaporated slowly. After 15 days, colourless crystals were obtained, washed by deionized water, and dried in air, yield 46% (based on 2-nitrobenzene-1,3-dicarboxylic acid).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ba1	0.72570 (3)	0.87498 (2)	1.06980 (2)	0.01690 (8)
C1	0.5241 (5)	0.6783 (4)	0.7761 (3)	0.0161 (8)
C2	0.3868 (5)	0.6013 (4)	0.6446 (3)	0.0167 (8)
C3	0.3309 (6)	0.4464 (4)	0.5854 (3)	0.0199 (8)
H3	0.370860	0.390036	0.629344	0.024*
C4	0.2167 (6)	0.3752 (4)	0.4619 (3)	0.0219 (9)
H4	0.178149	0.271167	0.424003	0.026*
C5	0.1600 (5)	0.4568 (4)	0.3950 (3)	0.0196 (8)
H5	0.087738	0.407297	0.310749	0.023*
C6	0.2074 (5)	0.6121 (4)	0.4494 (3)	0.0155 (8)
C7	0.1334 (5)	0.6949 (4)	0.3739 (3)	0.0192 (8)
C8	0.3204 (5)	0.6805 (4)	0.5760 (3)	0.0133 (7)
H1WA	0.569 (7)	0.794 (5)	1.271 (4)	0.049 (15)*
H1WB	0.382 (6)	0.794 (6)	1.213 (6)	0.08 (2)*
H3WA	0.134 (7)	0.377 (5)	−0.147 (4)	0.039 (14)*
H3WB	0.324 (10)	0.437 (7)	−0.149 (6)	0.10 (2)*
N1	0.3693 (5)	0.8431 (3)	0.6401 (3)	0.0182 (7)
N2	0.8448 (5)	1.2119 (3)	1.0303 (3)	0.0200 (7)
Na1	0.2277 (2)	0.51207 (16)	0.10439 (14)	0.0264 (4)
O1	0.6203 (4)	0.8153 (3)	0.8102 (2)	0.0239 (6)
O1W	0.4671 (5)	0.7566 (3)	1.2023 (3)	0.0315 (7)
O2	0.5386 (4)	0.6000 (3)	0.8440 (2)	0.0233 (6)
O2W	1.0874 (5)	0.9586 (4)	1.2265 (3)	0.0454 (9)
H2WA	1.191450	1.023720	1.227558	0.068*
H2WB	1.119071	0.929161	1.287239	0.068*
O3W	0.2432 (5)	0.3836 (3)	−0.1084 (3)	0.0226 (6)
O3	0.1202 (4)	0.8188 (3)	0.4295 (2)	0.0285 (7)
O4	0.0845 (4)	0.6295 (3)	0.2551 (2)	0.0310 (7)
O4W	0.7794 (5)	0.8825 (4)	0.3947 (4)	0.0603 (12)
H4WA	0.802036	0.967819	0.450951	0.090*
H4WB	0.888494	0.871829	0.400479	0.090*
O5	0.2789 (4)	0.8785 (3)	0.7193 (2)	0.0303 (7)
O6	0.4918 (4)	0.9311 (3)	0.6125 (3)	0.0288 (7)
O7	0.6713 (4)	1.1317 (3)	1.0091 (3)	0.0283 (7)
O8	0.9740 (4)	1.1640 (3)	1.0650 (3)	0.0294 (7)
O9	0.8891 (4)	1.3348 (3)	1.0167 (3)	0.0344 (8)

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropic (U_{iso}(H) = 1.2U_{eq}(C)) using a riding model with C–H = 0.93 Å. The H-atoms from O atoms positioned with Q peaks refined isotropically with the distance of O–H = 0.850 Å (U_{iso}(H) = 1.5U_{eq}(O)) except the distance of O1W–H1WA = 0.945 Å for freely refinement and O1W–H1WB = 0.833 Å with a dfix restraint for the reasonable hydrogen bond O1W–H1WB···O4 with the distance of H1WB···O4 = 2.547 Å.

Comment

Barium cation is adapted to coordinate with 1,3-dicarboxylic acid or the substituted benzene-1,3-dicarboxylic acids to build a variety of barium complexes [5–15].

The title compound crystallizes in the triclinic space group $P\bar{1}$ (no. 2), with the formula of $C_8H_{11}BaN_2NaO_{13}$. The asymmetric unit is made of one sodium cation, one Ba^{2+} cation, one fully deprotonated nitroisophthalato, one nitrate, three coordinated water molecules, and one crystal water molecule. The Ba^{2+} cation is ten-coordinated with nine oxygen atoms, including O1, O2 atoms from carboxyl groups, O5C atom (code C: 1 – *x*, 2 – *y*, 2 – *z*) from the nitro group O7, O7H, O8, O8D from nitrate, bridged–O1W and O2W, and O3WB (code B: 1 – *x*, 1 – *y*, 1 – *z*). The Na^+ cation is surrounded by six O atoms, O2B, bridged–O1WA (code A: *x*, *y*, –1 + *z*) and O3W, O4 from carboxy group, O9I (code I: 1 – *x*, 2 – *y*, 1 – *z*), and O9J (code J: –1 + *x*, –1 + *y*, –1 + *z*). The nitroisophthalato links Ba1, Ba1C (code C: 1 – *x*, 2 – *y*, 2 – *z*), Na1, and Na1B, combining with the nitrate (Ba1, Ba1, Ba1C, Ba1D (code D: 2 – *x*, 2 – *y*, 2 – *z*), Na1F (code F: 1 – *x*, 2 – *y*, 1 – *z*), Na1G (code G: 1 + *x*, 1 + *y*, 1 + *z*)) to generate one 3D structure. There are many complicated hydrogen bonds to reinforce this 3D structure. All the bond lengths are similar to the reported results [5–15].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. *CrysAlis^{PRO} 1.171.38.43f*; Rigaku OD: Oxfordshire, England, 2015.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. Using phases to determine the space group. *Acta Crystallogr.* 2018, *A74*, a353.
5. Bauer S., Stock N. Synthesis and characterization of four new metal 5-phosphonoisophthalates discovered by high-throughput experimentation. *J. Solid State Chem.* 2007, *180*, 3111–3120.
6. Shuai Q., Chen A., Gao S. Synthesis and structural investigation of two metal-organic frameworks constructed from alkaline earth metal cations and organic ligand 5-nitroisophthalic acid (5–NO_2–H_2BDC). *Struct. Chem.* 2007, *18*, 689–695.

7. Shuai Q., Chen S., Gao S. Structural and thermal properties of three novel alkaline earth metal coordination polymers based on 5-hydroxyisophthalate ligand. *Inorg. Chim. Acta* 2007, *360*, 1381–1387.
8. Chen S., Shuai Q., Gao S. Two three-dimensional metal-organic frameworks constructed from alkaline earth metal cations (Sr and Ba) and 5-nitroisophthalicacid–synthesis, charaterization, and thermochemistry. *Z. Anorg. Allg. Chem.* 2008, *634*, 1591–1596.
9. Wang Y.-L., Liu Q.-Y., Xu L. Syntheses, structures and photoluminescence of two 3D pillared metal-organic frameworks with unique $(4^{11}\cdot6^4)(4^{10}\cdot6^5)$ topology based on two kind of topologically nonequivalent nodes. *Inorg. Chem. Commun.* 2008, *11*, 851–854.
10. Wu C.-Y., Lin C.-H. Poly[(μ_5-5-aminoisophthalato) aquabarium]. *Acta Crystallogr.* 2011, *E67*, m1413.
11. Wu G., Wang X.-F., Wei H., Liu Z.-F. Synthesis, structure and physical properties of a barium(II) complex with 5-sulfoisophthalic acid sodium. *Chin. J. Struct. Chem.* 2011, *30*, 1095–1100.
12. Wu G., Yin F.-J., Wei H., Liu Z.-F., Wang X.-F. Synthesis, structure, and physical properties of a barium complex with 5-sulfoisophthalic acid sodium salt. *Z. Anorg. Allg. Chem.* 2011, *637*, 596–601.
13. Wu G., Feng J.-H., Wang X.-F. Synthesis, crystal structure, and physical properties of a barium(II) benzene-1,2,3-tricarboxylic acid complex. *Z. Anorg. Allg. Chem.* 2012, *638*, 1047–1052.
14. Wang J.-M. New heterometallic Cd(II)/Ba(II) coordination polymer based on isophthalic acid: synthesis, structure, and luminescent property. *Synth. React. Inorg, Met.-Org, Nano-Met. Chem.* 2015, *45*, 740–743.
15. Sokolov A. V., Vologzhanina A. V., Barabanova E. D., Stefanovich Y. S., Dorovatovskii P. V., Taydakov I. V., Alexandrov E. V. Coordination properties of hydroxyisophthalic acids: topological correlations, synthesis, structural analysis, and properties of new complexes. *Chem. Eur J.* 2021, *27*, 9180–9192.