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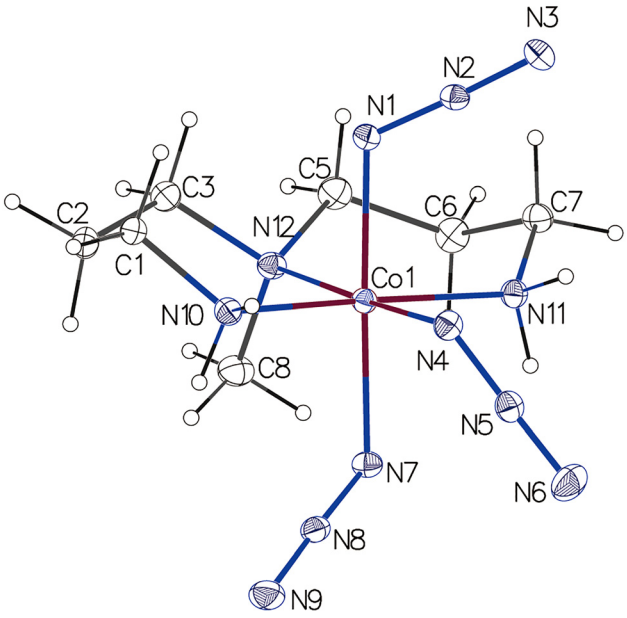
Crystal structure of tris(azido-κ^1*N*)-(*N*-(2-aminoethyl)-*N*-methyl-1,3-propanediamine-κ^3*N*,*N*′,*N*″)cobalt(III), $C_7H_{19}CoN_{12}$



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Abstract

$C_7H_{19}CoN_{12}$, monoclinic, $P2_1/c$ (no. 14), a = 7.7111(2) Å, b = 10.9909(3) Å, c = 17.5650(4) Å, β = 113.7570(10)°, V = 1362.52(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0266, $wR_{ref}(F^2)$ = 0.0718, T = 150(2) K.

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The molecular structure is shown in the Figure. 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:	Black block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.27 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22,308, 2793, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 2500
$N(param)_{refined}$:	182
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

All chemicals were commercially available and used without further purification. The title compound was synthesized with the following procedure: 0.0081 mL *N*′,*N*-bis(3-aminopropyl)methylamine (0.05 mmol) was stirred in 20 mL methanol at room temperature for 5 min. Then, 0.012 g $CoCl_2{\cdot}6H_2O$ (0.05 mmol) was added to the above solution and the resulting brown mixture was further stirred for another 30 min. Then 0.05 g $Ba(N_3)_2$ (0.23 mmol) was added and stirred for 1 h and filtered. The blocked reddish brown crystals of the title compound were obtained after three days by slow evaporation.

Experimental details

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model.

Comment

The study on azide ligand have attracted increasing interest in the field of coodination chemistry and crystal engineering, not only because the predictable structures, but also the potential applications, such as magnetism and nonlinear optics [5, 6].

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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}
C1	0.6913 (3)	0.49484 (19)	0.40998 (12)	0.0374 (4)
H1A	0.607661	0.536987	0.430168	0.045*
H1B	0.784119	0.552650	0.407652	0.045*
C2	0.7900 (3)	0.3924 (2)	0.46808 (12)	0.0420 (5)
H2A	0.883174	0.425726	0.519391	0.050*
H2B	0.856404	0.342421	0.442922	0.050*
C3	0.6518 (3)	0.3141 (2)	0.48757 (13)	0.0404 (5)
H3A	0.573095	0.367065	0.504479	0.049*
H3B	0.723541	0.262885	0.534805	0.049*
C5	0.3977 (3)	0.1765 (2)	0.45522 (13)	0.0416 (5)
H5A	0.341888	0.241306	0.475415	0.050*
H5B	0.476465	0.128493	0.502983	0.050*
C6	0.2380 (3)	0.0955 (2)	0.39753 (13)	0.0427 (5)
H6A	0.288220	0.039809	0.368589	0.051*
H6B	0.187761	0.047547	0.430368	0.051*
C7	0.0808 (3)	0.16932 (17)	0.33484 (13)	0.0359 (4)
H7A	−0.027153	0.117070	0.305801	0.043*
H7B	0.041236	0.231953	0.363356	0.043*
C8	0.6419 (3)	0.1354 (2)	0.40584 (14)	0.0462 (5)
H8A	0.562283	0.076696	0.367003	0.069*
H8B	0.712787	0.096344	0.458099	0.069*
H8C	0.727722	0.170015	0.384553	0.069*
Co1	0.37356 (3)	0.32842 (2)	0.30814 (2)	0.02593 (9)
N1	0.2817 (2)	0.43493 (15)	0.37329 (10)	0.0322 (3)
N2	0.1175 (2)	0.46119 (13)	0.34301 (9)	0.0299 (3)
N3	−0.0416 (2)	0.48680 (16)	0.31647 (11)	0.0373 (4)
N4	0.2337 (2)	0.42656 (14)	0.20873 (10)	0.0330 (3)
N5	0.1770 (2)	0.38121 (15)	0.14043 (10)	0.0351 (4)
N6	0.1192 (3)	0.3438 (2)	0.07346 (12)	0.0558 (5)
N7	0.4575 (2)	0.21847 (14)	0.24158 (10)	0.0322 (3)
N8	0.5645 (2)	0.25360 (14)	0.21172 (9)	0.0328 (3)
N9	0.6675 (3)	0.2825 (2)	0.18226 (12)	0.0492 (5)
N10	0.5809 (2)	0.44538 (14)	0.32626 (9)	0.0302 (3)
H10A	0.671699	0.405786	0.307209	0.036*
H10B	0.525849	0.515426	0.288849	0.036*
N11	0.1436 (2)	0.22663 (14)	0.27400 (9)	0.0308 (3)
H11A	0.036887	0.277338	0.236929	0.037*
H11B	0.159277	0.160138	0.239279	0.037*
N12	0.5229 (2)	0.23305 (14)	0.41799 (9)	0.0315 (3)

The asymmetric unit of the title structure contains one Co(III) atom, one *N′*,*N*-bis(3-aminopropyl)methylamine unit and three azide anions. The central Co(III) atom exhibits a distorted octahedral coordination geometry and is accomplished by three N atoms (N10, N11, N12) from *N′*,*N*-bis(3-aminopropyl) methylamine unit and three N atoms from azide anions (see the Figure). The bond lengths and angles are within the normal ranges and are comparable to related structures [7–9].

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

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