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Crystal structure of diaqua-monodimethylglycine-hexakis(μ_2-dimethylglycine-*O*:*O*)-μ_3-oxo-triiron(III) heptanitrate — water — dimethylgycine solvate (1:4:0.63), $Fe_3O(C_4H_9NO_2)_7(H_2O)_2(NO_3)_7 \cdot 4H_2O \cdot 0.63C_4H_9NO_2$

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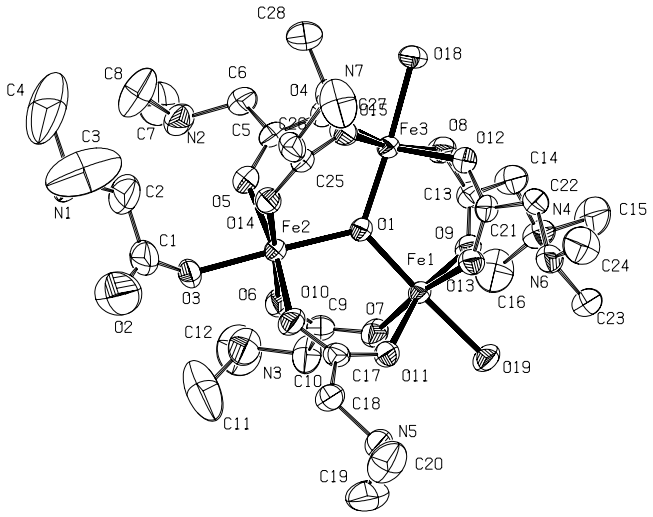

Abstract

$C_{30.20}H_{80.60}Fe_3N_{14.30}O_{43.26}$, triclinic, $P\bar{1}$ (No. 2), a = 12.424(6) Å, b = 16.565(5) Å, c = 17.627(3) Å, α = 90.59(4)°, β = 101.48(4)°, γ = 108.97(4)°, V = 3351.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.070, $wR_{ref}(F^2)$ = 0.261, T = 293 K.

Source of material

The title compound was obtained from an aqueous solution of dimethylglycine and iron nitrate in a 3:1 proportion. After many months of room temperature evaporation of the solvent small single crystals were grown. They showed hygroscopic behaviour.

Experimental details

The crystals were inserted in capillary and then tested by Laue photographic methods. The H atoms of the organic moieties were placed at calculated positions and refined as riding using SHELXL-97 [1] defaults. H atoms of the water molecules were not located.

Discussion

The title compound contains a triiron core linked by a μ_3-bridging oxide ion. Trinuclear iron compounds are of interest because oxo-bridged polynuclear Fe(III) centres perform important biological functions, such as oxygen storage and transport in a variety of proteins. It has been established that the iron atoms in the Fe_3O core are coupled antiferromagnetically both for Fe(III) and mixed valence (Fe(II)/Fe(III)), and spin frustation occurs due to the triangular geometry [2]. The magnetic Jahn-Teller effect has been observed in a few such compounds causing deviation of the clusters from an equilateral configuration [3].

In the title compound, the $(Fe_3O(dmgly)_7(H_2O)_2)^{7+}$ cation has no crystallographic imposed symmetry. Two of the Fe(III) ions are coordinated by four dimethylglycine molecules, one water molecule and the bridging oxide ion, while the third metal ion has the water molecule *trans* to the central oxide ion replaced by another dimethylglycine molecule. Each of the Fe(III) ions has a distorted octahedral geometry with distances to the basal planes of 0.192(2) Å, 0.101(2) Å and 0.202(2) Å for Fe1, Fe2, Fe3 respectively. The shortest Fe···Fe distance is 3.277(2) Å, too large to allow direct metal-metal interactions. A magnetic superexchange mechanism mainly through the central oxide ion has been used to explain the observed magnetic properties in similar compounds [2], the Fe—O1 distances range from 1.902(3) Å to 1.951(3) Å. The dimethylglycine molecules are present in neutral zwitterionic form, with the carboxylic group deprotonated and the amino group positively charged. The nitrate cations are disordered in the structure which accounts for their high displacement factors. One of the ions was refined in two different orientations. There is an extra dimethyl-glycine molecule that does not coordinate the metal core and its occupation refines to less than the unity. The oxygen atoms of four solvent water molecules were also located. These high solvent content and disorder is usual in these type of compounds [2], for instance, in the salt $[Cr_3O(OOCCH_3)_6(OH_2)_3]Cl \cdot 5H_2O$, the most studied compound of this kind, where the noncoordinated water molecules and the anions run in channels between the complex ions with deeply disordered arrangements [4?].

Table 1. Data collection and handling.

Crystal:	brown prism, size 0.29 × 0.49 × 0.49 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.50 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16128, 15307
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 10317
$N(param)_{refined}$:	837
Programs:	SHELXL-97 [1], SHELXS-97 [5], PLATON [6], ORTEPII [7]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	2*i*		–0.0900	0.1883	0.4196	0.096
H(2A)	2*i*		–0.0256	0.1397	0.3157	0.104
H(2B)	2*i*		0.0123	0.2347	0.2934	0.104
H(3A)	2*i*		–0.0007	0.3351	0.4236	0.248
H(3B)	2*i*		–0.1359	0.3092	0.4150	0.248
H(3C)	2*i*		–0.0808	0.3323	0.3421	0.248
H(4A)	2*i*		–0.2713	0.1964	0.3245	0.245
H(4B)	2*i*		–0.2426	0.1125	0.3118	0.245
H(4C)	2*i*		–0.2045	0.1883	0.2597	0.245
H(6A)	2*i*		–0.0336	0.0892	0.0534	0.055
H(6B)	2*i*		0.0616	0.0448	0.0673	0.055
H(8A)	2*i*		–0.0944	0.1148	0.1794	0.140
H(8B)	2*i*		–0.1521	0.0270	0.2118	0.140
H(8C)	2*i*		–0.1924	0.0411	0.1243	0.140
H(7A)	2*i*		–0.1451	–0.0727	0.0666	0.121
H(7B)	2*i*		–0.1309	–0.1028	0.1509	0.121
H(7C)	2*i*		–0.0311	–0.0906	0.1054	0.121
H(2)	2*i*		0.0161	0.0236	0.1912	0.063
H(3)	2*i*		0.2888	0.0039	0.2842	0.075
H(10A)	2*i*		0.4600	0.0396	0.2362	0.066
H(10B)	2*i*		0.5231	0.0647	0.3235	0.066
H(11A)	2*i*		0.4364	0.0084	0.4217	0.187
H(11B)	2*i*		0.3027	–0.0429	0.4076	0.187
H(11C)	2*i*		0.3459	0.0569	0.4055	0.187
H(12A)	2*i*		0.3473	–0.0978	0.2299	0.149
H(12B)	2*i*		0.2762	–0.1341	0.2935	0.149
H(12C)	2*i*		0.4125	–0.1070	0.3134	0.149
H(14A)	2*i*		0.4284	0.1592	–0.0361	0.057
H(14B)	2*i*		0.5003	0.2511	–0.0542	0.057
H(15A)	2*i*		0.5902	0.1303	–0.0935	0.108
H(15B)	2*i*		0.7178	0.1656	–0.0438	0.108
H(15C)	2*i*		0.6608	0.2284	–0.0876	0.108
H(16A)	2*i*		0.5127	0.0731	0.0378	0.113
H(16B)	2*i*		0.5933	0.1339	0.1103	0.113
H(16C)	2*i*		0.6477	0.0922	0.0545	0.113
H(4)	2*i*		0.6547	0.2372	0.0390	0.053
H(18A)	2*i*		0.5311	0.4795	0.4423	0.054
H(18B)	2*i*		0.5374	0.4030	0.4925	0.054
H(19A)	2*i*		0.7228	0.3968	0.5543	0.128
H(19B)	2*i*		0.8254	0.4276	0.5105	0.128
H(19C)	2*i*		0.7120	0.3519	0.4731	0.128
H(20A)	2*i*		0.7081	0.5469	0.5572	0.119
H(20B)	2*i*		0.6969	0.5899	0.4785	0.119
H(20C)	2*i*		0.8143	0.5761	0.5165	0.119
H(5)	2*i*		0.7120	0.4756	0.4186	0.054
H(22A)	2*i*		0.6013	0.5587	0.0693	0.045
H(22B)	2*i*		0.5170	0.5895	0.1083	0.045
H(6)	2*i*		0.6489	0.5944	0.2257	0.050
H(23A)	2*i*		0.7576	0.5206	0.1799	0.082
H(23B)	2*i*		0.8347	0.6065	0.2294	0.082
H(23C)	2*i*		0.8121	0.6027	0.1385	0.082
H(24A)	2*i*		0.7165	0.7153	0.1238	0.095
H(24B)	2*i*		0.7675	0.7315	0.2135	0.095
H(24C)	2*i*		0.6354	0.7178	0.1808	0.095
H(26A)	2*i*		0.1041	0.4558	0.2649	0.049
H(26B)	2*i*		0.2375	0.5086	0.2856	0.049
H(7)	2*i*		0.2099	0.5316	0.1509	0.055
H(27A)	2*i*		0.1561	0.6520	0.1743	0.118
H(27B)	2*i*		0.2667	0.6456	0.2314	0.118
H(27C)	2*i*		0.1464	0.6184	0.2563	0.118
H(28A)	2*i*		–0.0175	0.4904	0.1596	0.105
H(28B)	2*i*		0.0304	0.4427	0.1041	0.105
H(28C)	2*i*		0.0330	0.5367	0.0908	0.105
H(29A)	2*i*	0.627(8)	0.3144	0.2210	0.5063	0.116
H(29B)	2*i*	0.627	0.3903	0.2631	0.5883	0.116
H(8)	2*i*	0.627	0.3103	0.1255	0.6349	0.098
H(30A)	2*i*	0.627	0.1242	0.0427	0.5626	0.123
H(30B)	2*i*	0.627	0.2340	0.0304	0.5398	0.123
H(30C)	2*i*	0.627	0.1712	0.0879	0.4923	0.123
H(31A)	2*i*	0.627	0.1574	0.2104	0.5890	0.188
H(31B)	2*i*	0.627	0.2549	0.2353	0.6652	0.188
H(31C)	2*i*	0.627	0.1503	0.1492	0.6565	0.188
O(42)	2*i*		–0.0202(7)	0.2766(5)	–0.0601(5)	0.125(2)
O(43)	2*i*		0.685(1)	0.185(1)	0.4925(9)	0.247(6)
O(44)	2*i*		–0.046(1)	0.206(1)	–0.2141(8)	0.228(6)
O(45)	2*i*		0.102(2)	0.733(2)	0.351(1)	0.33(1)
N(15)	2*i*		0.2553(7)	0.0009(5)	0.0894(5)	0.086(2)
O(39A)	2*i*	0.53(1)	0.356(3)	0.047(2)	0.112(2)	0.21(1)
O(40A)	2*i*	0.53	0.222(2)	–0.013(1)	0.151(1)	0.133(6)
O(41A)	2*i*	0.53	0.208(2)	–0.027(1)	0.033(1)	0.139(6)
O(39B)	2*i*	0.47	0.171(1)	–0.0670(9)	0.0771(8)	0.096(4)
O(40B)	2*i*	0.47	0.309(2)	0.070(1)	0.123(1)	0.143(7)
O(41B)	2*i*	0.47	0.277(2)	0.011(1)	0.018(1)	0.118(6)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	2*i*		0.53012(5)	0.31717(4)	0.22012(3)	0.0228(3)	0.0347(3)	0.0264(3)	0.0117(2)	0.0056(2)	0.0021(2)
Fe(2)	2*i*		0.27967(5)	0.25190(4)	0.28008(3)	0.0237(3)	0.0392(3)	0.0285(3)	0.0129(2)	0.0071(2)	0.0071(2)
Fe(3)	2*i*		0.29178(5)	0.31959(4)	0.10178(3)	0.0238(3)	0.0372(3)	0.0258(3)	0.0121(2)	0.0053(2)	0.0037(2)
O(1)	2*i*		0.3667(2)	0.2958(2)	0.2001(2)	0.024(1)	0.037(1)	0.028(1)	0.013(1)	0.007(1)	0.004(1)
O(2)	2*i*		0.1317(6)	0.2447(5)	0.4590(3)	0.111(5)	0.193(7)	0.068(3)	0.073(5)	0.029(3)	–0.011(4)
O(3)	2*i*		0.1960(3)	0.2062(2)	0.3619(2)	0.041(2)	0.068(2)	0.043(2)	0.026(2)	0.021(1)	0.027(2)
N(1)	2*i*		–0.0874(5)	0.2153(4)	0.3750(5)	0.046(3)	0.070(3)	0.132(5)	0.022(2)	0.035(3)	0.010(3)
C(1)	2*i*		0.1157(6)	0.2168(5)	0.3899(4)	0.059(3)	0.107(5)	0.063(4)	0.043(3)	0.031(3)	0.034(3)
C(2)	2*i*		0.0024(6)	0.1990(6)	0.3365(5)	0.056(4)	0.117(6)	0.109(6)	0.044(4)	0.041(4)	0.047(5)
C(3)	2*i*		–0.075(1)	0.3059(7)	0.390(1)	0.130(9)	0.091(6)	0.26(2)	0.068(6)	–0.05(1)	–0.062(8)
C(4)	2*i*		–0.215(1)	0.1737(9)	0.311(1)	0.107(7)	0.15(1)	0.27(2)	0.061(7)	0.11(1)	0.04(1)
O(4)	2*i*		0.1548(3)	0.2114(2)	0.0848(2)	0.031(2)	0.045(2)	0.040(2)	0.007(1)	0.006(1)	0.007(1)
O(5)	2*i*		0.1401(3)	0.1681(2)	0.2034(2)	0.030(2)	0.044(2)	0.042(2)	0.007(1)	0.007(1)	0.005(1)
C(5)	2*i*		0.1136(4)	0.1595(3)	0.1312(3)	0.025(2)	0.041(2)	0.043(2)	0.012(2)	0.003(2)	0.002(2)
C(6)	2*i*		0.0235(4)	0.0763(3)	0.0929(3)	0.036(2)	0.048(3)	0.047(3)	0.009(2)	0.004(2)	0.003(2)
C(8)	2*i*		–0.1270(6)	0.0539(5)	0.1674(6)	0.050(4)	0.091(5)	0.135(7)	0.011(4)	0.034(4)	–0.019(5)
C(7)	2*i*		–0.0908(7)	–0.0691(4)	0.1146(5)	0.086(5)	0.036(3)	0.103(6)	0.001(3)	0.016(4)	0.006(3)
N(2)	2*i*		–0.0372(4)	0.0214(3)	0.1469(3)	0.039(2)	0.046(2)	0.064(3)	0.004(2)	0.007(2)	0.008(2)
O(7)	2*i*		0.5196(3)	0.2020(2)	0.2576(2)	0.033(2)	0.040(2)	0.055(2)	0.017(1)	0.012(1)	0.014(1)
O(6)	2*i*		0.3491(3)	0.1566(2)	0.2949(2)	0.036(2)	0.046(2)	0.047(2)	0.019(1)	0.015(1)	0.014(1)
C(9)	2*i*		0.4388(4)	0.1471(3)	0.2796(3)	0.037(2)	0.043(2)	0.040(2)	0.019(2)	0.007(2)	0.011(2)
N(3)	2*i*		0.3554(4)	–0.0045(3)	0.3093(3)	0.058(3)	0.048(2)	0.094(4)	0.027(2)	0.026(3)	0.032(2)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(10)	2*i*		0.4520(5)	0.0594(3)	0.2861(4)	0.054(3)	0.045(3)	0.082(4)	0.026(2)	0.033(3)	0.022(3)
C(11)	2*i*		0.361(1)	0.0053(7)	0.3934(6)	0.18(1)	0.123(7)	0.128(7)	0.089(7)	0.101(7)	0.071(6)
C(12)	2*i*		0.3471(9)	–0.0940(4)	0.2843(6)	0.120(7)	0.046(3)	0.139(8)	0.030(4)	0.041(6)	0.023(4)
O(9)	2*i*		0.5306(3)	0.2696(2)	0.1149(2)	0.030(1)	0.047(2)	0.032(2)	0.015(1)	0.008(1)	–0.004(1)
O(8)	2*i*		0.3620(3)	0.2600(2)	0.0356(2)	0.035(2)	0.056(2)	0.035(2)	0.022(1)	0.006(1)	–0.002(1)
C(13)	2*i*		0.4594(4)	0.2493(3)	0.0522(3)	0.033(2)	0.034(2)	0.037(2)	0.014(2)	0.009(2)	0.001(2)
C(14)	2*i*		0.4914(4)	0.2112(4)	–0.0141(3)	0.045(3)	0.065(3)	0.039(2)	0.028(2)	0.006(2)	–0.011(2)
C(15)	2*i*		0.6465(6)	0.1776(5)	–0.0600(4)	0.066(4)	0.098(5)	0.072(4)	0.045(3)	0.032(3)	–0.009(3)
C(16)	2*i*		0.5874(7)	0.1158(4)	0.0573(5)	0.079(4)	0.056(3)	0.107(6)	0.037(3)	0.031(4)	0.020(3)
N(4)	2*i*		0.6008(4)	0.1910(3)	0.0094(2)	0.040(2)	0.049(2)	0.046(2)	0.019(2)	0.004(2)	–0.009(2)
O(11)	2*i*		0.5736(3)	0.3690(2)	0.3307(2)	0.031(1)	0.054(2)	0.027(1)	0.013(1)	0.008(1)	–0.003(1)
O(10)	2*i*		0.4025(3)	0.3305(2)	0.3675(2)	0.028(2)	0.069(2)	0.034(2)	0.013(2)	0.007(1)	–0.006(2)
C(17)	2*i*		0.5089(4)	0.3709(3)	0.3763(2)	0.034(2)	0.045(2)	0.027(2)	0.018(2)	0.005(2)	0.001(2)
C(18)	2*i*		0.5617(4)	0.4324(4)	0.4483(3)	0.035(2)	0.068(3)	0.031(2)	0.018(2)	0.006(2)	–0.010(2)
C(19)	2*i*		0.7422(7)	0.4054(6)	0.5042(5)	0.063(4)	0.121(6)	0.077(5)	0.045(4)	0.000(4)	0.015(4)
C(20)	2*i*		0.7309(6)	0.5525(5)	0.5081(4)	0.057(4)	0.089(5)	0.073(4)	0.005(3)	0.010(3)	–0.036(4)
N(5)	2*i*		0.6902(3)	0.4678(3)	0.4651(2)	0.034(2)	0.065(3)	0.032(2)	0.011(2)	0.007(2)	–0.008(2)
O(12)	2*i*		0.4158(3)	0.4308(2)	0.0894(2)	0.031(2)	0.043(2)	0.036(2)	0.009(1)	0.008(1)	0.009(1)
O(13)	2*i*		0.5637(3)	0.4384(2)	0.1890(2)	0.034(2)	0.033(1)	0.035(2)	0.008(1)	0.005(1)	0.007(1)
C(21)	2*i*		0.5130(4)	0.4689(3)	0.1332(2)	0.031(2)	0.034(2)	0.035(2)	0.011(2)	0.012(2)	0.003(2)
C(22)	2*i*		0.5731(4)	0.5595(3)	0.1168(3)	0.037(2)	0.040(2)	0.036(2)	0.013(2)	0.008(2)	0.010(2)
N(6)	2*i*		0.6717(4)	0.6068(3)	0.1800(2)	0.040(2)	0.043(2)	0.037(2)	0.007(2)	0.008(2)	0.006(2)
C(23)	2*i*		0.7787(5)	0.5819(4)	0.1822(3)	0.035(2)	0.067(3)	0.057(3)	0.013(2)	0.005(2)	0.017(3)
C(24)	2*i*		0.7003(6)	0.7014(3)	0.1740(4)	0.072(4)	0.035(3)	0.069(4)	0.005(3)	0.009(3)	0.002(2)
O(14)	2*i*		0.2085(3)	0.3462(2)	0.2716(2)	0.036(2)	0.049(2)	0.038(2)	0.024(1)	0.014(1)	0.011(1)
O(15)	2*i*		0.2168(3)	0.3912(2)	0.1526(2)	0.043(2)	0.052(2)	0.033(2)	0.029(1)	0.012(1)	0.008(1)
C(25)	2*i*		0.2013(3)	0.3975(3)	0.2198(2)	0.025(2)	0.041(2)	0.032(2)	0.013(2)	0.007(2)	0.006(2)
C(26)	2*i*		0.1742(4)	0.4748(3)	0.2442(3)	0.048(2)	0.050(2)	0.038(2)	0.028(2)	0.019(2)	0.010(2)
N(7)	2*i*		0.1571(4)	0.5303(3)	0.1805(3)	0.053(2)	0.050(2)	0.054(2)	0.033(2)	0.026(2)	0.018(2)
C(27)	2*i*		0.1840(7)	0.6195(4)	0.2136(5)	0.111(5)	0.057(3)	0.097(5)	0.049(4)	0.054(4)	0.019(3)
C(28)	2*i*		0.0407(5)	0.4971(5)	0.1293(4)	0.053(3)	0.096(5)	0.071(4)	0.040(3)	0.009(3)	0.032(3)
O(16)	2*i*	0.627(8)	0.521(1)	0.2239(8)	0.502(1)	0.107(9)	0.109(9)	0.29(2)	0.022(7)	0.00(1)	0.09(1)
O(17)	2*i*	0.627	0.453(1)	0.108(1)	0.5689(8)	0.16(1)	0.16(1)	0.14(1)	0.089(9)	0.035(9)	0.029(9)
C(29)	2*i*	0.627	0.355(1)	0.2096(9)	0.556(1)	0.072(8)	0.083(8)	0.14(1)	0.025(7)	0.023(8)	0.009(8)
N(8)	2*i*	0.627	0.268(1)	0.1445(7)	0.5953(5)	0.093(7)	0.091(7)	0.052(5)	0.030(6)	0.001(5)	–0.002(5)
C(30)	2*i*	0.627	0.193(1)	0.0700(8)	0.5430(7)	0.094(9)	0.072(7)	0.079(8)	0.022(6)	0.027(7)	0.006(6)
C(31)	2*i*	0.627	0.202(2)	0.189(1)	0.6294(8)	0.13(1)	0.17(2)	0.062(8)	0.03(1)	0.017(9)	–0.001(9)
C(32)	2*i*	0.627	0.444(2)	0.174(1)	0.545(1)	0.19(2)	0.10(1)	0.13(1)	0.10(1)	0.05(1)	0.042(9)
O(18)	2*i*		0.2034(3)	0.3494(2)	0.0001(2)	0.037(2)	0.058(2)	0.029(2)	0.020(1)	0.002(1)	0.008(1)
O(19)	2*i*		0.7081(3)	0.3443(2)	0.2378(2)	0.026(1)	0.055(2)	0.043(2)	0.016(1)	0.008(1)	–0.001(1)
O(20)	2*i*		0.7889(3)	0.3066(3)	0.1146(3)	0.043(2)	0.093(3)	0.068(3)	0.029(2)	0.007(2)	–0.016(2)
O(21)	2*i*		0.8860(6)	0.2320(4)	0.0817(4)	0.104(4)	0.123(5)	0.130(5)	0.064(4)	0.053(4)	0.003(4)
O(22)	2*i*		0.9431(5)	0.3020(4)	0.1921(4)	0.060(3)	0.119(5)	0.104(4)	0.025(3)	–0.014(3)	0.018(4)
N(9)	2*i*		0.8746(4)	0.2811(3)	0.1293(3)	0.036(2)	0.066(3)	0.081(4)	0.018(2)	0.019(2)	0.008(3)
N(10)	2*i*		0.9212(4)	0.5199(4)	0.3652(3)	0.043(2)	0.071(3)	0.075(3)	0.006(2)	0.022(2)	–0.013(3)
O(23)	2*i*		0.8190(4)	0.4764(3)	0.3489(3)	0.046(2)	0.099(3)	0.066(3)	–0.001(2)	0.018(2)	–0.021(2)
O(24)	2*i*		0.9851(5)	0.5151(5)	0.3244(4)	0.076(3)	0.155(6)	0.128(5)	0.022(4)	0.060(4)	–0.024(4)
O(25)	2*i*		0.9546(7)	0.5662(6)	0.4252(5)	0.095(5)	0.202(8)	0.158(7)	–0.034(5)	0.049(5)	–0.108(6)
N(11)	2*i*		0.2931(4)	0.5692(3)	0.0191(3)	0.052(2)	0.057(2)	0.051(2)	0.029(2)	0.019(2)	0.015(2)
O(26)	2*i*		0.3580(4)	0.5804(3)	–0.0254(3)	0.076(3)	0.089(3)	0.069(3)	0.035(2)	0.044(2)	0.025(2)
O(27)	2*i*		0.3214(5)	0.6151(4)	0.0814(3)	0.086(3)	0.120(4)	0.071(3)	0.038(3)	0.025(3)	–0.015(3)
O(28)	2*i*		0.1988(4)	0.5092(3)	0.0078(3)	0.057(3)	0.052(2)	0.124(4)	0.023(2)	0.026(3)	0.021(2)
N(12)	2*i*		0.4963(5)	0.5785(3)	0.3109(3)	0.065(3)	0.071(3)	0.040(2)	0.028(2)	0.017(2)	0.004(2)
O(29)	2*i*		0.6042(4)	0.5908(4)	0.3257(3)	0.057(3)	0.107(4)	0.054(2)	0.020(2)	0.015(2)	–0.001(2)
O(30)	2*i*		0.4412(4)	0.5692(4)	0.3641(3)	0.076(3)	0.142(5)	0.049(2)	0.046(3)	0.028(2)	0.014(3)
O(31)	2*i*		0.4443(5)	0.5729(4)	0.2442(3)	0.080(3)	0.151(5)	0.048(2)	0.061(3)	0.021(2)	0.016(3)
N(13)	2*i*		0.3768(5)	0.0752(5)	0.7762(4)	0.063(3)	0.082(4)	0.085(4)	0.019(3)	0.015(3)	–0.009(4)
O(32)	2*i*		0.4046(7)	0.0342(6)	0.8277(5)	0.100(5)	0.154(6)	0.150(7)	0.041(5)	0.025(5)	0.073(6)
O(33)	2*i*		0.3338(9)	0.0365(6)	0.7145(5)	0.180(8)	0.124(6)	0.110(6)	0.041(6)	0.010(6)	–0.012(5)
O(34)	2*i*		0.388(1)	0.1473(7)	0.7814(7)	0.37(2)	0.132(7)	0.21(1)	0.157(9)	–0.09(1)	–0.058(7)
N(14)	2*i*		0.065(1)	–0.0265(8)	0.3210(7)	0.141(9)	0.17(1)	0.119(8)	0.089(8)	0.000(7)	0.020(7)
O(35)	2*i*		–0.023(1)	–0.0067(9)	0.3196(9)	0.164(9)	0.24(1)	0.31(2)	0.122(9)	0.07(1)	0.08(1)
O(36)	2*i*		0.092(2)	–0.053(1)	0.3868(8)	0.69(4)	0.38(2)	0.14(1)	0.34(3)	0.08(2)	0.08(1)
O(37)	2*i*		0.1119(6)	–0.0164(5)	0.2723(4)	0.083(4)	0.149(6)	0.100(5)	0.035(4)	0.015(4)	0.038(5)

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