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Crystal structure of iron(II)-benzenedisulfonate-triaqua-bis(*N*-methylpyrrolidone), Fe(*BDS*)$(H_2O)_3$(*NMP*)$_2$, $C_{16}H_{28}FeN_2O_{11}S_2$

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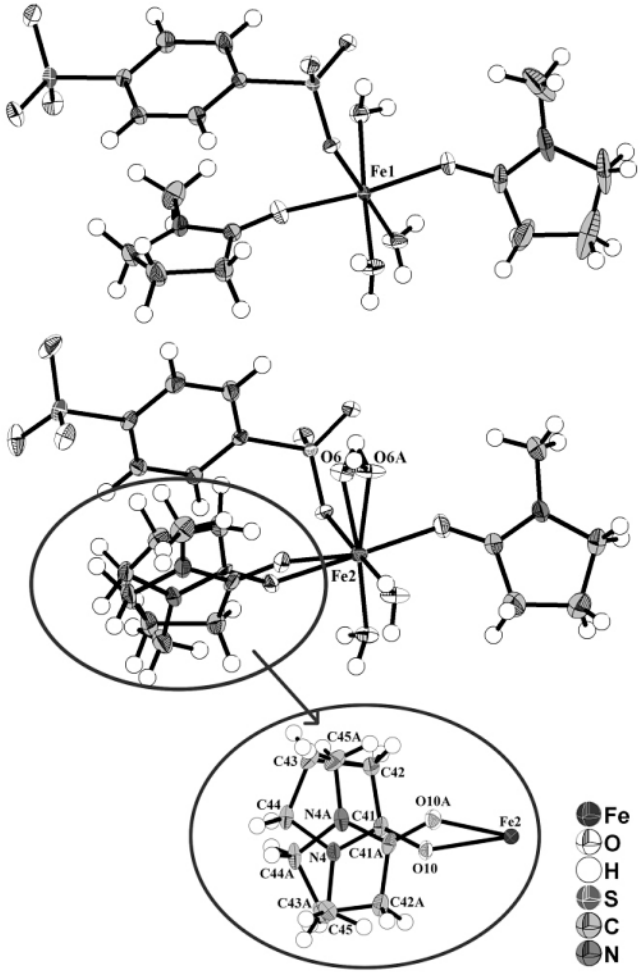

Abstract

$C_{16}H_{28}FeN_2O_{11}S_2$, monoclinic, *Pc* (no. 7), a = 14.6849(5) Å, b = 7.7856(2) Å, c = 20.1174(7) Å, β = 98.020(2)°, V = 2277.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.0346, $wR_{ref}(F^2)$ = 0.0675, T = 120 K.

Source of material

A mixture of 19 mg $FeCl_2$ (0.146 mmol), 20 mg $H_2BDS{\cdot}2\,H_2O$ (1,4-benzenedisulfonic acid dihydrate, 0.073 mmol) and 3 ml *N*-methylpyrrolidone (*NMP*) were sealed in a glass ampoule. The ampoule was placed in a resistance furnace and heated up to 120 °C within 6 h. After 36 h the furnace was slowly cooled to room temperature within 120 h and the compound Fe(*BDS*)(*NMP*)$_3$ was obtained as transparent single crystals which were separated from the excess liquid by decantation [1]. This compound is highly hygroscopic. Hydrolysis led to the title compound Fe(*BDS*)$(H_2O)_3$(*NMP*)$_2$ by absorption of water and recrystallization after several weeks.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.05×0.20×0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	67.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15297, 15671
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 13810
$N(param)_{refined}$:	693
Programs:	SHELX [2], X-RED [3], X-SHAPE[3], DIAMOND [4]

Experimental details

The structure could be successfully solved using Direct Methods of SHELXS [2]. The structure was expanded using Fourier techniques. Refinement of the structure with anisotropic displacement parameters for all atoms except for the hydrogen atoms was performed after the data were corrected for absorption effects. All calculations were performed with the SHELX program suite [2] and the data were processed with the programs X-RED [3] and X-Shape [4]. The Flack-X parameter is 0.078(8).

Discussion

One of our research projects is based on the design of coordination polymers with new sulfonic acids as linker. The number of known or commercially available sulfonic acids and the number of coordination polymers with sulfonic acids as a linker is limited [5-8]. Within a cooperation between organic and inorganic chemistry a large number of new sulfonic acids could already be synthesized [9-16]. Subsequently, on the basis of these new linkers several coordination polymers were successfully prepared [11-16]. A large number of coordination polymers are based on 1,4-benzenedisulfonic acid (*H$_2$BDS*) as a linker [15, 16]. This sulfonic acid was prepared as analogue to benzenedicarboxylic acid (*H$_2$BDC*, terephthalic acid), because it is a well-known linker in coordination and metal organic chemistry, especially in the synthesis of metal-organic frameworks (*MOFs*) and coordination polymers (*CP*) [17-26]. The hitherto known coordination polymers contain zinc, copper and rare earth elements as cation [11-16]. Now, for the first time iron could be introduced as node in the coordination polymer with the new sulfonate linker [1]. The presented compound is a hydrolysation product of Fe(*BDS*)(*NMP*)$_3$ [1]. The unit cell of Fe(*BDS*)(*NMP*)$_3$ contains two crystallographically distinguishable iron(II) ions. Both are octahedrally coordinated by six oxygen atoms originating from two *NMP* molecules, one benzenedisulfonate anion and three water molecules with Fe–O distances within normal ranges between

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2.050(2) and 2.191(2) Å [27]. Within the [FeO_6] octahedron three H_2O molecules and one BDS^{2-} anion define an equatorial plane with the apices being occupied by *NMP* molecules (Fig. 1). Since the 1,4-benzenedisulfonate anion is simply coordinated to one iron(II) ion, there is no link between the iron(II) centres resulting in a molecular structure of the composition Fe(*BDS*)$(H_2O)_3$-$(NMP)_2$. Although both iron atoms are equally coordinated, they are crystallographically different. For Fe2 a disorder of the coordinating ligands is found. One of the coordinating *NMP* ligands and one water molecule are distributed over two sites. The disorder was well resolved using split positions for the respective atoms leading to an occupancy ratio of 5.86:4.14 for the water and the NMP molecule, respectively.

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(1)	2*a*		1.073(2)	–0.210(2)	0.737(2)	0.031
H(2)	2*a*		1.078(2)	–0.059(3)	0.766(1)	0.031
H(3)	2*a*		1.072(2)	–0.273(3)	0.596(2)	0.037
H(4)	2*a*		1.082(2)	–0.170(4)	0.543(1)	0.037
H(5)	2*a*		1.059(2)	0.176(4)	0.521(1)	0.032
H(6)	2*a*		1.047(2)	0.290(3)	0.568(1)	0.032
H(21)	2*a*		0.8731	0.3593	0.7595	0.021
H(31)	2*a*		0.7124	0.3688	0.7349	0.021
H(51)	2*a*		0.7359	0.5585	0.5506	0.028
H(61)	2*a*		0.8963	0.5443	0.5748	0.028
H(141)	2*a*		1.2532	–0.2005	0.5866	0.062
H(142)	2*a*		1.2560	–0.2213	0.6666	0.062
H(151)	2*a*		1.4085	–0.2051	0.6850	0.088
H(152)	2*a*		1.4041	–0.2467	0.6063	0.088
H(161)	2*a*		1.4711	0.0431	0.6620	0.077
H(162)	2*a*		1.4381	0.0221	0.5825	0.077
H(171)	2*a*		1.3848	0.3441	0.6733	0.090
H(172)	2*a*		1.2819	0.3532	0.6347	0.090
H(173)	2*a*		1.3670	0.3458	0.5930	0.090
H(221)	2*a*		0.9016	0.0162	0.7663	0.032
H(222)	2*a*		0.8775	–0.1820	0.7487	0.032
H(231)	2*a*		0.7432	–0.1291	0.7868	0.043
H(232)	2*a*		0.7559	0.0756	0.7815	0.043
H(241)	2*a*		0.6643	–0.1452	0.6826	0.034
H(242)	2*a*		0.6513	0.0596	0.6886	0.034
H(251)	2*a*		0.6749	0.0969	0.5661	0.047
H(252)	2*a*		0.7114	–0.0896	0.5493	0.047
H(253)	2*a*		0.7778	0.0736	0.5498	0.047
H(7)	2*a*		0.618(2)	0.245(3)	0.429(1)	0.038
H(8)	2*a*		0.609(2)	0.332(4)	0.484(1)	0.038
H(9)	2*a*		0.578(2)	0.311(2)	0.297(1)	0.031
H(10)	2*a*		0.571(2)	0.454(3)	0.265(1)	0.031
H(11)	2*a*		0.590(2)	0.818(2)	0.465(2)	0.032
H(12)	2*a*		0.577(2)	0.705(3)	0.5069(8)	0.024(8)
H(81)	2*a*		0.3634	0.8565	0.2758	0.022
H(91)	2*a*		0.2134	0.8696	0.3018	0.022
H(111)	2*a*		0.3080	1.0977	0.4787	0.029
H(121)	2*a*		0.4575	1.0891	0.4519	0.026
H(321)	2*a*		0.8054	0.3223	0.4265	0.051
H(322)	2*a*		0.7923	0.3493	0.3464	0.051
H(331)	2*a*		0.9414	0.3688	0.3454	0.046
H(332)	2*a*		0.9532	0.2959	0.4210	0.046
H(341)	2*a*		0.9920	0.5538	0.4641	0.039
H(342)	2*a*		1.0039	0.6119	0.3889	0.039
H(351)	2*a*		0.9049	0.8658	0.4773	0.048
H(352)	2*a*		0.8175	0.8968	0.4215	0.048
H(353)	2*a*		0.9191	0.9094	0.4016	0.048
H(421)	2*a*	0.586(5)	0.3688	0.6784	0.4650	0.026
H(422)	2*a*	0.586(5)	0.4046	0.4923	0.4923	0.026
H(431)	2*a*	0.586(5)	0.2306	0.5894	0.4896	0.028
H(432)	2*a*	0.586(5)	0.2598	0.3899	0.4892	0.028
H(441)	2*a*	0.586(5)	0.1714	0.3857	0.3862	0.029
H(442)	2*a*	0.586(5)	0.1802	0.5906	0.3767	0.029
H(451)	2*a*	0.586(5)	0.3378	0.3970	0.2693	0.042
H(452)	2*a*	0.586(5)	0.2387	0.4864	0.2588	0.042
H(453)	2*a*	0.586(5)	0.2489	0.2902	0.2823	0.042
H(424)	2*a*	0.414(5)	0.3975	0.5334	0.2684	0.031
H(425)	2*a*	0.414(5)	0.3834	0.3335	0.2847	0.031
H(434)	2*a*	0.414(5)	0.2357	0.3766	0.2487	0.037
H(435)	2*a*	0.414(5)	0.2476	0.5819	0.2523	0.037
H(444)	2*a*	0.414(5)	0.1781	0.5692	0.3456	0.034
H(445)	2*a*	0.414(5)	0.1939	0.3645	0.3526	0.034
H(454)	2*a*	0.414(5)	0.2636	0.4439	0.4806	0.044
H(455)	2*a*	0.414(5)	0.2730	0.6463	0.4694	0.044
H(456)	2*a*	0.414(5)	0.3632	0.5315	0.4889	0.044

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*a*		1.05070(2)	0.03349(4)	0.64082(2)	0.0129(1)	0.0127(2)	0.0168(2)	0.0009(1)	0.0055(1)	0.0010(1)
O(1)	2*a*		1.0830(1)	–0.1106(2)	0.73042(9)	0.0296(9)	0.0136(8)	0.0177(9)	0.0001(7)	0.0014(7)	–0.0003(7)
O(2)	2*a*		1.0603(1)	–0.1819(2)	0.57822(9)	0.044(1)	0.0156(8)	0.019(1)	0.0020(8)	0.0172(8)	0.0023(7)
O(3)	2*a*		1.0272(1)	0.1963(2)	0.55293(9)	0.0263(9)	0.0197(9)	0.021(1)	0.0016(7)	0.0108(8)	0.0006(7)
S(1)	2*a*		1.02010(4)	0.43566(7)	0.69020(3)	0.0128(2)	0.0115(3)	0.0195(3)	0.0000(2)	0.0040(2)	0.0002(2)
S(2)	2*a*		0.58795(4)	0.46984(8)	0.61978(3)	0.0127(2)	0.0234(3)	0.0147(3)	0.0013(2)	0.0019(2)	–0.0027(2)
O(11)	2*a*		1.0397(1)	0.2547(2)	0.70655(8)	0.0181(7)	0.0120(7)	0.0188(8)	0.0030(6)	0.0034(7)	0.0017(6)
O(12)	2*a*		1.0440(1)	0.5474(2)	0.74753(9)	0.0201(8)	0.0139(8)	0.0240(9)	–0.0016(6)	0.0019(7)	–0.0049(7)
O(13)	2*a*		1.0587(1)	0.4921(2)	0.63042(9)	0.0192(8)	0.0154(8)	0.025(1)	0.0004(7)	0.0087(7)	0.0028(8)
O(14)	2*a*		0.5643(1)	0.3411(2)	0.56715(8)	0.0218(8)	0.0290(9)	0.0176(9)	–0.0020(7)	0.0014(7)	–0.0069(7)
O(15)	2*a*		0.5515(1)	0.4270(2)	0.68116(9)	0.0161(8)	0.035(1)	0.0166(8)	–0.0052(7)	0.0051(7)	–0.0013(8)
O(16)	2*a*		0.5664(1)	0.6441(2)	0.59616(9)	0.0191(8)	0.0270(9)	0.0238(9)	0.0065(7)	0.0035(7)	0.0009(8)
C(1)	2*a*		0.8986(2)	0.4487(3)	0.6692(1)	0.0143(9)	0.011(1)	0.019(1)	–0.0001(8)	0.0054(9)	–0.0012(9)
C(2)	2*a*		0.8448(2)	0.3984(3)	0.7168(1)	0.016(1)	0.022(1)	0.015(1)	–0.0002(9)	0.0015(9)	0.001(1)
C(3)	2*a*		0.7497(2)	0.4047(3)	0.7025(1)	0.017(1)	0.019(1)	0.020(1)	–0.0002(9)	0.0078(9)	0.0012(9)
C(4)	2*a*		0.7099(2)	0.4644(3)	0.6402(1)	0.0131(9)	0.016(1)	0.016(1)	0.0012(8)	0.0014(9)	–0.000(1)
C(5)	2*a*		0.7639(2)	0.5169(3)	0.5929(1)	0.020(1)	0.034(1)	0.016(1)	0.003(1)	0.003(1)	0.008(1)
C(6)	2*a*		0.8589(2)	0.5089(3)	0.6073(1)	0.019(1)	0.034(2)	0.019(1)	0.001(1)	0.009(1)	0.007(1)
O(7)	2*a*		1.1879(1)	0.1010(2)	0.6435(1)	0.0137(8)	0.031(1)	0.039(1)	0.0011(7)	0.0109(8)	0.0039(9)
C(13)	2*a*		1.2618(2)	0.0314(4)	0.6367(1)	0.012(1)	0.053(2)	0.020(1)	0.008(1)	0.003(1)	0.006(1)
C(14)	2*a*		1.2806(2)	–0.1555(4)	0.6309(2)	0.039(2)	0.059(2)	0.059(2)	0.029(2)	0.018(2)	0.014(2)
C(15)	2*a*		1.3841(2)	–0.1652(6)	0.6392(2)	0.034(2)	0.117(4)	0.066(3)	0.041(2)	–0.006(2)	–0.027(3)
C(16)	2*a*		1.4187(2)	0.0122(6)	0.6276(2)	0.016(1)	0.146(4)	0.034(2)	0.019(2)	0.013(1)	0.025(2)
C(17)	2*a*		1.3435(2)	0.3058(5)	0.6336(2)	0.039(2)	0.089(3)	0.051(2)	–0.034(2)	–0.003(2)	0.024(2)

Table 3. continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*a*		1.3388(1)	0.1202(4)	0.6330(1)	0.012(1)	0.083(2)	0.024(1)	–0.003(1)	0.0031(9)	0.012(1)
O(8)	2*a*		0.9116(1)	–0.0069(2)	0.63132(9)	0.0172(8)	0.029(1)	0.024(1)	–0.0045(7)	0.0062(7)	–0.0016(8)
C(21)	2*a*		0.8485(2)	–0.0209(3)	0.6659(1)	0.019(1)	0.015(1)	0.024(1)	–0.0002(9)	0.006(1)	–0.001(1)
C(22)	2*a*		0.8571(2)	–0.0619(4)	0.7400(1)	0.020(1)	0.036(2)	0.026(1)	0.004(1)	0.006(1)	0.006(1)
C(23)	2*a*		0.7601(2)	–0.0349(4)	0.7578(2)	0.026(1)	0.049(2)	0.034(2)	0.003(1)	0.014(1)	0.018(1)
C(24)	2*a*		0.6970(2)	–0.0343(3)	0.6904(2)	0.019(1)	0.027(1)	0.040(2)	–0.004(1)	0.012(1)	0.001(1)
C(25)	2*a*		0.7286(2)	0.0207(4)	0.5710(1)	0.027(1)	0.038(2)	0.026(1)	0.007(1)	–0.005(1)	–0.007(1)
N(2)	2*a*		0.7603(1)	–0.0071(3)	0.6416(1)	0.0132(9)	0.024(1)	0.029(1)	0.0009(8)	0.0027(9)	0.000(1)
Fe(2)	2*a*		0.58777(2)	0.55977(4)	0.38505(2)	0.0173(2)	0.0123(2)	0.0172(2)	–0.0004(1)	0.0030(1)	–0.0004(1)
O(4)	2*a*		0.6276(1)	0.3438(2)	0.44706(9)	0.046(1)	0.0144(8)	0.0168(9)	–0.0018(8)	0.0042(8)	–0.0019(7)
O(5)	2*a*		0.6003(1)	0.4109(2)	0.29938(9)	0.0293(9)	0.0150(8)	0.0172(9)	–0.0019(7)	0.0025(7)	–0.0003(7)
O(6)	2*a*	0.586(5)	0.5621(3)	0.7192(6)	0.4668(2)	0.029(2)	0.018(2)	0.016(2)	–0.005(2)	0.004(2)	–0.000(1)
O(6A)	2*a*	0.414(5)	0.6057(5)	0.7177(9)	0.4719(3)	0.054(4)	0.012(2)	0.013(3)	0.000(3)	–0.001(3)	–0.003(2)
S(3)	2*a*		0.53708(3)	0.96246(7)	0.33932(3)	0.0138(2)	0.0112(2)	0.0154(3)	0.0000(2)	0.0044(2)	–0.0001(2)
S(4)	2*a*		0.13472(4)	0.98567(8)	0.41628(3)	0.0151(2)	0.0279(3)	0.0163(3)	0.0043(2)	0.0072(2)	0.0044(3)
O(17)	2*a*		0.5512(1)	0.7840(2)	0.32092(8)	0.0183(8)	0.0120(7)	0.0177(8)	0.0021(6)	0.0038(7)	–0.0027(6)
O(18)	2*a*		0.5990(1)	1.0172(2)	0.39912(8)	0.0171(8)	0.0143(7)	0.0213(9)	–0.0019(6)	0.0022(7)	–0.0035(7)
O(19)	2*a*		0.5380(1)	1.0785(2)	0.28307(8)	0.0229(8)	0.0173(8)	0.0181(8)	–0.0010(6)	0.0071(7)	0.0031(7)
O(20)	2*a*		0.1320(1)	0.8396(2)	0.46105(9)	0.0233(9)	0.032(1)	0.022(1)	0.0041(7)	0.0116(7)	0.0096(8)
O(21)	2*a*		0.0718(1)	0.9682(3)	0.35468(9)	0.0160(8)	0.056(1)	0.0174(9)	0.0004(8)	0.0049(7)	0.0074(9)
O(22)	2*a*		0.1266(1)	1.1483(2)	0.4500(1)	0.0284(9)	0.029(1)	0.039(1)	0.0084(8)	0.0196(9)	–0.0015(9)
C(7)	2*a*		0.4247(2)	0.9721(3)	0.3615(1)	0.014(1)	0.011(1)	0.015(1)	0.0017(8)	0.0028(8)	0.0014(9)
C(8)	2*a*		0.3520(2)	0.9062(3)	0.3169(1)	0.017(1)	0.024(1)	0.017(1)	–0.0006(9)	0.0061(9)	–0.006(1)
C(9)	2*a*		0.2631(2)	0.9130(3)	0.3324(1)	0.015(1)	0.021(1)	0.019(1)	–0.0018(9)	0.0038(9)	–0.005(1)
C(10)	2*a*		0.2474(2)	0.9837(3)	0.3930(1)	0.015(1)	0.018(1)	0.014(1)	0.0039(8)	0.0066(9)	0.0042(9)
C(11)	2*a*		0.3194(2)	1.0490(3)	0.4373(1)	0.021(1)	0.034(1)	0.018(1)	0.005(1)	0.006(1)	–0.008(1)
C(12)	2*a*		0.4080(2)	1.0436(3)	0.4215(1)	0.016(1)	0.032(1)	0.018(1)	0.000(1)	0.0027(9)	–0.007(1)
O(9)	2*a*		0.7226(1)	0.6440(2)	0.3934(1)	0.0167(8)	0.0183(9)	0.073(2)	–0.0007(7)	–0.0105(9)	0.002(1)
C(31)	2*a*		0.8004(2)	0.5782(3)	0.3990(1)	0.019(1)	0.026(1)	0.027(1)	0.001(1)	–0.002(1)	–0.002(1)
C(32)	2*a*		0.8239(2)	0.3932(4)	0.3898(2)	0.027(2)	0.028(2)	0.071(2)	–0.001(1)	–0.000(2)	–0.005(2)
C(33)	2*a*		0.9257(2)	0.3887(4)	0.3911(2)	0.032(2)	0.039(2)	0.043(2)	0.009(1)	0.000(1)	–0.005(1)
C(34)	2*a*		0.9606(2)	0.5627(4)	0.4173(2)	0.018(1)	0.040(2)	0.040(2)	0.004(1)	0.005(1)	–0.001(1)
C(35)	2*a*		0.8800(2)	0.8497(3)	0.4299(2)	0.019(1)	0.031(2)	0.044(2)	–0.007(1)	0.002(1)	–0.005(1)
N(3)	2*a*		0.8776(1)	0.6671(3)	0.4139(1)	0.0155(9)	0.030(1)	0.027(1)	–0.0032(8)	0.0023(8)	–0.004(1)
O(10)	2*a*	0.586(5)	0.4461(2)	0.4909(4)	0.3644(2)	0.014(2)	0.023(2)	0.027(2)	–0.005(1)	0.006(2)	–0.008(2)
C(41)	2*a*	0.586(5)	0.3723(7)	0.501(1)	0.3887(4)	0.013(3)	0.007(2)	0.027(5)	0.004(2)	0.005(3)	–0.004(3)
C(42)	2*a*	0.586(5)	0.3602(3)	0.5530(6)	0.4587(2)	0.017(2)	0.026(2)	0.021(2)	–0.001(2)	0.002(2)	0.001(2)
C(43)	2*a*	0.586(5)	0.2612(4)	0.5007(7)	0.4653(3)	0.023(3)	0.026(3)	0.024(3)	0.001(2)	0.013(2)	0.002(2)
C(44)	2*a*	0.586(5)	0.2143(3)	0.4845(6)	0.3917(3)	0.012(2)	0.026(2)	0.036(3)	0.001(2)	0.005(2)	0.003(2)
C(45)	2*a*	0.586(5)	0.2782(5)	0.4035(8)	0.2858(4)	0.022(3)	0.036(4)	0.025(3)	–0.004(2)	–0.001(2)	–0.004(3)
N(4)	2*a*	0.586(5)	0.2917(2)	0.4580(5)	0.3552(2)	0.013(2)	0.022(2)	0.025(2)	–0.004(1)	0.003(2)	–0.003(2)
O(10A)	2*a*	0.414(5)	0.4616(3)	0.5103(6)	0.4023(3)	0.016(2)	0.025(2)	0.026(3)	0.004(2)	0.002(2)	–0.002(2)
C(41A)	2*a*	0.414(5)	0.385(1)	0.490(2)	0.3691(7)	0.014(5)	0.016(4)	0.033(7)	0.004(3)	0.009(4)	–0.001(5)
C(42A)	2*a*	0.414(5)	0.3646(4)	0.4520(8)	0.2944(3)	0.018(3)	0.026(3)	0.035(4)	–0.002(2)	0.008(3)	–0.002(3)
C(43A)	2*a*	0.414(5)	0.2622(7)	0.473(1)	0.2770(6)	0.017(4)	0.040(5)	0.033(5)	0.002(4)	–0.006(3)	–0.008(5)
C(44A)	2*a*	0.414(5)	0.2234(4)	0.4754(9)	0.3443(4)	0.011(3)	0.031(4)	0.043(5)	–0.005(2)	0.002(3)	0.004(3)
C(45A)	2*a*	0.414(5)	0.3010(6)	0.534(1)	0.4638(4)	0.032(5)	0.033(4)	0.026(4)	0.001(4)	0.015(4)	0.007(3)
N(4A)	2*a*	0.414(5)	0.3059(4)	0.5047(7)	0.3930(4)	0.015(3)	0.017(3)	0.036(4)	0.000(2)	0.001(3)	0.004(3)

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