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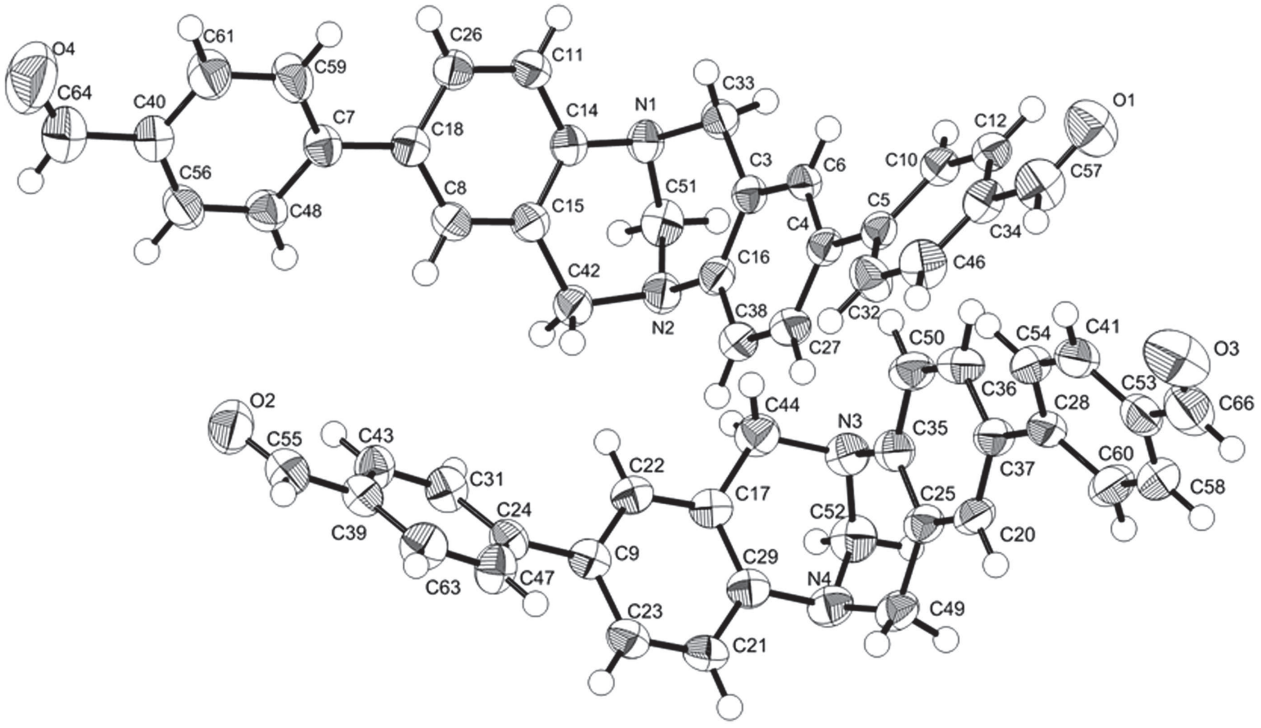
Feng Qiu, Yu Wan, Shu-ying Huang, Qiu-ju Zhou, Hao Cui, Yan-huan Chu, Sheng-Liang Zhou, Xiang-en Han* and Hui Wu*

Crystal structure of 1,1′-diformyl-4,4′-(6*H*,12*H*-5,11-methano-dibenzo[*b*,*f*][*11*,*5*]diazocine-2,8-diyl)dibenzene, $C_{29}H_{22}N_2O_2$



DOI 10.1515/ncrs-2015-0193
Received December 15, 2015; accepted January 11, 2016; available online February 16, 2016

***Corresponding authors: Xiang-en Han,** Institut School of Chemical Engineering and Technology, China University of Mining and Technology, Xuzhou 221116, Jiansu Province, P. R. China; and **Hui Wu,** Institut School of Chemistry and Chemical Engineering, Jiangsu Normal University, Xuzhou 221116, Jiangu Province, P. R. China, e-mail: xiangenh@163.com (X. Han); wuhui72@126.com (H. Wu)

Feng Qiu and Yan-huan Chu: School of Chemical Engineering and Technology, China University of Mining and Technology, Xuzhou 221116, Jiansu Province, P. R. China

Yu Wan, Shu-ying Huang, Qiu-ju Zhou, Hao Cui and Sheng-Liang Zhou: School of Chemistry and Chemical Engineering, Jiangsu Normal University, Xuzhou 221116, Jiangu Province, P. R. China

Abstract

$C_{29}H_{22}N_2O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 22.4995(6)$ Å, $b = 9.9370(3)$ Å, $c = 21.6466(6)$ Å, $\beta = 115.941(1)°$, $V = 4352.1(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0487$, $wR_{ref}(F^2) = 0.1473$, $T = 296$ K.

CCDC no.: 1446375

Table 1: Data collection and handling.

Crystal:	Colourl, block, size 0.2×0.2×0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.83 cm^{-1}
Diffractometer, scan mode:	CCD Area Detector, ω scans
$2\theta_{max}$:	55.17°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	37098, 9973
$N(param)_{refined}$:	605
Programs:	SHELX [23]

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(6)	4*e*	0.3962	0.9650	0.4938	0.046
H(8)	4*e*	0.0977	0.6473	0.3746	0.054
H(10)	4*e*	0.5045	0.9542	0.5521	0.050
H(11)	4*e*	0.1646	1.0852	0.4376	0.053
H(12)	4*e*	0.6037	0.9383	0.6474	0.053
H(26)	4*e*	0.0971	0.9812	0.4774	0.056
H(27)	4*e*	0.4095	0.5779	0.4513	0.062
H(32)	4*e*	0.4725	0.5615	0.5653	0.068
H(33A)	4*e*	0.2780	1.0563	0.4431	0.053
H(33B)	4*e*	0.2945	1.0971	0.3823	0.053
H(38)	4*e*	0.3104	0.5940	0.3557	0.063
H(42A)	4*e*	0.1943	0.6329	0.3402	0.060
H(42B)	4*e*	0.1402	0.7010	0.2741	0.060
H(46)	4*e*	0.5723	0.5454	0.6605	0.073
H(48)	4*e*	−0.0039	0.6164	0.3665	0.063
H(51A)	4*e*	0.1618	0.9318	0.2546	0.063
H(51B)	4*e*	0.2347	0.9780	0.2752	0.063
H(56)	4*e*	−0.0847	0.5324	0.3936	0.069
H(57)	4*e*	0.6768	0.6478	0.7444	0.080
H(59)	4*e*	0.0607	0.8487	0.5341	0.077
H(61)	4*e*	−0.0217	0.7683	0.5587	0.081
H(64)[a]	4*e*	−0.1422	0.5256	0.4652	0.089
H(64A)[b]	4*e*	−0.1092	0.6097	0.5387	0.089
H(20)	4*e*	0.4773	0.5536	0.2410	0.059
H(21)	4*e*	0.2566	0.3337	0.0930	0.059
H(22)	4*e*	0.1564	0.6513	0.1712	0.054
H(23)	4*e*	0.1875	0.2669	0.1396	0.059
H(31)	4*e*	0.0506	0.5496	0.1523	0.061
H(36)	4*e*	0.4510	0.8868	0.3291	0.067
H(41)	4*e*	0.6084	0.7295	0.5225	0.067
H(43)	4*e*	−0.0094	0.4940	0.2111	0.061
H(44A)	4*e*	0.1994	0.8223	0.0944	0.065
H(44B)	4*e*	0.2431	0.8156	0.1741	0.065
H(47)	4*e*	0.1808	0.2581	0.2506	0.068
H(49A)	4*e*	0.3916	0.5455	0.1008	0.064
H(49B)	4*e*	0.3685	0.4573	0.1463	0.064
H(50)	4*e*	0.3532	0.9274	0.2347	0.065
H(52A)	4*e*	0.3232	0.7407	0.0473	0.071
H(52B)	4*e*	0.2466	0.7314	0.0232	0.071
H(54)	4*e*	0.5122	0.7704	0.4267	0.062
H(55)	4*e*	0.0309	0.2370	0.3380	0.070
H(58)	4*e*	0.6852	0.5636	0.4049	0.081
H(60)	4*e*	0.5890	0.6075	0.3087	0.074
H(63)	4*e*	0.1213	0.2048	0.3103	0.071
H(66)	4*e*	0.7524	0.5685	0.5299	0.097

[a]Occupancy factor: 0.805; [b]Occupancy factor: 0.195.

Source of material

A mixture of 2,8 diboronic 6*H*,12*H*-5-11-methano-dibenzo[b,f][1,5]diazocine (1.2 mmol), 4-iodo-benzaldehyde (2.0 mmol), $Pd(PPh_3)_4$ (10%) and 2M K_2CO_3 (10 mL) was stirred in toluene (15 mL) under the argon atmosphere at 110 °C for 24 h, separated $Pd(PPh_3)_4$ by filtered and the filtrate was concentrated to give the crude product. This solid (30 mg) was recrystallized with acetone (2 mL) and kept at room temperature for seven days to give light yellow crystals of the title compound.

Experimental details

The structure was solved using direct methods and refined by full-matrix least-squares techniques.

All hydrogen atoms were added at calculated positions and refined using a riding model. The structure shows a disorder of one of the formyl groups (at O4).

Discussion

In 1887, Julius Tröger reported the synthesis of a compound which was named Tröger's base (TB) [1]. Since then, various of TB derivatives were synthesized and applied in the field of molecular recognition [2], chiral ligands [3–5], DNA probes [6, 7], stereo selective catalysis [8–12], drug development [13, 14], CO_2 capture [15, 16], bioorganic chemistry [17–19], electroluminescent materials [20, 21] and supra molecular chemistry [22] in the last two decades. Their wide range of applications is based on their huge rigidity, V-shaped twisted configuration, N-central chirality and C_2-symmetry. The types and applications of existing TB derivatives still can not meet the fast develops and requirements of chemistry and medicine. There are two crystallographic independent molecules in the asymmetric unit. Selected bond lengths are: N(1)—C(51) = 1.32(2) Å, N(2)—C(51) = 1.54(3) Å, N(1)—C(14) = 1.59(2) Å, N(1)—C(33) = 1.407(19) Å, N(2)—C(42) = 1.44(3) Å, N(2)—C(16) = 1.327(19) Å, C(3)—C(33) = 1.36(2) Å, C(42)—C(15) = 1.450(19) Å, C(4)—C(5) = 1.494(19) Å, C(7)—C(18) = 1.48(2) Å.

Acknowledgements: We are grateful to the foundation of the National Natural Sciences Foundation of China (No. 31300067), Major Project of Natural Science Research of 15KJA180002), Priority Academic Program Development of Jiangsu Higher Education Institutions for financial support. University in Jiangsu (No. 14KJA430003).

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.20688(6)	1.0036(1)	0.35057(6)	0.0403(6)	0.0484(8)	0.0431(7)	0.0019(6)	0.0171(5)	0.0064(6)
N(2)	4*e*	0.22884(6)	0.7930(1)	0.30861(6)	0.0439(7)	0.0591(9)	0.0421(7)	−0.0034(6)	0.0192(6)	−0.0065(6)
C(3)	4*e*	0.31680(7)	0.8997(2)	0.41070(7)	0.0402(7)	0.0401(8)	0.0410(8)	−0.0006(6)	0.0218(6)	0.0023(6)
C(4)	4*e*	0.41398(7)	0.7699(2)	0.48418(7)	0.0412(7)	0.0396(8)	0.0454(8)	0.0008(6)	0.0232(6)	0.0018(7)
C(5)	4*e*	0.47746(7)	0.7592(2)	0.54694(7)	0.0423(8)	0.0397(8)	0.0451(8)	0.0029(6)	0.0241(7)	0.0044(7)
C(6)	4*e*	0.37829(7)	0.8892(2)	0.46684(7)	0.0409(7)	0.0365(8)	0.0422(8)	−0.0040(6)	0.0222(6)	−0.0017(6)
C(7)	4*e*	0.03734(7)	0.7435(2)	0.44674(7)	0.0417(8)	0.0514(9)	0.0375(8)	−0.0013(7)	0.0139(6)	0.0024(7)
C(8)	4*e*	0.11042(7)	0.7363(2)	0.38655(7)	0.0441(8)	0.0453(9)	0.0409(8)	−0.0042(7)	0.0144(7)	−0.0036(7)
C(10)	4*e*	0.51804(7)	0.8713(2)	0.57360(7)	0.0450(8)	0.0380(8)	0.0447(8)	0.0054(6)	0.0213(7)	0.0072(7)
C(11)	4*e*	0.15021(7)	0.9977(2)	0.42382(7)	0.0433(8)	0.0411(9)	0.0445(8)	−0.0012(7)	0.0152(7)	−0.0019(7)
C(12)	4*e*	0.57747(7)	0.8621(2)	0.63082(7)	0.0455(8)	0.0425(9)	0.0458(8)	0.0029(7)	0.0213(7)	0.0011(7)
C(14)	4*e*	0.16995(7)	0.9309(2)	0.37924(7)	0.0357(7)	0.0442(9)	0.0372(7)	0.0023(6)	0.0117(6)	0.0040(6)
C(15)	4*e*	0.15190(7)	0.7969(2)	0.36259(7)	0.0387(7)	0.0477(9)	0.0377(7)	−0.0004(7)	0.0131(6)	−0.0022(7)
C(16)	4*e*	0.29126(7)	0.7880(2)	0.36812(7)	0.0408(8)	0.0491(9)	0.0422(8)	−0.0016(7)	0.0215(6)	−0.0038(7)
C(18)	4*e*	0.08721(7)	0.8039(2)	0.42778(7)	0.0410(8)	0.0494(9)	0.0354(7)	−0.0012(7)	0.0137(6)	0.0011(7)
C(26)	4*e*	0.10972(7)	0.9352(2)	0.44760(7)	0.0461(8)	0.053(1)	0.0401(8)	−0.0004(7)	0.0186(7)	−0.0040(7)
C(27)	4*e*	0.38676(8)	0.6593(2)	0.44112(9)	0.0485(9)	0.0409(9)	0.065(1)	0.0051(7)	0.0254(8)	−0.0040(8)
O(1)	4*e*	0.69773(6)	0.8268(2)	0.75114(7)	0.0616(8)	0.075(1)	0.0752(9)	0.0016(7)	0.0046(7)	0.0092(8)
C(32)	4*e*	0.49888(8)	0.6376(2)	0.58129(9)	0.055(1)	0.0412(9)	0.068(1)	−0.0025(8)	0.0204(9)	0.0096(8)
C(33)	4*e*	0.27606(7)	1.0265(2)	0.39954(8)	0.0411(8)	0.0410(9)	0.0502(8)	−0.0008(6)	0.0191(7)	0.0018(7)
C(34)	4*e*	0.59852(8)	0.7400(2)	0.66404(8)	0.0481(9)	0.052(1)	0.0470(9)	0.0090(7)	0.0208(7)	0.0098(8)
C(38)	4*e*	0.32708(8)	0.6685(2)	0.38403(9)	0.0499(9)	0.048(1)	0.059(1)	−0.0028(7)	0.0244(8)	−0.0160(8)
C(40)	4*e*	−0.06232(8)	0.6427(2)	0.47837(9)	0.0489(9)	0.056(1)	0.056(1)	−0.0031(8)	0.0236(8)	0.0051(8)
C(42)	4*e*	0.17680(7)	0.7187(2)	0.31851(8)	0.0449(8)	0.056(1)	0.0465(8)	−0.0071(7)	0.0187(7)	−0.0116(8)
C(46)	4*e*	0.55863(9)	0.6280(2)	0.63863(9)	0.063(1)	0.047(1)	0.066(1)	0.0075(8)	0.0210(9)	0.0209(9)
C(48)	4*e*	−0.00705(8)	0.6475(2)	0.40546(8)	0.0540(9)	0.055(1)	0.0485(9)	−0.0053(8)	0.0227(8)	−0.0054(8)
C(51)	4*e*	0.20632(8)	0.9316(2)	0.29148(8)	0.0500(9)	0.065(1)	0.0402(8)	0.0003(8)	0.0178(7)	0.0063(8)
C(56)	4*e*	−0.05582(8)	0.5976(2)	0.42158(9)	0.0531(9)	0.054(1)	0.063(1)	−0.0113(8)	0.0224(8)	−0.0057(9)
C(57)	4*e*	0.66249(9)	0.7321(2)	0.7249(1)	0.060(1)	0.064(1)	0.061(1)	0.012(1)	0.0134(9)	0.018(1)
C(59)	4*e*	0.03099(9)	0.7860(2)	0.50485(9)	0.063(1)	0.082(1)	0.0491(9)	−0.027(1)	0.0272(8)	−0.0147(9)
C(61)	4*e*	−0.01814(9)	0.7372(2)	0.51991(9)	0.072(1)	0.086(1)	0.054(1)	−0.022(1)	0.0360(9)	−0.013(1)
C(64)	4*e*	−0.1156(1)	0.5928(2)	0.4939(1)	0.068(1)	0.078(1)	0.084(1)	−0.013(1)	0.041(1)	−0.001(1)
O(4)[a]	4*e*	−0.1285(1)	0.6276(2)	0.5382(1)	0.091(1)	0.132(2)	0.109(2)	−0.028(1)	0.070(1)	−0.016(1)
C(9)	4*e*	0.16439(7)	0.4521(2)	0.16115(7)	0.0429(8)	0.0467(9)	0.0369(8)	0.0016(7)	0.0099(6)	0.0022(7)
N(3)	4*e*	0.29590(7)	0.7970(1)	0.11998(7)	0.0635(8)	0.0467(8)	0.0521(8)	0.0039(6)	0.0313(7)	0.0094(6)
C(17)	4*e*	0.22045(7)	0.6292(2)	0.12995(7)	0.0513(9)	0.0433(9)	0.0382(8)	0.0040(7)	0.0179(7)	0.0026(7)
N(4)	4*e*	0.29575(7)	0.5711(1)	0.07798(6)	0.0627(8)	0.0568(9)	0.0423(7)	0.0036(7)	0.0279(6)	−0.0002(6)
C(20)	4*e*	0.45225(8)	0.6288(2)	0.23981(8)	0.0581(9)	0.0434(9)	0.0557(9)	0.0019(7)	0.0348(8)	−0.0045(8)
C(21)	4*e*	0.23717(8)	0.3980(2)	0.10959(8)	0.0524(9)	0.0465(9)	0.0435(8)	0.0039(7)	0.0170(7)	−0.0070(7)
C(22)	4*e*	0.17727(7)	0.5870(2)	0.15635(7)	0.0492(8)	0.0455(9)	0.0400(8)	0.0056(7)	0.0181(7)	0.0001(7)
C(23)	4*e*	0.19532(7)	0.3581(2)	0.13704(8)	0.0492(9)	0.0412(9)	0.0473(9)	−0.0006(7)	0.0128(7)	−0.0011(7)
C(24)	4*e*	0.12309(7)	0.4116(2)	0.19563(7)	0.0402(8)	0.0474(9)	0.0404(8)	−0.0022(7)	0.0110(6)	0.0013(7)
C(25)	4*e*	0.39218(8)	0.6495(2)	0.18258(8)	0.0571(9)	0.0453(9)	0.0490(9)	−0.0007(7)	0.0328(8)	0.0002(7)
C(28)	4*e*	0.53930(8)	0.6926(2)	0.35673(8)	0.0556(9)	0.0373(8)	0.060(1)	−0.0054(7)	0.0322(8)	−0.0070(7)
C(29)	4*e*	0.25084(7)	0.5334(2)	0.10624(7)	0.0510(8)	0.0485(9)	0.0339(7)	0.0013(7)	0.0157(7)	−0.0005(7)
C(31)	4*e*	0.06516(7)	0.4802(2)	0.18435(8)	0.0471(8)	0.050(1)	0.0476(9)	0.0053(7)	0.0139(7)	0.0108(7)
C(35)	4*e*	0.35549(8)	0.7651(2)	0.17955(8)	0.060(1)	0.0427(9)	0.0514(9)	−0.0016(7)	0.0330(8)	0.0044(7)
C(36)	4*e*	0.43688(9)	0.8278(2)	0.29204(9)	0.070(1)	0.0424(9)	0.060(1)	0.0010(8)	0.0309(9)	−0.0081(8)
C(37)	4*e*	0.47582(8)	0.7168(2)	0.29512(8)	0.0573(9)	0.0413(9)	0.0548(9)	−0.0025(7)	0.0320(8)	−0.0035(7)
C(39)	4*e*	0.05024(8)	0.3442(2)	0.26758(8)	0.0478(8)	0.0481(9)	0.0451(8)	−0.0043(7)	0.0164(7)	0.0002(7)
C(41)	4*e*	0.60451(8)	0.7039(2)	0.47959(9)	0.061(1)	0.050(1)	0.058(1)	−0.0062(8)	0.0271(9)	−0.0098(8)
C(43)	4*e*	0.02916(8)	0.4472(2)	0.21952(8)	0.0423(8)	0.054(1)	0.0524(9)	0.0038(7)	0.0163(7)	0.0041(8)
C(44)	4*e*	0.23639(8)	0.7765(2)	0.13048(9)	0.061(1)	0.047(1)	0.059(1)	0.0067(8)	0.0302(8)	0.0075(8)
O(2)	4*e*	−0.03472(7)	0.3618(2)	0.30444(7)	0.0629(8)	0.097(1)	0.0731(8)	−0.0036(7)	0.0347(7)	−0.0046(8)
C(47)	4*e*	0.14289(8)	0.3068(2)	0.24294(9)	0.0474(9)	0.059(1)	0.060(1)	0.0114(8)	0.0199(8)	0.0156(9)
C(49)	4*e*	0.36503(8)	0.5457(2)	0.12600(8)	0.061(1)	0.057(1)	0.0502(9)	0.0014(8)	0.0329(8)	−0.0053(8)
C(50)	4*e*	0.37806(9)	0.8521(2)	0.23551(9)	0.067(1)	0.0384(9)	0.064(1)	0.0063(8)	0.0337(9)	0.0011(8)

Table 3 (continued)

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(52)	4*e*	0.28938(9)	0.7148(2)	0.06138(8)	0.073(1)	0.063(1)	0.0471(9)	0.0019(9)	0.0327(9)	0.0084(8)
C(53)	4*e*	0.65715(8)	0.6408(2)	0.4741(1)	0.052(1)	0.043(1)	0.076(1)	−0.0062(8)	0.0240(9)	−0.0046(9)
C(54)	4*e*	0.54701(8)	0.7286(2)	0.42209(8)	0.0539(9)	0.048(1)	0.060(1)	−0.0022(7)	0.0313(8)	−0.0076(8)
C(55)	4*e*	0.01421(9)	0.3083(2)	0.30740(9)	0.061(1)	0.063(1)	0.052(1)	−0.0101(9)	0.0241(8)	−0.0015(8)
C(58)	4*e*	0.65036(9)	0.6054(2)	0.4095(1)	0.058(1)	0.059(1)	0.093(1)	0.0034(9)	0.040(1)	−0.013(1)
C(60)	4*e*	0.59247(9)	0.6314(2)	0.3517(1)	0.064(1)	0.060(1)	0.069(1)	0.0000(9)	0.036(1)	−0.0142(9)
O(3)	4*e*	0.72691(8)	0.6326(2)	0.59381(9)	0.092(1)	0.087(1)	0.086(1)	−0.0040(9)	0.0020(9)	−0.003(1)
C(63)	4*e*	0.10709(8)	0.2745(2)	0.27845(9)	0.058(1)	0.058(1)	0.059(1)	0.0091(8)	0.0232(8)	0.0205(9)
C(66)	4*e*	0.7181(1)	0.6093(2)	0.5360(1)	0.061(1)	0.064(1)	0.101(2)	−0.004(1)	0.019(1)	−0.003(1)
O(4A)[b]	4*e*	−0.1617(4)	0.5390(8)	0.4624(5)	0.066(5)	0.093(6)	0.133(7)	−0.045(4)	0.049(5)	−0.035(5)

[a]Occupancy factor: 0.805; [b]Occupancy factor: 0.195.

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