Crystal structure of *N,N'*-di-5-nitrosalicylidene-(*R,R*)-1,2-cyclohexanediamine, $C_{20}H_{20}N_4O_6$

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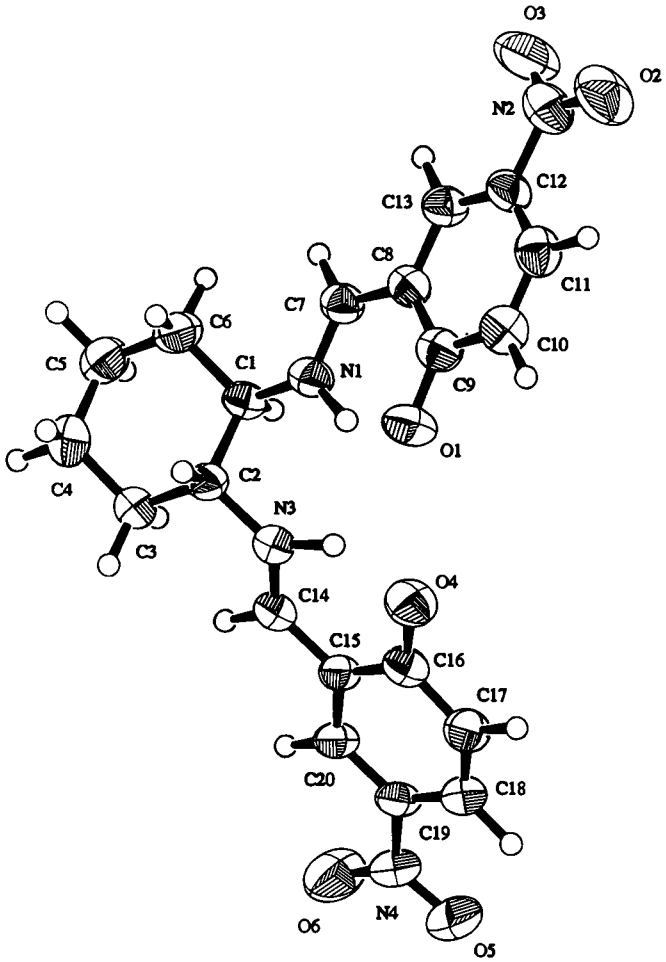

Abstract

$C_{20}H_{20}N_4O_6$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 7.2843(3)$ Å, $b = 10.8584(8)$ Å, $c = 24.407(2)$ Å, $V = 1930.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.037$, $wR_{ref}(F) = 0.034$, $T = 296$ K.

Source of material

The compound *N,N'*-di-5-nitrosalicylidene-(*R,R*)-1,2-cyclohexanediamine was prepared by condensation reaction at room temperature. The 1:2 molar mixture of (1*R*,2*R*)-diaminocyclohexane and 5-nitrosalicylaldehyde was stirred in diethyl ether for twelve hours and then extracted with ethyl acetate. The Schiff base was obtained as a yellow solid on evaporating the ethyl acetate solution and recrystallization in the same solvent yielded crystals having dimensions up to 0.35 mm^3.

Discussion

The displayed molecular entity is a roof shaped nonlinear optical chromophore in which a proton transfer has occurred between the acidic phenolic function and the basic nitrogen of the imine leading to a bis-zwitterionic form. Two intramolecular hydrogen bonds are formed between the iminium groups and anionic oxygen atoms of 4-nitrophenolate moieties. The protons involved in these internal H-bonds are located far enough from nitrogen atoms and close to oxygen atoms: N1–H11···O1 (1.21(5) Å; 1.46(5) Å; 146(4)°) and N3–H16···O4 (1.25(7) Å; 1.47(7) Å; 150(5)°), meaning that the proton transfer between phenol moieties and nitrogen atoms could be very sensitive with temperature or under laser beam. These zwitterions are piled up as herring-bone motifs alternating in opposite directions in the crystal so inducing a pseudo-centrosymmetric arrangement of NLO chromophores which results consequently in a weak macroscopic nonlinear optical efficiency as experimentally observed. Long C–H···O contacts participate to the cohesion of the alternated herringbone motifs: C11–H14···O3 (0.93(3) Å; 2.52(4) Å; 124(2)°), C1–H1···O1 (0.94(3)Å; 2.59(3) Å; 155(2)°), C14–H17···O5 (0.95(3) Å; 2.38(3) Å; 152(2)°), C18–H19···O6 (1.01(3) Å; 2.62(3) Å; 140(2)°), C20–H20···O5 (1.00(3) Å; 2.52(3) Å; 142(2)°).

Table 1. Data collection and handling.

Crystal:	yellow rectangular prism, size 0.16 × 0.24 × 0.40 mm
Wavelength:	Ag K_α radiation (0.5608 Å)
μ:	0.66 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, 180 exposures, $\Delta\varphi = 1°$
$2\theta_{max}$:	35.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1510, 1510
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 1\ \sigma(I_{obs})$, 1418
$N(param)_{refined}$:	351
Programs:	teXsan [1], SIR92 [2]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*a*	–0.519(3)	–0.053(2)	–0.0055(8)	0.049(6)
H(2)	4*a*	–0.890(3)	–0.089(2)	–0.0265(8)	0.047(6)
H(3)	4*a*	–0.833(3)	–0.002(2)	–0.1131(9)	0.055(7)
H(4)	4*a*	–0.630(3)	0.028(2)	–0.0985(8)	0.049(6)
H(5)	4*a*	–0.945(4)	0.128(2)	–0.041(1)	0.070(8)
H(6)	4*a*	–0.827(3)	0.207(2)	–0.0876(9)	0.064(7)
H(7)	4*a*	–0.725(4)	0.254(3)	0.001(1)	0.076(8)
H(8)	4*a*	–0.565(4)	0.185(2)	–0.027(1)	0.067(7)
H(9)	4*a*	–0.587(4)	0.095(2)	0.059(1)	0.081(8)
H(10)	4*a*	–0.810(4)	0.070(2)	0.0495(9)	0.068(7)
H(11)	4*a*	–0.710(6)	–0.255(4)	0.042(1)	0.17(1)
H(12)	4*a*	–0.551(3)	–0.064(2)	0.1086(9)	0.061(8)
H(13)	4*a*	–0.783(4)	–0.535(3)	0.146(1)	0.088(8)
H(14)	4*a*	–0.654(4)	–0.504(3)	0.234(1)	0.083(9)
H(15)	4*a*	–0.498(4)	–0.153(3)	0.201(1)	0.072(8)
H(16)	4*a*	–0.693(8)	–0.312(5)	–0.043(2)	0.25(2)
H(17)	4*a*	–0.635(3)	–0.153(2)	–0.1344(9)	0.055(7)
H(18)	4*a*	–0.588(3)	–0.636(2)	–0.1044(9)	0.063(7)
H(19)	4*a*	–0.463(3)	–0.647(2)	–0.1924(9)	0.068(8)
H(20)	4*a*	–0.522(3)	–0.270(2)	–0.2085(9)	0.061(7)

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

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	–0.7570(2)	–0.3706(2)	0.06880(7)	0.070(1)	0.074(1)	0.050(1)	–0.0095(9)	–0.0120(9)	–0.008(1)
O(2)	4*a*	–0.5076(4)	–0.4060(2)	0.31122(8)	0.126(2)	0.101(2)	0.061(1)	–0.004(1)	–0.009(1)	0.032(1)
O(3)	4*a*	–0.4090(4)	–0.2243(2)	0.29298(8)	0.196(3)	0.082(2)	0.069(2)	–0.022(2)	–0.055(2)	0.006(1)
O(4)	4*a*	–0.6888(2)	–0.4460(2)	–0.05080(6)	0.080(1)	0.065(1)	0.044(1)	–0.002(1)	0.0091(9)	0.0083(9)
O(5)	4*a*	–0.3432(3)	–0.5583(2)	–0.27812(7)	0.105(1)	0.075(1)	0.059(1)	0.016(1)	0.006(1)	–0.022(1)
O(6)	4*a*	–0.3967(3)	–0.3651(2)	–0.28825(8)	0.153(2)	0.065(1)	0.056(1)	0.001(2)	0.033(1)	0.003(1)
N(1)	4*a*	–0.6553(2)	–0.1499(2)	0.04837(7)	0.052(1)	0.059(1)	0.033(1)	0.001(1)	–0.0006(9)	–0.001(1)
N(2)	4*a*	–0.4901(4)	–0.3173(3)	0.2803(1)	0.099(2)	0.077(2)	0.049(2)	0.004(2)	–0.008(1)	0.015(2)
N(3)	4*a*	–0.7144(2)	–0.2052(2)	–0.06139(7)	0.044(1)	0.049(1)	0.036(1)	–0.0011(9)	–0.0010(9)	–0.0040(9)
N(4)	4*a*	–0.4012(3)	–0.4585(2)	–0.26079(8)	0.074(1)	0.060(2)	0.045(1)	0.002(1)	–0.001(1)	–0.017(1)
C(1)	4*a*	–0.6398(3)	–0.0502(2)	0.00769(9)	0.045(1)	0.051(2)	0.034(1)	–0.003(1)	0.003(1)	–0.000(1)
C(2)	4*a*	–0.7645(3)	–0.0827(2)	–0.04006(9)	0.038(1)	0.050(2)	0.038(1)	0.003(1)	0.003(1)	–0.003(1)
C(3)	4*a*	–0.7641(4)	0.0190(2)	–0.0830(1)	0.054(2)	0.054(2)	0.044(2)	0.003(1)	–0.010(1)	–0.000(1)
C(4)	4*a*	–0.8242(4)	0.1406(3)	–0.0578(1)	0.071(2)	0.051(2)	0.070(2)	0.008(1)	–0.002(2)	0.005(2)
C(5)	4*a*	–0.6941(5)	0.1755(3)	–0.0123(1)	0.078(2)	0.052(2)	0.066(2)	0.002(2)	–0.001(2)	–0.006(2)
C(6)	4*a*	–0.6853(5)	0.0766(2)	0.0311(1)	0.075(2)	0.053(2)	0.050(2)	–0.003(2)	0.001(2)	–0.007(1)
C(7)	4*a*	–0.6036(3)	–0.1407(3)	0.0988(1)	0.044(1)	0.055(2)	0.045(2)	0.001(1)	0.001(1)	–0.004(1)
C(8)	4*a*	–0.6189(3)	–0.2426(2)	0.13623(9)	0.040(1)	0.056(2)	0.039(1)	0.003(1)	0.003(1)	0.003(1)
C(9)	4*a*	–0.6989(3)	–0.3553(3)	0.1189(1)	0.043(1)	0.063(2)	0.045(2)	–0.000(1)	0.002(1)	–0.004(1)
C(10)	4*a*	–0.7142(3)	–0.4528(3)	0.1571(1)	0.060(2)	0.062(2)	0.059(2)	–0.003(2)	–0.000(1)	0.001(2)
C(11)	4*a*	–0.6486(4)	–0.4386(3)	0.2093(1)	0.062(2)	0.061(2)	0.059(2)	0.004(2)	0.005(1)	0.011(2)
C(12)	4*a*	–0.5680(3)	–0.3291(3)	0.22515(9)	0.063(2)	0.062(2)	0.036(1)	0.006(1)	0.000(1)	0.005(1)
C(13)	4*a*	–0.5538(3)	–0.2313(3)	0.1899(1)	0.056(2)	0.056(2)	0.041(2)	0.001(1)	0.002(1)	–0.002(1)
C(14)	4*a*	–0.6539(3)	–0.2212(2)	–0.1104(1)	0.045(1)	0.047(2)	0.038(2)	–0.003(1)	–0.004(1)	0.002(1)
C(15)	4*a*	–0.6036(3)	–0.3390(2)	–0.13212(8)	0.045(1)	0.042(2)	0.036(1)	–0.003(1)	–0.004(1)	–0.003(1)
C(16)	4*a*	–0.6249(3)	–0.4487(2)	–0.10119(9)	0.050(1)	0.053(2)	0.040(2)	–0.006(1)	–0.002(1)	0.002(1)
C(17)	4*a*	–0.5730(4)	–0.5607(3)	–0.1256(1)	0.076(2)	0.043(2)	0.050(2)	–0.008(1)	–0.006(1)	0.005(2)
C(18)	4*a*	–0.5001(4)	–0.5645(3)	–0.1772(1)	0.068(2)	0.050(2)	0.050(2)	0.005(2)	–0.009(1)	–0.012(2)
C(19)	4*a*	–0.4800(3)	–0.4553(2)	–0.20654(9)	0.056(1)	0.047(2)	0.033(1)	–0.004(1)	–0.005(1)	–0.006(1)
C(20)	4*a*	–0.5326(3)	–0.3449(3)	–0.18502(9)	0.056(1)	0.047(2)	0.036(2)	–0.006(1)	–0.002(1)	–0.004(1)

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Crystal structure of *N,N'*-di-5-nitrosalicylidene-(*R,S*)-1,2-cyclohexanediamine, $C_{20}H_{20}N_4O_6$

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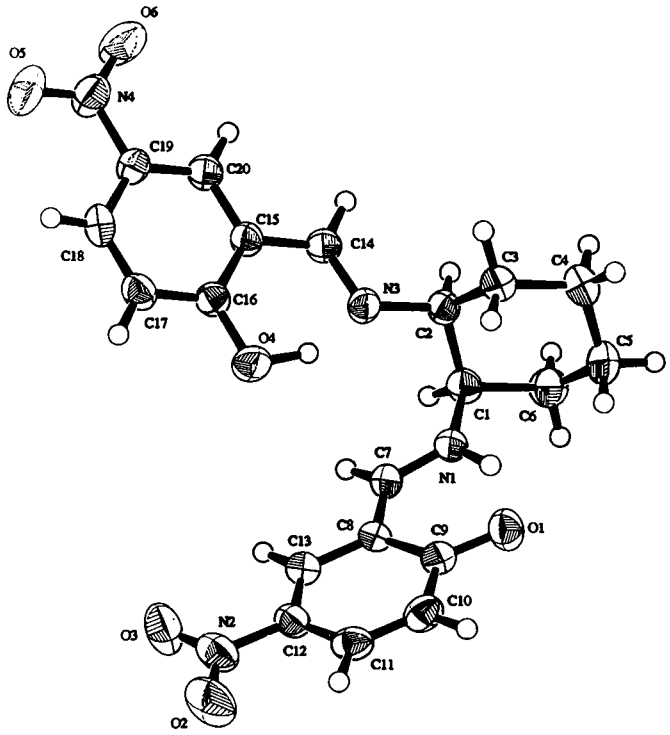

Abstract

$C_{20}H_{20}N_4O_6$, monoclinic, $P12_1/n1$ (No. 14), $a = 9.1865(4)$ Å, $b = 10.7102(7)$ Å, $c = 19.865(1)$ Å, $\beta = 92.099(3)°$, $V = 1953.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.045$, $wR_{ref}(F) = 0.042$, $T = 296$ K.

Source of material

The compound *N,N'*-di-5-nitrosalicylidene-(*R,S*)-1,2-cyclohexanediamine was prepared in the same manner as the optically active one [1], by condensation reaction at room temperature. The 1:2 molar mixture of *cis*-1,2-diaminocyclohexane and 5-nitrosalicylaldehyde was stirred in diethyl ether for twelve hours and then extracted with ethyl acetate. The Schiff base was obtained as a yellow solid on evaporating the ethyl acetate solution and recrystallization in the same solvent yielded crystals having dimensions up to 0.2 mm^3.

Discussion

This molecular compound is also a bis-dipolar roof shaped nonlinear optical chromophore displaying a simple zwitterionic structure in which two intramolecular short hydrogen bonds are observed: O4–H17···N3 (1.11(3) Å; 1.53(3) Å; 152(2)°), N1–H11···O1 (1.02(2) Å;1.65 Å; 144(2)°). Compared with the previous compound *N,N'*-di-5-nitrosalicylidene-(*R,R*)-1,2-cyclohexanediamine [1], here only one proton transfer has occurred between a 4-nitrophenol group and a imine function with the formation of an iminium site. The local centrosymmetric arrangement of these zwitterions in the cell and their three-dimensional cohesion is directed by multiple C–H···O bonds: C1–H1···O1 (0.99(2) Å; 2.53(2) Å; 156(1)°), C3–H3···O6 (1.00(2) Å; 2.64(2) Å; 164(1)°), C5–H7···O3 (0.98(2) Å; 2.64(2) Å; 160(2)°), C17–H18···O5 (0.98(2) Å; 2.54(2) Å; 158(1)°), C18–H19···O4 (0.93(2) Å; 2.57(2) Å; 167(1)°), C20–H20···O2 (0.94(2) Å; 2.37(2) Å; 157(1)°). Oxovanadium(IV) complexes derived from 5-nitrosalicylaldehyde and similar *R,S* diamines reveal the same herring-bone patterns [2].

Table 1. Data collection and handling.

Crystal:	yellow, oblique prism, size 0.16 × 0.16 × 0.29 mm
Wavelength:	Ag K_α radiation (0.5608 Å)
μ:	0.65 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, 180 exposures, $\Delta\varphi = 1$
$2\theta_{max}$:	38.5°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3485, 3485
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2487
$N(param)_{refined}$:	351
Programs:	teXsan [3], SIR92 [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.122(1)	0.091(1)	0.1823(7)	0.042(4)
H(2)	4*e*	0.190(2)	0.151(1)	0.0778(7)	0.046(4)
H(3)	4*e*	0.400(2)	0.079(1)	0.0266(8)	0.049(5)
H(4)	4*e*	0.428(2)	-0.020(1)	0.0889(8)	0.053(5)
H(5)	4*e*	0.187(2)	-0.026(1)	-0.0083(8)	0.064(5)
H(6)	4*e*	0.314(2)	-0.126(2)	-0.0031(8)	0.067(6)

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(7)	4e	0.101(2)	-0.204(2)	0.0491(9)	0.074(6)
H(8)	4e	0.241(2)	-0.205(1)	0.1018(8)	0.054(5)
H(9)	4e	0.035(2)	-0.112(1)	0.1523(8)	0.058(5)
H(10)	4e	0.008(2)	-0.015(1)	0.0935(8)	0.063(5)
H(11)	4e	0.321(2)	-0.118(2)	0.2004(9)	0.087(6)
H(12)	4e	0.288(1)	0.099(1)	0.2722(6)	0.028(4)
H(13)	4e	0.604(2)	-0.312(1)	0.3278(7)	0.049(5)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14)	4e	0.693(2)	-0.197(1)	0.4254(8)	0.055(5)
H(15)	4e	0.454(2)	0.100(1)	0.3704(7)	0.043(5)
H(16)	4e	0.319(2)	0.330(1)	0.0713(8)	0.054(5)
H(17)	4e	0.472(3)	0.206(2)	0.199(1)	0.132(8)
H(18)	4e	0.714(2)	0.434(1)	0.2707(8)	0.049(5)
H(19)	4e	0.736(2)	0.633(1)	0.2160(7)	0.048(5)
H(20)	4e	0.438(2)	0.531(1)	0.0739(7)	0.042(4)

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4e	0.4197(1)	-0.22763(9)	0.23885(6)	0.0638(8)	0.0392(7)	0.0480(8)	0.0066(6)	-0.0087(6)	-0.0080(6)
O(2)	4e	0.7275(1)	-0.0344(1)	0.50110(6)	0.0716(9)	0.107(1)	0.0585(9)	0.0009(8)	-0.0328(8)	0.0026(8)
O(3)	4e	0.6123(1)	0.1337(1)	0.47177(6)	0.081(1)	0.073(1)	0.0598(9)	-0.0042(8)	-0.0141(7)	-0.0202(8)
O(4)	4e	0.5491(1)	0.2580(1)	0.23271(6)	0.0652(8)	0.0441(7)	0.0485(8)	-0.0049(6)	-0.0186(6)	0.0132(6)
O(5)	4e	0.6912(2)	0.7948(1)	0.13398(7)	0.103(1)	0.0515(8)	0.092(1)	-0.0358(8)	-0.0129(9)	-0.0006(8)
O(6)	4e	0.5359(2)	0.7387(1)	0.05632(7)	0.096(1)	0.0523(9)	0.088(1)	-0.0132(7)	-0.0298(9)	0.0281(8)
N(1)	4e	0.2787(1)	-0.0307(1)	0.20516(6)	0.0439(8)	0.0328(8)	0.0325(8)	0.0001(6)	-0.0047(6)	0.0018(7)
N(2)	4e	0.6440(2)	0.0234(2)	0.46282(7)	0.0465(9)	0.076(1)	0.038(1)	-0.0102(9)	-0.0033(8)	0.0003(9)
N(3)	4e	0.3616(1)	0.1979(1)	0.13967(6)	0.0455(8)	0.0313(8)	0.0396(8)	-0.0014(6)	-0.0097(6)	0.0014(6)
N(4)	4e	0.6073(2)	0.7187(1)	0.10847(8)	0.062(1)	0.0394(9)	0.064(1)	-0.0031(8)	0.0008(9)	0.0013(9)
C(1)	4e	0.1794(2)	0.0317(2)	0.15649(8)	0.039(1)	0.038(1)	0.039(1)	0.0045(8)	-0.0078(8)	0.0007(8)
C(2)	4e	0.2654(2)	0.1074(1)	0.10519(8)	0.045(1)	0.0344(9)	0.036(1)	-0.0025(8)	-0.0133(8)	0.0018(8)
C(3)	4e	0.3521(2)	0.0238(2)	0.05980(9)	0.057(1)	0.045(1)	0.038(1)	-0.0092(9)	0.0005(9)	-0.0013(9)
C(4)	4e	0.2534(2)	-0.0712(2)	0.0237(1)	0.070(1)	0.054(1)	0.042(1)	-0.008(1)	-0.006(1)	-0.009(1)
C(5)	4e	0.1694(2)	-0.1481(2)	0.0733(1)	0.066(1)	0.049(1)	0.055(1)	-0.017(1)	-0.010(1)	-0.009(1)
C(6)	4e	0.0841(2)	-0.0649(2)	0.1198(1)	0.046(1)	0.057(1)	0.051(1)	-0.013(1)	-0.006(1)	0.002(1)
C(7)	4e	0.3246(2)	0.0184(2)	0.26128(8)	0.042(1)	0.031(1)	0.035(1)	0.0003(8)	0.0016(8)	-0.0029(8)
C(8)	4e	0.4252(2)	-0.0436(1)	0.30575(7)	0.0380(9)	0.0345(9)	0.0276(9)	-0.0011(7)	0.0002(7)	0.0017(7)
C(9)	4e	0.4704(2)	-0.1688(1)	0.29096(8)	0.0391(9)	0.037(1)	0.036(1)	-0.0029(8)	0.0023(8)	0.0016(8)
C(10)	4e	0.5729(2)	-0.2260(2)	0.33742(9)	0.044(1)	0.036(1)	0.052(1)	0.0028(8)	-0.0024(9)	0.0074(9)
C(11)	4e	0.6261(2)	-0.1640(2)	0.39226(9)	0.038(1)	0.053(1)	0.043(1)	-0.0044(9)	-0.0062(9)	0.014(1)
C(12)	4e	0.5812(2)	-0.0416(2)	0.40490(8)	0.0387(9)	0.050(1)	0.032(1)	-0.0062(8)	-0.0010(8)	0.0010(8)
C(13)	4e	0.4824(2)	0.0176(2)	0.36286(8)	0.044(1)	0.039(1)	0.036(1)	-0.0020(8)	0.0015(8)	0.0012(8)
C(14)	4e	0.3764(2)	0.3060(1)	0.11387(9)	0.044(1)	0.035(1)	0.038(1)	0.0049(8)	-0.0081(8)	0.0037(8)
C(15)	4e	0.4773(2)	0.3973(1)	0.14375(7)	0.0359(9)	0.0332(9)	0.035(1)	0.0013(7)	-0.0049(7)	-0.0004(8)
C(16)	4e	0.5603(2)	0.3695(1)	0.20279(8)	0.0392(9)	0.036(1)	0.036(1)	0.0043(8)	-0.0012(8)	0.0009(8)
C(17)	4e	0.6572(2)	0.4581(2)	0.23027(9)	0.045(1)	0.044(1)	0.036(1)	0.0024(8)	-0.0084(8)	-0.0025(9)
C(18)	4e	0.6722(2)	0.5714(2)	0.19946(9)	0.041(1)	0.041(1)	0.046(1)	-0.0016(8)	-0.0015(8)	-0.0140(9)
C(19)	4e	0.5910(2)	0.5975(1)	0.14103(8)	0.0402(9)	0.0294(9)	0.044(1)	0.0015(7)	0.0013(8)	-0.0008(8)
C(20)	4e	0.4947(2)	0.5131(1)	0.11334(9)	0.042(1)	0.037(1)	0.040(1)	0.0034(8)	-0.0080(9)	0.0024(8)

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