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Crystal structures of ethanol hemisolvates of (1*S*,3*R*,4*S*,8*S*,9*R*)-1*N*-boranyl-6′-methoxy-cinchonan-9-ol, $C_{20}H_{27}BN_2O_2 \cdot \frac{1}{2}C_2H_5OH$, and (1*S*,3*S*,4*S*,8*S*,9*R*)-1*N*-boranyl-10,11-didehydro-6′-methoxy-cinchonan-9-ol, $C_{20}H_{25}BN_2O_2 \cdot \frac{1}{2}C_2H_5OH$

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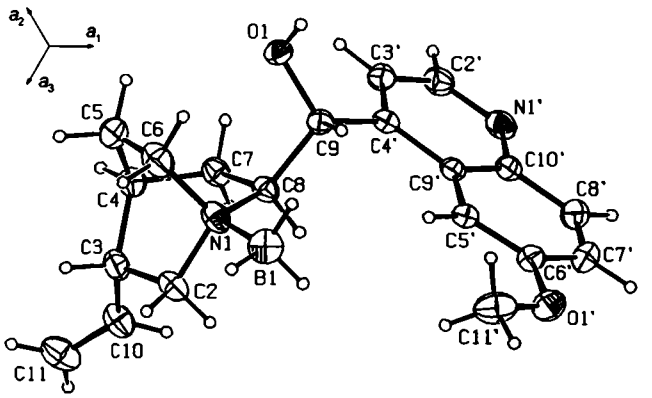

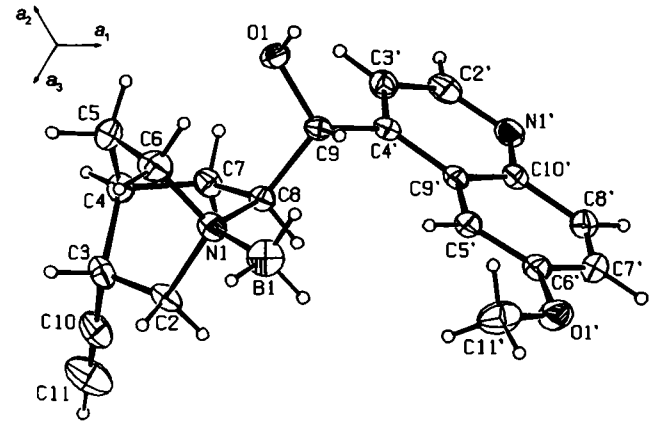

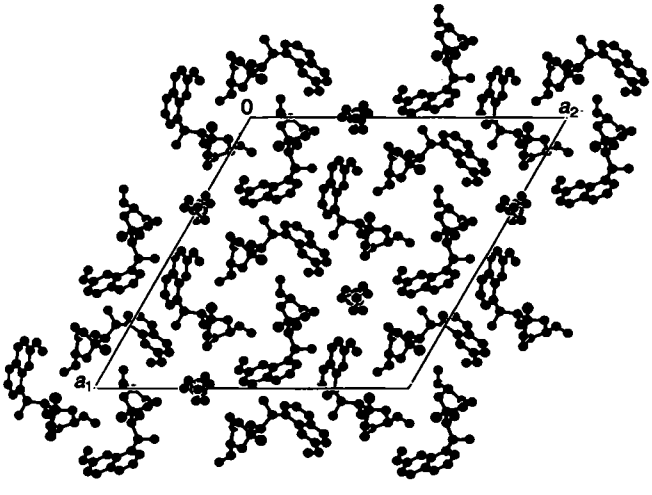

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Abstract

$C_{21}H_{30}BN_2O_{2.50}$, trigonal, *R*3 (no. 146), $a = 26.759(5)$ Å, $c = 7.431(2)$ Å, $V = 4608.1$ Å^3, $Z = 9$, $R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.094$, $T = 300$ K.

$C_{21}H_{28}BN_2O_{2.50}$, trigonal, *R*3 (no. 146), $a = 26.465(6)$ Å, $c = 7.375(2)$ Å, $V = 4473.5$ Å^3, $Z = 9$, $R_{gt}(F) = 0.039$, $wR_{ref}(F^2) = 0.062$, $T = 300$ K.

Source of materials

Compound 1: Quinine (2.24 g, 6.89 mmol) was dissolved in abs. THF (3 ml/mmol). $BH_3 \cdot$ THF (1 M, 6.9 ml) was added at 0 °C and the reaction mixture was stirred for 8 h at room temperature. Removal of the solvent and purification by column chromatography (MTBE) furnished the borane (1.88 g, 94 %), which was recrystallized from ethanol.

Compound 2: 10,11-Didehydroquinine (350 mg, 1.08 mmol) and $BH_3 \cdot$ THF (1 M, 1.1 ml) in abs. THF were allowed to react, according to the general procedure. Purification by column chromatography (MTBE/MeOH 10:1) afforded 10,11-didehydroquinineborane (310.3 mg, 85 %). Recrystallization from ethanol.

Discussion

In the group of the senior author many derivatives of *Cinchona* alkaloids were synthesized ([1,2] and references therein). Most of them crystallize in the space groups $P2_12_12_1$ or $P2_1$. But three compounds have been found which form an exception. They crystallize with higher symmetry in space group *R*3. The incorporation of solvent molecules seems to be essential for this arrangement but hydrogen bonds are probably important too. The structures of the two compounds (figure, top and middle) in which the quinuclidinium N atom is protected by the BH_3 group are presented here while the crystallographic data of the third compound, the bromoalkyne, in which the bromine atom has a nonbonded, but short contact to the N atom, was deposited ([1], compound 6c, and [2], fig. 5 ("quinidine" has to be replaced by "quinine" in the figure caption)). All three quasi isotypic compounds are derivatives of quinine.

There are three types of channels in the structure of the title compounds (figure, bottom; cell projection for compound 1). Type I (at 0,0,*z*) centered by a threefold axis is empty and is surrounded by alkaloid molecules which form a trigonal column. Type II (at ⅓,⅓,*z*) centered by a 3_1 screw axis is empty too and is surrounded by alkaloid molecules which are arranged along a right-handed helix. But the hydrogen bonds between these molecules connect molecules which are ⅓|c| apart from one another in [0001]-direction thus forming two interpenetrating left-handed helices.

Type III (at ⅓,0,z) centered by a 3_2 screw axis is the widest and contains the solvent molecules (ethanol). The short screw translation of only 2.46 Å demands that the solvent molecules are disordered and have a low occupation factor. An occupation factor of 0.25 was assumed as reasonable for two ethanol molecules per asymmetric unit which were introduced in the refinement applying several bond length and distance restraints. There is a particularity in the structure of the bromoalkyne [1,2]. The intermolecular short contact between N and Br (2.84 Å) connects molecules which differ by the symmetry operation $y-x,-x,1+z$. By this contact the trigonal column of molecules around channel I decomposes into three left-handed helices. There is some similarity of the structures presented here to the structure of quinol methanol clathrate [3]. But in the latter the solvent molecules are situated in channel I. Rosifoliol is an example of a structure crystallizing in a similar packing without solvent molecules [4].

1. (1*S*,3*R*,4*S*,8*S*,9*R*)-1*N*-Boranyl-6′-methoxy-cinchonan-9-ol ethanol hemisolvate, $C_{20}H_{27}BN_2O_2 \cdot \frac{1}{2}C_2H_5OH$

Table 1. Data collection and handling.

Crystal:	colorless hexagonal prism, size 0.63 × 0.74 × 0.96 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.76 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, 240 exposures, $\Delta\varphi = 1.5°$
$2\theta_{max}$:	52.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22063, 4023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2599
$N(param)_{refined}$:	281
Programs:	SHELXS-86 [5], SHELXL-93 [6], PLATON [7]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	9*b*		0.212(2)	0.309(2)	0.642(4)	0.13(1)
B(1)	9*b*		0.1059(2)	0.1415(2)	0.812(1)	0.089(1)
H(2)	9*b*		0.1161(2)	0.1136(2)	0.773(1)	0.134
H(3)	9*b*		0.0753(2)	0.1243(2)	0.899(1)	0.134
H(4)	9*b*		0.1389(2)	0.1737(2)	0.866(1)	0.134
H(5)	9*b*		0.0043(1)	0.0928(1)	0.666(1)	0.078
H(6)	9*b*		0.0415(1)	0.0844(1)	0.519(1)	0.078
H(7)	9*b*		−0.0328(1)	0.1257(1)	0.476(1)	0.077
H(8)	9*b*		0.0279(1)	0.2010(1)	0.267(1)	0.086
H(9)	9*b*		0.0128(2)	0.2250(1)	0.554(2)	0.116
H(10)	9*b*		0.0776(2)	0.2671(1)	0.504(2)	0.116
H(11)	9*b*		0.1011(2)	0.2396(2)	0.748(1)	0.108
H(12)	9*b*		0.0372(2)	0.1901(2)	0.779(1)	0.108
H(13)	9*b*		0.1274(1)	0.2370(1)	0.283(1)	0.071
H(14)	9*b*		0.0978(1)	0.1719(1)	0.225(1)	0.071
H(15)	9*b*		0.1331(1)	0.1524(1)	0.454(1)	0.057
H(16)	9*b*		0.1985(1)	0.2293(1)	0.659(1)	0.058
H(17)	9*b*		0.0094(1)	0.0771(1)	0.220(1)	0.093
H(18)	9*b*		−0.0964(2)	0.0678(2)	0.243(1)	0.111
H(19)	9*b*		−0.0766(2)	0.0351(2)	0.103(1)	0.111
H(20)	9*b*		0.2870(1)	0.2889(1)	0.020(1)	0.077
H(21)	9*b*		0.2298(1)	0.2943(1)	0.238(1)	0.068
H(22)	9*b*		0.2219(1)	0.1636(1)	0.670(1)	0.061
H(23)	9*b*		0.3254(1)	0.1111(1)	0.461(1)	0.088
H(24)	9*b*		0.3404(1)	0.1653(1)	0.213(1)	0.083
H(25)	9*b*		0.2296(2)	0.0688(2)	0.967(1)	0.138
H(26)	9*b*		0.1922(2)	0.0824(2)	0.835(1)	0.138
H(27)	9*b*		0.2463(2)	0.1329(2)	0.929(1)	0.138
O(21)	9*b*	0.25	0.3182(7)	−0.0059(7)	0.2359(8)	0.206(5)
H(201)	9*b*	0.25	0.3354(9)	0.0169(9)	0.3383(9)	0.309
C(21)	9*b*	0.25	0.3176(5)	0.0300(5)	0.091(1)	0.206
H(211)	9*b*	0.25	0.2969(8)	0.0482(7)	0.129(2)	0.309
H(212)	9*b*	0.25	0.3560(6)	0.0590(6)	0.066(2)	0.309
C(22)	9*b*	0.25	0.2898(6)	−0.0059(6)	−0.0777(9)	0.206
H(221)	9*b*	0.25	0.2896(8)	0.0177(8)	−0.171(1)	0.309
H(222)	9*b*	0.25	0.2515(6)	−0.0347(7)	−0.053(2)	0.309
H(223)	9*b*	0.25	0.311(1)	−0.0239(9)	−0.116(1)	0.309
O(31)	9*b*	0.25	0.3310(7)	−0.0035(7)	0.6791(7)	0.206
H(301)	9*b*	0.25	0.3102(8)	−0.0365(9)	0.736(1)	0.309
C(31)	9*b*	0.25	0.3424(5)	−0.0110(4)	0.492(1)	0.206
H(311)	9*b*	0.25	0.3061(6)	−0.0336(6)	0.434(1)	0.309
H(312)	9*b*	0.25	0.3613(9)	−0.0331(6)	0.491(3)	0.309
C(32)	9*b*	0.25	0.3785(6)	0.0446(6)	0.385(2)	0.206
H(321)	9*b*	0.25	0.3839(8)	0.0362(9)	0.266(2)	0.309
H(322)	9*b*	0.25	0.3596(9)	0.0664(7)	0.382(3)	0.309
H(323)	9*b*	0.25	0.4148(6)	0.0668(7)	0.440(2)	0.309

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)	9*b*	0.18729(8)	0.28815(7)	0.536(1)	0.062(1)	0.047(1)	0.076(1)	0.0304(9)	−0.0101(9)	−0.0130(9)
N(1)	9*b*	0.08468(9)	0.1636(1)	0.640(1)	0.049(1)	0.074(2)	0.050(1)	0.022(1)	0.003(1)	−0.008(1)
C(2)	9*b*	0.0311(1)	0.1116(1)	0.568(1)	0.047(2)	0.065(2)	0.066(2)	0.014(1)	0.004(1)	−0.004(1)
C(3)	9*b*	0.0016(1)	0.1275(1)	0.423(1)	0.045(2)	0.069(2)	0.075(2)	0.024(1)	−0.004(1)	−0.011(2)
C(4)	9*b*	0.0433(1)	0.1902(1)	0.369(1)	0.056(2)	0.065(2)	0.095(2)	0.030(2)	−0.013(2)	0.001(2)
C(5)	9*b*	0.0494(2)	0.2272(1)	0.529(2)	0.068(2)	0.069(2)	0.156(4)	0.037(2)	0.009(2)	−0.025(2)
C(6)	9*b*	0.0684(2)	0.2069(2)	0.692(1)	0.063(2)	0.093(2)	0.099(2)	0.027(2)	0.015(2)	−0.040(2)
C(7)	9*b*	0.1018(1)	0.1978(1)	0.323(1)	0.050(2)	0.062(2)	0.057(2)	0.022(1)	−0.007(1)	−0.001(1)
C(8)	9*b*	0.1275(1)	0.1847(1)	0.488(1)	0.043(1)	0.048(1)	0.045(1)	0.017(1)	0.003(1)	−0.005(1)
C(9)	9*b*	0.1880(1)	0.2357(1)	0.539(1)	0.050(2)	0.045(2)	0.048(1)	0.023(1)	−0.004(1)	−0.008(1)
C(10)	9*b*	−0.0176(1)	0.0856(1)	0.269(1)	0.059(2)	0.084(2)	0.072(2)	0.024(2)	−0.005(2)	−0.008(2)
C(11)	9*b*	−0.0682(2)	0.0605(2)	0.198(1)	0.077(2)	0.089(3)	0.079(2)	0.016(2)	−0.011(2)	0.002(2)
O(1′)	9*b*	0.26608(9)	0.09713(9)	0.730(1)	0.088(2)	0.070(1)	0.086(1)	0.045(1)	−0.002(1)	0.018(1)
N(1′)	9*b*	0.3032(1)	0.2335(1)	0.134(1)	0.055(1)	0.061(1)	0.060(1)	0.021(1)	0.016(1)	0.008(1)
C(2′)	9*b*	0.2795(1)	0.2662(1)	0.123(1)	0.059(2)	0.060(2)	0.066(2)	0.023(2)	0.011(1)	0.013(1)
C(3′)	9*b*	0.2442(1)	0.2693(1)	0.253(1)	0.054(2)	0.053(2)	0.061(2)	0.027(1)	0.007(1)	0.010(1)
C(4′)	9*b*	0.2305(1)	0.2361(1)	0.402(1)	0.041(1)	0.040(1)	0.049(1)	0.013(1)	−0.002(1)	−0.001(1)
C(5′)	9*b*	0.2460(1)	0.1647(1)	0.578(1)	0.046(2)	0.042(1)	0.060(2)	0.018(1)	0.000(1)	−0.001(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6′)	9*b*	0.2723(1)	0.1328(1)	0.589(1)	0.057(2)	0.051(2)	0.073(2)	0.027(1)	–0.005(1)	0.002(1)
C(7′)	9*b*	0.3079(1)	0.1335(1)	0.451(1)	0.069(2)	0.068(2)	0.099(2)	0.046(2)	0.005(2)	0.005(2)
C(8′)	9*b*	0.3170(1)	0.1659(1)	0.305(1)	0.063(2)	0.068(2)	0.081(2)	0.036(2)	0.015(2)	0.002(2)
C(9′)	9*b*	0.2554(1)	0.1999(1)	0.424(1)	0.037(1)	0.040(1)	0.051(1)	0.013(1)	–0.001(1)	–0.003(1)
C(10′)	9*b*	0.2917(1)	0.2010(1)	0.287(1)	0.044(1)	0.046(2)	0.064(2)	0.018(1)	0.002(1)	–0.006(1)
C(11′)	9*b*	0.2306(2)	0.0951(2)	0.877(1)	0.126(3)	0.073(2)	0.069(2)	0.044(2)	–0.007(2)	0.009(2)

2. (1*S*,3*S*,4*S*,8*S*,9*R*)-1*N*-Boranyl-10,11-didehydro-6′-methoxy-cinchonan-9-ol, ethanol hemisolvate, $C_{20}H_{25}BN_2O_2 \cdot \frac{1}{2}C_2H_5OH$

Table 4. Data collection and handling.

Crystal:	colorless hexagonal prism, size 0.30 × 0.31 × 0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.78 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, 240 exposures, $\Delta\varphi = 1.5°$
$2\theta_{max}$:	52.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21380, 3874
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2033
$N(param)_{refined}$:	281
Programs:	SHELXS-86 [5], SHELXL-93 [6], PLATON [7]

Table 5. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	9*b*		0.206(1)	0.305(1)	0.646(3)	0.13(1)
B(1)	9*b*		0.1054(2)	0.1384(2)	0.818(1)	0.092(1)
H(2)	9*b*		0.1142(2)	0.1093(2)	0.776(1)	0.138
H(3)	9*b*		0.0755(2)	0.1219(2)	0.908(1)	0.138
H(4)	9*b*		0.1399(2)	0.1706(2)	0.869(1)	0.138
H(5)	9*b*		0.0027(1)	0.0893(1)	0.683(1)	0.067
H(6)	9*b*		0.0376(1)	0.0826(1)	0.521(1)	0.067
H(7)	9*b*		–0.0361(1)	0.1282(1)	0.513(1)	0.075
H(8)	9*b*		0.0228(1)	0.2016(1)	0.286(1)	0.080
H(9)	9*b*		0.0159(1)	0.2308(1)	0.576(1)	0.104
H(10)	9*b*		0.0816(1)	0.2685(1)	0.517(1)	0.104
H(11)	9*b*		0.1031(1)	0.2377(1)	0.766(1)	0.093
H(12)	9*b*		0.0378(1)	0.1892(1)	0.797(1)	0.093

Table 5. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(13)	9*b*		0.0887(1)	0.1651(1)	0.233(1)	0.063
H(14)	9*b*		0.1227(1)	0.2321(1)	0.278(1)	0.063
H(15)	9*b*		0.13080(9)	0.1495(1)	0.456(1)	0.049
H(16)	9*b*		0.1968(1)	0.2285(1)	0.661(1)	0.052
H(17)	9*b*		–0.0676(2)	0.0246(2)	0.092(1)	0.139
H(18)	9*b*		0.2879(1)	0.2856(1)	0.018(1)	0.076
H(19)	9*b*		0.2278(1)	0.2907(1)	0.232(1)	0.066
H(20)	9*b*		0.2201(1)	0.1625(1)	0.675(1)	0.060
H(21)	9*b*		0.3258(1)	0.1087(1)	0.478(1)	0.089
H(22)	9*b*		0.3415(1)	0.1624(1)	0.225(1)	0.084
H(23)	9*b*		0.2293(2)	0.0687(1)	0.985(1)	0.137
H(24)	9*b*		0.1911(2)	0.0808(1)	0.848(1)	0.137
H(25)	9*b*		0.2452(2)	0.1330(1)	0.942(1)	0.137
O(21)	9*b*	0.25	0.3199(6)	–0.0054(7)	0.226(2)	0.204(4)
H(201)	9*b*	0.25	0.3274(8)	0.017(1)	0.341(2)	0.305
C(21)	9*b*	0.25	0.3038(6)	0.0231(6)	0.092(2)	0.204
H(211)	9*b*	0.25	0.270(1)	0.024(1)	0.130(3)	0.305
H(212)	9*b*	0.25	0.334(1)	0.0624(5)	0.077(3)	0.305
C(22)	9*b*	0.25	0.2915(7)	–0.0081(7)	–0.090(2)	0.204
H(221)	9*b*	0.25	0.281(1)	0.011(1)	–0.180(2)	0.305
H(222)	9*b*	0.25	0.260(1)	–0.0472(8)	–0.079(3)	0.305
H(223)	9*b*	0.25	0.325(1)	–0.009(1)	–0.132(3)	0.305
O(31)	9*b*	0.25	0.3330(7)	0.0028(7)	0.671(2)	0.204
H(301)	9*b*	0.25	0.3112(8)	–0.0351(9)	0.730(2)	0.305
C(31)	9*b*	0.25	0.3453(6)	–0.0064(5)	0.489(2)	0.204
H(311)	9*b*	0.25	0.3099(8)	–0.0308(8)	0.426(2)	0.305
H(312)	9*b*	0.25	0.367(1)	–0.0260(9)	0.489(3)	0.305
C(32)	9*b*	0.25	0.3796(7)	0.0504(7)	0.380(2)	0.204
H(321)	9*b*	0.25	0.3873(9)	0.043(1)	0.260(2)	0.305
H(322)	9*b*	0.25	0.358(1)	0.0703(9)	0.374(4)	0.305
H(323)	9*b*	0.25	0.4157(8)	0.0752(8)	0.439(3)	0.305

Table 6. 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)	9*b*	0.18561(7)	0.28709(7)	0.529(1)	0.059(1)	0.042(1)	0.064(2)	0.0269(9)	–0.005(1)	–0.003(1)
N(1)	9*b*	0.08295(8)	0.16124(9)	0.649(1)	0.048(1)	0.056(1)	0.046(2)	0.020(1)	0.004(1)	–0.005(1)
C(2)	9*b*	0.0280(1)	0.1094(1)	0.581(1)	0.043(2)	0.051(2)	0.053(2)	0.007(1)	0.002(2)	–0.004(2)
C(3)	9*b*	–0.0041(1)	0.1276(1)	0.448(1)	0.041(2)	0.072(2)	0.067(3)	0.022(2)	0.003(2)	–0.007(2)
C(4)	9*b*	0.0393(1)	0.1901(1)	0.387(1)	0.055(2)	0.062(2)	0.084(3)	0.030(2)	–0.004(2)	0.008(2)
C(5)	9*b*	0.0508(1)	0.2293(1)	0.546(1)	0.064(2)	0.066(2)	0.140(4)	0.040(2)	0.014(2)	–0.013(2)
C(6)	9*b*	0.0692(1)	0.2059(1)	0.709(1)	0.074(2)	0.068(2)	0.080(3)	0.028(2)	0.017(2)	–0.025(2)
C(7)	9*b*	0.0965(1)	0.1937(1)	0.327(1)	0.048(2)	0.057(2)	0.046(2)	0.021(2)	–0.004(1)	0.002(2)
C(8)	9*b*	0.12505(9)	0.1821(1)	0.490(1)	0.041(1)	0.043(2)	0.037(2)	0.019(1)	0.005(1)	–0.002(1)
C(9)	9*b*	0.1860(1)	0.2339(1)	0.538(1)	0.044(2)	0.039(2)	0.041(2)	0.016(1)	–0.005(1)	–0.004(1)
C(10)	9*b*	–0.0291(1)	0.0856(2)	0.299(1)	0.053(2)	0.092(3)	0.067(3)	0.020(2)	–0.011(2)	0.003(2)

Table 6. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(11)	9*b*	−0.0504(2)	0.0518(2)	0.185(1)	0.097(3)	0.105(3)	0.084(4)	0.005(3)	−0.014(2)	−0.002(3)
O(1′)	9*b*	0.26605(9)	0.09647(9)	0.747(1)	0.084(2)	0.063(1)	0.096(2)	0.039(1)	−0.013(1)	0.013(1)
N(1′)	9*b*	0.30410(9)	0.2304(1)	0.136(1)	0.047(1)	0.053(2)	0.071(2)	0.017(1)	0.014(1)	0.001(1)
C(2′)	9*b*	0.2799(1)	0.2630(1)	0.122(1)	0.054(2)	0.059(2)	0.059(3)	0.014(2)	0.009(2)	0.012(2)
C(3′)	9*b*	0.2429(1)	0.2661(1)	0.251(1)	0.049(2)	0.056(2)	0.058(3)	0.026(1)	0.008(2)	0.008(2)
C(4′)	9*b*	0.2289(1)	0.2335(1)	0.403(1)	0.039(2)	0.034(1)	0.042(2)	0.010(1)	−0.001(1)	0.001(1)
C(5′)	9*b*	0.2448(1)	0.1633(1)	0.585(1)	0.046(2)	0.046(2)	0.057(2)	0.021(1)	−0.006(1)	−0.004(1)
C(6′)	9*b*	0.2721(1)	0.1314(1)	0.603(1)	0.048(2)	0.047(2)	0.082(3)	0.020(2)	−0.012(2)	0.004(2)
C(7′)	9*b*	0.3081(1)	0.1312(1)	0.465(1)	0.057(2)	0.058(2)	0.117(4)	0.036(2)	−0.001(2)	0.002(2)
C(8′)	9*b*	0.3174(1)	0.1632(1)	0.315(1)	0.048(2)	0.055(2)	0.107(3)	0.025(2)	0.011(2)	−0.008(2)
C(9′)	9*b*	0.2543(1)	0.1975(1)	0.428(1)	0.033(1)	0.038(2)	0.050(2)	0.011(1)	−0.005(1)	−0.003(1)
C(10′)	9*b*	0.2915(1)	0.1983(1)	0.290(1)	0.036(2)	0.043(2)	0.066(3)	0.014(1)	−0.002(2)	−0.006(2)
C(11′)	9*b*	0.2300(2)	0.0946(1)	0.892(1)	0.126(3)	0.073(2)	0.075(3)	0.049(2)	−0.018(3)	0.007(2)

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