Crystal structure of 1-heptyl-4-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-1,2,4-triazolium bis(trifluoromethanesulfonyl)imide, $[C_{17}H_{21}F_{13}N_3][C_2F_6NO_4S_2]$

Verena Adamer, Klaus Wurst, Gerhard Laus and Herwig Schottenberger*

University of Innsbruck, Faculty of Chemistry and Pharmacy, Innrain 52a, 6020 Innsbruck, Austria

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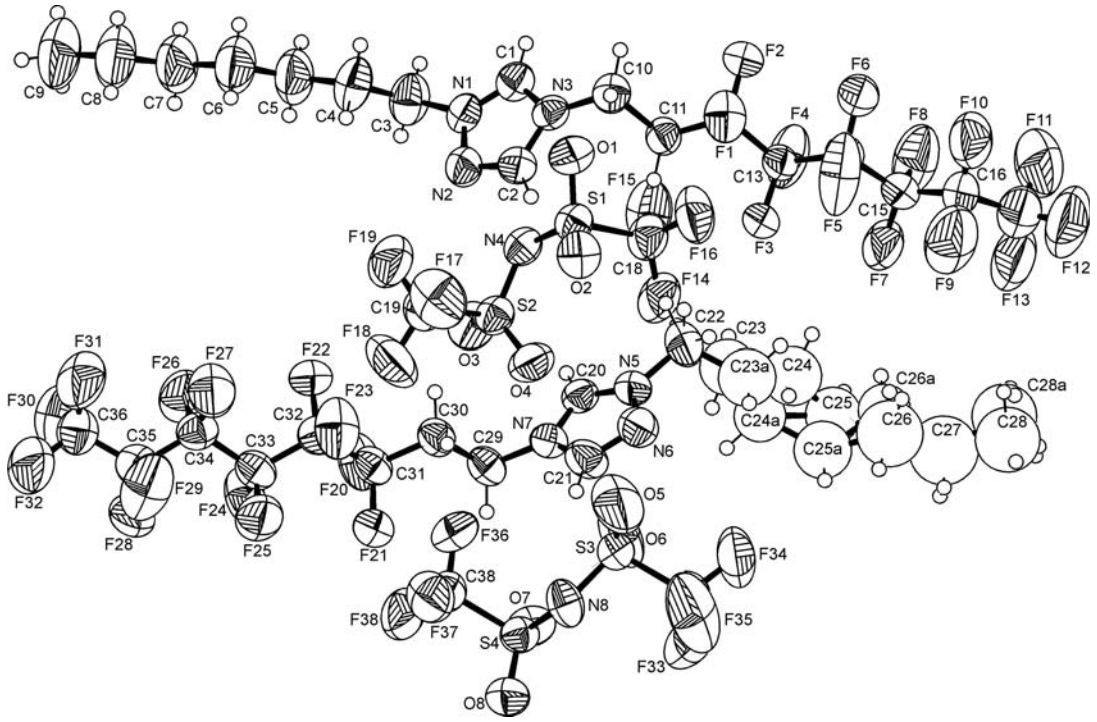

Abstract

$C_{19}H_{21}F_{19}N_4O_4S_2$, monoclinic, *C*1*c*1 (no. 9), a = 32.8918(2) Å, b = 20.3798(5) Å, c = 9.6098(5) Å, β = 99.754(2)°, V = 6348.6 Å^3, Z = 8, $R_{gt}(F)$ = 0.059, $wR_{ref}(F^2)$ = 0.164, T = 233 K.

Source of material

4-Amino-1-heptyl-1,2,4-triazolium bromide was prepared by refluxing a solution of 4-amino-1,2,4-triazole and 1-bromoheptane in acetonitrile for 24 h [1]. The crystalline product was deaminated using $NaNO_2$/HCl [2] to yield 1-heptyl-1,2,4-triazole as a yellowish liquid (n_D^{20} 1.4614). This intermediate was quaternized with 1-iodo-1*H*,1*H*,2*H*,2*H*-perfluorooctane in acetonitrile with microwave heating (2 h, 160 °C). The resulting 1-heptyl-4-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-1,2,4-triazolium iodide (m.p. 277 °C) was subjected to ion metathesis with lithium triflimide in a two-phase H_2O/CH_2Cl_2 mixture to give the title compound in 45 % overall yield (four steps). Suitable crystals were obtained by slow evaporation of a methanol solution (m.p. 65 - 66 °C). Spectroscopic characteristics are in accordance with published data [3,4].

Experimental details

The heptyl group of one cation is disordered and described with two split positions (approximate ratio 1:1). Atoms C23-C28 and C23A-C28A were refined with bond restraints and isotropic displacement parameters. Atoms C27 and C27A were refined with identical coordinates. Hydrogen atoms were calculated in ideal geometric positions. The Flack parameter x of 0.35(9) indicates a racemic twinning of this crystal. A refinement, including this twinning, offers a twin ratio of 2:1 without any change of the R value. Because the higher R value is influenced more by the disorder of the alkyl group, we decide on a refinement as a racemic twin.

Discussion

In the course of our search for liquid salts which combine affinity to fluorous solvents with a certain degree of hydrophilicity [5,6], we came across 1-heptyl-4-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-1,2,4-triazolium bis(trifluoromethanesulfonyl)imide [3,4] as a possible candidate with a reported melting point of 22 °C. However, we discovered that this compound, when sythesized by the old-fashioned regioselective method [2] (in contrast to the new method [3,4], which is prone to give a mixture of isomers), had a

* Correspondence author (e-mail: herwig.schottenberger@uibk.ac.at)

much higher melting point. Therefore, it cannot be classified as a room-temperature ionic liquid (RTIL), but allowed the determination of its crystal structure.

There are two independent ion pairs in the asymmetric unit of the title compound. In the first ion pair, the following interionic hydrogen bonds are observed (H···acceptor distance and donor···acceptor distance, donor–H···acceptor angle): C1–H···N4^{i} (2.533 and 3.387(7) Å, 151.2°), C2–H···O7^{ii} (2.544 and 3.389(7) Å, 149.8°), C2–H···O6^{ii} (2.413 and 3.194(9) Å, 140.4°). The second ion pair exhibits the following interactions: C20–H···O2 (2.611 and 3.317(7) Å, 132.2°), C20–H···O4 (2.431 and 3.300(8) Å, 153.6°), C21–H···N8^{iii} (2.527 and 3.459(9) Å, 172.3°). The heptyl chain of this second cation displays conformational disorder, as noted above. In addition, weak H···H contacts between the alkyl chains as well as weak F···F interactions between neighbouring fluoroalkyl chains are also observed. Finally, the triflimide anions adopt *anti* conformations with C–S···S–C torsion angles of 172.2° and 161.5°, respectively. Symmetry codes i: *x*,1–*y*,½+*z*; ii: *x*,*y*,1+*z*; iii: *x*,2–*y*,½+*z*.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.09 × 0.15 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.13 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ/ω
$2\theta_{max}$:	46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15979, 8544
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 7268
$N(param)_{refined}$:	855
Programs:	SHELXS-97, SHELXL-97 [7]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(1)	4*a*		0.5994	0.5357	1.5315	0.082
H(2)	4*a*		0.5967	0.7257	1.4555	0.091
H(3A)	4*a*		0.6693	0.5084	1.4536	0.113
H(3B)	4*a*		0.6822	0.5624	1.3505	0.113
H(4A)	4*a*		0.7076	0.5586	1.6438	0.123
H(4B)	4*a*		0.7175	0.6180	1.5490	0.123
H(5A)	4*a*		0.7468	0.4880	1.5268	0.153
H(5B)	4*a*		0.7552	0.5453	1.4249	0.153
H(6A)	4*a*		0.7929	0.5989	1.6190	0.164
H(6B)	4*a*		0.7870	0.5368	1.7116	0.164
H(7A)	4*a*		0.8303	0.5346	1.4870	0.204
H(7B)	4*a*		0.8263	0.4754	1.5899	0.204
H(8A)	4*a*		0.8714	0.5919	1.6700	0.206
H(8B)	4*a*		0.8664	0.5345	1.7767	0.206
H(9A)	4*a*		0.9299	0.5347	1.6867	0.339
H(9B)	4*a*		0.9041	0.5123	1.5402	0.339
H(9C)	4*a*		0.9064	0.4666	1.6745	0.339
H(10A)	4*a*		0.5437	0.6712	1.6237	0.081
H(10B)	4*a*		0.5373	0.5947	1.5982	0.081
H(11A)	4*a*		0.5087	0.6142	1.3595	0.078
H(11B)	4*a*		0.5162	0.6906	1.3819	0.078
H(20)	4*a*		0.5752	0.7820	1.0089	0.074
H(21)	4*a*		0.5691	0.9725	1.0436	0.100
H(29A)	4*a*		0.6282	0.9229	0.9085	0.085
H(29B)	4*a*		0.6339	0.8456	0.9180	0.085
H(30A)	4*a*		0.6611	0.8533	1.1574	0.086
H(30B)	4*a*		0.6532	0.9300	1.1544	0.086
H(22A)	4*a*	0.50	0.4812	0.8200	1.1434	0.115
H(22B)	4*a*	0.50	0.5095	0.7579	1.1338	0.115
C(23)	4*a*	0.50	0.4696(6)	0.776(1)	0.946(2)	0.148(7)
H(23A)	4*a*	0.50	0.4801	0.7932	0.8636	0.177
H(23B)	4*a*	0.50	0.4688	0.7279	0.9389	0.177
C(24)	4*a*	0.50	0.4271(6)	0.802(1)	0.949(3)	0.164(9)
H(24A)	4*a*	0.50	0.4140	0.7756	1.0136	0.196
H(24B)	4*a*	0.50	0.4295	0.8472	0.9856	0.196
C(25)	4*a*	0.50	0.4007(7)	0.803(1)	0.810(3)	0.149(8)
H(25A)	4*a*	0.50	0.4162	0.8212	0.7407	0.178
H(25B)	4*a*	0.50	0.3937	0.7571	0.7817	0.178
C(26)	4*a*	0.50	0.3637(7)	0.839(2)	0.806(4)	0.21(2)
H(26A)	4*a*	0.50	0.3556	0.8333	0.8988	0.252
H(26B)	4*a*	0.50	0.3710	0.8849	0.7981	0.252
C(27)	4*a*	0.50	0.3287(5)	0.828(1)	0.708(2)	0.228(7)
H(27A)	4*a*	0.50	0.3313	0.7832	0.6734	0.273
H(27B)	4*a*	0.50	0.3314	0.8570	0.6283	0.273
C(28)	4*a*	0.50	0.2866(8)	0.834(2)	0.728(5)	0.20(2)
H(28A)	4*a*	0.50	0.2684	0.8224	0.6409	0.300
H(28B)	4*a*	0.50	0.2813	0.8791	0.7523	0.300
H(28C)	4*a*	0.50	0.2817	0.8051	0.8029	0.300
H(22C)	4*a*	0.50	0.4975	0.7944	1.1847	0.115
H(22D)	4*a*	0.50	0.5023	0.7529	1.0487	0.115
C(23A)	4*a*	0.50	0.4567(7)	0.823(1)	1.008(2)	0.158(8)
H(23C)	4*a*	0.50	0.4347	0.8017	1.048	0.190
H(23D)	4*a*	0.50	0.4554	0.8702	1.0282	0.190
C(24A)	4*a*	0.50	0.4487(5)	0.815(1)	0.865(2)	0.128(6)
H(24C)	4*a*	0.50	0.4515	0.7682	0.8464	0.153
H(24D)	4*a*	0.50	0.4706	0.8377	0.8265	0.153
C(25A)	4*a*	0.50	0.4078(6)	0.837(2)	0.780(3)	0.158(9)
H(25C)	4*a*	0.50	0.4081	0.8268	0.6805	0.189
H(25D)	4*a*	0.50	0.4066	0.8853	0.7866	0.189
C(26A)	4*a*	0.50	0.3671(5)	0.809(1)	0.819(2)	0.131(7)
H(26C)	4*a*	0.50	0.3692	0.7615	0.8252	0.158
H(26D)	4*a*	0.50	0.3636	0.8262	0.9112	0.158
C(27A)	4*a*	0.50	0.3287(5)	0.828(1)	0.708(2)	0.228(7)
H(27C)	4*a*	0.50	0.3286	0.8757	0.6938	0.273
H(27D)	4*a*	0.50	0.3313	0.8075	0.6174	0.273
C(28A)	4*a*	0.50	0.2886(8)	0.808(2)	0.747(4)	0.20(2)
H(28D)	4*a*	0.50	0.2663	0.8316	0.6893	0.300
H(28E)	4*a*	0.50	0.2887	0.8182	0.8460	0.300
H(28F)	4*a*	0.50	0.2846	0.7612	0.7324	0.300

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}
S(1)	4*a*		0.56947(5)	0.60496(7)	1.0967(1)	0.0696(8)	0.0730(9)	0.0519(7)	0.0008(7)	0.0048(6)	–0.0053(7)
S(2)	4*a*		0.62225(5)	0.63840(8)	0.9091(2)	0.0720(9)	0.080(1)	0.0585(8)	0.0070(7)	0.0091(6)	–0.0016(7)
S(3)	4*a*		0.54433(5)	0.85419(9)	0.5818(2)	0.0654(9)	0.090(1)	0.083(1)	0.0040(8)	0.0154(7)	0.0205(9)
S(4)	4*a*		0.60816(5)	0.89302(7)	0.4422(2)	0.0729(9)	0.0646(8)	0.0633(8)	0.0053(7)	0.0170(6)	0.0017(7)
F(1)	4*a*		0.4690(1)	0.7019(2)	1.5748(4)	0.082(2)	0.119(3)	0.075(2)	0.003(2)	0.017(2)	–0.032(2)
F(2)	4*a*		0.4568(1)	0.6000(2)	1.5291(5)	0.077(2)	0.100(3)	0.111(3)	–0.003(2)	0.018(2)	0.045(2)
F(3)	4*a*		0.4464(1)	0.7266(3)	1.2858(6)	0.064(2)	0.182(5)	0.152(4)	–0.005(3)	0.015(2)	0.107(4)
F(4)	4*a*		0.4284(1)	0.6257(3)	1.2604(5)	0.086(3)	0.180(5)	0.090(3)	0.029(3)	–0.012(2)	–0.047(3)
F(5)	4*a*		0.3943(2)	0.7498(4)	1.4497(9)	0.081(3)	0.182(6)	0.238(7)	0.023(3)	–0.017(4)	–0.104(5)
F(6)	4*a*		0.3817(1)	0.6501(4)	1.4756(6)	0.080(3)	0.256(7)	0.131(4)	0.016(3)	0.030(3)	0.110(4)
F(7)	4*a*		0.3667(2)	0.7251(4)	1.1564(6)	0.080(3)	0.335(9)	0.113(3)	0.028(4)	0.014(3)	0.109(5)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(8)	4*a*		0.3448(2)	0.6356(3)	1.2201(8)	0.105(4)	0.139(5)	0.208(6)	0.006(3)	–0.035(4)	–0.044(4)
F(9)	4*a*		0.3220(2)	0.7945(3)	1.294(1)	0.136(5)	0.107(4)	0.34(1)	0.009(3)	–0.017(6)	0.011(5)
F(10)	4*a*		0.3084(2)	0.7123(4)	1.4212(6)	0.087(3)	0.228(6)	0.123(4)	0.021(3)	0.030(3)	0.024(4)
F(11)	4*a*		0.2642(2)	0.6580(5)	1.205(1)	0.112(5)	0.218(8)	0.29(1)	–0.056(5)	–0.012(5)	0.029(8)
F(12)	4*a*		0.2455(2)	0.7524(5)	1.2105(9)	0.098(4)	0.266(9)	0.237(8)	0.064(5)	0.011(5)	0.022(7)
F(13)	4*a*		0.2814(2)	0.7272(6)	1.0646(9)	0.098(4)	0.38(1)	0.149(6)	0.040(6)	–0.006(4)	0.073(7)
F(14)	4*a*		0.5144(1)	0.6269(3)	0.8717(5)	0.087(3)	0.177(4)	0.079(3)	0.007(3)	–0.013(2)	0.015(3)
F(15)	4*a*		0.5205(1)	0.5266(3)	0.9309(6)	0.101(3)	0.126(4)	0.149(4)	–0.010(3)	–0.012(3)	–0.062(3)
F(16)	4*a*		0.4903(1)	0.5945(3)	1.0487(5)	0.072(3)	0.193(5)	0.093(3)	–0.010(3)	0.022(2)	–0.018(3)
F(17)	4*a*		0.6587(2)	0.7043(3)	1.1266(6)	0.153(5)	0.153(4)	0.116(4)	–0.056(4)	0.007(3)	–0.044(4)
F(18)	4*a*		0.6900(2)	0.7053(3)	0.9529(7)	0.125(4)	0.157(5)	0.167(5)	–0.056(3)	0.050(4)	0.017(4)
F(19)	4*a*		0.6901(2)	0.6200(3)	1.0830(7)	0.096(3)	0.141(4)	0.179(5)	–0.018(3)	–0.039(3)	0.037(4)
F(20)	4*a*		0.7115(1)	0.8555(2)	0.9744(5)	0.083(3)	0.121(3)	0.110(3)	–0.004(2)	0.034(2)	–0.045(3)
F(21)	4*a*		0.7023(1)	0.9600(2)	0.9842(4)	0.079(2)	0.114(3)	0.102(3)	–0.011(2)	0.011(2)	0.037(2)
F(22)	4*a*		0.7423(1)	0.8470(2)	1.2398(6)	0.090(3)	0.126(4)	0.143(4)	–0.009(2)	0.003(3)	0.075(3)
F(23)	4*a*		0.7250(1)	0.9462(3)	1.2831(4)	0.093(3)	0.172(4)	0.085(2)	0.004(3)	0.025(2)	–0.053(3)
F(24)	4*a*		0.7862(1)	0.9129(3)	1.0338(4)	0.082(3)	0.206(5)	0.076(2)	–0.027(3)	0.031(2)	–0.029(3)
F(25)	4*a*		0.7767(2)	0.9988(2)	1.1483(6)	0.108(3)	0.077(3)	0.165(4)	–0.005(2)	–0.001(3)	0.022(3)
F(26)	4*a*		0.8264(2)	0.8539(2)	1.2502(7)	0.104(3)	0.072(3)	0.192(5)	0.007(2)	0.000(3)	0.007(3)
F(27)	4*a*		0.8066(2)	0.9199(3)	1.3978(4)	0.104(3)	0.231(6)	0.064(2)	–0.007(3)	0.018(2)	0.008(3)
F(28)	4*a*		0.8601(2)	0.9664(3)	1.1375(5)	0.100(3)	0.216(6)	0.096(3)	–0.034(3)	0.008(2)	0.065(3)
F(29)	4*a*		0.8495(2)	1.0149(3)	1.3266(8)	0.135(4)	0.098(3)	0.220(6)	0.014(3)	–0.030(4)	–0.043(4)
F(30)	4*a*		0.9079(2)	0.8786(3)	1.2915(7)	0.104(3)	0.153(4)	0.161(5)	0.028(3)	0.017(3)	–0.024(4)
F(31)	4*a*		0.8919(2)	0.9177(4)	1.4791(5)	0.115(4)	0.273(8)	0.072(3)	0.020(4)	0.002(2)	0.021(4)
F(32)	4*a*		0.9253(2)	0.9722(3)	1.3500(9)	0.103(4)	0.168(5)	0.234(7)	–0.037(4)	–0.041(4)	0.050(5)
F(33)	4*a*		0.4963(2)	0.9060(6)	0.380(1)	0.132(6)	0.39(2)	0.196(8)	0.069(7)	0.016(5)	0.161(9)
F(34)	4*a*		0.4655(1)	0.8608(3)	0.5274(8)	0.068(3)	0.123(4)	0.243(7)	–0.005(3)	0.028(3)	0.003(4)
F(35)	4*a*		0.4942(2)	0.9500(3)	0.564(1)	0.136(5)	0.095(4)	0.46(2)	0.010(3)	0.130(7)	–0.051(6)
F(36)	4*a*		0.6499(1)	0.8173(2)	0.6307(5)	0.105(3)	0.121(3)	0.098(3)	0.020(3)	0.002(2)	0.038(3)
F(37)	4*a*		0.6661(2)	0.9184(3)	0.6537(5)	0.109(3)	0.162(4)	0.092(3)	–0.036(3)	–0.003(2)	–0.001(3)
F(38)	4*a*		0.6847(2)	0.8608(3)	0.4865(6)	0.090(3)	0.231(6)	0.118(4)	0.050(4)	0.044(3)	0.051(4)
O(1)	4*a*		0.5706(2)	0.5546(3)	1.2016(5)	0.098(3)	0.108(3)	0.080(3)	0.001(3)	0.005(2)	0.024(3)
O(2)	4*a*		0.5661(1)	0.6711(2)	1.1361(5)	0.090(3)	0.079(3)	0.089(3)	0.003(2)	0.019(2)	–0.024(2)
O(3)	4*a*		0.6379(2)	0.6012(3)	0.8067(5)	0.107(4)	0.113(4)	0.077(3)	0.009(3)	0.016(3)	–0.014(3)
O(4)	4*a*		0.5985(2)	0.6950(2)	0.8683(5)	0.099(3)	0.092(3)	0.090(3)	0.023(3)	0.015(2)	0.024(2)
O(5)	4*a*		0.5427(2)	0.8518(7)	0.7250(7)	0.128(5)	0.42(2)	0.082(4)	–0.063(8)	0.008(4)	0.075(6)
O(6)	4*a*		0.5411(2)	0.7949(2)	0.503(1)	0.098(4)	0.065(3)	0.31(1)	–0.014(3)	0.089(5)	–0.027(4)
O(7)	4*a*		0.6010(2)	0.8389(2)	0.3473(4)	0.115(4)	0.080(3)	0.061(2)	0.011(2)	0.018(2)	–0.006(2)
O(8)	4*a*		0.6165(2)	0.9546(2)	0.3844(5)	0.092(3)	0.078(3)	0.099(3)	0.001(2)	0.020(2)	0.017(2)
N(1)	4*a*		0.6402(2)	0.5954(2)	1.4662(5)	0.058(3)	0.058(3)	0.073(3)	0.005(2)	–0.001(2)	–0.005(2)
N(2)	4*a*		0.6406(2)	0.6608(2)	1.4378(6)	0.063(3)	0.071(3)	0.095(4)	0.007(2)	0.014(3)	0.007(3)
N(3)	4*a*		0.5834(1)	0.6318(2)	1.5095(5)	0.065(3)	0.059(3)	0.053(2)	0.000(2)	0.001(2)	0.001(2)
N(4)	4*a*		0.6033(2)	0.5877(2)	1.0056(5)	0.069(3)	0.064(3)	0.071(3)	0.011(2)	0.008(2)	0.000(2)
N(5)	4*a*		0.5315(2)	0.8401(2)	1.0583(5)	0.066(3)	0.060(3)	0.079(3)	0.000(2)	0.015(2)	0.012(2)
N(6)	4*a*		0.5283(2)	0.9057(3)	1.0754(7)	0.078(3)	0.074(3)	0.109(4)	0.012(3)	0.032(3)	0.013(3)
N(7)	4*a*		0.5870(2)	0.8783(2)	1.0104(5)	0.069(3)	0.060(3)	0.063(3)	–0.001(2)	0.016(2)	0.004(2)
N(8)	4*a*		0.5778(2)	0.9043(3)	0.5471(6)	0.067(3)	0.083(3)	0.094(4)	–0.011(2)	0.027(3)	–0.026(3)
C(1)	4*a*		0.6065(2)	0.5784(3)	1.5073(6)	0.070(4)	0.060(3)	0.073(4)	0.002(3)	0.004(3)	–0.004(3)
C(2)	4*a*		0.6056(2)	0.6819(3)	1.4651(7)	0.071(4)	0.065(3)	0.089(4)	–0.003(3)	0.009(3)	0.012(3)
C(3)	4*a*		0.6761(2)	0.5549(4)	1.4456(9)	0.076(4)	0.083(4)	0.122(6)	0.017(3)	0.013(4)	–0.022(4)
C(4)	4*a*		0.7128(2)	0.5706(4)	1.5496(9)	0.070(4)	0.109(5)	0.123(6)	0.022(4)	0.000(4)	–0.017(5)
C(5)	4*a*		0.7515(2)	0.5353(4)	1.522(1)	0.070(5)	0.098(5)	0.21(1)	0.013(4)	0.018(6)	0.010(6)
C(6)	4*a*		0.7899(3)	0.5510(6)	1.616(1)	0.078(6)	0.147(8)	0.18(1)	0.016(5)	0.012(6)	0.008(7)
C(7)	4*a*		0.8283(3)	0.5233(5)	1.585(2)	0.083(6)	0.127(8)	0.30(2)	0.027(6)	0.039(8)	0.06(1)
C(8)	4*a*		0.8683(3)	0.5443(7)	1.678(2)	0.078(6)	0.19(1)	0.24(1)	0.011(7)	–0.004(7)	0.02(1)
C(9)	4*a*		0.9054(4)	0.5117(9)	1.642(2)	0.086(7)	0.25(2)	0.34(2)	0.048(9)	0.03(1)	0.06(2)
C(10)	4*a*		0.5433(2)	0.6361(3)	1.5538(6)	0.068(3)	0.081(4)	0.053(3)	0.001(3)	0.010(3)	–0.007(3)
C(11)	4*a*		0.5096(2)	0.6500(3)	1.4280(6)	0.059(3)	0.081(4)	0.054(3)	–0.005(3)	0.006(2)	–0.001(3)
C(12)	4*a*		0.4675(2)	0.6567(3)	1.4728(6)	0.068(3)	0.067(3)	0.064(3)	–0.011(3)	0.007(3)	0.008(3)
C(13)	4*a*		0.4334(2)	0.6757(3)	1.3519(6)	0.060(3)	0.088(4)	0.056(3)	–0.002(3)	0.015(2)	0.009(3)
C(14)	4*a*		0.3912(2)	0.6932(3)	1.3877(7)	0.072(4)	0.069(3)	0.085(4)	–0.010(3)	0.006(3)	0.001(3)
C(15)	4*a*		0.3549(2)	0.6989(4)	1.2626(8)	0.069(4)	0.100(5)	0.094(5)	–0.011(3)	0.012(3)	0.031(4)
C(16)	4*a*		0.3153(2)	0.7281(5)	1.293(1)	0.063(5)	0.142(8)	0.158(8)	0.015(5)	0.018(5)	0.062(6)
C(17)	4*a*		0.2772(3)	0.7194(7)	1.190(1)	0.094(7)	0.17(1)	0.126(8)	0.009(7)	0.009(6)	0.024(7)
C(18)	4*a*		0.5213(2)	0.5861(4)	0.9792(7)	0.077(4)	0.111(6)	0.071(4)	–0.001(4)	0.000(3)	–0.007(4)
C(19)	4*a*		0.6684(2)	0.6687(4)	1.0244(9)	0.082(5)	0.099(5)	0.089(5)	–0.010(4)	0.007(4)	0.001(4)
C(20)	4*a*		0.5662(2)	0.8249(3)	1.0232(6)	0.065(4)	0.055(3)	0.065(3)	0.008(3)	0.011(3)	0.007(2)
C(21)	4*a*		0.5622(2)	0.9278(3)	1.0438(8)	0.088(5)	0.054(3)	0.116(5)	0.003(3)	0.039(4)	0.009(3)
C(29)	4*a*		0.6279(2)	0.8847(3)	0.9703(7)	0.070(4)	0.075(4)	0.074(4)	–0.005(3)	0.028(3)	–0.003(3)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(30)	4*a*		0.6604(2)	0.8927(3)	1.0987(6)	0.067(4)	0.085(4)	0.066(3)	–0.003(3)	0.021(3)	0.003(3)
C(31)	4*a*		0.7023(2)	0.9040(3)	1.0603(6)	0.071(4)	0.068(3)	0.069(3)	–0.001(3)	0.030(3)	–0.006(3)
C(32)	4*a*		0.7376(2)	0.9076(3)	1.1868(6)	0.081(4)	0.074(4)	0.062(3)	0.008(3)	0.023(3)	–0.004(3)
C(33)	4*a*		0.7788(2)	0.9331(3)	1.1580(6)	0.079(4)	0.078(4)	0.069(4)	–0.004(3)	0.028(3)	–0.003(3)
C(34)	4*a*		0.8166(2)	0.9179(3)	1.2717(6)	0.084(4)	0.074(4)	0.065(4)	0.006(3)	0.016(3)	0.003(3)
C(35)	4*a*		0.8556(2)	0.9550(4)	1.2668(8)	0.094(5)	0.088(5)	0.092(5)	–0.007(4)	0.018(4)	0.000(4)
C(36)	4*a*		0.8948(2)	0.9313(4)	1.3495(8)	0.082(5)	0.099(5)	0.089(5)	0.000(4)	0.007(4)	–0.008(4)
C(37)	4*a*		0.4971(3)	0.8957(4)	0.508(1)	0.090(5)	0.096(5)	0.117(7)	0.001(4)	0.027(5)	0.009(5)
C(38)	4*a*		0.6552(2)	0.8705(4)	0.5599(7)	0.068(4)	0.114(5)	0.068(4)	0.009(4)	0.016(3)	0.011(4)
C(22)	4*a*	0.50	0.4978(2)	0.7971(4)	1.0831(9)	0.069(4)	0.098(5)	0.124(6)	–0.009(4)	0.022(4)	0.013(4)
C(22A)	4*a*	0.50	0.4978(2)	0.7971(4)	1.0831(9)	0.069(4)	0.098(5)	0.124(6)	–0.009(4)	0.022(4)	0.013(4)

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