Crystal structure of pentakis(diisobutylaminium) dichloride tris(tetrachloridoferrate(III)), $C_{40}H_{100}Cl_{14}Fe_3N_5$

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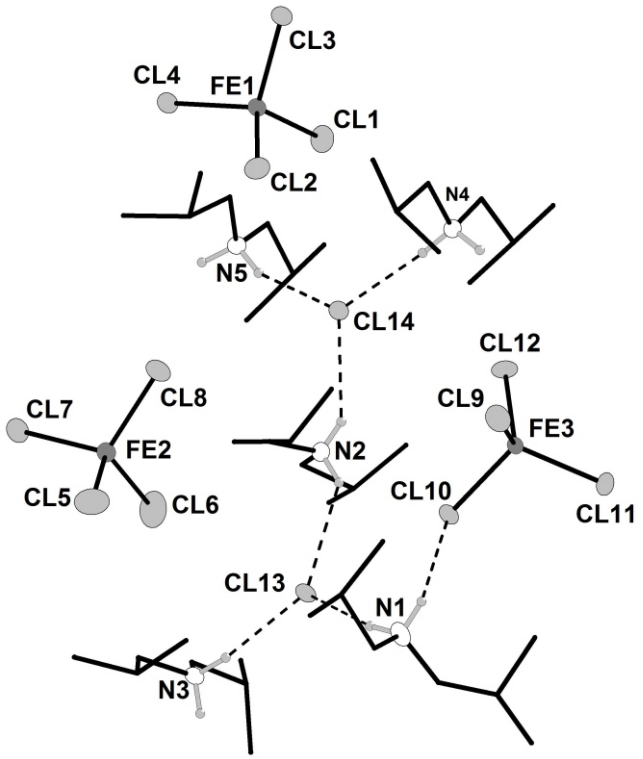

Abstract

$C_{40}H_{100}Cl_{14}Fe_3N_5$, monoclinic, $P2_1/n$ (no. 14), a = 19.549(2) Å, b = 20.031(2) Å, c = 19.705(2) Å, β = 117.388(4)°, V = 6851.3(12) Å^3, Z = 4, $R_{gt}(F)$ = 0.0220, $wR_{ref}(F^2)$ = 0.0529, T = 100 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.25 0.30 0.38 mm
Wavelength:	Mo K radiation (0.71073 Å)
μ:	12.02 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	59974, 12065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 10851
$N(param)_{refined}$:	610
Programs:	SHELX [12], DIAMOND [13], SADABS [14], SAINT [14]

Source of material

In a typical experiment 0.487 g (3 mmol) $FeCl_3$ was dissolved in concentrated hydrochloric acid supported by applying gentle heat. Diisobutylaminiumchloride (0.829 g; 5 mmol) was added to the warm solution and stirred receiving a yellow liquid. At the bottom of the vessel block-shaped, yellow crystals grow within a few days by isothermal evaporation.

Experimental details

All hydrogen atoms were identified in difference syntheses. (The H atoms of the methyl groups were allowed to rotate with a fixed angle about the C–C bond (HFIX 137 option of the SHELX-2013 program [12]) with U_{iso}(H) set to 1.5U_{eq}(C). Hydrogen atoms belonging to CH_2 groups were placed in calculated positions and were included in the refinement using a riding model with U_{iso}(H) set to 1.2U_{eq}(C). The hydrogen atoms of the NH_2 groups were refined freely with their U_{iso} values set to 1.2U_{eq}(N).

Discussion

Recently, simple dialkylaminium salts attracted considerable attention due to their ferroelectric properties [1, 2]. Furthermore, salts containing the tetrachloridoferrate(III) anion are of particular interest for their magnetic behavior [3, 4]. This contribution is part of our ongoing interest in salts of the system dialkylaminium / halogenometallate [5-7]. The asymmetric unit of the title structure consists of five diisobutylaminium cations (*dibu*H$^+$), two chloride and three tetrachloridoferrate(III) anions. The bond lengths within the *dibu*H$^+$ cations and the complex $[FeCl_4]^-$ anions are in the expected ranges. The conformation parameters of the five crystallographically independent *dibu*H$^+$ cations are partly extremely different to fulfill the needs of packing and hydrogen bonding. Strong to medium strong N–H···Cl hydrogen bonds (N···Cl distances range from 3.077(2) Å to 3.238(2) Å) connect the five *dibu*H$^+$ cations with the two chloride anions and one of the $[FeCl_4]^-$ anions to a moiety with the formal stoichiometry $\{(dibuH)_5Cl_2[FeCl_4]\}^{2+}$ as shown in the figure. Further N···Cl distances in the range of 3.277(2) Å to 3.426(2) Å may be interpreted as weak hydrogen bonds which connect all ions. The title structure is in excellent accord with related structures of double salts in the system aminium$^+$/$FeCl_4^-$/Cl^-. In these structures the chloride anions form the shortest hydrogen bonds whereas the $[FeCl_4]^-$ anions form exclusively weak hydrogen bonds [8-11].

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

Atom	Site	x	y	z	U_{iso}
H(11)	4*e*	0.218(1)	0.2755(9)	0.443(1)	0.030
H(12)	4*e*	0.215(1)	0.310(1)	0.380(1)	0.030
H(1A)	4*e*	0.2890	0.2333	0.3586	0.035
H(1B)	4*e*	0.2884	0.1886	0.4257	0.035
H(2A)	4*e*	0.3586	0.3178	0.4497	0.039
H(3A)	4*e*	0.4337	0.1906	0.4829	0.081
H(3B)	4*e*	0.4770	0.2611	0.4994	0.081
H(3C)	4*e*	0.4243	0.2373	0.4132	0.081

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(4A)	4*e*	0.4160	0.2912	0.5820	0.059
H(4B)	4*e*	0.3677	0.2232	0.5627	0.059
H(4C)	4*e*	0.3242	0.2936	0.5468	0.059
H(5A)	4*e*	0.1413	0.2270	0.2975	0.036
H(5B)	4*e*	0.0995	0.2657	0.3391	0.036
H(6A)	4*e*	0.1708	0.1373	0.3820	0.037
H(7A)	4*e*	0.0113	0.1731	0.3195	0.070
H(7B)	4*e*	0.0438	0.0985	0.3393	0.070
H(7C)	4*e*	0.0492	0.1384	0.2716	0.070
H(8A)	4*e*	0.1023	0.2135	0.4577	0.052
H(8B)	4*e*	0.1896	0.1880	0.4950	0.052
H(8C)	4*e*	0.1209	0.1356	0.4733	0.052
H(21)	4*e*	0.213(1)	0.477(1)	0.467(1)	0.021
H(22)	4*e*	0.223(1)	0.5240(9)	0.524(1)	0.021
H(9A)	4*e*	0.3273	0.5679	0.5247	0.025
H(9B)	4*e*	0.3082	0.5277	0.4477	0.025
H(10A)	4*e*	0.3333	0.4254	0.5182	0.030
H(11A)	4*e*	0.4276	0.4723	0.4913	0.064
H(11B)	4*e*	0.4689	0.4379	0.5737	0.064
H(11C)	4*e*	0.4590	0.5173	0.5668	0.064
H(12A)	4*e*	0.3905	0.5074	0.6518	0.044
H(12B)	4*e*	0.3999	0.4280	0.6526	0.044
H(12C)	4*e*	0.3162	0.4604	0.6232	0.044
H(13A)	4*e*	0.1797	0.5646	0.3796	0.022
H(13B)	4*e*	0.1783	0.6094	0.4462	0.022
H(14A)	4*e*	0.0782	0.4983	0.3755	0.025
H(15A)	4*e*	0.0189	0.5781	0.3161	0.060
H(15B)	4*e*	0.0503	0.5946	0.2966	0.060
H(15C)	4*e*	0.0400	0.6373	0.3600	0.060
H(16A)	4*e*	0.0967	0.5086	0.5012	0.055
H(16B)	4*e*	0.0105	0.5303	0.4432	0.055
H(16C)	4*e*	0.0741	0.5860	0.4878	0.055
H(31)	4*e*	0.207(1)	0.4189(9)	0.166(1)	0.021
H(32)	4*e*	0.223(1)	0.4313(9)	0.241(1)	0.021
H(17A)	4*e*	0.3178	0.5063	0.2561	0.026
H(17B)	4*e*	0.2954	0.4951	0.1681	0.026
H(18A)	4*e*	0.3412	0.3834	0.1956	0.028
H(19A)	4*e*	0.4493	0.4866	0.2784	0.052
H(19B)	4*e*	0.4221	0.4677	0.1908	0.052
H(18C)	4*e*	0.4738	0.4166	0.2571	0.052
H(20A)	4*e*	0.3291	0.3622	0.3067	0.055
H(20B)	4*e*	0.3916	0.4184	0.3524	0.055
H(20C)	4*e*	0.4172	0.3516	0.3255	0.055
H(21A)	4*e*	0.1638	0.5269	0.1297	0.031
H(21B)	4*e*	0.1802	0.5383	0.2163	0.031
H(22A)	4*e*	0.0841	0.4571	0.1989	0.042

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(23A)	4*e*	0.0803	0.3946	0.0973	0.061
H(23B)	4*e*	0.0548	0.4586	0.0425	0.061
H(23C)	4*e*	0.0033	0.4266	0.0701	0.061
H(24A)	4*e*	0.0311	0.5679	0.0948	0.095
H(24B)	4*e*	0.0548	0.5731	0.1840	0.095
H(24C)	4*e*	0.0192	0.5306	0.1284	0.095
H(41)	4*e*	0.782(1)	0.4632(9)	0.232(1)	0.019
H(42)	4*e*	0.773(1)	0.4108(9)	0.275(1)	0.019
H(25A)	4*e*	0.8246	0.3923	0.1666	0.024
H(25B)	4*e*	0.8242	0.3349	0.2228	0.024
H(26A)	4*e*	0.9195	0.4509	0.2777	0.027
H(27A)	4*e*	0.9620	0.3240	0.2414	0.056
H(27B)	4*e*	1.0215	0.3837	0.2814	0.056
H(27C)	4*e*	0.9547	0.3917	0.1956	0.056
H(28A)	4*e*	0.9320	0.3273	0.3552	0.055
H(28B)	4*e*	0.9012	0.3955	0.3737	0.055
H(28C)	4*e*	0.9892	0.3893	0.3905	0.055
H(29A)	4*e*	0.6829	0.3592	0.1651	0.024
H(29B)	4*e*	0.6904	0.4221	0.1190	0.024
H(30A)	4*e*	0.6305	0.4278	0.2274	0.028
H(31A)	4*e*	0.6869	0.5310	0.2280	0.047
H(31B)	4*e*	0.5953	0.5401	0.1847	0.047
H(31C)	4*e*	0.6425	0.5346	0.1367	0.047
H(32A)	4*e*	0.5105	0.4429	0.1197	0.048
H(32B)	4*e*	0.5481	0.3750	0.1097	0.048
H(32C)	4*e*	0.5500	0.4407	0.0644	0.048
H(51)	4*e*	0.715(1)	0.2221(9)	0.428(1)	0.022
H(52)	4*e*	0.732(1)	0.282(1)	0.400(1)	0.022
H(33A)	4*e*	0.6419	0.1897	0.2966	0.024
H(33B)	4*e*	0.6572	0.2646	0.2785	0.024
H(34A)	4*e*	0.5928	0.3011	0.3543	0.027
H(35A)	4*e*	0.4688	0.2884	0.2448	0.042
H(35B)	4*e*	0.5059	0.2365	0.2093	0.042
H(35C)	4*e*	0.5326	0.3130	0.2208	0.042
H(36A)	4*e*	0.5926	0.1982	0.4118	0.049
H(36B)	4*e*	0.5406	0.1655	0.3299	0.049
H(36C)	4*e*	0.5056	0.2225	0.3617	0.049
H(37A)	4*e*	0.7884	0.2166	0.3387	0.027
H(37B)	4*e*	0.7834	0.1567	0.3899	0.027
H(38A)	4*e*	0.8731	0.2753	0.4480	0.030
H(39A)	4*e*	0.8674	0.1655	0.5376	0.052
H(39B)	4*e*	0.8341	0.2384	0.5379	0.052
H(39C)	4*e*	0.9249	0.2276	0.5705	0.052
H(40A)	4*e*	0.9209	0.1973	0.3888	0.049
H(40B)	4*e*	0.9258	0.1417	0.4492	0.049
H(40C)	4*e*	0.9796	0.2065	0.4775	0.049

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}
Fe(1)	4*e*	0.22678(2)	0.47522(2)	0.02664(2)	0.0203(1)	0.0159(1)	0.0163(1)	0.00069(9)	0.00930(9)	0.00057(9)
Fe(2)	4*e*	0.70438(2)	0.30121(2)	0.65131(2)	0.0263(1)	0.0167(1)	0.0202(1)	0.00060(9)	0.0126(1)	0.00080(9)
Fe(3)	4*e*	0.18061(2)	0.34451(2)	0.62052(2)	0.0213(1)	0.0163(1)	0.0163(1)	0.00266(9)	0.0111(1)	0.00101(9)
Cl(13)	4*e*	0.20732(2)	0.41582(2)	0.34813(2)	0.0280(2)	0.0206(2)	0.0159(2)	0.0017(2)	0.0122(2)	0.0006(2)
Cl(14)	4*e*	0.75236(2)	0.38768(2)	0.37703(2)	0.0250(2)	0.0195(2)	0.0184(2)	0.0010(2)	0.0109(2)	0.0004(2)
Cl(1)	4*e*	0.35071(2)	0.48919(2)	0.01792(2)	0.0195(2)	0.0351(2)	0.0301(2)	0.0023(2)	0.0081(2)	0.0062(2)
Cl(2)	4*e*	0.17584(2)	0.56667(2)	0.00860(3)	0.0290(2)	0.0209(2)	0.0347(2)	0.0019(2)	0.0158(2)	0.0063(2)
Cl(3)	4*e*	0.17502(2)	0.45364(2)	0.14984(2)	0.0304(2)	0.0226(2)	0.0168(2)	0.0020(2)	0.0110(2)	0.0001(2)
Cl(4)	4*e*	0.20615(3)	0.38935(2)	0.03069(2)	0.0426(2)	0.0218(2)	0.0209(2)	0.0055(2)	0.0173(2)	0.0008(2)
Cl(5)	4*e*	0.63235(3)	0.38087(2)	0.65983(3)	0.0549(3)	0.0294(2)	0.0528(3)	0.0110(2)	0.0260(3)	0.0104(2)
Cl(6)	4*e*	0.82522(3)	0.32075(3)	0.72945(3)	0.0280(3)	0.0741(4)	0.0426(3)	0.0089(2)	0.0064(2)	0.0156(3)
Cl(7)	4*e*	0.67179(3)	0.20819(2)	0.68719(3)	0.0715(3)	0.0186(2)	0.0410(3)	0.0074(2)	0.0398(3)	0.0017(2)
Cl(8)	4*e*	0.68542(3)	0.29492(2)	0.53287(3)	0.0620(3)	0.0254(2)	0.0267(2)	0.0042(2)	0.0275(2)	0.0019(2)
Cl(9)	4*e*	0.29658(2)	0.30788(2)	0.69647(2)	0.0300(2)	0.0316(2)	0.0289(2)	0.0097(2)	0.0132(2)	0.0090(2)
Cl(10)	4*e*	0.16745(2)	0.36148(2)	0.50398(2)	0.0323(2)	0.0217(2)	0.0195(2)	0.0010(2)	0.0166(2)	0.0000(2)
Cl(11)	4*e*	0.09593(3)	0.27069(2)	0.61354(3)	0.0408(3)	0.0339(2)	0.0303(2)	0.0197(2)	0.0210(2)	0.0053(2)
Cl(12)	4*e*	0.15926(2)	0.44077(2)	0.65965(2)	0.0228(2)	0.0256(2)	0.0243(2)	0.0006(2)	0.0083(2)	0.0099(2)
N(1)	4*e*	0.21678(9)	0.26919(7)	0.39878(9)	0.0352(8)	0.0211(8)	0.0273(8)	0.0049(6)	0.0217(7)	0.0053(6)
C(1)	4*e*	0.2884(1)	0.23505(9)	0.4085(1)	0.039(1)	0.0254(9)	0.032(1)	0.0081(8)	0.0245(9)	0.0046(7)
C(2)	4*e*	0.3605(1)	0.27033(9)	0.4661(1)	0.035(1)	0.031(1)	0.040(1)	0.0081(8)	0.0246(9)	0.0096(8)
C(3)	4*e*	0.4301(1)	0.2368(1)	0.4654(1)	0.042(1)	0.073(2)	0.058(2)	0.017(1)	0.033(1)	0.008(1)

Table 3. continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	4*e*	0.3678(1)	0.2695(1)	0.5466(1)	0.039(1)	0.045(1)	0.036(1)	0.0067(9)	0.0190(9)	0.0026(9)
C(5)	4*e*	0.1427(1)	0.23529(9)	0.3477(1)	0.035(1)	0.032(1)	0.0267(9)	0.0024(8)	0.0167(8)	0.0027(8)
C(6)	4*e*	0.1304(1)	0.16953(9)	0.3788(1)	0.038(1)	0.0233(9)	0.037(1)	0.0013(8)	0.0235(9)	0.0004(8)
C(7)	4*e*	0.0517(1)	0.1424(1)	0.3223(1)	0.049(1)	0.045(1)	0.051(1)	0.012(1)	0.027(1)	0.011(1)
C(8)	4*e*	0.1363(1)	0.17734(9)	0.4583(1)	0.049(1)	0.027(1)	0.040(1)	0.0027(8)	0.030(1)	0.0042(8)
N(2)	4*e*	0.22515(7)	0.51701(7)	0.48283(8)	0.0172(7)	0.0176(7)	0.0191(7)	0.0005(5)	0.0086(6)	0.0003(6)
C(9)	4*e*	0.30678(9)	0.52533(8)	0.49726(9)	0.0147(8)	0.0252(9)	0.0224(8)	0.0034(6)	0.0089(7)	0.0022(7)
C(10)	4*e*	0.35766(9)	0.46821(9)	0.5442(1)	0.0178(8)	0.0268(9)	0.0255(9)	0.0017(7)	0.0063(7)	0.0047(7)
C(11)	4*e*	0.4352(1)	0.4745(1)	0.5440(1)	0.0200(9)	0.072(2)	0.034(1)	0.0099(9)	0.0111(8)	0.002(1)
C(12)	4*e*	0.3669(1)	0.46577(9)	0.6251(1)	0.0214(9)	0.033(1)	0.0288(9)	0.0038(7)	0.0072(8)	0.0061(8)
C(13)	4*e*	0.16977(9)	0.56376(8)	0.42451(9)	0.0190(8)	0.0181(8)	0.0177(8)	0.0025(6)	0.0078(7)	0.0033(6)
C(14)	4*e*	0.08645(9)	0.54382(8)	0.39882(9)	0.0186(8)	0.0217(8)	0.0210(8)	0.0007(6)	0.0077(7)	0.0008(7)
C(15)	4*e*	0.0348(1)	0.5928(1)	0.3374(1)	0.0227(9)	0.063(1)	0.033(1)	0.0107(9)	0.0110(8)	0.023(1)
C(16)	4*e*	0.0651(1)	0.5420(1)	0.4634(1)	0.0195(9)	0.064(1)	0.027(1)	0.0047(9)	0.0109(8)	0.0137(9)
N(3)	4*e*	0.22168(8)	0.44931(7)	0.20001(8)	0.0186(7)	0.0208(7)	0.0133(7)	0.0006(5)	0.0072(6)	0.0002(6)
C(17)	4*e*	0.29974(9)	0.47266(8)	0.21468(9)	0.0198(8)	0.0235(8)	0.0201(8)	0.0047(7)	0.0077(7)	0.0029(7)
C(18)	4*e*	0.35990(9)	0.41796(9)	0.23677(9)	0.0191(8)	0.0307(9)	0.0208(8)	0.0012(7)	0.0089(7)	0.0008(7)
C(19)	4*e*	0.4328(1)	0.4501(1)	0.2412(1)	0.0224(9)	0.048(1)	0.034(1)	0.0017(8)	0.0132(8)	0.0024(9)
C(20)	4*e*	0.3759(1)	0.3846(1)	0.3121(1)	0.027(1)	0.052(1)	0.033(1)	0.0120(9)	0.0160(9)	0.0141(9)
C(21)	4*e*	0.1641(1)	0.50476(9)	0.1748(1)	0.0269(9)	0.0267(9)	0.0205(8)	0.0091(7)	0.0087(7)	0.0003(7)
C(22)	4*e*	0.0834(1)	0.4818(1)	0.1545(1)	0.0215(9)	0.062(1)	0.0204(9)	0.0122(9)	0.0094(7)	0.0094(9)
C(23)	4*e*	0.0509(1)	0.4364(1)	0.0849(1)	0.0196(9)	0.036(1)	0.050(1)	0.0042(8)	0.0035(9)	0.0008(9)
C(24)	4*e*	0.0330(1)	0.5439(1)	0.1390(1)	0.034(1)	0.096(2)	0.043(1)	0.029(1)	0.003(1)	0.030(1)
N(4)	4*e*	0.77232(7)	0.42096(7)	0.23100(8)	0.0161(7)	0.0157(7)	0.0159(7)	0.0003(5)	0.0065(6)	0.0010(5)
C(25)	4*e*	0.83201(9)	0.38329(8)	0.21892(9)	0.0198(8)	0.0182(8)	0.0230(8)	0.0026(6)	0.0110(7)	0.0002(6)
C(26)	4*e*	0.91444(9)	0.40120(8)	0.27580(9)	0.0181(8)	0.0213(8)	0.0267(9)	0.0021(6)	0.0100(7)	0.0033(7)
C(27)	4*e*	0.9680(1)	0.3726(1)	0.2459(1)	0.024(1)	0.051(1)	0.040(1)	0.0087(9)	0.0175(9)	0.0060(9)
C(28)	4*e*	0.9361(1)	0.3761(1)	0.3559(1)	0.0235(9)	0.057(1)	0.026(1)	0.0055(9)	0.0084(8)	0.0091(9)
C(29)	4*e*	0.69281(9)	0.40788(8)	0.16821(9)	0.0166(8)	0.0222(8)	0.0177(8)	0.0030(6)	0.0043(7)	0.0015(6)
C(30)	4*e*	0.62939(9)	0.44354(9)	0.17869(9)	0.0152(8)	0.0330(9)	0.0191(8)	0.0018(7)	0.0067(7)	0.0029(7)
C(31)	4*e*	0.6394(1)	0.51902(9)	0.1824(1)	0.0212(9)	0.033(1)	0.035(1)	0.0064(7)	0.0088(8)	0.0063(8)
C(32)	4*e*	0.55268(9)	0.4238(1)	0.1122(1)	0.0167(8)	0.040(1)	0.032(1)	0.0016(8)	0.0052(8)	0.0036(8)
N(5)	4*e*	0.72197(7)	0.23886(7)	0.39098(8)	0.0176(7)	0.0205(7)	0.0162(7)	0.0022(6)	0.0073(6)	0.0013(6)
C(33)	4*e*	0.65026(9)	0.23617(8)	0.31600(9)	0.0167(8)	0.0236(8)	0.0167(8)	0.0031(6)	0.0054(7)	0.0039(6)
C(34)	4*e*	0.57948(9)	0.25964(8)	0.32256(9)	0.0196(8)	0.0235(9)	0.0220(8)	0.0007(7)	0.0084(7)	0.0043(7)
C(35)	4*e*	0.5159(1)	0.27584(9)	0.2421(1)	0.0226(9)	0.031(1)	0.0281(9)	0.0051(7)	0.0093(8)	0.0001(8)
C(36)	4*e*	0.5521(1)	0.2068(1)	0.3598(1)	0.0236(9)	0.046(1)	0.031(1)	0.0020(8)	0.0135(8)	0.0036(8)
C(37)	4*e*	0.78910(9)	0.20571(8)	0.38805(9)	0.0168(8)	0.0252(9)	0.0241(9)	0.0004(7)	0.0084(7)	0.0047(7)
C(38)	4*e*	0.86642(9)	0.22631(9)	0.4528(1)	0.0192(8)	0.0269(9)	0.0254(9)	0.0013(7)	0.0074(7)	0.0012(7)
C(39)	4*e*	0.8739(1)	0.2133(1)	0.5316(1)	0.0256(9)	0.046(1)	0.026(1)	0.0013(8)	0.0065(8)	0.0008(8)
C(40)	4*e*	0.9287(1)	0.1897(1)	0.4410(1)	0.0183(9)	0.041(1)	0.033(1)	0.0024(8)	0.0072(8)	0.0032(8)

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