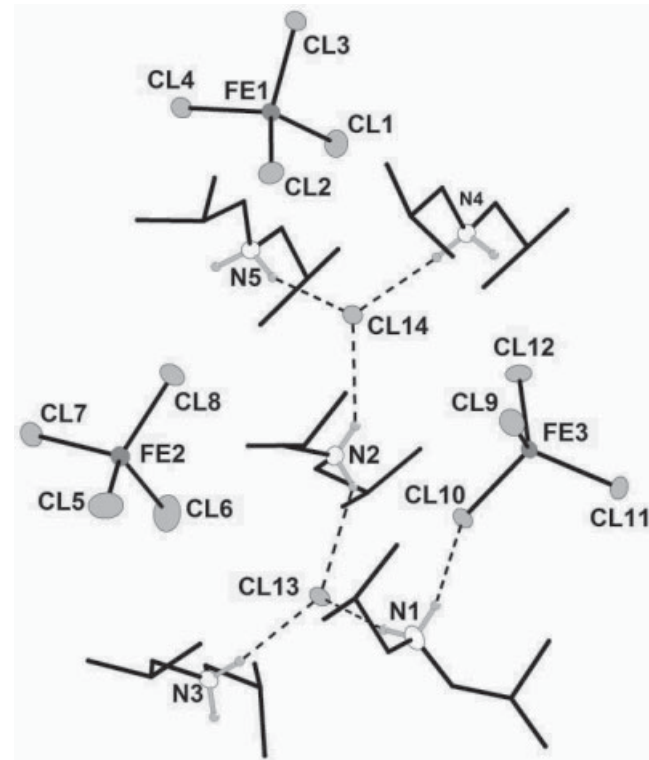


Crystal structure of pentakis(diisobutylamminium) dichloride tris(tetrachloridoferrate(III)), $C_{40}H_{100}Cl_{14}Fe_3N_5$

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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, $\beta = 117.388(4)^\circ$, $V = 6851.3(12)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0220$, $wR_{\text{ref}}(F^2) = 0.0529$, $T = 100$ K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.25×0.30×0.38 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	12.02 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	59974, 12065
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 10851
$N(\text{param})_{\text{refined}}$:	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. Diisobutylamminiumchloride (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 $U_{\text{iso}}(\text{H})$ set to $1.5U_{\text{eq}}(\text{C})$. Hydrogen atoms belonging to CH_2 groups were placed in calculated positions and were included in the refinement using a riding model with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The hydrogen atoms of the NH_2 groups were refined freely with their U_{iso} values set to $1.2U_{\text{eq}}(\text{N})$.

Discussion

Recently, simple dialkylamminium 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 dialkylamminium / halogenometallate [5–7]. The asymmetric unit of the title structure consists of five diisobutylamminium cations ($dibuH^+$), two chloride and three tetrachloridoferrate(III) anions. The bond lengths within the $dibuH^+$ cations and the complex $[FeCl_4]^-$ anions are in the expected ranges. The conformation parameters of the five crystallographically independent $dibuH^+$ 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 $dibuH^+$ 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 Å²).

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

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Table 2. continued.

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

Table 2. continued.

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

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Fe(1)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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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