Crystal structure of (*R*)-*N,N,N',N'',N''*-pentamethyl-*N*-(1-phenylethyl)-guanidinium iodide, [C₁₄H₂₄N₃]⁺I[−]

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Abstract

C₁₄H₂₄IN₃, monoclinic, *P*12₁1 (no. 4), *a* = 10.599(3) Å, *b* = 10.737(4) Å, *c* = 14.970(5) Å, β = 90.02(3)°, *V* = 1703.6 Å³, *Z* = 4, *R*_g(*F*) = 0.054, *wR*_{ref}(*F*²) = 0.149, *T* = 293 K.

Source of material

The title compound was prepared by methylation of (*R*)-*N,N,N',N'*-tetramethyl-*N*-(1-phenylethyl)guanidine [1] with methyl iodide. The crude product was recrystallized from acetonitrile/diethyl ether, m.p. 145–146 °C. The enantiomerically pure compound has [*α*]_D²⁰ = −14.3 (*c* = 1.00, acetonitrile).

Experimental details

The β angle of the unit cell is 90.02(3)°, which is close to the orthorhombic crystal system. All attempts solving the structure in the orthorhombic crystal system failed. Checking the Laue symmetry of some reflections had confirmed the monoclinic system in this case. H atoms were located on difference Fourier map, but refined with fixed individual displacement parameters using a riding model with *d*(C—H) ranging from 0.93 to 0.98 Å. In addition, the methyl groups were allowed to rotate but not to tip.

Discussion

The title compound crystallizes with two independent molecules in the asymmetric unit. The absolute configuration, as known from the starting material, (*R*)-*N,N,N',N'*-tetramethyl-*N*-(1-phenylethyl)guanidine, was confirmed by the absolute structure determination by X-ray data indicated by the Flack parameter of *x* = 0.00(3). The guanidinium ion is showing a planar conformation. In contrast, the methyl groups attached to the nitrogen atoms are forced out-of-plane. Both conformers (molecule A and molecule B) differ slightly in this distortion characterized by the dihedral angles between N1—C2—C3 and N2—C4—C5 of 58.4(5)° [A] and 58.2(6)° [B]. Between N2—C4—C5 and N3—C6—C7 the dihedral angles are 71.8(5)° [A] and 68.2(5)° [B]. The dihedral angles between N3—C6—C7 and N1—C2—C3 are 60.5(5)° [A] and 64.7(5)° [B].

Table 1. Data collection and handling.

Crystal:	colorless plates, size 0.35 × 0.70 × 1.20 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	18.70 cm ^{−1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff scan
2θ _{max} :	60°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	10696, 9905
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 7327
<i>N</i> (<i>param</i>) _{refined} :	338
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL-Plus [5]

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(2A1)	2a	1.1213	0.0432	−0.0017	0.102
H(2A2)	2a	1.0002	0.0273	0.0573	0.102
H(2A3)	2a	1.0966	−0.0827	0.0483	0.102
H(3A1)	2a	1.1290	−0.0862	0.2169	0.130
H(3A2)	2a	1.0239	0.0164	0.2213	0.130
H(3A3)	2a	1.1571	0.0418	0.2632	0.130
H(4A1)	2a	1.3691	0.0025	0.2760	0.122
H(4A2)	2a	1.4858	0.0124	0.2120	0.122
H(4A3)	2a	1.3626	−0.0547	0.1798	0.122
H(5A1)	2a	1.3600	0.3126	0.2011	0.102
H(5A2)	2a	1.4919	0.2476	0.1924	0.102
H(5A3)	2a	1.4136	0.2337	0.2807	0.102
H(6A1)	2a	1.1009	0.2532	−0.0195	0.110
H(6A2)	2a	1.1411	0.3736	0.0325	0.110
H(6A3)	2a	1.0665	0.2679	0.0818	0.110
H(7A)	2a	1.4312	0.1997	0.0273	0.069
H(9A)	2a	1.3522	0.0303	−0.0663	0.073
H(10A)	2a	1.2882	−0.0377	−0.2063	0.088
H(11A)	2a	1.2342	0.1058	−0.3132	0.092
H(12A)	2a	1.2335	0.3139	−0.2818	0.099

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(13A)	2a	1.2997	0.3839	-0.1410	0.083
H(14A)	2a	1.3395	0.4440	-0.0035	0.134
H(14B)	2a	1.4783	0.4025	-0.0241	0.134
H(14C)	2a	1.4301	0.4063	0.0748	0.134
H(2B1)	2a	1.0501	-0.1855	0.6106	0.150
H(2B2)	2a	1.1423	-0.0737	0.5939	0.150
H(2B3)	2a	1.1889	-0.1913	0.6465	0.150
H(3B1)	2a	1.1518	-0.0811	0.4280	0.141
H(3B2)	2a	1.0296	-0.1604	0.4433	0.141
H(3B3)	2a	1.1398	-0.2136	0.3848	0.141
H(4B1)	2a	1.0606	-0.4088	0.5859	0.102
H(4B2)	2a	1.1306	-0.4086	0.6782	0.102
H(4B3)	2a	1.1262	-0.5304	0.6205	0.102
H(5B1)	2a	1.3564	-0.5570	0.5767	0.095

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(5B2)	2a	1.3610	-0.4810	0.6661	0.095
H(5B3)	2a	1.4340	-0.4329	0.5818	0.095
H(6B1)	2a	1.3829	-0.1481	0.4464	0.126
H(6B2)	2a	1.3751	-0.2032	0.3496	0.126
H(6B3)	2a	1.4917	-0.2333	0.4104	0.126
H(7B)	2a	1.3039	-0.5114	0.4266	0.062
H(9B)	2a	1.4322	-0.3628	0.2355	0.083
H(10B)	2a	1.3224	-0.3241	0.1018	0.106
H(11B)	2a	1.1170	-0.3693	0.0934	0.137
H(12B)	2a	1.0093	-0.4543	0.2090	0.159
H(13B)	2a	1.1163	-0.4964	0.3448	0.116
H(14D)	2a	1.5329	-0.4334	0.3495	0.117
H(14E)	2a	1.4886	-0.5720	0.3595	0.117
H(14F)	2a	1.5235	-0.4941	0.4445	0.117

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
I(1A)	2a	0.76258(4)	0.18461(4)	0.12610(3)	0.0813(2)	0.0743(3)	0.0730(2)	0.0057(2)	0.0086(2)	-0.0128(2)
I(1B)	2a	0.76360(4)	-0.30102(4)	0.53683(3)	0.0910(3)	0.0673(2)	0.0658(2)	0.0120(2)	-0.0117(2)	-0.0065(2)
C(1A)	2a	1.2499(4)	0.1350(5)	0.1234(3)	0.045(2)	0.059(3)	0.032(2)	0.002(2)	-0.001(2)	-0.002(2)
N(1A)	2a	1.1568(4)	0.0528(5)	0.1292(3)	0.060(2)	0.073(3)	0.036(2)	-0.011(2)	0.003(2)	-0.001(2)
N(2A)	2a	1.3477(4)	0.1296(4)	0.1809(3)	0.059(2)	0.064(3)	0.042(2)	0.003(2)	-0.008(2)	0.003(2)
C(2A)	2a	1.0878(5)	0.0062(7)	0.0517(4)	0.060(3)	0.080(4)	0.064(3)	-0.016(3)	-0.008(2)	-0.008(3)
N(3A)	2a	1.2478(3)	0.2258(4)	0.0618(3)	0.049(2)	0.055(2)	0.049(2)	0.004(2)	-0.003(2)	0.005(2)
C(3A)	2a	1.1129(7)	0.0017(8)	0.2151(5)	0.093(5)	0.112(6)	0.055(3)	-0.025(4)	0.020(3)	0.016(4)
C(4A)	2a	1.3953(7)	0.0126(7)	0.2150(5)	0.095(5)	0.089(5)	0.061(4)	0.017(4)	-0.018(3)	0.022(3)
C(5A)	2a	1.4085(6)	0.2403(6)	0.2168(4)	0.073(3)	0.085(4)	0.045(3)	-0.013(3)	-0.008(2)	-0.014(3)
C(6A)	2a	1.1289(6)	0.2852(7)	0.0370(5)	0.066(3)	0.069(4)	0.084(4)	0.019(3)	-0.014(3)	0.003(3)
C(7A)	2a	1.3626(5)	0.2531(5)	0.0059(4)	0.055(3)	0.065(3)	0.053(3)	-0.009(2)	-0.001(2)	0.013(2)
C(8A)	2a	1.3307(4)	0.2131(5)	-0.0891(3)	0.060(2)	0.056(3)	0.046(2)	-0.001(2)	0.004(2)	0.011(2)
C(9A)	2a	1.3283(6)	0.0881(6)	-0.1094(4)	0.073(3)	0.057(3)	0.052(3)	0.009(2)	0.014(2)	0.010(2)
C(10A)	2a	1.2907(7)	0.0469(7)	-0.1934(5)	0.090(4)	0.067(4)	0.063(4)	-0.003(3)	0.024(3)	-0.002(3)
C(11A)	2a	1.2577(8)	0.1328(8)	-0.2566(5)	0.092(5)	0.090(5)	0.048(3)	-0.016(4)	0.007(3)	0.003(3)
C(12A)	2a	1.2584(8)	0.2570(9)	-0.2383(5)	0.106(5)	0.091(5)	0.050(4)	-0.012(4)	-0.004(3)	0.029(3)
C(13A)	2a	1.2970(7)	0.2991(6)	-0.1535(4)	0.100(4)	0.053(3)	0.054(3)	-0.007(3)	-0.004(3)	0.015(3)
C(14A)	2a	1.4068(8)	0.3894(8)	0.0140(5)	0.113(5)	0.085(5)	0.069(4)	-0.039(4)	-0.011(4)	0.008(4)
C(1B)	2a	1.2520(4)	-0.3172(5)	0.5114(3)	0.061(2)	0.040(2)	0.038(2)	-0.008(2)	-0.012(2)	-0.002(2)
N(1B)	2a	1.1765(5)	-0.2192(4)	0.5157(4)	0.089(3)	0.041(2)	0.063(3)	0.009(2)	-0.009(2)	-0.004(2)
N(2B)	2a	1.2476(4)	-0.4046(4)	0.5742(3)	0.057(2)	0.052(2)	0.037(2)	-0.003(2)	-0.006(2)	0.004(2)
C(2B)	2a	1.1360(9)	-0.1627(8)	0.5987(7)	0.120(7)	0.072(5)	0.108(7)	0.015(4)	-0.001(5)	-0.043(5)
C(3B)	2a	1.1195(8)	-0.1638(7)	0.4361(6)	0.121(6)	0.064(4)	0.098(6)	0.013(4)	-0.036(5)	0.015(4)
N(3B)	2a	1.3370(4)	-0.3287(4)	0.4445(2)	0.067(2)	0.046(2)	0.040(2)	-0.015(2)	-0.003(1)	0.001(2)
C(4B)	2a	1.1314(6)	-0.4412(7)	0.6184(5)	0.069(3)	0.082(4)	0.053(3)	-0.012(3)	0.007(3)	0.007(3)
C(5B)	2a	1.3594(5)	-0.4750(6)	0.6021(4)	0.062(3)	0.075(4)	0.054(3)	0.005(3)	-0.008(2)	0.019(3)
C(6B)	2a	1.4023(8)	-0.2189(6)	0.4098(5)	0.125(6)	0.061(3)	0.066(4)	-0.049(4)	0.002(4)	0.007(3)
C(7B)	2a	1.3502(5)	-0.4472(5)	0.3936(3)	0.063(3)	0.053(3)	0.038(2)	-0.008(2)	0.003(2)	0.001(2)
C(8B)	2a	1.2842(5)	-0.4307(6)	0.3042(4)	0.059(3)	0.067(3)	0.050(3)	0.001(2)	-0.001(2)	-0.018(2)
C(9B)	2a	1.3466(6)	-0.3811(7)	0.2314(4)	0.081(4)	0.081(4)	0.046(3)	-0.001(3)	-0.003(2)	-0.003(3)
C(10B)	2a	1.2809(9)	-0.3581(8)	0.1508(4)	0.125(6)	0.096(5)	0.043(3)	0.022(4)	-0.010(3)	-0.008(3)
C(11B)	2a	1.160(1)	-0.386(1)	0.1464(6)	0.136(8)	0.149(9)	0.058(4)	0.035(7)	-0.030(5)	-0.022(5)
C(12B)	2a	1.0946(8)	-0.436(1)	0.2151(8)	0.070(4)	0.21(1)	0.118(8)	0.005(6)	-0.033(5)	-0.065(8)
C(13B)	2a	1.1589(7)	-0.461(1)	0.2970(5)	0.075(4)	0.140(8)	0.075(5)	-0.015(4)	-0.003(3)	-0.030(5)
C(14B)	2a	1.4863(6)	-0.4907(8)	0.3861(4)	0.071(4)	0.103(5)	0.061(4)	0.008(3)	-0.003(3)	0.006(3)

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