Crystal structure of (1*R**,7*S**,9*S**,15*R**)-2,6,10,14-tetraaza-tricyclo[13.1.0.0^{7,9}]hexadeca-2,4,10,12-tetraene bis(hydrobromide), C₁₂H₁₆N₄(HBr)₂

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C₁₂H₁₆Br₂N₄, monoclinic, *P*2₁/*n* (No. 14), *a* = 6.173(1) Å, *b* = 9.252(2) Å, *c* = 13.779(3) Å, β = 99.55(3)°, *V* = 776.1 Å³, *Z* = 2, *R*(*F*) = 0.035, *R*_w(*F*) = 0.032.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.45 x 0.35 x 0.30 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	52.10 cm ⁻¹
Diffractometer:	Syntex P3
Scan mode:	ω
<i>T</i> _{measurement} :	293 K
2θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{unique} :	1770
Criterion for <i>F</i> _o :	<i>F</i> _o > 3 σ(<i>F</i> _o)
<i>N</i> (<i>param</i>) _{refined} :	118
Program:	SHELXTL-plus

Source of material: The title compound was prepared, according to ref. 1, from *trans*-1,2-cyclopropanediammonium dibromide and 1,5-diphenyl-1,5-diazapenta-1,3-diene hydroperchlorate in ethanol solution in the presence of sodium acetate (3 d, 298 K). Evaporation of the solvent and cooling to 273 K yielded a precipitate which was treated with hot ethanol/water (1:1), isolated by filtration, and recrystallized from water to afford colorless crystals (51%), mp 569 K (decomposition).

The title compound, which exhibits symmetry in solution as shown by proton and carbon-13 spectroscopy, adopts a symmetrical structure in the crystalline state as well. All carbon-nitrogen bonds in the chains between the cyclopropane rings exist in the *Z* configuration while the carbon-carbon bonds of both chains adopt the *E* configuration. The chains represent two separate 6π systems with complete delocalization of the positive charge. For the diastereomeric bis(hydroperchlorate), which is obtained from *cis*-1,2-cyclopropanediammonium dibromide, see ref. 2.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.246(5)	1.714(4)	0.543(2)	0.06(1)
H(2)	4e	0.483(6)	1.585(4)	0.701(3)	0.08(1)
H(3)	4e	0.495(6)	1.376(4)	0.641(3)	0.06(1)
H(4)	4e	0.150(5)	1.465(4)	0.484(2)	0.05(1)
H(5)	4e	0.360(5)	1.199(4)	0.527(2)	0.046(9)
H(6)	4e	0.145(7)	1.104(4)	0.406(3)	0.09(2)
H(7)	4e	-0.024(5)	1.328(4)	0.305(2)	0.05(1)
H(8A)	4e	-0.339(6)	1.189(4)	0.275(3)	0.05(1)
H(8B)	4e	-0.263(6)	1.078(5)	0.360(3)	0.08(1)

Table 3. Final 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
Br	4e	0.22896(7)	0.86727(4)	0.35143(3)	0.0691(3)	0.0528(2)	0.0451(2)	0.0220(2)	-0.0100(2)	-0.0035(2)
C(1)	4e	0.2579(6)	1.6967(4)	0.6039(3)	0.052(2)	0.044(2)	0.038(2)	0.001(2)	0.003(2)	-0.001(2)
N(2)	4e	0.3862(5)	1.5742(3)	0.6442(2)	0.046(2)	0.051(2)	0.038(2)	0.006(1)	-0.003(1)	-0.004(1)
C(3)	4e	0.3793(5)	1.4460(4)	0.6039(2)	0.042(2)	0.045(2)	0.044(2)	0.007(2)	0.004(1)	0.002(2)
C(4)	4e	0.2463(5)	1.4020(4)	0.5183(3)	0.039(2)	0.044(2)	0.044(2)	0.008(1)	0.001(1)	0.001(2)
C(5)	4e	0.2520(6)	1.2598(4)	0.4910(3)	0.044(2)	0.047(2)	0.053(2)	0.011(2)	0.003(2)	-0.000(2)
N(6)	4e	0.1232(5)	1.1968(4)	0.4174(2)	0.053(2)	0.044(2)	0.057(2)	0.011(1)	-0.002(1)	-0.010(2)
C(7)	4e	-0.0571(6)	1.2636(4)	0.3542(3)	0.053(2)	0.050(2)	0.042(2)	0.004(2)	0.006(2)	-0.007(2)
C(8)	4e	-0.2584(7)	1.1764(5)	0.3307(3)	0.062(3)	0.050(2)	0.066(3)	0.003(2)	-0.010(2)	-0.015(2)

References

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