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(*S*)-2-(Pyrrolidinium-2-ylmethyl)-isoquinolinium dibromide

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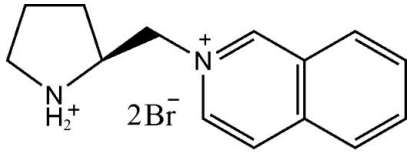
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.023; wR factor = 0.052; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $C_{14}H_{18}N_2^{+}\cdot 2Br^{-}$, molecules are linked by N—H···Br hydrogen bonds.

Related literature

For related literature, see: List & Lerner (2000); List & Pojarliev (2001); Notz & Sakthivel (2001).



Experimental

Crystal data

$C_{14}H_{18}N_2^{+}\cdot 2Br^{-}$
M_r = 374.12
Triclinic, $P1$
a = 6.1326 (6) Å
b = 7.3174 (7) Å
c = 9.8781 (10) Å
α = 93.817 (2)°
β = 104.335 (2)°
γ = 114.408 (1)°
V = 383.86 (7) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 5.27 mm^{-1}
T = 298 (2) K
0.34 × 0.23 × 0.19 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.256, T_{max} = 0.375
2761 measured reflections
2423 independent reflections
2281 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.052
S = 0.96
2423 reflections
163 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 1061 Friedel pairs
Flack parameter: 0.062 (1)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Br1	0.90	2.30	3.203 (3)	178
N2—H2B···Br2	0.90	2.30	3.180 (3)	166

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

We acknowledge the Analytic Centre of Wenzhou University for access to their diffractometer. We are also grateful for financial support from the Catalytic Hydrogenation Center of Zhejiang University of Technology

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2162).

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supplementary materials

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(*S*)-2-(Pyrrolidinium-2-ylmethyl)isoquinolinium dibromide

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Comment

The title compound is a relatively new structural class of organocatalysts that play an important role in asymmetric reactions. For example, *L*-proline is an efficient organocatalyst and has been defined as a universal catalyst because of its utility in enantioselective aldol (List *et al.*, 2000), Mannich (Notz *et al.*, 2001) and Michael (List *et al.*, 2001) reactions.

The crystallographic asymmetric unit of (I) consists of an isoquinoline cation and a bromide anion (Fig. 1) which are linked by an N—H···Br hydrogen bond. The angle of C11, C10 and N1 is 111.4 (3)° (Table 1). The isoquinoline group lies above the pyrrolidine five-membered ring.

Experimental

(*S*)-2-(Bromomethyl)pyrrolidine hydrobromide (20 mmol), prepared by reaction of proline with sodium borohydride, was added slowly to isoquinoline (22 mmol) in methanol (50 ml) at 338 K. The mixture was stirred for 12 h and then the solvent was removed to give the title compound. Crystals suitable for X-ray analysis were obtained from diethyl ether by slow evaporation.

Refinement

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

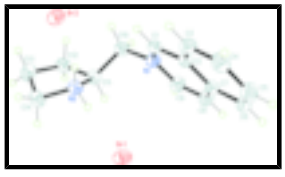

Fig. 1. View of the asymmetric unit in (1), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

(*S*)-2-(Pyrrolidinium-2-ylmethyl)isoquinolinium dibromide

Crystal data

$C_{14}H_{18}N_2^+ \cdot 2Br^-$	$Z = 1$
$M_r = 374.12$	$F_{000} = 186$

Triclinic, *P*1	D_x = 1.618 Mg m^{-3}
Hall symbol: P 1	Mo *K*α radiation λ = 0.71073 Å
a = 6.1326 (6) Å	Cell parameters from 1985 reflections
b = 7.3174 (7) Å	θ = 3.1–24.9º
c = 9.8781 (10) Å	μ = 5.27 mm^{-1}
α = 93.817 (2)º	*T* = 298 (2) K
β = 104.335 (2)º	Blcok, colorless
γ = 114.408 (1)º	0.34 × 0.23 × 0.19 mm
V = 383.86 (7) Å^3	

Data collection

Bruker APEX area-detector diffractometer	2423 independent reflections
Radiation source: fine-focus sealed tube	2281 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.014
T = 298(2) K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	*h* = −7→7
T_{min} = 0.256, T_{max} = 0.375	*k* = −8→8
2761 measured reflections	*l* = −11→11

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.023	$w = 1/[\sigma^2(F_o^2) + (0.0026P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.052	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.96	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
2423 reflections	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
163 parameters	Extinction correction: none
3 restraints	Absolute structure: Flack (1983), 1061 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.062 (1)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.27632 (5)	0.78925 (4)	0.83981 (4)	0.05390 (15)
Br2	0.23689 (5)	0.26066 (4)	1.18024 (4)	0.05562 (16)
N1	0.5178 (8)	0.3772 (7)	0.8240 (4)	0.0441 (11)
N2	0.5946 (5)	0.6520 (4)	1.0843 (3)	0.0396 (7)
H2A	0.5052	0.6927	1.0172	0.048*
H2B	0.4916	0.5288	1.0967	0.048*
C1	0.2876 (7)	0.2516 (6)	0.8219 (4)	0.0431 (9)
H1	0.2616	0.1918	0.9003	0.052*
C2	0.0848 (7)	0.2085 (5)	0.7033 (4)	0.0397 (8)
C3	−0.1629 (8)	0.0811 (6)	0.7041 (4)	0.0502 (10)
H3	−0.1896	0.0203	0.7820	0.060*
C4	−0.3627 (8)	0.0481 (7)	0.5888 (4)	0.0590 (11)
H4	−0.5258	−0.0345	0.5886	0.071*
C5	−0.3201 (9)	0.1394 (7)	0.4713 (5)	0.0598 (11)
H5	−0.4568	0.1164	0.3938	0.072*
C6	−0.0830 (8)	0.2611 (6)	0.4678 (4)	0.0537 (11)
H6	−0.0597	0.3189	0.3884	0.064*
C7	0.1259 (7)	0.2989 (6)	0.5846 (4)	0.0434 (9)
C8	0.3782 (8)	0.4274 (6)	0.5923 (4)	0.0531 (11)
H8	0.4126	0.4867	0.5150	0.064*
C9	0.5690 (8)	0.4647 (6)	0.7107 (4)	0.0505 (10)
H9	0.7339	0.5492	0.7149	0.061*
C10	0.7300 (9)	0.4310 (8)	0.9574 (6)	0.0465 (14)
H10A	0.8726	0.4284	0.9330	0.056*
H10B	0.6790	0.3297	1.0163	0.056*
C11	0.8078 (9)	0.6415 (8)	1.0411 (5)	0.0408 (12)
H11	0.8652	0.7447	0.9831	0.049*
C12	1.0133 (10)	0.6959 (9)	1.1816 (6)	0.0498 (14)
H12A	1.0432	0.5784	1.2000	0.060*
H12B	1.1687	0.8062	1.1787	0.060*
C13	0.9224 (8)	0.7617 (7)	1.2967 (4)	0.0606 (11)
H13A	0.8656	0.6542	1.3503	0.073*
H13B	1.0557	0.8840	1.3618	0.073*
C14	0.7107 (7)	0.8025 (6)	1.2197 (4)	0.0514 (10)
H14A	0.5919	0.7812	1.2731	0.062*
H14B	0.7715	0.9413	1.2024	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0522 (3)	0.0563 (3)	0.0681 (4)	0.0318 (3)	0.0261 (3)	0.0247 (3)
Br2	0.0613 (4)	0.0498 (3)	0.0561 (3)	0.0177 (3)	0.0282 (3)	0.0203 (3)
N1	0.046 (2)	0.046 (2)	0.044 (2)	0.026 (2)	0.011 (2)	0.0081 (19)
N2	0.0307 (16)	0.0364 (17)	0.0496 (18)	0.0126 (13)	0.0126 (13)	0.0081 (14)
C1	0.054 (3)	0.035 (2)	0.041 (2)	0.0191 (19)	0.0170 (19)	0.0072 (17)
C2	0.046 (2)	0.037 (2)	0.039 (2)	0.0214 (18)	0.0132 (17)	0.0043 (16)
C3	0.052 (2)	0.047 (2)	0.048 (2)	0.020 (2)	0.013 (2)	0.0112 (19)
C4	0.051 (3)	0.058 (3)	0.061 (3)	0.022 (2)	0.012 (2)	0.002 (2)
C5	0.059 (3)	0.068 (3)	0.050 (2)	0.034 (2)	0.005 (2)	0.001 (2)
C6	0.068 (3)	0.058 (3)	0.038 (2)	0.033 (2)	0.012 (2)	0.009 (2)
C7	0.055 (2)	0.040 (2)	0.040 (2)	0.0252 (19)	0.0163 (19)	0.0061 (17)
C8	0.066 (3)	0.060 (3)	0.043 (2)	0.029 (2)	0.030 (2)	0.017 (2)
C9	0.050 (2)	0.056 (3)	0.049 (2)	0.022 (2)	0.024 (2)	0.011 (2)
C10	0.044 (3)	0.049 (3)	0.052 (3)	0.028 (2)	0.010 (2)	0.010 (2)
C11	0.032 (2)	0.042 (3)	0.048 (3)	0.014 (2)	0.016 (2)	0.009 (2)
C12	0.042 (3)	0.046 (3)	0.057 (3)	0.021 (3)	0.006 (2)	0.002 (3)
C13	0.051 (3)	0.066 (3)	0.052 (3)	0.020 (2)	0.007 (2)	0.007 (2)
C14	0.045 (2)	0.047 (2)	0.061 (2)	0.0160 (19)	0.023 (2)	0.002 (2)

Geometric parameters (Å, °)

N1—C1	1.322 (6)	C6—H6	0.9300
N1—C9	1.366 (5)	C7—C8	1.419 (6)
N1—C10	1.498 (6)	C8—C9	1.352 (5)
N2—C14	1.481 (5)	C8—H8	0.9300
N2—C11	1.501 (6)	C9—H9	0.9300
N2—H2A	0.9000	C10—C11	1.521 (5)
N2—H2B	0.9000	C10—H10A	0.9700
C1—C2	1.387 (5)	C10—H10B	0.9700
C1—H1	0.9300	C11—C12	1.523 (7)
C2—C7	1.410 (5)	C11—H11	0.9800
C2—C3	1.419 (5)	C12—C13	1.516 (6)
C3—C4	1.373 (6)	C12—H12A	0.9700
C3—H3	0.9300	C12—H12B	0.9700
C4—C5	1.404 (6)	C13—C14	1.488 (6)
C4—H4	0.9300	C13—H13A	0.9700
C5—C6	1.366 (6)	C13—H13B	0.9700
C5—H5	0.9300	C14—H14A	0.9700
C6—C7	1.408 (5)	C14—H14B	0.9700
C1—N1—C9	122.6 (4)	C8—C9—N1	119.4 (4)
C1—N1—C10	118.8 (4)	C8—C9—H9	120.3
C9—N1—C10	118.6 (4)	N1—C9—H9	120.3
C14—N2—C11	106.1 (3)	N1—C10—C11	111.4 (3)
C14—N2—H2A	110.5	N1—C10—H10A	109.3

C11—N2—H2A	110.5	C11—C10—H10A	109.3
C14—N2—H2B	110.5	N1—C10—H10B	109.3
C11—N2—H2B	110.5	C11—C10—H10B	109.3
H2A—N2—H2B	108.7	H10A—C10—H10B	108.0
N1—C1—C2	120.3 (4)	N2—C11—C10	111.4 (3)
N1—C1—H1	119.8	N2—C11—C12	104.0 (4)
C2—C1—H1	119.8	C10—C11—C12	112.7 (4)
C1—C2—C7	119.5 (3)	N2—C11—H11	109.5
C1—C2—C3	120.1 (4)	C10—C11—H11	109.5
C7—C2—C3	120.4 (3)	C12—C11—H11	109.5
C4—C3—C2	119.4 (4)	C13—C12—C11	106.9 (4)
C4—C3—H3	120.3	C13—C12—H12A	110.3
C2—C3—H3	120.3	C11—C12—H12A	110.3
C3—C4—C5	119.8 (4)	C13—C12—H12B	110.3
C3—C4—H4	120.1	C11—C12—H12B	110.3
C5—C4—H4	120.1	H12A—C12—H12B	108.6
C6—C5—C4	121.7 (4)	C14—C13—C12	105.2 (3)
C6—C5—H5	119.2	C14—C13—H13A	110.7
C4—C5—H5	119.2	C12—C13—H13A	110.7
C5—C6—C7	120.0 (4)	C14—C13—H13B	110.7
C5—C6—H6	120.0	C12—C13—H13B	110.7
C7—C6—H6	120.0	H13A—C13—H13B	108.8
C6—C7—C2	118.7 (3)	N2—C14—C13	103.6 (3)
C6—C7—C8	124.0 (4)	N2—C14—H14A	111.0
C2—C7—C8	117.3 (3)	C13—C14—H14A	111.0
C9—C8—C7	120.8 (4)	N2—C14—H14B	111.0
C9—C8—H8	119.6	C13—C14—H14B	111.0
C7—C8—H8	119.6	H14A—C14—H14B	109.0
C9—N1—C1—C2	−2.1 (6)	C2—C7—C8—C9	−1.7 (6)
C10—N1—C1—C2	176.0 (4)	C7—C8—C9—N1	0.1 (6)
N1—C1—C2—C7	0.3 (5)	C1—N1—C9—C8	1.9 (6)
N1—C1—C2—C3	−177.1 (4)	C10—N1—C9—C8	−176.2 (4)
C1—C2—C3—C4	176.7 (4)	C1—N1—C10—C11	−101.6 (5)
C7—C2—C3—C4	−0.7 (6)	C9—N1—C10—C11	76.6 (5)
C2—C3—C4—C5	0.4 (6)	C14—N2—C11—C10	149.9 (3)
C3—C4—C5—C6	0.2 (7)	C14—N2—C11—C12	28.2 (5)
C4—C5—C6—C7	−0.5 (6)	N1—C10—C11—N2	60.3 (5)
C5—C6—C7—C2	0.2 (6)	N1—C10—C11—C12	176.7 (5)
C5—C6—C7—C8	−178.2 (4)	N2—C11—C12—C13	−7.2 (6)
C1—C2—C7—C6	−177.1 (3)	C10—C11—C12—C13	−128.0 (4)
C3—C2—C7—C6	0.4 (5)	C11—C12—C13—C14	−16.0 (6)
C1—C2—C7—C8	1.5 (5)	C11—N2—C14—C13	−38.6 (4)
C3—C2—C7—C8	178.9 (4)	C12—C13—C14—N2	33.2 (5)
C6—C7—C8—C9	176.8 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···Br1	0.90	2.30	3.203 (3)	178

N2—H2B···Br2	0.90	2.30	3.180 (3)	166

Fig. 1