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## 2-(2-Quinoly)quinolinium nitrate

Anita Abedi,<sup>a</sup> Arezoo Bahrami Shabestari<sup>a</sup> and Vahid Amani<sup>b\*</sup><sup>a</sup>Department of Chemistry, Islamic Azad University, North Tehran Branch, Tehran, Iran, and <sup>b</sup>Department of Chemistry, Islamic Azad University, Shahr-e-Rey Branch, Tehran, Iran

Correspondence e-mail: v\_amani2002@yahoo.com

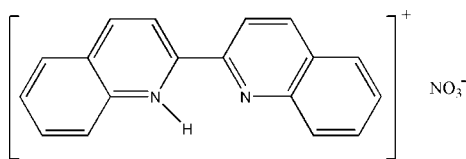
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; disorder in solvent or counterion;  $R$  factor = 0.057;  $wR$  factor = 0.137; data-to-parameter ratio = 16.3.

In the cation of the title compound,  $\text{C}_{18}\text{H}_{13}\text{N}_2^+\cdot\text{NO}_3^-$ , the two bicyclic ring systems form a dihedral angle of  $3.84$  ( $4$ )°. The nitrate anion is disordered over two orientations in a 0.9:0.1 ratio. In the crystal structure, the cations form stacks along the  $a$  axis, with short intermolecular contacts [ $\text{C}\cdots\text{C} = 3.330$  (3) and  $3.345$  (4) Å], and link to the anions *via*  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

## Related literature

For related literature, see: Smith *et al.* (1999); Zafar *et al.* (2000); Rafizadeh *et al.* (2006); Yousefi *et al.* (2007); Parlow & Hartl (1979).



## Experimental

## Crystal data

 $\text{C}_{18}\text{H}_{13}\text{N}_2^+\cdot\text{NO}_3^-$   
 $M_r = 319.31$ 

 Monoclinic,  $P2_1/c$   
 $a = 6.9756$  (6) Å

 $b = 10.6408$  (9) Å  
 $c = 19.1226$  (15) Å  
 $\beta = 94.399$  (2)°  
 $V = 1415.2$  (2) Å<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 120$  (2) K  
 $0.45 \times 0.30 \times 0.25$  mm

## Data collection

 Bruker SMART 1000 CCD area-detector diffractometer  
 Absorption correction: none  
 15078 measured reflections

 3739 independent reflections  
 2115 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.057$   
 $wR(F^2) = 0.136$   
 $S = 0.97$   
 3739 reflections  
 229 parameters

 3 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.29$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2N}\cdots\text{O1}$	0.91	1.92	2.766 (2)	153

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2401).

## References

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**supplementary materials**

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## 2-(2-Quinolyl)quinolinium nitrate

A. Abedi, A. Bahrami Shabestari and V. Amani

### Comment

In recent years, there has been considerable interest in proton transfer systems and their structures (Smith *et al.*, 1999; Zafar *et al.*, 2000; Rafizadeh *et al.*, 2006; Yousefi *et al.*, 2007). To our knowledge, there is only one proton-transfer system with 2,2'-biquinolyl, such as [(biq.H)(I<sub>2</sub>Cl<sub>3</sub>)] [biq.H = 2-(2-quinolyl)quinolinium], which was structurally characterized (Parlow & Hartl, 1979). Herewith we report the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I) contains one cation and one anion (Fig. 1). In the cation, two bicycles form a dihedral angle of 3.84 (4)°. In the crystal structure, the cations form stacks along the *a* axis with short intermolecular contacts [C...C = 3.330 (3) and 3.345 (4)Å] linking the anions *via* N—H...O hydrogen bonds.

### Experimental

For the preparation of the title compound, (I), a solution of 2,2'-biquinolyl (0.20 g, 0.78 mmol) in HNO<sub>3</sub> 0.5 M (10 ml) was added to a solution of La(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O, (0.11 g, 0.26 mmol) in water (5 ml) and the resulting yellow solution was stirred at 333 K for 2 h. Then, it was left to evaporate slowly at room temperature. The suitable crystals for X-ray diffraction experiment were obtained by methanol diffusion in a solution of yellow precipitated in DMSO after one week (yield 0.19 g, 76.2%, m.p 496–497 K).

### Refinement

C-bound H atoms were geometrically positioned (C-H 0.95 Å). The H atom of NH group was located on a difference Fourier map, but placed in idealized position (N-H 0.91 Å). All H atoms were refined in riding model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}$  of the parent atom. The NO<sub>3</sub> anion was treated as disordered between two orientations with the occupancies fixed to 0.9 and 0.1, respectively.

### Figures

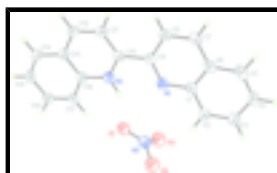


Fig. 1. The content of asymmetric unit of (I) showing the atomic numbering and 50% probability displacement ellipsoids. Only major part of the disordered nitrate anion is shown.

## 2-(2-Quinoly)quinolinium nitrate

### Crystal data

$C_{18}H_{13}N_2^+ \cdot NO_3^-$	$F_{000} = 664$
$M_r = 319.31$	$D_x = 1.499 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 6.9756 (6) \text{ \AA}$	Cell parameters from 937 reflections
$b = 10.6408 (9) \text{ \AA}$	$\theta = 3\text{--}29^\circ$
$c = 19.1226 (15) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$\beta = 94.399 (2)^\circ$	$T = 120 (2) \text{ K}$
$V = 1415.2 (2) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.45 \times 0.30 \times 0.25 \text{ mm}$

### Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2115 reflections with $I > 2\sigma(I)$
Radiation source: normal-focus sealed tube	$R_{\text{int}} = 0.038$
Monochromator: graphite	$\theta_{\text{max}} = 29.0^\circ$
$T = 120(2) \text{ K}$	$\theta_{\text{min}} = 2.1^\circ$
$\omega$ scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -14 \rightarrow 14$
15078 measured reflections	$l = -25 \rightarrow 26$
3739 independent reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.057$	H-atom parameters constrained
$wR(F^2) = 0.136$	$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.46P]$
$S = 0.97$	where $P = (F_o^2 + 2F_c^2)/3$
3739 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
229 parameters	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
3 restraints	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.2846 (2)	0.12303 (14)	0.47447 (7)	0.0261 (3)	
N2	0.21783 (19)	-0.05200 (13)	0.56913 (7)	0.0245 (3)	
H2N	0.2249	0.0316	0.5802	0.029*	
C1	0.3241 (2)	0.21681 (17)	0.42885 (9)	0.0265 (4)	
C2	0.3386 (3)	0.34167 (17)	0.45393 (10)	0.0320 (4)	
H2A	0.3180	0.3593	0.5015	0.038*	
C3	0.3821 (3)	0.43694 (19)	0.41010 (10)	0.0352 (5)	
H3A	0.3920	0.5205	0.4274	0.042*	
C4	0.4124 (3)	0.4124 (2)	0.33902 (10)	0.0347 (5)	
H4A	0.4448	0.4794	0.3093	0.042*	
C5	0.3955 (3)	0.29307 (19)	0.31289 (10)	0.0329 (4)	
H5A	0.4127	0.2780	0.2648	0.040*	
C6	0.3524 (2)	0.19166 (18)	0.35718 (9)	0.0283 (4)	
C7	0.3393 (3)	0.06604 (19)	0.33471 (9)	0.0319 (4)	
H7A	0.3553	0.0460	0.2871	0.038*	
C8	0.3037 (3)	-0.02721 (18)	0.38088 (9)	0.0307 (4)	
H8A	0.2981	-0.1126	0.3663	0.037*	
C9	0.2754 (2)	0.00614 (17)	0.45085 (9)	0.0243 (4)	
C10	0.2313 (2)	-0.09095 (17)	0.50285 (9)	0.0249 (4)	
C11	0.2028 (3)	-0.21806 (17)	0.48705 (10)	0.0309 (4)	
H11A	0.2123	-0.2471	0.4405	0.037*	
C12	0.1613 (3)	-0.30107 (17)	0.53859 (10)	0.0321 (4)	
H12A	0.1413	-0.3873	0.5273	0.038*	
C13	0.1480 (2)	-0.26001 (17)	0.60813 (9)	0.0272 (4)	
C14	0.1044 (2)	-0.34060 (17)	0.66293 (10)	0.0313 (4)	
H14A	0.0826	-0.4274	0.6539	0.038*	
C15	0.0931 (3)	-0.29430 (18)	0.72935 (10)	0.0332 (4)	
H15A	0.0621	-0.3492	0.7660	0.040*	
C16	0.1272 (3)	-0.16592 (18)	0.74375 (10)	0.0326 (4)	
H16A	0.1207	-0.1355	0.7902	0.039*	
C17	0.1696 (2)	-0.08437 (17)	0.69156 (9)	0.0285 (4)	
H17A	0.1928	0.0020	0.7015	0.034*	
C18	0.1781 (2)	-0.13100 (17)	0.62339 (9)	0.0248 (4)	
N3	0.2376 (3)	0.27946 (17)	0.62588 (8)	0.0385 (4)	
O1	0.1506 (3)	0.17769 (16)	0.63037 (10)	0.0433 (5)	0.90
O2	0.1410 (3)	0.37683 (15)	0.60925 (9)	0.0502 (5)	0.90
O3	0.4157 (3)	0.2874 (2)	0.63555 (9)	0.0555 (5)	0.90
O1'	0.0850 (18)	0.2168 (18)	0.6230 (13)	0.060 (7)*	0.10

## supplementary materials

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O2'	0.264 (3)	0.3897 (8)	0.6450 (11)	0.077 (6)*	0.10
O3'	0.3749 (19)	0.2024 (14)	0.6346 (9)	0.054 (4)*	0.10

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0253 (8)	0.0271 (8)	0.0258 (8)	0.0003 (6)	0.0022 (6)	0.0036 (6)
N2	0.0255 (8)	0.0229 (7)	0.0249 (8)	0.0000 (6)	0.0011 (6)	-0.0003 (6)
C1	0.0239 (9)	0.0306 (10)	0.0249 (9)	0.0021 (7)	0.0017 (7)	0.0031 (8)
C2	0.0371 (11)	0.0314 (10)	0.0280 (10)	0.0010 (8)	0.0063 (8)	0.0017 (8)
C3	0.0368 (11)	0.0306 (10)	0.0384 (11)	0.0008 (8)	0.0044 (9)	0.0054 (8)
C4	0.0289 (10)	0.0431 (12)	0.0323 (10)	0.0004 (8)	0.0027 (8)	0.0144 (9)
C5	0.0274 (10)	0.0473 (12)	0.0239 (9)	0.0000 (9)	0.0010 (7)	0.0069 (8)
C6	0.0214 (9)	0.0393 (11)	0.0243 (9)	0.0021 (8)	0.0014 (7)	0.0027 (8)
C7	0.0286 (10)	0.0442 (12)	0.0230 (9)	-0.0017 (8)	0.0035 (7)	-0.0013 (8)
C8	0.0329 (10)	0.0318 (10)	0.0273 (10)	-0.0010 (8)	0.0029 (8)	-0.0046 (8)
C9	0.0214 (8)	0.0288 (9)	0.0226 (8)	0.0006 (7)	0.0001 (7)	-0.0005 (7)
C10	0.0203 (8)	0.0292 (10)	0.0251 (9)	0.0016 (7)	0.0013 (7)	-0.0017 (7)
C11	0.0332 (10)	0.0299 (10)	0.0295 (10)	0.0002 (8)	0.0026 (8)	-0.0047 (8)
C12	0.0310 (10)	0.0240 (9)	0.0412 (11)	-0.0010 (8)	0.0029 (8)	-0.0041 (8)
C13	0.0215 (9)	0.0282 (9)	0.0319 (10)	0.0015 (7)	0.0027 (7)	0.0018 (8)
C14	0.0280 (10)	0.0241 (9)	0.0417 (11)	-0.0014 (7)	0.0024 (8)	0.0046 (8)
C15	0.0290 (10)	0.0336 (10)	0.0375 (11)	-0.0009 (8)	0.0053 (8)	0.0118 (8)
C16	0.0339 (10)	0.0364 (11)	0.0278 (10)	0.0036 (8)	0.0048 (8)	0.0024 (8)
C17	0.0287 (10)	0.0288 (9)	0.0282 (9)	0.0013 (8)	0.0027 (7)	-0.0003 (8)
C18	0.0203 (8)	0.0258 (9)	0.0283 (9)	0.0016 (7)	0.0017 (7)	0.0036 (7)
N3	0.0534 (11)	0.0360 (10)	0.0272 (9)	-0.0011 (9)	0.0102 (8)	-0.0040 (7)
O1	0.0697 (12)	0.0249 (9)	0.0380 (10)	-0.0089 (10)	0.0211 (9)	-0.0020 (8)
O2	0.0790 (13)	0.0276 (9)	0.0460 (10)	0.0114 (9)	0.0186 (9)	0.0005 (7)
O3	0.0514 (12)	0.0705 (14)	0.0440 (11)	-0.0145 (10)	0.0011 (8)	0.0099 (9)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—C9	1.323 (2)	C10—C11	1.397 (3)
N1—C1	1.367 (2)	C11—C12	1.371 (3)
N2—C10	1.344 (2)	C11—H11A	0.9500
N2—C18	1.380 (2)	C12—C13	1.410 (3)
N2—H2N	0.910	C12—H12A	0.9500
C1—C2	1.413 (3)	C13—C14	1.405 (2)
C1—C6	1.425 (2)	C13—C18	1.416 (3)
C2—C3	1.364 (3)	C14—C15	1.370 (3)
C2—H2A	0.9500	C14—H14A	0.9500
C3—C4	1.416 (3)	C15—C16	1.410 (3)
C3—H3A	0.9500	C15—H15A	0.9500
C4—C5	1.366 (3)	C16—C17	1.372 (3)
C4—H4A	0.9500	C16—H16A	0.9500
C5—C6	1.418 (3)	C17—C18	1.400 (2)
C5—H5A	0.9500	C17—H17A	0.9500
C6—C7	1.405 (3)	N3—O2'	1.239 (5)

C7—C8	1.364 (3)	N3—O3	1.245 (2)
C7—H7A	0.9500	N3—O1	1.247 (2)
C8—C9	1.413 (2)	N3—O1'	1.253 (5)
C8—H8A	0.9500	N3—O3'	1.263 (5)
C9—C10	1.483 (2)	N3—O2	1.263 (2)
N1...C18 <sup>i</sup>	3.606 (3)	C6...C14 <sup>i</sup>	3.550 (4)
N1...C10 <sup>ii</sup>	3.388 (3)	C18...C6 <sup>ii</sup>	3.330 (3)
C1...C13 <sup>i</sup>	3.345 (4)		
C9—N1—C1	118.36 (15)	N2—C10—C9	116.81 (15)
C10—N2—C18	123.64 (15)	C11—C10—C9	124.27 (16)
C10—N2—H2N	120.8	C12—C11—C10	120.19 (17)
C18—N2—H2N	115.4	C12—C11—H11A	119.9
N1—C1—C2	118.83 (16)	C10—C11—H11A	119.9
N1—C1—C6	121.75 (16)	C11—C12—C13	120.75 (17)
C2—C1—C6	119.42 (16)	C11—C12—H12A	119.6
C3—C2—C1	120.24 (17)	C13—C12—H12A	119.6
C3—C2—H2A	119.9	C14—C13—C12	123.24 (17)
C1—C2—H2A	119.9	C14—C13—C18	118.36 (17)
C2—C3—C4	120.60 (19)	C12—C13—C18	118.40 (16)
C2—C3—H3A	119.7	C15—C14—C13	120.20 (17)
C4—C3—H3A	119.7	C15—C14—H14A	119.9
C5—C4—C3	120.51 (18)	C13—C14—H14A	119.9
C5—C4—H4A	119.7	C14—C15—C16	120.56 (17)
C3—C4—H4A	119.7	C14—C15—H15A	119.7
C4—C5—C6	120.33 (17)	C16—C15—H15A	119.7
C4—C5—H5A	119.8	C17—C16—C15	120.89 (18)
C6—C5—H5A	119.8	C17—C16—H16A	119.6
C7—C6—C5	123.57 (17)	C15—C16—H16A	119.6
C7—C6—C1	117.54 (16)	C16—C17—C18	118.69 (17)
C5—C6—C1	118.88 (17)	C16—C17—H17A	120.7
C8—C7—C6	120.34 (17)	C18—C17—H17A	120.7
C8—C7—H7A	119.8	N2—C18—C17	120.62 (16)
C6—C7—H7A	119.8	N2—C18—C13	118.09 (16)
C7—C8—C9	118.43 (17)	C17—C18—C13	121.29 (16)
C7—C8—H8A	120.8	O3—N3—O1	122.3 (2)
C9—C8—H8A	120.8	O2'—N3—O1'	128.7 (14)
N1—C9—C8	123.57 (16)	O2'—N3—O3'	118.7 (14)
N1—C9—C10	115.67 (15)	O1'—N3—O3'	107.1 (13)
C8—C9—C10	120.77 (16)	O3—N3—O2	119.2 (2)
N2—C10—C11	118.92 (16)	O1—N3—O2	118.5 (2)
C9—N1—C1—C2	-178.56 (16)	N1—C9—C10—N2	-4.2 (2)
C9—N1—C1—C6	0.9 (2)	C8—C9—C10—N2	176.18 (15)
N1—C1—C2—C3	178.55 (17)	N1—C9—C10—C11	175.52 (16)
C6—C1—C2—C3	-1.0 (3)	C8—C9—C10—C11	-4.1 (3)
C1—C2—C3—C4	0.3 (3)	N2—C10—C11—C12	0.2 (3)
C2—C3—C4—C5	1.1 (3)	C9—C10—C11—C12	-179.57 (16)
C3—C4—C5—C6	-1.7 (3)	C10—C11—C12—C13	-0.4 (3)
C4—C5—C6—C7	-177.72 (18)	C11—C12—C13—C14	179.46 (17)

## supplementary materials

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C4—C5—C6—C1	1.0 (3)	C11—C12—C13—C18	0.2 (3)
N1—C1—C6—C7	-0.3 (3)	C12—C13—C14—C15	-179.80 (17)
C2—C1—C6—C7	179.14 (16)	C18—C13—C14—C15	-0.5 (3)
N1—C1—C6—C5	-179.15 (16)	C13—C14—C15—C16	-0.7 (3)
C2—C1—C6—C5	0.3 (3)	C14—C15—C16—C17	0.9 (3)
C5—C6—C7—C8	177.74 (17)	C15—C16—C17—C18	0.1 (3)
C1—C6—C7—C8	-1.0 (3)	C10—N2—C18—C17	178.81 (15)
C6—C7—C8—C9	1.7 (3)	C10—N2—C18—C13	-0.6 (2)
C1—N1—C9—C8	-0.2 (2)	C16—C17—C18—N2	179.18 (16)
C1—N1—C9—C10	-179.75 (15)	C16—C17—C18—C13	-1.4 (3)
C7—C8—C9—N1	-1.1 (3)	C14—C13—C18—N2	-178.98 (15)
C7—C8—C9—C10	178.41 (16)	C12—C13—C18—N2	0.3 (2)
C18—N2—C10—C11	0.4 (2)	C14—C13—C18—C17	1.6 (2)
C18—N2—C10—C9	-179.86 (14)	C12—C13—C18—C17	-179.08 (16)

Symmetry codes: (i)  $-x, -y, -z+1$ ; (ii)  $-x+1, -y, -z+1$ .

### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2N $\cdots$ O1	0.91	1.92	2.766 (2)	153

Fig. 1

