

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2,3,3-Trimethyl-1-[4-(2,3,3-trimethyl-3H-indol-1-ium-1-yl)butyl]-3H-indol-1-ium diiodide

Di Wu,<sup>a</sup> Li-Xia Shen,<sup>a</sup> Yun-Yin Niu<sup>a</sup> and Seik Weng Ng<sup>b,c,\*</sup>

<sup>a</sup>College of Chemistry and Molecular Engineering, Zhengzhou University, Zhengzhou, People's Republic of China, <sup>b</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and <sup>c</sup>Chemistry Department, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia  
Correspondence e-mail: seikweng@um.edu.my

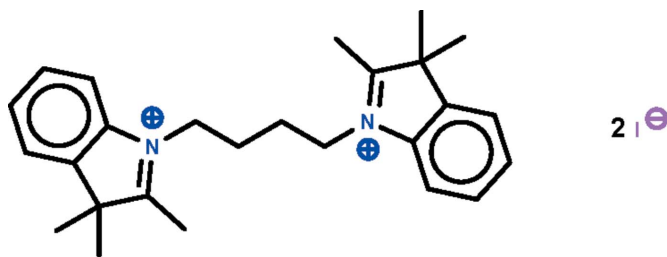
Received 31 July 2012; accepted 9 August 2012

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å; disorder in solvent or counterion;  $R$  factor = 0.027;  $wR$  factor = 0.087; data-to-parameter ratio = 21.1.

In the crystal of the title salt,  $\text{C}_{26}\text{H}_{34}\text{N}_2^{2+}\cdot 2\text{I}^-$ , the dication lies on a center of inversion that exists along the mid-point of the butyl chain; its five-membered ring is approximately planar (r.m.s. deviation = 0.011 Å). In the crystal, the iodide anion is disordered over two positions in a 1:1 ratio.

### Related literature

For the synthesis, see: Yang *et al.* (2005). For industrial applications of Mannich products, see: Su *et al.* (2005).



### Experimental

#### Crystal data

$\text{C}_{26}\text{H}_{34}\text{N}_2^{2+}\cdot 2\text{I}^-$   
 $M_r = 628.35$   
 Monoclinic,  $P2_1/c$   
 $a = 13.9414$  (14) Å  
 $b = 7.6013$  (8) Å  
 $c = 13.8261$  (15) Å  
 $\beta = 113.011$  (2)°  
 $V = 1348.6$  (2) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 2.35$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.35 \times 0.30 \times 0.25$  mm

#### Data collection

Bruker SMART APEX diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.494$ ,  $T_{\max} = 0.592$   
 8426 measured reflections  
 3077 independent reflections  
 2602 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.017$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.087$   
 $S = 1.04$   
 3077 reflections  
 146 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.94$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2010).

We thank the National Natural Science Foundation of China (No. J1103309), the Students' Innovative Undertaking Training Project of Zhengzhou University (No. 121045919) and the Ministry of Higher Education of Malaysia (grant No. UM·C/HIR/MOHE/SC/12) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5603).

### References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
 Bruker (2005). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Su, J.-H., Meng, F.-S., Tian, H., Li, C., Wang, H.-L. & Chen, K.-C. (2005). *Faming Zhuanli Shenqing* (Chin. Patent) 1563201 A 20050112.  
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.  
 Yang, Z.-F., An, J., Yang, Y.-M. & Cheng, W. (2005). *Changchun Gongye Daxue Xuebao, Ziran Kexueban*, **26**, 8–10.

## supporting information

*Acta Cryst.* (2012). E68, o2714 [doi:10.1107/S1600536812035234]

## 2,3,3-Trimethyl-1-[4-(2,3,3-trimethyl-3*H*-indol-1-ium-1-yl)butyl]-3*H*-indol-1-ium diiodide

Di Wu, Li-Xia Shen, Yun-Yin Niu and Seik Weng Ng

### S1. Comment

The salt (Scheme I) can be synthesized in several steps from benzohydrazine, methyl isopropyl ketone and 1,4-diiodobutane, and is an intermediate in the synthesis of a bis-spiro-naphthoxazine (Yang *et al.*, 2005). It is available commercially in milligram quantities. The methyl group on the carbon atom adjacent to the quaternary nitrogen is acidic, and the methyl group undergoes a Mannich-type of reaction to yield compounds that have been patented for use as high-density recording media (Su *et al.*, 2005).

In the salt,  $C_{26}H_{34}N_2^+ 2I^-$ , the dicationic species lies on a center-of-inversion that exists along the mid-point of the butyl chain; its five-membered ring is planar (r.m.s. deviation 0.011 Å). The iodide anion is disordered over two positions in a 1:1 ratio (Fig. 1).

### S2. Experimental

3,3-Dimethyl-3*H*-indole (40 mmol, 5.81 g), 1,4-dibromobutane (20 mmol, 4.32 g) and potassium iodide (50 mmol, 8.310) were added to acetonitrile (40 ml). The mixture was heated under reflux for 4 days. The mixture was collected and the yellow solid recrystallized from a methanol-ether mixture; yield 40%. The salt is soluble in most organic solvents.

### S3. Refinement

Carbon-, nitrogen- and oxygen-bound H-atoms were placed in calculated positions (C–H 0.93 to 0.97, N–H 0.88, O–H 0.84 Å) and were included in the refinement in the riding model approximation, with  $U(H)$  set to 1.2 to 1.5 $U(C,N,O)$ .

The iodine atom is disordered over two positions; as the disorder refined to a nearly 1:1 ratio, the occupancy was then fixed as exactly 0.5.

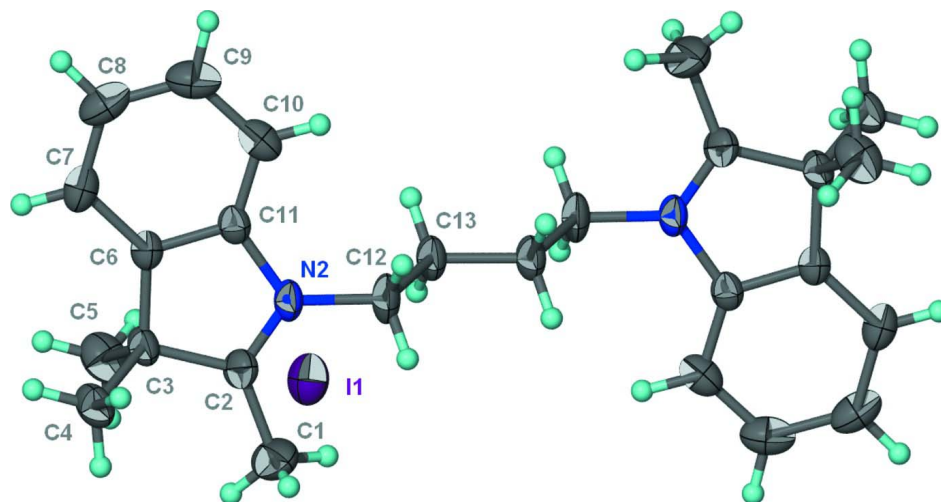


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of  $C_{26}H_{34}N_2^{2+} 2I^-$  at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The disorder in the iodine atom is not shown.

### 2,3,3-Trimethyl-1-[4-(2,3,3-trimethyl-3H-indol-1-ium-1-yl)butyl]- 3H-indol-1-ium diiodide

#### Crystal data

$C_{26}H_{34}N_2^{2+} \cdot 2I^-$

$M_r = 628.35$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P 2_1/c$

$a = 13.9414 (14) \text{ \AA}$

$b = 7.6013 (8) \text{ \AA}$

$c = 13.8261 (15) \text{ \AA}$

$\beta = 113.011 (2)^\circ$

$V = 1348.6 (2) \text{ \AA}^3$

$Z = 2$

$F(000) = 620$

$D_x = 1.547 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3950 reflections

$\theta = 3.0\text{--}28.3^\circ$

$\mu = 2.35 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prism, colorless

$0.35 \times 0.30 \times 0.25 \text{ mm}$

#### Data collection

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.494$ ,  $T_{\max} = 0.592$

8426 measured reflections

3077 independent reflections

2602 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.0^\circ$

$h = -13 \rightarrow 17$

$k = -8 \rightarrow 9$

$l = -17 \rightarrow 14$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.087$

$S = 1.04$

3077 reflections

146 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.3658P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.94 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
I1	0.3111 (2)	0.6289 (3)	0.77695 (19)	0.0536 (2)	0.50
I1'	0.3220 (2)	0.6038 (3)	0.7930 (2)	0.0800 (6)	0.50
N2	0.31190 (14)	0.3641 (2)	0.55053 (14)	0.0382 (4)	
C1	0.3943 (2)	0.1407 (5)	0.6859 (3)	0.0789 (11)	
H1A	0.3772	0.0208	0.6943	0.118*	
H1B	0.4081	0.2039	0.7501	0.118*	
H1C	0.4550	0.1435	0.6693	0.118*	
C2	0.30596 (17)	0.2235 (3)	0.59984 (18)	0.0437 (5)	
C3	0.19566 (18)	0.1564 (3)	0.56180 (19)	0.0398 (5)	
C4	0.1613 (2)	0.1429 (3)	0.6543 (2)	0.0514 (6)	
H4A	0.1669	0.2562	0.6867	0.077*	
H4B	0.2051	0.0604	0.7049	0.077*	
H4C	0.0902	0.1035	0.6292	0.077*	
C5	0.1875 (3)	-0.0238 (4)	0.5086 (2)	0.0621 (7)	
H5A	0.2092	-0.0131	0.4510	0.093*	
H5B	0.1166	-0.0640	0.4830	0.093*	
H5C	0.2316	-0.1067	0.5586	0.093*	
C6	0.13926 (16)	0.2961 (3)	0.48332 (16)	0.0358 (4)	
C7	0.03506 (19)	0.3190 (3)	0.4208 (2)	0.0484 (5)	
H7	-0.0147	0.2402	0.4239	0.058*	
C8	0.0064 (2)	0.4603 (4)	0.3539 (2)	0.0585 (7)	
H8	-0.0635	0.4773	0.3112	0.070*	
C9	0.0803 (3)	0.5787 (4)	0.3488 (2)	0.0585 (7)	
H9	0.0590	0.6731	0.3025	0.070*	
C10	0.1846 (2)	0.5586 (3)	0.41111 (19)	0.0475 (5)	
H10	0.2345	0.6375	0.4084	0.057*	
C11	0.21086 (17)	0.4165 (3)	0.47728 (16)	0.0352 (4)	
C12	0.40689 (18)	0.4695 (4)	0.56978 (19)	0.0491 (6)	
H12	0.4593	0.4384	0.6380	0.059*	
H12B	0.3904	0.5931	0.5719	0.059*	
C13	0.45182 (18)	0.4435 (3)	0.48771 (18)	0.0435 (5)	
H13	0.4001	0.4747	0.4191	0.052*	
H13B	0.4699	0.3206	0.4858	0.052*	

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
I1	0.0481 (5)	0.0585 (3)	0.0626 (3)	-0.0023 (3)	0.0307 (3)	-0.0126 (3)
I1'	0.0531 (7)	0.0987 (14)	0.1030 (14)	-0.0154 (8)	0.0466 (9)	-0.0430 (8)
N2	0.0294 (9)	0.0511 (10)	0.0395 (9)	-0.0101 (7)	0.0193 (7)	-0.0044 (7)
C1	0.0413 (16)	0.107 (3)	0.083 (2)	0.0150 (15)	0.0190 (15)	0.0413 (18)

C2	0.0320 (11)	0.0575 (14)	0.0466 (12)	0.0011 (9)	0.0206 (9)	0.0075 (10)
C3	0.0342 (11)	0.0396 (11)	0.0514 (12)	-0.0026 (8)	0.0230 (9)	0.0066 (9)
C4	0.0516 (15)	0.0559 (14)	0.0590 (15)	-0.0019 (11)	0.0348 (12)	0.0082 (11)
C5	0.0748 (19)	0.0467 (14)	0.0763 (19)	0.0018 (13)	0.0420 (16)	-0.0007 (13)
C6	0.0317 (10)	0.0376 (10)	0.0406 (10)	-0.0031 (8)	0.0168 (8)	-0.0027 (8)
C7	0.0343 (12)	0.0543 (13)	0.0556 (14)	-0.0054 (10)	0.0166 (10)	-0.0083 (11)
C8	0.0460 (15)	0.0679 (17)	0.0498 (14)	0.0172 (13)	0.0058 (11)	-0.0049 (12)
C9	0.075 (2)	0.0531 (14)	0.0478 (14)	0.0189 (13)	0.0246 (13)	0.0090 (11)
C10	0.0623 (15)	0.0396 (11)	0.0493 (12)	-0.0011 (11)	0.0313 (11)	0.0025 (10)
C11	0.0354 (11)	0.0383 (10)	0.0365 (10)	-0.0052 (8)	0.0191 (8)	-0.0036 (8)
C12	0.0368 (12)	0.0683 (16)	0.0487 (12)	-0.0237 (11)	0.0237 (10)	-0.0136 (11)
C13	0.0343 (11)	0.0568 (13)	0.0459 (12)	-0.0135 (10)	0.0226 (9)	-0.0053 (10)

*Geometric parameters (Å, °)*

N2—C2	1.287 (3)	C6—C11	1.381 (3)
N2—C11	1.433 (3)	C6—C7	1.381 (3)
N2—C12	1.480 (3)	C7—C8	1.371 (4)
C1—C2	1.478 (4)	C7—H7	0.9300
C1—H1A	0.9600	C8—C9	1.390 (4)
C1—H1B	0.9600	C8—H8	0.9300
C1—H1C	0.9600	C9—C10	1.378 (4)
C2—C3	1.507 (3)	C9—H9	0.9300
C3—C6	1.502 (3)	C10—C11	1.370 (3)
C3—C4	1.535 (3)	C10—H10	0.9300
C3—C5	1.538 (4)	C12—C13	1.508 (3)
C4—H4A	0.9600	C12—H12	0.9700
C4—H4B	0.9600	C12—H12B	0.9700
C4—H4C	0.9600	C13—C13 <sup>i</sup>	1.517 (4)
C5—H5A	0.9600	C13—H13	0.9700
C5—H5B	0.9600	C13—H13B	0.9700
C5—H5C	0.9600		
C2—N2—C11	110.85 (18)	C11—C6—C7	119.1 (2)
C2—N2—C12	126.3 (2)	C11—C6—C3	109.00 (19)
C11—N2—C12	122.67 (19)	C7—C6—C3	131.9 (2)
C2—C1—H1A	109.5	C8—C7—C6	118.6 (2)
C2—C1—H1B	109.5	C8—C7—H7	120.7
H1A—C1—H1B	109.5	C6—C7—H7	120.7
C2—C1—H1C	109.5	C7—C8—C9	121.0 (2)
H1A—C1—H1C	109.5	C7—C8—H8	119.5
H1B—C1—H1C	109.5	C9—C8—H8	119.5
N2—C2—C1	125.0 (2)	C10—C9—C8	121.2 (2)
N2—C2—C3	111.39 (19)	C10—C9—H9	119.4
C1—C2—C3	123.5 (2)	C8—C9—H9	119.4
C6—C3—C2	100.79 (17)	C11—C10—C9	116.5 (2)
C6—C3—C4	113.8 (2)	C11—C10—H10	121.8
C2—C3—C4	109.9 (2)	C9—C10—H10	121.8

C6—C3—C5	111.4 (2)	C10—C11—C6	123.5 (2)
C2—C3—C5	110.1 (2)	C10—C11—N2	128.6 (2)
C4—C3—C5	110.5 (2)	C6—C11—N2	107.90 (18)
C3—C4—H4A	109.5	N2—C12—C13	113.52 (18)
C3—C4—H4B	109.5	N2—C12—H12	108.9
H4A—C4—H4B	109.5	C13—C12—H12	108.9
C3—C4—H4C	109.5	N2—C12—H12B	108.9
H4A—C4—H4C	109.5	C13—C12—H12B	108.9
H4B—C4—H4C	109.5	H12—C12—H12B	107.7
C3—C5—H5A	109.5	C12—C13—C13 <sup>i</sup>	110.4 (2)
C3—C5—H5B	109.5	C12—C13—H13	109.6
H5A—C5—H5B	109.5	C13 <sup>i</sup> —C13—H13	109.6
C3—C5—H5C	109.5	C12—C13—H13B	109.6
H5A—C5—H5C	109.5	C13 <sup>i</sup> —C13—H13B	109.6
H5B—C5—H5C	109.5	H13—C13—H13B	108.1
C11—N2—C2—C1	-175.6 (3)	C3—C6—C7—C8	-178.7 (2)
C12—N2—C2—C1	-0.1 (4)	C6—C7—C8—C9	-0.1 (4)
C11—N2—C2—C3	2.1 (3)	C7—C8—C9—C10	-0.4 (4)
C12—N2—C2—C3	177.6 (2)	C8—C9—C10—C11	0.2 (4)
N2—C2—C3—C6	-2.7 (3)	C9—C10—C11—C6	0.3 (4)
C1—C2—C3—C6	175.0 (3)	C9—C10—C11—N2	-179.6 (2)
N2—C2—C3—C4	-123.1 (2)	C7—C6—C11—C10	-0.7 (3)
C1—C2—C3—C4	54.6 (3)	C3—C6—C11—C10	178.7 (2)
N2—C2—C3—C5	114.9 (2)	C7—C6—C11—N2	179.17 (19)
C1—C2—C3—C5	-67.3 (3)	C3—C6—C11—N2	-1.4 (2)
C2—C3—C6—C11	2.4 (2)	C2—N2—C11—C10	179.5 (2)
C4—C3—C6—C11	120.0 (2)	C12—N2—C11—C10	3.7 (3)
C5—C3—C6—C11	-114.4 (2)	C2—N2—C11—C6	-0.4 (3)
C2—C3—C6—C7	-178.3 (2)	C12—N2—C11—C6	-176.17 (19)
C4—C3—C6—C7	-60.7 (3)	C2—N2—C12—C13	104.5 (3)
C5—C3—C6—C7	65.0 (3)	C11—N2—C12—C13	-80.5 (3)
C11—C6—C7—C8	0.6 (4)	N2—C12—C13—C13 <sup>i</sup>	179.6 (3)

Symmetry code: (i)  $-x+1, -y+1, -z+1$ .