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## Structure Reports

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# Low-temperature redetermination of benzofurazan 1-oxide

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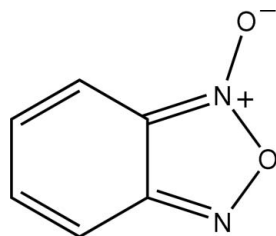
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 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.035;  $wR$  factor = 0.110; data-to-parameter ratio = 11.8.

In the six-membered ring of the low-temperature crystal structure of benzofurazan 1-oxide,  $\text{C}_6\text{H}_4\text{N}_2\text{O}_2$ , the two C atoms adjacent to the N atoms are linked by a delocalized aromatic bond [1.402 (2) Å]; each is connected to its neighbour by a longer, more localized, bond [1.420 (2), 1.430 (2) Å]. However, the next two bonds in the ring approximate double bonds [1.357 (2), 1.366 (2) Å]. As such, the six-membered ring is better described as a cyclohexadiene system, in contrast to the description in the room-temperature structure reported by Britton & Olson (1979) [*Acta Cryst. B* **35**, 3076–3078].

## Related literature

For the room-temperature structure in the  $P\bar{1}$  setting [6.772 (3), 7.515 (4), 7.759 (4) Å, 99.08 (3), 114.94 (3), 112.67 (3)°], see: Britton & Olson (1979). For the geometry-optimized structure, see: Friedrichsen, 1995; Ponder *et al.* (1994); Rauhut (1996). For details of the synthesis, see: Terrian *et al.* (1992); Wolthius (1979). For work mentioning the original structure, see: Ammon & Bhattacharjee (1982); Bird (1993); Cerecetto & González (2007); Ojala *et al.* (1999); Ramm *et al.* (1991).



## Experimental

### Crystal data

 $\text{C}_6\text{H}_4\text{N}_2\text{O}_2$ 
 $M_r = 136.11$ 

 Triclinic,  $P\bar{1}$ 
 $a = 6.6751$  (2) Å

 $b = 7.3256$  (2) Å

 $c = 7.6842$  (2) Å

 $\alpha = 100.710$  (2)°

 $\beta = 114.265$  (2)°

 $\gamma = 111.747$  (2)°

 $V = 291.71$  (1) Å<sup>3</sup>
 $Z = 2$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.12$  mm<sup>-1</sup>
 $T = 100$  K

 $0.30 \times 0.25 \times 0.10$  mm

### Data collection

 Bruker SMART APEX  
 diffractometer  
 Absorption correction: none  
 1952 measured reflections

 1276 independent reflections  
 1110 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.012$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$ 
 $wR(F^2) = 0.110$ 
 $S = 1.03$ 

1276 reflections

108 parameters

4 restraints

All H-atom parameters refined

 $\Delta\rho_{\text{max}} = 0.33$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>
**Table 1**

Selected bond lengths (Å).

O1–N1	1.230 (1)	C1–C2	1.430 (2)
O2–N2	1.381 (1)	C2–C3	1.357 (2)
O2–N1	1.443 (2)	C3–C4	1.436 (2)
N1–C6	1.336 (2)	C4–C5	1.366 (2)
N2–C1	1.327 (2)	C5–C6	1.420 (2)
C1–C6	1.409 (2)		

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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, 2009).

I thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2443).

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## supporting information

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## Low-temperature redetermination of benzofurazan 1-oxide

Seik Weng Ng

### S1. Comment

Researchers have used the published structure of benzofurazan 1-oxide (Britton & Olson, 1979) in, for example, studies on packing (Ammon & Bhattacharjee, 1982; Ojala *et al.*, 1999; Ramm *et al.*, 1991), influence of *N*-oxide formation on heteroaromaticity (Bird, 1993), and reactivity and biology (Cerecetto & González, 2007). Bond dimensions from geometry-optimization calculations (Friedrichsen, 1995; Ponder *et al.*, 1994; Rauhut, 1996) have also been compared with values taken from the solid-state structure.

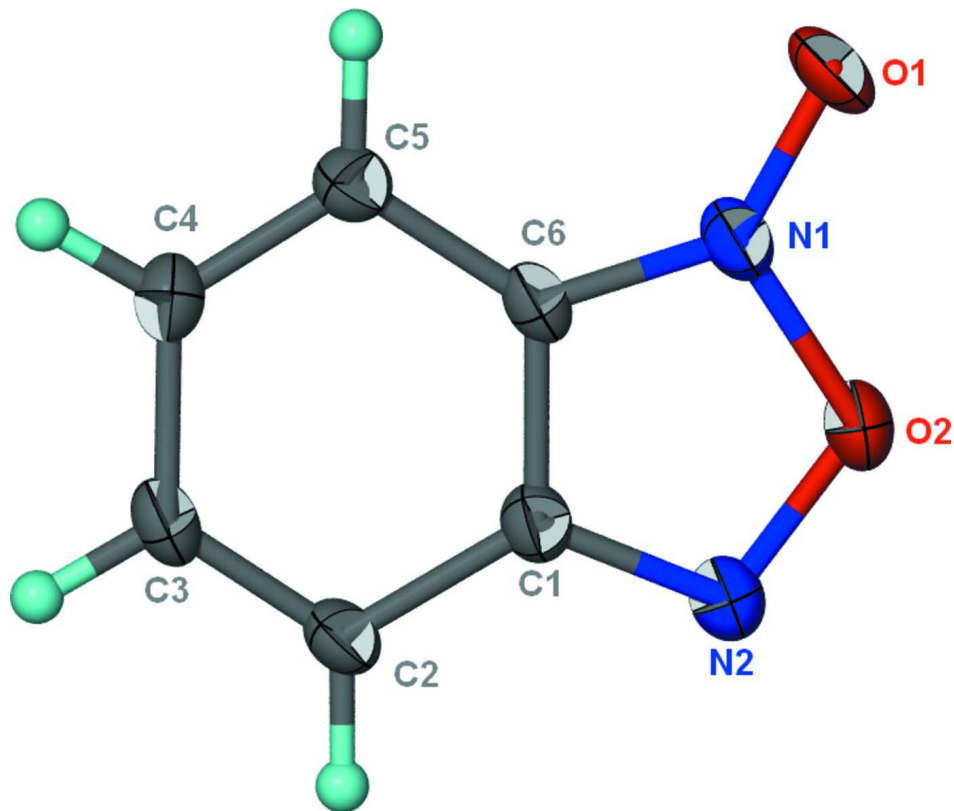
The present low-temperature structure (Fig. 1 & Table 1) reveals features quite distinct from those disclosed in the original, room-temperature, analysis (Britton & Olson, 1979). In the six-membered ring, the two carbon atoms adjacent to the nitrogen atoms are linked by a delocalized aromatic bond [1.402 (2) Å]; each is connected to its neighbor by a longer, more localized, bond [1.420 (2), 1.430 (2) Å]. However, the next two bonds in the ring approximate double-bonds [1.357 (2), 1.366 (2) Å]. As such, the six-membered ring is better described as a cyclohexadiene system.

### S2. Experimental

The compound was synthesized according to a reported procedure (Terrian *et al.*, 1992; Wolthius, 1979). Crystals were grown with THF as solvent.

### S3. Refinement

The carbon-bound H-atoms were restrained to C—H 0.95±0.01 Å; their temperature factors were freely refined.

**Figure 1**

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 70% probability level, and hydrogen atoms are drawn as spheres of arbitrary radius.

**(I)***Crystal data*

$C_6H_4N_2O_2$

$M_r = 136.11$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 6.6751(2)\ \text{\AA}$

$b = 7.3256(2)\ \text{\AA}$

$c = 7.6842(2)\ \text{\AA}$

$\alpha = 100.710(2)^\circ$

$\beta = 114.265(2)^\circ$

$\gamma = 111.747(2)^\circ$

$V = 291.71(1)\ \text{\AA}^3$

$Z = 2$

$F(000) = 140$

$D_x = 1.550\ \text{Mg m}^{-3}$

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

Cell parameters from 1320 reflections

$\theta = 3.2\text{--}28.3^\circ$

$\mu = 0.12\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Irregular block, yellow-orange

$0.30 \times 0.25 \times 0.10\ \text{mm}$

*Data collection*

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

1952 measured reflections

1276 independent reflections

1110 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\text{max}} = 27.5^\circ$ ,  $\theta_{\text{min}} = 3.2^\circ$

$h = -7 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -9 \rightarrow 9$

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.035$  $wR(F^2) = 0.110$  $S = 1.03$ 

1276 reflections

108 parameters

4 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

All H-atom parameters refined

 $w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.0855P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL*, $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$ 

Extinction coefficient: 0.03 (1)

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.1183 (2)	0.2141 (2)	-0.0082 (1)	0.0274 (3)
O2	0.5178 (2)	0.2611 (2)	0.1146 (1)	0.0271 (3)
N1	0.3061 (2)	0.2329 (2)	0.1385 (2)	0.0211 (3)
N2	0.7112 (2)	0.2839 (2)	0.2955 (2)	0.0257 (3)
C1	0.6232 (2)	0.2701 (2)	0.4219 (2)	0.0188 (3)
C2	0.7496 (3)	0.2863 (2)	0.6308 (2)	0.0202 (3)
C3	0.6182 (3)	0.2669 (2)	0.7284 (2)	0.0207 (3)
C4	0.3643 (3)	0.2321 (2)	0.6312 (2)	0.0209 (3)
C5	0.2382 (3)	0.2174 (2)	0.4325 (2)	0.0199 (3)
C6	0.3750 (2)	0.2377 (2)	0.3296 (2)	0.0181 (3)
H2	0.919 (2)	0.310 (3)	0.693 (3)	0.030 (4)*
H3	0.701 (3)	0.278 (3)	0.867 (2)	0.037 (5)*
H4	0.281 (3)	0.221 (3)	0.706 (2)	0.027 (4)*
H5	0.072 (2)	0.197 (3)	0.369 (2)	0.031 (4)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0242 (5)	0.0333 (6)	0.0172 (5)	0.0139 (4)	0.0039 (4)	0.0130 (4)
O2	0.0270 (6)	0.0398 (6)	0.0190 (5)	0.0172 (5)	0.0137 (4)	0.0157 (4)
N1	0.0213 (6)	0.0237 (6)	0.0158 (5)	0.0105 (5)	0.0077 (5)	0.0098 (4)
N2	0.0237 (6)	0.0361 (7)	0.0202 (6)	0.0155 (5)	0.0120 (5)	0.0144 (5)
C1	0.0198 (6)	0.0199 (6)	0.0171 (6)	0.0096 (5)	0.0095 (5)	0.0090 (5)
C2	0.0187 (6)	0.0231 (6)	0.0175 (6)	0.0109 (5)	0.0072 (5)	0.0103 (5)
C3	0.0240 (7)	0.0215 (6)	0.0147 (6)	0.0109 (5)	0.0083 (5)	0.0093 (5)
C4	0.0246 (7)	0.0228 (6)	0.0192 (6)	0.0120 (5)	0.0137 (6)	0.0102 (5)
C5	0.0188 (6)	0.0213 (6)	0.0200 (6)	0.0104 (5)	0.0096 (5)	0.0096 (5)
C6	0.0200 (6)	0.0175 (6)	0.0136 (5)	0.0086 (5)	0.0066 (5)	0.0073 (4)

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

O1—N1	1.230 (1)	C3—C4	1.436 (2)
O2—N2	1.381 (1)	C4—C5	1.366 (2)

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O2—N1	1.443 (2)	C5—C6	1.420 (2)
N1—C6	1.336 (2)	C2—H2	0.956 (9)
N2—C1	1.327 (2)	C3—H3	0.948 (9)
C1—C6	1.409 (2)	C4—H4	0.946 (9)
C1—C2	1.430 (2)	C5—H5	0.947 (9)
C2—C3	1.357 (2)		
N2—O2—N1	109.4 (1)	N1—C6—C1	106.9 (1)
O1—N1—C6	136.0 (1)	N1—C6—C5	129.7 (1)
O1—N1—O2	117.7 (1)	C1—C6—C5	123.5 (1)
C6—N1—O2	106.3 (1)	C3—C2—H2	124 (1)
C1—N2—O2	105.0 (1)	C1—C2—H2	119 (1)
N2—C1—C6	112.5 (1)	C2—C3—H3	117 (1)
N2—C1—C2	128.0 (1)	C4—C3—H3	120 (1)
C6—C1—C2	119.5 (1)	C5—C4—H4	118 (1)
C3—C2—C1	116.8 (1)	C3—C4—H4	120 (1)
C2—C3—C4	122.9 (1)	C4—C5—H5	122 (1)
C5—C4—C3	121.9 (1)	C6—C5—H5	122 (1)
C4—C5—C6	115.4 (1)		
N2—O2—N1—O1	178.7 (1)	O1—N1—C6—C1	-178.3 (1)
N2—O2—N1—C6	-0.4 (1)	O2—N1—C6—C1	0.6 (1)
N1—O2—N2—C1	0.1 (1)	O1—N1—C6—C5	1.2 (2)
O2—N2—C1—C6	0.3 (2)	O2—N1—C6—C5	-179.9 (1)
O2—N2—C1—C2	-179.1 (1)	N2—C1—C6—N1	-0.5 (2)
N2—C1—C2—C3	179.9 (1)	C2—C1—C6—N1	178.9 (1)
C6—C1—C2—C3	0.6 (2)	N2—C1—C6—C5	179.9 (1)
C1—C2—C3—C4	-0.1 (2)	C2—C1—C6—C5	-0.7 (2)
C2—C3—C4—C5	-0.4 (2)	C4—C5—C6—N1	-179.2 (1)
C3—C4—C5—C6	0.3 (2)	C4—C5—C6—C1	0.2 (2)

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