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## Structure Reports

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## *N*-(4-Azobenzoyl)-5-norbornene-2,3-dicarboximide

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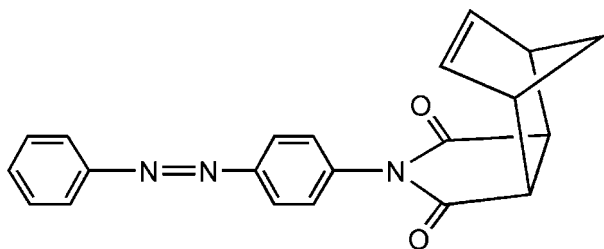
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Key indicators: single-crystal X-ray study;  $T = 292$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å; disorder in main residue;  $R$  factor = 0.059;  $wR$  factor = 0.139; data-to-parameter ratio = 14.9.

The title compound,  $\text{C}_{21}\text{H}_{17}\text{N}_3\text{O}_2$ , is a norbornene derivative containing an azo group which is disordered over two sites in an approximate 2:1 ratio. The two aromatic rings are almost coplanar [dihedral angle  $9.25$  ( $10^\circ$ )]. The crystal packing is stabilized by an intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bond.

### Related literature

For related literature, see: Tian *et al.* (2007); Zhao *et al.* (1999).



### Experimental

#### Crystal data

$\text{C}_{21}\text{H}_{17}\text{N}_3\text{O}_2$   
 $M_r = 343.38$

Monoclinic,  $C2/c$   
 $a = 25.190$  (2) Å

$b = 6.5076$  (5) Å  
 $c = 24.2792$  (19) Å  
 $\beta = 119.567$  ( $1^\circ$ )  
 $V = 3461.7$  (5) Å<sup>3</sup>  
 $Z = 8$

Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 292$  (2) K  
 $0.20 \times 0.20 \times 0.10$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
Absorption correction: none  
13859 measured reflections

3778 independent reflections  
2363 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.041$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$   
 $wR(F^2) = 0.139$   
 $S = 1.06$   
3778 reflections  
254 parameters

2 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                          | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C9}-\text{H9}\cdots\text{O2}^i$ | 0.93  | 2.53        | 3.155 (3)   | 125           |

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

Data collection: *SMART* (Bruker 2001); cell refinement: *SAINTE* (Bruker 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 2001); software used to prepare material for publication: *SHELXTL*.

The authors thank Dr Meng Xianggao (College of Chemistry, Central China Normal University) for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2500).

### References

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Zhao, W., Wu, C. X. & Iwamoto, M. (1999). *Chem. Phys. Lett.* **312**, 572–577.

**supplementary materials**

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## *N*-(4-Azobenzoyl)-5-norbornene-2,3-dicarboximide

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### Comment

The family of azobenzenes has been investigated intensively due to their remarkable photochemically induced *trans* to *cis* isomerism. Azo polymers are of interest in various fields (Zhao *et al.*, 1999). As a part of our investigation of preparing azo polymers, we report the crystal structure of the title compound (Fig. 1).

Bond lengths and angles of the norbornene moiety are similar to those in other norbornene derivatives (Tian *et al.*, 2007). By means of C—H···O hydrogen bonds, the molecules are linked into a ribbon. (Tab. 1, Fig. 2). The two aromatic rings are almost coplanar [dihedral angle 9.25 (10)°]. The dihedral angle between the rings C13—C14—C15—C16—N13 and C7—C8—C9—C10—C11—C12 is 64.32 (7)° (Fig. 1).

### Experimental

The title compound was prepared from norbornene dianhydride and 4-azobenzene according to the literature method (Tian *et al.*, 2007). Single crystals were obtained by slow evaporation of an ethanol solution.

### Refinement

All hydrogen atoms were located in a difference Fourier map, but they were constrained to ride on their parent atoms with C—H<sub>aryl</sub>, C—H<sub>methylene</sub> and C—H<sub>methine-H</sub> distances of 0.93, 0.97 and 0.98 Å, respectively.  $U_{\text{iso}}(\text{H})$  was set to  $1.2U_{\text{eq}}(\text{C})$ . The two N atoms of the azo moiety are disordered over two sites with occupancies of 0.681 (12) and 0.319 (12). The C—N bond distances of the disordered N atoms were restrained to be equal within 0.01 Å.

### Figures

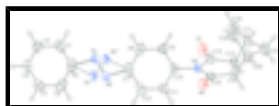


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

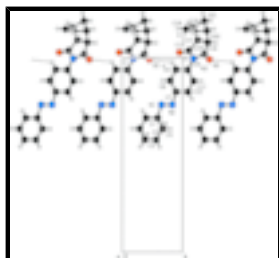


Fig. 2. Crystal packing of the title compound.

## *N*-(4-Azobenzoyl)-5-norbornene-2,3-dicarboximide

### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{21}H_{17}N_3O_2$           | $F_{000} = 1440$                          |
| $M_r = 343.38$                 | $D_x = 1.318 \text{ Mg m}^{-3}$           |
| Monoclinic, $C2/c$             | Mo $K\alpha$ radiation                    |
| Hall symbol: $-C 2yc$          | $\lambda = 0.71073 \text{ \AA}$           |
| $a = 25.190 (2) \text{ \AA}$   | Cell parameters from 1899 reflections     |
| $b = 6.5076 (5) \text{ \AA}$   | $\theta = 3.2\text{--}20.8^\circ$         |
| $c = 24.2792 (19) \text{ \AA}$ | $\mu = 0.09 \text{ mm}^{-1}$              |
| $\beta = 119.567 (1)^\circ$    | $T = 292 (2) \text{ K}$                   |
| $V = 3461.7 (5) \text{ \AA}^3$ | Block, red                                |
| $Z = 8$                        | $0.20 \times 0.20 \times 0.10 \text{ mm}$ |

### Data collection

|  |  |
|--|--|
| CCD area detector diffractometer         | 2363 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{\text{int}} = 0.041$               |
| Monochromator: graphite                  | $\theta_{\text{max}} = 27.0^\circ$     |
| $T = 292(2) \text{ K}$                   | $\theta_{\text{min}} = 1.9^\circ$      |
| $\varphi$ and $\omega$ scans             | $h = -32 \rightarrow 32$               |
| Absorption correction: none              | $k = -8 \rightarrow 7$                 |
| 13859 measured reflections               | $l = -31 \rightarrow 29$               |
| 3778 independent reflections             |  |

### Refinement

|  |  |
|--|--|
| Refinement on $F^2$  | Secondary atom site location: difference Fourier map     |
| Least-squares matrix: full                                     | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.059$                                | H-atom parameters constrained                            |
| $wR(F^2) = 0.139$  | $w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 1.3571P]$        |
| $S = 1.06$   | where $P = (F_o^2 + 2F_c^2)/3$                           |
| 3778 reflections   | $(\Delta/\sigma)_{\text{max}} = 0.001$                   |
| 254 parameters   | $\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$      |
| 2 restraints   | $\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$     |
| 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  $F^2 > 2\sigma(F^2)$  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) |
|-----|---------------|------------|--------------|----------------------------------|-----------|
| C1  | 0.08004 (11)  | 0.3958 (5) | 0.69015 (11) | 0.0732 (7)                       |           |
| H1  | 0.1091        | 0.3373     | 0.7281       | 0.088*                           |           |
| C2  | 0.06036 (11)  | 0.2911 (4) | 0.63479 (12) | 0.0678 (7)                       |           |
| H2  | 0.0767        | 0.1626     | 0.6351       | 0.081*                           |           |
| C3  | 0.01680 (10)  | 0.3736 (4) | 0.57875 (10) | 0.0602 (6)                       |           |
| H3  | 0.0032        | 0.3005     | 0.5412       | 0.072*                           |           |
| C4  | -0.00677 (10) | 0.5639 (4) | 0.57788 (11) | 0.0618 (6)                       |           |
| H4  | -0.0363       | 0.6205     | 0.5398       | 0.074*                           |           |
| C5  | 0.01360 (11)  | 0.6716 (4) | 0.63394 (13) | 0.0679 (7)                       |           |
| H5  | -0.0022       | 0.8010     | 0.6337       | 0.081*                           |           |
| C6  | 0.05724 (11)  | 0.5864 (4) | 0.68998 (11) | 0.0688 (7)                       |           |
| C7  | 0.09946 (11)  | 0.8942 (5) | 0.82488 (11) | 0.0725 (8)                       |           |
| C8  | 0.13603 (12)  | 0.7963 (4) | 0.88181 (12) | 0.0699 (7)                       |           |
| H8  | 0.1504        | 0.6646     | 0.8821       | 0.084*                           |           |
| C9  | 0.15139 (10)  | 0.8937 (3) | 0.93864 (10) | 0.0558 (6)                       |           |
| H9  | 0.1767        | 0.8295     | 0.9771       | 0.067*                           |           |
| C10 | 0.12857 (8)   | 1.0860 (3) | 0.93703 (9)  | 0.0430 (5)                       |           |
| C11 | 0.09270 (9)   | 1.1846 (4) | 0.88030 (9)  | 0.0554 (6)                       |           |
| H11 | 0.0779        | 1.3159     | 0.8798       | 0.067*                           |           |
| C12 | 0.07893 (10)  | 1.0891 (5) | 0.82462 (10) | 0.0707 (7)                       |           |
| H12 | 0.0555        | 1.1570     | 0.7864       | 0.085*                           |           |
| C13 | 0.12212 (9)   | 1.1082 (3) | 1.03599 (9)  | 0.0504 (5)                       |           |
| C14 | 0.14222 (9)   | 1.2552 (3) | 1.09033 (9)  | 0.0522 (6)                       |           |
| H14 | 0.1070        | 1.3146     | 1.0914       | 0.063*                           |           |
| C15 | 0.17851 (9)   | 1.4214 (3) | 1.07873 (9)  | 0.0522 (5)                       |           |
| H15 | 0.1604        | 1.5573     | 1.0756       | 0.063*                           |           |
| C16 | 0.17573 (9)   | 1.3640 (3) | 1.01730 (9)  | 0.0470 (5)                       |           |
| C17 | 0.18936 (10)  | 1.1676 (4) | 1.15637 (9)  | 0.0629 (6)                       |           |
| H17 | 0.1725        | 1.0893     | 1.1787       | 0.075*                           |           |
| C18 | 0.23697 (11)  | 1.0607 (4) | 1.14686 (10) | 0.0648 (7)                       |           |
| H18 | 0.2435        | 0.9197     | 1.1484       | 0.078*                           |           |
| C19 | 0.26790 (10)  | 1.2020 (4) | 1.13589 (10) | 0.0639 (6)                       |           |
| H19 | 0.2998        | 1.1786     | 1.1278       | 0.077*                           |           |

## supplementary materials

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|      |              |             |              |             |            |
|------|--------------|-------------|--------------|-------------|------------|
| C20  | 0.24259 (10) | 1.4069 (4)  | 1.13884 (10) | 0.0607 (6)  |            |
| H20  | 0.2696       | 1.5250      | 1.1471       | 0.073*      |            |
| C21  | 0.22356 (11) | 1.3637 (4)  | 1.18850 (11) | 0.0750 (7)  |            |
| H21A | 0.2580       | 1.3396      | 1.2304       | 0.090*      |            |
| H21B | 0.1972       | 1.4693      | 1.1902       | 0.090*      |            |
| N1   | 0.08481 (19) | 0.6515 (8)  | 0.7579 (2)   | 0.0587 (14) | 0.681 (12) |
| N2   | 0.07375 (17) | 0.8321 (8)  | 0.75791 (19) | 0.0555 (14) | 0.681 (12) |
| N1'  | 0.0608 (4)   | 0.7694 (12) | 0.7278 (5)   | 0.053 (3)   | 0.319 (12) |
| N2'  | 0.0990 (4)   | 0.7111 (15) | 0.7852 (4)   | 0.054 (3)   | 0.319 (12) |
| N3   | 0.14098 (7)  | 1.1850 (2)  | 0.99484 (7)  | 0.0426 (4)  |            |
| O1   | 0.09449 (8)  | 0.9498 (3)  | 1.02764 (7)  | 0.0773 (5)  |            |
| O2   | 0.19937 (8)  | 1.4485 (2)  | 0.99100 (8)  | 0.0686 (5)  |            |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1  | 0.0621 (14) | 0.108 (2)   | 0.0493 (14) | 0.0060 (14)  | 0.0272 (11) | -0.0015 (14) |
| C2  | 0.0740 (16) | 0.0677 (16) | 0.0706 (17) | 0.0052 (13)  | 0.0426 (14) | -0.0057 (13) |
| C3  | 0.0613 (13) | 0.0777 (17) | 0.0491 (13) | -0.0168 (12) | 0.0331 (11) | -0.0194 (12) |
| C4  | 0.0522 (13) | 0.0806 (17) | 0.0563 (14) | -0.0011 (12) | 0.0296 (11) | 0.0076 (13)  |
| C5  | 0.0664 (15) | 0.0599 (15) | 0.101 (2)   | -0.0058 (12) | 0.0591 (15) | -0.0159 (14) |
| C6  | 0.0588 (14) | 0.099 (2)   | 0.0625 (15) | -0.0196 (14) | 0.0404 (13) | -0.0319 (15) |
| C7  | 0.0657 (15) | 0.113 (2)   | 0.0516 (15) | -0.0391 (15) | 0.0388 (13) | -0.0333 (15) |
| C8  | 0.0912 (18) | 0.0579 (15) | 0.0824 (18) | -0.0183 (13) | 0.0594 (16) | -0.0232 (14) |
| C9  | 0.0693 (14) | 0.0482 (13) | 0.0553 (13) | -0.0041 (11) | 0.0349 (11) | -0.0018 (11) |
| C10 | 0.0445 (10) | 0.0465 (12) | 0.0406 (11) | -0.0080 (9)  | 0.0229 (9)  | -0.0039 (9)  |
| C11 | 0.0469 (11) | 0.0739 (15) | 0.0454 (12) | 0.0032 (10)  | 0.0227 (10) | 0.0055 (11)  |
| C12 | 0.0510 (13) | 0.119 (2)   | 0.0402 (12) | -0.0088 (14) | 0.0210 (10) | -0.0045 (14) |
| C13 | 0.0465 (11) | 0.0610 (14) | 0.0441 (11) | -0.0087 (10) | 0.0226 (9)  | -0.0034 (10) |
| C14 | 0.0428 (11) | 0.0709 (15) | 0.0468 (12) | 0.0027 (10)  | 0.0251 (9)  | -0.0102 (10) |
| C15 | 0.0613 (13) | 0.0399 (12) | 0.0546 (12) | 0.0064 (10)  | 0.0279 (10) | -0.0065 (10) |
| C16 | 0.0519 (12) | 0.0352 (11) | 0.0517 (12) | 0.0022 (9)   | 0.0238 (10) | 0.0038 (9)   |
| C17 | 0.0655 (14) | 0.0846 (17) | 0.0416 (12) | -0.0100 (13) | 0.0286 (11) | -0.0020 (12) |
| C18 | 0.0693 (15) | 0.0596 (15) | 0.0498 (13) | 0.0124 (12)  | 0.0174 (11) | 0.0061 (11)  |
| C19 | 0.0440 (12) | 0.0839 (18) | 0.0554 (13) | 0.0080 (12)  | 0.0181 (10) | -0.0037 (13) |
| C20 | 0.0565 (13) | 0.0640 (15) | 0.0551 (13) | -0.0147 (11) | 0.0226 (11) | -0.0184 (11) |
| C21 | 0.0697 (15) | 0.099 (2)   | 0.0496 (13) | -0.0036 (14) | 0.0244 (12) | -0.0237 (14) |
| N1  | 0.061 (3)   | 0.067 (3)   | 0.047 (3)   | 0.010 (2)    | 0.025 (2)   | 0.000 (2)    |
| N2  | 0.057 (2)   | 0.062 (3)   | 0.046 (3)   | -0.0013 (17) | 0.025 (2)   | 0.0017 (19)  |
| N1' | 0.052 (4)   | 0.067 (6)   | 0.030 (5)   | 0.005 (4)    | 0.014 (4)   | -0.007 (4)   |
| N2' | 0.056 (5)   | 0.068 (7)   | 0.024 (5)   | 0.003 (4)    | 0.010 (4)   | -0.009 (4)   |
| N3  | 0.0480 (9)  | 0.0428 (10) | 0.0373 (8)  | -0.0031 (7)  | 0.0213 (7)  | -0.0001 (7)  |
| O1  | 0.0894 (12) | 0.0902 (13) | 0.0616 (10) | -0.0448 (10) | 0.0443 (9)  | -0.0165 (9)  |
| O2  | 0.0898 (11) | 0.0520 (9)  | 0.0711 (10) | -0.0171 (8)  | 0.0450 (9)  | 0.0034 (8)   |

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

|       |           |         |           |
|-------|-----------|---------|-----------|
| C1—C2 | 1.363 (3) | C12—H12 | 0.9300    |
| C1—C6 | 1.366 (4) | C13—O1  | 1.204 (2) |

|            |           |             |             |
|------------|-----------|-------------|-------------|
| C1—H1      | 0.9300    | C13—N3      | 1.393 (2)   |
| C2—C3      | 1.368 (3) | C13—C14     | 1.501 (3)   |
| C2—H2      | 0.9300    | C14—C15     | 1.529 (3)   |
| C3—C4      | 1.369 (3) | C14—C17     | 1.558 (3)   |
| C3—H3      | 0.9300    | C14—H14     | 0.9800      |
| C4—C5      | 1.385 (3) | C15—C16     | 1.505 (3)   |
| C4—H4      | 0.9300    | C15—C20     | 1.555 (3)   |
| C5—C6      | 1.375 (3) | C15—H15     | 0.9800      |
| C5—H5      | 0.9300    | C16—O2      | 1.200 (2)   |
| C6—N1'     | 1.480 (7) | C16—N3      | 1.397 (2)   |
| C6—N1      | 1.500 (5) | C17—C18     | 1.499 (3)   |
| C7—C12     | 1.369 (4) | C17—C21     | 1.522 (3)   |
| C7—C8      | 1.381 (4) | C17—H17     | 0.9800      |
| C7—N2      | 1.479 (5) | C18—C19     | 1.314 (3)   |
| C7—N2'     | 1.529 (9) | C18—H18     | 0.9300      |
| C8—C9      | 1.389 (3) | C19—C20     | 1.495 (3)   |
| C8—H8      | 0.9300    | C19—H19     | 0.9300      |
| C9—C10     | 1.370 (3) | C20—C21     | 1.528 (3)   |
| C9—H9      | 0.9300    | C20—H20     | 0.9800      |
| C10—C11    | 1.377 (3) | C21—H21A    | 0.9700      |
| C10—N3     | 1.432 (2) | C21—H21B    | 0.9700      |
| C11—C12    | 1.368 (3) | N1—N2       | 1.207 (9)   |
| C11—H11    | 0.9300    | N1'—N2'     | 1.298 (18)  |
| C2—C1—C6   | 120.2 (2) | C15—C14—C17 | 103.14 (16) |
| C2—C1—H1   | 119.9     | C13—C14—H14 | 110.9       |
| C6—C1—H1   | 119.9     | C15—C14—H14 | 110.9       |
| C1—C2—C3   | 120.5 (2) | C17—C14—H14 | 110.9       |
| C1—C2—H2   | 119.7     | C16—C15—C14 | 105.49 (16) |
| C3—C2—H2   | 119.7     | C16—C15—C20 | 115.72 (17) |
| C2—C3—C4   | 120.0 (2) | C14—C15—C20 | 102.83 (17) |
| C2—C3—H3   | 120.0     | C16—C15—H15 | 110.8       |
| C4—C3—H3   | 120.0     | C14—C15—H15 | 110.8       |
| C3—C4—C5   | 119.7 (2) | C20—C15—H15 | 110.8       |
| C3—C4—H4   | 120.2     | O2—C16—N3   | 123.68 (19) |
| C5—C4—H4   | 120.2     | O2—C16—C15  | 128.39 (19) |
| C6—C5—C4   | 119.7 (2) | N3—C16—C15  | 107.92 (17) |
| C6—C5—H5   | 120.1     | C18—C17—C21 | 100.03 (19) |
| C4—C5—H5   | 120.1     | C18—C17—C14 | 105.81 (17) |
| C1—C6—C5   | 119.9 (2) | C21—C17—C14 | 99.88 (18)  |
| C1—C6—N1'  | 146.4 (5) | C18—C17—H17 | 116.2       |
| C5—C6—N1'  | 93.7 (5)  | C21—C17—H17 | 116.2       |
| C1—C6—N1   | 106.7 (3) | C14—C17—H17 | 116.2       |
| C5—C6—N1   | 133.2 (3) | C19—C18—C17 | 107.8 (2)   |
| C12—C7—C8  | 119.7 (2) | C19—C18—H18 | 126.1       |
| C12—C7—N2  | 106.8 (3) | C17—C18—H18 | 126.1       |
| C8—C7—N2   | 133.5 (3) | C18—C19—C20 | 107.7 (2)   |
| C12—C7—N2' | 146.5 (5) | C18—C19—H19 | 126.1       |
| C8—C7—N2'  | 93.8 (5)  | C20—C19—H19 | 126.1       |
| C7—C8—C9   | 120.4 (2) | C19—C20—C21 | 100.4 (2)   |

## supplementary materials

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|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C7—C8—H8        | 119.8        | C19—C20—C15     | 106.20 (17)  |
| C9—C8—H8        | 119.8        | C21—C20—C15     | 99.63 (18)   |
| C10—C9—C8       | 118.8 (2)    | C19—C20—H20     | 116.1        |
| C10—C9—H9       | 120.6        | C21—C20—H20     | 116.1        |
| C8—C9—H9        | 120.6        | C15—C20—H20     | 116.1        |
| C9—C10—C11      | 120.9 (2)    | C17—C21—C20     | 93.94 (17)   |
| C9—C10—N3       | 119.84 (18)  | C17—C21—H21A    | 112.9        |
| C11—C10—N3      | 119.27 (18)  | C20—C21—H21A    | 112.9        |
| C12—C11—C10     | 119.8 (2)    | C17—C21—H21B    | 112.9        |
| C12—C11—H11     | 120.1        | C20—C21—H21B    | 112.9        |
| C10—C11—H11     | 120.1        | H21A—C21—H21B   | 110.4        |
| C11—C12—C7      | 120.4 (2)    | N2—N1—C6        | 107.1 (4)    |
| C11—C12—H12     | 119.8        | N1—N2—C7        | 106.9 (4)    |
| C7—C12—H12      | 119.8        | N2'—N1'—C6      | 102.5 (7)    |
| O1—C13—N3       | 124.03 (19)  | N1'—N2'—C7      | 102.7 (8)    |
| O1—C13—C14      | 127.59 (19)  | C13—N3—C16      | 112.89 (16)  |
| N3—C13—C14      | 108.38 (18)  | C13—N3—C10      | 123.70 (16)  |
| C13—C14—C15     | 105.19 (16)  | C16—N3—C10      | 123.35 (16)  |
| C13—C14—C17     | 115.26 (18)  |                 |              |
| C6—C1—C2—C3     | -1.3 (4)     | C14—C17—C18—C19 | 69.5 (2)     |
| C1—C2—C3—C4     | 0.9 (4)      | C17—C18—C19—C20 | 0.9 (2)      |
| C2—C3—C4—C5     | -0.2 (3)     | C18—C19—C20—C21 | 32.3 (2)     |
| C3—C4—C5—C6     | -0.1 (3)     | C18—C19—C20—C15 | -71.1 (2)    |
| C2—C1—C6—C5     | 1.0 (4)      | C16—C15—C20—C19 | -48.1 (3)    |
| C2—C1—C6—N1'    | -174.7 (6)   | C14—C15—C20—C19 | 66.3 (2)     |
| C2—C1—C6—N1     | 176.3 (3)    | C16—C15—C20—C21 | -152.03 (19) |
| C4—C5—C6—C1     | -0.3 (4)     | C14—C15—C20—C21 | -37.6 (2)    |
| C4—C5—C6—N1'    | 177.3 (3)    | C18—C17—C21—C20 | 49.8 (2)     |
| C4—C5—C6—N1     | -174.1 (3)   | C14—C17—C21—C20 | -58.39 (19)  |
| C12—C7—C8—C9    | 0.8 (3)      | C19—C20—C21—C17 | -49.44 (19)  |
| N2—C7—C8—C9     | -177.6 (3)   | C15—C20—C21—C17 | 59.2 (2)     |
| N2'—C7—C8—C9    | -178.1 (4)   | C1—C6—N1—N2     | 168.1 (3)    |
| C7—C8—C9—C10    | 1.4 (3)      | C5—C6—N1—N2     | -17.6 (5)    |
| C8—C9—C10—C11   | -2.2 (3)     | N1'—C6—N1—N2    | -4.2 (6)     |
| C8—C9—C10—N3    | 176.58 (18)  | C6—N1—N2—C7     | 178.4 (3)    |
| C9—C10—C11—C12  | 0.8 (3)      | C12—C7—N2—N1    | -170.9 (3)   |
| N3—C10—C11—C12  | -177.98 (18) | C8—C7—N2—N1     | 7.7 (5)      |
| C10—C11—C12—C7  | 1.5 (3)      | N2'—C7—N2—N1    | 8.4 (6)      |
| C8—C7—C12—C11   | -2.2 (3)     | C1—C6—N1'—N2'   | -7.1 (10)    |
| N2—C7—C12—C11   | 176.6 (2)    | C5—C6—N1'—N2'   | 176.6 (6)    |
| N2'—C7—C12—C11  | 175.8 (7)    | N1—C6—N1'—N2'   | 6.4 (6)      |
| O1—C13—C14—C15  | 176.6 (2)    | C6—N1'—N2'—C7   | -176.3 (5)   |
| N3—C13—C14—C15  | -3.3 (2)     | C12—C7—N2'—N1'  | -4.1 (11)    |
| O1—C13—C14—C17  | 63.8 (3)     | C8—C7—N2'—N1'   | 174.2 (6)    |
| N3—C13—C14—C17  | -116.16 (19) | N2—C7—N2'—N1'   | -5.3 (5)     |
| C13—C14—C15—C16 | 1.6 (2)      | O1—C13—N3—C16   | -175.9 (2)   |
| C17—C14—C15—C16 | 122.73 (17)  | C14—C13—N3—C16  | 4.0 (2)      |
| C13—C14—C15—C20 | -120.09 (17) | O1—C13—N3—C10   | 1.3 (3)      |
| C17—C14—C15—C20 | 1.1 (2)      | C14—C13—N3—C10  | -178.78 (16) |



|                 |             |                |             |
|-----------------|-------------|----------------|-------------|
| C14—C15—C16—O2  | -177.8 (2)  | O2—C16—N3—C13  | 175.6 (2)   |
| C20—C15—C16—O2  | -64.9 (3)   | C15—C16—N3—C13 | -2.9 (2)    |
| C14—C15—C16—N3  | 0.6 (2)     | O2—C16—N3—C10  | -1.7 (3)    |
| C20—C15—C16—N3  | 113.55 (19) | C15—C16—N3—C10 | 179.84 (16) |
| C13—C14—C17—C18 | 46.5 (2)    | C9—C10—N3—C13  | -62.1 (3)   |
| C15—C14—C17—C18 | -67.5 (2)   | C11—C10—N3—C13 | 116.6 (2)   |
| C13—C14—C17—C21 | 150.03 (18) | C9—C10—N3—C16  | 114.8 (2)   |
| C15—C14—C17—C21 | 36.0 (2)    | C11—C10—N3—C16 | -66.4 (2)   |
| C21—C17—C18—C19 | -33.9 (2)   |                |             |

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

| <i>D</i> —H $\cdots$ <i>A</i>  | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C9—H9 $\cdots$ O2 <sup>i</sup> | 0.93        | 2.53                | 3.155 (3)                  | 125                           |

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

Fig. 1

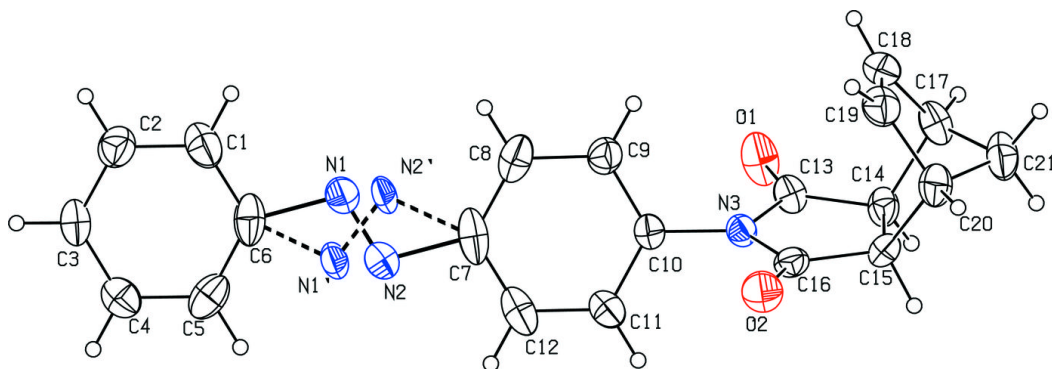


Fig. 2

