Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# (3a*R*,7a*R*)-Bis(4-nitrophenyl) 2-oxoperhydrobenzo[*d*]imidazole-1,3-dicarboxylate

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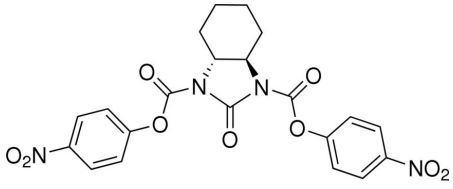
Received 14 July 2007; accepted 10 August 2007

Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.042; $wR$ factor = 0.103; data-to-parameter ratio = 8.9.

The title compound, $C_{21}H_{18}N_4O_9$, is a chiral cyclic urea derivative with an approximate local $C_2$ axis. The crystal structure is mainly stablized by weak interactions since the molecule does not possess hydrogen-bonding donors. The molecules stack along the *a* axis.

## Related literature

For geometry, see: Allen *et al.* (1987). For similar structures, see: Long (2006); Siegler & Long (2006).



## Experimental

### Crystal data

$C_{21}H_{18}N_4O_9$
$M_r$ = 470.39
Orthorhombic, $P2_12_12_1$
$a$ = 6.059 (1) Å
$b$ = 17.956 (3) Å
$c$ = 18.982 (3) Å
$V$ = 2065.2 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.12 mm$^{-1}$
$T$ = 90.0 (2) K
0.35 × 0.20 × 0.10 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.959, $T_{max}$ = 0.988
9218 measured reflections
2724 independent reflections
2209 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.103
$S$ = 1.04
2724 reflections
307 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

SL is grateful to Dr Sean Parkin for providing support and laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2150).

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 doi:10.1107/S1600536807039785  

# supplementary materials

*Acta Cryst.* (2007). E**63**, o3835 [ **doi:10.1107/S1600536807039785** ]

# (3a*R*,7a*R*)-Bis(4-nitrophenyl) 2-oxoperhydrobenzo[*d*]imidazole-1,3-dicarboxylate

**Y.-H. Xu, M. Siegler and S. Long**

## Comment

The title compound, (I), a chiral *C*2 symmetric cyclic urea derivative, was obtained as a by-product during an attempt to synthesize a linker for the preparation of chiral oligoureas (Long, 2006). Here we report the structure of it.

The asymmetric unit of (I) (Fig. 1) contains one molecule and the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The chirality of the C atoms (C1 *R*, C6 *R*) was assigned based on the known chirality of the starting material, with the assumption that no chirality change took place during the reaction. The cyclohexane ring has a chair conformation and the cyclic C1/C6/N2/C7/N1 urea unit has a twisted envolope conformation with no four atoms in the same plane, similar to the structure of (4*R*,5*R*)-4,5-Diphenylimidazolidin-2-one (Siegler and Long, 2006). Since the molecule has no hydrogen bonding donors, the structure is stablized by weak interactions including π stacking. The molecules stack along the *a* axis, and the distance between the face-to-face π stacking aromatic rings is 6.059 Å. In addition, intramolecular O···O interaction exists as indicated by the distances (O3···O1 = 2.615 Å, and O1···O6 = 2.662 Å) (Fig. 2).

## Experimental

n-BuLi(8.6 ml, 21.5 mmol) was added dropwise to a round-bottom flask containing *tert*-butyl (1*R*,2*R*)-cyclohexane-1,2-diyldicarbamate (3.14 g, 10 mmol) in 20 ml THF cooled with dry ice. After the resulted solutuion was warmed to ambient temperature, 4-nitrophenyl [1,2-diphenyl-2-(2,2,2-trifluoroacetylamino)ethyl]- carbamate (4.03 g, 20 mmol) in 20 ml THF was added dropwise. The mixture was stirred at room temperature overnight. The solution was washed with 1 N NaOH, water and brine, and then dried with anhydrous $Na_2SO_4$. After removal of the solvent, the product was recovered as a colorless solid (4.3 g, 91%). Crystals of (I) were obtained by recrystallization from ethyl acetate as colorless blocks.

## Refinement

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained C—H distances of 0.95 Å ($C_{Ar}H$), 0.99 Å ($CH_2$), and 1.00 Å ($CH_1$). $U_{iso}$(H) values were set to 1.2$U_{eq}$ for all H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged prior to refinement.

## Figures

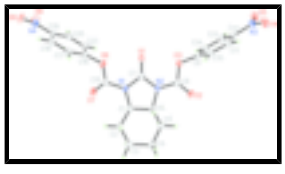
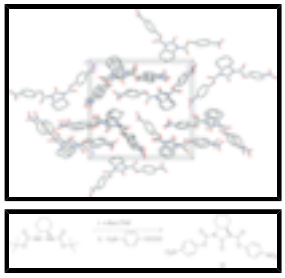


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).
Fig. 2. A packing diagram of (I) along *a* axis.



**(3aR,7aR)-Bis(4-nitrophenyl) 2-oxoperhydrobenzo[*d*]imidazole-1,3-dicarboxylate**

*Crystal data*

| | |
|---|---|
| $C_{21}H_{18}N_4O_9$ | $D_x = 1.513$ Mg m$^{-3}$ |
| $M_r = 470.39$ | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| Orthorhombic, $P2_12_12_1$ | Cell parameters from 4880 reflections |
| $a = 6.059$ (1) Å | $\theta = 1.0$–$27.5$º |
| $b = 17.956$ (3) Å | $\mu = 0.12$ mm$^{-1}$ |
| $c = 18.982$ (3) Å | $T = 90.0$ (2) K |
| $V = 2065.2$ (6) Å$^3$ | Block, colourless |
| $Z = 4$ | 0.35 × 0.20 × 0.10 mm |
| $F_{000} = 976$ | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 2724 independent reflections |
| Radiation source: fine-focus sealed tube | 2209 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.049$ |
| Detector resolution: 18 pixels mm$^{-1}$ | $\theta_{max} = 27.5$º |
| $T = 90.0(2)$ K | $\theta_{min} = 1.6$º |
| ω scans at fixed $\chi = 55$° | $h = -7 \rightarrow 7$ |
| Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997) | $k = -23 \rightarrow 23$ |
| $T_{min} = 0.959$, $T_{max} = 0.988$ | $l = -24 \rightarrow 23$ |
| 9218 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.067P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2) = 0.103$ | $\Delta\rho_{max} = 0.21$ e Å$^{-3}$ |
| $S = 1.04$ | $\Delta\rho_{min} = -0.31$ e Å$^{-3}$ |
| 2724 reflections | Extinction correction: none |

307 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

*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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.7327 (4) | 0.07569 (12) | 0.30668 (13) | 0.0216 (5) |
| H1 | 0.6373 | 0.0305 | 0.3025 | 0.026* |
| C2 | 0.9611 (4) | 0.05185 (14) | 0.32890 (13) | 0.0255 (6) |
| H2A | 0.9565 | 0.0284 | 0.3761 | 0.031* |
| H2B | 1.0611 | 0.0954 | 0.3309 | 0.031* |
| C3 | 1.0425 (5) | −0.00426 (14) | 0.27372 (13) | 0.0300 (6) |
| H3A | 0.9539 | −0.0504 | 0.2776 | 0.036* |
| H3B | 1.1980 | −0.0172 | 0.2841 | 0.036* |
| C4 | 1.0270 (5) | 0.02536 (14) | 0.19768 (13) | 0.0292 (6) |
| H4A | 1.1389 | 0.0649 | 0.1911 | 0.035* |
| H4B | 1.0627 | −0.0156 | 0.1647 | 0.035* |
| C5 | 0.7995 (5) | 0.05674 (13) | 0.17903 (13) | 0.0254 (6) |
| H5A | 0.8026 | 0.0802 | 0.1318 | 0.030* |
| H5B | 0.6877 | 0.0165 | 0.1788 | 0.030* |
| C6 | 0.7444 (4) | 0.11400 (13) | 0.23481 (12) | 0.0200 (5) |
| H6 | 0.8658 | 0.1517 | 0.2362 | 0.024* |
| C7 | 0.4719 (4) | 0.17341 (12) | 0.30407 (12) | 0.0204 (5) |
| C8 | 0.4582 (4) | 0.19100 (12) | 0.17450 (12) | 0.0198 (5) |
| C9 | 0.1934 (4) | 0.27412 (14) | 0.12998 (12) | 0.0230 (6) |
| C10 | 0.3021 (5) | 0.33867 (14) | 0.10993 (13) | 0.0262 (6) |
| H10 | 0.4412 | 0.3513 | 0.1297 | 0.031* |
| C11 | 0.2022 (5) | 0.38445 (13) | 0.06004 (13) | 0.0260 (6) |
| H11 | 0.2729 | 0.4286 | 0.0441 | 0.031* |
| C12 | −0.0020 (5) | 0.36406 (13) | 0.03427 (12) | 0.0225 (6) |
| C13 | −0.1070 (4) | 0.29878 (14) | 0.05265 (13) | 0.0246 (6) |
| H13 | −0.2452 | 0.2856 | 0.0326 | 0.030* |
| C14 | −0.0045 (4) | 0.25308 (13) | 0.10121 (13) | 0.0244 (6) |

| | | | | |
|---|---|---|---|---|
| H14 | −0.0709 | 0.2073 | 0.1146 | 0.029* |
| C15 | 0.5755 (4) | 0.12054 (13) | 0.41966 (12) | 0.0235 (6) |
| C16 | 0.3959 (5) | 0.16923 (13) | 0.51801 (12) | 0.0220 (5) |
| C17 | 0.5086 (5) | 0.19901 (13) | 0.57421 (12) | 0.0232 (5) |
| H17 | 0.6464 | 0.2232 | 0.5677 | 0.028* |
| C18 | 0.4146 (4) | 0.19267 (12) | 0.64138 (13) | 0.0220 (6) |
| H18 | 0.4871 | 0.2124 | 0.6816 | 0.026* |
| C19 | 0.2146 (4) | 0.15713 (13) | 0.64735 (12) | 0.0205 (5) |
| C20 | 0.1017 (4) | 0.12713 (12) | 0.59117 (13) | 0.0220 (5) |
| H20 | −0.0354 | 0.1024 | 0.5978 | 0.026* |
| C21 | 0.1940 (5) | 0.13401 (13) | 0.52468 (13) | 0.0239 (6) |
| H21 | 0.1199 | 0.1149 | 0.4844 | 0.029* |
| N1 | 0.6143 (4) | 0.13238 (10) | 0.34801 (10) | 0.0202 (5) |
| N2 | 0.5302 (4) | 0.15399 (11) | 0.23445 (10) | 0.0207 (4) |
| N3 | −0.1138 (4) | 0.41454 (12) | −0.01561 (11) | 0.0276 (5) |
| N4 | 0.1167 (4) | 0.14975 (11) | 0.71830 (11) | 0.0244 (5) |
| O1 | 0.3305 (3) | 0.21570 (10) | 0.32218 (9) | 0.0259 (4) |
| O2 | 0.5490 (3) | 0.18582 (9) | 0.11838 (8) | 0.0239 (4) |
| O3 | 0.2732 (3) | 0.23065 (9) | 0.18638 (9) | 0.0262 (4) |
| O4 | −0.0114 (4) | 0.46898 (9) | −0.03702 (10) | 0.0365 (5) |
| O5 | −0.3052 (4) | 0.40039 (11) | −0.03286 (10) | 0.0372 (5) |
| O6 | 0.4816 (3) | 0.18046 (8) | 0.44960 (8) | 0.0248 (4) |
| O7 | 0.6268 (4) | 0.06473 (10) | 0.44975 (10) | 0.0385 (5) |
| O8 | 0.2036 (3) | 0.18496 (9) | 0.76670 (9) | 0.0290 (4) |
| O9 | −0.0414 (3) | 0.10836 (10) | 0.72600 (9) | 0.0327 (5) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0264 (13) | 0.0189 (11) | 0.0196 (12) | 0.0002 (10) | 0.0028 (11) | 0.0005 (10) |
| C2 | 0.0247 (13) | 0.0286 (12) | 0.0232 (13) | 0.0028 (12) | 0.0020 (12) | 0.0036 (11) |
| C3 | 0.0289 (14) | 0.0309 (13) | 0.0302 (14) | 0.0079 (13) | 0.0015 (13) | 0.0012 (12) |
| C4 | 0.0328 (15) | 0.0281 (12) | 0.0267 (14) | 0.0069 (12) | 0.0098 (13) | −0.0013 (11) |
| C5 | 0.0312 (14) | 0.0237 (12) | 0.0213 (13) | 0.0017 (12) | 0.0022 (12) | −0.0008 (11) |
| C6 | 0.0225 (13) | 0.0208 (12) | 0.0167 (11) | 0.0003 (10) | 0.0015 (11) | 0.0028 (10) |
| C7 | 0.0218 (12) | 0.0212 (11) | 0.0183 (12) | 0.0010 (11) | 0.0024 (11) | −0.0004 (10) |
| C8 | 0.0220 (12) | 0.0202 (11) | 0.0173 (12) | −0.0008 (10) | −0.0036 (11) | 0.0013 (9) |
| C9 | 0.0272 (14) | 0.0286 (13) | 0.0132 (11) | 0.0076 (12) | 0.0014 (11) | 0.0022 (10) |
| C10 | 0.0274 (14) | 0.0300 (13) | 0.0211 (12) | 0.0010 (12) | −0.0020 (12) | −0.0019 (11) |
| C11 | 0.0338 (15) | 0.0237 (12) | 0.0204 (12) | −0.0005 (12) | 0.0016 (12) | −0.0032 (11) |
| C12 | 0.0315 (15) | 0.0227 (11) | 0.0132 (11) | 0.0066 (12) | 0.0018 (11) | 0.0006 (9) |
| C13 | 0.0225 (13) | 0.0324 (13) | 0.0189 (12) | 0.0001 (11) | −0.0017 (12) | 0.0012 (11) |
| C14 | 0.0259 (13) | 0.0265 (12) | 0.0207 (12) | −0.0002 (11) | 0.0053 (12) | 0.0022 (10) |
| C15 | 0.0292 (14) | 0.0240 (12) | 0.0174 (11) | 0.0015 (11) | 0.0019 (11) | 0.0031 (10) |
| C16 | 0.0313 (14) | 0.0209 (11) | 0.0138 (11) | 0.0038 (11) | 0.0025 (11) | 0.0000 (10) |
| C17 | 0.0267 (14) | 0.0223 (11) | 0.0207 (12) | −0.0006 (11) | 0.0015 (11) | 0.0012 (10) |
| C18 | 0.0281 (14) | 0.0221 (12) | 0.0158 (11) | −0.0017 (11) | −0.0015 (11) | 0.0010 (10) |
| C19 | 0.0263 (14) | 0.0211 (11) | 0.0141 (11) | 0.0023 (11) | 0.0010 (11) | 0.0000 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C20 | 0.0211 (12) | 0.0220 (11) | 0.0230 (12) | −0.0008 (11) | −0.0004 (11) | −0.0001 (10) |
| C21 | 0.0328 (15) | 0.0203 (12) | 0.0186 (12) | 0.0010 (12) | −0.0050 (12) | −0.0017 (10) |
| N1 | 0.0258 (11) | 0.0205 (9) | 0.0144 (9) | 0.0053 (9) | 0.0028 (9) | 0.0011 (8) |
| N2 | 0.0239 (11) | 0.0239 (10) | 0.0143 (10) | 0.0040 (9) | 0.0008 (9) | 0.0020 (8) |
| N3 | 0.0346 (14) | 0.0267 (11) | 0.0214 (11) | 0.0053 (11) | −0.0009 (11) | −0.0009 (9) |
| N4 | 0.0297 (12) | 0.0228 (10) | 0.0206 (11) | 0.0008 (10) | 0.0022 (10) | 0.0004 (9) |
| O1 | 0.0294 (10) | 0.0306 (9) | 0.0175 (9) | 0.0074 (9) | 0.0019 (8) | 0.0022 (8) |
| O2 | 0.0292 (10) | 0.0268 (9) | 0.0156 (8) | 0.0015 (8) | 0.0025 (8) | −0.0010 (7) |
| O3 | 0.0251 (9) | 0.0360 (9) | 0.0174 (8) | 0.0087 (8) | 0.0012 (8) | 0.0082 (8) |
| O4 | 0.0520 (14) | 0.0241 (9) | 0.0333 (11) | −0.0013 (10) | −0.0014 (11) | 0.0062 (8) |
| O5 | 0.0337 (12) | 0.0447 (12) | 0.0332 (11) | 0.0078 (10) | −0.0082 (10) | 0.0078 (9) |
| O6 | 0.0363 (11) | 0.0229 (8) | 0.0150 (8) | 0.0035 (8) | 0.0055 (8) | 0.0013 (7) |
| O7 | 0.0573 (14) | 0.0358 (10) | 0.0223 (9) | 0.0176 (10) | 0.0088 (10) | 0.0091 (9) |
| O8 | 0.0342 (10) | 0.0347 (10) | 0.0180 (9) | −0.0028 (9) | 0.0027 (9) | −0.0059 (8) |
| O9 | 0.0354 (11) | 0.0338 (10) | 0.0290 (10) | −0.0123 (9) | 0.0093 (10) | −0.0017 (8) |

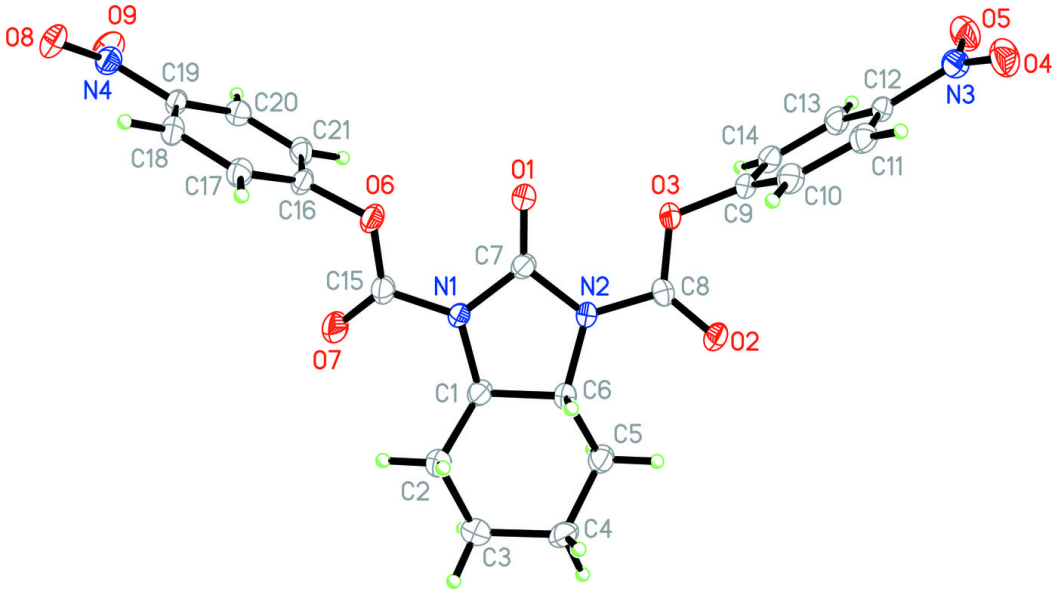
*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.472 (3) | C10—H10 | 0.9500 |
| C1—C2 | 1.509 (4) | C11—C12 | 1.380 (4) |
| C1—C6 | 1.530 (3) | C11—H11 | 0.9500 |
| C1—H1 | 1.0000 | C12—C13 | 1.378 (3) |
| C2—C3 | 1.535 (3) | C12—N3 | 1.476 (3) |
| C2—H2A | 0.9900 | C13—C14 | 1.381 (3) |
| C2—H2B | 0.9900 | C13—H13 | 0.9500 |
| C3—C4 | 1.541 (3) | C14—H14 | 0.9500 |
| C3—H3A | 0.9900 | C15—O7 | 1.194 (3) |
| C3—H3B | 0.9900 | C15—O6 | 1.343 (3) |
| C4—C5 | 1.531 (4) | C15—N1 | 1.397 (3) |
| C4—H4A | 0.9900 | C16—C17 | 1.375 (4) |
| C4—H4B | 0.9900 | C16—C21 | 1.383 (4) |
| C5—C6 | 1.513 (3) | C16—O6 | 1.413 (3) |
| C5—H5A | 0.9900 | C17—C18 | 1.401 (3) |
| C5—H5B | 0.9900 | C17—H17 | 0.9500 |
| C6—N2 | 1.484 (3) | C18—C19 | 1.374 (4) |
| C6—H6 | 1.0000 | C18—H18 | 0.9500 |
| C7—O1 | 1.195 (3) | C19—C20 | 1.377 (3) |
| C7—N1 | 1.408 (3) | C19—N4 | 1.477 (3) |
| C7—N2 | 1.412 (3) | C20—C21 | 1.386 (3) |
| C8—O2 | 1.202 (3) | C20—H20 | 0.9500 |
| C8—O3 | 1.347 (3) | C21—H21 | 0.9500 |
| C8—N2 | 1.388 (3) | N3—O4 | 1.227 (3) |
| C9—C14 | 1.371 (4) | N3—O5 | 1.232 (3) |
| C9—C10 | 1.386 (4) | N4—O9 | 1.222 (3) |
| C9—O3 | 1.410 (3) | N4—O8 | 1.233 (3) |
| C10—C11 | 1.393 (4) | | |
| N1—C1—C2 | 119.6 (2) | C12—C11—C10 | 118.3 (2) |
| N1—C1—C6 | 100.78 (17) | C12—C11—H11 | 120.9 |
| C2—C1—C6 | 109.5 (2) | C10—C11—H11 | 120.9 |

| | | | |
|---|---|---|---|
| N1—C1—H1 | 108.8 | C13—C12—C11 | 123.3 (2) |
| C2—C1—H1 | 108.8 | C13—C12—N3 | 118.2 (2) |
| C6—C1—H1 | 108.8 | C11—C12—N3 | 118.4 (2) |
| C1—C2—C3 | 106.9 (2) | C12—C13—C14 | 117.8 (2) |
| C1—C2—H2A | 110.3 | C12—C13—H13 | 121.1 |
| C3—C2—H2A | 110.3 | C14—C13—H13 | 121.1 |
| C1—C2—H2B | 110.3 | C9—C14—C13 | 119.7 (2) |
| C3—C2—H2B | 110.3 | C9—C14—H14 | 120.2 |
| H2A—C2—H2B | 108.6 | C13—C14—H14 | 120.2 |
| C2—C3—C4 | 113.1 (2) | O7—C15—O6 | 125.4 (2) |
| C2—C3—H3A | 109.0 | O7—C15—N1 | 123.3 (2) |
| C4—C3—H3A | 108.9 | O6—C15—N1 | 111.2 (2) |
| C2—C3—H3B | 108.9 | C17—C16—C21 | 123.1 (2) |
| C4—C3—H3B | 108.9 | C17—C16—O6 | 118.4 (2) |
| H3A—C3—H3B | 107.8 | C21—C16—O6 | 118.3 (2) |
| C5—C4—C3 | 113.5 (2) | C16—C17—C18 | 118.2 (2) |
| C5—C4—H4A | 108.9 | C16—C17—H17 | 120.9 |
| C3—C4—H4A | 108.9 | C18—C17—H17 | 120.9 |
| C5—C4—H4B | 108.9 | C19—C18—C17 | 118.1 (2) |
| C3—C4—H4B | 108.9 | C19—C18—H18 | 120.9 |
| H4A—C4—H4B | 107.7 | C17—C18—H18 | 120.9 |
| C6—C5—C4 | 106.7 (2) | C18—C19—C20 | 123.8 (2) |
| C6—C5—H5A | 110.4 | C18—C19—N4 | 118.1 (2) |
| C4—C5—H5A | 110.4 | C20—C19—N4 | 118.1 (2) |
| C6—C5—H5B | 110.4 | C19—C20—C21 | 118.0 (2) |
| C4—C5—H5B | 110.4 | C19—C20—H20 | 121.0 |
| H5A—C5—H5B | 108.6 | C21—C20—H20 | 121.0 |
| N2—C6—C5 | 121.2 (2) | C16—C21—C20 | 118.7 (2) |
| N2—C6—C1 | 100.43 (19) | C16—C21—H21 | 120.6 |
| C5—C6—C1 | 109.19 (18) | C20—C21—H21 | 120.6 |
| N2—C6—H6 | 108.4 | C15—N1—C7 | 123.6 (2) |
| C5—C6—H6 | 108.4 | C15—N1—C1 | 119.72 (19) |
| C1—C6—H6 | 108.4 | C7—N1—C1 | 110.17 (19) |
| O1—C7—N1 | 127.0 (2) | C8—N2—C7 | 124.80 (19) |
| O1—C7—N2 | 127.3 (2) | C8—N2—C6 | 120.69 (19) |
| N1—C7—N2 | 105.78 (19) | C7—N2—C6 | 109.51 (19) |
| O2—C8—O3 | 124.8 (2) | O4—N3—O5 | 123.5 (2) |
| O2—C8—N2 | 123.1 (2) | O4—N3—C12 | 118.0 (2) |
| O3—C8—N2 | 112.2 (2) | O5—N3—C12 | 118.4 (2) |
| C14—C9—C10 | 122.5 (2) | O9—N4—O8 | 123.9 (2) |
| C14—C9—O3 | 116.7 (2) | O9—N4—C19 | 118.6 (2) |
| C10—C9—O3 | 120.5 (2) | O8—N4—C19 | 117.5 (2) |
| C9—C10—C11 | 118.3 (2) | C8—O3—C9 | 116.79 (19) |
| C9—C10—H10 | 120.9 | C15—O6—C16 | 115.47 (17) |
| C11—C10—H10 | 120.9 | | |
| N1—C1—C2—C3 | −177.3 (2) | N2—C7—N1—C15 | −162.8 (2) |
| C6—C1—C2—C3 | −61.9 (3) | O1—C7—N1—C1 | 168.4 (2) |
| C1—C2—C3—C4 | 53.0 (3) | N2—C7—N1—C1 | −11.4 (3) |
| C2—C3—C4—C5 | −51.1 (3) | C2—C1—N1—C15 | −57.4 (3) |

| | | | |
|---|---|---|---|
| C3—C4—C5—C6 | 53.6 (3) | C6—C1—N1—C15 | −177.4 (2) |
| C4—C5—C6—N2 | −177.9 (2) | C2—C1—N1—C7 | 149.8 (2) |
| C4—C5—C6—C1 | −62.1 (3) | C6—C1—N1—C7 | 29.9 (3) |
| N1—C1—C6—N2 | −34.9 (2) | O2—C8—N2—C7 | 161.2 (2) |
| C2—C1—C6—N2 | −161.84 (19) | O3—C8—N2—C7 | −20.9 (3) |
| N1—C1—C6—C5 | −163.4 (2) | O2—C8—N2—C6 | 8.9 (3) |
| C2—C1—C6—C5 | 69.7 (3) | O3—C8—N2—C6 | −173.17 (18) |
| C14—C9—C10—C11 | 1.8 (4) | O1—C7—N2—C8 | 12.1 (4) |
| O3—C9—C10—C11 | −172.1 (2) | N1—C7—N2—C8 | −168.1 (2) |
| C9—C10—C11—C12 | 1.4 (4) | O1—C7—N2—C6 | 167.0 (2) |
| C10—C11—C12—C13 | −3.5 (4) | N1—C7—N2—C6 | −13.2 (2) |
| C10—C11—C12—N3 | 176.6 (2) | C5—C6—N2—C8 | −52.9 (3) |
| C11—C12—C13—C14 | 2.3 (4) | C1—C6—N2—C8 | −173.1 (2) |
| N3—C12—C13—C14 | −177.8 (2) | C5—C6—N2—C7 | 151.0 (2) |
| C10—C9—C14—C13 | −3.0 (4) | C1—C6—N2—C7 | 30.8 (2) |
| O3—C9—C14—C13 | 171.1 (2) | C13—C12—N3—O4 | −173.2 (2) |
| C12—C13—C14—C9 | 1.0 (4) | C11—C12—N3—O4 | 6.7 (3) |
| C21—C16—C17—C18 | 0.6 (4) | C13—C12—N3—O5 | 7.6 (3) |
| O6—C16—C17—C18 | 175.2 (2) | C11—C12—N3—O5 | −172.5 (2) |
| C16—C17—C18—C19 | −0.1 (3) | C18—C19—N4—O9 | −168.4 (2) |
| C17—C18—C19—C20 | 0.3 (4) | C20—C19—N4—O9 | 10.7 (3) |
| C17—C18—C19—N4 | 179.4 (2) | C18—C19—N4—O8 | 10.1 (3) |
| C18—C19—C20—C21 | −0.8 (4) | C20—C19—N4—O8 | −170.7 (2) |
| N4—C19—C20—C21 | −179.9 (2) | O2—C8—O3—C9 | −5.8 (3) |
| C17—C16—C21—C20 | −1.1 (4) | N2—C8—O3—C9 | 176.2 (2) |
| O6—C16—C21—C20 | −175.8 (2) | C14—C9—O3—C8 | 113.1 (2) |
| C19—C20—C21—C16 | 1.2 (3) | C10—C9—O3—C8 | −72.7 (3) |
| O7—C15—N1—C7 | 146.1 (3) | O7—C15—O6—C16 | −14.2 (4) |
| O6—C15—N1—C7 | −36.0 (3) | N1—C15—O6—C16 | 167.9 (2) |
| O7—C15—N1—C1 | −2.8 (4) | C17—C16—O6—C15 | 106.5 (3) |
| O6—C15—N1—C1 | 175.1 (2) | C21—C16—O6—C15 | −78.6 (3) |
| O1—C7—N1—C15 | 16.9 (4) | | |

**Fig. 1**


O8
O9
N4
C19
C20
C21
C18
C17
C16
O6
O1
O3
C7
C15
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N2
C8
O7
O2
C1
C6
C5
C2
C4
C3
C14
C13
C12
O5
O4
N3
C11
C10
C9

**Fig. 2**

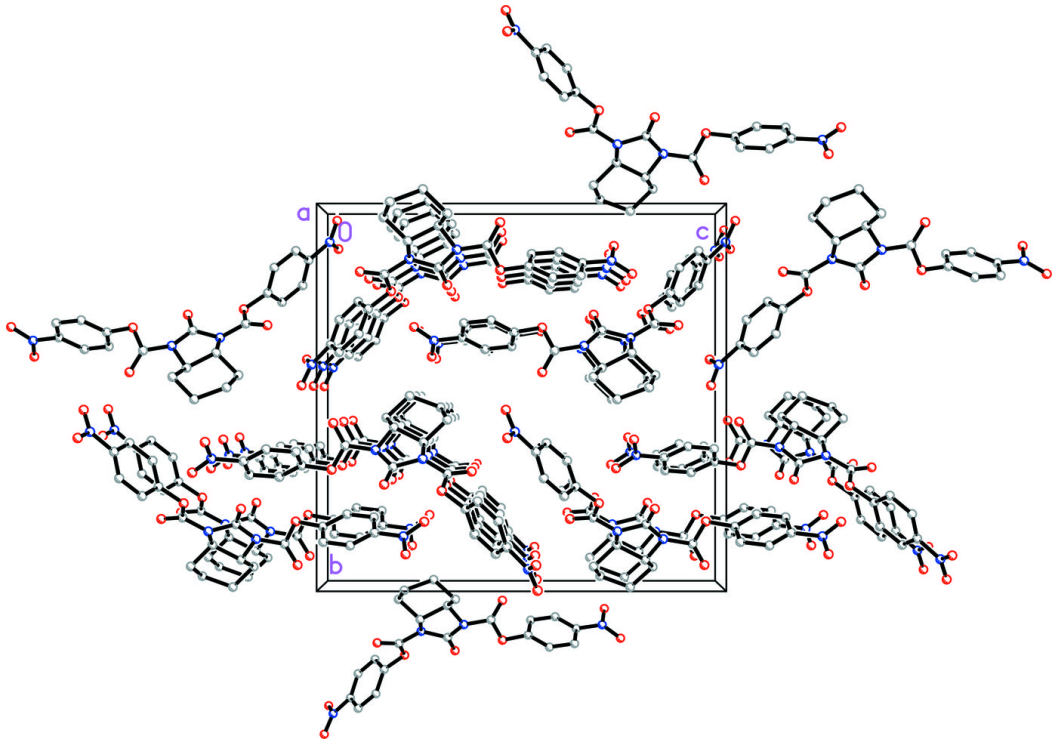
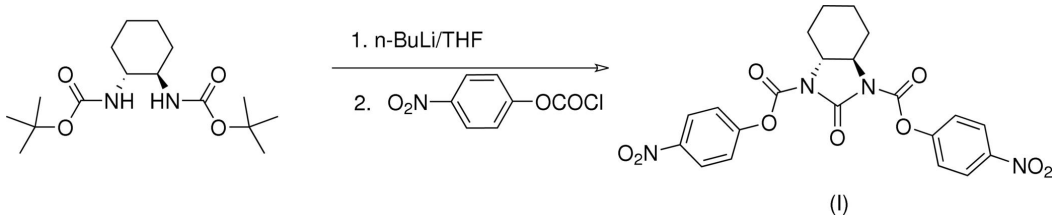


**Fig. 3**




(I)