

RESEARCH OUTPUTS / RÉSULTATS DE RECHERCHE

4-Nitro-N-phthalyl-L-tryptophan

Tilborg, A.; Boittiaux, I.; Norberg, B.; Lambert, Didier; Wouters, J.

Published in:

Acta crystallographica. Section E: Structure reports

DOI:

[10.1107/S1600536811029138](https://doi.org/10.1107/S1600536811029138)

Publication date:

2011

Document Version

Early version, also known as pre-print

[Link to publication](#)

Citation for published version (HARVARD):

Tilborg, A, Boittiaux, I, Norberg, B, Lambert, D & Wouters, J 2011, '4-Nitro-N-phthalyl-L-tryptophan', *Acta crystallographica. Section E: Structure reports*, vol. 67, no. 8, pp. o2116.
<https://doi.org/10.1107/S1600536811029138>

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal ?

Take down policy

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-Nitro-*N*-phthalyl-L-tryptophan

Anaëlle Tilborg,^a Irving Boittiaux,^b Bernadette Norberg,^a
Didier Lambert^b and Johan Wouters^{a*}

^aDepartment of Chemistry, University of Namur, 61, Rue de Bruxelles, B-5000 Namur, Belgium, and ^bLouvain Drug Research Institute (LDRI), UCL, 50, Avenue Mounier, B-1200 Woluwe-Saint-Lambert, Belgium
Correspondence e-mail: johan.wouters@fundp.ac.be

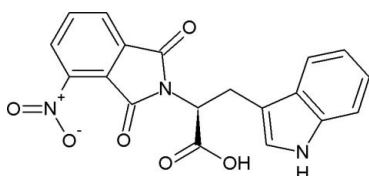
Received 1 July 2011; accepted 19 July 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.031; wR factor = 0.091; data-to-parameter ratio = 11.3.

The crystal structure of the title compound [systematic name: (2*R*)-3-(1*H*-indol-3-yl)-2-(4-nitro-1,3-dioxisoindolin-2-yl)propanoic acid], $\text{C}_{19}\text{H}_{13}\text{N}_3\text{O}_6$, an analogue of epigenetic modulator RG108, is constrained by strong hydrogen bonds between the indole N—H group and a carbonyl O atom of the phthalimide ring of a symmetry-related molecule, and between the protonated O atom of the carboxyl group and a carbonyl O atom of the phthalimide ring. π - π stacking interactions with centroid-centroid distances of 3.638 (1) and 3.610 (1) Å are also observed between indole and phthalimide rings.

Related literature

For crystallographic information and details about the RG108 analogue, see: Braun *et al.* (2010) and details of the biological evaluation, see: Brueckner *et al.* (2005).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{13}\text{N}_3\text{O}_6$
 $M_r = 379.33$
Monoclinic, $P2_1$
 $a = 7.0569$ (3) Å
 $b = 15.5302$ (8) Å
 $c = 7.6947$ (4) Å
 $\beta = 95.415$ (4)°

$V = 839.54$ (7) Å³
 $Z = 2$
Cu $K\alpha$ radiation
 $\mu = 0.97$ mm⁻¹
 $T = 293$ K
0.22 × 0.10 × 0.03 mm

Data collection

Oxford Diffraction Xcalibur Ruby
Gemini ultra diffractometer
Absorption correction: multi-scan
(*CrysAlis PRO*; Oxford
Diffraction, 2009)
 $T_{\min} = 0.815$, $T_{\max} = 0.972$
9007 measured reflections
2966 independent reflections
2735 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.091$
 $S = 1.06$
2966 reflections
262 parameters
1 restraint

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.13$ e Å⁻³
Absolute structure: Flack (1983),
1371 Friedel pairs
Flack parameter: -0.1 (2)

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.86 (4)	2.28 (4)	3.002 (3)	142 (4)
$\text{O4}-\text{H1}\cdots\text{O2}^{\text{ii}}$	1.00 (4)	1.78 (4)	2.716 (2)	154 (3)

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z$; (ii) $x, y, z - 1$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

This work was supported in part by the Fonds National de la Recherche Scientifique (FRS – FNRS, Belgium) by a Télévie grant (IB). AT acknowledges the FRIFA (Fonds pour la formation à la Recherche dans l'Industrie et dans l'Agriculture) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2109).

References

- Altomare, A., Casciarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Braun, J., Boittiaux, I., Tilborg, A., Lambert, D. & Wouters, J. (2010). *Acta Cryst. E* **66**, o3175–o3176.
Brueckner, B., Boy, R. G., Siedlecki, P., Munsch, T., Kliem, H. C., Zielenkiewicz, P., Suhai, S., Wiessler, M. & Lyko, F. (2005). *Cancer Res.* **65**, 6305–6311.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.
Oxford Diffraction (2009). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.

supplementary materials

Acta Cryst. (2011). E67, o2116 [doi:10.1107/S1600536811029138]

4-Nitro-*N*-phthalyl-L-tryptophan

A. Tilborg, I. Boittiaux, B. Norberg, D. Lambert and J. Wouters

Comment

4-Nitro-*N*-phthalyl-L-tryptophan is an analog of RG108, a DNA methyltransferase (DNMT) inhibitor discovered by virtual screening (Brueckner *et al.* (2005)). Introduction of a nitro functional group on the phthalimide moiety is expected to improve inhibition ability (unpublished results).

Isomer *S* (C9) of 4-nitro-*N*-phthalyl-tryptophan is obtained from L-tryptophan and nitrophthalic anhydride in DMF.

Two main types of H-bonding interactions are observed in the structure: one between N—H of the indole ring (N2) and O1 of the phthalimide ring, and the other between the protonated oxygen (O4) from the carboxylic moiety and O2 from the phthalimide ring (see Table 1).

In addition to H-bonds, crystal packing organization is further stabilized by π - π -stacking interactions involving symmetry-related molecules, in particular between the 6-membered coupled rings of nitrophthalimide and indole moiety (see Table 2). No interactions of this type are present in the packing of the dicyclohexylamine salt of RG108 (Braun *et al.* (2010)), because of the presence of the ammonium counter-cation and water molecules included in the crystalline network.

In contrast to the structure of the dicyclohexylamine salt of RG108 (Braun *et al.* (2010)), where the compound conformation is constrained by strong (charge-assisted) H-bonds with the dicyclohexylammonium ion and extra water molecules, the angle between the two fused rings is 14.23 (4)° in the present structure compared to 58.35 (4)° in the case of the RG108 salt. The torsion angle of the chain between the two aromatic moieties (N1—C9—C10—C11) is also distinct: -155.66 (17)° and -61.93 (17)° for the title and RG108 salt structures, respectively.

Experimental

Synthesis of the compound was accomplished by micro-wave heating of L-tryptophan (1 mmol, 204 mg) and 4-nitro phthalic anhydride (1 mmol, 193 mg) in 5 ml of DMF. The mixture was then poured in cold aqueous buffer solution (pH = 2) and extracted with ethyl acetate. After drying with Na₂SO₄, the organic phase is evaporated and the residue is purified by flash chromatography (dichloromethane and methanol: 9/1; yield = 60%, 230 mg).

Refinement

H1 and H2, bound to O4 and N2 respectively and involved in hydrogen bonds, were located from ΔF Fourier difference maps and their position refined freely. All other remaining H-atoms were placed at idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.93 – 0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

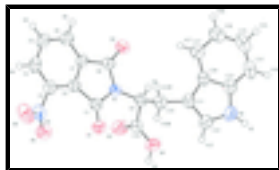


Fig. 1. ORTEP view and atom numbering of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

(2R)-3-(1H-indol-3-yl)-2-(4-nitro-1,3-dioxisoindolin-2-yl)propanoic acid

Crystal data

$C_{19}H_{13}N_3O_6$

$M_r = 379.33$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 7.0569$ (3) Å

$b = 15.5302$ (8) Å

$c = 7.6947$ (4) Å

$\beta = 95.415$ (4)°

$V = 839.54$ (7) Å³

$Z = 2$

$F(000) = 392$

$D_x = 1.501$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 4268 reflections

$\theta = 2.8$ – 67.9 °

$\mu = 0.97$ mm⁻¹

$T = 293$ K

Prism, yellow

$0.22 \times 0.10 \times 0.03$ mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini ultra diffractometer

Radiation source: fine-focus sealed tube graphite

Detector resolution: 10.3712 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (*Crys.Alis PRO*; Oxford Diffraction, 2009)

$T_{\min} = 0.815$, $T_{\max} = 0.972$

9007 measured reflections

2966 independent reflections

2735 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 68.0$ °, $\theta_{\min} = 5.7$ °

$h = -8$ → 8

$k = -18$ → 17

$l = -9$ → 8

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.091$

$S = 1.06$

2966 reflections

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.1477P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.15$ e Å⁻³

$\Delta\rho_{\min} = -0.13$ e Å⁻³

262 parameters
 1 restraint
 0 constraints
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map

Extinction correction: *SHELXL97* (Sheldrick, 2008),
 $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0080 (8)
 Absolute structure: Flack (1983), 1371 Friedel pairs
 Flack parameter: -0.1 (2)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C1	0.7937 (3)	0.58969 (13)	0.3293 (3)	0.0401 (4)
C2	0.8237 (3)	0.64858 (13)	0.4829 (3)	0.0385 (4)
C3	0.7949 (3)	0.73572 (14)	0.5072 (3)	0.0427 (5)
C4	0.8157 (3)	0.77052 (15)	0.6745 (3)	0.0481 (5)
H4	0.7881	0.8283	0.6913	0.058*
C5	0.8772 (3)	0.71950 (17)	0.8159 (3)	0.0533 (6)
H5	0.8943	0.7437	0.9269	0.064*
C6	0.9135 (3)	0.63265 (16)	0.7939 (3)	0.0497 (5)
H6	0.9581	0.5984	0.8881	0.060*
C7	0.8815 (3)	0.59866 (14)	0.6286 (3)	0.0410 (4)
C8	0.8976 (3)	0.50848 (14)	0.5709 (2)	0.0402 (4)
C9	0.8329 (3)	0.42945 (12)	0.2879 (3)	0.0391 (4)
H9	0.9070	0.3854	0.3557	0.047*
C10	0.6282 (3)	0.39570 (14)	0.2619 (3)	0.0479 (5)
H10A	0.5655	0.4077	0.3661	0.057*
H10B	0.5601	0.4265	0.1656	0.057*
C11	0.6167 (3)	0.30101 (14)	0.2249 (3)	0.0437 (5)
C12	0.5649 (3)	0.26268 (17)	0.0691 (3)	0.0532 (6)
H12	0.5355	0.2917	-0.0357	0.064*
C13	0.6095 (3)	0.15405 (15)	0.2600 (3)	0.0488 (5)
C14	0.6192 (3)	0.07499 (16)	0.3441 (4)	0.0607 (7)
H14	0.5920	0.0241	0.2830	0.073*
C15	0.6705 (3)	0.07453 (17)	0.5213 (4)	0.0650 (7)
H15	0.6779	0.0224	0.5810	0.078*
C16	0.7115 (3)	0.15063 (19)	0.6124 (4)	0.0590 (6)
H16	0.7468	0.1482	0.7318	0.071*

supplementary materials

C17	0.7012 (3)	0.22977 (16)	0.5303 (3)	0.0479 (5)
H17	0.7294	0.2801	0.5928	0.058*
C18	0.6472 (3)	0.23231 (14)	0.3505 (3)	0.0413 (4)
C19	0.9328 (3)	0.44380 (13)	0.1227 (3)	0.0433 (5)
N1	0.8488 (2)	0.50773 (11)	0.3922 (2)	0.0397 (4)
N2	0.5622 (3)	0.17470 (15)	0.0885 (3)	0.0591 (6)
N3	0.7496 (3)	0.79406 (12)	0.3599 (3)	0.0518 (5)
O1	0.7311 (2)	0.60263 (10)	0.18047 (19)	0.0526 (4)
O2	0.9409 (2)	0.44385 (10)	0.65541 (19)	0.0518 (4)
O3	1.0857 (3)	0.47711 (14)	0.1246 (2)	0.0659 (5)
O4	0.8377 (3)	0.41420 (14)	-0.0186 (2)	0.0709 (6)
O5	0.8221 (3)	0.77922 (12)	0.2254 (2)	0.0683 (5)
O6	0.6475 (3)	0.85593 (12)	0.3807 (3)	0.0768 (6)
H1	0.913 (5)	0.425 (2)	-0.120 (5)	0.093 (11)*
H2	0.525 (6)	0.138 (3)	0.008 (5)	0.095 (12)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0449 (10)	0.0354 (11)	0.0400 (11)	0.0023 (8)	0.0046 (8)	0.0025 (8)
C2	0.0382 (9)	0.0391 (11)	0.0391 (10)	-0.0004 (8)	0.0080 (8)	-0.0029 (8)
C3	0.0408 (10)	0.0409 (11)	0.0475 (11)	-0.0011 (8)	0.0092 (8)	-0.0035 (9)
C4	0.0440 (11)	0.0449 (12)	0.0576 (13)	-0.0067 (9)	0.0161 (9)	-0.0150 (10)
C5	0.0539 (12)	0.0625 (14)	0.0451 (12)	-0.0119 (11)	0.0133 (10)	-0.0138 (11)
C6	0.0527 (12)	0.0579 (15)	0.0397 (11)	-0.0068 (10)	0.0100 (9)	-0.0027 (10)
C7	0.0434 (10)	0.0442 (11)	0.0363 (10)	-0.0038 (8)	0.0083 (8)	-0.0055 (8)
C8	0.0414 (10)	0.0436 (11)	0.0362 (10)	0.0003 (8)	0.0078 (8)	0.0037 (9)
C9	0.0482 (10)	0.0314 (10)	0.0381 (10)	0.0020 (8)	0.0068 (8)	0.0011 (8)
C10	0.0477 (12)	0.0417 (12)	0.0549 (13)	0.0001 (9)	0.0085 (9)	0.0007 (10)
C11	0.0409 (10)	0.0400 (11)	0.0511 (12)	-0.0037 (8)	0.0087 (9)	-0.0006 (9)
C12	0.0575 (13)	0.0598 (15)	0.0422 (11)	-0.0117 (11)	0.0034 (10)	-0.0017 (10)
C13	0.0442 (11)	0.0413 (11)	0.0618 (14)	-0.0044 (9)	0.0104 (10)	-0.0042 (10)
C14	0.0490 (12)	0.0396 (12)	0.095 (2)	-0.0061 (9)	0.0157 (13)	-0.0023 (12)
C15	0.0497 (12)	0.0517 (15)	0.095 (2)	0.0012 (11)	0.0143 (13)	0.0218 (14)
C16	0.0486 (12)	0.0678 (16)	0.0610 (15)	0.0017 (11)	0.0068 (10)	0.0146 (12)
C17	0.0430 (10)	0.0505 (12)	0.0506 (12)	0.0002 (9)	0.0060 (9)	0.0034 (10)
C18	0.0371 (9)	0.0404 (11)	0.0474 (11)	-0.0035 (8)	0.0088 (8)	-0.0003 (9)
C19	0.0553 (12)	0.0359 (10)	0.0388 (10)	-0.0026 (9)	0.0053 (9)	-0.0012 (8)
N1	0.0527 (10)	0.0349 (9)	0.0315 (8)	0.0015 (7)	0.0053 (7)	-0.0001 (7)
N2	0.0653 (13)	0.0558 (14)	0.0568 (13)	-0.0161 (10)	0.0081 (10)	-0.0152 (10)
N3	0.0536 (10)	0.0382 (10)	0.0639 (13)	-0.0008 (8)	0.0069 (9)	-0.0001 (8)
O1	0.0717 (10)	0.0440 (8)	0.0401 (8)	0.0082 (7)	-0.0046 (7)	-0.0003 (7)
O2	0.0694 (10)	0.0456 (9)	0.0406 (8)	0.0043 (7)	0.0057 (7)	0.0065 (7)
O3	0.0628 (11)	0.0857 (13)	0.0511 (9)	-0.0194 (9)	0.0148 (8)	-0.0044 (8)
O4	0.0945 (13)	0.0828 (13)	0.0363 (8)	-0.0362 (11)	0.0113 (9)	-0.0052 (8)
O5	0.1017 (14)	0.0533 (10)	0.0511 (10)	0.0083 (9)	0.0135 (9)	0.0002 (8)
O6	0.0812 (12)	0.0447 (10)	0.1084 (16)	0.0170 (9)	0.0289 (11)	0.0126 (10)

Geometric parameters (Å, °)

C1—O1	1.205 (3)	C10—H10B	0.9700
C1—N1	1.403 (3)	C11—C12	1.358 (3)
C1—C2	1.493 (3)	C11—C18	1.442 (3)
C2—C3	1.384 (3)	C12—N2	1.375 (3)
C2—C7	1.392 (3)	C12—H12	0.9300
C3—C4	1.391 (3)	C13—N2	1.369 (3)
C3—N3	1.463 (3)	C13—C14	1.386 (4)
C4—C5	1.382 (4)	C13—C18	1.413 (3)
C4—H4	0.9300	C14—C15	1.377 (4)
C5—C6	1.386 (4)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.391 (4)
C6—C7	1.376 (3)	C15—H15	0.9300
C6—H6	0.9300	C16—C17	1.381 (4)
C7—C8	1.477 (3)	C16—H16	0.9300
C8—O2	1.219 (2)	C17—C18	1.401 (3)
C8—N1	1.386 (3)	C17—H17	0.9300
C9—N1	1.455 (2)	C19—O3	1.195 (3)
C9—C19	1.527 (3)	C19—O4	1.306 (3)
C9—C10	1.532 (3)	N2—H2	0.86 (4)
C9—H9	0.9800	N3—O5	1.219 (3)
C10—C11	1.499 (3)	N3—O6	1.221 (3)
C10—H10A	0.9700	O4—H1	1.00 (4)
O1—C1—N1	122.92 (18)	C12—C11—C10	127.1 (2)
O1—C1—C2	131.44 (18)	C18—C11—C10	126.7 (2)
N1—C1—C2	105.61 (16)	C11—C12—N2	110.2 (2)
C3—C2—C7	118.11 (19)	C11—C12—H12	124.9
C3—C2—C1	133.98 (19)	N2—C12—H12	124.9
C7—C2—C1	107.76 (18)	N2—C13—C14	130.9 (2)
C2—C3—C4	120.1 (2)	N2—C13—C18	106.9 (2)
C2—C3—N3	121.67 (19)	C14—C13—C18	122.2 (2)
C4—C3—N3	118.2 (2)	C15—C14—C13	117.6 (2)
C5—C4—C3	120.2 (2)	C15—C14—H14	121.2
C5—C4—H4	119.9	C13—C14—H14	121.2
C3—C4—H4	119.9	C14—C15—C16	121.2 (2)
C4—C5—C6	120.6 (2)	C14—C15—H15	119.4
C4—C5—H5	119.7	C16—C15—H15	119.4
C6—C5—H5	119.7	C17—C16—C15	121.8 (2)
C7—C6—C5	118.1 (2)	C17—C16—H16	119.1
C7—C6—H6	121.0	C15—C16—H16	119.1
C5—C6—H6	121.0	C16—C17—C18	118.3 (2)
C6—C7—C2	122.7 (2)	C16—C17—H17	120.8
C6—C7—C8	129.2 (2)	C18—C17—H17	120.8
C2—C7—C8	108.11 (17)	C17—C18—C13	118.9 (2)
O2—C8—N1	123.29 (19)	C17—C18—C11	133.8 (2)
O2—C8—C7	129.99 (18)	C13—C18—C11	107.29 (19)
N1—C8—C7	106.71 (17)	O3—C19—O4	123.7 (2)

supplementary materials

N1—C9—C19	108.63 (16)	O3—C19—C9	122.70 (19)
N1—C9—C10	112.37 (17)	O4—C19—C9	113.59 (18)
C19—C9—C10	116.40 (17)	C8—N1—C1	111.65 (16)
N1—C9—H9	106.3	C8—N1—C9	123.58 (16)
C19—C9—H9	106.3	C1—N1—C9	124.30 (16)
C10—C9—H9	106.3	C13—N2—C12	109.5 (2)
C11—C10—C9	113.22 (18)	C13—N2—H2	125 (3)
C11—C10—H10A	108.9	C12—N2—H2	125 (3)
C9—C10—H10A	108.9	O5—N3—O6	124.1 (2)
C11—C10—H10B	108.9	O5—N3—C3	117.55 (18)
C9—C10—H10B	108.9	O6—N3—C3	118.3 (2)
H10A—C10—H10B	107.7	C19—O4—H1	109 (2)
C12—C11—C18	106.1 (2)		
O1—C1—C2—C3	-1.5 (4)	C16—C17—C18—C13	-1.3 (3)
N1—C1—C2—C3	-179.2 (2)	C16—C17—C18—C11	178.1 (2)
O1—C1—C2—C7	173.8 (2)	N2—C13—C18—C17	-179.45 (18)
N1—C1—C2—C7	-3.9 (2)	C14—C13—C18—C17	1.8 (3)
C7—C2—C3—C4	-2.8 (3)	N2—C13—C18—C11	1.0 (2)
C1—C2—C3—C4	172.1 (2)	C14—C13—C18—C11	-177.7 (2)
C7—C2—C3—N3	174.80 (18)	C12—C11—C18—C17	-179.8 (2)
C1—C2—C3—N3	-10.3 (3)	C10—C11—C18—C17	-3.3 (4)
C2—C3—C4—C5	4.2 (3)	C12—C11—C18—C13	-0.4 (2)
N3—C3—C4—C5	-173.46 (19)	C10—C11—C18—C13	176.2 (2)
C3—C4—C5—C6	-1.9 (3)	N1—C9—C19—O3	45.1 (3)
C4—C5—C6—C7	-1.7 (3)	C10—C9—C19—O3	173.1 (2)
C5—C6—C7—C2	3.2 (3)	N1—C9—C19—O4	-136.7 (2)
C5—C6—C7—C8	-176.1 (2)	C10—C9—C19—O4	-8.7 (3)
C3—C2—C7—C6	-0.9 (3)	O2—C8—N1—C1	176.27 (19)
C1—C2—C7—C6	-177.10 (18)	C7—C8—N1—C1	-2.7 (2)
C3—C2—C7—C8	178.52 (17)	O2—C8—N1—C9	3.8 (3)
C1—C2—C7—C8	2.4 (2)	C7—C8—N1—C9	-175.13 (18)
C6—C7—C8—O2	0.6 (4)	O1—C1—N1—C8	-173.90 (19)
C2—C7—C8—O2	-178.8 (2)	C2—C1—N1—C8	4.1 (2)
C6—C7—C8—N1	179.5 (2)	O1—C1—N1—C9	-1.5 (3)
C2—C7—C8—N1	0.1 (2)	C2—C1—N1—C9	176.45 (17)
N1—C9—C10—C11	-155.69 (18)	C19—C9—N1—C8	-134.89 (18)
C19—C9—C10—C11	78.1 (2)	C10—C9—N1—C8	94.9 (2)
C9—C10—C11—C12	-105.8 (3)	C19—C9—N1—C1	53.6 (2)
C9—C10—C11—C18	78.3 (3)	C10—C9—N1—C1	-76.7 (2)
C18—C11—C12—N2	-0.4 (3)	C14—C13—N2—C12	177.3 (2)
C10—C11—C12—N2	-176.9 (2)	C18—C13—N2—C12	-1.3 (3)
N2—C13—C14—C15	-179.5 (2)	C11—C12—N2—C13	1.1 (3)
C18—C13—C14—C15	-1.1 (3)	C2—C3—N3—O5	-34.6 (3)
C13—C14—C15—C16	-0.1 (4)	C4—C3—N3—O5	143.1 (2)
C14—C15—C16—C17	0.5 (4)	C2—C3—N3—O6	147.9 (2)
C15—C16—C17—C18	0.2 (3)	C4—C3—N3—O6	-34.5 (3)

Hydrogen-bond geometry (Å, °)

<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>
N2—H2 \cdots O1 ⁱ	0.86 (4)	2.28 (4)	3.002 (3)	142 (4)
O4—H1 \cdots O2 ⁱⁱ	1.00 (4)	1.78 (4)	2.716 (2)	154 (3)

Symmetry codes: (i) $-x+1, y-1/2, -z$; (ii) $x, y, z-1$.

Table 2

π - π stacking interactions between six-membered rings from indole (C13—C18; ring centroid Cg(1)) and nitrophthalimide (C2—C7; ring centroid Cg(2)).

Cg-Cg : distance (Å) between ring centroids; α : dihedral angle(°) between ring planes 1 and 2 ; β : angle (°) between Cg(1)-->Cg(2) vector and normal to ring plane 1 ; γ : angle (°) between Cg(1)-->Cg(2) vector and normal to ring plane 2 ; Cg1_Perp : perpendicular distance (Å) of Cg(1) on ring plane 2 ; Cg2_Perp : perpendicular distance (Å) of Cg(2) on ring plane 1.

Cg(I)-Cg(J)	sym (J)	Cg-Cg	α	β	γ	Cg1_Perp	Cg2_Perp
Cg(1)-Cg(2)	(i)	3.638 (1)	7.24	18.69	20.72	-3.403 (1)	3.446 (1)
Cg(1)-Cg(2)	(ii)	3.610 (1)	7.24	19.41	12.26	3.528 (1)	3.405 (1)

Symmetry codes : (i) $1 - x, 1/2 + y, 1 - z$, (ii) $2 - x, 1/2 + y, 1 - z$

Fig. 1

