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# *N*-Methyl-1,2-diphenylcyclobuteno-[3,4-*a*]naphthalene-2,3-dicarboximide

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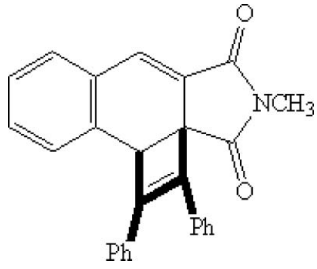
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.058; $wR$ factor = 0.123; data-to-parameter ratio = 13.7.

The title compound, $C_{27}H_{19}NO_2$, is the product of a photochemical reaction between *N*-methylnaphthalene-2,3-dicarboximide and diphenylacetylene. The cyclobutene ring in the molecule is almost perpendicular to the plane of the dihydronaphthalimide system; the interplanar angle between the cyclobutene ring and the plane through the two six-membered rings of the naphthalimide system is 70.66 (9)°. The interplanar angle between the two phenyl substituents on the cyclobutene ring is 42.47 (8)°.

## Related literature

For information on the photochemistry of naphthalene-dicarboximides with alkynes see Liu *et al.* (2006) and for related structures see Liu *et al.* (2003, 2006).




## Experimental

### *Crystal data*

$C_{27}H_{19}NO_2$
$M_r$ = 389.43
Monoclinic, $P2_1/c$
$a$ = 10.763 (2) Å
$b$ = 7.9925 (15) Å
$c$ = 23.438 (5) Å
$\beta$ = 97.326 (3)°
$V$ = 1999.8 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 298 (2) K
0.28 × 0.22 × 0.08 mm

### *Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
10516 measured reflections
3715 independent reflections
2456 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.062

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.123
$S$ = 1.01
3715 reflections
272 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

The author gratefully acknowledges Professor Jianhua Xu of Nanjing University for his help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2314).

## References

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o2901 [ **doi:10.1107/S1600536807022568** ]

# *N*-Methyl-1,2-diphenylcyclobuteno[3,4-*a*]naphthalene-2,3-dicarboximide

**Q.-J. Liu**

## Comment

In the studies of photochemistry of naphthalenedicarboximides with alkynes, it has been found that the photoinduced cycloadditions of *N*-methyl-1,8-naphthalenedicarboximides (NI18) with alkynes followed the pathway of NI18 with alkenes (Liu *et al.*, 2006), but that is not the case for *N*-methylnaphthalene-2,3-dicarboximide (NI23). UV irradiation of NI23 in the presence of diphenylacetylene in benzene solution resulted in the novel adduct (I) and its structure is reported here (Figure 1).

The bond lengths and angles in (I) show normal values except for the geometry of the cyclobutene ring (Table 1). The C20—C21 bond length of 1.345 (3)Å is nearly the same as that in 4-methyl-1,2-diphenylbenzo[de]cyclobut[i]isoquinoline-3,5(2*H*)-dione(1.346 (3) Å) (Liu *et al.*, 2006), but is longer than those of 4,5,9,10-tetrahydro-4- methyl-2-phenyl-9,10-epoxy-3H,10aH-cyclobuta[benzo[2,3,4-de]- isoquinoline-3,5-dione (1.324 (2) Å) (Liu *et al.*, 2003), Such elongation is considered to be caused by the steric effect of the bulky phenyl substituents on C20 and C21, respectively. The cyclobutene ring in the molecule is almost perpendicular to the plane of dihydronaphthalimide, the interplanar angle between the cyclobutene ring and the plane through the two six membered rings of the naphthalimide is 70.66 (9). The interplanar angle between the two benzene substituents on the cyclobutene ring is 42.47 (8).

## Experimental

The title compound, (I), was prepared by irradiation of a benzene (150 ml) solution of *N*-methylnaphthalene-2,3-dicarboximide (633 mg, 3.0 mmol) with diphenylacetylene (1.068 g, 6 mmol) with UV light of wavelength longer than 300 nm purged with nitrogen, and isolated by flash column chromatography on silica gel, eluting with petroleum-ethyl acetate (4:1 v/v). Single crystals (m.p. 505–506 K) suitable for X-ray diffraction were obtained by recrystallization from ethanol-ethyl acetate solution (1:95 v/v).

## Refinement

Hydrogen atoms were placed in geometrically idealized positions and refined using a riding model with d (C—H) = 0.93 Å, $U_{iso}$ =1.2$U_{eq}$ (C) for aromatic 0.98 Å, $U_{iso}$ = 1.2$U_{eq}$ (C) for CH and 0.96 Å, $U_{iso}$ = 1.5$U_{eq}$ (C) for $CH_3$ atoms.

## Figures

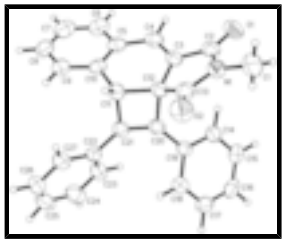

Fig. 1. The structure of (I) with the atom-numbering scheme and ellipsoids drawn at the 30% probability level.

Fig. 2. The formation of (I).

### *N*-Methyl-1,2-diphenylcyclobuteno[3,4-a]naphthalene-2,3-dicarboximide

*Crystal data*

| | |
|---|---|
| $C_{27}H_{19}NO_2$ | $F_{000}$ = 816 |
| $M_r$ = 389.43 | $D_x$ = 1.293 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Melting point: 505 K |
| Hall symbol: -P 2ybc | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 10.763 (2) Å | Cell parameters from 1602 reflections |
| $b$ = 7.9925 (15) Å | θ = 2.4–20.9º |
| $c$ = 23.438 (5) Å | μ = 0.08 mm$^{-1}$ |
| β = 97.326 (3)º | $T$ = 298 (2) K |
| $V$ = 1999.8 (7) Å$^3$ | Plate, colourless |
| $Z$ = 4 | 0.28 × 0.22 × 0.08 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 2456 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int}$ = 0.062 |
| Monochromator: graphite | $\theta_{max}$ = 25.5º |
| $T$ = 298(2) K | $\theta_{min}$ = 1.9º |
| φ and ω scans | $h$ = −11→13 |
| Absorption correction: none | $k$ = −9→7 |
| 10516 measured reflections | $l$ = −28→24 |
| 3715 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.058 | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2)$ = 0.123 | $\Delta\rho_{max}$ = 0.17 e Å$^{-3}$ |
| $S$ = 1.01 | $\Delta\rho_{min}$ = −0.15 e Å$^{-3}$ |
| 3715 reflections | Extinction correction: none |
| 272 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 esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal estimating esds 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 $F^2$>2sigma ($F^2$) 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 | 0.5270 (3) | 0.7812 (4) | 0.55850 (12) | 0.0659 (9) |
| H1A | 0.5237 | 0.7875 | 0.5174 | 0.099* |
| H1B | 0.6028 | 0.7257 | 0.5743 | 0.099* |
| H1C | 0.5256 | 0.8921 | 0.5741 | 0.099* |
| C2 | 0.4147 (2) | 0.5119 (3) | 0.57395 (9) | 0.0415 (6) |
| C3 | 0.2884 (2) | 0.4690 (3) | 0.58640 (9) | 0.0362 (6) |
| C4 | 0.2254 (2) | 0.3278 (3) | 0.57293 (9) | 0.0417 (6) |
| H4 | 0.2692 | 0.2324 | 0.5647 | 0.050* |
| C5 | 0.0900 (2) | 0.3192 (3) | 0.57082 (9) | 0.0406 (6) |
| C6 | 0.0276 (3) | 0.1680 (3) | 0.55931 (10) | 0.0528 (7) |
| H6 | 0.0738 | 0.0705 | 0.5568 | 0.063* |
| C7 | −0.1008 (3) | 0.1601 (4) | 0.55158 (11) | 0.0608 (8) |
| H7 | −0.1408 | 0.0576 | 0.5448 | 0.073* |
| C8 | −0.1705 (3) | 0.3042 (4) | 0.55384 (11) | 0.0577 (8) |
| H8 | −0.2576 | 0.3000 | 0.5480 | 0.069* |
| C9 | −0.1089 (2) | 0.4554 (4) | 0.56487 (10) | 0.0501 (7) |
| H9 | −0.1560 | 0.5526 | 0.5658 | 0.060* |
| C10 | 0.0197 (2) | 0.4662 (3) | 0.57446 (9) | 0.0389 (6) |
| C11 | 0.0817 (2) | 0.6310 (3) | 0.59191 (10) | 0.0384 (6) |
| H11 | 0.0499 | 0.7255 | 0.5677 | 0.046* |
| C12 | 0.2275 (2) | 0.6241 (3) | 0.60469 (9) | 0.0361 (6) |
| C13 | 0.3101 (2) | 0.7618 (3) | 0.58514 (10) | 0.0428 (6) |
| C14 | 0.4107 (2) | 0.5646 (3) | 0.72463 (10) | 0.0410 (6) |
| H14 | 0.4304 | 0.5042 | 0.6931 | 0.049* |
| C15 | 0.4926 (2) | 0.5629 (3) | 0.77487 (11) | 0.0477 (7) |
| H15 | 0.5656 | 0.4997 | 0.7773 | 0.057* |
| C16 | 0.4659 (2) | 0.6550 (3) | 0.82139 (11) | 0.0506 (7) |
| H16 | 0.5206 | 0.6538 | 0.8555 | 0.061* |
| C17 | 0.3578 (2) | 0.7489 (3) | 0.81730 (11) | 0.0496 (7) |
| H17 | 0.3406 | 0.8131 | 0.8484 | 0.060* |
| C18 | 0.2754 (2) | 0.7483 (3) | 0.76758 (10) | 0.0435 (6) |
| H18 | 0.2023 | 0.8114 | 0.7655 | 0.052* |
| C19 | 0.2998 (2) | 0.6539 (3) | 0.71998 (9) | 0.0342 (6) |
| C20 | 0.2084 (2) | 0.6472 (3) | 0.66827 (10) | 0.0360 (6) |

| | | | | |
|---|---|---|---|---|
| C21 | 0.0834 (2) | 0.6619 (3) | 0.65621 (10) | 0.0373 (6) |
| C22 | −0.0208 (2) | 0.6824 (3) | 0.69000 (10) | 0.0386 (6) |
| C23 | −0.0146 (2) | 0.6137 (3) | 0.74466 (11) | 0.0475 (7) |
| H23 | 0.0551 | 0.5512 | 0.7594 | 0.057* |
| C24 | −0.1108 (3) | 0.6373 (3) | 0.77724 (12) | 0.0585 (8) |
| H24 | −0.1054 | 0.5916 | 0.8140 | 0.070* |
| C25 | −0.2141 (3) | 0.7276 (4) | 0.75599 (15) | 0.0628 (9) |
| H25 | −0.2781 | 0.7451 | 0.7785 | 0.075* |
| C26 | −0.2234 (3) | 0.7922 (4) | 0.70151 (15) | 0.0635 (9) |
| H26 | −0.2948 | 0.8510 | 0.6866 | 0.076* |
| C27 | −0.1271 (2) | 0.7703 (3) | 0.66871 (12) | 0.0521 (7) |
| H27 | −0.1338 | 0.8152 | 0.6318 | 0.062* |
| N1 | 0.41972 (19) | 0.6876 (3) | 0.57308 (8) | 0.0442 (5) |
| O1 | 0.50087 (17) | 0.4229 (2) | 0.56464 (7) | 0.0553 (5) |
| O2 | 0.28991 (17) | 0.9106 (2) | 0.58275 (8) | 0.0599 (5) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.056 (2) | 0.076 (2) | 0.068 (2) | −0.0136 (16) | 0.0180 (15) | −0.0095 (16) |
| C2 | 0.0456 (17) | 0.0540 (18) | 0.0244 (13) | 0.0114 (13) | 0.0026 (11) | 0.0012 (12) |
| C3 | 0.0397 (15) | 0.0406 (15) | 0.0282 (13) | 0.0099 (12) | 0.0038 (11) | 0.0018 (11) |
| C4 | 0.0517 (18) | 0.0376 (15) | 0.0352 (14) | 0.0127 (13) | 0.0033 (12) | 0.0013 (12) |
| C5 | 0.0518 (17) | 0.0427 (16) | 0.0270 (13) | 0.0008 (13) | 0.0044 (11) | 0.0015 (12) |
| C6 | 0.066 (2) | 0.0465 (17) | 0.0441 (17) | −0.0019 (15) | 0.0011 (14) | −0.0001 (13) |
| C7 | 0.074 (2) | 0.062 (2) | 0.0461 (17) | −0.0197 (17) | 0.0068 (15) | −0.0055 (15) |
| C8 | 0.0507 (18) | 0.082 (2) | 0.0396 (16) | −0.0136 (17) | 0.0035 (13) | −0.0123 (16) |
| C9 | 0.0482 (18) | 0.066 (2) | 0.0352 (15) | 0.0011 (14) | 0.0032 (12) | −0.0057 (14) |
| C10 | 0.0445 (16) | 0.0473 (16) | 0.0247 (13) | 0.0014 (13) | 0.0037 (11) | −0.0009 (11) |
| C11 | 0.0418 (16) | 0.0352 (15) | 0.0369 (14) | 0.0096 (11) | 0.0000 (11) | 0.0009 (11) |
| C12 | 0.0393 (15) | 0.0360 (14) | 0.0333 (14) | 0.0066 (11) | 0.0064 (11) | 0.0002 (11) |
| C13 | 0.0472 (17) | 0.0475 (17) | 0.0331 (15) | 0.0040 (14) | 0.0025 (12) | −0.0012 (13) |
| C14 | 0.0384 (16) | 0.0432 (15) | 0.0424 (15) | 0.0003 (12) | 0.0084 (12) | −0.0016 (12) |
| C15 | 0.0352 (16) | 0.0543 (18) | 0.0522 (17) | −0.0018 (13) | −0.0001 (13) | 0.0018 (14) |
| C16 | 0.0435 (17) | 0.0651 (19) | 0.0406 (16) | −0.0135 (14) | −0.0051 (12) | 0.0041 (14) |
| C17 | 0.0517 (18) | 0.0574 (18) | 0.0405 (16) | −0.0093 (14) | 0.0084 (13) | −0.0111 (13) |
| C18 | 0.0406 (16) | 0.0454 (15) | 0.0451 (16) | 0.0014 (12) | 0.0082 (12) | −0.0047 (13) |
| C19 | 0.0350 (14) | 0.0331 (14) | 0.0354 (14) | −0.0008 (11) | 0.0077 (11) | 0.0003 (11) |
| C20 | 0.0385 (15) | 0.0330 (14) | 0.0367 (14) | 0.0056 (11) | 0.0050 (11) | −0.0019 (11) |
| C21 | 0.0411 (16) | 0.0328 (14) | 0.0378 (14) | 0.0070 (11) | 0.0039 (11) | −0.0013 (11) |
| C22 | 0.0358 (15) | 0.0355 (14) | 0.0439 (16) | 0.0039 (11) | 0.0030 (11) | −0.0107 (12) |
| C23 | 0.0424 (17) | 0.0458 (17) | 0.0556 (18) | 0.0050 (12) | 0.0110 (13) | 0.0006 (14) |
| C24 | 0.063 (2) | 0.0551 (19) | 0.0621 (19) | −0.0040 (15) | 0.0253 (16) | −0.0019 (15) |
| C25 | 0.051 (2) | 0.058 (2) | 0.086 (3) | −0.0054 (16) | 0.0312 (17) | −0.0236 (18) |
| C26 | 0.0395 (18) | 0.067 (2) | 0.083 (2) | 0.0123 (14) | 0.0045 (16) | −0.0260 (18) |
| C27 | 0.0470 (18) | 0.0579 (18) | 0.0501 (17) | 0.0131 (14) | 0.0016 (13) | −0.0109 (14) |
| N1 | 0.0416 (13) | 0.0496 (14) | 0.0422 (13) | −0.0013 (11) | 0.0084 (10) | −0.0006 (10) |
| O1 | 0.0509 (12) | 0.0680 (13) | 0.0486 (12) | 0.0213 (10) | 0.0125 (9) | 0.0012 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.0661 (14) | 0.0384 (11) | 0.0755 (14) | 0.0037 (10) | 0.0095 (10) | 0.0017 (10) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.453 (3) | C13—O2 | 1.208 (3) |
| C1—H1A | 0.9600 | C13—N1 | 1.382 (3) |
| C1—H1B | 0.9600 | C14—C15 | 1.378 (3) |
| C1—H1C | 0.9600 | C14—C19 | 1.383 (3) |
| C2—O1 | 1.210 (3) | C14—H14 | 0.9300 |
| C2—N1 | 1.406 (3) | C15—C16 | 1.376 (3) |
| C2—C3 | 1.467 (3) | C15—H15 | 0.9300 |
| C3—C4 | 1.333 (3) | C16—C17 | 1.378 (3) |
| C3—C12 | 1.491 (3) | C16—H16 | 0.9300 |
| C4—C5 | 1.453 (3) | C17—C18 | 1.371 (3) |
| C4—H4 | 0.9300 | C17—H17 | 0.9300 |
| C5—C6 | 1.392 (3) | C18—C19 | 1.399 (3) |
| C5—C10 | 1.407 (3) | C18—H18 | 0.9300 |
| C6—C7 | 1.372 (4) | C19—C20 | 1.461 (3) |
| C6—H6 | 0.9300 | C20—C21 | 1.345 (3) |
| C7—C8 | 1.379 (4) | C21—C22 | 1.462 (3) |
| C7—H7 | 0.9300 | C22—C27 | 1.381 (3) |
| C8—C9 | 1.387 (3) | C22—C23 | 1.388 (3) |
| C8—H8 | 0.9300 | C23—C24 | 1.376 (3) |
| C9—C10 | 1.376 (3) | C23—H23 | 0.9300 |
| C9—H9 | 0.9300 | C24—C25 | 1.365 (4) |
| C10—C11 | 1.509 (3) | C24—H24 | 0.9300 |
| C11—C21 | 1.525 (3) | C25—C26 | 1.369 (4) |
| C11—C12 | 1.560 (3) | C25—H25 | 0.9300 |
| C11—H11 | 0.9800 | C26—C27 | 1.378 (4) |
| C12—C13 | 1.522 (3) | C26—H26 | 0.9300 |
| C12—C20 | 1.541 (3) | C27—H27 | 0.9300 |
| N1—C1—H1A | 109.5 | N1—C13—C12 | 107.3 (2) |
| N1—C1—H1B | 109.5 | C15—C14—C19 | 121.6 (2) |
| H1A—C1—H1B | 109.5 | C15—C14—H14 | 119.2 |
| N1—C1—H1C | 109.5 | C19—C14—H14 | 119.2 |
| H1A—C1—H1C | 109.5 | C16—C15—C14 | 119.8 (2) |
| H1B—C1—H1C | 109.5 | C16—C15—H15 | 120.1 |
| O1—C2—N1 | 123.5 (2) | C14—C15—H15 | 120.1 |
| O1—C2—C3 | 130.5 (3) | C15—C16—C17 | 119.8 (2) |
| N1—C2—C3 | 105.9 (2) | C15—C16—H16 | 120.1 |
| C4—C3—C2 | 127.4 (2) | C17—C16—H16 | 120.1 |
| C4—C3—C12 | 122.9 (2) | C18—C17—C16 | 120.4 (2) |
| C2—C3—C12 | 108.4 (2) | C18—C17—H17 | 119.8 |
| C3—C4—C5 | 121.7 (2) | C16—C17—H17 | 119.8 |
| C3—C4—H4 | 119.1 | C17—C18—C19 | 120.8 (2) |
| C5—C4—H4 | 119.1 | C17—C18—H18 | 119.6 |
| C6—C5—C10 | 119.1 (3) | C19—C18—H18 | 119.6 |
| C6—C5—C4 | 120.3 (2) | C14—C19—C18 | 117.6 (2) |
| C10—C5—C4 | 120.2 (2) | C14—C19—C20 | 121.9 (2) |

| | | | |
|---|---|---|---|
| C7—C6—C5 | 121.2 (3) | C18—C19—C20 | 120.5 (2) |
| C7—C6—H6 | 119.4 | C21—C20—C19 | 136.1 (2) |
| C5—C6—H6 | 119.4 | C21—C20—C12 | 93.44 (19) |
| C6—C7—C8 | 120.0 (3) | C19—C20—C12 | 130.4 (2) |
| C6—C7—H7 | 120.0 | C20—C21—C22 | 135.4 (2) |
| C8—C7—H7 | 120.0 | C20—C21—C11 | 94.53 (19) |
| C7—C8—C9 | 119.0 (3) | C22—C21—C11 | 129.8 (2) |
| C7—C8—H8 | 120.5 | C27—C22—C23 | 118.3 (2) |
| C9—C8—H8 | 120.5 | C27—C22—C21 | 121.3 (2) |
| C10—C9—C8 | 122.2 (3) | C23—C22—C21 | 120.4 (2) |
| C10—C9—H9 | 118.9 | C24—C23—C22 | 120.5 (2) |
| C8—C9—H9 | 118.9 | C24—C23—H23 | 119.8 |
| C9—C10—C5 | 118.4 (2) | C22—C23—H23 | 119.8 |
| C9—C10—C11 | 120.0 (2) | C25—C24—C23 | 120.4 (3) |
| C5—C10—C11 | 121.5 (2) | C25—C24—H24 | 119.8 |
| C10—C11—C21 | 111.00 (19) | C23—C24—H24 | 119.8 |
| C10—C11—C12 | 114.87 (19) | C24—C25—C26 | 119.9 (3) |
| C21—C11—C12 | 86.05 (17) | C24—C25—H25 | 120.0 |
| C10—C11—H11 | 114.0 | C26—C25—H25 | 120.0 |
| C21—C11—H11 | 114.0 | C25—C26—C27 | 120.1 (3) |
| C12—C11—H11 | 114.0 | C25—C26—H26 | 120.0 |
| C3—C12—C13 | 102.7 (2) | C27—C26—H26 | 120.0 |
| C3—C12—C20 | 119.68 (19) | C26—C27—C22 | 120.7 (3) |
| C13—C12—C20 | 111.06 (19) | C26—C27—H27 | 119.6 |
| C3—C12—C11 | 116.3 (2) | C22—C27—H27 | 119.6 |
| C13—C12—C11 | 121.9 (2) | C13—N1—C2 | 113.0 (2) |
| C20—C12—C11 | 85.83 (17) | C13—N1—C1 | 123.5 (2) |
| O2—C13—N1 | 124.5 (2) | C2—N1—C1 | 123.4 (2) |
| O2—C13—C12 | 128.1 (2) | | |
| O1—C2—C3—C4 | −23.2 (4) | C16—C17—C18—C19 | −0.6 (4) |
| N1—C2—C3—C4 | 155.4 (2) | C15—C14—C19—C18 | 2.4 (3) |
| O1—C2—C3—C12 | 170.0 (2) | C15—C14—C19—C20 | −175.7 (2) |
| N1—C2—C3—C12 | −11.4 (2) | C17—C18—C19—C14 | −1.3 (3) |
| C2—C3—C4—C5 | −160.1 (2) | C17—C18—C19—C20 | 176.8 (2) |
| C12—C3—C4—C5 | 4.9 (3) | C14—C19—C20—C21 | 150.3 (3) |
| C3—C4—C5—C6 | −176.9 (2) | C18—C19—C20—C21 | −27.7 (4) |
| C3—C4—C5—C10 | 9.7 (3) | C14—C19—C20—C12 | −31.9 (4) |
| C10—C5—C6—C7 | −0.1 (4) | C18—C19—C20—C12 | 150.1 (2) |
| C4—C5—C6—C7 | −173.6 (2) | C3—C12—C20—C21 | −121.1 (2) |
| C5—C6—C7—C8 | 1.5 (4) | C13—C12—C20—C21 | 119.6 (2) |
| C6—C7—C8—C9 | −1.0 (4) | C11—C12—C20—C21 | −3.11 (19) |
| C7—C8—C9—C10 | −0.9 (4) | C3—C12—C20—C19 | 60.5 (3) |
| C8—C9—C10—C5 | 2.3 (4) | C13—C12—C20—C19 | −58.9 (3) |
| C8—C9—C10—C11 | −174.1 (2) | C11—C12—C20—C19 | 178.5 (2) |
| C6—C5—C10—C9 | −1.8 (3) | C19—C20—C21—C22 | −4.2 (5) |
| C4—C5—C10—C9 | 171.7 (2) | C12—C20—C21—C22 | 177.5 (3) |
| C6—C5—C10—C11 | 174.6 (2) | C19—C20—C21—C11 | −178.5 (3) |
| C4—C5—C10—C11 | −11.9 (3) | C12—C20—C21—C11 | 3.18 (19) |
| C9—C10—C11—C21 | 81.3 (3) | C10—C11—C21—C20 | 112.0 (2) |

| | | | |
|---|---|---|---|
| C5—C10—C11—C21 | −95.0 (2) | C12—C11—C21—C20 | −3.14 (19) |
| C9—C10—C11—C12 | 176.8 (2) | C10—C11—C21—C22 | −62.8 (3) |
| C5—C10—C11—C12 | 0.5 (3) | C12—C11—C21—C22 | −177.9 (2) |
| C4—C3—C12—C13 | −151.6 (2) | C20—C21—C22—C27 | 148.6 (3) |
| C2—C3—C12—C13 | 16.0 (2) | C11—C21—C22—C27 | −38.8 (4) |
| C4—C3—C12—C20 | 84.9 (3) | C20—C21—C22—C23 | −31.2 (4) |
| C2—C3—C12—C20 | −107.6 (2) | C11—C21—C22—C23 | 141.4 (2) |
| C4—C3—C12—C11 | −15.9 (3) | C27—C22—C23—C24 | −1.9 (4) |
| C2—C3—C12—C11 | 151.61 (19) | C21—C22—C23—C24 | 177.9 (2) |
| C10—C11—C12—C3 | 12.6 (3) | C22—C23—C24—C25 | 0.6 (4) |
| C21—C11—C12—C3 | 123.9 (2) | C23—C24—C25—C26 | 1.3 (4) |
| C10—C11—C12—C13 | 139.1 (2) | C24—C25—C26—C27 | −1.8 (4) |
| C21—C11—C12—C13 | −109.5 (2) | C25—C26—C27—C22 | 0.4 (4) |
| C10—C11—C12—C20 | −108.6 (2) | C23—C22—C27—C26 | 1.4 (4) |
| C21—C11—C12—C20 | 2.74 (16) | C21—C22—C27—C26 | −178.4 (2) |
| C3—C12—C13—O2 | 168.5 (2) | O2—C13—N1—C2 | −174.5 (2) |
| C20—C12—C13—O2 | −62.4 (3) | C12—C13—N1—C2 | 8.8 (3) |
| C11—C12—C13—O2 | 36.1 (4) | O2—C13—N1—C1 | 3.0 (4) |
| C3—C12—C13—N1 | −15.0 (2) | C12—C13—N1—C1 | −173.8 (2) |
| C20—C12—C13—N1 | 114.1 (2) | O1—C2—N1—C13 | −179.9 (2) |
| C11—C12—C13—N1 | −147.4 (2) | C3—C2—N1—C13 | 1.5 (3) |
| C19—C14—C15—C16 | −1.6 (4) | O1—C2—N1—C1 | 2.7 (4) |
| C14—C15—C16—C17 | −0.3 (4) | C3—C2—N1—C1 | −175.9 (2) |
| C15—C16—C17—C18 | 1.4 (4) | | |

**Fig. 1**

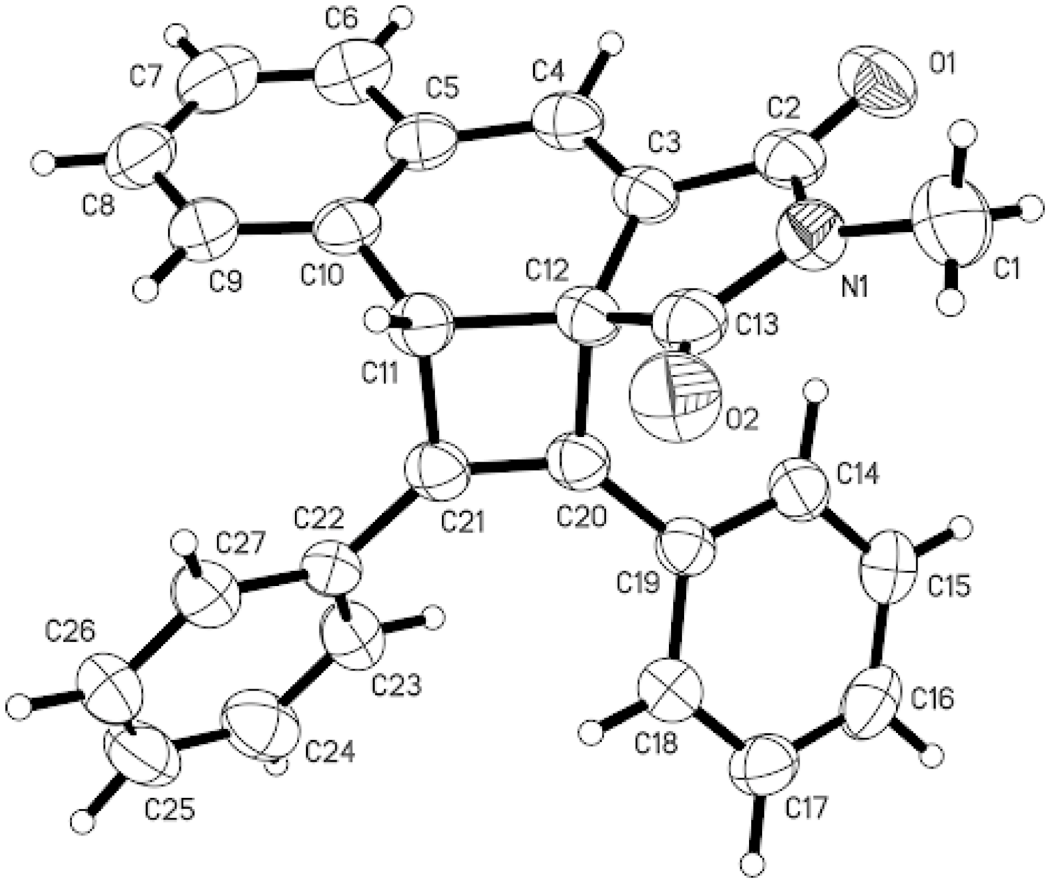
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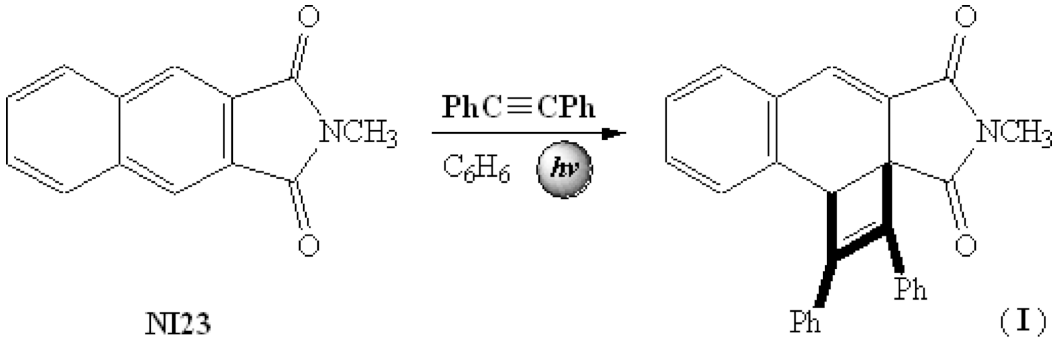
**Fig. 2**




NI23 (I)