Absorption correction:
empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.832$, $T_{\max} = 0.998$
2992 measured reflections
2812 independent reflections

$\theta_{\max} = 60°$
$h = 0 \rightarrow 38$
$k = 0 \rightarrow 10$
$l = -15 \rightarrow 15$
3 standard reflections
every 200 reflections
frequency: 120 min
intensity decay: <1.3%

Refinement

Refinement on *F*
$R = 0.06$
$wR = 0.082$
$S = 0.878$
2454 reflections
346 parameters
All H atoms refined
$w = 1/[\sigma^2(F) + 0.019F^2]$

$(\Delta/\sigma)_{\max} = 0.023$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

C1—C2	1.392 (5)	C10—N11	1.462 (5)
C1—C6	1.408 (4)	N11—C12	1.472 (3)
C1—O23	1.363 (3)	N11—C14	1.463 (3)
C2—C3	1.387 (4)	C12—C13	1.521 (4)
C2—C15	1.521 (4)	C15—N16	1.464 (5)
C3—C4	1.402 (4)	N16—C17	1.469 (4)
C4—C5	1.395 (5)	N16—C21	1.473 (3)
C4—C24	1.430 (4)	C17—C18	1.515 (5)
C5—C6	1.373 (4)	C18—N19	1.454 (4)
C6—C7	1.523 (5)	N19—C20	1.464 (4)
C7—N8	1.480 (3)	N19—C22	1.470 (6)
N8—C9	1.478 (3)	C20—C21	1.522 (5)
N8—C13	1.472 (5)	C24—N25	1.151 (4)
C9—C10	1.517 (4)		
C6—C1—O23	120.7 (3)	C9—C10—N11	110.7 (2)
C2—C1—O23	118.1 (3)	C10—N11—C14	109.9 (3)
C2—C1—C6	121.2 (3)	C10—N11—C12	109.6 (2)
C1—C2—C15	118.2 (2)	C12—N11—C14	110.5 (2)
C1—C2—C3	119.7 (3)	N11—C12—C13	110.4 (2)
C3—C2—C15	122.1 (3)	N8—C13—C12	109.4 (3)
C2—C3—C4	119.2 (3)	C2—C15—N16	112.6 (2)
C3—C4—C24	120.0 (3)	C15—N16—C21	110.3 (2)
C3—C4—C5	120.5 (2)	C15—N16—C17	110.7 (2)
C5—C4—C24	119.4 (3)	C17—N16—C21	109.3 (2)
C4—C5—C6	120.7 (3)	N16—C17—C18	109.9 (2)
C1—C6—C5	118.7 (3)	C17—C18—N19	109.9 (3)
C5—C6—C7	119.7 (3)	C18—N19—C22	111.4 (3)
C1—C6—C7	121.6 (2)	C18—N19—C20	109.1 (3)
C6—C7—N8	111.5 (3)	C20—N19—C22	110.6 (3)
C7—N8—C13	110.5 (2)	N19—C20—C21	110.3 (2)
C7—N8—C9	111.8 (2)	N16—C21—C20	109.8 (2)
C9—N8—C13	109.1 (2)	C4—C24—N25	177.8 (3)
N8—C9—C10	109.3 (2)		
C9—N8—C13—C12	−60.3 (3)	C17—N16—C21—C20	−58.0 (3)
C13—N8—C9—C10	60.3 (3)	C21—N16—C17—C18	58.9 (3)
N8—C9—C10—N11	−59.7 (3)	N16—C17—C18—N19	−60.6 (3)
C9—C10—N11—C12	58.3 (3)	C17—C18—N19—C20	60.3 (3)
C10—N11—C12—C13	−58.0 (3)	C18—N19—C20—C21	−59.6 (3)
N11—C12—C13—N8	59.4 (3)	N19—C20—C21—N16	58.8 (3)

Refinement was performed by full-matrix least-squares methods. All the H atoms were located from the difference Fourier map, except the two of the water molecule (O*W*), and were included in the refinement. Calculations were performed using VAX730 and MicroVAX II computers.

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: PT1043). Services for accessing these data are described at the back of the journal.

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3-(1,8-Naphthalenedicarboximido)benzonitrile [or *N*-(3-Cyanophenyl)-1,8-naphthalimide], $C_{19}H_{10}N_2O_2$

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Abstract

The reaction of 3-cyanoaniline and naphthalic anhydride in refluxing dimethylformamide yielded the title compound. In the crystal, the naphthalimide and benzonitrile ring planes are inclined at 69.36(16)°. The molecules pack in columnar stacks parallel to the *c* axis.

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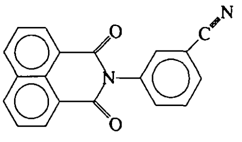
Comment

The title compound, (I), was synthesized as a precursor for the formation of cyclic 1,3,5-triazines with the potential to act as molecular tweezers in host–guest interactions (Whitlock & Sheridan, 1988; Zimmerman & van Zyl, 1987). Complexes containing the 1,4,5,8-naphthalenetetracarboxylic acid diimide units have been shown to act as receptors in inclusion complexes (Jazwinski *et al.*, 1987).



(I)

In the title compound, the naphthalimide and benzonitrile ring systems are linked through the N1—C13 bond which is 1.448 (4) Å long and the angle between the respective ring planes is 69.36 (16)° (Spek, 1995). The corresponding N—C bonds in 3-cyanoaniline, 1.408 (7) Å (Merlino & Sartori, 1982), and *N*-(3-cyanophenyl)acetohydroxamic acid, 1.417 (5) Å (Mocharla, Powell & van der Helm, 1984), are significantly shorter. The increase in N—C bond length may reflect the steric requirements of the carbonyl substituents on the dicarboximide ring or be caused by the loss of π-conjugation in the twisted naphthalimide. The only other aryl-substituted 1,8-naphthalenedicarboximide to have been structurally characterized is the 2-benzamide (Lindeman, Ponomarev & Rusanov, 1995), where the N—C bond is again long, 1.4485 (14) Å, but, in addition, the corresponding interplanar angle is increased to 87.05 (4)° in order to minimize steric interactions between the dicarboximide carbonyl groups and the *ortho*-benzamide substituent on the aryl ring.

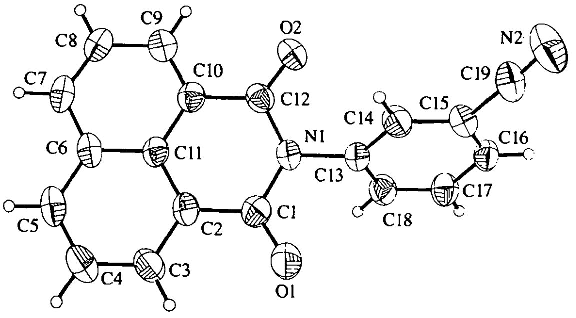


Fig. 1. Perspective drawing of the title molecule showing the atom-numbering scheme with displacement ellipsoids drawn at the 50% probability level.

Bond lengths and angles in the naphthalenedicarboximide system are very similar to those found in the benzamide (Lindeman *et al.*, 1995) and *N*-ethyl derivatives (Easton, Gulbis, Hoskins, Scharfbillig & Tiekink, 1992), and the ethyldimethylammonium cation (Clark & Hall, 1989). There is some indication of a pattern of long and short bond lengths in the naphthalene rings noted previously (Clark & Hall, 1989), but the variations are barely significant in this determination. The C15—C19, 1.444 (6), and C19—N2, 1.138 (5) Å, distances and C15—C19—N2, 178.6 (5)°, angle are comparable with those found in related benzonitrile derivatives (Merlino & Sartori, 1982; Mocharla *et al.*, 1984).

The structure is stabilized by a combination of hydrogen-bond and columnar interactions. Pairs of molecules form *via* C—H···O($-x$, y, $\frac{1}{2}-z$) intermolecular hydrogen bonds parallel to the *c* axis [H14···O2 2.46, C14···O2 3.370 (6) Å and C14—H14···O2 167°] (Fig. 2). These pairs stack further from alternate faces *via* van der Waals interactions. Mean perpendicular distances are 3.509 (4) Å between the ring planes of the hydrogen-bonded pairs and 3.615 (1) Å between the naphthalimide planes of adjacent pairs (Spek, 1995). Additional columnar interactions are possible between the aryl rings of the benzonitrile substituent parallel to the *a* axis with a mean interplanar separation of 3.401 (1) Å.

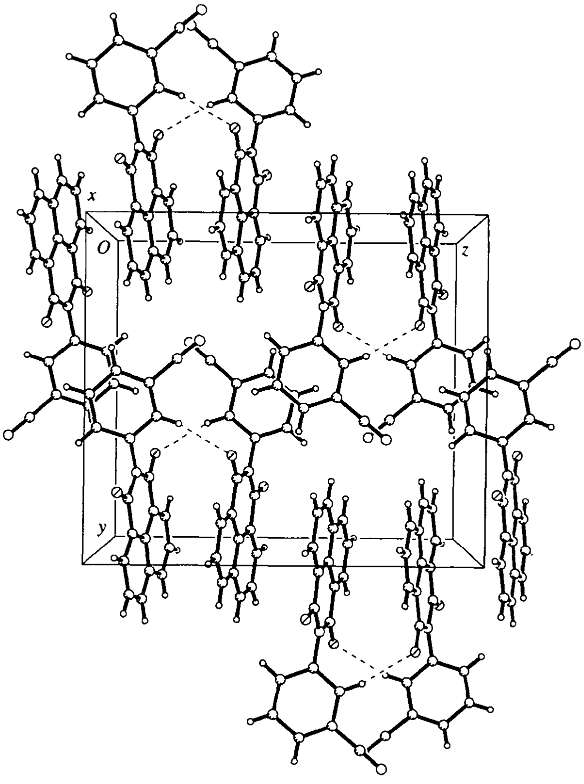


Fig. 2. Projection of the unit-cell contents down the *a* axis.

Experimental

The title compound was synthesized by refluxing 1,8-naphthalic anhydride (1.00 g, 5.04 mmol) and 3-cyanoaniline

(0.6 g, 5.08 mmol) in dimethylformamide (5 ml). The reaction mixture was poured onto crushed ice, stirred for 5 min and the resulting solid filtered and dried *in vacuo*. Column chromatography on silica with CH_2Cl_2/CH_3OH as eluant gave the product as a brown powder in 66% yield. ^{1}H NMR ($CDCl_3$): δ 8.65 (2H, *d*), 8.31 (2H, *d*), 7.86–7.60 (6H, *m*). Pale-yellow truncated octahedral crystals were obtained from CH_2Cl_2/hexane solution.

Crystal data

$C_{19}H_{10}N_2O_2$
$M_r = 298.29$
Orthorhombic
Pbcn
$a = 14.997\ (6)$ Å
$b = 12.898\ (9)$ Å
$c = 14.743\ (14)$ Å
$V = 2852\ (4)$ Å^3
$Z = 8$
$D_x = 1.390$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.05–17.43°$
$\mu = 0.092$ mm^{-1}
$T = 293\ (2)$ K
Truncated octahedron
0.46 × 0.46 × 0.40 mm
Pale yellow

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
2520 measured reflections
2520 independent reflections
1162 reflections with $I > 2\sigma(I)$

$\theta_{max} = 25.05°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 17$
3 standard reflections every 97 reflections intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.163$
$S = 0.926$
2520 reflections
208 parameters
H atoms riding using *AFIX* in *SHELXL*93 (Sheldrick, 1993)

$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.176$ e Å^{-3}
$\Delta\rho_{min} = -0.258$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.216 (4)	C7—C8	1.351 (5)
C1—N1	1.405 (5)	C8—C9	1.401 (5)
C1—C2	1.471 (5)	C9—C10	1.357 (6)
C2—C3	1.375 (5)	C10—C11	1.414 (5)
C2—C11	1.409 (5)	C10—C12	1.470 (5)
C3—C4	1.400 (5)	C12—O2	1.219 (4)
C4—C5	1.367 (6)	C12—N1	1.403 (5)
C5—C6	1.409 (5)	N1—C13	1.448 (4)
C6—C7	1.411 (5)	C15—C19	1.444 (6)
C6—C11	1.416 (5)	C19—N2	1.138 (5)
O1—C1—N1	119.6 (4)	N1—C12—C10	117.0 (3)
O1—C1—C2	123.8 (4)	C12—N1—C1	125.0 (3)
N1—C1—C2	116.6 (4)	C12—N1—C13	117.1 (3)
C11—C2—C1	120.3 (3)	C1—N1—C13	117.8 (3)
C11—C10—C12	119.9 (4)	C14—C13—N1	120.4 (3)
C2—C11—C10	121.1 (3)	C18—C13—N1	119.1 (3)
O2—C12—N1	119.8 (3)	C16—C15—C19	119.3 (3)
O2—C12—C10	123.2 (4)	N2—C19—C15	178.6 (5)

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1259). Services for accessing these data are described at the back of the journal.

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1,8-Bis(dimethylphosphino)naphthalene: an Unusual Case of Pseudosymmetry

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Abstract

The title compound, $C_{14}H_{18}P_2$, crystallizes in the rare tetragonal space group $P4_2bc$ with two independent mol-