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Key indicators

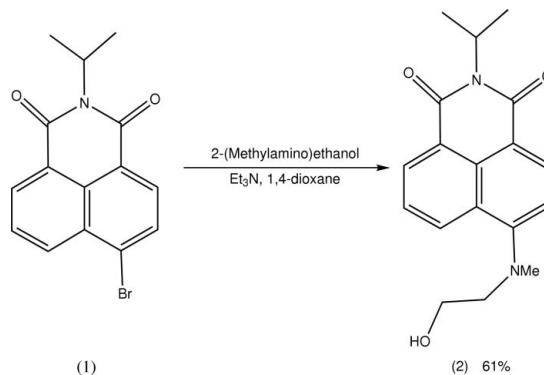
Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.054
 wR factor = 0.161
Data-to-parameter ratio = 12.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-[*N*-(2-Hydroxyethyl)-*N*-methylamino]-*N*-isopropyl-1,8-naphthalimide

In the title crystal structure, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_3$, molecules are linked into centrosymmetric dimers *via* $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{H}\cdots\text{O} = 1.92(3)$ Å]. These dimers are, in turn, linked by weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{H}\cdots\text{O} = 2.42$ Å], forming a two-dimensional network.

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Comment

1,8-Naphthalimide derivatives are highly fluorescent and photostable. They have been used as fluorescent dyes for synthetic polymers and textile materials, in fluorescent solar energy collectors, as liquid-crystal additives, as electro-optically sensitive materials, in laser technology and as fluorescent markers in medicine and biology (Grabtchev *et al.*, 1996). 4-Amino-1,8-naphthalimide derivatives are used as yellow components for daylight fluorescent pigments; most applications of 1,8-naphthalimide derivatives are as fluorescent brighteners in detergents, textiles, paper, plastics and paints (Dorlars *et al.*, 1975; Gold, 1971). 1,8-Naphthalimide derivatives are used as fluorescent dichroic dyes in liquid crystal displays (Wolarz *et al.*, 1992; Marty'nski, Mykowska & Bauman, 1994; Marty'nski, Mykowska, Stolarski & Bauman, 1994), and their high fluorescent quantum yield and photostability makes them excellent candidates for dye lasers, where a high quantum yield and low level of re-absorption are extremely important. More recently, 1,8-naphthalimide derivatives have been studied as DNA intercalators (Tao *et al.*, 1996) and they have been proposed for use in the quantitation of paramagnetic transition metal cations by fluorescent emission enhancement (Chang *et al.*, 1992; Mitchell *et al.*, 1998). Here we report the crystal structure of the title naphthalimide, derived from the reaction of 2-(methylamino)ethanol with 4-bromo-*N*-isopropyl-1,8-naphthalimide in the presence of triethylamine as a base.



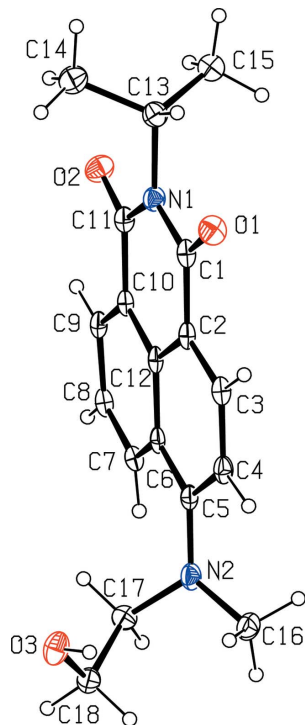


Figure 1
Molecular structure of (2), showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

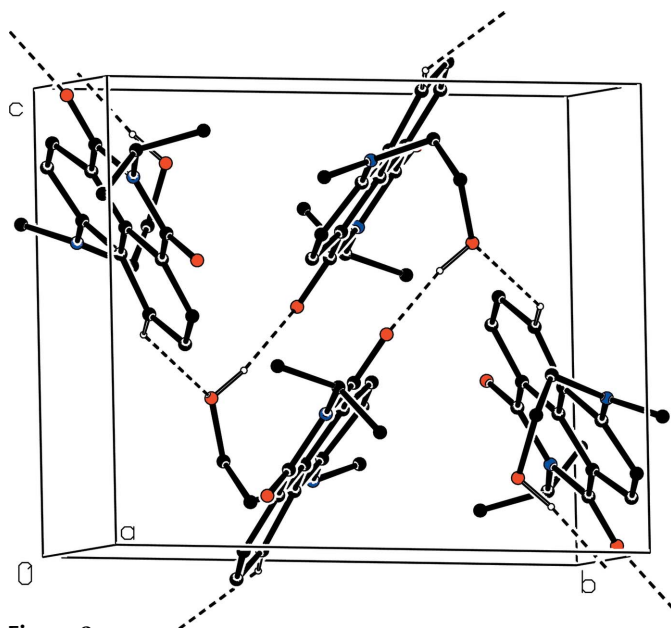


Figure 2
A partial packing plot (Spek, 2003) of (2), showing hydrogen bonds as dashed lines.

geometry at atom N2 is slightly pyramidal as indicated by the sum of the angles at this atom [357.1°] compared to the sum of the angles at N1 [360.0°]. In the crystal structure, molecules are linked *via* O—H···O hydrogen bonds into centrosymmetric dimers, forming $R_2^2(22)$ rings (Bernstein, *et al.*, 1995). These dimers are, in turn, linked by weak C—H···O hydrogen bonds to form a two-dimensional network perpendicular to the *a* axis (Table 2 and Fig. 2).

Experimental

Synthesis of (1): in a round-bottomed flask equipped with a magnet and condenser, 4-bromo-1,8-naphthalic anhydride (5 mmol, 1.4 g) was dissolved in 1,4-dioxane (20 ml) and isopropylamine (4 ml) was added at room temperature. The solution was refluxed for 5 h, and the course of the reaction was monitored using TLC on silica gel with *n*-heptane-acetone (3:1) as eluent. The solvent was evaporated under vacuum and the product was separated by column chromatography on silica gel. Yield 72%; m.p. = 337–338 K.

Synthesis of (2): in a round-bottomed flask equipped with a magnet and condenser, 4-bromo-*N*-isopropyl-naphthalimide (1) (1 mmol, 0.317 g) was dissolved in 1,4-dioxane (20 cm³) at room temperature and then 2-(methylamino)-ethanol amine (5 ml) and triethylamine (5 ml) were added. The solution was refluxed and the course of the reaction was monitored using TLC on silica gel with *n*-heptane-acetone (3:1) as eluent. After 12 h, the reaction was completed and product (2) was isolated by pouring into water. After filtration, the resulting crystals were washed with water and recrystallized in ethanol. The crystals were dried under vacuum at 313 K. Yield 61%; mp 420–421 K.

Crystal data

C₁₈H₂₀N₂O₃
M_r = 312.36
 Monoclinic, *P*2₁/*c*
a = 13.6894 (4) Å
b = 11.2312 (8) Å
c = 10.2511 (9) Å
 β = 106.086 (4)°
V = 1514.38 (18) Å³
Z = 4

D_x = 1.370 Mg m⁻³
 Mo Kα radiation
 Cell parameters from 9870 reflections
 θ = 2.8–25.0°
 μ = 0.09 mm⁻¹
T = 150 (1) K
 Block, orange
 0.25 × 0.20 × 0.16 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
 φ scans and ω scans with κ offsets
 Absorption correction: none
 9870 measured reflections
 2660 independent reflections

1688 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.104
 θ_{max} = 25.0°
h = −16 → 16
k = −13 → 13
l = −12 → 11

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.054
wR(*F*²) = 0.161
S = 1.03
 2660 reflections
 215 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.0395P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δσ)_{max} = 0.002
 Δρ_{max} = 0.23 e Å⁻³
 Δρ_{min} = −0.36 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N1—C11	1.402 (3)	N2—C5	1.384 (3)
N1—C1	1.405 (3)	N2—C16	1.459 (3)
N1—C13	1.493 (3)	N2—C17	1.465 (3)
C11—N1—C1	123.3 (2)	C5—N2—C16	119.0 (2)
C11—N1—C13	118.9 (2)	C5—N2—C17	122.6 (2)
C1—N1—C13	117.75 (19)	C16—N2—C17	115.5 (2)

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O3-H3O\cdots O1^i$	0.92 (3)	1.92 (3)	2.816 (3)	166 (3)
$C7-H7A\cdots O3^{ii}$	0.95	2.42	3.162 (3)	135

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

H atoms bonded to C atoms were placed in calculated positions with C–H distances ranging from 0.95 to 1.00 Å. They were included in the refinement in riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms. The hydroxyl H atom (H3O) was refined independently with an isotropic displacement parameter.

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2001); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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