

Absorption correction: $R_{\text{int}} = 0.073$
 empirical (SADABS; $\theta_{\text{max}} = 30.00^\circ$
 Sheldrick, 1996a) $h = -14 \rightarrow 20$
 $T_{\text{min}} = 0.842$, $T_{\text{max}} = 0.996$ $k = -10 \rightarrow 10$
 8482 measured reflections $l = -20 \rightarrow 21$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0509P)^2$
 $R[F^2 > 2\sigma(F^2)] = 0.077$ $+ 0.2428P]$
 $wR(F^2) = 0.169$ where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.036$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 3390 reflections $\Delta\rho_{\text{max}} = 0.220 \text{ e } \text{\AA}^{-3}$
 199 parameters $\Delta\rho_{\text{min}} = -0.285 \text{ e } \text{\AA}^{-3}$
 All H-atom parameters Extinction correction: none
 refined Scattering factors from
International Tables for
Crystallography (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

S—C1	1.653 (3)	N3—C1	1.386 (3)
O1—N5	1.207 (4)	N3—C2	1.387 (4)
O2—N5	1.212 (4)	N3—N4	1.391 (3)
N1—C1	1.341 (4)	N4—C4	1.261 (4)
N1—N2	1.373 (4)	N5—C8	1.477 (4)
N2—C2	1.302 (4)		
C1—N3—N4	132.7 (2)	N1—C1—S	127.4 (2)
C4—N4—N3	119.5 (2)	N2—C2—C3	125.9 (3)
O1—N5—O2	123.1 (3)	N4—C4—C5	118.2 (3)
O1—N5—C8	118.1 (4)	C7—C8—N5	118.9 (3)
O2—N5—C8	118.8 (4)		

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—HN1...O1 ¹	0.88 (4)	2.38 (5)	3.066 (5)	136 (4)
N1—HN1...O2 ¹	0.88 (4)	2.42 (4)	3.290 (4)	170 (4)

Symmetry code: (i) $x - 1, y, z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω . The crystal-to-detector distance was 4 cm and the detector swing angle was -35° . Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The structure was solved by direct methods and refined by full-matrix least-squares procedures. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: SMART (Siemens, 1996a). Cell refinement: SAINT (Siemens, 1996b). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Sheldrick, 1996b). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL and PARST (Nardelli, 1995).

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2-Hydroxy-5-methyl-3-(morpholinomethyl)-benzaldehyde, (I), and 4,4'-dimethyl-6,6'-bis(morpholinomethyl)-2,2'-ethylenedinitrilodimethyldiphenol, (II)

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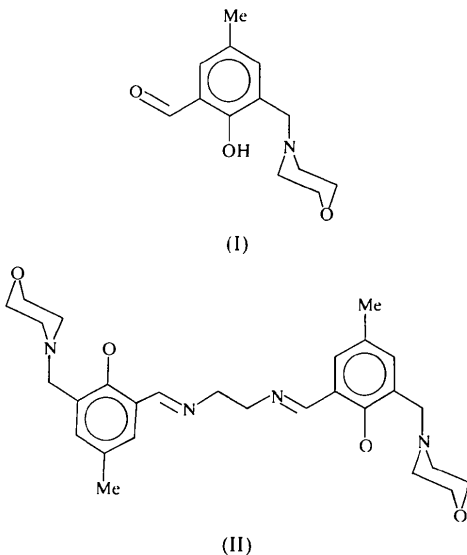
Abstract

The title compounds, (I) (C₁₃H₁₇NO₃) and (II) (C₂₈H₃₈N₄O₄), are in the monoclinic space group $P2_1/c$ with $Z = 4$ and 2, respectively. Molecule (II) has a crys-

tallographic centre of inversion. The morpholino ring adopts a chair conformation in both with equatorial substituents. Both molecules have an intramolecular O—H...N bond.

Comment

We are interested in the synthesis and structures of ligands which can yield binuclear metal complexes. Such



information is relevant to the preparation of metal complexes which are simple models for magnetically coupled binuclear metalloproteins (Kurtz, 1990; Soloman *et al.*, 1992). Two such ligands, (I) and (II), are presented here.

The morpholine ring in both the title compounds adopts the chair conformation with puckering amplitude $Q_T = 0.570(2)$ for (I) and $0.578(3)$ Å for (II), respectively. The best plane passes through the central C atoms leaving N and O atoms displaced at $-0.676(1)$ and $0.639(2)$ Å for (I), and $0.676(2)$ and $-0.6477(2)$ Å for (II), respectively. The phenyl ring makes a dihedral angle of $79.79(5)$ and $58.78(9)^\circ$ with the morpholino ring in (I) and (II), respectively. The bond lengths and bond angles are comparable with the literature values (Allen *et al.*, 1987).

In compound (I) the phenyloxy-H atom makes an intramolecular hydrogen bond with the morpholine ring,

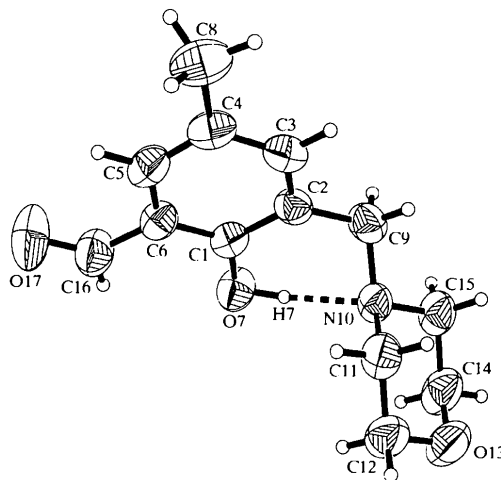


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids for non-H atoms. H atoms are shown as spheres of an arbitrary radius.

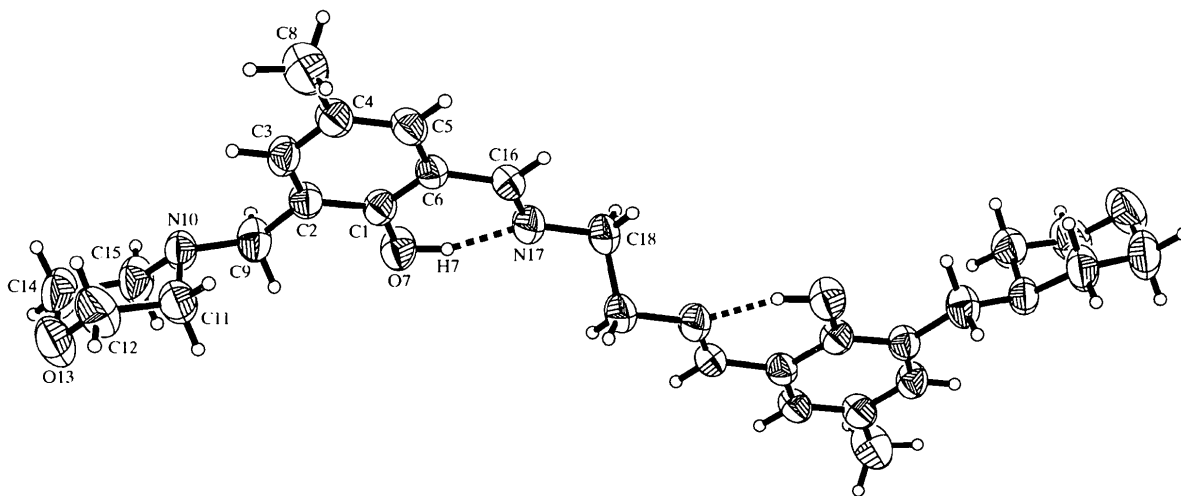


Fig. 2. The structure of (II) showing 50% probability displacement ellipsoids for non-H atoms. Symmetry-related atoms are not labelled. H atoms are shown as circles of an arbitrary radius.

while in compound (II) it is hydrogen bonded to the imino-N atom. The formyl group of (I) is involved in an intermolecular C—H···O hydrogen bond, but otherwise the structures are stabilized by van der Waals interactions.

Experimental

Compound (I) was synthesized by stirring paraformaldehyde (0.66 g, 0.022 mol) and morpholine (2.1 ml, 0.020 mol) in acetic acid overnight; 5-methylsalicylaldehyde (2.7 g, 0.019 mol) was then added and the solution stirred continuously for one day and then refluxed for 8 h. The resulting compound was neutralized with saturated Na₂CO₃ and extracted with CHCl₃. The CHCl₃ extract was evaporated and the product recrystallized from methanol by slow evaporation (m.p. 448 K, yield 70%).

Compound (II) was synthesized by adding 1,2-diaminoethane (0.7 ml, 0.01 mol) to compound (I) (4.70 g, 0.02 mol) in methanol (30 ml) whilst stirring and then refluxed for 2 h. The solvent was evaporated at room temperature and the product recrystallized from CHCl₃/MeOH by slow evaporation at room temperature (m.p. 466 K, yield = 70%).

Compound (I)

Crystal data

C₁₃H₁₇NO₃

M_r = 235.28

Monoclinic

*P*2₁/*c*

a = 5.3172 (1) Å

b = 15.3099 (3) Å

c = 15.3406 (1) Å

β = 91.846 (1)°

V = 1248.17 (3) Å³

Z = 4

D_x = 1.252 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 4735

reflections

θ = 1.37–33.22°

μ = 0.089 mm⁻¹

T = 293 (2) K

Parallelepiped

0.52 × 0.42 × 0.30 mm

Yellow

Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction: none

7859 measured reflections

2832 independent reflections

2094 reflections with

I > 2σ(*I*)

R_{int} = 0.021

θ_{max} = 27.49°

h = -6 → 6

k = 0 → 19

l = 0 → 19

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.049

wR(*F*²) = 0.149

S = 1.070

2832 reflections

159 parameters

H atoms: see text

w = 1/[σ²(*F_o*²) + (0.0663*P*)²

+ 0.2732*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.264 e Å⁻³

Δρ_{min} = -0.201 e Å⁻³

Extinction correction:

SHELXTL

Extinction coefficient:

0.0391 (52)

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °) for (I)

C1—O7	1.356 (2)	C12—O13	1.422 (2)
N10—C11	1.461 (2)	O13—C14	1.410 (2)
N10—C15	1.468 (2)	C14—C15	1.499 (3)
C11—C12	1.496 (3)	C16—O17	1.208 (2)
N10—C9—C2	111.91 (13)	O13—C12—C11	111.3 (2)
C11—N10—C15	108.55 (13)	C14—O13—C12	109.91 (14)
C11—N10—C9	111.65 (14)	O13—C14—C15	112.2 (2)
C15—N10—C9	111.60 (13)	N10—C15—C14	110.21 (15)
N10—C11—C12	109.8 (2)	O17—C16—C6	123.7 (2)
C15—N10—C11—C12	-57.9 (2)	C12—O13—C14—C15	57.2 (2)
N10—C11—C12—O13	59.7 (2)	C11—N10—C15—C14	56.5 (2)
C11—C12—O13—C14	-58.4 (2)	O13—C14—C15—N10	-57.2 (2)

Table 2. Hydrogen-bonding geometry (Å, °) for (I)

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
O7—H7···N10	0.91 (3)	1.81 (3)	2.655 (2)	153 (2)
C9—H9A···O17	0.97 (2)	2.43 (3)	3.381 (3)	165 (2)

Symmetry code: (i) -*x*, $\frac{1}{2}$ + *y*, $\frac{1}{2}$ - *z*.

Compound (II)

Crystal data

C₂₈H₃₈N₄O₄

M_r = 494.62

Monoclinic

*P*2₁/*c*

a = 11.1356 (5) Å

b = 9.5825 (5) Å

c = 12.4721 (6) Å

β = 98.828 (2)°

V = 1315.09 (11) Å³

Z = 2

D_x = 1.249 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 2700

reflections

θ = 1.37–33.22°

μ = 0.084 mm⁻¹

T = 293 (2) K

Block

0.40 × 0.26 × 0.18 mm

Yellow

Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction: none

8310 measured reflections

3017 independent reflections

1671 reflections with

I > 2σ(*I*)

R_{int} = 0.043

θ_{max} = 27.50°

h = -14 → 14

k = 0 → 12

l = 0 → 16

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.065

wR(*F*²) = 0.175

S = 1.037

3012 reflections

167 parameters

H atoms: see text

w = 1/[σ²(*F_o*²) + (0.0648*P*)²

+ 0.4623*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.219 e Å⁻³

Δρ_{min} = -0.207 e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 3. Selected geometric parameters (Å, °) for (II)

C1—O7	1.361 (3)	O13—C14	1.414 (4)
N10—C11	1.457 (3)	C14—C15	1.508 (4)
N10—C15	1.461 (3)	C16—N17	1.274 (3)
C11—C12	1.507 (4)	N17—C18	1.460 (3)
C12—O13	1.422 (4)	C18—C18'	1.519 (5)

C11—N10—C15	108.1 (2)	C14—O13—C12	109.3 (2)
C11—N10—C9	112.2 (2)	O13—C14—C15	112.5 (3)
C15—N10—C9	109.3 (2)	N10—C15—C14	110.6 (2)
N10—C11—C12	109.6 (2)	N17—C18—C18'	109.7 (2)
O13—C12—C11	110.4 (3)		
C15—N10—C11—C12	-59.1 (3)	C11—N10—C15—C14	55.9 (3)
N10—C11—C12—O13	62.1 (3)	O13—C14—C15—N10	-56.2 (3)
C11—C12—O13—C14	-59.6 (3)	C16—N17—C18—C18'	130.4 (3)
C12—O13—C14—C15	57.0 (3)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Table 4. Hydrogen-bonding geometry (\AA , $^\circ$) for (II)

D—H...A	D—H	H...A	D...A	D—H...A
O7—H7...N17	0.94 (4)	1.69 (4)	2.577 (3)	156 (3)

Each data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω . The crystal-to-detector distance was 4 cm and the detector swing angle was -35° . Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The structures were solved by direct methods and refined by full-matrix least-squares techniques. All the H atoms were geometrically fixed and allowed to ride on the parent C atoms except the phenyloxy-H atom (H7) which was located from the difference Fourier map and refined isotropically in both the compounds.

For both compounds, data collection: *SMART* (Siemens, 1996a); cell refinement: *SAINT* (Siemens, 1996b); data reduction: *SAINT*; program(s) used to solve structures: *SHELXTL* (Sheldrick, 1996); program(s) used to refine structures: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1231). Services for accessing these data are described at the back of the journal.

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5-Benzoyl-1-ethyl-4-phenyl-1H-pyrimidin-2-one

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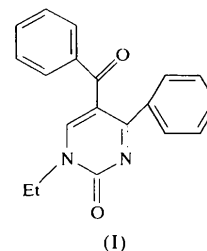
Abstract

In the title compound, $\text{C}_{19}\text{H}_{16}\text{N}_2\text{O}_2$, the pyrimidine ring is distorted from planarity. The dihedral angle between the two phenyl rings is $88.3(1)^\circ$. The two phenyl rings form dihedral angles of $114.6(1)$ and $141.2(1)^\circ$ with the pyrimidine ring.

Comment

Pyrimidines in general are of great biological and medicinal interest and for this reason their chemistry has been extensively investigated (Brown, 1984, 1985). In particular, various analogues of thiopyrimidines possess effective antibacterial, antifungal, antiviral, insecticidal and mitocidal activity (Sankyo Co., 1984; Akçamur *et al.*, 1988; Özbey *et al.*, 1991; Akkurt *et al.*, 1992; Akkurt & Hiller, 1993).

The bond lengths and bond angles observed in the title structure, (I), show normal values (Öztürk *et al.*, 1997). The pyrimidine ring is distorted from



planarity with a maximum deviation of $0.095(4) \text{\AA}$ for C2. The dihedral angle between the two phenyl rings is $88.3(1)^\circ$ and they form dihedral angles of $114.6(1)$ and $141.2(1)^\circ$ with the pyrimidine ring. The shortest intermolecular contact observed in the structure is $3.209(5) \text{\AA}$ between C1 and $\text{O1}(x, \frac{1}{2} - y, z - \frac{1}{2})$. The crystal structure is stabilized by van der Waals contacts.