

***N,N'*-Bis(2,5-dimethoxyphenyl)oxamide**Yong-Hong Wen, Kun Zhang,
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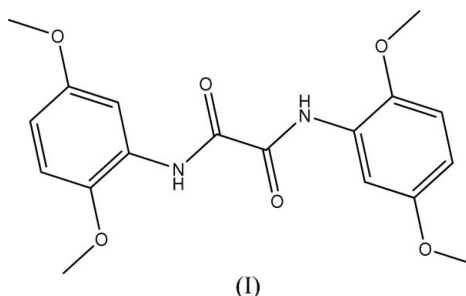
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Key indicatorsSingle-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.064
 wR factor = 0.109
Data-to-parameter ratio = 7.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_6$, has two independent molecules in the asymmetric unit. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds make the molecules essentially planar. The crystal packing is stabilized by $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions.

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In our ongoing studies of oxalamide derivatives, the title compound, (I), was obtained from the reaction of 2,5-dimethoxyaniline and oxalyl chloride.



The asymmetric unit of (I) consists of two crystallographically independent molecules *A* and *B* (Fig. 1). The bond lengths and angles in *A* and *B* agree with each other and are within normal ranges (Allen *et al.*, 1987). However, the $\text{C7}-\text{C8}$ [1.549 (7) Å] and $\text{C25}-\text{C26}$ [1.517 (8) Å] bond lengths are greater than the typical $\text{Csp}^2-\text{Csp}^2$ single bond distance (1.460 Å) and are comparable to that observed in *N,N'*-bis(2-ethoxyphenyl)oxalamide [1.535 (3) Å; Wen *et al.*, 2006]. Each independent molecule is almost planar, with the four methyl fragments oriented in opposite directions owing to the steric effect of the carbonyl groups. In each molecule, there exist intramolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1) which contribute to the planarity of the molecule.

In the crystal structure, molecules are linked into a three-dimensional network by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions. In Table 1, Cg1 and Cg2 denote the centroids of the $\text{C19}-\text{C24}$ and $\text{C27}-\text{C32}$ rings, respectively.

Experimental

To a solution of 2,5-dimethoxyaniline (30.6 g, 0.2 mol) in benzene (70 ml) was added dropwise a solution of oxalyl chloride (6.4 g, 0.05 mol) in benzene (30 ml), and the mixture was stirred at 343 K for 9 h. After cooling to room temperature, water (50 ml) was added to the reaction mixture and the organic phase was washed three times with water and dried at room temperature for 3 d to obtain (I) as a

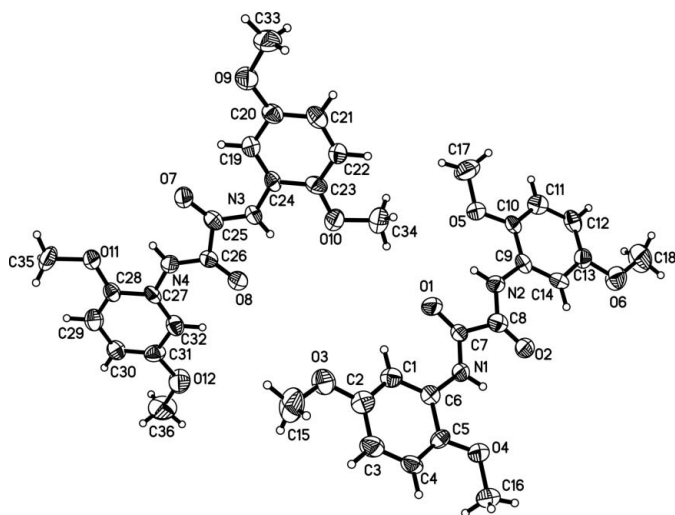


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atom numbering scheme.

solid. Single crystals of (I) suitable for X-ray diffraction study were obtained by slow evaporation of an *N,N*-dimethylformamide solution over a period of 24 h.

Crystal data

$C_{18}H_{20}N_2O_6$
 $M_r = 360.36$
Monoclinic, $P2_1$
 $a = 6.597$ (3) Å
 $b = 17.427$ (7) Å
 $c = 15.433$ (6) Å
 $\beta = 91.496$ (9)°
 $V = 1773.7$ (13) Å³

$Z = 4$
 $D_x = 1.350$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ (2) K
Needle, colourless
 $0.46 \times 0.06 \times 0.05$ mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.954$, $T_{\max} = 0.995$

10019 measured reflections
3611 independent reflections
1861 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.089$
 $\theta_{\text{max}} = 26.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.110$
 $S = 0.99$
3611 reflections
469 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.028P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O2	0.86	2.23	2.665 (7)	112
N1—H1A···O4	0.86	2.20	2.615 (6)	109
N2—H2A···O1	0.86	2.22	2.657 (6)	112
N2—H2A···O5	0.86	2.19	2.607 (6)	110
N3—H3A···O8	0.86	2.21	2.656 (6)	112
N3—H3A···O10	0.86	2.19	2.604 (7)	110
N4—H4A···O7	0.86	2.22	2.663 (6)	112
N4—H4A···O11	0.86	2.18	2.599 (6)	110
C1—H1···O1	0.93	2.34	2.936 (7)	122
C14—H14···O2	0.93	2.32	2.923 (7)	122
C16—H16A···O3 ⁱ	0.96	2.47	3.367 (8)	155
C18—H18A···O9 ⁱⁱ	0.96	2.60	3.302 (8)	131
C19—H19···O7	0.93	2.34	2.945 (7)	122
C32—H32···O8	0.93	2.34	2.938 (7)	121
C34—H34A···O1	0.96	2.52	3.243 (8)	132
C35—H35A···O12 ⁱⁱⁱ	0.96	2.31	3.228 (8)	160
C4—H4···Cg1 ⁱ	0.93	2.77	3.572	145
C12—H12···Cg2 ^{iv}	0.93	2.83	3.603	141

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

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H = 0.93 or 0.96 Å, N—H = 0.86 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ and $1.5U_{\text{eq}}(\text{methyl C})$. 3215 Friedel reflections were merged before the final refinement because of the absence of any significant anomalous scattering effects.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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