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(*E*)-4-Methyl-*N*-(2,3,4-trimethoxy-6-methylbenzylidene)aniline

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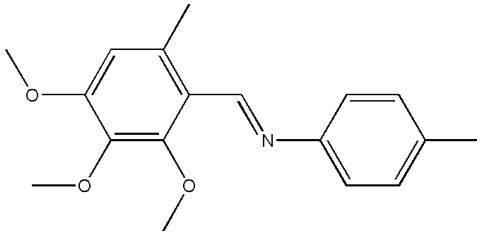
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.053; wR factor = 0.150; data-to-parameter ratio = 14.6.

In the title molecule, $C_{18}H_{21}NO_3$, the dihedral angle between the two benzene rings is 42.2 (2)° and it adopts a *trans* configuration with respect to the central C=N bond.

Related literature

For the structure of the related compound (*E*)-*N*-(2,3,4-trimethoxy-6-methylbenzylidene)naphthalen-1-amine, see: Wang (2009).



Experimental

Crystal data

$C_{18}H_{21}NO_3$
M_r = 299.36
Monoclinic, $P2_1/c$
a = 7.7239 (9) Å
b = 27.287 (2) Å
c = 8.4128 (11) Å
β = 111.529 (2)°
V = 1649.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.45 × 0.43 × 0.40 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.968
8258 measured reflections
2899 independent reflections
1475 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.150
S = 1.02
2899 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2770).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, C.-Y. (2009). *Acta Cryst.* E**65**, o56.

supporting information

Acta Cryst. (2009). E**65**, o493 [doi:10.1107/S1600536809004115]

(*E*)-4-Methyl-*N*-(2,3,4-trimethoxy-6-methylbenzylidene)aniline

Cheng-Yun Wang

S1. Comment

The preparation, properties and applications of Schiff bases are important in the development of coordination chemistry. In this paper, the structure of the title compound, (I), is reported. The molecular structure of (I) is illustrated in Fig. 1. The bond lengths and angles of the title compound agree with those in the related compound (*E*)-*N*-(2,3,4-trimethoxy-6-methylbenzylidene)naphthalen-1-amine (Wang, 2009), as representative example. The dihedral angle between between the two phenyl rings is 137.8 (2) °. The molecule adopts a *trans* configuration about the central C=N functional bond. In the crystal structure, molecules pack in a 'herring-bone′ fashion along the b axis direction (see fig. 2).

S2. Experimental

A mixture of *p*-toluidine (0.535 g, 5 mmol) and 2,3,4-trimethoxy-6-methylbenzaldehyde (1.04 g, 5 mmol) in ethyl alcohol (20 ml) was stirred magnetically for 2 h at reflux temperature. After cooling the precipitate was filtered and dried. The crude product of 20 mg was dissolved in a 20 ml of ethylalcohol by heating on a magnetic stirrer. The solution was filtered to remove impurities, and then left to crystallize at room temperature. After a week single crystals suitable for the X-ray crystal structure determination were obtained.

S3. Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5 U_{eq}(methyl C).

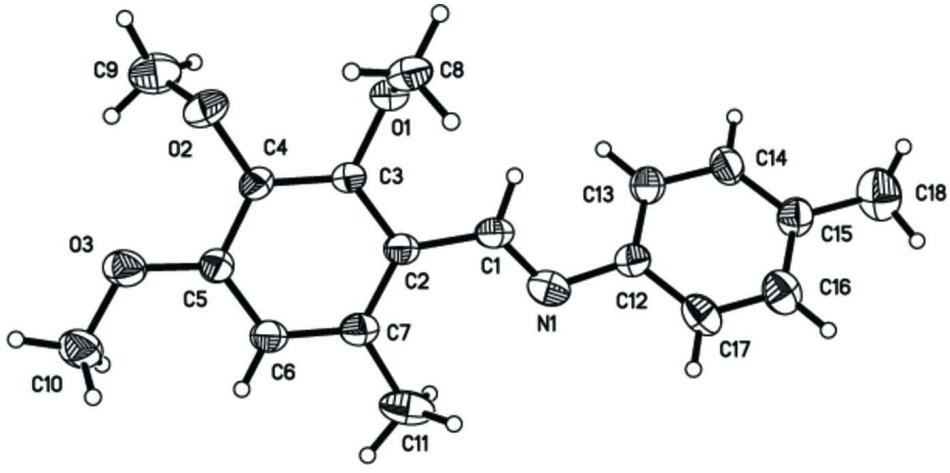


Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids.

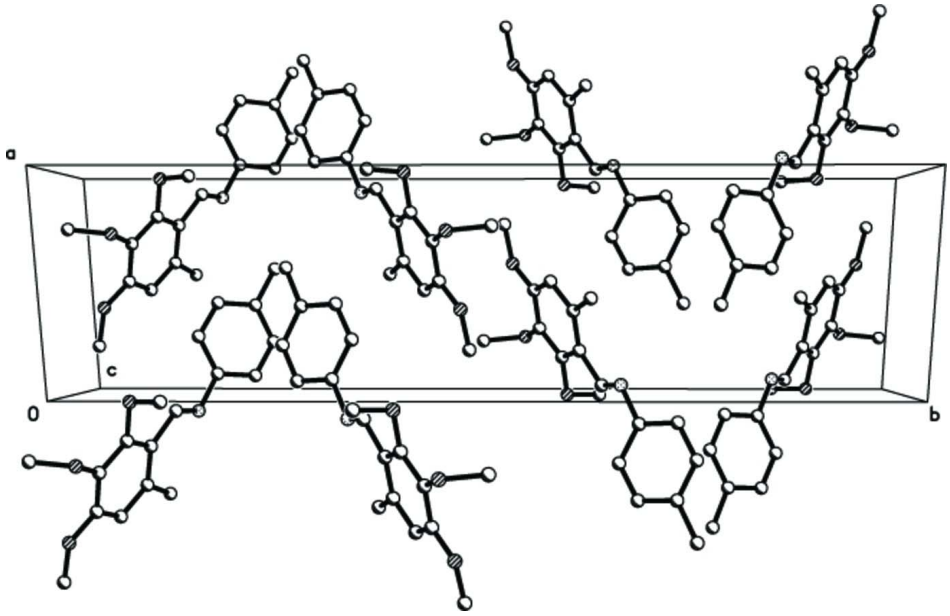


Figure 2

Part of the crystal structure of (I).

(*E*)-4-Methyl-*N*-(2,3,4-trimethoxy-6-methylbenzylidene)aniline

Crystal data

$C_{18}H_{21}NO_3$
M_r = 299.36
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.7239 (9) Å
b = 27.287 (2) Å
c = 8.4128 (11) Å
β = 111.529 (2)°
V = 1649.4 (3) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.206 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1422 reflections
θ = 2.7–20.0°
μ = 0.08 mm^{-1}
T = 298 K
Block, brown
0.45 × 0.43 × 0.40 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.968
8258 measured reflections
2899 independent reflections
1475 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 25.0°, θ_{min} = 1.5°
h = −9→9
k = −30→32
l = −9→5

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.150
S = 1.02
2899 reflections
199 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.6503P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.9325 (3)	0.15463 (10)	0.3804 (4)	0.0607 (7)
O1	0.9997 (3)	0.09590 (7)	−0.0287 (2)	0.0536 (6)
O2	0.7379 (3)	0.04533 (8)	−0.2839 (3)	0.0628 (6)
O3	0.3972 (3)	0.02973 (8)	−0.2724 (3)	0.0640 (6)
C1	0.9412 (4)	0.13395 (11)	0.2492 (4)	0.0520 (8)
H1	1.0549	0.1351	0.2348	0.062*
C2	0.7898 (4)	0.10855 (10)	0.1187 (4)	0.0448 (7)
C3	0.8252 (4)	0.08912 (10)	−0.0207 (4)	0.0451 (7)
C4	0.6955 (4)	0.06230 (10)	−0.1484 (4)	0.0466 (7)
C5	0.5201 (4)	0.05545 (10)	−0.1404 (4)	0.0481 (8)
C6	0.4808 (4)	0.07464 (11)	−0.0057 (4)	0.0506 (8)
H6	0.3632	0.0698	−0.0019	0.061*
C7	0.6114 (4)	0.10096 (10)	0.1238 (4)	0.0512 (8)
C8	1.0063 (5)	0.13609 (12)	−0.1356 (5)	0.0742 (11)
H8A	0.9214	0.1302	−0.2502	0.111*
H8B	1.1303	0.1393	−0.1350	0.111*
H8C	0.9717	0.1657	−0.0933	0.111*
C9	0.7579 (5)	−0.00633 (14)	−0.2860 (5)	0.0790 (11)
H9A	0.8493	−0.0169	−0.1793	0.119*
H9B	0.7973	−0.0154	−0.3777	0.119*
H9C	0.6408	−0.0217	−0.3023	0.119*
C10	0.2178 (4)	0.01986 (14)	−0.2686 (4)	0.0737 (11)
H10A	0.2309	0.0048	−0.1617	0.111*
H10B	0.1515	−0.0018	−0.3607	0.111*
H10C	0.1499	0.0500	−0.2808	0.111*
C11	0.5545 (5)	0.11989 (13)	0.2664 (5)	0.0774 (11)
H11A	0.4251	0.1131	0.2402	0.116*
H11B	0.5749	0.1546	0.2779	0.116*
H11C	0.6275	0.1040	0.3715	0.116*
C12	1.0977 (4)	0.17428 (11)	0.5029 (4)	0.0501 (8)

C13	1.2692 (4)	0.15170 (11)	0.5499 (4)	0.0566 (9)
H13	1.2816	0.1228	0.4965	0.068*
C14	1.4218 (4)	0.17184 (12)	0.6755 (4)	0.0623 (9)
H14	1.5363	0.1562	0.7045	0.075*
C15	1.4108 (4)	0.21420 (12)	0.7595 (4)	0.0596 (9)
C16	1.2394 (5)	0.23596 (12)	0.7127 (4)	0.0678 (10)
H16	1.2272	0.2647	0.7668	0.081*
C17	1.0854 (5)	0.21645 (12)	0.5881 (4)	0.0653 (10)
H17	0.9708	0.2319	0.5607	0.078*
C18	1.5798 (5)	0.23466 (14)	0.8988 (5)	0.0950 (13)
H18A	1.6665	0.2465	0.8501	0.143*
H18B	1.6375	0.2094	0.9806	0.143*
H18C	1.5431	0.2612	0.9546	0.143*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0504 (16)	0.0693 (19)	0.0638 (19)	−0.0076 (14)	0.0229 (15)	−0.0111 (15)
O1	0.0471 (12)	0.0616 (14)	0.0591 (14)	−0.0011 (10)	0.0279 (10)	0.0078 (11)
O2	0.0767 (15)	0.0720 (16)	0.0501 (14)	−0.0086 (12)	0.0355 (12)	−0.0009 (11)
O3	0.0516 (13)	0.0881 (17)	0.0500 (14)	−0.0170 (12)	0.0159 (11)	−0.0071 (12)
C1	0.0486 (19)	0.0546 (19)	0.061 (2)	−0.0062 (15)	0.0293 (17)	0.0001 (17)
C2	0.0424 (17)	0.0433 (17)	0.0525 (19)	−0.0031 (14)	0.0220 (15)	−0.0009 (14)
C3	0.0414 (17)	0.0468 (18)	0.0517 (19)	−0.0009 (14)	0.0225 (15)	0.0067 (15)
C4	0.0497 (19)	0.0529 (19)	0.0403 (18)	−0.0016 (15)	0.0200 (15)	0.0060 (15)
C5	0.0437 (18)	0.0512 (19)	0.0470 (19)	−0.0039 (15)	0.0140 (15)	0.0036 (15)
C6	0.0449 (18)	0.0546 (19)	0.056 (2)	−0.0047 (15)	0.0224 (16)	0.0009 (16)
C7	0.0529 (19)	0.0495 (19)	0.059 (2)	−0.0036 (15)	0.0292 (17)	−0.0030 (16)
C8	0.068 (2)	0.075 (2)	0.095 (3)	−0.0027 (19)	0.048 (2)	0.023 (2)
C9	0.090 (3)	0.081 (3)	0.082 (3)	−0.008 (2)	0.051 (2)	−0.016 (2)
C10	0.053 (2)	0.099 (3)	0.063 (2)	−0.0190 (19)	0.0128 (18)	−0.003 (2)
C11	0.067 (2)	0.088 (3)	0.095 (3)	−0.021 (2)	0.052 (2)	−0.033 (2)
C12	0.0501 (19)	0.0525 (19)	0.0501 (19)	−0.0024 (16)	0.0212 (16)	0.0005 (16)
C13	0.062 (2)	0.0442 (19)	0.067 (2)	0.0038 (17)	0.0269 (19)	−0.0008 (16)
C14	0.049 (2)	0.059 (2)	0.072 (2)	0.0071 (17)	0.0150 (18)	0.0057 (19)
C15	0.059 (2)	0.051 (2)	0.061 (2)	−0.0044 (17)	0.0131 (18)	−0.0014 (17)
C16	0.068 (2)	0.056 (2)	0.075 (2)	0.0058 (19)	0.021 (2)	−0.0140 (18)
C17	0.052 (2)	0.066 (2)	0.076 (3)	0.0088 (17)	0.0201 (19)	−0.0124 (19)
C18	0.077 (3)	0.087 (3)	0.091 (3)	−0.009 (2)	−0.005 (2)	−0.012 (2)

Geometric parameters (Å, º)

N1—C1	1.263 (3)	C9—H9B	0.9600
N1—C12	1.419 (4)	C9—H9C	0.9600
O1—C3	1.386 (3)	C10—H10A	0.9600
O1—C8	1.431 (3)	C10—H10B	0.9600
O2—C4	1.377 (3)	C10—H10C	0.9600
O2—C9	1.419 (4)	C11—H11A	0.9600

O3—C5	1.361 (3)	C11—H11B	0.9600
O3—C10	1.423 (3)	C11—H11C	0.9600
C1—C2	1.453 (4)	C12—C17	1.377 (4)
C1—H1	0.9300	C12—C13	1.380 (4)
C2—C3	1.402 (4)	C13—C14	1.376 (4)
C2—C7	1.409 (4)	C13—H13	0.9300
C3—C4	1.380 (4)	C14—C15	1.373 (4)
C4—C5	1.393 (4)	C14—H14	0.9300
C5—C6	1.380 (4)	C15—C16	1.370 (4)
C6—C7	1.384 (4)	C15—C18	1.505 (4)
C6—H6	0.9300	C16—C17	1.372 (4)
C7—C11	1.512 (4)	C16—H16	0.9300
C8—H8A	0.9600	C17—H17	0.9300
C8—H8B	0.9600	C18—H18A	0.9600
C8—H8C	0.9600	C18—H18B	0.9600
C9—H9A	0.9600	C18—H18C	0.9600

C1—N1—C12	118.8 (3)	O3—C10—H10A	109.5
C3—O1—C8	113.1 (2)	O3—C10—H10B	109.5
C4—O2—C9	113.8 (2)	H10A—C10—H10B	109.5
C5—O3—C10	118.2 (2)	O3—C10—H10C	109.5
N1—C1—C2	125.9 (3)	H10A—C10—H10C	109.5
N1—C1—H1	117.0	H10B—C10—H10C	109.5
C2—C1—H1	117.0	C7—C11—H11A	109.5
C3—C2—C7	117.6 (3)	C7—C11—H11B	109.5
C3—C2—C1	117.5 (3)	H11A—C11—H11B	109.5
C7—C2—C1	124.9 (3)	C7—C11—H11C	109.5
C4—C3—O1	118.3 (3)	H11A—C11—H11C	109.5
C4—C3—C2	122.9 (3)	H11B—C11—H11C	109.5
O1—C3—C2	118.8 (3)	C17—C12—C13	117.8 (3)
O2—C4—C3	119.9 (3)	C17—C12—N1	118.6 (3)
O2—C4—C5	121.7 (3)	C13—C12—N1	123.5 (3)
C3—C4—C5	118.3 (3)	C14—C13—C12	120.2 (3)
O3—C5—C6	124.6 (3)	C14—C13—H13	119.9
O3—C5—C4	115.4 (3)	C12—C13—H13	119.9
C6—C5—C4	120.0 (3)	C15—C14—C13	122.3 (3)
C5—C6—C7	121.8 (3)	C15—C14—H14	118.9
C5—C6—H6	119.1	C13—C14—H14	118.9
C7—C6—H6	119.1	C16—C15—C14	116.9 (3)
C6—C7—C2	119.4 (3)	C16—C15—C18	122.2 (3)
C6—C7—C11	117.5 (3)	C14—C15—C18	120.9 (3)
C2—C7—C11	123.1 (3)	C15—C16—C17	121.8 (3)
O1—C8—H8A	109.5	C15—C16—H16	119.1
O1—C8—H8B	109.5	C17—C16—H16	119.1
H8A—C8—H8B	109.5	C16—C17—C12	121.0 (3)
O1—C8—H8C	109.5	C16—C17—H17	119.5
H8A—C8—H8C	109.5	C12—C17—H17	119.5
H8B—C8—H8C	109.5	C15—C18—H18A	109.5

O2—C9—H9A	109.5	C15—C18—H18B	109.5
O2—C9—H9B	109.5	H18A—C18—H18B	109.5
H9A—C9—H9B	109.5	C15—C18—H18C	109.5
O2—C9—H9C	109.5	H18A—C18—H18C	109.5
H9A—C9—H9C	109.5	H18B—C18—H18C	109.5
H9B—C9—H9C	109.5		

C12—N1—C1—C2	174.7 (3)	O3—C5—C6—C7	−179.3 (3)
N1—C1—C2—C3	178.1 (3)	C4—C5—C6—C7	0.1 (4)
N1—C1—C2—C7	−3.5 (5)	C5—C6—C7—C2	0.1 (4)
C8—O1—C3—C4	83.9 (3)	C5—C6—C7—C11	−179.3 (3)
C8—O1—C3—C2	−97.8 (3)	C3—C2—C7—C6	0.7 (4)
C7—C2—C3—C4	−1.6 (4)	C1—C2—C7—C6	−177.7 (3)
C1—C2—C3—C4	176.9 (3)	C3—C2—C7—C11	−180.0 (3)
C7—C2—C3—O1	−179.8 (2)	C1—C2—C7—C11	1.6 (5)
C1—C2—C3—O1	−1.3 (4)	C1—N1—C12—C17	145.4 (3)
C9—O2—C4—C3	110.9 (3)	C1—N1—C12—C13	−39.0 (4)
C9—O2—C4—C5	−72.6 (3)	C17—C12—C13—C14	−1.3 (5)
O1—C3—C4—O2	−3.4 (4)	N1—C12—C13—C14	−177.0 (3)
C2—C3—C4—O2	178.4 (3)	C12—C13—C14—C15	0.6 (5)
O1—C3—C4—C5	180.0 (2)	C13—C14—C15—C16	0.0 (5)
C2—C3—C4—C5	1.8 (4)	C13—C14—C15—C18	178.7 (3)
C10—O3—C5—C6	−2.9 (4)	C14—C15—C16—C17	0.2 (5)
C10—O3—C5—C4	177.7 (3)	C18—C15—C16—C17	−178.5 (3)
O2—C4—C5—O3	1.9 (4)	C15—C16—C17—C12	−0.9 (5)
C3—C4—C5—O3	178.4 (2)	C13—C12—C17—C16	1.5 (5)
O2—C4—C5—C6	−177.5 (3)	N1—C12—C17—C16	177.4 (3)
C3—C4—C5—C6	−1.0 (4)		