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3,6-Diacetyl-1,4-diphenyl-1,4-dihydro-1,2,4,5-tetrazine

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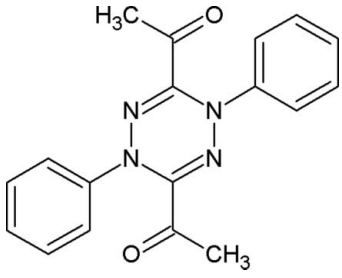
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.108; data-to-parameter ratio = 13.6.

In the title compound, $C_{18}H_{16}N_4O_2$, the central six-membered ring has a boat conformation.

Related literature

For background, see: Sauer (1996). For further synthetic details, see: Al-Noaimi *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{16}N_4O_2$
M_r = 320.35
Monoclinic, $P2_1/c$
a = 11.7853 (6) Å
b = 14.7217 (7) Å
c = 9.5113 (4) Å
β = 92.350 (1)°
V = 1648.82 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.28 × 0.23 × 0.12 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.973, T_{max} = 0.989
24032 measured reflections
2985 independent reflections
2090 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.02
2985 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *XS* in *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *XL* in *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *XCIF* in *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2696).

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supplementary materials

Acta Cryst. (2008). E**64**, o551 [**doi:10.1107/S1600536808003115**]

3,6-Diacetyl-1,4-diphenyl-1,4-dihydro-1,2,4,5-tetrazine

M. Al-Noaimi and S. F. Haddad

Comment

1,2,4,5-Tetrazine derivatives have high potential for biological activity, possessing a wide spectrum of antiviral and antitumor properties. They have been widely used in pesticides and herbicides (Sauer, 1996).

As part of our studies in this area, the title compound, (I), was prepared and its structure determined by X-ray diffraction (Fig. 1).

In (I), atoms N1, C3, N4 and C6 are coplanar [r.m.s. deviation = 0.010 Å] and atoms N2 and N5 deviate from the plane by 0.457 and 0.451 Å, respectively. Thus, the central tetrazine ring in (I) has a boat conformation.

The dihedral angle between the plane defined by atoms N1 C3 N4 C6 and the C11—C16 benzene ring plane is 28.27 (9)°. The dihedral angle between the same atoms and the C17—C22 benzene ring is 40.11 (5)°. The dihedral angle between the C11—C16 and C17—C22 benzene rings is 66.19 (9)°.

Experimental

A solution of 1-phenylhydrazono-1-chloropropanone (10 g, 55 mmol) (Al-Noaimi *et al.*, 2007) and triethylamine (7.2 g, 71 mmol) in ethanol (80 ml) was refluxed for 2 h, and then the solvent was partially removed under reduced pressure. A solution of the compound in ethanol was concentrated gradually at room temperature to afford red blocks of (I) (m.p. 439–441 K).

Refinement

The H atoms were geometrically placed (C—H = 0.93–0.97 Å) and refined as riding with with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

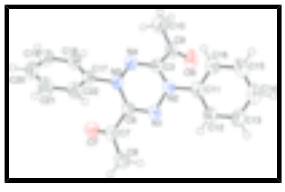


Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

3,6-Diacetyl-1,4-diphenyl-1,4-dihydro-1,2,4,5-tetrazine

Crystal data

$C_{18}H_{16}N_4O_2$ F_{000} = 672

M_r = 320.35	D_x = 1.291 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4916 reflections
a = 11.7853 (6) Å	θ = 2.2–23.9º
b = 14.7217 (7) Å	μ = 0.09 mm^{-1}
c = 9.5113 (4) Å	T = 298 (2) K
β = 92.350 (1)º	Block, red
V = 1648.82 (13) Å^3	0.28 × 0.23 × 0.12 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	2985 independent reflections
Radiation source: normal-focus sealed tube	2090 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
Detector resolution: 8.3 pixels mm^{-1}	θ_{max} = 25.3º
T = 298(2) K	θ_{min} = 1.7º
ω scans	h = −14→14
Absorption correction: multi-scan (SADABS; Bruker, 2004)	k = −17→17
T_{min} = 0.973, T_{max} = 0.989	l = −11→11
24032 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.1852P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
2985 reflections	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
219 parameters	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
C3	0.19860 (13)	0.04264 (11)	0.24262 (17)	0.0476 (4)
C6	0.06963 (13)	0.17718 (11)	0.30979 (16)	0.0443 (4)
C7	0.01584 (14)	0.23941 (12)	0.41383 (18)	0.0485 (4)
C8	0.03913 (16)	0.33770 (12)	0.3984 (2)	0.0615 (5)
H8A	0.0063	0.3704	0.4740	0.092*
H8B	0.0065	0.3588	0.3102	0.092*
H8C	0.1197	0.3476	0.4009	0.092*
C9	0.29164 (15)	−0.02517 (13)	0.27788 (19)	0.0566 (5)
C10	0.25803 (17)	−0.12250 (14)	0.2816 (3)	0.0861 (7)
H10A	0.3228	−0.1588	0.3093	0.129*
H10B	0.2298	−0.1409	0.1899	0.129*
H10C	0.1997	−0.1306	0.3481	0.129*
C11	0.31755 (13)	0.15724 (12)	0.12166 (17)	0.0479 (4)
C12	0.36333 (14)	0.24291 (13)	0.14032 (19)	0.0567 (5)
H12	0.3338	0.2825	0.2057	0.068*
C13	0.45313 (16)	0.26945 (15)	0.0613 (2)	0.0681 (5)
H13	0.4833	0.3275	0.0726	0.082*
C14	0.49833 (16)	0.21120 (16)	−0.0338 (2)	0.0707 (6)
H14	0.5600	0.2291	−0.0852	0.085*
C15	0.45218 (16)	0.12619 (15)	−0.0529 (2)	0.0666 (5)
H15	0.4831	0.0865	−0.1171	0.080*
C16	0.36055 (15)	0.09929 (13)	0.02212 (19)	0.0582 (5)
H16	0.3277	0.0426	0.0061	0.070*
C17	−0.09434 (13)	0.07208 (11)	0.28912 (17)	0.0466 (4)
C18	−0.12547 (14)	−0.00429 (12)	0.36200 (18)	0.0533 (5)
H18	−0.0705	−0.0437	0.3996	0.064*
C19	−0.23938 (16)	−0.02164 (14)	0.3785 (2)	0.0660 (5)
H19	−0.2611	−0.0730	0.4277	0.079*
C20	−0.32094 (16)	0.03634 (17)	0.3228 (2)	0.0744 (6)
H20	−0.3974	0.0246	0.3351	0.089*
C21	−0.28909 (16)	0.11175 (16)	0.2489 (2)	0.0730 (6)
H21	−0.3443	0.1509	0.2112	0.088*
C22	−0.17587 (15)	0.12967 (13)	0.2303 (2)	0.0603 (5)
H22	−0.1545	0.1801	0.1787	0.072*
N1	0.16981 (11)	0.19907 (9)	0.27549 (14)	0.0495 (4)
N2	0.22031 (11)	0.13056 (9)	0.19490 (14)	0.0496 (4)
N4	0.09917 (11)	0.02070 (9)	0.28117 (14)	0.0489 (4)
N5	0.02219 (10)	0.09373 (9)	0.26874 (14)	0.0460 (4)
O7	−0.04003 (12)	0.20784 (9)	0.50509 (14)	0.0728 (4)
O9	0.38657 (10)	0.00113 (9)	0.30439 (15)	0.0717 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.0384 (9)	0.0512 (11)	0.0535 (10)	0.0003 (8)	0.0079 (7)	−0.0038 (8)
C6	0.0394 (9)	0.0460 (10)	0.0481 (10)	0.0020 (7)	0.0072 (7)	0.0017 (7)
C7	0.0423 (9)	0.0533 (11)	0.0504 (10)	0.0028 (8)	0.0069 (8)	−0.0011 (8)
C8	0.0665 (12)	0.0545 (12)	0.0640 (12)	0.0037 (9)	0.0095 (9)	−0.0061 (9)
C9	0.0407 (10)	0.0647 (12)	0.0653 (12)	0.0063 (9)	0.0125 (8)	0.0026 (9)
C10	0.0592 (13)	0.0587 (13)	0.141 (2)	0.0137 (10)	0.0099 (13)	0.0077 (13)
C11	0.0369 (9)	0.0583 (11)	0.0488 (10)	0.0014 (8)	0.0060 (7)	0.0026 (8)
C12	0.0455 (10)	0.0626 (12)	0.0625 (12)	−0.0040 (9)	0.0093 (8)	−0.0045 (9)
C13	0.0515 (12)	0.0737 (14)	0.0797 (14)	−0.0133 (10)	0.0126 (10)	0.0048 (11)
C14	0.0479 (11)	0.0961 (17)	0.0691 (14)	−0.0026 (11)	0.0165 (10)	0.0149 (12)
C15	0.0573 (12)	0.0870 (16)	0.0567 (12)	0.0107 (11)	0.0176 (9)	0.0008 (10)
C16	0.0536 (11)	0.0616 (12)	0.0604 (12)	0.0014 (9)	0.0136 (9)	−0.0035 (9)
C17	0.0373 (9)	0.0525 (10)	0.0505 (10)	0.0004 (7)	0.0088 (7)	−0.0066 (8)
C18	0.0476 (10)	0.0567 (11)	0.0563 (11)	−0.0033 (8)	0.0101 (8)	−0.0060 (9)
C19	0.0550 (12)	0.0711 (14)	0.0732 (13)	−0.0173 (10)	0.0170 (10)	−0.0112 (10)
C20	0.0398 (11)	0.0975 (17)	0.0867 (15)	−0.0139 (11)	0.0098 (10)	−0.0248 (13)
C21	0.0415 (11)	0.0894 (16)	0.0875 (15)	0.0130 (10)	−0.0023 (10)	−0.0096 (12)
C22	0.0443 (11)	0.0639 (12)	0.0728 (13)	0.0083 (9)	0.0045 (9)	0.0018 (10)
N1	0.0442 (8)	0.0493 (8)	0.0560 (9)	0.0011 (6)	0.0128 (6)	−0.0039 (7)
N2	0.0406 (8)	0.0492 (8)	0.0601 (9)	0.0001 (6)	0.0163 (6)	−0.0047 (7)
N4	0.0373 (8)	0.0484 (8)	0.0615 (9)	0.0036 (6)	0.0076 (6)	−0.0027 (7)
N5	0.0344 (7)	0.0457 (8)	0.0585 (9)	0.0038 (6)	0.0088 (6)	−0.0009 (7)
O7	0.0844 (10)	0.0649 (9)	0.0717 (9)	−0.0061 (7)	0.0377 (8)	−0.0061 (7)
O9	0.0402 (8)	0.0865 (10)	0.0887 (10)	0.0030 (7)	0.0061 (7)	0.0111 (8)

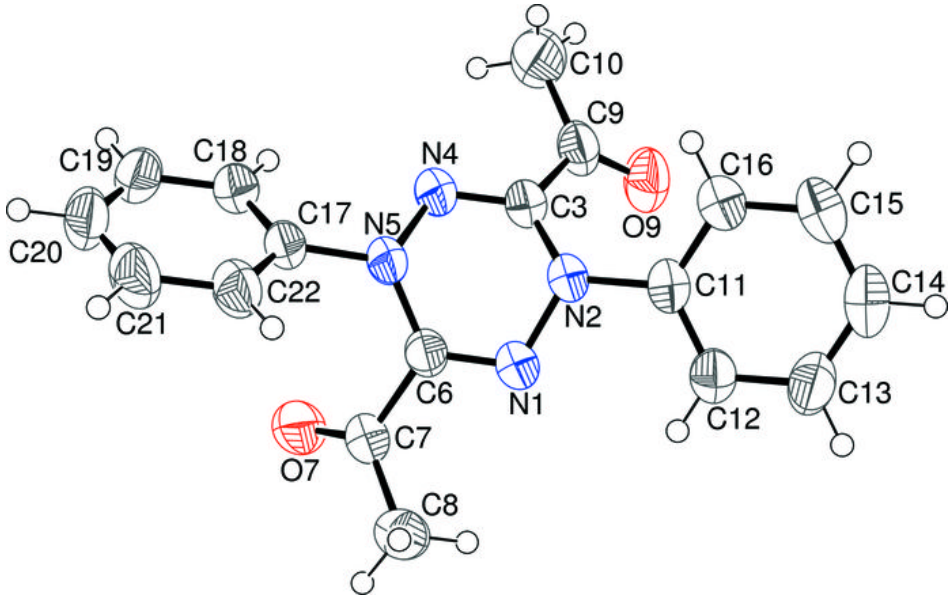
Geometric parameters (Å, °)

C3—N4	1.2834 (19)	C13—C14	1.370 (3)
C3—N2	1.399 (2)	C13—H13	0.9300
C3—C9	1.510 (2)	C14—C15	1.374 (3)
C6—N1	1.2790 (19)	C14—H14	0.9300
C6—N5	1.399 (2)	C15—C16	1.377 (2)
C6—C7	1.507 (2)	C15—H15	0.9300
C7—O7	1.2041 (19)	C16—H16	0.9300
C7—C8	1.481 (2)	C17—C18	1.378 (2)
C8—H8A	0.9600	C17—C22	1.382 (2)
C8—H8B	0.9600	C17—N5	1.431 (2)
C8—H8C	0.9600	C18—C19	1.382 (2)
C9—O9	1.201 (2)	C18—H18	0.9300
C9—C10	1.487 (3)	C19—C20	1.375 (3)
C10—H10A	0.9600	C19—H19	0.9300
C10—H10B	0.9600	C20—C21	1.375 (3)
C10—H10C	0.9600	C20—H20	0.9300
C11—C12	1.380 (2)	C21—C22	1.379 (3)
C11—C16	1.386 (2)	C21—H21	0.9300

C11—N2	1.4207 (19)	C22—H22	0.9300
C12—C13	1.380 (2)	N1—N2	1.4130 (18)
C12—H12	0.9300	N4—N5	1.4086 (17)
N4—C3—N2	120.46 (15)	C13—C14—H14	120.2
N4—C3—C9	115.67 (15)	C15—C14—H14	120.2
N2—C3—C9	122.88 (14)	C14—C15—C16	120.54 (19)
N1—C6—N5	120.85 (14)	C14—C15—H15	119.7
N1—C6—C7	115.58 (15)	C16—C15—H15	119.7
N5—C6—C7	122.89 (14)	C15—C16—C11	119.58 (18)
O7—C7—C8	123.90 (16)	C15—C16—H16	120.2
O7—C7—C6	119.72 (16)	C11—C16—H16	120.2
C8—C7—C6	116.35 (15)	C18—C17—C22	120.59 (16)
C7—C8—H8A	109.5	C18—C17—N5	121.77 (15)
C7—C8—H8B	109.5	C22—C17—N5	117.63 (15)
H8A—C8—H8B	109.5	C17—C18—C19	119.19 (18)
C7—C8—H8C	109.5	C17—C18—H18	120.4
H8A—C8—H8C	109.5	C19—C18—H18	120.4
H8B—C8—H8C	109.5	C20—C19—C18	120.6 (2)
O9—C9—C10	123.52 (17)	C20—C19—H19	119.7
O9—C9—C3	119.68 (17)	C18—C19—H19	119.7
C10—C9—C3	116.77 (16)	C21—C20—C19	119.81 (18)
C9—C10—H10A	109.5	C21—C20—H20	120.1
C9—C10—H10B	109.5	C19—C20—H20	120.1
H10A—C10—H10B	109.5	C20—C21—C22	120.40 (19)
C9—C10—H10C	109.5	C20—C21—H21	119.8
H10A—C10—H10C	109.5	C22—C21—H21	119.8
H10B—C10—H10C	109.5	C21—C22—C17	119.41 (19)
C12—C11—C16	119.90 (16)	C21—C22—H22	120.3
C12—C11—N2	120.56 (15)	C17—C22—H22	120.3
C16—C11—N2	119.32 (15)	C6—N1—N2	111.85 (13)
C13—C12—C11	119.55 (17)	C3—N2—N1	113.44 (12)
C13—C12—H12	120.2	C3—N2—C11	125.24 (13)
C11—C12—H12	120.2	N1—N2—C11	115.77 (13)
C14—C13—C12	120.68 (19)	C3—N4—N5	112.11 (13)
C14—C13—H13	119.7	C6—N5—N4	113.47 (12)
C12—C13—H13	119.7	C6—N5—C17	122.19 (13)
C13—C14—C15	119.68 (18)	N4—N5—C17	115.92 (13)
N1—C6—C7—O7	−140.54 (17)	N5—C6—N1—N2	0.3 (2)
N5—C6—C7—O7	30.1 (2)	C7—C6—N1—N2	171.13 (13)
N1—C6—C7—C8	37.6 (2)	N4—C3—N2—N1	42.1 (2)
N5—C6—C7—C8	−151.76 (15)	C9—C3—N2—N1	−126.06 (16)
N4—C3—C9—O9	−147.50 (17)	N4—C3—N2—C11	−165.46 (15)
N2—C3—C9—O9	21.2 (3)	C9—C3—N2—C11	26.4 (2)
N4—C3—C9—C10	30.7 (2)	C6—N1—N2—C3	−40.54 (19)
N2—C3—C9—C10	−160.58 (17)	C6—N1—N2—C11	164.27 (14)
C16—C11—C12—C13	−1.2 (3)	C12—C11—N2—C3	−146.46 (17)
N2—C11—C12—C13	−175.75 (17)	C16—C11—N2—C3	39.0 (2)
C11—C12—C13—C14	−1.0 (3)	C12—C11—N2—N1	5.4 (2)

C12—C13—C14—C15	1.5 (3)	C16—C11—N2—N1	−169.15 (15)
C13—C14—C15—C16	0.4 (3)	N2—C3—N4—N5	−0.1 (2)
C14—C15—C16—C11	−2.6 (3)	C9—C3—N4—N5	168.86 (14)
C12—C11—C16—C15	3.0 (3)	N1—C6—N5—N4	41.6 (2)
N2—C11—C16—C15	177.59 (17)	C7—C6—N5—N4	−128.49 (15)
C22—C17—C18—C19	−1.5 (3)	N1—C6—N5—C17	−171.60 (15)
N5—C17—C18—C19	179.92 (15)	C7—C6—N5—C17	18.3 (2)
C17—C18—C19—C20	0.1 (3)	C3—N4—N5—C6	−40.05 (18)
C18—C19—C20—C21	0.6 (3)	C3—N4—N5—C17	170.98 (14)
C19—C20—C21—C22	−0.1 (3)	C18—C17—N5—C6	−124.46 (17)
C20—C21—C22—C17	−1.3 (3)	C22—C17—N5—C6	56.9 (2)
C18—C17—C22—C21	2.1 (3)	C18—C17—N5—N4	21.6 (2)
N5—C17—C22—C21	−179.28 (16)	C22—C17—N5—N4	−157.09 (15)

Fig. 1


C10
C9
C19
C18
N4
C16
C15
C17
N5
C3
O9
C20
C22
N2
C11
C14
C21
C6
N1
C13
C12
O7
C7
C8