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

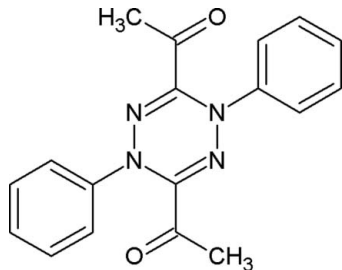
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.040; wR factor = 0.108; data-to-parameter ratio = 13.6.

 In the title compound, $\text{C}_{18}\text{H}_{16}\text{N}_4\text{O}_2$, 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

 $\text{C}_{18}\text{H}_{16}\text{N}_4\text{O}_2$
 $M_r = 320.35$

 Monoclinic, $P2_1/c$
 $a = 11.7853$ (6) Å

 $b = 14.7217$ (7) Å
 $c = 9.5113$ (4) Å
 $\beta = 92.350$ (1)°
 $V = 1648.82$ (13) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ (2) K
 $0.28 \times 0.23 \times 0.12$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

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

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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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

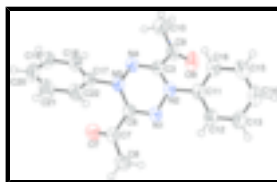


Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

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

Crystal data

C₁₈H₁₆N₄O₂

$F_{000} = 672$

supplementary materials

$M_r = 320.35$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.7853$ (6) Å

$b = 14.7217$ (7) Å

$c = 9.5113$ (4) Å

$\beta = 92.350$ (1)°

$V = 1648.82$ (13) Å³

$Z = 4$

$D_x = 1.291$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4916 reflections

$\theta = 2.2$ – 23.9 °

$\mu = 0.09$ mm⁻¹

$T = 298$ (2) K

Block, red

$0.28 \times 0.23 \times 0.12$ mm

Data collection

Bruker SMART APEX
diffractometer

Radiation source: normal-focus sealed tube

Monochromator: graphite

Detector resolution: 8.3 pixels mm⁻¹

$T = 298$ (2) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2004)

$T_{\min} = 0.973$, $T_{\max} = 0.989$

24032 measured reflections

2985 independent reflections

2090 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\text{max}} = 25.3$ °

$\theta_{\text{min}} = 1.7$ °

$h = -14 \rightarrow 14$

$k = -17 \rightarrow 17$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.107$

$S = 1.02$

2985 reflections

219 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.1852P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.12$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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)

supplementary materials

Atomic displacement parameters (\AA^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 (\AA , $^\circ$)

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)

supplementary materials

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

