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# (1*E*,2*E*)-1,2-Bis(2,2-diphenylhydrazin-1-ylidene)ethane

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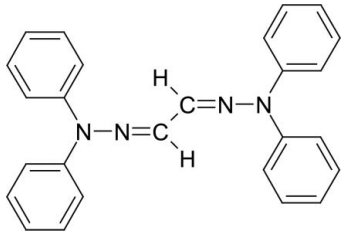
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.113; data-to-parameter ratio = 13.8.

In the crystal structure of the title compound, $C_{26}H_{22}N_4$, the molecule is located on an inversion centre and shows an $E$ configuration with respect to each C=N bond. The dihedral angle between the phenyl rings in the diphenylhydrazone group is 83.69 (11)°. These two rings make dihedral angles of 30.53 (15) and 84.53 (16)° with the central N—N=C—C=N—N dihydrazonoethane plane. Intermolecular C—H···$\pi$ interactions are observed.

## Related literature

For applications of hydrazones, see: Angell *et al.* (2006); Ibañez *et al.* (2002). For related structures, see: Clulow *et al.* (2008); Mendoza *et al.* (2010). For bond-length data, see: Allen *et al.* (1987).



## Experimental

### Crystal data

$C_{26}H_{22}N_4$
$M_r$ = 390.48
Monoclinic, $P2_1/n$
$a$ = 12.2210 (19) Å
$b$ = 5.612 (1) Å
$c$ = 15.731 (3) Å
$\beta$ = 103.924 (16)°
$V$ = 1047.2 (3) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 0.58 mm$^{-1}$
$T$ = 298 K
0.19 × 0.11 × 0.05 mm

### Data collection

Oxford Xcalibur Atlas Gemini diffractometer
Absorption correction: analytical (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min}$ = 0.978, $T_{max}$ = 0.993
3621 measured reflections
1892 independent reflections
1163 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.113
$S$ = 1.01
1892 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C7–C12 rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C3—H3···*Cg*2$^{i}$ | 0.93 | 2.85 | 3.728 (3) | 159 |
| C8—H8···*Cg*1$^{ii}$ | 0.93 | 2.88 | 3.785 (3) | 164 |

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x-1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2589).

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# supporting information

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## (1*E*,2*E*)-1,2-Bis(2,2-diphenylhydrazin-1-ylidene)ethane

**Angel Mendoza, Blanca M. Cabrera-Vivas, Ruth Meléndrez-Luevano, Juan C. Ramírez and Marcos Flores-Alamo**

### S1. Comment

Among the most interesting applications of hydrazones, molecular sensing is worth mentioning. They are being used widely to detect chemical and biological species (Angell *et al.*, 2006). Also, hydrazones are being applied as plasticizer agents, polymerization initiators and antioxidants (Ibañez *et al.*, 2002). There are pigments, as 1-fenilazo-2-naftol, that show an azo/hydrazone tautomery in which the main tautomer exist as hydrazone form.

The asymmetric unit of the title compound **I** consist of $C_{13}H_{11}N_2$ with a $Z'$ = 0.5 showing a centrosymmetrical structure. The compound **I** ($C_{26}H_{22}N_4$) present an *E* configuration for each C═N double bond (Fig. 1), with *N*,*N*-diphenyl group opposite to second C═N group. The molecule shows a non-planar structure for phenyl rings respect to N—N group, with a torsion angle between them C2—C1—N1—C7 = 46.6 (3)°. The torsion angle of phenyl ring C1/C2/C3/C4/C5/C6 to N—N═C group is -173.48 (18)°, and the other ring C7/C8/C9/C10/C11/C12 shows a torsion angle of -14.9 (3)° to the same group. The N—N distance [1.364 (2) Å] is shorter than found in free diphenylhydrazine [1.418 (2) Å] (Clulow *et al.*, 2008). Imine bond distance, N2═C13 [1.287 (2) Å], is longer than N═C typical bond (Allen *et al.*, 1987), but similar [1.286 (3) Å] to related structures with *N*,*N*-diphenylhidrazone group (Mendoza *et al.*, 2010).

### S2. Experimental

*N*,*N*-diphenylhydrazine (2.74 mg, 12.4 mmol) was dissolved in ethanol and acetic acid (0.5 ml) was added slowly into this solution while stirring. Glyoxal (300 mg, 5.1 mmol) was added drop by drop into the above solution with strong stirring and the resulting mixture was kept at atmospheric temperature until it became yellow solution. After three hours, the amber solution turns to be precipitated. The mixture was separated with filtration in vacuum system and the precipitate was washed three times with cold methanol. Recrystallization was performed several times with acetonitrile, to obtain needle crystals suitable for X-ray analysis. Yield: 1.79 g (90%) at 25 °C, mp. 185–189 °C. FT–IR (film): ($cm^{-1}$): 3062 *ν*(C—H), 1750–2000 *ν*(Ph), 1591, 1544, 1490 *ν*(C═N). EI–MS: m/*z* 390 $M^+$.

### S3. Refinement

H atoms were placed in geometrical idealized positions (C—H = 0.93 Å) and refined as riding on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C).

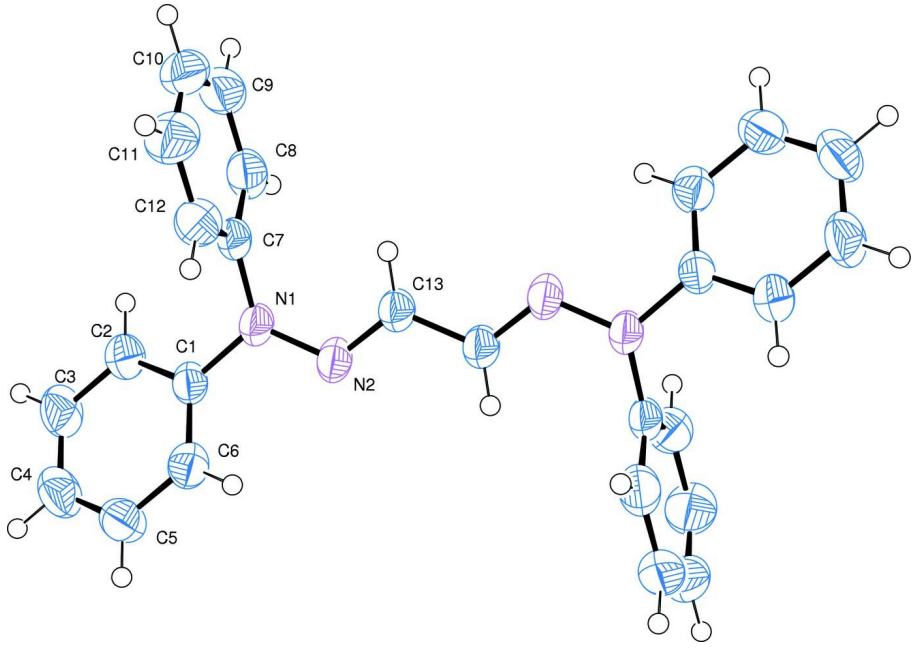


**Figure 1**

The molecular structure of compound **I**, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

**(1*E*,2*E*)-1,2-Bis(2,2-diphenylhydrazin-1-ylidene)ethane**

*Crystal data*

$C_{26}H_{22}N_4$
$M_r = 390.48$
Monoclinic, $P2_1/n$
$a = 12.2210\ (19)$ Å
$b = 5.612\ (1)$ Å
$c = 15.731\ (3)$ Å
$\beta = 103.924\ (16)°$
$V = 1047.2\ (3)$ Å$^3$
$Z = 2$
$F(000) = 412$
$D_x = 1.238$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 864 reflections
$\theta = 3.7–68.0°$
$\mu = 0.58$ mm$^{-1}$
$T = 298$ K
Prism, colourless
0.19 × 0.11 × 0.05 mm

*Data collection*

Oxford Xcalibur Atlas Gemini diffractometer
Graphite monochromator
Detector resolution: 10.4685 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: analytical (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.978$, $T_{max} = 0.993$
3621 measured reflections
1892 independent reflections
1163 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 68.2°$, $\theta_{min} = 4.1°$
$h = -14\rightarrow14$
$k = -4\rightarrow6$
$l = -18\rightarrow10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.113$
$S = 1.01$
1892 reflections
137 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
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.0674P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0134 (9)

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N2 | 0.09341 (13) | 0.1892 (3) | 0.46419 (10) | 0.0451 (5) |
| N1 | 0.20416 (13) | 0.2544 (3) | 0.48657 (10) | 0.0481 (5) |
| C1 | 0.23555 (16) | 0.4384 (4) | 0.43534 (12) | 0.0421 (5) |
| C13 | 0.05715 (15) | 0.0396 (4) | 0.51307 (13) | 0.0451 (5) |
| H13 | 0.1046 | −0.0161 | 0.5646 | 0.054* |
| C12 | 0.27665 (18) | 0.3696 (4) | 0.63743 (14) | 0.0541 (6) |
| H12 | 0.2307 | 0.5037 | 0.6265 | 0.065* |
| C7 | 0.27471 (15) | 0.2080 (4) | 0.57158 (12) | 0.0415 (5) |
| C6 | 0.16000 (18) | 0.6077 (4) | 0.39339 (13) | 0.0494 (5) |
| H6 | 0.0859 | 0.6044 | 0.3988 | 0.059* |
| C2 | 0.34652 (17) | 0.4494 (4) | 0.42837 (13) | 0.0532 (6) |
| H2 | 0.3988 | 0.3382 | 0.4574 | 0.064* |
| C5 | 0.1938 (2) | 0.7827 (4) | 0.34321 (14) | 0.0587 (6) |
| H5 | 0.1422 | 0.8956 | 0.3147 | 0.07* |
| C8 | 0.33966 (18) | 0.0069 (4) | 0.58755 (15) | 0.0572 (6) |
| H8 | 0.3374 | −0.1049 | 0.5435 | 0.069* |
| C3 | 0.3793 (2) | 0.6255 (4) | 0.37837 (15) | 0.0623 (7) |
| H3 | 0.4538 | 0.6319 | 0.3739 | 0.075* |
| C4 | 0.3036 (2) | 0.7904 (4) | 0.33537 (15) | 0.0640 (7) |
| H4 | 0.3261 | 0.9069 | 0.3011 | 0.077* |
| C10 | 0.4116 (2) | 0.1382 (6) | 0.73586 (17) | 0.0726 (8) |
| H10 | 0.4585 | 0.115 | 0.7913 | 0.087* |
| C9 | 0.40936 (19) | −0.0277 (5) | 0.67093 (19) | 0.0722 (8) |
| H9 | 0.4544 | −0.163 | 0.6827 | 0.087* |
| C11 | 0.3458 (2) | 0.3350 (5) | 0.71930 (15) | 0.0708 (8) |
| H11 | 0.3473 | 0.4465 | 0.7633 | 0.085* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N2 | 0.0382 (9) | 0.0534 (11) | 0.0436 (10) | −0.0137 (8) | 0.0099 (7) | −0.0026 (8) |
| N1 | 0.0372 (9) | 0.0609 (11) | 0.0446 (10) | −0.0163 (8) | 0.0066 (7) | 0.0080 (9) |
| C1 | 0.0439 (11) | 0.0468 (12) | 0.0357 (10) | −0.0134 (10) | 0.0096 (8) | −0.0006 (9) |
| C13 | 0.0391 (10) | 0.0537 (13) | 0.0428 (11) | −0.0106 (10) | 0.0101 (9) | 0.0036 (11) |
| C12 | 0.0544 (13) | 0.0597 (14) | 0.0500 (13) | −0.0014 (12) | 0.0161 (10) | 0.0011 (12) |
| C7 | 0.0352 (10) | 0.0470 (12) | 0.0438 (11) | −0.0101 (10) | 0.0125 (8) | 0.0036 (10) |
| C6 | 0.0480 (12) | 0.0532 (13) | 0.0470 (12) | −0.0083 (11) | 0.0113 (10) | −0.0035 (11) |
| C2 | 0.0461 (12) | 0.0587 (14) | 0.0564 (13) | −0.0081 (11) | 0.0157 (10) | 0.0069 (11) |
| C5 | 0.0705 (16) | 0.0501 (14) | 0.0523 (13) | −0.0041 (12) | 0.0087 (11) | 0.0069 (11) |
| C8 | 0.0512 (13) | 0.0509 (13) | 0.0713 (16) | −0.0074 (12) | 0.0184 (12) | 0.0017 (13) |
| C3 | 0.0574 (14) | 0.0731 (16) | 0.0640 (15) | −0.0158 (13) | 0.0297 (12) | 0.0068 (13) |
| C4 | 0.0806 (17) | 0.0612 (16) | 0.0540 (14) | −0.0189 (14) | 0.0234 (12) | 0.0076 (12) |
| C10 | 0.0544 (14) | 0.102 (2) | 0.0554 (16) | −0.0156 (16) | 0.0024 (12) | 0.0226 (16) |
| C9 | 0.0484 (13) | 0.0676 (17) | 0.098 (2) | 0.0028 (13) | 0.0116 (14) | 0.0304 (16) |
| C11 | 0.0731 (16) | 0.092 (2) | 0.0463 (14) | −0.0098 (16) | 0.0125 (12) | −0.0014 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N2—C13 | 1.287 (2) | C2—C3 | 1.381 (3) |
| N2—N1 | 1.364 (2) | C2—H2 | 0.93 |
| N1—C1 | 1.418 (2) | C5—C4 | 1.377 (3) |
| N1—C7 | 1.430 (2) | C5—H5 | 0.93 |
| C1—C6 | 1.377 (3) | C8—C9 | 1.395 (3) |
| C1—C2 | 1.388 (3) | C8—H8 | 0.93 |
| C13—C13$^{i}$ | 1.429 (4) | C3—C4 | 1.367 (3) |
| C13—H13 | 0.93 | C3—H3 | 0.93 |
| C12—C7 | 1.373 (3) | C4—H4 | 0.93 |
| C12—C11 | 1.373 (3) | C10—C11 | 1.354 (3) |
| C12—H12 | 0.93 | C10—C9 | 1.377 (4) |
| C7—C8 | 1.368 (3) | C10—H10 | 0.93 |
| C6—C5 | 1.384 (3) | C9—H9 | 0.93 |
| C6—H6 | 0.93 | C11—H11 | 0.93 |
| | | | |
| C13—N2—N1 | 118.93 (16) | C4—C5—C6 | 120.3 (2) |
| N2—N1—C1 | 115.85 (16) | C4—C5—H5 | 119.9 |
| N2—N1—C7 | 122.01 (14) | C6—C5—H5 | 119.9 |
| C1—N1—C7 | 118.63 (15) | C7—C8—C9 | 118.9 (2) |
| C6—C1—C2 | 119.11 (19) | C7—C8—H8 | 120.5 |
| C6—C1—N1 | 122.18 (18) | C9—C8—H8 | 120.5 |
| C2—C1—N1 | 118.71 (19) | C4—C3—C2 | 120.8 (2) |
| N2—C13—C13$^{i}$ | 119.0 (2) | C4—C3—H3 | 119.6 |
| N2—C13—H13 | 120.5 | C2—C3—H3 | 119.6 |
| C13$^{i}$—C13—H13 | 120.5 | C3—C4—C5 | 119.5 (2) |
| C7—C12—C11 | 120.6 (2) | C3—C4—H4 | 120.2 |
| C7—C12—H12 | 119.7 | C5—C4—H4 | 120.2 |

| | | | |
|---|---|---|---|
| C11—C12—H12 | 119.7 | C11—C10—C9 | 120.2 (2) |
| C8—C7—C12 | 120.2 (2) | C11—C10—H10 | 119.9 |
| C8—C7—N1 | 120.98 (19) | C9—C10—H10 | 119.9 |
| C12—C7—N1 | 118.86 (19) | C10—C9—C8 | 120.1 (2) |
| C1—C6—C5 | 120.3 (2) | C10—C9—H9 | 119.9 |
| C1—C6—H6 | 119.8 | C8—C9—H9 | 119.9 |
| C5—C6—H6 | 119.8 | C10—C11—C12 | 119.9 (2) |
| C3—C2—C1 | 120.0 (2) | C10—C11—H11 | 120.1 |
| C3—C2—H2 | 120 | C12—C11—H11 | 120.1 |
| C1—C2—H2 | 120 | | |

| | | | |
|---|---|---|---|
| C13—N2—N1—C1 | −173.48 (18) | N1—C1—C6—C5 | −179.26 (18) |
| C13—N2—N1—C7 | −14.9 (3) | C6—C1—C2—C3 | −1.3 (3) |
| N2—N1—C1—C6 | 26.8 (3) | N1—C1—C2—C3 | 179.48 (18) |
| C7—N1—C1—C6 | −132.5 (2) | C1—C6—C5—C4 | −0.6 (3) |
| N2—N1—C1—C2 | −154.06 (18) | C12—C7—C8—C9 | −1.5 (3) |
| C7—N1—C1—C2 | 46.6 (3) | N1—C7—C8—C9 | 178.36 (18) |
| N1—N2—C13—C13$^{i}$ | −176.3 (2) | C1—C2—C3—C4 | 0.0 (3) |
| C11—C12—C7—C8 | 1.7 (3) | C2—C3—C4—C5 | 1.0 (4) |
| C11—C12—C7—N1 | −178.09 (18) | C6—C5—C4—C3 | −0.8 (3) |
| N2—N1—C7—C8 | 93.9 (2) | C11—C10—C9—C8 | 0.4 (4) |
| C1—N1—C7—C8 | −108.1 (2) | C7—C8—C9—C10 | 0.4 (3) |
| N2—N1—C7—C12 | −86.3 (2) | C9—C10—C11—C12 | −0.1 (4) |
| C1—N1—C7—C12 | 71.7 (2) | C7—C12—C11—C10 | −0.9 (3) |
| C2—C1—C6—C5 | 1.6 (3) | | |

Symmetry code: (i) −*x*, −*y*, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C3—H3···*Cg*2$^{ii}$ | 0.93 | 2.85 | 3.728 (3) | 159 |
| C8—H8···*Cg*1$^{iii}$ | 0.93 | 2.88 | 3.785 (3) | 164 |

Symmetry codes: (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*.