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4,4-Dimethyl-2-(2-methyl-1-propenyl)-2,6-diphenyl-2,3,4,5-tetrahydropyrimidine

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Key indicators

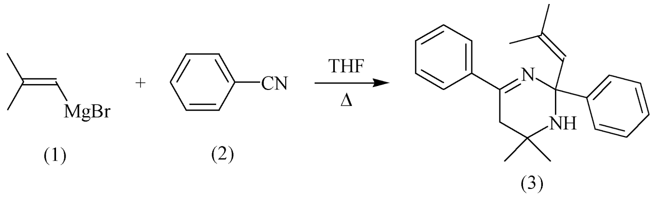
Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.118
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{22}H_{26}N_2$, the tetrahydropyrimidine ring is planar except for the C atom bearing the two methyl groups. The NH group is not involved in classical hydrogen bonding, but instead displays a contact of 2.83 Å to the midpoint of a phenyl C—C bond.

Comment

In an attempt to prepare 3-methyl-1-phenyl-but-2-en-1-one by the addition of 2-methyl-1-propenyl magnesium bromide, (1), to benzonitrile, (2), in refluxing tetrahydrofuran, we noted that the initially formed oily product (1-aza-4-methyl-2-phenyl-1,3-pentadiene) began to form colourless crystals in *ca*. 12% yield after 10 h. This compound was identified as the title compound, (3), by spectroscopic data (Lautenbach, 1991) and the structure analysis reported here. We propose that the mechanism for the formation of (3) involves a hetero Diels–Alder reaction of the initially formed imine, followed by a 1,3-hydrogen shift.



The molecule of (3) is shown in Fig. 1. Bond lengths and angles may be regarded as normal. Five atoms of the tetrahydropyrimidine ring are coplanar (r.m.s. deviation 0.03 Å), with C4 lying 0.561 (2) Å out of the plane.

The NH group is not involved in classical hydrogen bonding; instead, the molecules are linked in pairs (*via* the operator $1 - x$, $1 - y$, $1 - z$) by the contacts N3—H03$\cdots$Cent(C13,14), with H$\cdots$Cent 2.83 Å and an angle of 171°, and C8—H8*A*$\cdots$Cent(C9–14), with H$\cdots$Cent 2.90 Å and an angle of 170°; these could be classified as N—H$\cdots\pi$ or C—H$\cdots\pi$ interactions (Cent = centroid or midpoint).

Experimental

The compound was synthesized as reported by Lautenbach (1991) and recrystallized by evaporation from *tert*-butyl methyl ether.

Crystal data

$C_{22}H_{26}N_2$
M_r = 318.45
Monoclinic, $P2_1/c$
a = 10.268 (3) Å
b = 8.401 (3) Å
c = 21.281 (7) Å
β = 96.45 (3)°
V = 1824.1 (10) Å^3
Z = 4

D_x = 1.160 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 50 reflections
θ = 10–11.5°
μ = 0.07 mm^{-1}
T = 178 (2) K
Prism, pale violet
0.70 × 0.25 × 0.20 mm

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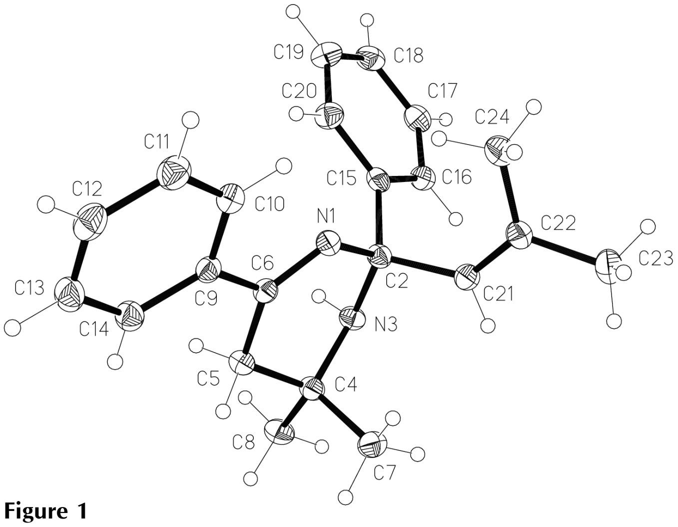

Figure 1
The molecule of compound (3). Displacement ellipsoids are drawn at the 30% probability level. H atom radii are arbitrary.

Data collection

Nicolet *R*3 diffractometer
ω scans
6491 measured reflections
3204 independent reflections
2169 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 25.0°$
$h = -6 \rightarrow 12$
$k = -9 \rightarrow 9$
$l = -25 \rightarrow 25$
3 standard reflections
every 147 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.118$
$S = 1.01$
3204 reflections
225 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.2435P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.278 (2)	N3—C4	1.471 (2)
N1—C2	1.471 (2)	C4—C5	1.532 (2)
C2—N3	1.477 (2)	C5—C6	1.509 (2)
C6—N1—C2—N3	4.9 (2)	N3—C4—C5—C6	−42.63 (19)
N1—C2—N3—C4	−32.5 (2)	C2—N1—C6—C5	1.2 (3)
C2—N3—C4—C5	50.80 (19)	C4—C5—C6—N1	18.5 (2)

The H atom at N3 was refined freely. Methyl H atoms were identified in difference syntheses, idealized and then refined using rigid methyl groups (C—H 0.98 Å, H—C—H 109.5°) Other H atoms were included using a riding model, with fixed C—H bond lengths of 0.95 Å (sp^2) or 0.99 Å ($\mathrm{CH_2}$); $U_{\rm iso}$(H) values were fixed at 1.2 times $U_{\rm eq}$ of the parent atom.

Data collection: *P*3 (Nicolet, 1987); cell refinement: *P*3; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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