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Tris(cyclohexylmethyl)amine

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The title compound, $C_{21}H_{39}N$, is a sterically hindered amine with three cyclohexane rings, all having chair conformations. The bond angles at the N atom are 111.3 (2), 111.58 (19) and 110.3 (2)°.

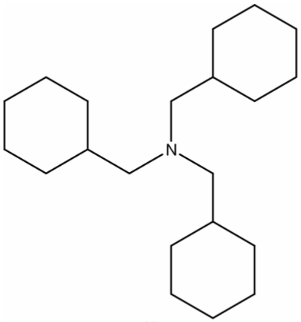
Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.004 Å
R factor = 0.034
wR factor = 0.065
Data-to-parameter ratio = 10.5

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

Comment

In order to study the rhodium-catalysed hydroaminomethylation (Rische & Eilbracht, 1997) of urea with cyclohexene, different conditions were used. Under acidic conditions crystals of the title compound (Katritzky *et al.*, 1989; Striegler & Weber, 1965), (I), were surprisingly obtained, but only in poor yields and small amounts.



(I)

The molecular structure of (I) is illustrated in Fig. 1. On performing the same reaction under optimized conditions, (I)

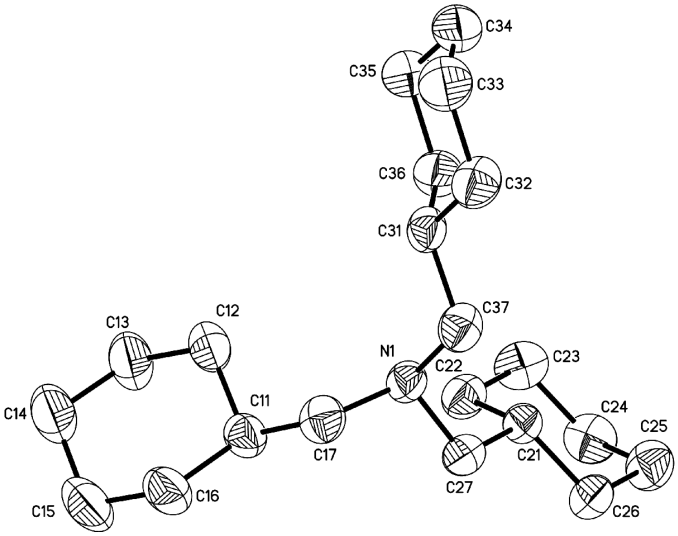


Figure 1
View of the title compound (*XP*; Sheldrick, 1991), showing the labelling of all non-H atoms. Displacement ellipsoids are shown at 30% probability levels. H atoms have been omitted for clarity.

can be obtained as the only product in 74% yield. The formation of (I) occurs by hydrolysis of the urea to ammonia, which was alkylated by a series of condensation and reduction reactions to the tertiary amine. Due to the low concentration of ammonia, the tertiary amine is selectively formed. This is in contrast to the direct conversion of ammonia, which leads preferentially to the formation of the secondary amine (Rische *et al.*, 1998).

Experimental

A solution of 0.821 g (10 mmol) cyclohexene in 90 ml dioxane, 9 ml of methanol and 1 ml of glacial acetic acid in the presence of 0.606 g (10 mmol) urea was treated over a period of 3 d under hydroaminomethylation conditions, under 80 bar syngas (40 bar hydrogen, 40 bar carbon monoxide) at 393 K with 0.3 mol% of the $[Rh(cod)Cl]_2$ (cod is 1,5-cyclooctadiene) catalyst. The solvent was evaporated and the aquous mixture treated with 20 ml of a concentrated solution of sodium hydroxide and 20 ml of water. The mixture was extracted with ethyl acetate, and the organic layer was dried over sodium sulfate and evaporated to obtain the crude material, which was purified by column chromatography on neutral aluminium oxide (act. III) with cyclohexane/methyl *tert*-butyl ether in a 10:1 ratio as eluant to obtain 0.752 g (2.5 mmol) of (I) in a yield of 74%. Product (I) was recrystallized from a mixture of diethyl ether/methanol in a 1:1 ratio. The purity of (I) (m.p. 366–367 K) was confirmed by elemental analysis (calculated: C 82.55, H 12.86, N 4.59%; found: C 82.4, H 12.6, N 4.5%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.01 (*d*, 6H, ^3J = 7.0 Hz, H17, H27, H37), 1.64–1.79 (15H, ring CH_2), 1.13–1.38 (12H, ring CH_2), 0.75–0.82 (6H, ring CH_2); ^{13}C NMR (125 MHz, $CDCl_3$): δ 63.26, (C17, C27, C37), 36.27 (C11, C21, C31), 31.90 (ring CH_2), 27.03 (ring CH_2), 26.28 (ring CH_2); IR (Film, NaCl): $\tilde{\nu}$ [cm^{-1}] 2922 (*vs*), 2846 (*vs*), 2792 (*s*), 1448 (*s*), 1275 (*m*), 1261 (*m*), 1244 (*m*), 1171 (*m*), 1115 (*m*), 1074 (*m*), 1061 (*m*), 1051 (*m*), 966 (*m*), 868 (*m*), 841 (*m*), 582 (*w*); ESI MS (*m*/*z*): 306.25 ($[M + (H^+)]$); GC–MS (EI, 70 eV): m/*z* (%) = 305 (M^+, 7), 222 (100), 140 (51), 58 (51), 55 (51).

Crystal data

$C_{21}H_{39}N$
$M_r = 305.53$
Orthorhombic, $P2_12_12_1$
$a = 11.050$ (3) Å
$b = 11.2116$ (4) Å
$c = 16.3949$ (6) Å
$V = 2031.2$ (6) Å^3
$Z = 4$
$D_x = 0.999$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 10 616 reflections
$\theta = 3.1$–25.3°
$\mu = 0.06$ mm^{-1}
$T = 291$ (1) K
Block, colourless
0.12 × 0.12 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: none
10 616 measured reflections
2101 independent reflections
882 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.037$
$\theta_{\rm max} = 25.3°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.065$
$S = 0.88$
2101 reflections
200 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0162P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.08$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.08$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0226 (10)

H atoms were placed in calculated positions, with C—H = 0.97–0.98 Å, and were refined as riding, with $U_{\rm iso} = 1.2U_{\rm eq}$(C) of the carrier atom. In the absence of significant anomalous cattering, Friedel pairs were merged.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97, *PARST*95 (Nardelli, 1995) and *PLATON* (Spek, 2001).

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