

map calculations, full-matrix least squares (SHELX76; Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms and isotropic ones for H atoms, $w = [\sigma^2(|F_o|) + 0.0040|F_o|^2]^{-1}$; all H atoms were located on difference Fourier map; final $R = 0.048$ and $wR = 0.057$ for 3380 observed reflections; $(\Delta/\sigma)_{\max} = 0.08$; final difference map showed $\Delta\rho < 0.13 \text{ e } \text{Å}^{-3}$; atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV); calculations were carried out on an IBM 4381-R24 computer at this university. Final atomic coordinates are listed in Table 1 and selected bond distances and angles in Table 2.* The molecule with

* Lists of anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms, bond distances and angles involving H atoms, least-squares-planes data and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53199 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the atom numbering is shown in Fig. 1. The crystal structure is shown in Fig. 2.

Related literature. Mechanistic study of the valence tautomerism: Lown & Matsumoto (1970), and Padwa & Vega (1975).

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N,N'-Dicyclohexyl-*N*-4-phenylbutyrylurea

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Abstract. $\text{C}_{23}\text{H}_{34}\text{N}_2\text{O}_2$, $M_r = 370.54$, monoclinic, $P2_1/c$, $a = 11.0578$ (8), $b = 23.740$ (3), $c = 8.8991$ (9) Å, $\beta = 111.117$ (8)°, $V = 2179.2$ Å³, $Z = 4$, $D_x = 1.129 \text{ Mg m}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 0.53 \text{ mm}^{-1}$, $F(000) = 808$, $T = 296 \text{ K}$, $R(F) = 0.059$ for 3060 observed [$I \geq 3\sigma(I)$] reflections. Bond distances and angles within the molecule are normal. There is one close intermolecular hydrogen-bonding distance between O(1) and N(1) of 2.841 (1) Å. The C(1)—N(1)—C(7)—O(1) and C(8)—N(2)—C(14)—O(2) torsion angles are -10.6 (3) and -10.7 (3)° respectively.

Experimental. The title compound was prepared as a side product in a reaction to prepare *N*-methyl-*N*-(2-methylphenyl)-4-phenylbutanamide by reacting 4-phenylbutyric acid with dicyclohexylcarbodiimide and 2-methyl-*N*-methylaniline in acetonitrile. Re-

crystallization from ethanol/water yielded diffraction-quality crystals.

Crystal $0.13 \times 0.21 \times 0.44 \text{ mm}$. Enraf–Nonius CAD-4 diffractometer. Lattice parameters determined using 24 reflections with $18 < 2\theta < 40^\circ$. Lorentz–polarization correction applied; absorption correction using empirical method (absorption surface) (Walker & Stuart, 1983). Maximum–minimum correction coefficients applied to F_o were 1.2219 and 0.6630. Intensity measurements in range $0 < 2\theta < 140^\circ$ (index limits: $h + 10$, $k + 28$, $l \pm 13$). Intensity standards: two reflections remeasured every 60 min of X-ray exposure time showed changes in intensity of $< 0.1\%$. No decay correction. Total reflections measured: 4520; unique: 4242; 1182 unobserved [$I < 3\sigma(I)$]. R factor for averaging equivalent reflections 0.020. Structure solved by direct methods using SHELXS86 (Sheldrick, 1985). Full-matrix least-

Table 1. Positional ($\times 10^4$) and thermal ($\times 10^2$) parameters

	x	y	z	$U(\text{\AA}^2)^*$
O(1)	1719 (1)	2931.9 (7)	769 (2)	7.01 (5)
O(2)	4974 (1)	2742.3 (7)	-1001 (2)	8.90 (6)
N(1)	1224 (1)	2365.8 (7)	-1413 (2)	5.82 (5)
N(2)	3071 (1)	2922.8 (7)	-704 (2)	5.79 (5)
C(1)	144 (2)	2067.9 (8)	-1177 (2)	5.62 (6)
C(2)	167 (2)	1454 (1)	-1622 (3)	8.26 (9)
C(3)	-951 (3)	1132 (1)	-1400 (4)	9.9 (1)
C(4)	-2234 (2)	1392 (1)	-2321 (3)	9.3 (1)
C(5)	-2256 (2)	2007 (1)	-1858 (4)	10.6 (1)
C(6)	-1142 (2)	2334 (1)	-2102 (4)	9.3 (1)
C(7)	1958 (2)	2737.9 (8)	-368 (2)	5.40 (6)
C(8)	3256 (2)	3535.0 (8)	-828 (2)	6.04 (6)
C(9)	2048 (3)	3819 (1)	-1954 (3)	7.83 (9)
C(10)	2315 (3)	4431 (1)	-2170 (3)	9.4 (1)
C(11)	2850 (3)	4736 (1)	-600 (3)	9.5 (1)
C(12)	4040 (3)	4449 (1)	537 (4)	10.0 (1)
C(13)	3770 (3)	3829 (1)	789 (3)	8.16 (9)
C(14)	4053 (2)	2566.9 (9)	-684 (2)	6.29 (6)
C(15)	4000 (2)	1959.8 (9)	-203 (3)	6.49 (7)
C(16)	5325 (2)	1685.1 (9)	405 (3)	6.65 (7)
C(17)	5265 (2)	1079 (1)	890 (3)	8.24 (9)
C(18)	6563 (2)	794.5 (9)	1549 (3)	6.71 (7)
C(19)	6844 (2)	335 (1)	795 (3)	8.09 (9)
C(20)	8030 (3)	68 (1)	1409 (3)	9.4 (1)
C(21)	8968 (3)	257 (1)	2769 (3)	10.0 (1)
C(22)	8719 (3)	719 (1)	3526 (3)	10.4 (1)
C(23)	7536 (3)	987 (1)	2931 (3)	8.8 (1)

* The equivalent isotropic thermal parameter U is given by $U = \frac{1}{3} \sum_{i=1}^3 r_i^2$, where r_i are the principal values of the root-mean-square amplitudes of vibration.

Table 2. Selected interatomic distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

O(1)—C(7)	1.224 (1)	C(9)—C(10)	1.508 (2)
O(2)—C(14)	1.224 (2)	C(10)—C(11)	1.493 (3)
N(1)—C(1)	1.467 (2)	C(11)—C(12)	1.503 (3)
N(1)—C(7)	1.327 (2)	C(12)—C(13)	1.535 (2)
N(2)—C(7)	1.436 (2)	C(14)—C(15)	1.510 (2)
N(2)—C(8)	1.477 (2)	C(15)—C(16)	1.514 (2)
N(2)—C(14)	1.371 (2)	C(16)—C(17)	1.511 (2)
C(1)—C(2)	1.513 (2)	C(17)—C(18)	1.501 (2)
C(1)—C(6)	1.501 (2)	C(18)—C(19)	1.373 (2)
C(2)—C(3)	1.526 (2)	C(18)—C(23)	1.387 (2)
C(3)—C(4)	1.492 (3)	C(19)—C(20)	1.380 (2)
C(4)—C(5)	1.519 (3)	C(20)—C(21)	1.356 (3)
C(5)—C(6)	1.535 (2)	C(21)—C(22)	1.365 (3)
C(8)—C(9)	1.510 (2)	C(22)—C(23)	1.378 (3)
C(8)—C(13)	1.514 (2)		
C(1)—N(1)—C(7)	123.0 (1)	C(9)—C(10)—C(11)	112.1 (1)
C(7)—N(2)—C(8)	117.9 (1)	C(10)—C(11)—C(12)	111.5 (2)
C(7)—N(2)—C(14)	122.9 (1)	C(11)—C(12)—C(13)	111.3 (1)
C(8)—N(2)—C(14)	118.5 (1)	C(8)—C(13)—C(12)	109.2 (1)
N(1)—C(1)—C(2)	109.6 (1)	O(2)—C(14)—N(2)	120.5 (1)
N(1)—C(1)—C(6)	112.2 (1)	O(2)—C(14)—C(15)	120.7 (1)
C(2)—C(1)—C(6)	111.3 (1)	N(2)—C(14)—C(15)	118.7 (1)
C(1)—C(2)—C(3)	111.0 (1)	C(14)—C(15)—C(16)	112.4 (1)
C(2)—C(3)—C(4)	112.0 (2)	C(15)—C(16)—C(17)	112.1 (1)
C(3)—C(4)—C(5)	110.7 (1)	C(16)—C(17)—C(18)	114.0 (1)
C(4)—C(5)—C(6)	110.8 (2)	C(17)—C(18)—C(19)	121.3 (1)
C(1)—C(6)—C(5)	111.1 (1)	C(17)—C(18)—C(23)	121.7 (2)
O(1)—C(7)—N(1)	124.8 (1)	C(19)—C(18)—C(23)	117.1 (2)
O(1)—C(7)—N(2)	120.9 (1)	C(18)—C(19)—C(20)	121.5 (2)
N(1)—C(7)—N(2)	114.3 (1)	C(19)—C(20)—C(21)	120.8 (2)
N(2)—C(8)—C(9)	112.1 (1)	C(20)—C(21)—C(22)	118.8 (2)
N(2)—C(8)—C(13)	113.4 (1)	C(21)—C(22)—C(23)	120.9 (2)
C(9)—C(8)—C(13)	111.4 (1)	C(18)—C(23)—C(22)	120.9 (2)
C(8)—C(9)—C(10)	110.4 (1)		

squares refinement using F magnitudes. H atoms located at calculated positions (C—H 0.95 \AA). H-atom thermal parameters fixed at 1.2 times those of the attached atom. Positional and anisotropic thermal parameters for all non-H atoms were refined. For 279 variables refined: $R = 0.059$, $wR = 0.090$, $S = 2.70$. Weights of $1/\sigma^2(F^2)$ with $\sigma(F)$ as defined by Stout & Jensen (1968, equation H.14) and the instability factor defined to be 0.04. $(\Delta/\sigma)_{\text{max}} = 0.03$. Maximum peak height in final difference Fourier map 0.17 (4) e \AA^{-3} . Included as a variable was a secondary-extinction coefficient which refined to a value of 1.01×10^{-6} . Neutral-atom atomic scattering factors and f' and f'' anomalous-dispersion values from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computer programs used include the Enraf-Nonius (1985) *SDP-Plus* software and *ORTEPII* (Johnson, 1976). All calculations performed on a Sun Microsystems 3/260 computer.

Positional and thermal parameters are listed in Table 1; selected distances and angles are presented in Table 2.* The molecule is depicted in Fig. 1 with the numbering scheme employed.

Related literature. The bonding geometry within the core of the molecule [C(1), N(1), C(7), O(1), N(2), C(8), C(14), O(2) and C(15)] is in good agreement

* Lists of structure factors, anisotropic thermal parameters and H-atom positional and thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52866 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

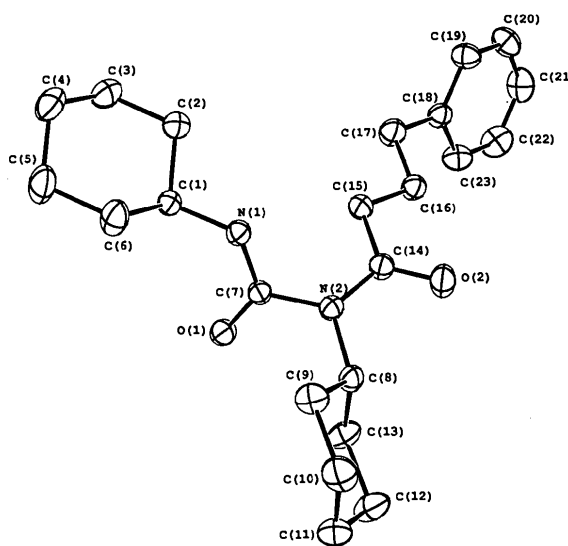


Fig. 1. Perspective view of the molecule with 20% probability ellipsoids.

with the values observed in three related structures: Ishida, Yamashita, Takai & Inoue (1983), Bechtel, Bideau & Cotrait (1979) and Lovell & Perkinson (1978).

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Sequential Reaction of 2-Lithio-2-trimethylsilyl-1,3-dithiane with Dimethylformamide and Ammonia: Structure of Bis[(1,3-dithian-2-ylidene)methyl]amine

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Abstract. $C_{10}H_{15}NS_4$, $M_r = 277.48$, monoclinic, $P2_1/c$, $a = 12.522$ (3), $b = 17.81$ (1), $c = 6.140$ (1) Å, $\beta = 100.41$ (1)°, $V = 1346.7$ Å³, $Z = 4$, $D_x = 1.37$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 6.48$ cm⁻¹, $F(000) = 584$, room temperature, $R = 0.038$ for 1492 observed reflections. The bridging C=C—N—C=C group atoms are planar to within 0.014 Å; the C—N—C bond angle is 125°. The S—C distances range from 1.759 to 1.824 Å.

Experimental. Compound isolated unexpectedly (in 50% yield) from reaction between 2-lithio-2-trimethylsilyl-1,3-dithiane (generated from 2-trimethylsilyl-1,3-dithiane and *n*-butyllithium) and dimethylformamide following work-up with aqueous ammonium chloride solution. Crystals obtained by recrystallization from ethanol. Large block cut to ca 0.35 × 0.3 × 0.3 mm and mounted on a glass fibre; Stoe Stadi-2 2-circle diffractometer, 18 centred reflections ($3.3 < \theta < 17.3^\circ$), graphite-monochromated Mo $K\alpha$; for data collection: $\theta_{\text{max}} = 25^\circ$, ω scans in 0.01° steps, ω -scan width ($2.00 + 0.6 \sin\mu / \tan\theta$)°, h 0 to 15, k 0 to 21, l -7 to 7 (collected in layers of k), intensity-control reflections (one per layer) varied $\pm 3\%$, yielding 2383 unique reflections of which 1492 with $F > 6\sigma(F)$ were retained. Space group $P2_1/c$ from systematic absences ($h0l$: $l = 2n$; $0k0$: $k = 2n$).

Atoms located by automatic direct methods (*SHELXS86*; Sheldrick, 1986) followed by difference-Fourier syntheses with full-matrix, least-squares refinement (*SHELX76*; Sheldrick, 1976), $w = [\sigma^2(F) + 0.000164 F^2]^{-1}$, anisotropic thermal parameters for all non-H atoms. H atoms were located from difference Fourier synthesis but in the later stages of refinement were constrained at fixed bond distances (C—H, 1.00 Å; N—H, 0.95 Å) and were refined with a common isotropic thermal parameter (0.0791 Å² at convergence). $R = 0.038$, $wR = 0.035$, $S = 1.192$, data:variable = 7.3:1, $(\Delta\rho)_{\text{max}} = 0.22$, $(\Delta\rho)_{\text{min}} = -0.20$ e Å⁻³. Max Δ/σ in final cycle was 0.018. Scattering factors were from *SHELX76*. Other programs used were *CHEM3D* and *CALC* (Gould, 1984).

Atomic parameters are listed in Table 1 and bond lengths and angles in Table 2.* The molecule and numbering scheme are shown in Fig. 1. Atoms C(4), C(8), C(9), C(10), and N(1) are planar to within 0.014 Å; deviations from the best plane are as follows: C(4), -0.012 (3); C(8), 0.002 (4); C(9), 0.014 (3); C(10), -0.014 (4); N(1), 0.009 (3) Å.

Related literature. The compound $C_{10}H_{15}NS_4$ is a dienamine in which the β -carbon atoms of the vinyl

* Lists of structure factors, anisotropic thermal parameters, full bond lengths, angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53178 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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