$S = 2.29$
1628 reflections
227 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ge1	0.62926 (1)	0.50268 (2)	0.86369 (4)	0.0404 (2)
C1	0.6291 (2)	0.3761 (2)	0.8150 (6)	0.058 (3)
C2	0.5974 (2)	0.3383 (2)	0.9738 (6)	0.068 (3)
C3	0.5444 (2)	0.3685 (3)	1.0006 (7)	0.086 (3)
C4	0.5840 (1)	0.3981 (3)	1.1373 (6)	0.068 (2)
C5	0.6022 (1)	0.4915 (2)	1.1242 (5)	0.056 (2)
C6	0.5954 (1)	0.5767 (2)	0.6746 (4)	0.039 (2)
C7	0.6315 (1)	0.6342 (2)	0.5939 (4)	0.041 (2)
C8	0.6156 (1)	0.6927 (2)	0.4535 (5)	0.053 (2)
C9	0.5655 (1)	0.6952 (2)	0.3948 (5)	0.056 (2)
C10	0.5302 (1)	0.6395 (2)	0.4749 (5)	0.053 (2)
C11	0.5454 (1)	0.5802 (2)	0.6137 (5)	0.047 (2)
C12	0.6922 (1)	0.5666 (2)	0.8180 (4)	0.043 (2)
C13	0.7397 (1)	0.5601 (2)	0.9037 (5)	0.057 (2)
C14	0.7791 (1)	0.6136 (3)	0.8455 (6)	0.066 (3)
C15	0.7716 (1)	0.6737 (2)	0.7009 (6)	0.065 (2)
C16	0.7247 (1)	0.6827 (2)	0.6156 (5)	0.055 (2)
C17	0.6843 (1)	0.6288 (2)	0.6732 (4)	0.042 (2)

Table 2. *Selected geometric parameters* (Å, °)

Ge1—C1	1.956 (3)	Ge1—C5	1.952 (3)
Ge1—C6	1.945 (3)	Ge1—C12	1.940 (3)
C1—C2	1.496 (6)	C2—C3	1.474 (7)
C3—C4	1.477 (6)	C2—C4	1.497 (6)
C4—C5	1.502 (5)		
C1—Ge1—C5	94.2 (2)	C6—Ge1—C12	89.2 (1)
Ge1—C1—C2	104.6 (3)	C1—C2—C3	119.7 (4)
C2—C3—C4	61.0 (3)	C3—C4—C5	118.2 (3)
Ge1—C5—C4	104.7 (2)	C1—C2—C4	117.1 (3)
C2—C4—C5	117.0 (3)		
Ge1—C1—C2—C3	57.5 (4)	Ge1—C1—C2—C4	−11.3 (5)
C1—C2—C3—C4	−105.9 (4)	C1—C2—C4—C5	1.9 (5)
C2—C3—C4—C5	106.4 (4)	C2—C4—C5—Ge1	8.7 (4)
C3—C4—C5—Ge1	−59.3 (4)	C4—C5—Ge1—C1	−12.7 (2)
C5—Ge1—C1—C2	13.6 (3)	C1—C2—Ge1—C6	−114.2 (3)
C4—C5—Ge1—C6	112.2 (2)	C1—C2—Ge1—C12	141.2 (3)
C4—C5—Ge1—C12	−138.5 (2)		

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *TEXSAN*: *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1113). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1339–1342

1-(3-Benzoylphenyl)ethanone (I) and 3-Benzoyl-α-methylbenzeneacetamide Methylene Chloride Solvate (2/1) (II)

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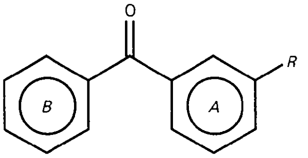
Abstract

While the acetyl group in (I) (*m*-benzoylacetophenone, $C_{15}H_{12}O_2$) is nearly coplanar with the phenyl ring [O2—C14—C1—C2 6.8 (5)°] and positioned opposite the C7═O1 group, the 2-propanamido group in (II) [2-(3-benzoylphenyl)propanamide, $C_{16}H_{15}NO_2.0.5CH_2Cl_2$] is almost perpendicular to the corresponding phenyl ring [C16—C14—C1—C2 134.0 (4), C15—C14—C1—C2 −102.4 (5)°] and occupies the same side as the C7═O1 group.

Comment

The compounds 1-(3-benzoylphenyl)ethanone (I) and 3-benzoyl-α-methylbenzeneacetamide (III) are intermediates (Shimizu, Matsumura & Arai, 1988; Cerbelaud &

Pétré, 1989) and degradation products (Pietta, Manera & Ceva, 1987) of ketoprofene, (IV), which is a well known and widely prescribed non-steroidal anti-inflammatory drug (Brodgen, Speight & Avery, 1974; Huskisson, 1983). In addition, (III) is also known for its anti-inflammatory activity, most probably as the pro-drug form of ketoprofene (Spickett, Vega, Prieto, Moragues, Marquez & Roberts, 1976). Thus, the structure determinations of (I) and (III) were undertaken in order to gain more structural information on ketoprofene-related species, to complement that available for ketoprofene (Briard & Rossi, 1990). Details of the synthetic work will be published elsewhere (Dumić, Eškinja, Matak, Petrović, Rogić, Starčević & Vinković, 1994). Since it was not possible to prepare a good quality single crystal of the propanamide (III) itself, well developed crystals were obtained as its 2:1 solvate with methylene chloride, (II).



(I) $R = -COCH_3$
(II) $R = (III).\frac{1}{2}CH_2Cl_2$
(III) $R = -CH(CH_3)CONH_2$
(IV) $R = -CH(CH_3)COOH$

The bond lengths in the structures of (I) and (II) are within the expected ranges of values. The benzoylphenyl segments are twisted and make torsion angles of 25.2 (5)° for C4—C3—C7—O1 and 30.6 (5)° for O1—C7—C8—C9 in (I) and 32.6 (4)° for C2—C3—C7—O1 and 34.9 (4)° for O1—C7—C8—C9 in (II). Superposition of the benzoylphenyl segments of the structures of (I), (II) and (IV) is shown in Fig. 3. The acetyl group in (I) is nearly coplanar with the phenyl ring [O2—C14—C1—C2 6.8 (5)°] and is positioned opposite the C7═O1 group. The 2-propanamido group in (II), however, is almost perpendicular to the phenyl group [C16—C14—C1—C2 134.0 (4), C15—C14—C1—C2 −102.4 (5)°] and is positioned on the same side as the C7═O1 group. The orientation of the polar parts of the 2-propanato and the 2-propanamido groups with respect to the nonpolar aromatic parts of the benzoyl groups is significantly different, being *anti* in (IV) and *syn* in (II).

The crystal structure of (I) is characterized by stacks of phenyl groups extended along the crystallographic direction $\{10\bar{2}\}$, as shown in Fig. 4. The phenyl groups *A* and *B* alternate in the sequence *AABB*.... However, the phenyl groups *A* and *B* are closer (minimum interatomic distance 3.6 Å) than *A* and *A* or *B* and *B* (minimum interatomic distance > 4 Å).

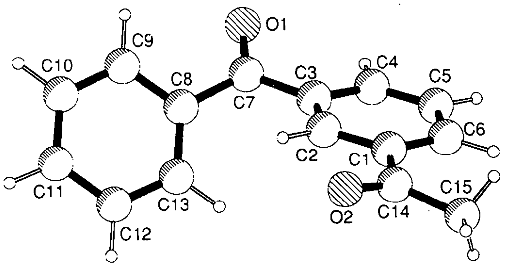


Fig. 1. *PLUTON* (Spek, 1982) drawing of (I) with labelling scheme for non-H atoms

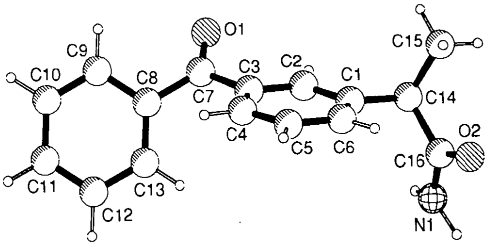


Fig. 2. *PLUTON* (Spek, 1982) drawing of (II) with labelling scheme for non-H atoms.

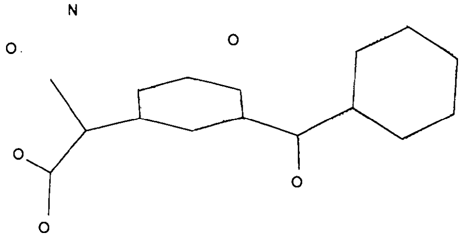


Fig. 3. Superposition of structures (I), (II) and (IV).

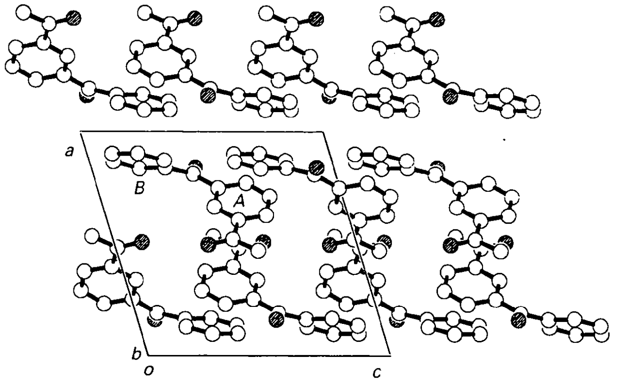


Fig. 4. The molecular packing in compound (I).

In the crystal structure of (II), the molecules are linked together to form dimers by hydrogen bonds [N1—H101···O1^{i} 3.119 (4) Å; symmetry code: (i) $\frac{1}{2}-x, -\frac{1}{2}-y$, $1 - z$], as shown in Fig. 5. These dimers are linked into infinite chains by further hydrogen bonding [N1—H201···O2^{ii} 2.976 (4) Å; symmetry code: (ii) $-x$, $-1 - y$, $1 - z$]. In this way the hydrogen-bonded molecules within the crystal structure build cages, which are filled with the methylene chloride solvent molecules in the molar ratio 2:1.

The methylene chloride molecules are situated on twofold axes and are held within the crystal structure by van der Waals interactions. Thermogravimetric analysis showed that the solvent molecules are lost from the crystals at 343 K [m.p. of (II) 348 K, b.p. of CH_2Cl_2 318 K (Dumić *et al.*, 1994)].

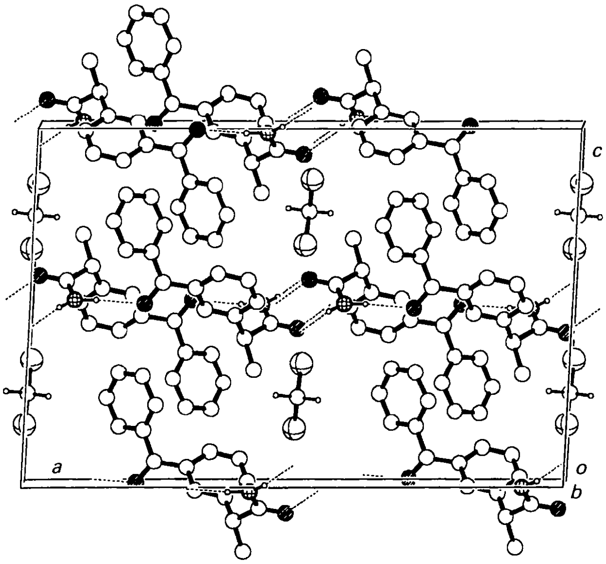


Fig. 5. The molecular packing in (II) with the hydrogen-bonding network represented by dashed lines.

Experimental

Compound (I)

Crystal data

$C_{15}H_{12}O_2$
$M_r = 224.26$
Monoclinic
$P2_1/c$
$a = 10.681$ (1) Å
$b = 10.509$ (1) Å
$c = 11.157$ (1) Å
$\beta = 107.38$ (1)°
$V = 1195.2$ (2) Å^3
$Z = 4$
$D_x = 1.22$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10$–23°
$\mu = 0.6211$ mm^{-1}
$T = 293$ K
Irregular
0.5 × 0.5 × 0.2 mm
Colourless

Data collection

Philips PW1100 diffractometer modified by Stoe
ω scans
Absorption correction: none
2791 measured reflections
1957 independent reflections
1227 observed reflections [$F > 3.0\sigma(F)$]

$R_{\rm int} = 0.0796$
$\theta_{\rm max} = 64°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity variation: 12%

Refinement

Refinement on F
$R = 0.070$
$wR = 0.099$
$S = 0.305$
1227 reflections
166 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F) + 0.1200F^2]$
$(\Delta/\sigma)_{\rm max} = 0.002$

$\Delta\rho_{\rm max} = 0.286$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.446$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (I)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.1612 (3)	−0.1854 (2)	1.0718 (3)	0.017 (2)
O2	0.5055 (3)	0.3019 (2)	1.1146 (2)	0.089 (1)
C1	0.3956 (3)	0.1546 (3)	0.9600 (3)	0.058 (9)
C2	0.3364 (3)	0.0985 (3)	1.0421 (3)	0.059 (1)
C3	0.2523 (3)	−0.0054 (3)	1.0037 (3)	0.065 (1)
C4	0.2303 (3)	−0.0524 (3)	0.8814 (4)	0.081 (2)
C5	0.2897 (4)	0.0024 (4)	0.8022 (3)	0.088 (2)
C6	0.3738 (3)	0.1065 (3)	0.8397 (3)	0.072 (1)
C7	0.1903 (3)	−0.0718 (3)	1.0887 (3)	0.077 (1)
C8	0.1624 (3)	−0.0039 (2)	1.1957 (3)	0.060 (1)
C9	0.1661 (3)	−0.0729 (3)	1.3035 (4)	0.079 (1)
C10	0.1350 (4)	−0.0172 (4)	1.4012 (4)	0.094 (2)
C11	0.0997 (4)	0.1080 (4)	1.3958 (4)	0.092 (2)
C12	0.0978 (3)	0.1793 (3)	1.2905 (4)	0.082 (2)
C13	0.1289 (3)	0.1239 (3)	1.1910 (3)	0.066 (1)
C14	0.4807 (3)	0.2690 (3)	1.0067 (3)	0.063 (1)
C15	0.5313 (4)	0.3433 (4)	0.9160 (4)	0.089 (2)

Table 2. *Selected geometric parameters* (Å, °) *for* (I)

C3—C2	1.396 (4)	C7—O1	1.232 (4)
C3—C4	1.403 (5)	C8—C13	1.385 (4)
C3—C7	1.482 (5)	C8—C9	1.394 (5)
C2—C1	1.389 (5)	C13—C12	1.378 (6)
C1—C6	1.387 (5)	C12—C11	1.388 (6)
C1—C14	1.502 (4)	C11—C10	1.363 (6)
C6—C5	1.397 (5)	C10—C9	1.362 (7)
C5—C4	1.359 (6)	C14—C15	1.500 (6)
C7—C8	1.494 (5)	C14—O2	1.203 (4)
C4—C3—C7	118.7 (3)	C7—C8—C9	118.7 (2)
C2—C3—C7	122.7 (3)	C7—C8—C13	122.8 (3)
C2—C3—C4	118.6 (3)	C13—C8—C9	118.4 (3)
C3—C2—C1	120.4 (3)	C8—C13—C12	121.0 (3)
C2—C1—C6	120.4 (3)	C13—C12—C11	120.5 (3)
C6—C1—C14	122.4 (3)	C12—C11—C10	119.4 (4)
C1—C6—C5	118.8 (3)	C11—C10—C9	120.6 (4)
C6—C5—C4	121.3 (3)	C8—C9—C10	121.1 (3)
C3—C4—C5	120.5 (3)	C1—C14—O2	120.4 (3)
C3—C7—O1	119.8 (3)	C1—C14—C15	118.9 (3)
C3—C7—C8	121.3 (3)	C15—C14—O2	120.6 (3)
C8—C7—O1	118.8 (3)		

Compound (II)

Crystal data

$C_{16}H_{15}NO_2.0.5CH_2Cl_2$
$M_r = 295.09$
Monoclinic
$C2/c$
$a = 23.902$ (10) Å
$b = 8.064$ (2) Å
$c = 15.669$ (3) Å
$\beta = 93.11$ (2)°
$V = 3016$ (1) Å^3

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 28 reflections
$\theta = 12$–16°
$\mu = 0.252$ mm^{-1}
$T = 293$ K
Prism
0.51 × 0.43 × 0.40 mm

$Z = 8$
$D_x = 1.30$ Mg m^{-3}
Colourless

Data collection

Philips PW1100 diffractometer modified by Stoe
ω scans
Absorption correction: none
7072 measured reflections
4393 independent reflections
1947 observed reflections [$F > 4.0\sigma(F)$]
$R_{\rm int} = 0.0492$
$\theta_{\rm max} = 30°$
$h = -34 \rightarrow 34$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 22$
3 standard reflections frequency: 60 min intensity variation: 9.3%

Refinement

Refinement on F
$R = 0.0826$
$wR = 0.1210$
$S = 0.4883$
1947 reflections
202 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F) + 0.0669F^2]$
$(\Delta/\sigma)_{\rm max} = 0.056$
$\Delta\rho_{\rm max} = 0.679$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.481$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.2919 (1)	0.0425 (4)	0.5014 (1)	0.0539 (9)
O2	0.0109 (1)	−0.3296 (4)	0.4248 (3)	0.071 (1)
N1	0.0786 (1)	−0.4902 (4)	0.4883 (2)	0.056 (1)
C1	0.1256 (1)	−0.1265 (5)	0.4650 (2)	0.045 (1)
C2	0.1819 (1)	−0.0950 (4)	0.4833 (2)	0.0400 (9)
C3	0.1996 (1)	0.0254 (4)	0.5445 (2)	0.039 (1)
C4	0.1594 (1)	0.1143 (4)	0.5864 (2)	0.047 (1)
C5	0.1035 (1)	0.0882 (5)	0.5669 (3)	0.056 (1)
C6	0.0866 (1)	−0.0301 (5)	0.5059 (3)	0.053 (1)
C7	0.2606 (1)	0.0565 (4)	0.5599 (2)	0.0387 (9)
C8	0.2839 (1)	0.0994 (4)	0.6465 (2)	0.038 (1)
C9	0.3304 (1)	0.2081 (5)	0.6545 (2)	0.046 (1)
C10	0.3552 (2)	0.2385 (5)	0.7348 (3)	0.058 (1)
C11	0.3351 (2)	0.1631 (6)	0.8069 (3)	0.066 (2)
C12	0.2897 (2)	0.0582 (6)	0.7992 (3)	0.061 (1)
C13	0.2633 (2)	0.0282 (5)	0.7190 (2)	0.050 (1)
C14	0.1069 (2)	−0.2583 (5)	0.4008 (3)	0.051 (1)
C15	0.0868 (3)	−0.1855 (7)	0.3146 (3)	0.083 (2)
C16	0.0611 (1)	−0.3632 (5)	0.4388 (3)	0.049 (1)
C17	0	−0.3648 (9)	3/4	0.109 (4)
Cl1	0.0036 (1)	−0.2436 (3)	0.6591 (1)	0.1145 (8)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

O2—C16	1.238 (4)	C4—C5	1.371 (4)
O1—C7	1.220 (4)	C5—C6	1.395 (6)
N1—C16	1.338 (5)	C7—C8	1.480 (4)
C16—C14	1.530 (6)	C8—C9	1.416 (4)
C14—C15	1.526 (7)	C8—C13	1.387 (5)
C14—C1	1.514 (6)	C9—C10	1.383 (6)
C1—C2	1.384 (4)	C10—C11	1.391 (7)
C1—C6	1.396 (5)	C11—C12	1.376 (7)
C2—C3	1.413 (4)	C12—C13	1.396 (6)
C3—C4	1.392 (4)	Cl1—C17	1.733 (5)
C3—C7	1.486 (4)		
O2—C16—N1	122.7 (3)	C4—C5—C6	120.0 (3)
N1—C16—C14	116.2 (3)	C1—C6—C5	121.3 (3)
O2—C16—C14	121.2 (3)	O1—C7—C3	119.9 (3)
C16—C14—C1	108.7 (4)	C3—C7—C8	120.4 (3)
C16—C14—C15	111.1 (4)	O1—C7—C8	119.7 (3)
C15—C14—C1	112.7 (4)	C7—C8—C13	121.5 (3)
C14—C1—C6	121.0 (3)	C7—C8—C9	118.7 (3)
C14—C1—C2	121.0 (3)	C9—C8—C13	119.7 (3)
C2—C1—C6	118.0 (3)	C8—C9—C10	119.1 (3)
C1—C2—C3	121.2 (3)	C9—C10—C11	120.8 (4)
C2—C3—C7	118.8 (3)	C10—C11—C12	120.2 (4)
C2—C3—C4	119.0 (3)	C11—C12—C13	120.1 (4)
C4—C3—C7	122.2 (3)	C8—C13—C12	120.2 (4)
C3—C4—C5	120.4 (3)		

Table 5. *Hydrogen-bonding geometry* (Å, °) *for* (II)

D	H	A	$D\cdots A$	D—H$\cdots A$
N1	H101	O1$^{\rm i}$	3.119 (4)	166
N1	H201	O2$^{\rm ii}$	2.976 (4)	158

Symmetry codes: (i) $\frac{1}{2}-x, -\frac{1}{2}-y, 1-z$; (ii) $-x, -1-y, 1-z$.

Both structures were solved by direct methods using *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) and refined by full-matrix least-squares calculations using *SHELX*76 (Sheldrick, 1976). Non-H atoms were refined with anisotropic displacement parameters while the H atoms were positioned on geometrical grounds (C—H 1.0 Å), except those of the methyl and amino groups. The latter were found in a difference Fourier map and were refined isotropically. Data collection: *PWD*4 (Stoe & Cie, 1992*a*). Cell refinement: *PWD*4. Data reduction: *REDU*4*S* (Stoe & Cie, 1992*b*). Molecular graphics: *PLUTON* (Spek, 1982).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1058). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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