
Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OH1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dimethyl 2-Methylcubane-1,4-dicarboxylate and Dimethyl 2,3-Dimethylcubane-1,4-dicarboxylate

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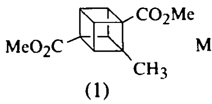
Abstract

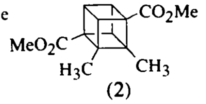
The title compounds, dimethyl 2-methylcubane-1,4-dicarboxylate, $C_{13}H_{14}O_4$, and dimethyl 2,3-dimethylcubane-1,4-dicarboxylate, $C_{14}H_{16}O_4$, are rare examples of alkyl-substituted cubanes. The 1,2-disubstituted cubane C—C bond lengths of both compounds are generally found to be longer than the other cubane C—C bonds. Both compounds show differences in the cubane C—C distances, as well as differences in the conformations of the methyl carboxylate groups.

Comment

The renaissance in cubane chemistry in recent years is in large part due to the discovery (Eaton & Castaldi, 1985) that amide-directed *ortho*-metallation methodology (Snieckus, 1990) could be employed to extend both the range and pattern of substitution of available cubanes (Eaton, Xiong & Gillardi, 1993). This approach, however, is not suitable for the introduction of alkyl groups onto the cubane nucleus. Herein, we report the structures of the title compounds, dimethyl 2-methylcubane-1,4-dicarboxylate, (1) (Lowe, Moorhouse, Walter & Tsanaktsidis, 1994), and dimethyl 2,3-dimethylcubane-1,4-dicarboxylate, (2) (Cristiano & Tsanaktsidis, 1994).



(1)



(2)

The centre of the cubane cage of the monomethyl derivative (1) lies on a centre of symmetry, which results in the methyl substitutent being disordered over two symmetry-related positions. The observed C—CH_3 bond length of 1.384 (6) Å is much shorter than the corresponding value of 1.494 (2) Å found in (2), and is presumably a consequence of this disorder. All other bond lengths are comparable to those found in other cubanes. The cubane C—C distances for (1) (1.550–1.574 Å) are generally found to be slightly shorter than the corresponding values found for (2) (1.553–1.581 Å). In addition, there are significant differences in the conformations of the methyl carboxylate groups, with the torsion angle C(2)—C(1)—C(9)—O(2*A*) being 92 (3)° for (1) and the corresponding value for (2) being −119.4 (2)°. For both compounds, the 1,2-disubstituted C—C distances involving the methyl carboxylate and the methyl groups, 1.574 (2) for (1) and 1.581 (2) Å for (2), are significantly longer than the other cubane C—C distances, while the bond involving the two methyl substitutents in (2) is somewhat shorter, being

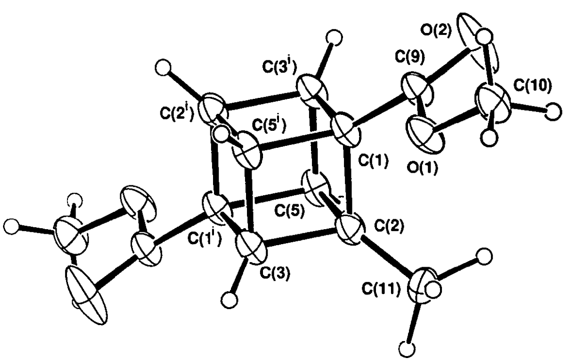


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1). Displacement ellipsoids are drawn at the 50% probability level. Symmetry transformation to generate equivalent atoms: (i) −*x*, −*y*, −*z*.

1.570 (2) Å in length. These distances are longer than that of 1.564 Å found for the 1,2-disubstituted C—C bonds in 2-methoxycarbonyl-1,4-dinitrocubane (Ammon & Bashir-Hashemi, 1993) and approach the values of 1.598 and 1.607 Å found for 1-*tert*-butyl-2-cubylcubane (Gilardi, Maggini & Eaton, 1988) and 1,4-[bis-(diisopropylamino)-methyl]-2,7-diphenylcubane (Bashir-Hashemi, Ammon & Choi, 1990).

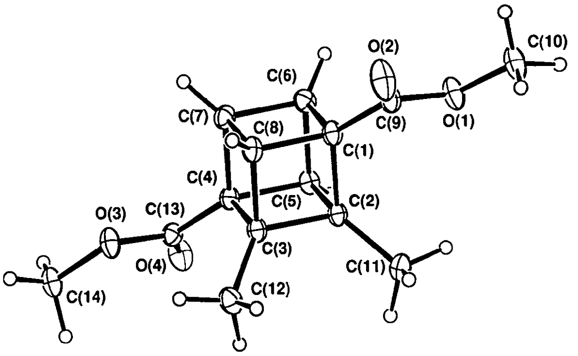


Fig. 2. *ORTEP*II (Johnson, 1976) drawing of (2). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Compound (1) was prepared by the method of Lowe, Moorhouse, Walter & Tsanaktsidis (1994) and compound (2) was prepared by the method of Cristiano & Tsanaktsidis (1994). Crystals of both compounds were obtained by slow evaporation from light petroleum.

Compound (1)

Crystal data

$C_{13}H_{14}O_4$
$M_r = 234.24$
Monoclinic
$P2_1/c$
$a = 7.9455\ (11)$ Å
$b = 5.8831\ (7)$ Å
$c = 12.560\ (2)$ Å
$\beta = 100.822\ (11)°$
$V = 576.67\ (14)$ Å^3
$Z = 2$
$D_x = 1.349$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8.0–19.0°$
$\mu = 0.100$ mm^{-1}
$T = 293\ (1)$ K
Chunky plate
0.40 × 0.35 × 0.21 mm
Colourless

Data collection

Enraf–Nonius CAD-4 MachS diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3152 measured reflections
1321 independent reflections
1069 observed reflections $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0154$
$\theta_{\max} = 27.47°$
$h = -10 \rightarrow 10$
$k = -1 \rightarrow 7$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 160 min
intensity decay: 10%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0465$
$wR(F^2) = 0.1233$
$S = 1.179$
1321 reflections
127 parameters
x, y, z of H(3) and H(5) refined; other H atoms riding (see below)
$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.1467P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$

$\Delta\rho_{\max} = 0.214$ e Å^{-3}
$\Delta\rho_{\min} = -0.218$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.051 (8)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
O(1)	−0.3200 (2)	−0.3152 (2)	0.09353 (11)	0.0671 (4)
O(2*A*)†	−0.343 (5)	0.037 (3)	0.154 (4)	0.115 (7)
O(2*B*)‡	−0.392 (2)	0.035 (3)	0.1153 (16)	0.069 (4)
O(2*C*)§	−0.406 (7)	0.035 (4)	0.101 (5)	0.072 (11)
C(1)	−0.1404 (2)	−0.0425 (3)	0.04325 (14)	0.0532 (5)
C(2)	0.0489 (2)	−0.1127 (3)	0.09386 (14)	0.0555 (5)
C(3)	0.0762 (2)	−0.1979 (3)	−0.01962 (15)	0.0560 (5)
C(5)	0.1104 (2)	0.1297 (3)	0.06953 (14)	0.0558 (5)
C(11)¶	0.0895 (5)	−0.2294 (9)	0.1910 (5)	0.0664 (13)
C(9)	−0.2912 (2)	−0.0944 (3)	0.09263 (14)	0.0555 (4)
C(10)	−0.4621 (3)	−0.3890 (4)	0.1403 (2)	0.0696 (6)

† Occupancy 0.46 (5).
‡ Occupancy 0.40 (7).
§ Occupancy 0.14 (7).
¶ Occupancy 0.5 (see below).

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

O(1)—C(9)	1.319 (2)	C(1)—C(2)	1.574 (2)
O(1)—C(10)	1.434 (2)	C(2)—C(11)	1.384 (6)
O(2*A*)—C(9)	1.216 (14)	C(2)—C(5)	1.556 (2)
O(2*B*)—C(9)	1.18 (2)	C(2)—C(3)	1.564 (2)
O(2*C*)—C(9)	1.21 (3)	C(3)—C(5$^{\text{i}}$)	1.550 (3)
C(1)—C(9)	1.480 (2)	C(3)—C(1$^{\text{i}}$)	1.551 (2)
C(1)—C(3$^{\text{i}}$)	1.551 (2)	C(5)—C(3$^{\text{i}}$)	1.550 (3)
C(1)—C(5$^{\text{i}}$)	1.566 (2)	C(5)—C(1$^{\text{i}}$)	1.566 (2)
C(9)—O(1)—C(10)	116.94 (14)	C(5$^{\text{i}}$)—C(3)—C(1$^{\text{i}}$)	90.60 (14)
C(9)—C(1)—C(3$^{\text{i}}$)	126.12 (14)	C(5$^{\text{i}}$)—C(3)—C(2)	89.98 (12)
C(9)—C(1)—C(5$^{\text{i}}$)	125.72 (15)	C(1$^{\text{i}}$)—C(3)—C(2)	89.36 (13)
C(3$^{\text{i}}$)—C(1)—C(5$^{\text{i}}$)	90.82 (12)	C(3$^{\text{i}}$)—C(5)—C(2)	90.28 (13)
C(9)—C(1)—C(2)	124.30 (15)	C(3$^{\text{i}}$)—C(5)—C(1$^{\text{i}}$)	90.90 (13)
C(3$^{\text{i}}$)—C(1)—C(2)	89.58 (12)	C(2)—C(5)—C(1$^{\text{i}}$)	89.10 (13)
C(5$^{\text{i}}$)—C(1)—C(2)	89.03 (12)	O(2*B*)—C(9)—O(1)	120.5 (7)
C(11)—C(2)—C(5)	126.7 (2)	O(2*C*)—C(9)—O(1)	119.1 (17)
C(11)—C(2)—C(3)	126.5 (2)	O(2*A*)—C(9)—O(1)	122.7 (8)
C(5)—C(2)—C(3)	90.71 (12)	O(2*B*)—C(9)—C(1)	127.4 (7)
C(11)—C(2)—C(1)	122.2 (2)	O(2*C*)—C(9)—C(1)	126.1 (11)
C(5)—C(2)—C(1)	89.53 (13)	O(2*A*)—C(9)—C(1)	122.0 (9)
C(3)—C(2)—C(1)	90.09 (13)	O(1)—C(9)—C(1)	111.26 (14)

Symmetry code: (i) $-x, -y, -z$.

Compound (2)

Crystal data

$C_{14}H_{16}O_4$
$M_r = 248.27$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Triclinic
$P\bar{1}$
$a = 6.5835\ (12)$ Å
$b = 9.487\ (2)$ Å
$c = 10.546\ (2)$ Å
$\alpha = 74.10\ (2)°$
$\beta = 83.81\ (2)°$
$\gamma = 87.89\ (2)°$
$V = 629.8\ (2)$ Å^3
$Z = 2$
$D_x = 1.309$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 9.5–17.5°$
$\mu = 0.096$ mm^{-1}
$T = 293\ (1)$ K
Thin plate
0.58 × 0.30 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4 MachS diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3687 measured reflections
2900 independent reflections
2281 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0082$
$\theta_{\rm max} = 27.47°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -2 \rightarrow 13$
3 standard reflections
frequency: 160 min
intensity decay: 17%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0439$
$wR(F^2) = 0.1256$
$S = 1.144$
2900 reflections
228 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 0.0854P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.001$

$\Delta\rho_{\rm max} = 0.341$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.250$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.056 (8)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
O(1)	0.3157 (2)	0.22326 (11)	0.44545 (10)	0.0515 (3)
O(2)	0.2600 (2)	0.00199 (13)	0.58404 (12)	0.0696 (4)
O(3)	0.6704 (2)	0.26555 (10)	1.04285 (10)	0.0469 (3)
O(4)	0.7304 (2)	0.48621 (11)	0.90249 (11)	0.0541 (3)
C(1)	0.4180 (2)	0.18423 (13)	0.65602 (13)	0.0357 (3)
C(2)	0.3297 (2)	0.32056 (13)	0.70355 (12)	0.0334 (3)
C(3)	0.3622 (2)	0.23338 (13)	0.84971 (12)	0.0336 (3)
C(4)	0.5833 (2)	0.30103 (13)	0.82844 (12)	0.0333 (3)
C(5)	0.5503 (2)	0.38515 (14)	0.68333 (12)	0.0350 (3)
C(6)	0.6377 (2)	0.25132 (15)	0.63494 (14)	0.0385 (3)
C(7)	0.6706 (2)	0.16545 (14)	0.78056 (13)	0.0382 (3)
C(8)	0.4512 (2)	0.10028 (14)	0.80099 (13)	0.0371 (3)
C(9)	0.3234 (2)	0.12367 (14)	0.56140 (13)	0.0391 (3)
C(10)	0.2223 (4)	0.1791 (2)	0.3455 (2)	0.0603 (5)
C(11)	0.1411 (2)	0.4046 (2)	0.6606 (2)	0.0458 (4)
C(12)	0.2097 (2)	0.2229 (2)	0.96750 (15)	0.0462 (4)
C(13)	0.6699 (2)	0.36389 (13)	0.92480 (12)	0.0342 (3)
C(14)	0.7393 (3)	0.3161 (2)	1.1480 (2)	0.0503 (4)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

O(1)—C(9)	1.329 (2)	C(2)—C(5)	1.568 (2)
O(1)—C(10)	1.437 (2)	C(2)—C(3)	1.570 (2)
O(2)—C(9)	1.195 (2)	C(3)—C(12)	1.494 (2)
O(3)—C(13)	1.336 (2)	C(3)—C(8)	1.561 (2)
O(3)—C(14)	1.443 (2)	C(3)—C(4)	1.581 (2)
O(4)—C(13)	1.194 (2)	C(4)—C(13)	1.482 (2)
C(1)—C(9)	1.479 (2)	C(4)—C(5)	1.554 (2)
C(1)—C(8)	1.553 (2)	C(4)—C(7)	1.574 (2)
C(1)—C(6)	1.571 (2)	C(5)—C(6)	1.563 (2)
C(1)—C(2)	1.581 (2)	C(6)—C(7)	1.563 (2)
C(2)—C(11)	1.494 (2)	C(7)—C(8)	1.562 (2)
C(9)—O(1)—C(10)	116.70 (12)	C(5)—C(4)—C(7)	90.13 (10)
C(13)—O(3)—C(14)	116.52 (11)	C(13)—C(4)—C(3)	124.23 (11)
C(9)—C(1)—C(8)	125.67 (11)	C(5)—C(4)—C(3)	90.56 (9)
C(9)—C(1)—C(6)	126.10 (11)	C(7)—C(4)—C(3)	89.22 (9)
C(8)—C(1)—C(6)	90.47 (10)	C(4)—C(5)—C(6)	90.18 (9)
C(9)—C(1)—C(2)	123.84 (11)	C(4)—C(5)—C(2)	90.01 (9)
C(8)—C(1)—C(2)	90.35 (9)	C(6)—C(5)—C(2)	90.17 (9)
C(6)—C(1)—C(2)	89.39 (9)	C(5)—C(6)—C(7)	90.25 (10)
C(11)—C(2)—C(5)	125.75 (11)	C(5)—C(6)—C(1)	90.50 (10)
C(11)—C(2)—C(3)	125.33 (12)	C(7)—C(6)—C(1)	89.22 (10)
C(5)—C(2)—C(3)	90.47 (9)	C(8)—C(7)—C(6)	90.42 (10)
C(11)—C(2)—C(1)	125.16 (11)	C(8)—C(7)—C(4)	90.31 (10)
C(5)—C(2)—C(1)	89.94 (9)	C(6)—C(7)—C(4)	89.44 (9)
C(3)—C(2)—C(1)	88.93 (9)	C(1)—C(8)—C(3)	90.27 (9)
C(12)—C(3)—C(8)	125.01 (11)	C(1)—C(8)—C(7)	89.89 (10)
C(12)—C(3)—C(2)	125.14 (11)	C(3)—C(8)—C(7)	90.38 (9)
C(8)—C(3)—C(2)	90.44 (9)	O(2)—C(9)—O(1)	123.28 (13)
C(12)—C(3)—C(4)	126.00 (11)	O(2)—C(9)—C(1)	125.55 (13)
C(8)—C(3)—C(4)	90.09 (9)	O(1)—C(9)—C(1)	111.17 (11)
C(2)—C(3)—C(4)	88.96 (9)	O(4)—C(13)—O(3)	123.43 (12)
C(13)—C(4)—C(5)	125.48 (10)	O(4)—C(13)—C(4)	125.49 (12)
C(13)—C(4)—C(7)	126.14 (10)	O(3)—C(13)—C(4)	111.07 (10)

Refinement was performed using *SHELXL*93 (Sheldrick, 1993) by full-matrix least squares, with anisotropic displacement parameters for all non-H atoms, and isotropic for the H atoms. For (1), O(2) was found to be disordered and was refined over three sites with site occupation factors refining to 0.46 (5), 0.40 (7) and 0.14 (7). The positional parameters of H(3) and H(5) were refined. All other H atoms were included at calculated positions and refined using a riding model; H atoms on C(10) were refined over two sites with relative site occupancies of 0.66 (4) and 0.34 (4), while the C(2)—H(2) bond length was restrained at 0.98 Å. C(11) and H(2) were each given site occupancies of 0.5. All calculations were carried out on a VAXstation 4000VLC computer system.

For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KH1021). 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. (1995). C**51**, 1661–1663

3-(3-Nitroanilino)cyclohex-2-en-1-one: an Unexpected Product from the Co-Crystallization of *m*-Nitroaniline with 1,3-Cyclohexanedione

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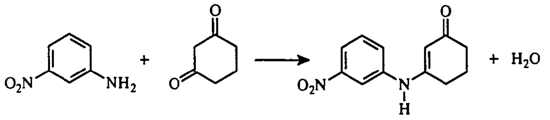
Abstract

The title compound, $C_{12}H_{12}N_2O_3$, is composed of a nitroanilino moiety and a cyclic α,β-unsaturated ketone moiety. The nitro group is oriented *anti* to the carbonyl group and the cyclic ketone ring is in a puckered conformation with C9 as the out-of-plane atom. The shortened C11—N2 and C7—C12 bond lengths suggest that the amino group is conjugated with the α,β-unsaturated ketone group. Intermolecular hydrogen bonds between the carbonyl O atom and the amino group link the molecules into infinite chains [N—H 0.88 (3), H$\cdots$O$^{\rm i}$ 1.98 (3), N$\cdots$O$^{\rm i}$ 2.858 (3) Å, N—H$\cdots$O$^{\rm i}$ 175 (1)°; symmetry code: (i) $1+x$, y, z].

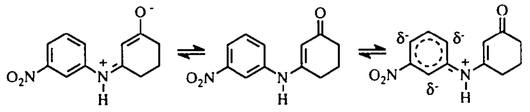
Comment

Hydrogen-bond directed co-crystallization has been demonstrated as a useful way to study the molecular recognition and hydrogen-bond properties of a class of related host molecules (Etter & Baures, 1988; Etter, Urbanczyk-Lipkowska, Zia-Ebrahimi & Panunto, 1990; Etter & Reutzel, 1991). As part of a comprehensive study of the hydrogen-bond properties of nitroanilines (Panunto, Urbanczyk-Lipkowska, Johnson & Etter, 1987; Etter & Huang, 1992), the title compound, (I), was obtained unexpectedly during an attempt to prepare an *m*-nitroaniline/1,3-cyclohexanedione co-crystal. The reaction is believed to proceed by a standard enamine formation in which nucleophilic addition to the carbonyl group of 1,3-cyclohexanedione by the amino group of *m*-nitroaniline is followed by dehydration, as shown in the scheme below.



(I)

The title compound is composed of a nitroanilino moiety and a cyclic α,β-unsaturated ketone moiety. The cyclic ketone ring is in a puckered conformation where C9 is out of the plane of the remaining atoms, a common conformation for cyclohexyl rings containing three adjacent sp^2-hybridized C atoms and three adjacent sp^3-hybridized C atoms (Katrusiak, 1990). The anilino group and the α,β-unsaturated ketone group are not coplanar. The nitro group is oriented *anti* to the carbonyl group and is at an angle of 28.3° to the phenyl ring least-squares plane, and the phenyl ring least-squares plane is at an angle of 32.8° to the α,β-unsaturated ketone least-squares plane to reduce the H atom–H atom repulsion between C5 and C12. The C11—N2 bond distance of 1.366 (3) Å is considerably shorter than that of C4—N2 [1.400 (3) Å], suggesting that the lone pair of electrons in the amino group is more easily delocalized into the cyclic α,β-unsaturated ketone group than into the nitrophenyl group; thus, the resonance form *A* is more favorable than the resonance form *B*, shown below.



Form *A* Form *B*

(II)

This is understandable since the nitro group in the *meta* position cannot interact directly with the charge in the ring. Because of the resonance effect, the C7—C12 bond distance of 1.423 (3) Å is much shorter than a normal sp^2–sp^2 C—C single bond [1.48 Å] (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987), C7—C8 [1.503 (4) Å] or C10—C11 [1.499 (3) Å]. The molecules form infinite chains *via* intermolecular hydrogen bonds between the carbonyl O atom and the amino group [N—H 0.88 (3), H$\cdots$O$^{\rm i}$ 1.98 (3), N$\cdots$O$^{\rm i}$ 2.858 (3) Å, N—H$\cdots$O$^{\rm i}$ 175 (1)°; symmetry code: (i) $1+x$, y, z]. This type of hydrogen-bond pattern is designated $C_1^1(6)$ according to Etter notation (Etter, MacDonald & Bernstein, 1990). [This notation indicates the chain nature (*C*) of the hydrogen-bonded aggregates, the one proton donor and one proton acceptor (as subscript and superscript) involved in the bonding and the chain length (in parentheses).]

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