Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Ethyl 3-[(3,5-dimethylphenyl)aminocarbonyl]propanoate

**B. Thimme Gowda,[a]* Sabine Foro,[b] B. S. Saraswathi[a] and Hartmut Fuess[b]**

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

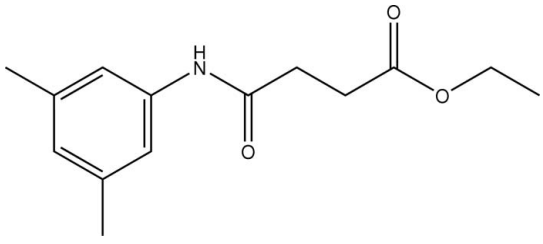
Received 22 July 2009; accepted 24 July 2009

Key indicators: single-crystal X-ray study; $T$ = 299 K; mean $\sigma$(C–C) = 0.003 Å; some non-H atoms missing; $R$ factor = 0.058; $wR$ factor = 0.169; data-to-parameter ratio = 11.4.

The non-H atoms in the title compound, $C_{14}H_{19}NO_3$, lie on a mirror plane. The amide O and ester carbonyl O atoms are *trans* to each other. Furthermore, the C=O and O—$CH_2$ bonds of the ester group are *syn* with respect to each other. In the crystal, molecules are packed into centrosymmetric dimers through intermolecular N—H···O hydrogen bonds.

## Related literature

For related structures, see: Gowda *et al.* (2009*a*,*b*,*c*).



## Experimental

### Crystal data

$C_{14}H_{19}NO_3$ $M_r$ = 249.30
Tetragonal, $I4/m$
$a$ = 19.938 (2) Å
$c$ = 7.0367 (9) Å
$V$ = 2797.3 (5) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 0.67 mm$^{-1}$
$T$ = 299 K
0.40 × 0.28 × 0.25 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
4040 measured reflections
1367 independent reflections
1201 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.169
$S$ = 1.11
1367 reflections
120 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$N$···O2$^{i}$ | 0.85 (3) | 2.15 (3) | 2.995 (3) | 176 (3) |

Symmetry code: (i) $-x+1, -y, -z$.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

BTG thanks the Alexander von Humboldt Foundation, Bonn, Germany, for a resumption of his research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2515).

## References

Enraf–Nonius (1996). *CAD-4-PC*. Enraf–Nonius, Delft, The Netherlands.
Gowda, B. T., Foro, S., Saraswathi, B. S., Terao, H. & Fuess, H. (2009*a*). *Acta Cryst.* E**65**, o399.
Gowda, B. T., Foro, S., Saraswathi, B. S., Terao, H. & Fuess, H. (2009*b*). *Acta Cryst.* E**65**, o466.
Gowda, B. T., Foro, S., Saraswathi, B. S., Terao, H. & Fuess, H. (2009*c*). *Acta Cryst.* E**65**, o873.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Stoe & Cie (1987). *REDU4*. Stoe & Cie GmbH, Darmstadt, Germany.

# supporting information

*Acta Cryst.* (2009). E**65**, o2039 [doi:10.1107/S1600536809029511]

## Ethyl 3-[(3,5-dimethylphenyl)aminocarbonyl]propanoate

**B. Thimme Gowda, Sabine Foro, B. S. Saraswathi and Hartmut Fuess**

### S1. Comment

As a part of studying the effect of ring and side chain substitutions on the structures of the substituted amides (Gowda *et al.*, 2009*a,b,c*), the crystal structure of ethyl *N*-(3,5-dimethylphenyl)succinamate (I) has been determined. The non-hydrogen atoms lie on a crystallographic mirror plane. The conformations of N—H and C=O bonds in the amide segment of the structure are *trans* to each other (Fig. 1). Likewise, the amide-O atom and ester carbonyl-O atoms are *trans* to each other. The C=O and O—$CH_2$ bonds of the ester group are in *syn* positions to each other, similar to that observed between the C=O and O—H bonds in the crystal structures of *N*-(2,6-dimethylphenyl)succinamic acid (Gowda *et al.*, 2009*b*) and *N*-(2-chlorophenyl)succinamic acid (Gowda *et al.*, 2009*a*).

The presence of N—H···O hydrogen bonds connect the molecules into centrosymmetric dimers (Table 1).

### S2. Experimental

A solution of succinic anhydride (0.025 mole) in toluene (25 ml) was treated dropwise with a solution of 3,5-dimethyl-aniline (0.025 mole) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about one hour and set aside for an additional hour at room temperature for the completion of reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 3,5-dimethylaniline. The resultant solid *N*-(3,5-dimethylphenyl)-succinamic acid was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. *N*-(3,5-Dimethylphenyl)succinamic acid was recrystallized into ethyl *N*-(3,5-dimethylphenyl)-succinamate (I) from hot ethanol. The rod like colourless single crystals of (I) were grown in hot ethanolic solution by slow evaporation.

### S3. Refinement

The H atoms of the NH group, of C11 and C12 were located in a difference map and their position refined [N—H = 0.85 (3) Å, C—H = 0.98 (4)–1.03 (3) Å]. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93–0.97 Å]. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the $U_{eq}$ of the parent atom).

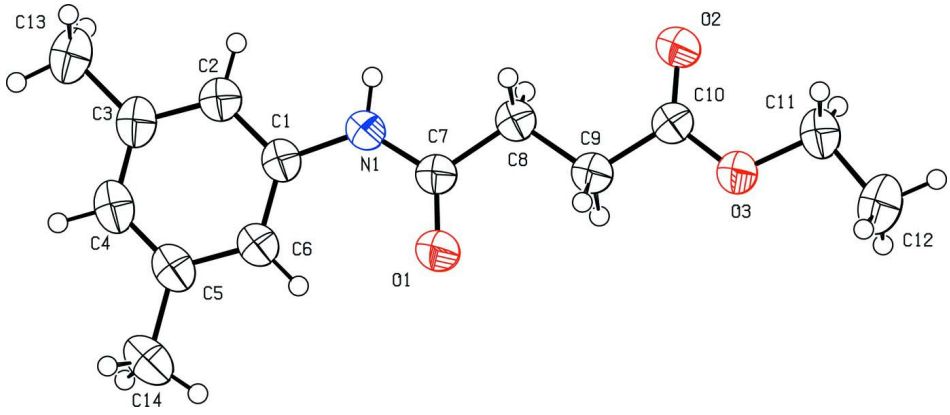



**Figure 1**

Molecular structure of (I), showing the atom labelling and the displacement ellipsoids are at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.

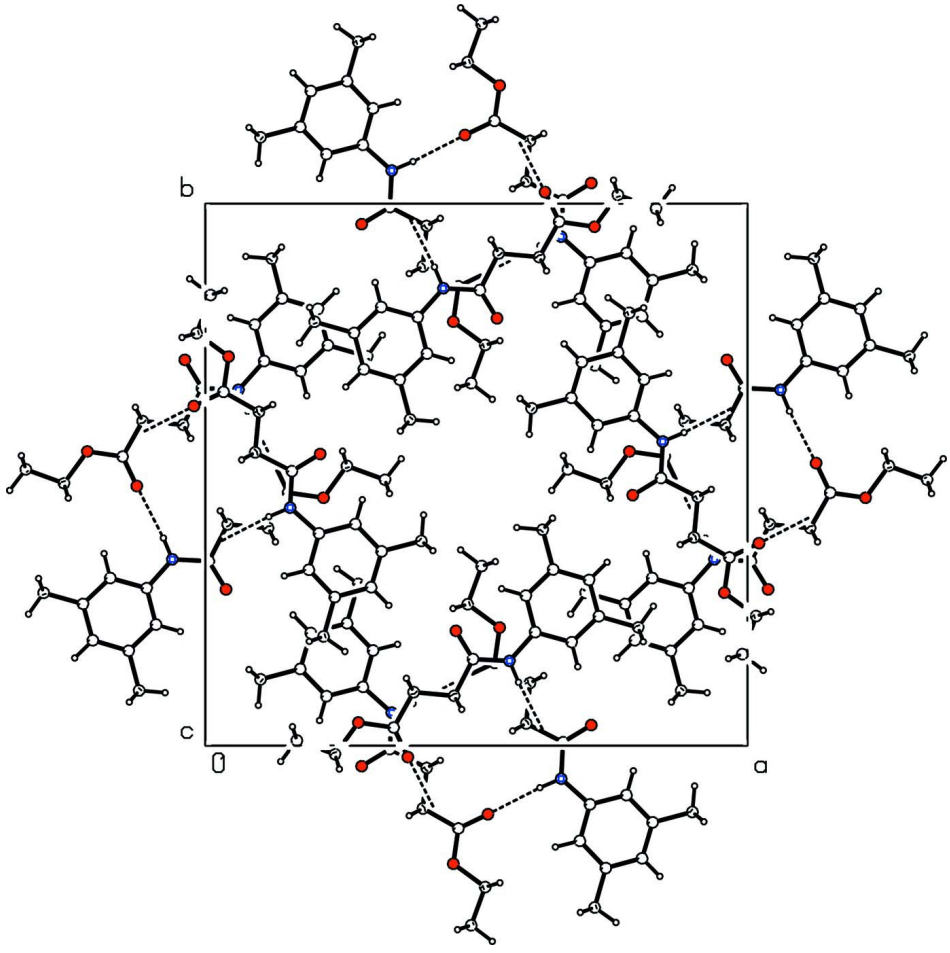



**Figure 2**

Molecular packing of (I) with hydrogen bonds shown as dashed lines.

**Ethyl 3-[(3,5-dimethylphenyl)aminocarbonyl]propanoate**

*Crystal data*

$C_{14}H_{19}NO_3$
$M_r = 249.30$
Tetragonal, $I4/m$
Hall symbol: -I 4
$a = 19.938$ (2) Å
$c = 7.0367$ (9) Å
$V = 2797.3$ (5) Å$^3$
$Z = 8$
$F(000) = 1072$
$D_x = 1.184$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 4.4$–$20.5°$
$\mu = 0.67$ mm$^{-1}$
$T = 299$ K
Rod, colourless
0.40 × 0.28 × 0.25 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
4040 measured reflections
1367 independent reflections
1201 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 67.0°$, $\theta_{min} = 3.1°$
$h = -16\rightarrow23$
$k = -16\rightarrow23$
$l = -3\rightarrow8$
3 standard reflections every 120 min
intensity decay: 1.0%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.169$
$S = 1.11$
1367 reflections
120 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1022P)^2 + 1.0554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0012 (3)

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.60839 (11) | 0.20945 (11) | 0.0000 | 0.0465 (6) | |
| C2 | 0.67561 (12) | 0.19085 (12) | 0.0000 | 0.0513 (6) | |
| H2 | 0.6869 | 0.1456 | 0.0000 | 0.062* | |

| | | | | | |
|---|---|---|---|---|---|
| C3 | 0.72584 (12) | 0.23863 (13) | 0.0000 | 0.0532 (6) | |
| C4 | 0.70778 (13) | 0.30566 (14) | 0.0000 | 0.0579 (7) | |
| H4 | 0.7411 | 0.3383 | 0.0000 | 0.070* | |
| C5 | 0.64153 (13) | 0.32504 (12) | 0.0000 | 0.0562 (7) | |
| C6 | 0.59123 (12) | 0.27666 (12) | 0.0000 | 0.0522 (6) | |
| H6 | 0.5464 | 0.2894 | 0.0000 | 0.063* | |
| C7 | 0.49362 (12) | 0.15957 (11) | 0.0000 | 0.0506 (6) | |
| C8 | 0.45900 (11) | 0.09187 (12) | 0.0000 | 0.0543 (7) | |
| H8A | 0.4728 | 0.0668 | −0.1115 | 0.065* | 0.50 |
| H8B | 0.4728 | 0.0668 | 0.1115 | 0.065* | 0.50 |
| C9 | 0.38416 (12) | 0.09873 (11) | 0.0000 | 0.0515 (6) | |
| H9A | 0.3708 | 0.1242 | 0.1113 | 0.062* | 0.50 |
| H9B | 0.3708 | 0.1242 | −0.1113 | 0.062* | 0.50 |
| C10 | 0.34741 (11) | 0.03335 (12) | 0.0000 | 0.0491 (6) | |
| C11 | 0.23912 (14) | −0.01607 (15) | 0.0000 | 0.0666 (8) | |
| H11 | 0.2470 (10) | −0.0432 (11) | 0.118 (3) | 0.080* | |
| C12 | 0.16867 (16) | 0.0096 (2) | 0.0000 | 0.0849 (10) | |
| H12A | 0.139 (2) | −0.029 (2) | 0.0000 | 0.102* | |
| H12B | 0.1605 (12) | 0.0349 (13) | 0.126 (4) | 0.102* | |
| C13 | 0.79850 (13) | 0.21804 (16) | 0.0000 | 0.0652 (7) | |
| H13A | 0.8077 | 0.1916 | −0.1110 | 0.078* | 0.50 |
| H13B | 0.8078 | 0.1920 | 0.1118 | 0.078* | 0.50 |
| H13C | 0.8263 | 0.2573 | −0.0007 | 0.078* | |
| C14 | 0.62237 (18) | 0.39835 (14) | 0.0000 | 0.0829 (10) | |
| H14A | 0.6403 | 0.4196 | −0.1113 | 0.099* | 0.50 |
| H14B | 0.6402 | 0.4196 | 0.1115 | 0.099* | 0.50 |
| H14C | 0.5744 | 0.4024 | −0.0003 | 0.099* | |
| N1 | 0.56095 (10) | 0.15635 (10) | 0.0000 | 0.0519 (6) | |
| H1N | 0.5776 (15) | 0.1170 (16) | 0.0000 | 0.062* | |
| O1 | 0.46202 (9) | 0.21173 (9) | 0.0000 | 0.0821 (8) | |
| O2 | 0.37347 (9) | −0.02118 (8) | 0.0000 | 0.0701 (7) | |
| O3 | 0.28217 (8) | 0.04272 (8) | 0.0000 | 0.0614 (6) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0444 (12) | 0.0435 (12) | 0.0516 (12) | −0.0050 (9) | 0.000 | 0.000 |
| C2 | 0.0475 (13) | 0.0481 (13) | 0.0583 (13) | −0.0009 (9) | 0.000 | 0.000 |
| C3 | 0.0465 (12) | 0.0632 (15) | 0.0498 (12) | −0.0103 (10) | 0.000 | 0.000 |
| C4 | 0.0567 (15) | 0.0593 (15) | 0.0578 (14) | −0.0177 (11) | 0.000 | 0.000 |
| C5 | 0.0621 (15) | 0.0464 (13) | 0.0601 (14) | −0.0105 (10) | 0.000 | 0.000 |
| C6 | 0.0494 (13) | 0.0426 (12) | 0.0645 (14) | −0.0036 (9) | 0.000 | 0.000 |
| C7 | 0.0433 (12) | 0.0411 (12) | 0.0673 (15) | 0.0014 (9) | 0.000 | 0.000 |
| C8 | 0.0432 (13) | 0.0422 (13) | 0.0775 (16) | −0.0015 (9) | 0.000 | 0.000 |
| C9 | 0.0447 (13) | 0.0425 (12) | 0.0673 (15) | 0.0006 (9) | 0.000 | 0.000 |
| C10 | 0.0430 (12) | 0.0449 (12) | 0.0594 (14) | 0.0013 (9) | 0.000 | 0.000 |
| C11 | 0.0497 (14) | 0.0559 (15) | 0.094 (2) | −0.0124 (11) | 0.000 | 0.000 |
| C12 | 0.0490 (16) | 0.094 (3) | 0.112 (3) | −0.0109 (15) | 0.000 | 0.000 |

| | | | | | | |
|---|---|---|---|---|---|---|
| C13 | 0.0460 (14) | 0.0815 (19) | 0.0681 (16) | −0.0094 (12) | 0.000 | 0.000 |
| C14 | 0.083 (2) | 0.0443 (15) | 0.121 (3) | −0.0118 (13) | 0.000 | 0.000 |
| N1 | 0.0424 (11) | 0.0367 (10) | 0.0768 (14) | −0.0013 (8) | 0.000 | 0.000 |
| O1 | 0.0481 (10) | 0.0416 (10) | 0.157 (2) | 0.0021 (7) | 0.000 | 0.000 |
| O2 | 0.0514 (10) | 0.0397 (10) | 0.1193 (18) | 0.0015 (7) | 0.000 | 0.000 |
| O3 | 0.0420 (9) | 0.0458 (9) | 0.0965 (14) | −0.0017 (7) | 0.000 | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.383 (3) | C9—H9A | 0.9700 |
| C1—C2 | 1.391 (3) | C9—H9B | 0.9700 |
| C1—N1 | 1.420 (3) | C10—O2 | 1.205 (3) |
| C2—C3 | 1.382 (3) | C10—O3 | 1.314 (3) |
| C2—H2 | 0.9300 | C11—H11$^{i}$ | 1.00 (2) |
| C3—C4 | 1.384 (4) | C11—O3 | 1.453 (3) |
| C3—C13 | 1.506 (4) | C11—C12 | 1.495 (4) |
| C4—C5 | 1.376 (4) | C11—H11 | 1.00 (2) |
| C4—H4 | 0.9300 | C12—H12B$^{i}$ | 1.03 (3) |
| C5—C6 | 1.392 (3) | C12—H12A | 0.98 (4) |
| C5—C14 | 1.511 (4) | C12—H12B | 1.03 (3) |
| C6—H6 | 0.9300 | C13—H13A | 0.9600 |
| C7—O1 | 1.216 (3) | C13—H13B | 0.9600 |
| C7—N1 | 1.344 (3) | C13—H13C | 0.9600 |
| C7—C8 | 1.516 (3) | C14—H14A | 0.9600 |
| C8—C9 | 1.498 (3) | C14—H14B | 0.9600 |
| C8—H8A | 0.9700 | C14—H14C | 0.9600 |
| C8—H8B | 0.9700 | N1—H1N | 0.85 (3) |
| C9—C10 | 1.495 (3) | | |
| | | | |
| C6—C1—C2 | 119.8 (2) | H9A—C9—H9B | 107.6 |
| C6—C1—N1 | 123.9 (2) | O2—C10—O3 | 123.7 (2) |
| C2—C1—N1 | 116.3 (2) | O2—C10—C9 | 125.1 (2) |
| C3—C2—C1 | 121.0 (2) | O3—C10—C9 | 111.17 (19) |
| C3—C2—H2 | 119.5 | H11$^{i}$—C11—O3 | 110.1 (12) |
| C1—C2—H2 | 119.5 | H11$^{i}$—C11—C12 | 109.4 (12) |
| C2—C3—C4 | 118.5 (2) | O3—C11—C12 | 106.2 (2) |
| C2—C3—C13 | 120.6 (3) | H11$^{i}$—C11—H11 | 112 (3) |
| C4—C3—C13 | 120.9 (2) | O3—C11—H11 | 110.1 (12) |
| C5—C4—C3 | 121.4 (2) | C12—C11—H11 | 109.4 (12) |
| C5—C4—H4 | 119.3 | H12B$^{i}$—C12—C11 | 108.4 (14) |
| C3—C4—H4 | 119.3 | H12B$^{i}$—C12—H12A | 106.9 (16) |
| C4—C5—C6 | 119.8 (2) | C11—C12—H12A | 107 (2) |
| C4—C5—C14 | 121.0 (2) | H12B$^{i}$—C12—H12B | 118 (3) |
| C6—C5—C14 | 119.2 (3) | C11—C12—H12B | 108.4 (14) |
| C1—C6—C5 | 119.6 (2) | H12A—C12—H12B | 106.9 (16) |
| C1—C6—H6 | 120.2 | C3—C13—H13A | 109.5 |
| C5—C6—H6 | 120.2 | C3—C13—H13B | 109.5 |
| O1—C7—N1 | 123.9 (2) | H13A—C13—H13B | 109.5 |

| | | | |
|---|---|---|---|
| O1—C7—C8 | 121.7 (2) | C3—C13—H13C | 109.5 |
| N1—C7—C8 | 114.3 (2) | H13A—C13—H13C | 109.5 |
| C9—C8—C7 | 111.8 (2) | H13B—C13—H13C | 109.5 |
| C9—C8—H8A | 109.2 | C5—C14—H14A | 109.5 |
| C7—C8—H8A | 109.2 | C5—C14—H14B | 109.5 |
| C9—C8—H8B | 109.2 | H14A—C14—H14B | 109.5 |
| C7—C8—H8B | 109.2 | C5—C14—H14C | 109.5 |
| H8A—C8—H8B | 107.9 | H14A—C14—H14C | 109.5 |
| C10—C9—C8 | 114.1 (2) | H14B—C14—H14C | 109.5 |
| C10—C9—H9A | 108.7 | C7—N1—C1 | 129.0 (2) |
| C8—C9—H9A | 108.7 | C7—N1—H1N | 116 (2) |
| C10—C9—H9B | 108.7 | C1—N1—H1N | 115 (2) |
| C8—C9—H9B | 108.7 | C10—O3—C11 | 118.0 (2) |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 0.0 | C7—C8—C9—C10 | 180.0 |
| N1—C1—C2—C3 | 180.0 | C8—C9—C10—O2 | 0.0 |
| C1—C2—C3—C4 | 0.0 | C8—C9—C10—O3 | 180.0 |
| C1—C2—C3—C13 | 180.0 | H11$^{i}$—C11—C12—H12B$^{i}$ | −54 (2) |
| C2—C3—C4—C5 | 0.0 | O3—C11—C12—H12B$^{i}$ | 64.8 (16) |
| C13—C3—C4—C5 | 180.0 | O1—C7—N1—C1 | 0.0 |
| C3—C4—C5—C6 | 0.0 | C8—C7—N1—C1 | 180.0 |
| C3—C4—C5—C14 | 180.0 | C6—C1—N1—C7 | 0.0 |
| C2—C1—C6—C5 | 0.0 | C2—C1—N1—C7 | 180.0 |
| N1—C1—C6—C5 | 180.0 | O2—C10—O3—C11 | 0.0 |
| C4—C5—C6—C1 | 0.0 | C9—C10—O3—C11 | 180.0 |
| C14—C5—C6—C1 | 180.0 | H11$^{i}$—C11—O3—C10 | −61.7 (13) |
| O1—C7—C8—C9 | 0.0 | C12—C11—O3—C10 | 180.0 |
| N1—C7—C8—C9 | 180.0 | | |

Symmetry code: (i) *x*, *y*, −*z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O2$^{ii}$ | 0.85 (3) | 2.15 (3) | 2.995 (3) | 176 (3) |

Symmetry code: (ii) −*x*+1, −*y*, −*z*.