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# *rac*-Ethyl (2*Z*)-3-{2-[(*Z*)-4-ethoxy-4-oxobut-2-en-2-ylamino]cyclohexylamino}but-2-enoate

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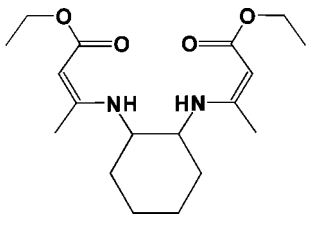
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.034; $wR$ factor = 0.106; data-to-parameter ratio = 15.4.

The asymmetric unit of the title compound, $C_{18}H_{30}N_2O_4$, contains two independent molecules. In each molecule, the cyclohexane ring adopts a chair conformation with equatorial orientation of the substituents, and the conformation is stabilized by two intramolecular N—H···O hydrogen bonds, forming rings of $S$(6) graph-set motif. One ethoxy group and one ethyl group are disordered over two sets of sites with refined occupancy ratios of 0.704 (2):0.296 (2) and 0.505 (3):0.495 (3), respectively. In the crystal, a weak intermolecular C—H···O hydrogen interaction is observed, involving the O atom of the major component of the disordered ethoxy group.

## Related literature

For the synthesis and applications of $\beta$-enaminoesters, see: Spivey *et al.* (2003); Eddington *et al.* (2003); Elaridi, Thaqi *et al.* (2005); Cornils & Herrmann (1996); Venter *et al.* (2009); Elaridi, Jackson & Robinson (2005); Harrad *et al.* (2010). For related structures, see: McCann *et al.* (2001); Huang *et al.* (2008). For puckering parameters, see: Cremer & Pople (1975). For graph-set notation, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{18}H_{30}N_2O_4$
$M_r$ = 338.44
Triclinic, $P\overline{1}$
$a$ = 11.1424 (9) Å
$b$ = 12.7445 (6) Å
$c$ = 16.3298 (11) Å
$\alpha$ = 90.904 (6)°
$\beta$ = 109.222 (6)°
$\gamma$ = 114.048 (5)°
$V$ = 1969.1 (3) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.65 mm$^{-1}$
$T$ = 294 K
0.18 × 0.15 × 0.10 mm

### Data collection

Siemens AED diffractometer
7461 measured reflections
7189 independent reflections
5826 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.006
3 standard reflections every 100 reflections
intensity decay: 0.02%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.106
$S$ = 1.16
7189 reflections
467 parameters
8 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*···O1 | 0.850 (13) | 2.048 (14) | 2.754 (2) | 140.1 (12) |
| N2—H2*N*···O3 | 0.865 (12) | 2.009 (12) | 2.720 (2) | 138.7 (13) |
| N3—H3*N*···O5 | 0.846 (17) | 2.018 (15) | 2.724 (2) | 140.3 (12) |
| N4—H4*N*···O7 | 0.811 (17) | 2.112 (15) | 2.754 (2) | 136.1 (14) |
| C4—H4*A*···O6*A*$^{i}$ | 0.97 | 2.48 | 3.400 (3) | 158 |

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

Data collection: *AED* (Belletti *et al.*, 1993); cell refinement: *AED*; data reduction: *AED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *SCHAKAL97* (Keller, 1997); software used to prepare material for publication: *SHELXL97* and *PARST95* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2371).

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# supplementary materials

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# *rac*-Ethyl (2*Z*)-3-{2-[(*Z*)-4-ethoxy-4-oxobut-2-en-2-ylamino]cyclohexylamino}but-2-enoate

**M. A. Harrad, B. Boualy, M. A. Ali, L. E. Firdoussi and C. Rizzoli**

## Comment

β-Enaminoesters have been extensively studied because of their applications in pharmaceutical, biochemical, biomedical and immunochemical research (Spivey *et al.*, 2003; Eddington *et al.*, 2003). These compounds are also used as precursors for the preparation of a large number of heterocyclic derivatives (Elaridi, Thaqi *et al.*, 2005) and novel organometallic complexes (Cornils & Herrmann, 1996; Venter *et al.*, 2009). Furthermore, chiral β-amino acid derivatives were prepared by enantioselective hydrogenation of β-enaminoesters (Elaridi, Jackson & Robinson, 2005). In the course of our studies in this field, we have recently reported an efficient method for the synthesis of various β-enaminoesters (Harrad *et al.*, 2010). Following our catalysis objective on the coupling of amines and keto-ester compounds, we describe herein the crystal structure of a new β-enaminoester which has been prepared using our previously mentioned method by the dicondensation of trans-cyclohexane-1,2-diamine with 3-oxo-butyric acid ethyl ester under solvent-free conditions.

The asymmetric unit of the title compound (Fig. 1) consists of two independent molecules differing mainly in the orientation of the aminobutenoate groups (Fig. 2), as indicated by the dihedral angles of 82.23 (3) and 51.57 (3)° between the mean planes through N1/O1/O2/C8—C10 and N2/O3/O4/C13—C16 in one molecule, and through N3/O5/O6A/O6B/C25—C28 and N4/O7/O8/C31—C24 in the other molecule. The molecular conformations, where the substituents are equatorially oriented with respect to the cyclohexane rings, are similar to those observed for the related compound *trans*-*N*,*N*'-bis(4-oxopent-2-en-2-yl)-1,2-diaminocyclohexane (McCann *et al.*, 2001; Huang *et al.*, 2009). In each aminobutenoate group an intramolecular N—H···O hydrogen bond (Table 1) is present, forming a ring of *S*(6) graph-set motif (Bernstein *et al.*, 1995). The cyclohexane rings adopt a chair conformations with puckering parameters *Q*, θ and φ (Cremer & Pople, 1975) of 0.5691 (11) Å, 179.21 (19)°, -105 (8)° and 0.5723 (15) Å, 178.86 (15)°, -67 (8)° for rings C1–C6 and C19–C24, respectively. In one molecule, the O6/C29–C30 ethoxy group and the C35–C36 ethyl group are disordered over two orientations with refined site occupancy ratios of 0.704 (2):0.296 (2) and 0.505 (3):0.495 (3), respectively. In the crystal structure (Fig. 3), one weak C—H···O hydrogen bond (Table 1) is observed, involving the oxygen atom of the major component of the disordered ethoxy group as acceptor.

## Experimental

To a stirred mixture of 3-oxo-butyric acid ethyl ester (1.7 mmol), *trans*-cyclohexane-1,2-diamine (0.85 mmol) and $Ca(CF_3COO)_2$ (0.17 mmol) at room temperature 10 ml of distilled water was added, and the residue extracted with diethyl ether (3 × 25 ml). The organic layer was dried over $Na_2SO_4$ and the solvent removed under reduced pressure. The title β-enaminoester was obtained by column chromatography over silica gel using a mixture of *n*-hexane/ethyl acetate (5:95 *v*/*v*) as eluent (yield 90%; m. p. 160 °C). Crystals suitable for X-ray analysis were obtained on slow evaporation of the solvent at room temperature.

### Refinement

The amine H atoms were located in a difference Fourier map and refined freely. All other H atoms were placed at calculated positions and refined using a riding model approximation, with C—H = 0.93–0.98 Å, and with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for methyl H atoms. One ethoxy group (O6/C29–C30) and one ethyl group (C35–C36) are disordered over two orientations (called A and B) with refined site occupancy ratios of 0.704 (2):0.296 (2) and 0.505 (3):0.495 (3), respectively. During the refinement, the O—C and C—C bond distances within the disordered groups were restrained to be 1.45 (1) and 1.49 (1) Å, respectively, and the anisotropic displacement parameters of the pairs of the disordered atoms were set equal by the command EADP (Sheldrick, 2008).

### Figures

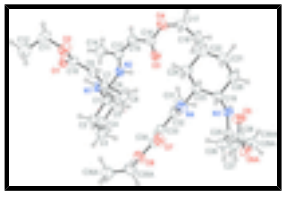

Fig. 1. The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level. Only the major components of disorder are shown.

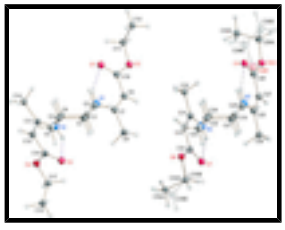

Fig. 2. Comparison of the conformations of the two independent molecules of the title compound.

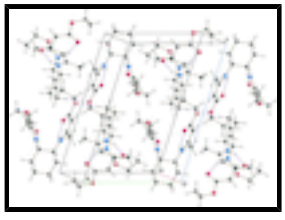

Fig. 3. Crystal packing of the title compound with intra- and intermolecular hydrogen bonds shown as dashed lines.

### *rac*-Ethyl (2*Z*)-3-{2-[(*Z*)-4-ethoxy- 4-oxobut-2-en-2-ylamino]cyclohexylamino}but-2-enoate

*Crystal data*

| | |
|---|---|
| $C_{18}H_{30}N_2O_4$ | $Z = 4$ |
| $M_r = 338.44$ | $F(000) = 736$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.142$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å |
| $a = 11.1424$ (9) Å | Cell parameters from 48 reflections |
| $b = 12.7445$ (6) Å | $\theta = 18.4$–$29.8°$ |
| $c = 16.3298$ (11) Å | $\mu = 0.65$ mm$^{-1}$ |
| $\alpha = 90.904$ (6)° | $T = 294$ K |
| $\beta = 109.222$ (6)° | Irregular block, colourless |
| $\gamma = 114.048$ (5)° | 0.18 × 0.15 × 0.10 mm |

$V$ = 1969.1 (3) Å$^3$

*Data collection*

| | |
|---|---|
| Siemens AED diffractometer | $R_{int}$ = 0.006 |
| Radiation source: fine-focus sealed tube | $\theta_{max}$ = 68.0°, $\theta_{min}$ = 2.9° |
| graphite | $h$ = −13→12 |
| θ/2θ scans | $k$ = −15→9 |
| 7461 measured reflections | $l$ = −18→19 |
| 7189 independent reflections | 3 standard reflections every 100 reflections |
| 5826 reflections with $I > 2\sigma(I)$ | intensity decay: 0.02% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.034 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.106 | $w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.0296P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.16 | $(\Delta/\sigma)_{max} < 0.001$ |
| 7189 reflections | $\Delta\rho_{max}$ = 0.19 e Å$^{-3}$ |
| 467 parameters | $\Delta\rho_{min}$ = −0.14 e Å$^{-3}$ |
| 8 restraints | Extinction correction: *SHELXL97* (Sheldrick, 2008) |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.0100 (5) |

*Special details*

**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) |
|---|---|---|---|---|---|
| O1 | 0.65218 (9) | 0.84797 (6) | 0.08853 (5) | 0.0814 (2) | |
| O2 | 0.58185 (10) | 0.74147 (7) | −0.04449 (5) | 0.0864 (2) | |
| O3 | 0.11165 (8) | 0.64888 (7) | 0.18740 (5) | 0.0774 (2) | |
| O4 | −0.03250 (9) | 0.73472 (8) | 0.13437 (6) | 0.0912 (3) | |
| O5 | −0.19007 (10) | 0.18776 (8) | 0.38766 (5) | 0.0895 (3) | |
| O6A | −0.1211 (2) | 0.10330 (16) | 0.50344 (14) | 0.0829 (5) | 0.704 (2) |
| O6B | −0.0647 (6) | 0.1425 (4) | 0.5121 (4) | 0.0829 (5) | 0.296 (2) |
| O7 | 0.34330 (8) | 0.27768 (7) | 0.26921 (6) | 0.0846 (2) | |
| O8 | 0.52581 (9) | 0.38869 (8) | 0.39309 (7) | 0.0963 (3) | |

| | | | | |
|---|---|---|---|---|
| N1 | 0.55037 (10) | 0.72198 (8) | 0.20595 (6) | 0.0684 (2) |
| H1N | 0.5994 (13) | 0.7854 (12) | 0.1927 (8) | 0.082 (4)* |
| N2 | 0.38841 (10) | 0.79819 (8) | 0.27442 (7) | 0.0727 (2) |
| H2N | 0.3211 (14) | 0.7281 (12) | 0.2553 (8) | 0.084 (4)* |
| N3 | −0.13927 (10) | 0.13606 (8) | 0.24363 (6) | 0.0687 (2) |
| H3N | −0.1697 (13) | 0.1707 (11) | 0.2705 (8) | 0.080 (4)* |
| N4 | 0.11045 (10) | 0.32223 (9) | 0.23049 (6) | 0.0719 (2) |
| H4N | 0.1472 (13) | 0.2852 (11) | 0.2165 (8) | 0.082 (4)* |
| C1 | 0.55684 (11) | 0.71559 (9) | 0.29601 (6) | 0.0635 (2) |
| H1 | 0.4829 | 0.6400 | 0.2960 | 0.076* |
| C2 | 0.69934 (12) | 0.72291 (10) | 0.35581 (8) | 0.0756 (3) |
| H2A | 0.7131 | 0.6592 | 0.3338 | 0.091* |
| H2B | 0.7744 | 0.7955 | 0.3542 | 0.091* |
| C3 | 0.70863 (14) | 0.71697 (11) | 0.45078 (8) | 0.0846 (3) |
| H3A | 0.6395 | 0.6415 | 0.4536 | 0.102* |
| H3B | 0.8018 | 0.7256 | 0.4871 | 0.102* |
| C4 | 0.68137 (14) | 0.81250 (11) | 0.48572 (8) | 0.0863 (3) |
| H4A | 0.7565 | 0.8879 | 0.4890 | 0.104* |
| H4B | 0.6816 | 0.8045 | 0.5447 | 0.104* |
| C5 | 0.53939 (13) | 0.80654 (11) | 0.42641 (8) | 0.0819 (3) |
| H5A | 0.4639 | 0.7346 | 0.4285 | 0.098* |
| H5B | 0.5272 | 0.8710 | 0.4487 | 0.098* |
| C6 | 0.52777 (11) | 0.81168 (9) | 0.33084 (7) | 0.0664 (2) |
| H6 | 0.5987 | 0.8876 | 0.3285 | 0.080* |
| C7 | 0.48965 (10) | 0.63196 (8) | 0.13905 (6) | 0.0608 (2) |
| C8 | 0.40309 (15) | 0.51312 (10) | 0.15353 (9) | 0.0937 (4) |
| H8A | 0.4628 | 0.4896 | 0.1987 | 0.141* |
| H8B | 0.3609 | 0.4582 | 0.0998 | 0.141* |
| H8C | 0.3302 | 0.5155 | 0.1714 | 0.141* |
| C9 | 0.50447 (10) | 0.64528 (9) | 0.05978 (7) | 0.0648 (2) |
| H9 | 0.4586 | 0.5793 | 0.0162 | 0.078* |
| C10 | 0.58526 (11) | 0.75309 (9) | 0.03918 (7) | 0.0655 (2) |
| C11 | 0.65923 (17) | 0.84598 (13) | −0.07360 (9) | 0.0971 (4) |
| H11A | 0.6510 | 0.9113 | −0.0488 | 0.117* |
| H11B | 0.6176 | 0.8358 | −0.1372 | 0.117* |
| C12 | 0.80899 (19) | 0.87259 (17) | −0.04739 (13) | 0.1255 (6) |
| H12A | 0.8566 | 0.9426 | −0.0674 | 0.188* |
| H12B | 0.8177 | 0.8091 | −0.0732 | 0.188* |
| H12C | 0.8508 | 0.8836 | 0.0156 | 0.188* |
| C13 | 0.34359 (13) | 0.88097 (10) | 0.25392 (8) | 0.0779 (3) |
| C14 | 0.45322 (18) | 1.00673 (12) | 0.28361 (13) | 0.1315 (7) |
| H14A | 0.4072 | 1.0572 | 0.2707 | 0.197* |
| H14B | 0.5175 | 1.0224 | 0.2531 | 0.197* |
| H14C | 0.5046 | 1.0204 | 0.3459 | 0.197* |
| C15 | 0.20513 (13) | 0.85458 (10) | 0.20757 (7) | 0.0795 (3) |
| H15 | 0.1805 | 0.9157 | 0.1951 | 0.095* |
| C16 | 0.09706 (12) | 0.73879 (10) | 0.17749 (7) | 0.0699 (3) |
| C17 | −0.15032 (13) | 0.61954 (12) | 0.10095 (8) | 0.0878 (4) |
| H17A | −0.1213 | 0.5689 | 0.0757 | 0.105* |

| | | | | | |
|---|---|---|---|---|---|
| H17B | −0.2272 | 0.6252 | 0.0544 | 0.105* | |
| C18 | −0.20206 (15) | 0.56635 (16) | 0.17051 (10) | 0.1087 (5) | |
| H18A | −0.2798 | 0.4907 | 0.1452 | 0.163* | |
| H18B | −0.2327 | 0.6153 | 0.1950 | 0.163* | |
| H18C | −0.1270 | 0.5587 | 0.2161 | 0.163* | |
| C19 | −0.13341 (11) | 0.16744 (9) | 0.15936 (7) | 0.0646 (2) | |
| H19 | −0.0979 | 0.1202 | 0.1354 | 0.077* | |
| C20 | −0.28197 (12) | 0.14077 (10) | 0.09474 (7) | 0.0760 (3) | |
| H20A | −0.3426 | 0.0579 | 0.0856 | 0.091* | |
| H20B | −0.3214 | 0.1824 | 0.1197 | 0.091* | |
| C21 | −0.27846 (14) | 0.17667 (11) | 0.00643 (8) | 0.0837 (3) | |
| H21A | −0.2467 | 0.1302 | −0.0209 | 0.100* | |
| H21B | −0.3730 | 0.1616 | −0.0324 | 0.100* | |
| C22 | −0.18062 (14) | 0.30461 (12) | 0.01838 (8) | 0.0833 (3) | |
| H22A | −0.2169 | 0.3513 | 0.0410 | 0.100* | |
| H22B | −0.1776 | 0.3245 | −0.0382 | 0.100* | |
| C23 | −0.03151 (13) | 0.33309 (12) | 0.08190 (8) | 0.0817 (3) | |
| H23A | 0.0276 | 0.4162 | 0.0908 | 0.098* | |
| H23B | 0.0085 | 0.2926 | 0.0565 | 0.098* | |
| C24 | −0.03252 (11) | 0.29689 (9) | 0.17064 (7) | 0.0652 (2) | |
| H24 | −0.0665 | 0.3432 | 0.1971 | 0.078* | |
| C25 | −0.07559 (10) | 0.07824 (8) | 0.29520 (7) | 0.0607 (2) | |
| C26 | −0.00907 (14) | 0.01806 (11) | 0.25829 (8) | 0.0788 (3) | |
| H26A | 0.0647 | 0.0746 | 0.2427 | 0.118* | |
| H26B | −0.0793 | −0.0381 | 0.2069 | 0.118* | |
| H26C | 0.0300 | −0.0209 | 0.3017 | 0.118* | |
| C27 | −0.07262 (11) | 0.07126 (9) | 0.37959 (7) | 0.0654 (2) | |
| H27 | −0.0317 | 0.0267 | 0.4114 | 0.078* | |
| C28 | −0.12888 (13) | 0.12870 (9) | 0.42072 (7) | 0.0725 (3) | |
| C29A | −0.1870 (3) | 0.14951 (19) | 0.54811 (14) | 0.0902 (5) | 0.704 (2) |
| H29A | −0.2160 | 0.0993 | 0.5888 | 0.108* | 0.704 (2) |
| H29B | −0.2707 | 0.1505 | 0.5052 | 0.108* | 0.704 (2) |
| C30A | −0.0877 (3) | 0.2695 (2) | 0.59710 (16) | 0.1157 (7) | 0.704 (2) |
| H30A | −0.1333 | 0.2985 | 0.6259 | 0.174* | 0.704 (2) |
| H30B | −0.0598 | 0.3194 | 0.5568 | 0.174* | 0.704 (2) |
| H30C | −0.0056 | 0.2683 | 0.6403 | 0.174* | 0.704 (2) |
| C29B | −0.0883 (6) | 0.2124 (5) | 0.5686 (3) | 0.0902 (5) | 0.296 (2) |
| H29C | −0.0699 | 0.2880 | 0.5509 | 0.108* | 0.296 (2) |
| H29D | −0.0242 | 0.2246 | 0.6288 | 0.108* | 0.296 (2) |
| C30B | −0.2357 (7) | 0.1538 (6) | 0.5640 (4) | 0.1157 (7) | 0.296 (2) |
| H30D | −0.2509 | 0.2013 | 0.6020 | 0.174* | 0.296 (2) |
| H30E | −0.2532 | 0.0796 | 0.5824 | 0.174* | 0.296 (2) |
| H30F | −0.2988 | 0.1424 | 0.5045 | 0.174* | 0.296 (2) |
| C31 | 0.18551 (11) | 0.39366 (9) | 0.30854 (7) | 0.0653 (2) | |
| C32 | 0.11850 (15) | 0.45730 (13) | 0.34209 (8) | 0.0911 (4) | |
| H32A | 0.0876 | 0.5000 | 0.2987 | 0.137* | |
| H32B | 0.1863 | 0.5105 | 0.3955 | 0.137* | |
| H32C | 0.0389 | 0.4020 | 0.3535 | 0.137* | |
| C33 | 0.32099 (11) | 0.41186 (9) | 0.35799 (7) | 0.0692 (3) | |

| | | | | | |
|---|---|---|---|---|---|
| H33 | 0.3689 | 0.4659 | 0.4101 | 0.083* | |
| C34 | 0.39224 (11) | 0.35293 (9) | 0.33421 (8) | 0.0701 (3) | |
| C35A | 0.6064 (10) | 0.3370 (8) | 0.3684 (8) | 0.1107 (14) | 0.505 (3) |
| H35A | 0.5625 | 0.3052 | 0.3059 | 0.133* | 0.505 (3) |
| H35B | 0.6079 | 0.2739 | 0.4005 | 0.133* | 0.505 (3) |
| C36A | 0.7530 (4) | 0.4278 (3) | 0.3891 (3) | 0.1138 (9) | 0.505 (3) |
| H36A | 0.8069 | 0.3938 | 0.3736 | 0.171* | 0.505 (3) |
| H36B | 0.7958 | 0.4591 | 0.4509 | 0.171* | 0.505 (3) |
| H36C | 0.7510 | 0.4891 | 0.3561 | 0.171* | 0.505 (3) |
| C35B | 0.6115 (10) | 0.3321 (9) | 0.3864 (8) | 0.1107 (14) | 0.495 (3) |
| H35C | 0.5535 | 0.2491 | 0.3655 | 0.133* | 0.495 (3) |
| H35D | 0.6832 | 0.3434 | 0.4435 | 0.133* | 0.495 (3) |
| C36B | 0.6779 (4) | 0.3848 (3) | 0.3239 (3) | 0.1138 (9) | 0.495 (3) |
| H36D | 0.7310 | 0.3454 | 0.3148 | 0.171* | 0.495 (3) |
| H36E | 0.7401 | 0.4658 | 0.3472 | 0.171* | 0.495 (3) |
| H36F | 0.6062 | 0.3777 | 0.2688 | 0.171* | 0.495 (3) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0996 (6) | 0.0589 (4) | 0.0826 (5) | 0.0259 (4) | 0.0411 (4) | 0.0138 (4) |
| O2 | 0.1099 (6) | 0.0779 (5) | 0.0716 (5) | 0.0353 (5) | 0.0407 (4) | 0.0190 (4) |
| O3 | 0.0683 (4) | 0.0726 (5) | 0.0910 (5) | 0.0352 (4) | 0.0234 (4) | 0.0129 (4) |
| O4 | 0.0796 (5) | 0.0919 (6) | 0.0928 (6) | 0.0482 (5) | 0.0072 (4) | 0.0111 (4) |
| O5 | 0.1190 (7) | 0.1038 (6) | 0.0856 (5) | 0.0769 (6) | 0.0486 (5) | 0.0328 (5) |
| O6A | 0.1189 (17) | 0.0739 (12) | 0.0744 (7) | 0.0502 (11) | 0.0471 (12) | 0.0226 (9) |
| O6B | 0.1189 (17) | 0.0739 (12) | 0.0744 (7) | 0.0502 (11) | 0.0471 (12) | 0.0226 (9) |
| O7 | 0.0722 (5) | 0.0796 (5) | 0.0976 (6) | 0.0342 (4) | 0.0253 (4) | 0.0048 (4) |
| O8 | 0.0653 (5) | 0.0818 (5) | 0.1212 (7) | 0.0336 (4) | 0.0085 (4) | −0.0023 (5) |
| N1 | 0.0743 (5) | 0.0544 (5) | 0.0685 (5) | 0.0177 (4) | 0.0301 (4) | 0.0123 (4) |
| N2 | 0.0631 (5) | 0.0594 (5) | 0.0851 (6) | 0.0232 (4) | 0.0194 (4) | 0.0076 (4) |
| N3 | 0.0763 (6) | 0.0798 (6) | 0.0707 (5) | 0.0477 (5) | 0.0340 (4) | 0.0227 (4) |
| N4 | 0.0635 (5) | 0.0806 (6) | 0.0752 (6) | 0.0370 (5) | 0.0231 (4) | 0.0075 (5) |
| C1 | 0.0623 (5) | 0.0550 (5) | 0.0642 (5) | 0.0178 (4) | 0.0227 (4) | 0.0114 (4) |
| C2 | 0.0709 (6) | 0.0699 (6) | 0.0818 (7) | 0.0314 (5) | 0.0220 (5) | 0.0158 (5) |
| C3 | 0.0831 (8) | 0.0752 (7) | 0.0766 (7) | 0.0302 (6) | 0.0122 (6) | 0.0179 (6) |
| C4 | 0.0909 (8) | 0.0780 (7) | 0.0644 (6) | 0.0235 (6) | 0.0152 (6) | 0.0080 (5) |
| C5 | 0.0844 (8) | 0.0805 (7) | 0.0737 (7) | 0.0289 (6) | 0.0302 (6) | 0.0055 (5) |
| C6 | 0.0609 (5) | 0.0579 (5) | 0.0688 (6) | 0.0187 (4) | 0.0194 (5) | 0.0080 (4) |
| C7 | 0.0515 (5) | 0.0560 (5) | 0.0645 (5) | 0.0194 (4) | 0.0144 (4) | 0.0089 (4) |
| C8 | 0.1030 (9) | 0.0618 (7) | 0.0816 (8) | 0.0023 (6) | 0.0360 (7) | 0.0035 (5) |
| C9 | 0.0624 (6) | 0.0586 (5) | 0.0640 (6) | 0.0223 (4) | 0.0173 (4) | 0.0070 (4) |
| C10 | 0.0709 (6) | 0.0646 (6) | 0.0658 (6) | 0.0342 (5) | 0.0248 (5) | 0.0148 (5) |
| C11 | 0.1316 (12) | 0.0907 (9) | 0.0856 (8) | 0.0509 (9) | 0.0557 (8) | 0.0361 (7) |
| C12 | 0.1146 (13) | 0.1228 (13) | 0.1317 (14) | 0.0328 (10) | 0.0597 (11) | 0.0457 (11) |
| C13 | 0.0854 (7) | 0.0616 (6) | 0.0734 (7) | 0.0286 (5) | 0.0176 (6) | 0.0133 (5) |
| C14 | 0.1176 (12) | 0.0606 (8) | 0.1555 (15) | 0.0270 (8) | −0.0074 (11) | 0.0155 (8) |
| C15 | 0.0893 (8) | 0.0714 (7) | 0.0753 (7) | 0.0441 (6) | 0.0156 (6) | 0.0138 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.0733 (6) | 0.0781 (7) | 0.0618 (6) | 0.0411 (6) | 0.0186 (5) | 0.0114 (5) |
| C17 | 0.0700 (7) | 0.1016 (9) | 0.0771 (7) | 0.0386 (7) | 0.0084 (6) | 0.0057 (6) |
| C18 | 0.0777 (8) | 0.1517 (14) | 0.0942 (9) | 0.0514 (9) | 0.0268 (7) | 0.0191 (9) |
| C19 | 0.0687 (6) | 0.0691 (6) | 0.0656 (6) | 0.0388 (5) | 0.0250 (5) | 0.0139 (5) |
| C20 | 0.0694 (6) | 0.0742 (7) | 0.0768 (7) | 0.0324 (5) | 0.0167 (5) | 0.0085 (5) |
| C21 | 0.0901 (8) | 0.0898 (8) | 0.0687 (6) | 0.0505 (7) | 0.0123 (6) | 0.0086 (6) |
| C22 | 0.0974 (8) | 0.0956 (8) | 0.0738 (7) | 0.0589 (7) | 0.0297 (6) | 0.0291 (6) |
| C23 | 0.0850 (8) | 0.0902 (8) | 0.0843 (7) | 0.0467 (7) | 0.0369 (6) | 0.0329 (6) |
| C24 | 0.0631 (6) | 0.0708 (6) | 0.0684 (6) | 0.0358 (5) | 0.0237 (5) | 0.0129 (5) |
| C25 | 0.0551 (5) | 0.0535 (5) | 0.0707 (6) | 0.0238 (4) | 0.0196 (4) | 0.0098 (4) |
| C26 | 0.0933 (8) | 0.0823 (7) | 0.0818 (7) | 0.0561 (7) | 0.0344 (6) | 0.0194 (6) |
| C27 | 0.0709 (6) | 0.0588 (5) | 0.0686 (6) | 0.0329 (5) | 0.0223 (5) | 0.0141 (4) |
| C28 | 0.0880 (7) | 0.0653 (6) | 0.0714 (6) | 0.0385 (6) | 0.0314 (5) | 0.0177 (5) |
| C29A | 0.1123 (16) | 0.0960 (15) | 0.0813 (11) | 0.0553 (12) | 0.0459 (12) | 0.0170 (10) |
| C30A | 0.1343 (18) | 0.1182 (16) | 0.1043 (15) | 0.0694 (14) | 0.0388 (13) | −0.0099 (12) |
| C29B | 0.1123 (16) | 0.0960 (15) | 0.0813 (11) | 0.0553 (12) | 0.0459 (12) | 0.0170 (10) |
| C30B | 0.1343 (18) | 0.1182 (16) | 0.1043 (15) | 0.0694 (14) | 0.0388 (13) | −0.0099 (12) |
| C31 | 0.0716 (6) | 0.0657 (6) | 0.0641 (6) | 0.0310 (5) | 0.0297 (5) | 0.0196 (5) |
| C32 | 0.0950 (9) | 0.1066 (10) | 0.0821 (8) | 0.0563 (8) | 0.0301 (7) | 0.0042 (7) |
| C33 | 0.0715 (6) | 0.0686 (6) | 0.0658 (6) | 0.0298 (5) | 0.0238 (5) | 0.0167 (5) |
| C34 | 0.0623 (6) | 0.0603 (6) | 0.0805 (7) | 0.0228 (5) | 0.0225 (5) | 0.0189 (5) |
| C35A | 0.0754 (10) | 0.0835 (10) | 0.157 (4) | 0.0428 (8) | 0.0135 (18) | 0.0046 (19) |
| C36A | 0.102 (2) | 0.0986 (19) | 0.163 (3) | 0.0561 (17) | 0.0601 (18) | 0.0378 (19) |
| C35B | 0.0754 (10) | 0.0835 (10) | 0.157 (4) | 0.0428 (8) | 0.0135 (18) | 0.0046 (19) |
| C36B | 0.102 (2) | 0.0986 (19) | 0.163 (3) | 0.0561 (17) | 0.0601 (18) | 0.0378 (19) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C10 | 1.2273 (12) | C15—H15 | 0.9300 |
| O2—C10 | 1.3584 (13) | C17—C18 | 1.490 (2) |
| O2—C11 | 1.4482 (15) | C17—H17A | 0.9700 |
| O3—C16 | 1.2272 (13) | C17—H17B | 0.9700 |
| O4—C16 | 1.3605 (13) | C18—H18A | 0.9600 |
| O4—C17 | 1.4520 (16) | C18—H18B | 0.9600 |
| O5—C28 | 1.2294 (13) | C18—H18C | 0.9600 |
| O6A—C28 | 1.377 (2) | C19—C20 | 1.5327 (14) |
| O6A—C29A | 1.453 (3) | C19—C24 | 1.5368 (15) |
| O6B—C28 | 1.398 (6) | C19—H19 | 0.9800 |
| O6B—C29B | 1.429 (6) | C20—C21 | 1.5277 (16) |
| O7—C34 | 1.2264 (14) | C20—H20A | 0.9700 |
| O8—C34 | 1.3558 (13) | C20—H20B | 0.9700 |
| O8—C35B | 1.439 (7) | C21—C22 | 1.5121 (19) |
| O8—C35A | 1.455 (7) | C21—H21A | 0.9700 |
| N1—C7 | 1.3448 (13) | C21—H21B | 0.9700 |
| N1—C1 | 1.4538 (13) | C22—C23 | 1.5264 (17) |
| N1—H1N | 0.850 (13) | C22—H22A | 0.9700 |
| N2—C13 | 1.3435 (14) | C22—H22B | 0.9700 |
| N2—C6 | 1.4577 (13) | C23—C24 | 1.5298 (14) |
| N2—H2N | 0.865 (13) | C23—H23A | 0.9700 |

| | | | |
|---|---|---|---|
| N3—C25 | 1.3446 (13) | C23—H23B | 0.9700 |
| N3—C19 | 1.4537 (13) | C24—H24 | 0.9800 |
| N3—H3N | 0.847 (13) | C25—C27 | 1.3727 (14) |
| N4—C31 | 1.3392 (14) | C25—C26 | 1.4979 (14) |
| N4—C24 | 1.4639 (13) | C26—H26A | 0.9600 |
| N4—H4N | 0.811 (13) | C26—H26B | 0.9600 |
| C1—C2 | 1.5293 (14) | C26—H26C | 0.9600 |
| C1—C6 | 1.5332 (15) | C27—C28 | 1.4197 (15) |
| C1—H1 | 0.9800 | C27—H27 | 0.9300 |
| C2—C3 | 1.5251 (16) | C29A—C30A | 1.484 (3) |
| C2—H2A | 0.9700 | C29A—H29A | 0.9700 |
| C2—H2B | 0.9700 | C29A—H29B | 0.9700 |
| C3—C4 | 1.5145 (18) | C30A—H30A | 0.9600 |
| C3—H3A | 0.9700 | C30A—H30B | 0.9600 |
| C3—H3B | 0.9700 | C30A—H30C | 0.9600 |
| C4—C5 | 1.5288 (18) | C29B—C30B | 1.475 (7) |
| C4—H4A | 0.9700 | C29B—H29C | 0.9700 |
| C4—H4B | 0.9700 | C29B—H29D | 0.9700 |
| C5—C6 | 1.5277 (15) | C30B—H30D | 0.9600 |
| C5—H5A | 0.9700 | C30B—H30E | 0.9600 |
| C5—H5B | 0.9700 | C30B—H30F | 0.9600 |
| C6—H6 | 0.9800 | C31—C33 | 1.3751 (15) |
| C7—C9 | 1.3626 (14) | C31—C32 | 1.5048 (15) |
| C7—C8 | 1.5003 (15) | C32—H32A | 0.9600 |
| C8—H8A | 0.9600 | C32—H32B | 0.9600 |
| C8—H8B | 0.9600 | C32—H32C | 0.9600 |
| C8—H8C | 0.9600 | C33—C34 | 1.4231 (15) |
| C9—C10 | 1.4202 (14) | C33—H33 | 0.9300 |
| C9—H9 | 0.9300 | C35A—C36A | 1.485 (9) |
| C11—C12 | 1.465 (2) | C35A—H35A | 0.9700 |
| C11—H11A | 0.9700 | C35A—H35B | 0.9700 |
| C11—H11B | 0.9700 | C36A—H36A | 0.9600 |
| C12—H12A | 0.9600 | C36A—H36B | 0.9600 |
| C12—H12B | 0.9600 | C36A—H36C | 0.9600 |
| C12—H12C | 0.9600 | C35B—C36B | 1.468 (9) |
| C13—C15 | 1.3662 (17) | C35B—H35C | 0.9700 |
| C13—C14 | 1.5119 (18) | C35B—H35D | 0.9700 |
| C14—H14A | 0.9600 | C36B—H36D | 0.9600 |
| C14—H14B | 0.9600 | C36B—H36E | 0.9600 |
| C14—H14C | 0.9600 | C36B—H36F | 0.9600 |
| C15—C16 | 1.4172 (16) | | |
| C10—O2—C11 | 117.28 (10) | C20—C19—H19 | 108.5 |
| C16—O4—C17 | 116.58 (9) | C24—C19—H19 | 108.5 |
| C28—O6A—C29A | 117.05 (18) | C21—C20—C19 | 111.11 (10) |
| C28—O6B—C29B | 119.8 (5) | C21—C20—H20A | 109.4 |
| C34—O8—C35B | 121.2 (5) | C19—C20—H20A | 109.4 |
| C34—O8—C35A | 114.2 (5) | C21—C20—H20B | 109.4 |
| C7—N1—C1 | 126.83 (9) | C19—C20—H20B | 109.4 |
| C7—N1—H1N | 111.8 (8) | H20A—C20—H20B | 108.0 |

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|---|---|---|---|
| C1—N1—H1N | 120.6 (8) | C22—C21—C20 | 111.03 (10) |
| C13—N2—C6 | 128.79 (10) | C22—C21—H21A | 109.4 |
| C13—N2—H2N | 113.1 (8) | C20—C21—H21A | 109.4 |
| C6—N2—H2N | 117.7 (8) | C22—C21—H21B | 109.4 |
| C25—N3—C19 | 128.67 (9) | C20—C21—H21B | 109.4 |
| C25—N3—H3N | 112.5 (8) | H21A—C21—H21B | 108.0 |
| C19—N3—H3N | 117.2 (8) | C21—C22—C23 | 111.17 (10) |
| C31—N4—C24 | 128.22 (9) | C21—C22—H22A | 109.4 |
| C31—N4—H4N | 115.3 (9) | C23—C22—H22A | 109.4 |
| C24—N4—H4N | 116.3 (9) | C21—C22—H22B | 109.4 |
| N1—C1—C2 | 111.09 (9) | C23—C22—H22B | 109.4 |
| N1—C1—C6 | 110.54 (8) | H22A—C22—H22B | 108.0 |
| C2—C1—C6 | 110.93 (8) | C22—C23—C24 | 111.12 (9) |
| N1—C1—H1 | 108.0 | C22—C23—H23A | 109.4 |
| C2—C1—H1 | 108.0 | C24—C23—H23A | 109.4 |
| C6—C1—H1 | 108.0 | C22—C23—H23B | 109.4 |
| C3—C2—C1 | 111.95 (9) | C24—C23—H23B | 109.4 |
| C3—C2—H2A | 109.2 | H23A—C23—H23B | 108.0 |
| C1—C2—H2A | 109.2 | N4—C24—C23 | 110.76 (9) |
| C3—C2—H2B | 109.2 | N4—C24—C19 | 110.83 (8) |
| C1—C2—H2B | 109.2 | C23—C24—C19 | 111.46 (9) |
| H2A—C2—H2B | 107.9 | N4—C24—H24 | 107.9 |
| C4—C3—C2 | 110.45 (10) | C23—C24—H24 | 107.9 |
| C4—C3—H3A | 109.6 | C19—C24—H24 | 107.9 |
| C2—C3—H3A | 109.6 | N3—C25—C27 | 121.75 (9) |
| C4—C3—H3B | 109.6 | N3—C25—C26 | 118.67 (9) |
| C2—C3—H3B | 109.6 | C27—C25—C26 | 119.57 (9) |
| H3A—C3—H3B | 108.1 | C25—C26—H26A | 109.5 |
| C3—C4—C5 | 111.02 (10) | C25—C26—H26B | 109.5 |
| C3—C4—H4A | 109.4 | H26A—C26—H26B | 109.5 |
| C5—C4—H4A | 109.4 | C25—C26—H26C | 109.5 |
| C3—C4—H4B | 109.4 | H26A—C26—H26C | 109.5 |
| C5—C4—H4B | 109.4 | H26B—C26—H26C | 109.5 |
| H4A—C4—H4B | 108.0 | C25—C27—C28 | 123.28 (9) |
| C6—C5—C4 | 112.53 (10) | C25—C27—H27 | 118.4 |
| C6—C5—H5A | 109.1 | C28—C27—H27 | 118.4 |
| C4—C5—H5A | 109.1 | O5—C28—O6A | 120.10 (13) |
| C6—C5—H5B | 109.1 | O5—C28—O6B | 121.0 (2) |
| C4—C5—H5B | 109.1 | O5—C28—C27 | 126.94 (10) |
| H5A—C5—H5B | 107.8 | O6A—C28—C27 | 112.70 (12) |
| N2—C6—C5 | 111.60 (9) | O6B—C28—C27 | 108.9 (3) |
| N2—C6—C1 | 109.13 (8) | O6A—C29A—C30A | 110.8 (2) |
| C5—C6—C1 | 110.09 (9) | O6A—C29A—H29A | 109.5 |
| N2—C6—H6 | 108.7 | C30A—C29A—H29A | 109.5 |
| C5—C6—H6 | 108.7 | O6A—C29A—H29B | 109.5 |
| C1—C6—H6 | 108.7 | C30A—C29A—H29B | 109.5 |
| N1—C7—C9 | 122.42 (9) | H29A—C29A—H29B | 108.1 |
| N1—C7—C8 | 118.00 (10) | C29A—C30A—H30A | 109.5 |
| C9—C7—C8 | 119.59 (9) | C29A—C30A—H30B | 109.5 |

| | | | |
|---|---|---|---|
| C7—C8—H8A | 109.5 | H30A—C30A—H30B | 109.5 |
| C7—C8—H8B | 109.5 | C29A—C30A—H30C | 109.5 |
| H8A—C8—H8B | 109.5 | H30A—C30A—H30C | 109.5 |
| C7—C8—H8C | 109.5 | H30B—C30A—H30C | 109.5 |
| H8A—C8—H8C | 109.5 | O6B—C29B—C30B | 109.9 (5) |
| H8B—C8—H8C | 109.5 | O6B—C29B—H29C | 109.7 |
| C7—C9—C10 | 124.40 (9) | C30B—C29B—H29C | 109.7 |
| C7—C9—H9 | 117.8 | O6B—C29B—H29D | 109.7 |
| C10—C9—H9 | 117.8 | C30B—C29B—H29D | 109.7 |
| O1—C10—O2 | 121.51 (10) | H29C—C29B—H29D | 108.2 |
| O1—C10—C9 | 126.35 (10) | C29B—C30B—H30D | 109.5 |
| O2—C10—C9 | 112.14 (9) | C29B—C30B—H30E | 109.5 |
| O2—C11—C12 | 112.02 (12) | H30D—C30B—H30E | 109.5 |
| O2—C11—H11A | 109.2 | C29B—C30B—H30F | 109.5 |
| C12—C11—H11A | 109.2 | H30D—C30B—H30F | 109.5 |
| O2—C11—H11B | 109.2 | H30E—C30B—H30F | 109.5 |
| C12—C11—H11B | 109.2 | N4—C31—C33 | 122.59 (10) |
| H11A—C11—H11B | 107.9 | N4—C31—C32 | 118.16 (10) |
| C11—C12—H12A | 109.5 | C33—C31—C32 | 119.25 (10) |
| C11—C12—H12B | 109.5 | C31—C32—H32A | 109.5 |
| H12A—C12—H12B | 109.5 | C31—C32—H32B | 109.5 |
| C11—C12—H12C | 109.5 | H32A—C32—H32B | 109.5 |
| H12A—C12—H12C | 109.5 | C31—C32—H32C | 109.5 |
| H12B—C12—H12C | 109.5 | H32A—C32—H32C | 109.5 |
| N2—C13—C15 | 122.28 (11) | H32B—C32—H32C | 109.5 |
| N2—C13—C14 | 117.39 (11) | C31—C33—C34 | 123.87 (10) |
| C15—C13—C14 | 120.33 (11) | C31—C33—H33 | 118.1 |
| C13—C14—H14A | 109.5 | C34—C33—H33 | 118.1 |
| C13—C14—H14B | 109.5 | O7—C34—O8 | 121.61 (10) |
| H14A—C14—H14B | 109.5 | O7—C34—C33 | 126.26 (10) |
| C13—C14—H14C | 109.5 | O8—C34—C33 | 112.13 (10) |
| H14A—C14—H14C | 109.5 | O8—C35A—C36A | 109.2 (6) |
| H14B—C14—H14C | 109.5 | O8—C35A—H35A | 109.8 |
| C13—C15—C16 | 123.31 (10) | C36A—C35A—H35A | 109.8 |
| C13—C15—H15 | 118.3 | O8—C35A—H35B | 109.8 |
| C16—C15—H15 | 118.3 | C36A—C35A—H35B | 109.8 |
| O3—C16—O4 | 120.92 (10) | H35A—C35A—H35B | 108.3 |
| O3—C16—C15 | 126.59 (10) | C35A—C36A—H36A | 109.5 |
| O4—C16—C15 | 112.49 (10) | C35A—C36A—H36B | 109.5 |
| O4—C17—C18 | 112.64 (11) | H36A—C36A—H36B | 109.5 |
| O4—C17—H17A | 109.1 | C35A—C36A—H36C | 109.5 |
| C18—C17—H17A | 109.1 | H36A—C36A—H36C | 109.5 |
| O4—C17—H17B | 109.1 | H36B—C36A—H36C | 109.5 |
| C18—C17—H17B | 109.1 | O8—C35B—C36B | 107.3 (7) |
| H17A—C17—H17B | 107.8 | O8—C35B—H35C | 110.3 |
| C17—C18—H18A | 109.5 | C36B—C35B—H35C | 110.3 |
| C17—C18—H18B | 109.5 | O8—C35B—H35D | 110.3 |
| H18A—C18—H18B | 109.5 | C36B—C35B—H35D | 110.3 |
| C17—C18—H18C | 109.5 | H35C—C35B—H35D | 108.5 |

| | | | |
|---|---|---|---|
| H18A—C18—H18C | 109.5 | C35B—C36B—H36D | 109.5 |
| H18B—C18—H18C | 109.5 | C35B—C36B—H36E | 109.5 |
| N3—C19—C20 | 109.91 (9) | H36D—C36B—H36E | 109.5 |
| N3—C19—C24 | 110.78 (8) | C35B—C36B—H36F | 109.5 |
| C20—C19—C24 | 110.58 (8) | H36D—C36B—H36F | 109.5 |
| N3—C19—H19 | 108.5 | H36E—C36B—H36F | 109.5 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1N···O1 | 0.850 (13) | 2.048 (14) | 2.754 (2) | 140.1 (12) |
| N2—H2N···O3 | 0.865 (12) | 2.009 (12) | 2.720 (2) | 138.7 (13) |
| N3—H3N···O5 | 0.846 (17) | 2.018 (15) | 2.724 (2) | 140.3 (12) |
| N4—H4N···O7 | 0.811 (17) | 2.112 (15) | 2.754 (2) | 136.1 (14) |
| C4—H4A···O6A$^{i}$ | 0.97 | 2.48 | 3.400 (3) | 158 |

Symmetry codes: (i) *x*+1, *y*+1, *z*.

**Fig. 1**

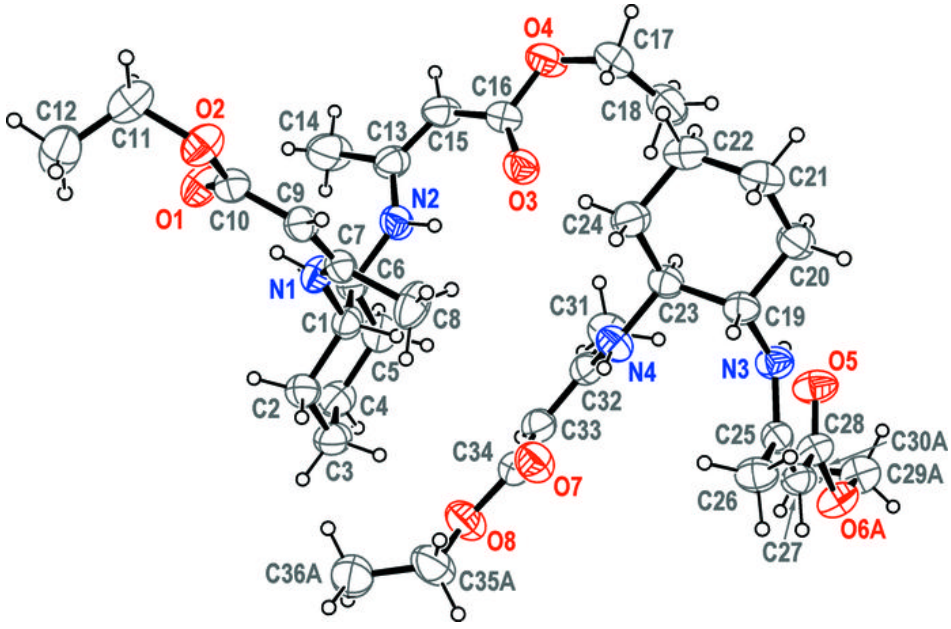
O4
C17
C16
C18
C12
O2
C11
C14
C13
C15
C22
C21
O3
C9
N2
C24
O1
C10
C7
C6
C20
N1
C31
C23
C19
C1
C8
N4
N3
O5
C5
C32
C2
C4
C33
C25
C28
C30A
C34
O7
C29A
C3
C26
O6A
O8
C27
C36A
C35A

**Fig. 2**

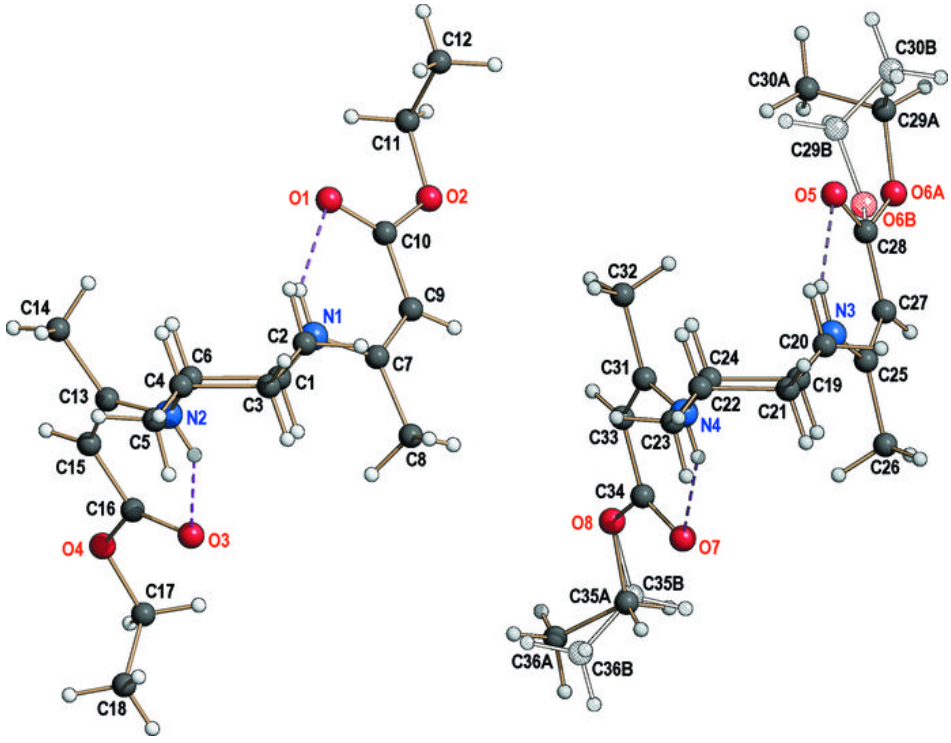
C12
C11
O1
O2
C10
C9
C14
N1
C2
C6
C4
C7
C13
C1
C3
N2
C5
C8
C15
C16
O4
O3
C17
C18
C30B
C30A
C29A
C29B
O5
O6A
O6B
C28
C32
C27
N3
C20
C31
C24
C25
C19
C22
C21
C33
C23
N4
C26
C34
O8
O7
C35A
C35B
C36A
C36B

**Fig. 3**