rac-4-Carbamoylpiperidinium

* cis-2-carboxycyclohexane-1-carboxylate*

Graham Smith and Urs D. Wermuth

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In the title racemic salt, \( \text{C}_8\text{H}_{13}\text{N}_2\text{O}^+\cdot\text{C}_8\text{H}_{11}\text{O}_4^- \), formed from the reaction of cis-cyclohexane-1,2-dicarboxylic anhydride with isonpecotamide, the cations are linked into duplex chain substructures through both centrosymmetric cyclic head-to-head ‘amide motif’ hydrogen-bonding associations [graph set \( R_2(8) \)] and ‘side-by-side’ \( R_2(14) \) associations. The anions are incorporated into the chains through cyclic \( R_3(10) \) interactions involving amide and piperidinium N—H⋯O
{\text{carboxyl}} hydrogen bonds which, together with inter-anion carboxylic acid O—H⋯O
{\text{carboxyl}} hydrogen bonds, give a two-dimensional layered structure extending along \( \alpha \).

**Related literature**

For examples of structures of 1:1 Lewis base salts of cis-cyclohexane-1,2-dicarboxylic acid, see: Smith & Wermuth (2011a,b). For examples of isonpecotamide proton-transfer salts, see: Smith & Wermuth (2010). For graph-set analysis, see: Etter et al. (1990). For hydrogen-bonding motifs, see: Allen et al. (1998).

**Table 1**

| Hydrogen-bond geometry (Å, °) |
|-----------------------------|
| \( D—H⋯A \) | \( D—H \) | \( H—A \) | \( D⋯A \) | \( D—H⋯A \) |
|-----------------------------|
| N1A—H11A⋯O41i | 0.97 (3) | 1.95 (3) | 2.861 (3) | 155 (2) |
| N1A—H12A⋯O11 | 0.99 (4) | 1.64 (4) | 2.588 (4) | 158 (3) |
| N41A—H41A⋯O14ii | 0.86 (3) | 2.14 (4) | 2.936 (3) | 174 (2) |
| N41A—H42A⋯O12iv | 0.77 (3) | 2.11 (3) | 2.882 (4) | 177 (3) |
| O22—H22⋯O12vii | 0.95 (3) | 1.65 (4) | 2.571 (3) | 173 (4) |

Symmetry codes:
(i) \( x, −y + 1, −z \);
(ii) \( −x, −y, −z \);
(iii) \( x, y − 1, z \);
(iv) \( x, −y + 1, z + 1 \).

Data collection: CrysAlis PRO (Oxford Diffraction, 2010); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2139).

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rac-4-Carbamoypiperidinium cis-2-carboxycyclohexane-1-carboxylate

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Comment
cis-Cyclohexane-1,2-dicarboxylic anhydride (cis-CHDC anhydride) forms racemic 1:1 salts with some Lewis bases and the structures of a few of these have been reported, e.g. with 2-aminopyridine (Smith & Wermuth, 2011a) and 4-amino-pyridine (Smith & Wermuth, 2011b). The 1:1 stoichiometric reaction of cis-CHDC anhydride with piperidine-4-carboxamide (isonipecotamide) also gave a racemic salt, the title compound, C₆H₁₂N₂O⁺C₈H₁₁O₄⁻ and the structure is reported here.

In this compound (Fig. 1) the cis-configuration of the anion is found as expected, with the cations linked into duplex ribbon substructures through both centrosymmetric cyclic head-to-head hydrogen-bonding associations [the ‘amide’ motif (Allen et al., 1998)] [graph set R₂⁺(8) (Etter et al., 1990)] and ‘side-by-side’ R₂⁺(14) associations (Table 1, Fig. 2). Both of these associations have been found in the structures of Lewis base salts of isonipecotamide (Smith & Wermuth, 2010). In the present structure, the monoanions are incorporated into the ribbons through cyclic R³⁺(10) amide and piperidinium N—H···Ocarboxyl associations and together with inter-anion carboxylic acid O—H···Ocarboxyl hydrogen bonds down c (Fig. 3), give a two-dimensional layered structure extending along (011).

Experimental
The title compound was synthesized by heating together under reflux for 15 min, 1 mmol quantities of cyclohexane-1,2-dicarboxylic anhydride and piperidine-4-carboxamide (isonipecotamide) in 50 ml of methanol. After volume reduction to 30 ml, the hot-filtered solution was allowed evaporate to dryness at room temperature, giving a white amorphous powder. Minor colourless crystal plates were obtained in the residual viscous residue after evaporation of a solution of the compound in 80% propane-2-ol–water.

Refinement
H atoms potentially involved in hydrogen-bonding associations were located in a difference Fourier analysis and their positional and isotropic displacement parameters were refined. Other H atoms were included in the refinement at calculated positions [C—H = 0.97–0.98 Å] with Uiso(H) = 1.2Ueq(C), using a riding-model approximation.

Computing details
Data collection: CrysAlis PRO (Oxford Diffraction, 2010); cell refinement: CrysAlis PRO (Oxford Diffraction, 2010); data reduction: CrysAlis PRO (Oxford Diffraction, 2010); program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON (Spek, 2009).
Figure 1
Molecular conformation of the cation and anion in the title compound, with the inter-ion hydrogen bond shown as a dashed line. Displacement ellipsoids are drawn at the 40% probability level.

Figure 2
The hydrogen-bonded ribbon substructure in the title salt showing the isonipicotamide cation $R_{2}^{2}(8)$ and $R_{2}^{2}(14)$ cyclic associations and the $R_{3}^{4}(10)$ incorporation of the monoanion. For symmetry codes, see Table 1.
A view of the two-dimensional hydrogen-bonded layered structure looking down the \( b \) axial direction, showing the inter-ribbon carboxylic acid···carboxyl hydrogen-bonding extensions down \( c \).

**rac-4-Carbamoylpiperidinium cis-2-carboxycyclohexane-1-carboxylate**

**Crystal data**

\[
\begin{align*}
C_5H_{13}N_2O^+ \cdot C_8H_{11}O_4^- & \\
M_r & = 300.35 \\
\text{Monoclinic, } P2_1/c & \\
\text{Hall symbol: } -P 2yb & \\
a & = 19.0097 \text{ (14) Å} & \\
b & = 9.0667 \text{ (7) Å} & \\
c & = 9.1999 \text{ (8) Å} & \\
\beta & = 92.022 \text{ (7)°} & \\
V & = 1584.7 \text{ (2) Å} & \\
Z & = 4 \\

F(000) & = 644 \\
D_x & = 1.255 \text{ Mg m}^{-3} & \\
\lambda & = 0.71073 \text{ Å} & \\
\text{Cell parameters from 3793 reflections} & \\
\theta & = 3.2–28.9° & \\
\mu & = 0.10 \text{ mm}^{-1} & \\
T & = 200 \text{ K} & \\
\text{Plate, colourless} & \\
\text{0.40 × 0.35 × 0.10 mm} & \\
\end{align*}
\]

**Data collection**

Oxford Gemini-S CCD area-detector diffractometer

Radiation source: Enhance (Mo) X-ray source

Graphite monochromator

Detector resolution: 16.077 pixels mm\(^{-1}\)

\( \omega \) scans

Absorption correction: multi-scans

(CrysAlis PRO; Oxford Diffraction, 2010)

\( T_{\text{min}} = 0.86, T_{\text{max}} = 0.98 \)

**Refinement**

Refinement on \( F^2 \)

Least-squares matrix: full

\( R[F^2 > 2\sigma(F^2)] = 0.075 \)

\( wR(F^2) = 0.182 \)

\( S = 1.06 \)

3100 reflections

210 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

\[
w = 1/\left[\sigma^2(F_o^2) + (0.0845P)^2 + 0.9949P\right]
\]

where \( P = (F_o^2 + 2F_c^2)/3 \)

\( \Delta \sigma_{\text{max}} = 0.002 \)

\( \Delta \rho_{\text{max}} = 0.43 \text{ e Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -0.20 \text{ e Å}^{-3} \)
supplementary materials

Special details

**Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 ($\AA^2$)

|    | $x$           | $y$           | $z$           | $U_{is}$/$U_{eq}$ |
|----|---------------|---------------|---------------|-------------------|
| O11| 0.21833 (13)  | 0.8455 (3)    | 0.0243 (3)    | 0.0522 (9)        |
| O12| 0.23582 (11)  | 1.0432 (3)    | −0.1098 (2)   | 0.0415 (8)        |
| O21| 0.25201 (12)  | 1.1290 (3)    | 0.2369 (3)    | 0.0487 (9)        |
| O22| 0.31747 (13)  | 1.3299 (3)    | 0.2117 (3)    | 0.0485 (9)        |
| C1 | 0.33119 (16)  | 0.9592 (3)    | 0.0442 (4)    | 0.0362 (10)       |
| C2 | 0.35236 (15)  | 1.1183 (4)    | 0.0836 (4)    | 0.0337 (10)       |
| C3 | 0.42799 (19)  | 1.1251 (5)    | 0.1462 (5)    | 0.0634 (16)       |
| C4 | 0.4392 (2)    | 1.0232 (6)    | 0.2741 (6)    | 0.086 (2)         |
| C5 | 0.4211 (2)    | 0.8668 (6)    | 0.2325 (6)    | 0.085 (2)         |
| C6 | 0.3456 (2)    | 0.8519 (4)    | 0.1711 (5)    | 0.0586 (14)       |
| C11| 0.25621 (15)  | 0.9481 (3)    | −0.0177 (3)   | 0.0306 (9)        |
| C21| 0.30223 (16)  | 1.1903 (4)    | 0.1864 (3)    | 0.0337 (10)       |
| O41A| −0.00706 (10) | 0.2052 (2)    | −0.0339 (2)   | 0.0298 (7)        |
| N1A | 0.13089 (14)  | 0.6330 (3)    | −0.0339 (3)   | 0.0293 (8)        |
| N41A| 0.08655 (16)  | 0.0589 (3)    | −0.0669 (3)   | 0.0286 (8)        |
| C2A | 0.11125 (16)  | 0.5811 (3)    | −0.1821 (3)   | 0.0290 (9)        |
| C3A | 0.06735 (15)  | 0.4418 (3)    | −0.1727 (3)   | 0.0257 (9)        |
| C4A | 0.10386 (14)  | 0.3218 (3)    | −0.0845 (3)   | 0.0251 (8)        |
| C5A | 0.12773 (16)  | 0.3800 (3)    | 0.0645 (3)    | 0.0292 (9)        |
| C6A | 0.17115 (17)  | 0.5201 (3)    | 0.0524 (3)    | 0.0325 (10)       |
| C41A| 0.05649 (14)  | 0.1901 (3)    | −0.0612 (3)   | 0.0236 (8)        |
| H1  | 0.36180       | 0.92850       | −0.03370      | 0.0430*           |
| H2  | 0.35070       | 1.17570       | −0.00670      | 0.0400*           |
| H22 | 0.288 (3)     | 1.369 (5)     | 0.281 (5)     | 0.082 (15)*       |
| H31 | 0.46020       | 1.09850       | 0.07100       | 0.0760*           |
| H32 | 0.43860       | 1.22540       | 0.17640       | 0.0760*           |
| H41 | 0.41000       | 1.05460       | 0.35270       | 0.1030*           |
| H42 | 0.48800       | 1.02810       | 0.30850       | 0.1030*           |
| H51 | 0.42710       | 0.80410       | 0.31750       | 0.1020*           |
| H52 | 0.45340       | 0.83290       | 0.16040       | 0.1020*           |
| H61 | 0.31300       | 0.87170       | 0.24740       | 0.0700*           |
| H62 | 0.33780       | 0.75160       | 0.13760       | 0.0700*           |
| H4A | 0.14530       | 0.28900       | −0.13600      | 0.0300*           |
| H11A| 0.0869 (17)   | 0.663 (3)     | 0.007 (3)     | 0.027 (8)*        |
| H12A| 0.161 (2)     | 0.722 (4)     | −0.035 (4)    | 0.061 (11)*       |
| H21A| 0.15340       | 0.56100       | −0.23520      | 0.0350*           |
| H22A| 0.08450       | 0.65690       | −0.23390      | 0.0350*           |
| H31A| 0.02300       | 0.46550       | −0.12910      | 0.0310*           |
H32A  0.05690   0.40530   −0.27020   0.0310*  
H41A  0.0623 (18) −0.017 (4) −0.045 (3)  0.037 (10)*  
H42A  0.1263 (17)  0.054 (3)  −0.082 (3)  0.017 (8)*  
H51A  0.08680   0.40010   0.12130   0.0350*  
H52A  0.15560   0.30510   0.11510   0.0350*  
H61A  0.18260   0.55830   0.14880   0.0390*  
H62A  0.21480   0.49810   0.00550   0.0390*  

Atomic displacement parameters (Å²)

|       | U₁₁   | U₂₂   | U₃₃   | U₁₂   | U₁₃   | U₂₃   |
|-------|-------|-------|-------|-------|-------|-------|
| O11   | 0.0398 (14) | 0.0352 (14) | 0.0811 (19) | −0.0182 (11) | −0.0039 (13) | 0.0027 (13) |
| O12   | 0.0305 (12) | 0.0545 (16) | 0.0393 (13) | −0.0007 (11) | −0.0015 (10) | 0.0068 (11) |
| O21   | 0.0385 (14) | 0.0496 (15) | 0.0590 (16) | −0.0112 (11) | 0.0170 (12)  | −0.0067 (12) |
| O22   | 0.0473 (15) | 0.0479 (16) | 0.0512 (15) | −0.0127 (12) | 0.0156 (12)  | −0.0159 (12) |
| C1    | 0.0240 (16) | 0.0363 (19) | 0.0483 (19) | 0.0031 (14)  | 0.0016 (14)  | −0.0011 (15) |
| C2    | 0.0211 (15) | 0.0402 (19) | 0.0398 (18) | −0.0042 (13) | 0.0006 (13)  | −0.0027 (14) |
| C3    | 0.028 (2)   | 0.074 (3)   | 0.088 (3)   | −0.0069 (19) | −0.001 (2)   | −0.025 (2)   |
| C4    | 0.043 (3)   | 0.111 (5)   | 0.102 (4)   | 0.009 (3)    | −0.035 (3)   | −0.007 (3)   |
| C5    | 0.053 (3)   | 0.097 (4)   | 0.103 (4)   | 0.028 (3)    | −0.022 (3)   | 0.026 (3)    |
| C6    | 0.046 (2)   | 0.047 (2)   | 0.082 (3)   | 0.0090 (18)  | −0.008 (2)   | 0.020 (2)    |
| C11   | 0.0273 (16) | 0.0242 (16) | 0.0403 (17) | −0.0002 (13) | −0.0005 (13) | −0.0073 (14) |
| C21   | 0.0277 (17) | 0.0436 (19) | 0.0294 (16) | −0.0014 (15) | −0.0036 (13) | −0.0003 (14) |
| O41A  | 0.0228 (11) | 0.0265 (11) | 0.0401 (12) | −0.0011 (9)  | 0.0010 (9)   | 0.0013 (9)   |
| N1A   | 0.0274 (15) | 0.0253 (14) | 0.0351 (14) | 0.0002 (12)  | −0.0004 (11) | −0.0009 (11) |
| N41A  | 0.0188 (14) | 0.0291 (15) | 0.0381 (15) | −0.0049 (12) | 0.0026 (11)  | 0.0020 (11)  |
| C2A   | 0.0288 (16) | 0.0317 (16) | 0.0264 (15) | 0.0014 (13)  | −0.0010 (12) | 0.0075 (13)  |
| C3A   | 0.0257 (15) | 0.0310 (16) | 0.0200 (14) | −0.0011 (13) | −0.0046 (11) | 0.0013 (12)  |
| C4A   | 0.0217 (14) | 0.0258 (15) | 0.0278 (15) | 0.0000 (12)  | −0.0006 (12) | 0.0001 (12)  |
| C5A   | 0.0326 (17) | 0.0284 (16) | 0.0259 (15) | 0.0002 (13)  | −0.0092 (12) | 0.0037 (12)  |
| C6A   | 0.0378 (18) | 0.0270 (16) | 0.0318 (16) | −0.0027 (14) | −0.0123 (13) | 0.0051 (13)  |
| C41A  | 0.0240 (15) | 0.0253 (15) | 0.0211 (14) | −0.0014 (12) | −0.0063 (11) | 0.0014 (12)  |

Geometric parameters (Å, °)

|       |       | C3—H31 | 0.9700 |
|-------|-------|--------|-------|
| O11—C11 | 1.246 (4) | 1.260 (4) | 1.211 (4) | 1.317 (4) | 0.93 (5) | 1.250 (3) | 1.490 (4) | 1.478 (4) | 1.322 (4) | 0.99 (4) | 0.97 (3) | 0.86 (3) | 0.77 (3) | 1.538 (5) | 1.537 (5) | 0.9700 |

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| Bond            | Distance (Å) | Bond Angle (°) | Value  |
|-----------------|--------------|----------------|--------|
| C1—C11          | 1.520 (4)    |                | 0.9700 |
| C2—C3           | 1.531 (5)    |                | 0.9700 |
| C2—C21          | 1.514 (5)    |                | 0.9800 |
| C3—C4           | 1.505 (7)    |                | 0.9700 |
| C4—C5           | 1.506 (8)    |                | 0.9700 |
| C5—C6           | 1.530 (6)    |                | 0.9700 |
| C1—H1           | 0.9800       |                |        |
| C2—H2           | 0.9800       |                |        |
| C21—O22—H22     | 111 (3)      |                | 109.00 |
| C2A—N1A—C6A     | 112.4 (2)    |                | 112.7 (2) |
| C6A—N1A—H11A    | 114.9 (16)   |                | 109.00 |
| C11A—N1A—H12A   | 106 (3)      |                | 109.00 |
| C6A—N1A—H12A    | 106 (2)      |                | 109.00 |
| C2A—N1A—H12A    | 112 (2)      |                | 109.00 |
| C6A—N1A—H11A    | 104.7 (17)   |                | 109.00 |
| H41A—N41A—H42A  | 122 (3)      |                | 109.00 |
| C41A—N41A—H42A  | 119 (2)      |                | 109.00 |
| C2A—C3—C4       | 112.5 (3)    |                | 112.7 (2) |
| C2—C6—C5        | 112.1 (3)    |                | 112.3 (2) |
| C1—C11—C21      | 112.6 (2)    |                | 110.5 (2) |
| C3—C4—C5        | 112.7 (3)    |                | 107.8 (2) |
| C1—C2—C3        | 110.6 (3)    |                | 111.6 (2) |
| C1—C11—C21      | 111.2 (3)    |                | 110.1 (2) |
| C2—C3—C4        | 112.0 (3)    |                | 121.7 (2) |
| C3—C4—C5        | 110.8 (4)    |                | 116.3 (2) |
| C4—C5—C6        | 112.3 (4)    |                | 121.9 (3) |
| C1—C6—C5        | 111.4 (3)    |                | 110.00 |
| O11—C11—O12     | 123.5 (3)    |                | 110.00 |
| O11—C11—C1      | 118.6 (3)    |                | 110.00 |
| O12—C11—C1      | 118.0 (3)    |                | 110.00 |
| O21—C21—C2      | 124.3 (3)    |                | 108.00 |
| O22—C21—C2      | 112.7 (3)    |                | 109.00 |
| O21—C21—O22     | 123.0 (3)    |                | 109.00 |
| C6—C1—H1        | 106.00       |                | 109.00 |
| C2—C1—H1        | 106.00       |                | 109.00 |
| C11—C1—H1       | 106.00       |                | 109.00 |
| C3—C2—H2        | 107.00       |                | 109.00 |
| C1—C2—H2        | 107.00       |                | 109.00 |
| C21—C2—H2       | 107.00       |                | 109.00 |
| C4—C3—H31       | 109.00       |                | 109.00 |
| C2—C3—H31       | 109.00       |                | 109.00 |
| H31—C3—H32      | 108.00       |                | 109.00 |
| C4—C3—H32       | 109.00       |                | 109.00 |
| C2—C3—H32       | 109.00       |                | 109.00 |
| C5—C4—H42       | 110.00       |                | 110.00 |
| C3—C4—H42       | 110.00       |                | 110.00 |

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C5—C4—H41 110.00 C5A—C6A—H62A 110.00
C3—C4—H41 109.00 H61A—C6A—H62A 108.00
C6—C5—H51 109.00

C2A—N1A—C6A—C5A −58.9 (3) C3—C2—C21—O22 59.5 (4)
C6A—N1A—C2A—C3A 58.5 (3) C1—C2—C21—O21 2.3 (5)
C11—C1—C2—C3 179.7 (3) C2—C3—C4—C5 57.2 (5)
C11—C1—C2—C21 55.0 (4) C3—C4—C5—C6 −56.7 (5)
C6—C1—C2—C21 −73.1 (3) C4—C5—C6—C1 53.8 (5)
C11—C1—C6—C5 −179.3 (3) N1A—C2A—C3A—C4A −55.6 (3)
C6A—N1A—C2A—C3A 58.5 (3) C2A—C3A—C4A—C5A 53.0 (3)
C21—C2—C3—C4 43.4 (4) C2A—C3A—C4A—C41A 173.4 (2)
C21—C2—C1—C11 −9.1 (4) C41A—C4A—C5A—C6A −175.6 (2)
C6—C1—C11—O11 171.3 (3) C3A—C4A—C41A—O41A −40.5 (4)
C6—C1—C11—O12 51.7 (4) C3A—C4A—C41A—N41A 141.6 (3)
C2—C1—C11—O11 −137.0 (3) C5A—C4A—C41A—O41A 81.5 (3)
C2—C1—C11—O12 −54.8 (5) C5A—C4A—C41A—N41A −96.5 (3)
C6—C1—C6—C5 51.7 (4) C3A—C4A—C5A—C6A −52.5 (3)
C1—C2—C21—O22 −175.4 (3) C4A—C5A—C6A—N1A 55.2 (3)
C3—C2—C21—O21 −122.8 (4)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|-------|-------|---------|
| N1A—H11A···O41Aα | 0.97 (3) | 1.95 (3) | 2.861 (3) | 155 (2) |
| N1A—H12A···O11 | 0.99 (4) | 1.64 (4) | 2.588 (4) | 158 (3) |
| N41A—H41A···O41Aβ | 0.86 (3) | 2.14 (4) | 2.996 (3) | 174 (2) |
| N41A—H42A···O12β | 0.77 (3) | 2.11 (3) | 2.882 (4) | 177 (3) |
| O22—H22···O12ν | 0.93 (5) | 1.64 (5) | 2.571 (3) | 173 (4) |
| C44A—H44A···O21ου | 0.98 | 2.49 | 3.340 (4) | 145 |
| C24A—H24A···O21υ | 0.97 | 2.57 | 3.389 (4) | 143 |
| C24A—H22A···O41Aυ | 0.97 | 2.59 | 3.413 (3) | 143 |
| C3—H32···O22 | 0.97 | 2.52 | 2.884 (5) | 102 |
| C64A—H61A···O12ω | 0.97 | 2.58 | 3.351 (3) | 137 |

Symmetry codes: (i) x, −y+1, −z; (ii) −x, −y, −z; (iii) x, y−1, z; (iv) x, −y+5/2, z+1/2; (v) x, −y+3/2, z−1/2; (vi) −x, y+1/2, −z−1/2; (vii) x, −y+3/2, z+1/2.