O(38)—C(29)	1.222 (4)	C(2)—C(11)	1.560 (6)
O(47)—C(46)	1.221 (4)	C(4)—C(5)	1.492 (6)
N(3)—C(2)	1.448 (5)	C(7)—C(8)	1.547 (5)
N(3)—C(4)	1.472 (5)	C(7)—C(22)	1.528 (6)
N(3)—C(18)	1.349 (6)	C(11)—C(12)	1.500 (7)
N(6)—C(1)	1.364 (5)	C(18)—C(20)	1.523 (6)
N(6)—C(5)	1.461 (5)	C(22)—C(23)	1.517 (6)
N(6)—C(7)	1.465 (5)	C(29)—C(30)	1.511 (6)
N(21)—C(20)	1.468 (5)	C(30)—C(39)	1.547 (6)
N(21)—C(36)	1.326 (5)	C(32)—C(33)	1.473 (6)
N(31)—C(30)	1.473 (4)	C(35)—C(36)	1.549 (5)
N(31)—C(32)	1.460 (5)	C(35)—C(50)	1.529 (6)
N(31)—C(46)	1.337 (5)	C(39)—C(40)	1.508 (6)
N(34)—C(29)	1.350 (5)	C(46)—C(48)	1.525 (5)
N(34)—C(33)	1.471 (4)	C(50)—C(51)	1.514 (6)

C(2)—N(3)—C(4)	113.2 (3)	C(8)—C(7)—C(22)	111.3 (3)
C(2)—N(3)—C(18)	127.2 (3)	O(9)—C(8)—N(49)	122.2 (4)
C(4)—N(3)—C(18)	119.0 (4)	O(9)—C(8)—C(7)	122.0 (3)
C(1)—N(6)—C(5)	123.7 (3)	N(49)—C(8)—C(7)	115.9 (3)
C(1)—N(6)—C(7)	118.5 (3)	O(19)—C(18)—N(3)	121.4 (4)
C(5)—N(6)—C(7)	117.6 (3)	O(19)—C(18)—C(20)	117.9 (4)
C(20)—N(21)—C(36)	119.2 (3)	N(3)—C(18)—C(20)	120.7 (4)
C(30)—N(31)—C(32)	112.9 (3)	N(21)—C(20)—C(18)	109.4 (3)
C(30)—N(31)—C(46)	118.4 (3)	O(38)—C(29)—N(34)	122.4 (4)
C(32)—N(31)—C(46)	128.7 (3)	O(38)—C(29)—C(30)	117.8 (3)
C(29)—N(34)—C(33)	122.7 (3)	N(34)—C(29)—C(30)	119.7 (3)
C(29)—N(34)—C(35)	119.8 (3)	N(31)—C(30)—C(29)	112.6 (3)
C(33)—N(34)—C(35)	116.8 (3)	N(31)—C(30)—C(39)	111.1 (3)
C(8)—N(49)—C(48)	121.5 (3)	C(29)—C(30)—C(39)	110.9 (3)
O(10)—C(1)—N(6)	123.2 (4)	N(31)—C(32)—C(33)	110.0 (3)
O(10)—C(1)—C(2)	118.0 (3)	N(34)—C(33)—C(32)	111.9 (3)
N(6)—C(1)—C(2)	118.9 (3)	N(34)—C(35)—C(36)	107.5 (3)
N(3)—C(2)—C(1)	114.3 (3)	N(34)—C(35)—C(50)	112.3 (3)
N(3)—C(2)—C(11)	112.2 (4)	C(36)—C(35)—C(50)	115.6 (3)
C(1)—C(2)—C(11)	108.2 (3)	O(37)—C(36)—C(35)	119.4 (3)
N(3)—C(4)—C(5)	110.0 (3)	N(21)—C(36)—C(35)	118.0 (3)
N(6)—C(5)—C(4)	111.4 (3)	O(47)—C(46)—C(48)	119.8 (3)
N(6)—C(7)—C(8)	110.1 (3)	N(31)—C(46)—C(48)	118.0 (3)
N(6)—C(7)—C(22)	112.5 (3)	N(49)—C(48)—C(46)	106.3 (3)

N(3)—C(2)—C(1)—N(6)	−10.7 (5)
N(3)—C(4)—C(5)—N(6)	51.1 (5)
N(3)—C(18)—C(20)—N(21)	116.4 (4)
N(6)—C(7)—C(8)—N(49)	62.1 (4)
N(21)—C(36)—C(35)—N(34)	−133.4 (3)
N(31)—C(30)—C(29)—N(34)	−19.4 (5)
N(31)—C(32)—C(33)—N(34)	50.9 (5)
N(31)—C(46)—C(48)—N(49)	−87.2 (4)
C(1)—N(6)—C(5)—C(4)	−23.2 (5)
C(1)—N(6)—C(7)—C(8)	−128.5 (3)
C(1)—C(2)—N(3)—C(4)	40.4 (5)
C(1)—C(2)—N(3)—C(18)	−130.8 (4)
C(1)—C(2)—C(11)—C(12)	165.2 (4)
C(2)—N(3)—C(4)—C(5)	−61.7 (5)
C(2)—C(1)—N(6)—C(5)	2.6 (5)
C(8)—N(49)—C(48)—C(46)	−169.0 (4)
C(8)—C(7)—C(22)—C(23)	−168.9 (3)
C(18)—C(20)—N(21)—C(36)	−80.9 (5)
C(29)—N(34)—C(33)—C(32)	−27.4 (5)
C(29)—N(34)—C(35)—C(36)	−111.8 (4)
C(29)—C(30)—N(31)—C(32)	44.7 (4)
C(29)—C(30)—N(31)—C(46)	−134.0 (4)
C(29)—C(30)—C(39)—C(40)	85.9 (4)
C(30)—N(31)—C(32)—C(33)	−61.9 (4)
C(30)—C(29)—N(34)—C(33)	11.5 (6)
C(36)—C(35)—C(50)—C(51)	−168.2 (4)

Program used for data collection and cell refinement: *MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). Programs used to solve structure: *SHELXS*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens *et al.*, 1992). Program used for all calculations, including data reduction: *TEXSAN* (Molecular Structure Corporation, 1992).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1202). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hydrogen-Bonding Pattern of a β-Keto Acid: (±)-10-Oxo-3,4-benzobicyclo[4.3.1]dec-3-ene-1-carboxylic Acid

Marie L. Cote, Hugh W. Thompson and Roger A. Lalancette*

Carl A. Olson Memorial Laboratories, Department of Chemistry, Rutgers University, Newark, NJ 07102, USA

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Abstract

In the title compound, $C_{15}H_{16}O_3$, enantiomeric pairs of molecules form centrosymmetric dimers by mutual hydrogen bonding of carboxy groups, with the ketone not involved in the hydrogen bonding. The plane of the carboxy group [O(2)—C(11)—O(3)] makes a

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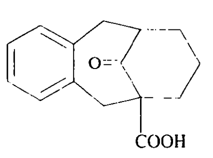
dihedral angle of 86.3 (2)° with the plane of the ketone {O(1)—C(10)[—C(1)]—C(6)}. The observed C—O bond lengths and angles in the carboxy group are equivalent due to disorder. There is a reciprocal intermolecular 2.570 (4) Å contact between an aromatic H atom [H(14*A*)] and the ketone O atom [O(1)].

Comment

Almost all simple crystalline carboxylic acids exist as mutually hydrogen-bonded pairs (Leiserowitz, 1976; Berkovitch-Yellin & Leiserowitz, 1982), but in the presence of additional functional groups capable of hydrogen bonding, other patterns may become favored. We have studied simple crystalline keto-carboxylic acids, which have four known hydrogen-bonding arrangements dictating or affecting the intermolecular association. The commonest retains the simple dimer pattern, in which the ketone is not involved (Lalancette, Vanderhoff & Thompson, 1990; Lalancette, Thompson & Vanderhoff, 1991; Thompson, Vanderhoff & Lalancette, 1991; Thompson, Lalancette & Vanderhoff, 1992). Less commonly, an intermolecular carboxy-to-ketone hydrogen bond repeats infinitely along one cell axis to yield a chain, or catemer (Vanderhoff, Thompson & Lalancette, 1986; Lalancette, Slifer & Thompson, 1989; Lalancette, Vanderhoff & Thompson, 1990, 1991; Lalancette, Thompson & Vanderhoff, 1991). A third, rare arrangement is an internal hydrogen bond (Griffe, Durant & Pieret, 1972; Sheldrick & Trowitzsch, 1983; Abell, Morris & McKee, 1990; Halfpenny, 1990), and one instance is known of acid-to-ketone dimerization (Abell, Trent & Morris, 1991).

In β-keto acids the geometry appears favorable for internal hydrogen bonding (Toffoli, Khodadad & Rodier, 1988). A considerable volume of literature suggests the importance of such hydrogen bonding in solution, where it has been implicated indirectly in the thermal instability of β-keto acids (Hay & Bond, 1967; Grande & Rosenfeld, 1980). Their thermal decarboxylation is known to proceed by a six-centered internal H-atom transfer, for which the corresponding internally hydrogen-bonded structure seems a logical precursor (Logue, Pollack & Vitullo, 1975). Crystallographically, however, to our knowledge, this hydrogen-bonding mode has been found only in γ- or 4-keto acids, requiring a seven-membered hydrogen-bonding ring. Although such internal hydrogen bonding occurs in several β-oxo acids where the oxo function is actually part of a vinylogous amide or ester, no evidence for it in simple crystalline β-keto acids has been found, but thermal instability makes β-keto acids, as a class, less available, so few crystal structures are known. However, β-keto acids in which decarboxylation introduces serious strain (*e.g.* Bredt's rule) are adequately stable, as in our title compound, (I), m.p. 448 K, which was prepared *via* condensation of 2-carbethoxycyclohexanone with dibromo-*o*-xylene.



(I)

Fig. 1 presents a view of the molecule, with its numbering scheme. The cyclohexanone is in a chair form, with the aromatic ring flexed in the direction of the ketone. The only completely free bond rotation available is around the bond C(1)—C(11), joining the carboxy group to the benzobicyclic cage. The preferred plane of the carboxy group [O(2)—C(11)—O(3)] is at a dihedral angle of 86.3 (2)° to the plane of the ketone {O(1)—C(10)[—C(1)]—C(6)}. In the few other true β-keto acids with known X-ray structures, the corresponding dihedral angle is found to vary between 58 and 90°: BKGLUT, 79.7° (Martuscelli & Avitabile, 1967); GIMSIE, 60.0° (Ermer & Lindenberg, 1988); KIGCAE, 63.3°, ZZZBLSO1, 67.3° (Lalancette, Vanderhoff & Thompson, 1991); TANPIH, 90° (Thompson, Lalancette & Vanderhoff, 1992); TOXGLU10, 78.9 and 87.8° (Avitabile, Ganis & Martuscelli, 1969); TOXGLU11, 58.0 and 83.2° (Avitabile, Ganis & Lepore, 1971).

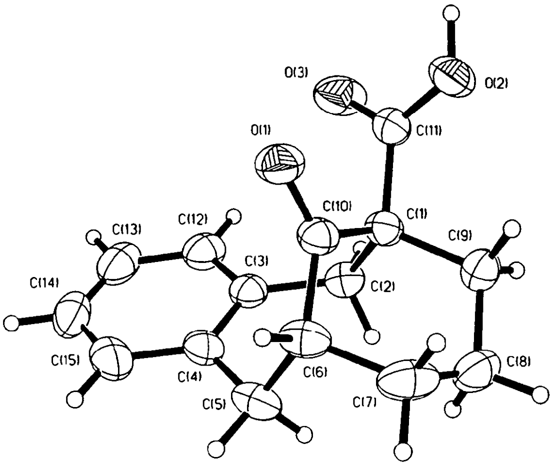


Fig. 1. A view of the title compound with the numbering scheme shown with ellipsoids at the 40% probability level. An idealized H atom is added to the carboxy group for clarity (even though it was never found in difference maps).

Fig. 2 illustrates the centrosymmetric dimers formed through carboxy-pairing of enantiomers across the *a* cell edge. The carboxy C—O bond lengths are found to be intermediate between single- and double-bond lengths, and essentially equal to each other because of the disordering of the O-atom positions. The bond lengths are 1.252 (4) and 1.259 (4) Å, and the C—C—O angles are 118.5 (3) and 119.3 (3)°, in contrast to typical lengths of 1.21 and 1.31 Å, and angles of 112 and

123° in ordered carboxy dimers (Borthwick, 1980). We have previously discussed implications and experimental expectations associated with such disorder (Thompson, Lalancette & Vanderhoff, 1992). Because the present data were acquired at 297 K, several of the phenomena expected to result from such disordering (*e.g.* doubling of the acid H atoms) were not observed.

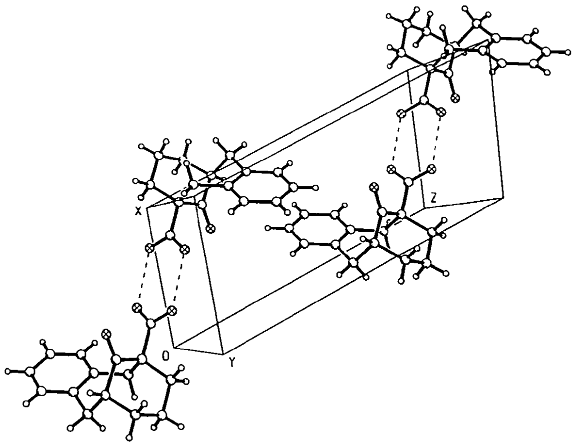


Fig. 2. A partial packing diagram for the title compound. Extracellular molecules illustrate the centrosymmetric hydrogen-bonded carboxy dimer (in its disordered state without the carboxy H atoms), centered on the *a* cell edge. Also shown is the 2.570 (4) Å reciprocal intermolecular contact across the *ac* cell face between the ketonic O atom, O(1), and the aromatic H atom, H(14*A*).

Apart from the dimerization, the closest intermolecular contact occurs reciprocally between the ketonic O atom, O(1), and an aromatic H atom, H(14*A*), of the enantiomeric molecule related centrosymmetrically across the *ac* cell face [2.570 (4) Å], illustrated in Fig. 2. This probably represents a significant polar attraction contributing to the packing forces (Leiserowitz, 1976; Jönsson, 1972; Berkovitch-Yellin & Leiserowitz, 1982). The parallel aromatic rings lie in planes 3.284 Å apart.

Although models of the title compound can be arranged convincingly for an internal hydrogen bond, that conformation is not found. In fact, in the absence of additional hydrogen-bonding constraints (Destro & Marsh, 1984), every β-keto acid we know of whose hydrogen-bonding mode has been determined crystallographically forms hydrogen bonds either by carboxy dimerization (referenced above for BKGLUT, GIMSIE, TANPIH and TOXGLU) or by catemerization (Lalancette, Vanderhoff & Thompson, 1991; Thompson, Lalancette & Vanderhoff, 1992).

The solid-state (KBr) IR spectrum of the title compound displays C=O stretching absorptions at 1712 cm^{-1} (ketone) and 1694 cm^{-1} (carboxy), normal for keto acids with this type of hydrogen-bonding pattern.

Experimental

Crystal data

$C_{15}H_{16}O_3$
$M_r = 244.279$
Triclinic
$P\overline{1}$
$a = 6.629\ (1)$ Å
$b = 7.908\ (1)$ Å
$c = 12.985\ (1)$ Å
$\alpha = 82.556\ (8)°$
$\beta = 76.504\ (9)°$
$\gamma = 67.621\ (7)°$
$V = 611.5\ (2)$ Å^3
$Z = 2$
$D_x = 1.327$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 34 reflections
$\theta = 6.7–18.9°$
$\mu = 0.091$ mm^{-1}
$T = 297$ K
Parallelepiped
0.60 × 0.48 × 0.08 mm
Colorless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: analytical
$T_{\min} = 0.955$, $T_{\max} = 0.988$
2351 measured reflections
2147 independent reflections
1419 observed reflections [$F > 4\sigma(F)$]

$R_{\text{int}} = 0.0433$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 7$
$k = -8 \rightarrow 9$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 47 reflections
intensity decay: 1.14%

Refinement

Refinement on *F*
$R = 0.0450$
$wR = 0.0581$
$S = 1.20$
1419 reflections
167 parameters
H atoms refined riding on associated C atoms
$w = 1/[\sigma^2(F) + 0.00070F^2]$
$(\Delta/\sigma)_{\max} = 0.018$

$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Extinction correction: empirical isotropic
Extinction coefficient: 0.0142
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)*

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

	x	*y*	*z*	U_{eq}
O(1)	0.3823 (3)	0.2854 (3)	0.7466 (1)	0.057 (1)
O(2)	0.2849 (3)	0.2033 (3)	0.9873 (1)	0.067 (1)
O(3)	0.3877 (3)	−0.0533 (2)	0.9071 (2)	0.069 (1)
C(1)	0.0853 (4)	0.2033 (3)	0.8553 (2)	0.040 (1)
C(2)	0.0105 (4)	0.0599 (3)	0.8184 (2)	0.044 (1)
C(3)	0.1325 (4)	−0.0167 (3)	0.7118 (2)	0.044 (1)
C(4)	0.1079 (4)	0.0982 (3)	0.6197 (2)	0.048 (1)
C(5)	−0.0420 (4)	0.2961 (3)	0.6299 (2)	0.055 (1)
C(6)	0.0370 (4)	0.4127 (3)	0.6874 (2)	0.050 (1)
C(7)	−0.1574 (4)	0.5600 (3)	0.7537 (2)	0.062 (1)
C(8)	−0.2772 (4)	0.4785 (3)	0.8495 (2)	0.058 (1)
C(9)	−0.1163 (4)	0.3537 (3)	0.9186 (2)	0.052 (1)
C(10)	0.1908 (4)	0.2965 (3)	0.7598 (2)	0.042 (1)
C(11)	0.2642 (4)	0.1106 (3)	0.9212 (2)	0.044 (1)
C(12)	0.2633 (4)	−0.2006 (4)	0.7024 (2)	0.056 (1)
C(13)	0.3699 (5)	−0.2744 (5)	0.6048 (3)	0.070 (1)
C(14)	0.3485 (5)	−0.1619 (5)	0.5150 (3)	0.073 (2)
C(15)	0.2182 (5)	0.0230 (4)	0.5220 (2)	0.063 (1)

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

O(1)—C(10)	1.210 (3)	O(2)—C(11)	1.259 (4)
O(3)—C(11)	1.252 (3)	C(1)—C(2)	1.563 (4)
C(1)—C(9)	1.551 (3)	C(1)—C(10)	1.524 (3)
C(1)—C(11)	1.525 (3)	C(2)—C(3)	1.503 (3)
C(3)—C(4)	1.406 (3)	C(3)—C(12)	1.385 (3)
C(4)—C(5)	1.504 (3)	C(4)—C(15)	1.389 (4)
C(5)—C(6)	1.553 (5)	C(6)—C(7)	1.535 (3)
C(6)—C(10)	1.510 (3)	C(7)—C(8)	1.522 (4)
C(8)—C(9)	1.527 (4)	C(12)—C(13)	1.381 (4)
C(13)—C(14)	1.372 (5)	C(14)—C(15)	1.389 (5)
O(2)···O(3$^{\rm i}$)	2.647 (3)		
C(2)—C(1)—C(9)	111.0 (2)	C(2)—C(1)—C(10)	110.3 (2)
C(9)—C(1)—C(10)	107.9 (2)	C(2)—C(1)—C(11)	111.0 (2)
C(9)—C(1)—C(11)	110.5 (2)	C(10)—C(1)—C(11)	105.9 (2)
C(1)—C(2)—C(3)	115.9 (2)	C(2)—C(3)—C(4)	119.7 (2)
C(2)—C(3)—C(12)	120.9 (2)	C(4)—C(3)—C(12)	119.3 (2)
C(3)—C(4)—C(5)	119.2 (2)	C(3)—C(4)—C(15)	118.6 (2)
C(5)—C(4)—C(15)	122.2 (2)	C(4)—C(5)—C(6)	115.2 (2)
C(5)—C(6)—C(7)	112.7 (2)	C(5)—C(6)—C(10)	112.0 (2)
C(7)—C(6)—C(10)	107.9 (2)	C(6)—C(7)—C(8)	112.4 (2)
C(7)—C(8)—C(9)	111.5 (2)	C(1)—C(9)—C(8)	112.8 (2)
O(1)—C(10)—C(1)	121.8 (2)	O(1)—C(10)—C(6)	122.5 (2)
C(1)—C(10)—C(6)	115.6 (2)	O(2)—C(11)—O(3)	122.1 (2)
O(2)—C(11)—C(1)	118.5 (2)	O(3)—C(11)—C(1)	119.3 (3)
C(3)—C(12)—C(13)	121.6 (3)	C(12)—C(13)—C(14)	119.1 (3)
C(13)—C(14)—C(15)	120.5 (3)	C(4)—C(15)—C(14)	120.9 (3)
C(11)—O(2)···O(3$^{\rm i}$)	119.2 (2)	C(11)—O(3)···O(2$^{\rm i}$)	118.6 (2)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
C(14)—H(14A)···O(1$^{\rm ii}$)	2.570 (4)	3.513 (4)	169.4 (1)

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

The structure was solved by direct methods. Integrated intensities were corrected for Lorentz and polarization effects. Full-matrix least-squares refinement using *SHELXTL/PC* (Sheldrick, 1990) was performed. All non-H atoms were refined anisotropically. Although most H atoms were found in difference maps, they were nonetheless replaced by H atoms in calculated positions and refined with a riding model, which gave a slightly better goodness-of-fit.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1015). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl 3-(*RS*)-Hydroxy-4-[(*RS*)-3,4,5,6-tetrahydro-2*H*-2-pyranyloxy]cyclohexanecarboxylate

MADI BIDYASAGAR,[a] KRISHNAN RAVIKUMAR,[a] SAID JAWED HASSAN[b] AND J. S. YADAV[b]

[a]*Laboratory of Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and* [b]*Organic Chemistry Division, Indian Institute of Chemical Technology, Hyderabad 500 007, India*

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Abstract

In the title compound, $C_{13}H_{22}O_5$, an intramolecular hydrogen bond between the hydroxy group and the O atom of the tetrahydropyranyl moiety results in a seven-membered ring which displays a twist-chair conformation.

Comment

Interest in the structure of substituted cyclohexanes arises from their presence in immunosuppressant macrolides like FK 506 and rapamycin. An *ORTEP*II

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