C(9*A*)	0.3782 (5)	0.0747 (1)	0.4710 (2)	0.044 (1)
C(10*A*)	0.5195 (5)	0.0717 (1)	0.3773 (3)	0.057 (1)
C(11*A*)	0.4422 (7)	0.0829 (1)	0.2560 (3)	0.074 (2)
C(12*A*)	0.2270 (7)	0.0979 (1)	0.2268 (3)	0.072 (1)
C(13*A*)	0.0884 (6)	0.1014 (1)	0.3184 (3)	0.064 (1)
C(14*A*)	0.1611 (5)	0.0896 (1)	0.4401 (3)	0.051 (1)
O(1*B*)	−0.5164 (4)	0.1857 (1)	0.2330 (2)	0.072 (1)
O(2*B*)	−0.2231 (4)	0.1416 (1)	0.0544 (2)	0.067 (1)
O(3*B*)	−0.4897 (4)	0.1662 (1)	−0.0867 (2)	0.073 (1)
C(1*B*)	−0.2383 (5)	0.2002 (1)	0.0518 (2)	0.047 (1)
C(2*B*)	−0.1398 (5)	0.2021 (1)	0.1934 (2)	0.044 (1)
C(3*B*)	−0.0833 (5)	0.2381 (1)	0.2279 (3)	0.055 (1)
C(4*B*)	0.0961 (6)	0.2520 (1)	0.1506 (3)	0.063 (1)
C(5*B*)	0.0183 (6)	0.2475 (1)	0.0085 (3)	0.068 (1)
C(6*B*)	−0.0527 (6)	0.2126 (1)	−0.0260 (3)	0.062 (1)
C(7*B*)	−0.3179 (5)	0.1666 (1)	0.0081 (3)	0.048 (1)
C(8*B*)	−0.3080 (5)	0.1877 (1)	0.2754 (3)	0.048 (1)
C(9*B*)	−0.2195 (5)	0.1765 (1)	0.4075 (3)	0.045 (1)
C(10*B*)	−0.3706 (5)	0.1592 (1)	0.4726 (3)	0.057 (1)
C(11*B*)	−0.2980 (6)	0.1489 (1)	0.5957 (3)	0.067 (1)
C(12*B*)	−0.0751 (6)	0.1558 (1)	0.6563 (3)	0.067 (1)
C(13*B*)	0.0764 (6)	0.1726 (1)	0.5935 (3)	0.068 (1)
C(14*B*)	0.0071 (5)	0.1825 (1)	0.4690 (3)	0.057 (1)

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

O(1*A*)—C(8*A*)	1.221 (3)	O(1*B*)—C(8*B*)	1.220 (3)
O(2*A*)—C(7*A*)	1.232 (4)	O(2*B*)—C(7*B*)	1.225 (4)
O(3*A*)—C(7*A*)	1.301 (3)	O(3*B*)—C(7*B*)	1.299 (3)
C(1*A*)—C(2*A*)	1.531 (3)	C(1*B*)—C(2*B*)	1.527 (3)
C(1*A*)—C(6*A*)	1.526 (4)	C(1*B*)—C(6*B*)	1.540 (4)
C(1*A*)—C(7*A*)	1.490 (4)	C(1*B*)—C(7*B*)	1.494 (4)
C(2*A*)—C(3*A*)	1.524 (4)	C(2*B*)—C(3*B*)	1.535 (4)
C(2*A*)—C(8*A*)	1.507 (4)	C(2*B*)—C(8*B*)	1.521 (4)
C(3*A*)—C(4*A*)	1.519 (5)	C(3*B*)—C(4*B*)	1.530 (5)
C(4*A*)—C(5*A*)	1.522 (4)	C(4*B*)—C(5*B*)	1.519 (4)
C(5*A*)—C(6*A*)	1.523 (5)	C(5*B*)—C(6*B*)	1.511 (5)
C(8*A*)—C(9*A*)	1.487 (4)	C(8*B*)—C(9*B*)	1.491 (4)
C(9*A*)—C(10*A*)	1.389 (4)	C(9*B*)—C(10*B*)	1.388 (4)
C(9*A*)—C(14*A*)	1.385 (5)	C(9*B*)—C(14*B*)	1.390 (4)
C(10*A*)—C(11*A*)	1.377 (4)	C(10*B*)—C(11*B*)	1.378 (4)
C(12*A*)—C(13*A*)	1.363 (5)	C(11*B*)—C(12*B*)	1.375 (5)
C(11*A*)—C(12*A*)	1.377 (5)	C(12*B*)—C(13*B*)	1.367 (5)
C(13*A*)—C(14*A*)	1.384 (4)	C(13*B*)—C(14*B*)	1.383 (4)
O(3*A*)···O(2B^{i})	2.655 (5)	O(3*B*)···O(2A^{ii})	2.630 (5)
H(16)···O(2A^{ii})	1.634 (4)	H(15)···O(2B^{i})	1.663 (4)
H(15)—O(3*A*)	0.997(4)	H(16)—O(3*B*)	1.000 (4)
C(2*A*)—C(1*A*)—C(6*A*)	110.3 (2)	C(2*B*)—C(1*B*)—C(6*B*)	109.4 (2)
C(2*A*)—C(1*A*)—C(7*A*)	112.0 (2)	C(2*B*)—C(1*B*)—C(7*B*)	113.7 (2)
C(6*A*)—C(1*A*)—C(7*A*)	111.1 (2)	C(6*B*)—C(1*B*)—C(7*B*)	109.8 (2)
C(1*A*)—C(2*A*)—C(3*A*)	110.3 (2)	C(1*B*)—C(2*B*)—C(3*B*)	108.4 (2)
C(1*A*)—C(2*A*)—C(8*A*)	112.3 (2)	C(1*B*)—C(2*B*)—C(8*B*)	112.1 (2)
C(3*A*)—C(2*A*)—C(8*A*)	108.8 (2)	C(3*B*)—C(2*B*)—C(8*B*)	111.1 (2)
C(2*A*)—C(3*A*)—C(4*A*)	112.0 (2)	C(2*B*)—C(3*B*)—C(4*B*)	111.2 (3)
C(3*A*)—C(4*A*)—C(5*A*)	111.0 (3)	C(3*B*)—C(4*B*)—C(5*B*)	111.9 (3)
C(4*A*)—C(5*A*)—C(6*A*)	111.1 (3)	C(4*B*)—C(5*B*)—C(6*B*)	112.4 (3)
C(1*A*)—C(6*A*)—C(5*A*)	111.1 (3)	C(1*B*)—C(6*B*)—C(5*B*)	111.3 (3)
O(2*A*)—C(7*A*)—O(3*A*)	122.2 (3)	O(2*B*)—C(7*B*)—O(3*B*)	122.6 (3)
O(2*A*)—C(7*A*)—C(1*A*)	122.5 (2)	O(2*B*)—C(7*B*)—C(1*B*)	122.8 (2)
O(3*A*)—C(7*A*)—C(1*A*)	115.3 (3)	O(3*B*)—C(7*B*)—C(1*B*)	114.5 (3)
O(1*A*)—C(8*A*)—C(2*A*)	120.4 (2)	O(1*B*)—C(8*B*)—C(2*B*)	119.7 (2)
O(1*A*)—C(8*A*)—C(9*A*)	120.0 (3)	O(1*B*)—C(8*B*)—C(9*B*)	120.0 (3)
C(2*A*)—C(8*A*)—C(9*A*)	119.5 (2)	C(2*B*)—C(8*B*)—C(9*B*)	120.3 (2)
C(8*A*)—C(9*A*)—C(10*A*)	118.1 (3)	C(8*B*)—C(9*B*)—C(10*B*)	118.5 (2)
C(8*A*)—C(9*A*)—C(14*A*)	123.1 (3)	C(8*B*)—C(9*B*)—C(14*B*)	123.2 (3)
C(10*A*)—C(9*A*)—C(14*A*)	118.7 (2)	C(10*B*)—C(9*B*)—C(14*B*)	118.3 (3)
C(9*A*)—C(10*A*)—C(11*A*)	120.2 (3)	C(9*B*)—C(10*B*)—C(11*B*)	120.6 (3)
C(10*A*)—C(11*A*)—C(12*A*)	120.5 (3)	C(10*B*)—C(11*B*)—C(12*B*)	120.4 (3)
C(11*A*)—C(12*A*)—C(13*A*)	119.7 (3)	C(11*B*)—C(12*B*)—C(13*B*)	119.8 (3)
C(12*A*)—C(13*A*)—C(14*A*)	120.6 (3)	C(12*B*)—C(13*B*)—C(14*B*)	120.3 (3)
C(9*A*)—C(14*A*)—C(13*A*)	120.3 (3)	C(9*B*)—C(14*B*)—C(13*B*)	120.6 (3)
O(2A^{ii})···H(16)—O(3*B*)	173.8 (2)	O(2B^{i})···H(15)—O(3*A*)	172.4 (2)
C(7*A*)—O(2*A*)···H(16^{ii})	122.5 (2)	C(7*B*)—O(2*B*)···H(15^{ii})	121.0 (2)
C(7*A*)—O(3*A*)—H(15)	108.4 (3)	C(7*B*)—O(3*B*)—H(16)	109.5 (3)

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

All diffractometer software was provided by Siemens Analytical X-ray Instruments Inc., including *SHELXTL/PC* (Sheldrick, 1990). The structure was solved by direct methods. Integrated intensities were corrected for Lorentz and polarization effects. Full-matrix least-squares refinement was performed. All non-H atoms were refined anisotropically. Both carboxyl H atoms were located in difference maps and allowed to refine as riding models on their associated O atoms. All other H atoms were placed in calculated positions and allowed to refine as riding models on their associated C atoms for optimum *R* factor and goodness-of-fit.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1013). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2*E*,4′*S*)-2-(4′-Benzyl-2′-oxo-3′-oxazolidinylcarbonyl)-3-phenylacrylic Acid Methyl Ester

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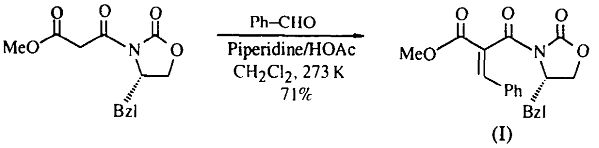
Abstract

The crystal structure of the title compound, $C_{21}H_{19}NO_5$, has been determined at 153 K in order to confirm the configuration of the double bond.

Comment

This work was part of our studies on the synthesis of alkylidene-1,3-dicarbonlyl compounds which are further used for the synthesis of *enantiopure trans*-1,2-disubstituted cyclopentanes and cyclohexanes. The five-membered oxazolidine ring of the title compound, (I), adopts a twist conformation with the twist axis through C2. Ring puckering parameters $q = 0.226$ and $\varphi = 301.9$ were calculated using the program *PUCKER* (Gould & Taylor, 1994). All bond lengths and angles observed are in the expected range.



(I)

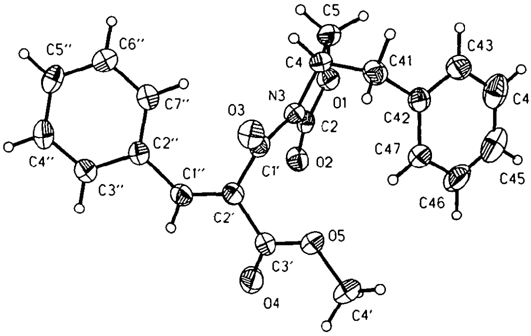


Fig. 1. Crystal structure of the title compound showing 50% probability displacement ellipsoids.

Experimental

The title compound was obtained *via* a Knoevenagel condensation of (4′*S*)-2-(4′-benzyl-2′-oxo-3′-oxazolidinylcarbonyl)-acetic acid methyl ester with benzaldehyde. Further details of the reaction are published in Tietze & Schünke (1995). The compound was crystallized from a mixture of diethyl ether and pentane at room temperature.

Crystal data

$C_{21}H_{19}NO_5$
$M_r = 365.37$
Monoclinic
$P2_1$
$a = 9.929\,(2)$ Å
$b = 8.750\,(2)$ Å
$c = 11.230\,(2)$ Å
$\beta = 107.21\,(2)°$
$V = 932.0\,(3)$ Å^3
$Z = 2$
$D_x = 1.302$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10$–$12.5°$
$\mu = 0.093$ mm^{-1}
$T = 153\,(2)$ K
Block
$0.8 \times 0.4 \times 0.4$ mm
Colourless

Data collection

Stoe–Huber four-circle diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction: none
3302 measured reflections
2469 independent reflections
2405 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0119$
$\theta_{\max} = 22.55°$
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 9$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0287$
$wR(F^2) = 0.0693$
$S = 1.058$
2469 reflections
245 parameters
H atoms refined using riding model
$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.1108P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.098$ e Å^{-3}
$\Delta\rho_{\min} = -0.151$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: $x = 0.3\,(9)$ (Flack, 1983)

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

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

	x	y	z	$U_{\rm eq}$
O1	0.19504 (12)	0.0719 (2)	0.56924 (11)	0.0339 (3)
C2	0.0984 (2)	0.0439 (2)	0.62812 (15)	0.0281 (4)
O2	0.06681 (14)	−0.08143 (15)	0.65189 (12)	0.0360 (3)
N3	0.04566 (15)	0.1828 (2)	0.65506 (12)	0.0265 (3)
C4	0.1287 (2)	0.3110 (2)	0.6308 (2)	0.0304 (4)
C5	0.1984 (2)	0.2334 (2)	0.5426 (2)	0.0350 (5)
C41	0.2313 (2)	0.3743 (2)	0.7506 (2)	0.0357 (5)
C42	0.3459 (2)	0.2645 (2)	0.8172 (2)	0.0330 (4)
C43	0.4779 (2)	0.2709 (3)	0.7997 (2)	0.0415 (5)
C44	0.5831 (2)	0.1700 (3)	0.8600 (2)	0.0526 (6)
C45	0.5565 (3)	0.0603 (3)	0.9366 (2)	0.0591 (6)
C46	0.4260 (3)	0.0526 (3)	0.9554 (2)	0.0624 (7)
C47	0.3221 (2)	0.1553 (3)	0.8969 (2)	0.0461 (6)
C1′	−0.0734 (2)	0.2035 (2)	0.69329 (15)	0.0276 (4)
O3	−0.10822 (13)	0.33082 (15)	0.71393 (12)	0.0364 (3)
C2′	−0.1561 (2)	0.0643 (2)	0.7050 (2)	0.0312 (4)
C3′	−0.1037 (2)	−0.0278 (3)	0.8198 (2)	0.0398 (5)
O4	−0.1502 (2)	−0.1492 (2)	0.8377 (2)	0.0625 (5)
O5	−0.00047 (13)	0.0462 (2)	0.90374 (11)	0.0453 (4)
C4′	0.0569 (3)	−0.0291 (4)	1.0227 (2)	0.0672 (8)
C1″	−0.2816 (2)	0.0280 (2)	0.6261 (2)	0.0316 (4)
C2″	−0.3625 (2)	0.0959 (2)	0.5073 (2)	0.0280 (4)
C3″	−0.5044 (2)	0.0536 (2)	0.4604 (2)	0.0343 (4)
C4″	−0.5873 (2)	0.1089 (2)	0.3481 (2)	0.0385 (5)
C5″	−0.5309 (2)	0.2066 (2)	0.2793 (2)	0.0361 (5)
C6″	−0.3914 (2)	0.2495 (2)	0.3239 (2)	0.0338 (4)
C7″	−0.3077 (2)	0.1958 (2)	0.4370 (2)	0.0318 (4)

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

O1—C2	1.340 (2)	C46—C47	1.379 (3)
O1—C5	1.446 (2)	C1′—O3	1.209 (2)
C2—O2	1.192 (2)	C1′—C2′	1.496 (3)
C2—N3	1.391 (2)	C2′—C1″	1.337 (3)
N3—C1′	1.384 (2)	C2′—C3′	1.478 (3)
N3—C4	1.465 (2)	C3′—O4	1.198 (3)
C4—C5	1.525 (3)	C3′—O5	1.336 (2)
C4—C41	1.531 (3)	O5—C4′	1.447 (3)
C2—O1—C5	110.16 (14)	O3—C1′—N3	119.9 (2)
O2—C2—O1	123.6 (2)	O3—C1′—C2′	122.5 (2)
O2—C2—N3	127.9 (2)	N3—C1′—C2′	117.5 (2)

O1—C2—N3	108.5 (2)	C1″—C2′—C3′	117.9 (2)
C1′—N3—C2	126.4 (2)	C1″—C2′—C1′	124.3 (2)
C1′—N3—C4	122.47 (15)	C3′—C2′—C1′	117.4 (2)
C2—N3—C4	111.09 (14)	O4—C3′—O5	124.3 (2)
N3—C4—C5	99.70 (14)	O4—C3′—C2′	125.2 (2)
N3—C4—C41	112.26 (14)	O5—C3′—C2′	110.5 (2)
C5—C4—C41	114.8 (2)	C3′—O5—C4′	116.2 (2)
O1—C5—C4	104.94 (14)		

Intensities were measured with a 'learnt profile' method (Clegg, 1981). The space group was determined from the Laue symmetry, the systematically absent reflections and the average value of $|E^2 - 1|$ using program *XPREP* (Sheldrick, 1994). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). All non-H atoms were refined anisotropically using *SHELXL*93 (Sheldrick, 1993). H atoms were refined using a riding model. The isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameter of the atom they are attached to. The absolute structure could not be determined as no significant anomalous scatterer was present. The known absolute structure of the educt was thus assumed (Tietze & Schünke, 1995). The floating origin restraint was applied (Flack & Schwarzenbach, 1988).

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1277). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Amino-3-methylpyridinium Bromide Monohydrate

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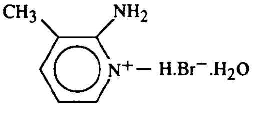
Abstract

The title compound, $C_6H_9N_2^+.Br^-.H_2O$, was crystallized from water by slow evaporation. The 2-amino-3-methylpyridinium cation shows bond lengths and angles comparable with those observed in more complex counterions.

Comment

As a result of our interest in low-dimensional magnetic lattices, we have been studying complexes of the family $(LH)_2MX_4$, where M is a 2+ first row transition metal ion, X is Cl or Br, and L is an organic base. These compounds are well known to pack in the crystal lattice such that low-dimensional magnetic lattices, arising from van der Waals contacts between the MX_4 ions, are frequently obtained (for examples see Place & Willett, 1987*a*,*b*). The nature of these contacts is determined by the organic base L. Several complexes, where L is 2-amino-3-methylpyridine, have been prepared and we were interested in seeing whether the pronounced effect of L on the packing of the anions was reciprocated by the anions altering the structure of L in the crystal lattice. The crystal structure determination of 2-amino-3-methylpyridinium bromide monohydrate, (I), was thus undertaken.



(I)

The general structure of the title material is unremarkable, with bond lengths and angles comparable with other structures containing the protonated moiety, where the anions are copper or palladium-based complex ions (Place & Willett, 1987*b*; Grigereit *et al.*, 1987; Roman, Beitia & Luque, 1993). The bonds were slightly longer than those of the neutral compound (Espenbetov, Struchkov, Poplavskaya & Kurman-

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