Le groupement $\frac{C}{C} > NO$ est légèrement pyramidal: l'angle du NO avec le plan $\frac{C}{C} > N$ vaut 7,6° pour $B(R)$ et 4,5° pour $B(E)$. Ces valeurs sont du même ordre de grandeur que celles trouvées dans d'autres cycles oxazolidiniques (7,2° pour $A$) et dans certains nitroxydes pyrrolidiniques (Chion & Lajzérowicz, 1975; Chion, Lajzérowicz, Bordeaux, Collet & Jacques, 1978). Les longueurs de liaison N–O de 1,27 Å sont également classiques.

La cristallisation des mélanges racémiques des épimères $A(R)$ et $B(R)$ est très différente: l'un cristallise sous forme de conglomérat, l'autre sous forme de racémate. Nous étudions actuellement les structures des mélanges $A(R)$ et $B(R)$ qui cristallisent dans certaines conditions et dans une large gamme de concentration $A(R)_x B(R)_{1-x}$ sous forme de solution solide; une démixtion entre énantiomères est observée au cours du temps (étude en cours).

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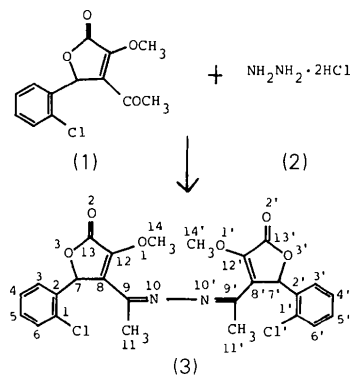
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# Structures of Two Isomeric Azines of 3-Acetyl-4-(2-chlorophenyl)-4-hydroxy-2-methoxycrotonic Acid Lactone, $C_{26}H_{22}Cl_2N_2O_6$

BY TOSHIMASA ISHIDA, MASATOSHI INOUE, KEIKO NASU AND TAKUSHI KURIHARA

*Osaka College of Pharmacy, 2-10-65 Kawai, Matsubara, Osaka 580, Japan*

**Abstract.** $M_r = 529.37$. Isomer (I): orthorhombic, *Pbcn*, $a = 20.132$ (4), $b = 6.162$ (1), $c = 19.954$ (3) Å, $V = 2475$ (2) Å$^3$ $Z = 4$, $D_m = 1.414$ (1), $D_x = 1.421$ Mg m$^{-3}$, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 2.76$ mm$^{-1}$, $T = 293$ K. Isomer (II): orthorhombic, *Pbca*, $a = 18.911$ (5), $b = 17.245$ (3), $c = 15.463$ (4) Å, $V = 5043$ (2) Å$^3$, $Z = 8$, $D_m = 1.400$ (1), $D_x = 1.394$ Mg m$^{-3}$, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 2.76$ mm$^{-1}$, $T = 293$ K. The block-diagonal least-squares refinements for isomers (I) and (II) converged to $R = 0.072$ and 0.075 for 1834 and 4291 unique reflections, respectively. The stereochemical difference for isomers (I) and (II) primarily involves the $=N-N=$ bond; the conformation of the $-C=N-N=C-$ bond is completely *trans* planar for isomer (I), and –anticlinal for isomer (II). This twisted bonding of isomer (II) is structurally more stable than the planar one of isomer (I), as shown by the results of energy calculations and thermal analyses.

**Introduction.** As is shown below, we recently reported that the reaction of 3-acetyl-4-(2-chlorophenyl)-4-hydroxy-2-methoxycrotonic acid lactone (1) with hydrazine dihydrochloride (2) gave two kinds of azines (3)

as red (isomer I) and yellow (isomer II) needle crystals (Kurihara, Sakamoto, Mori & Sakaki, 1978; Kurihara, Nasu, Inoue & Ishida, 1982). Their mass spectrometry and elemental analysis showed that both molecules have the same chemical formula. $^1$H NMR spectra in $(C^2H_3)_2SO$ solution clearly demonstrated that all the protons of both molecules are isochronous to each other. Therefore, it could be thought that a different stereochemistry should be assigned to both molecules.



In order to confirm these assignments, we determined the crystal structures of isomers (I) and (II).

**Experimental.** Co-crystallized from ethyl acetate and separated under a microscope as red needles (isomer I) and yellow prismatic needles (isomer II); $0.3 \times 0.6 \times 0.4$ mm (isomer I) and $0.4 \times 0.4 \times 0.6$ mm (isomer II); Rigaku automated four-circle diffractometer, graphite monochromator, cell parameters obtained by least-squares fit to $2\theta$ and $-2\theta$ values of 40 independent reflections; $D_m$ by flotation method in $CCl_4/C_6H_6$ mixture; intensity data of isomers (I) and (II) up to $2\theta = 130°$ collected by means of $\omega/2\theta$ scanning, scan speed $4°(2\theta)$ min$^{-1}$, scan width $(2\theta)$ $(1.2 + 0.15 \tan\theta)°$ at 40 kV and 150mA; four reference reflections monitored at 100-reflection intervals showed no intensity deterioration; Lorentz and polarization corrections applied, but absorption ignored; structures for both isomers solved by Patterson and Fourier methods, refined by block-diagonal least squares with anisotropic thermal parameters for all non-H atoms; H atoms located from a difference Fourier map, and included in the refinement; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 0.51$ for $F_o = 0.0$, $w = 1.0$ for $0.0 < F_o \leq 11.0$, and $w = 1.0/[1.0 + 0.115 (F_o - 11.0)]$ for $F_o > 11.0$ (isomer I), $w = 0.54$ for $F_o = 0.0$, $w = 1.0$ for $0.0 < F_o \leq 32.0$, $w = 1.0/[1.0 + 0.112 (F_o - 32.0)]$ for $F_o > 32.0$ (isomer II); final $R = 0.072$ (isomer I), $0.075$ (isomer II) and $R_w = 0.095$ (isomer I), $0.089$ (isomer II); maximum shift of parameters in the last cycle $1\sigma$, the residual fluctuations in the difference maps within the range of $-0.3$ to $0.3$ e Å$^{-3}$ for both isomers; atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974); $F(000) = 1096$ (isomer I),

2192 (isomer II); programs used: *MFPA* (Yasuoka & Tanaka, 1979), *SFFR* and *HBLS* V (Ashida, 1979), *MOLCON* (Fujii, 1979) and *ORTEP* (Johnson, 1976).

The energy calculations were carried out using the CNDO/2 method (Pople & Segal, 1966). The electronic energy was converged by iteration.

All the numerical calculations were carried out at the Computation Center of Osaka University, and at the Crystallographic Research Center, Institute for Protein Research, Osaka University.

**Discussion.** The final atomic parameters are in Table 1.* Stereoscopic views of isomers (I) and (II) are shown in Fig. 1, and their crystal structures in Fig. 2. Bond lengths and angles between the non-H atoms are given in Table 2.

The C—H bond lengths are from 0.89 [C(11)—H(11c)] to 1.08 Å [C(6)—H(6)] for isomer (I) and from 0.90 [C(11')—H(11'b)] to 1.11 Å [C(7')—H(7')] for (II). Although the bond lengths and angles of both isomers are almost in the ranges of accepted values (Kennard, 1968), the bonding parameters concerning the =N—N= bond appear to be significantly different from each other; isomer (I) has longer bond lengths for C(9)—N(10) and C(9')—N(10') and smaller bond angles for C(9)—N(10)—N(10') and C(9')—N(10')—N(10) than those of isomer (II), while the N(10)—N(10') bond lengths are almost identical in (I) and (II).

Selected torsion angles are listed in Table 3. In the ring moieties consisting of non-primed atoms of both isomers, the signs of the torsion angles around C(2)—C(7) and C(8)—C(9) are reversed, while the moieties consisting of primed atoms hold the same conformation in both.

The significant difference between isomers (I) and (II) primarily involves the N(10)—N(10') bond: the torsion angle C(9)—N(10)—N(10')—C(9') $(\varphi)$ is completely in the *trans* region for isomer (I) and in the $-$anticlinal region for (II). In order to clarify the relationship between $\varphi$ and its bonding energy, CNDO/2 energy calculations were carried out as a variable of $\varphi$ using the $H_2C=N—N=CH_2$ molecule: the molecular conformation $\varphi = -115°$ (corresponding to isomer II) was energetically more stable than $\varphi = 180°$ (corresponding to isomer I), and the difference of their energy values was 15 kJ mol$^{-1}$.

On the other hand, it was reported that the thermal-degradation process of isomer (II) occurs in the range 463–483 K, while that of isomer (I) is in the

## Table 1. *Fractional coordinates* (× 10⁴) *and isotropic temperature factors of the non-hydrogen atoms*

The $B$ values are the equivalent isotropic temperature factors calculated from the deposited anisotropic thermal parameters ($B_{ij}$) using the equation $B = (4/3) (B_{11}a^2 + B_{22}b^2 + B_{33}c^2)$.

| | $x$ | $y$ | $z$ | $B_{eq}(Å^2)$ |
|---|---|---|---|---|
| Isomer (I) | | | | |
| Cl | 4706 (1) | 10273 (3) | 3358 (1) | 7·0 (1) |
| C(1) | 4115 (2) | 8258 (7) | 3452 (2) | 4·2 (3) |
| C(2) | 3797 (2) | 7928 (6) | 4060 (2) | 3·4 (3) |
| C(3) | 3331 (2) | 6301 (7) | 4102 (2) | 4·6 (3) |
| C(4) | 3186 (2) | 4990 (8) | 3558 (3) | 5·8 (4) |
| C(5) | 3525 (3) | 5303 (9) | 2964 (2) | 6·0 (4) |
| C(6) | 3996 (2) | 6953 (8) | 2905 (2) | 5·4 (4) |
| C(7) | 3969 (2) | 9312 (6) | 4662 (2) | 3·6 (3) |
| C(8) | 3971 (2) | 8172 (6) | 5328 (2) | 3·5 (3) |
| C(9) | 4400 (2) | 6309 (6) | 5441 (2) | 3·4 (3) |
| N(10) | 4800 (1) | 5894 (6) | 4949 (1) | 4·0 (2) |
| C(11) | 4351 (2) | 5033 (7) | 6074 (2) | 4·5 (3) |
| C(12) | 3550 (2) | 9205 (6) | 5737 (2) | 3·5 (3) |
| C(13) | 3233 (2) | 11006 (6) | 5370 (2) | 3·8 (3) |
| C(14) | 2981 (2) | 9940 (8) | 6769 (2) | 5·5 (3) |
| O(1) | 3443 (1) | 8722 (5) | 6385 (1) | 4·9 (2) |
| O(2) | 2836 (1) | 12346 (5) | 5552 (2) | 5·4 (2) |
| O(3) | 3465 (1) | 11000 (4) | 4737 (1) | 4·4 (2) |
| | | | | |
| Isomer (II) | | | | |
| Cl | 11703 (1) | 32 (1) | 158 (1) | 5·6 (1) |
| C(1) | 11800 (2) | 282 (2) | −922 (2) | 3·8 (3) |
| C(2) | 11293 (2) | 73 (2) | −1531 (2) | 3·5 (3) |
| C(3) | 11402 (2) | 307 (2) | −2383 (2) | 4·6 (3) |
| C(4) | 11991 (2) | 742 (3) | −2609 (3) | 5·8 (3) |
| C(5) | 12479 (2) | 949 (3) | −1987 (3) | 5·4 (3) |
| C(6) | 12386 (2) | 723 (2) | −1140 (3) | 4·8 (3) |
| C(7) | 10641 (2) | −376 (2) | −1298 (2) | 3·4 (2) |
| C(8) | 9949 (2) | 5 (2) | −1551 (2) | 3·5 (2) |
| C(9) | 9759 (2) | 773 (2) | −1247 (2) | 3·7 (2) |
| N(10) | 10103 (1) | 1006 (1) | −582 (2) | 3·4 (1) |
| C(11) | 9212 (2) | 1243 (2) | −1701 (3) | 5·4 (3) |
| C(12) | 9588 (2) | −495 (2) | −2034 (2) | 4·0 (2) |
| C(13) | 10001 (2) | −1202 (2) | −2163 (3) | 4·8 (3) |
| C(14) | 8473 (2) | −961 (3) | −2484 (6) | 8·9 (4) |
| O(1) | 8931 (1) | −377 (2) | −2376 (2) | 6·1 (2) |
| O(2) | 9865 (2) | −1792 (2) | −2543 (3) | 7·1 (2) |
| O(3) | 10630 (1) | −1108 (1) | −1759 (2) | 4·5 (1) |
| Cl' | 8013 (1) | 2036 (1) | 974 (1) | 5·5 (1) |
| C(1') | 8163 (2) | 2506 (2) | −4 (2) | 3·8 (2) |
| C(2') | 8767 (1) | 2952 (2) | −128 (2) | 3·3 (2) |
| C(3') | 8835 (2) | 3327 (2) | −917 (2) | 4·5 (3) |
| C(4') | 8332 (2) | 3246 (3) | −1563 (2) | 5·0 (3) |
| C(5') | 7758 (2) | 2780 (3) | −1429 (3) | 5·0 (3) |
| C(6') | 7664 (2) | 2410 (2) | −650 (2) | 4·4 (2) |
| C(7') | 9342 (2) | 3028 (2) | 550 (2) | 3·5 (2) |
| C(8') | 10090 (2) | 2956 (2) | 207 (2) | 3·3 (2) |
| C(9') | 10324 (1) | 2260 (2) | −239 (2) | 3·2 (2) |
| N(10') | 9892 (1) | 1694 (2) | −214 (2) | 3·6 (2) |
| C(11') | 11009 (2) | 2242 (2) | −739 (3) | 4·5 (3) |
| C(12') | 10433 (2) | 3609 (2) | 397 (2) | 3·8 (2) |
| C(13') | 9961 (2) | 4156 (2) | 837 (2) | 4·2 (2) |
| C(14') | 11348 (2) | 4479 (3) | −28 (4) | 6·4 (3) |
| O(1') | 11132 (1) | 3744 (1) | 294 (2) | 4·6 (1) |
| O(2') | 10073 (1) | 4795 (2) | 1114 (2) | 5·4 (2) |
| O(3') | 9317 (1) | 3811 (1) | 908 (2) | 4·2 (2) |

## Table 2. *Bond lengths* (Å) *and angles* (°) *involving non-hydrogen atoms*

Those involving the primed atoms of isomer (II) are listed in square brackets.

| | Isomer (I) | Isomer (II) |
|---|---|---|
| Cl–C(1) | 1·731 (4) | 1·734 (4) [1·739 (4)] |
| C(1)–C(2) | 1·387 (5) | 1·392 (5) [1·390 (5)] |
| C(1)–C(6) | 1·377 (7) | 1·386 (5) [1·385 (5)] |
| C(2)–C(3) | 1·375 (6) | 1·393 (5) [1·387 (5)] |
| C(2)–C(7) | 1·514 (5) | 1·500 (4) [1·514 (4)] |
| C(3)–C(4) | 1·385 (7) | 1·387 (6) [1·387 (6)] |
| C(4)–C(5) | 1·381 (7) | 1·381 (7) [1·367 (6)] |
| C(5)–C(6) | 1·396 (7) | 1·378 (6) [1·374 (6)] |
| C(7)–C(8) | 1·504 (5) | 1·516 (4) [1·515 (4)] |
| C(7)–O(3) | 1·462 (5) | 1·450 (4) [1·461 (4)] |
| C(8)–C(9) | 1·453 (5) | 1·450 (5) [1·454 (4)] |
| C(8) C(12) | 1·337 (5) | 1·329 (5) [1·333 (4)] |
| C(9)–N(10) | 1·296 (5) | 1·282 (4) [1·274 (4)] |

## Table 2 (cont.)

| | Isomer (I) | Isomer (II) |
|---|---|---|
| C(9)–C(11) | 1·490 (6) | 1·490 (5) [1·509 (5)] |
| C(12)–C(13) | 1·475 (5) | 1·463 (5) [1·466 (5)] |
| C(12)–O(1) | 1·345 (5) | 1·366 (5) [1·352 (4)] |
| C(13)–O(2) | 1·206 (5) | 1·203 (6) [1·202 (5)] |
| C(13)–O(3) | 1·346 (5) | 1·352 (5) [1·360 (4)] |
| C(14)–O(1) | 1·420 (6) | 1·337 (9) [1·421 (7)] |
| N(10)–N(10') | 1·379 (5) | 1·376 (4) |
| | | |
| Cl–C(1)–C(2) | 121·2 (3) | 121·0 (3) [120·8 (3)] |
| Cl–C(1)–C(6) | 116·9 (3) | 117·1 (3) [117·4 (3)] |
| C(2)–C(1)–C(6) | 121·9 (4) | 121·9 (3) [121·8 (3)] |
| C(1)–C(2)–C(3) | 118·4 (4) | 117·6 (3) [117·0 (3)] |
| C(1)–C(2)–C(7) | 120·4 (3) | 122·5 (3) [122·9 (3)] |
| C(3)–C(2)–C(7) | 121·2 (3) | 120·0 (3) [120·1 (3)] |
| C(2)–C(3)–C(4) | 121·4 (4) | 120·9 (4) [121·6 (9)] |
| C(3)–C(4)–C(5) | 119·2 (5) | 120·1 (4) [119·6 (4)] |
| C(4)–C(5)–C(6) | 120·7 (5) | 120·2 (4) [120·6 (4)] |
| C(1)–C(6)–C(5) | 118·4 (5) | 119·3 (4) [119·2 (3)] |
| C(2)–C(7)–C(8) | 116·1 (3) | 115·0 (3) [114·9 (3)] |
| C(2)–C(7)–O(3) | 108·8 (3) | 110·0 (3) [108·7 (3)] |
| C(8)–C(7)–O(3) | 104·1 (3) | 103·7 (2) [103·8 (2)] |
| C(7)–C(8)–C(9) | 120·5 (3) | 121·7 (3) [121·2 (3)] |
| C(7)–C(8)–C(12) | 108·3 (3) | 107·9 (3) [108·0 (3)] |
| C(9)–C(8)–C(12) | 131·2 (3) | 130·3 (3) [130·8 (3)] |
| C(8)–C(9)–N(10) | 114·1 (3) | 114·9 (9) [115·0 (3)] |
| C(8)–C(9)–C(11) | 120·6 (3) | 121·1 (3) [121·5 (3)] |
| N(10)–C(9)–C(11) | 125·3 (3) | 124·1 (3) [123·4 (3)] |
| C(9)–N(10)–N(10') | 114·1 (3) | 117·0 (3) [117·5 (3)] |
| C(8)–C(12)–C(13) | 109·3 (3) | 110·1 (3) [110·4 (3)] |
| C(8)–C(12)–O(1) | 125·6 (3) | 126·0 (3) [126·6 (3)] |
| C(13)–C(12)–O(1) | 125·1 (3) | 123·9 (3) [122·7 (3)] |
| C(12)–C(13)–O(2) | 130·7 (4) | 131·1 (4) [130·5 (3)] |
| C(12)–C(13)–O(3) | 108·3 (3) | 107·8 (3) [107·6 (3)] |
| O(2)–C(13)–O(3) | 121·0 (4) | 121·1 (4) [121·9 (3)] |
| C(7)–O(3)–C(13) | 109·8 (3) | 110·2 (3) [110·2 (3)] |
| C(12)–O(1)–C(14) | 120·4 (3) | 121·7 (5) [118·3 (3)] |

## Table 3. *Selected torsion angles* (°)

| | Isomer (I) | Isomer (II) |
|---|---|---|
| C(1)–C(2)–C(7)–C(8) | −143·0 (4) | 124·3 (3) |
| C(3)–C(2)–C(7)–C(8) | 36·0 (5) | −54·8 (4) |
| C(7)–C(8)–C(9)–N(10) | 5·6 (5) | −18·7 (4) |
| C(7)–C(8)–C(9)–C(11) | −173·5 (3) | 106·2 (3) |
| C(8)–C(12)–O(1)–C(14) | −179·9 (4) | −147·7 (6) |
| C(8)–C(9)–N(10)–N(10') | −179·0 (3) | −173·3 (3) |
| C(9)–N(10)–N(10')–C(9') | 180·0 (3) | −114·8 (3) |
| C(8')–C(9')–N(10')–N(10) | 179·0 (3) | −176·3 (3) |
| C(7')–C(8')–C(9')–N(10') | −5·6 (5) | −9·0 (4) |
| C(7')–C(8')–C(9')–C(11') | 173·5 (3) | 167·3 (3) |
| C(8')–C(12')–O(1')–C(14') | 179·9 (4) | 139·1 (4) |
| C(1')–C(2')–C(7')–C(8') | 143·0 (4) | 135·3 (3) |
| C(3')–C(2')–C(7')–C(8') | −36·0 (5) | −43·9 (4) |



(a)



(b)

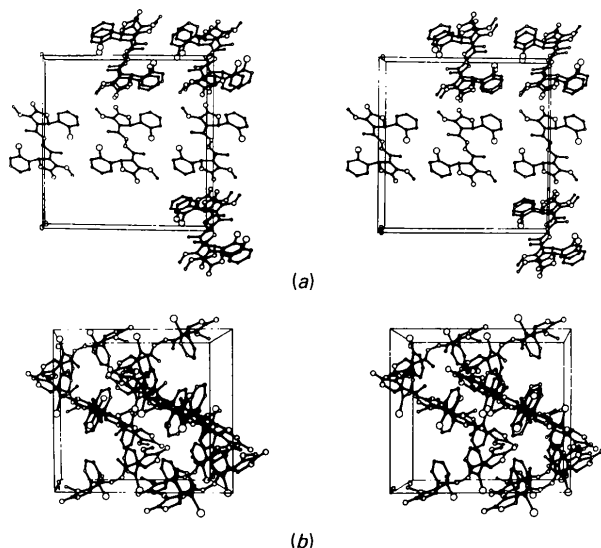Fig. 1. Stereoscopic views of (a) isomer (I) and (b) isomer (II).

Fig. 2. Stereoscopic views of the crystal packing of (*a*) isomer (I) and (*b*) isomer (II).

range 435–483 K (Kurihara, Nasu, Inoue & Ishida, 1982). This observation is comparable with the result of the energy calculation that the twisted conformation

around the =N—N= bond is more stable than the planar one.

There are no intermolecular contacts closer than the sum of the appropriate van der Waals radii for both isomers in their crystal structures.

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# Structure of 2-(Methylsulphinyl)-1,2-diphenylethyl Acetate, C17H18O3S

BY M. A. HOYOS-GUERRERO

*Departamento de Geología y Geoquímica, Universidad Autónoma, Madrid*

AND S. MARTÍNEZ-CARRERA AND S. GARCÍA-BLANCO

*Departamento de Rayos X, Instituto 'Rocasolano', CSIC, Serrano 119, Madrid-6, Spain*

**Abstract.** $M_r = 302.4$, monoclinic, space group $P2_1/c$, $a = 8.878$ (1), $b = 21.703$ (4), $c = 8.408$ (1) Å, $\beta = 93.47$ (1)°, $V = 1617.1$ (4) Å³, $Z = 4$, $D_x = 1.242$ Mg m⁻³, $\lambda$(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.197$ mm⁻¹, $F(000) = 640$. Final $R = 0.082$ for 2496 observed reflections. The three chiral centres, C(1),C(2),S, are *S,R,S* (or *R,S,R*) respectively.

**Introduction.** The present study is a continuation of the investigations we are carrying out into the structure of sulphoxides.

**Experimental.** Crystals prepared by Dr Rodriguez-Ramos; colourless crystal, $0.2 \times 0.3 \times 0.2$ mm, moun-ted at random on a Philips PW1100 four-circle diffractometer, was used for both cell-parameter determinations and intensity measurements; cell dimensions obtained by a least-squares fit of settings for 54 reflections ($\pm hkl$), intensities recorded in $\omega/2\theta$ scan mode with $\theta$ ranging between 2 and 35°, graphite-monochromated Mo $K\alpha$ radiation, 4729 unique reflections, 2496 considered observed with $I > 2\sigma(I)$ and retained for use in structure analysis; intensities and $\omega$ angles of two reference reflections measured periodically every 90 min showed no appreciable variation during course of data collection; Lorentz and polarization but no absorption correction; direct-methods program *MULTAN* 78 (Main, Woolfson,