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earlier publications (Michel, Michel-Dewez, Roughton, Springer & Hoogsteen, 1989; Michel, Drouin, Michel-Dewez, Roughton & Deslongchamps, 1991; Drouin, Lamothe & Michel, 1992). More recently, a new series of 14-membered ring compounds have been synthesized using intramolecular Michael addition (Stork, Winkler & Saccomano, 1983), which leads to completely controlled diene geometries. Indeed, compounds (I) and (II) were obtained in low (30%) and very high (90%) yields, respectively (Crevisy, Couturier, Dugave, Dory & Deslongchamps, 1995), via intramolecular Michael addition involving the β -keto ester and conjugated olefinic ketone moieties.

Acta Cryst. (1996). **C52**, 2084–2087

Two 14-Membered Macrocycles with *trans-trans* and *trans-cis* Dienes. Trimethyl (2*E*,4*E*)-*cis*- and Trimethyl (2*Z*,4*E*)-*trans*-11,15-Dioxobicyclo[12.3.0]heptadeca-2,4-diene-7,7,14-tricarboxylate

MARC DROUIN,^a MICHEL COUTURIER,^b CHRISTOPHE CREVISY,^b YVES L. DORY^b AND PIERRE DESLONGCHAMPS^b

^aLaboratoire de Diffraction des Rayons-X, Département de Chimie, Université de Sherbrooke, Sherbrooke, Québec, Canada J1K 2R1, and ^bLaboratoire de Synthèse Organique, Département de Chimie, Université de Sherbrooke, Sherbrooke, Québec, Canada J1K 2R1. E-mail: mdrouin@structure.chimie.usherb.ca

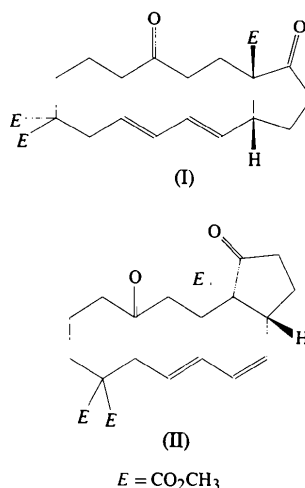
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Abstract

Two new isomeric 14-membered ring compounds, C₂₃H₃₀O₈, were synthesized based on Michael addition macrocyclization, which leads to completely controlled diene geometries, *trans-trans* and *trans-cis*. The crystal structures show the respective *trans-trans* and *trans-cis* diene geometries and their corresponding *cis* and *trans* ring junctions.

Comment

The transannular Diels–Alder cycloaddition represents a powerful approach towards the syntheses of several classes of natural products such as terpenes, triterpenes and steroids (Deslongchamps, 1991). This strategy for the construction of polycyclic macromolecules (Lamothe, Ndibwami & Deslongchamps, 1988; Marinier & Deslongchamps, 1988) involves the traditionally difficult synthesis of large rings. The conformational properties of such macrocycles change with each olefinic geometry combination and substituents as shown in



We present here the results of crystallographic investigations of compounds (I) and (II), undertaken to determine the ring-junction and diene geometries of these compounds as well as their exact conformations. The results clearly show that (I) and (II) have *trans-trans* and *trans-cis* olefin geometries, respectively, and their corresponding *cis* and *trans* ring junctions. Both macrocycles have carbonyl groups at C11 and at C17. Two methyl esters are attached at C1 and one at C8 in both molecules. The torsion-angle values for the olefin moieties show large deviations from ideally unstrained systems in (I). Indeed, C2—C3—C4—C5 and C4—C5—C6—C7 have respective values of $-168.3(4)$ and $-167.9(4)^\circ$ for (I) and $-178.9(10)$ and $-3.7(4)^\circ$ for (II). This shows that the olefinic system in (I) is severely strained compared to (II) and could explain the major difference in the yields of the two compounds. The conjugation of the diene moiety is partially broken in both molecules as shown by the C3—C4—C5—C6 torsion-angle values of $161.7(4)$ and $168.2(11)^\circ$ for (I) and (II), respectively. The torsion-angle values of the 14-membered ring are similar in both compounds, which show great similarities in global conformation. Puckering analysis (Cremer & Pople, 1975) shows that the five-membered ring adopts a conformation halfway between envelope (*E*) and twist (*T*) (*C*₂ half chair with C17 on the twist axis).

In (I), C7 is 0.156(8) Å above the plane defined by atoms C8, C16, C17 and O2, whereas C15 is 0.468(10) Å below. In (II), C7 is 0.423(17) Å above the same plane and C15 is 0.138(19) Å below. Torsion-angle values of the five-membered rings are listed in Table 2. No abnormally short contacts were observed.

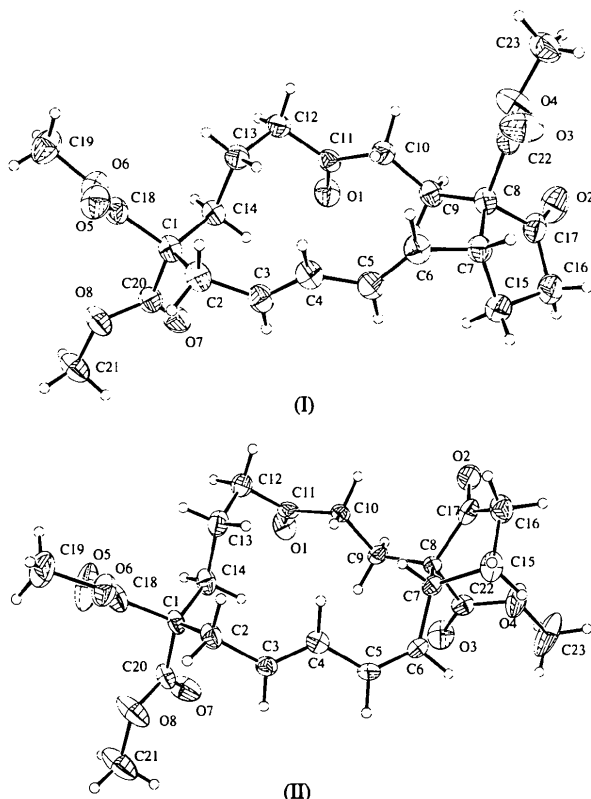


Fig. 1. ORTEP (Johnson, 1976) perspective views showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level; H atoms are drawn as small circles of arbitrary radii.

Experimental

Compounds (I) and (II) were obtained from methyl 2-[(1*E*,3*E*)-6,6-bis(methoxycarbonyl)-10-oxododeca-1,3,11-trienyl]-5-oxocyclopentane-1-carboxylate and methyl 2-[(1*Z*,3*E*)-6,6-bis(methoxycarbonyl)-10-oxododeca-1,3,11-trienyl]-5-oxocyclopentane-1-carboxylate, respectively, *via* intramolecular Michael addition involving the β -keto ester and conjugated olefinic ketone moieties. Detailed syntheses of the precursors are described in Crevisy, Couturier, Dugave, Dory & Deslongchamps (1995).

Compound (I)

Crystal data

$C_{23}H_{30}O_8$
 $M_r = 434.47$
 Monoclinic
 $P2_1$

Cu $K\alpha$ radiation
 $\lambda = 1.54184$ Å
 Cell parameters from 24 reflections

$a = 9.8355(4)$ Å
 $b = 8.2688(4)$ Å
 $c = 14.7126(8)$ Å
 $\beta = 107.567(4)^\circ$
 $V = 1140.74(10)$ Å³
 $Z = 2$
 $D_x = 1.265$ Mg m⁻³
 D_m not measured

$\theta = 20\text{--}25^\circ$
 $\mu = 0.792$ mm⁻¹
 $T = 293(2)$ K
 Irregular
 $0.30 \times 0.25 \times 0.25$ mm
 Colorless

Data collection

Enraf-Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none
 2499 measured reflections
 2397 independent reflections
 1978 observed reflections
 $[I > 2\sigma(I)]$

$R_{int} = 0.010$
 $\theta_{max} = 71.66^\circ$
 $h = -12 \rightarrow 11$
 $k = 0 \rightarrow 10$
 $l = 0 \rightarrow 18$
 2 standard reflections
 frequency: 60 min
 intensity decay: <1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0413$
 $wR(F^2) = 0.1055$
 $S = 1.101$
 2397 reflections
 283 parameters
 H-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.0268P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = -0.001$

$\Delta\rho_{max} = 0.176$ e Å⁻³
 $\Delta\rho_{min} = -0.143$ e Å⁻³
 Extinction correction:
 SHELXL93 (Sheldrick, 1993)
 Extinction coefficient:
 0.0107(12)
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (I)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C1	0.3114 (3)	0.8898 (6)	0.2547 (2)	0.0534 (7)
C2	0.4131 (3)	0.7638 (6)	0.2289 (2)	0.0618 (8)
C3	0.3459 (3)	0.6762 (6)	0.1375 (2)	0.0609 (8)
C4	0.3935 (3)	0.6897 (6)	0.0626 (2)	0.0617 (8)
C5	0.3211 (3)	0.6339 (6)	-0.0323 (2)	0.0609 (8)
C6	0.3525 (3)	0.6855 (6)	-0.1083 (2)	0.0591 (8)
C7	0.2703 (3)	0.6605 (6)	-0.2106 (2)	0.0585 (8)
C8	0.1752 (3)	0.8072 (6)	-0.2569 (2)	0.0547 (7)
C9	0.0968 (3)	0.8912 (6)	-0.1933 (2)	0.0537 (7)
C10	0.1814 (3)	1.0182 (5)	-0.1238 (2)	0.0511 (7)
C11	0.1172 (3)	1.0636 (5)	-0.0465 (2)	0.0493 (6)
C12	0.2014 (3)	1.1768 (6)	0.0305 (2)	0.0561 (7)
C13	0.2990 (3)	1.0877 (6)	0.1171 (2)	0.0548 (7)
C14	0.2177 (3)	0.9816 (6)	0.1673 (2)	0.0533 (7)
C15	0.1687 (5)	0.5156 (7)	-0.2327 (3)	0.0779 (11)
C16	0.0768 (6)	0.5474 (7)	-0.3341 (3)	0.0920 (14)
C17	0.0597 (4)	0.7272 (6)	-0.3408 (2)	0.0675 (10)
C18	0.4020 (3)	1.0058 (6)	0.3290 (2)	0.0575 (8)
C19	0.3946 (5)	1.2442 (7)	0.4164 (3)	0.0970 (15)
C20	0.2150 (3)	0.7948 (6)	0.2992 (2)	0.0557 (7)
C21	0.2002 (4)	0.6741 (7)	0.4426 (3)	0.0815 (11)
C22	0.2560 (3)	0.9263 (6)	-0.2990 (2)	0.0558 (7)
C23	0.2320 (5)	1.1547 (8)	-0.3973 (3)	0.0921 (13)
O1	0.0029 (2)	1.0100 (5)	-0.04507 (15)	0.0679 (6)
O2	-0.0312 (3)	0.7987 (6)	-0.4000 (2)	0.0983 (10)
O3	0.3772 (3)	0.9118 (5)	-0.2975 (2)	0.0971 (10)

O4	0.1757 (3)	1.0477 (5)	-0.3407 (2)	0.0871 (9)
O5	0.5292 (2)	1.0000	0.3618 (2)	0.0766 (7)
O6	0.3214 (2)	1.1192 (5)	0.3505 (2)	0.0731 (7)
O7	0.0958 (2)	0.7512 (5)	0.2581 (2)	0.0749 (7)
O8	0.2791 (2)	0.7614 (5)	0.3905 (2)	0.0741 (7)

$\omega/2\theta$ scans	$h = -13 \rightarrow 13$
Absorption correction:	$k = 0 \rightarrow 15$
none	$l = 0 \rightarrow 10$
3587 measured reflections	2 standard reflections
2336 independent reflections	frequency: 60 min
1239 observed reflections	intensity decay: <1%
[$I > 1.8\sigma(I)$]	

Table 2. Selected geometric parameters (\AA , $^\circ$) for (I)

C1—C20	1.523 (4)	C11—O1	1.214 (3)
C1—C18	1.523 (4)	C11—C12	1.509 (4)
C1—C14	1.538 (4)	C12—C13	1.533 (4)
C1—C2	1.568 (5)	C13—C14	1.519 (4)
C2—C3	1.494 (5)	C15—C16	1.516 (6)
C3—C4	1.325 (4)	C16—C17	1.496 (6)
C4—C5	1.439 (5)	C17—O2	1.199 (5)
C5—C6	1.317 (4)	C18—O5	1.198 (4)
C6—C7	1.492 (4)	C18—O6	1.327 (4)
C7—C15	1.530 (5)	C19—O6	1.451 (5)
C7—C8	1.557 (5)	C20—O7	1.201 (3)
C8—C22	1.510 (4)	C20—O8	1.329 (3)
C8—C9	1.546 (4)	C21—O8	1.439 (4)
C8—C17	1.551 (4)	C22—O3	1.192 (4)
C9—C10	1.525 (4)	C22—O4	1.310 (4)
C10—C11	1.507 (4)	C23—O4	1.435 (5)
C20—C1—C18	108.8 (2)	O1—C11—C12	121.0 (3)
C20—C1—C14	108.7 (2)	C10—C11—C12	117.4 (2)
C18—C1—C14	111.2 (3)	C11—C12—C13	112.9 (3)
C20—C1—C2	106.7 (3)	C14—C13—C12	113.1 (2)
C18—C1—C2	108.4 (2)	C13—C14—C1	114.9 (2)
C14—C1—C2	112.9 (2)	C16—C15—C7	103.3 (3)
C3—C2—C1	113.5 (2)	C17—C16—C15	105.0 (3)
C4—C3—C2	122.4 (3)	O2—C17—C16	125.6 (4)
C3—C4—C5	125.8 (3)	O2—C17—C8	125.0 (4)
C6—C5—C4	123.1 (3)	C16—C17—C8	109.3 (3)
C5—C6—C7	128.1 (3)	O5—C18—O6	124.0 (3)
C6—C7—C15	116.3 (3)	O5—C18—C1	125.3 (3)
C6—C7—C8	113.9 (3)	O6—C18—C1	110.7 (2)
C15—C7—C8	104.5 (3)	O7—C20—O8	122.7 (3)
C22—C8—C9	112.0 (3)	O7—C20—C1	125.3 (3)
C22—C8—C17	107.6 (2)	O8—C20—C1	111.9 (2)
C9—C8—C17	107.1 (2)	O3—C22—O4	122.9 (3)
C22—C8—C7	111.5 (2)	O3—C22—C8	125.0 (3)
C9—C8—C7	115.4 (2)	O4—C22—C8	112.0 (3)
C17—C8—C7	102.4 (3)	C22—O4—C23	117.5 (3)
C10—C9—C8	116.4 (2)	C18—O6—C19	116.8 (3)
C11—C10—C9	114.2 (2)	C20—O8—C21	118.3 (3)
O1—C11—C10	121.5 (3)		
C15—C7—C8—C17	-28.5 (3)	C15—C16—C17—O2	-160.9 (4)
C8—C7—C15—C16	39.8 (3)	C15—C16—C17—C8	17.2 (4)
C7—C15—C16—C17	-35.0 (4)	C7—C8—C17—C16	7.1 (4)

Refinement

Refinement on F	$\Delta\rho_{\max} = 0.26 \text{ e \AA}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.072$	$\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$
$wR(F^2) = 0.077$	Extinction correction: Larson (1970)
$S = 1.32$	Extinction coefficient: $0.5 (1) \times 10^{-6}$
1239 reflections	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV, Table 2.2B)
281 parameters	
H-atom parameters not refined	
$w = 1/[\sigma^2(F) + 0.0010F^2]$	
$(\Delta/\sigma)_{\max} = 0.001$	

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (II)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
C1	0.3941 (7)	0.3554 (7)	0.8640 (9)	0.042 (7)
C2	0.3385 (8)	0.4217 (7)	0.9394 (10)	0.051 (8)
C3	0.2586 (7)	0.4682 (7)	0.8608 (9)	0.042 (7)
C4	0.1628 (7)	0.4532 (7)	0.8586 (9)	0.042 (7)
C5	0.0871 (8)	0.4981 (6)	0.7789 (10)	0.046 (7)
C6	-0.0066 (8)	0.4764 (7)	0.7577 (11)	0.047 (7)
C7	-0.0572 (7)	0.3987 (6)	0.8036 (9)	0.036 (7)
C8	-0.0801 (7)	0.3297 (6)	0.7022 (8)	0.033 (6)
C9	0.0065 (7)	0.2740 (6)	0.6729 (9)	0.042 (7)
C10	0.0597 (7)	0.2257 (6)	0.7832 (9)	0.037 (6)
C11	0.1484 (7)	0.1781 (6)	0.7471 (10)	0.041 (7)
C12	0.2236 (8)	0.1534 (7)	0.8531 (11)	0.055 (8)
C13	0.2819 (7)	0.2299 (7)	0.9096 (10)	0.057 (8)
C14	0.3270 (7)	0.2830 (7)	0.8103 (9)	0.052 (8)
C15	-0.1561 (8)	0.4158 (7)	0.8599 (9)	0.050 (7)
C16	-0.2031 (8)	0.3277 (8)	0.8561 (11)	0.065 (8)
C17	-0.1634 (7)	0.2778 (7)	0.7536 (10)	0.051 (7)
C18	0.4846 (8)	0.3152 (8)	0.9434 (11)	0.065 (8)
C19	0.5866 (10)	0.3045 (11)	1.1238 (13)	0.118 (12)
C20	0.4374 (7)	0.4032 (8)	0.7584 (10)	0.053 (8)
C21	0.5529 (10)	0.5086 (10)	0.7142 (13)	0.110 (12)
C22	-0.1256 (7)	0.3694 (7)	0.5778 (9)	0.046 (7)
C23	-0.2707 (11)	0.4261 (10)	0.4678 (12)	0.123 (12)
O1	0.1579 (5)	0.1598 (5)	0.6396 (7)	0.064 (5)
O2	-0.1884 (5)	0.2071 (4)	0.7205 (7)	0.067 (5)
O3	-0.0809 (6)	0.3802 (6)	0.4885 (6)	0.077 (6)
O4	-0.2192 (5)	0.3894 (6)	0.5823 (7)	0.079 (6)
O5	0.5355 (6)	0.2644 (7)	0.8969 (8)	0.117 (8)
O6	0.4954 (5)	0.3417 (5)	1.0583 (7)	0.074 (6)
O7	0.4123 (6)	0.3960 (5)	0.6474 (7)	0.080 (6)
O8	0.5048 (6)	0.4591 (6)	0.8033 (7)	0.090 (7)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (II)

C1—C2	1.554 (15)	C11—C12	1.499 (14)
C1—C14	1.530 (14)	C11—O1	1.202 (13)
C1—C18	1.557 (14)	C12—C13	1.527 (16)
C1—C20	1.521 (15)	C13—C14	1.524 (16)
C2—C3	1.494 (14)	C15—C16	1.519 (16)
C3—C4	1.323 (14)	C16—C17	1.490 (16)
C4—C5	1.450 (14)	C17—O2	1.200 (12)
C5—C6	1.318 (15)	C18—O5	1.194 (15)
C6—C7	1.503 (15)	C18—O6	1.290 (13)
C7—C8	1.540 (13)	C19—O6	1.482 (14)

Compound (II)

Crystal data

C₂₃H₃₀O₈ $M_r = 434.48$

Monoclinic

 $P2_1/c$ $a = 13.6059 (6) \text{ \AA}$ $b = 15.6454 (6) \text{ \AA}$ $c = 10.6840 (5) \text{ \AA}$ $\beta = 95.735 (4)^\circ$ $V = 2262.71 (2) \text{ \AA}^3$ $Z = 4$ $D_x = 1.275 \text{ Mg m}^{-3}$ D_m not measured

Data collection

Enraf-Nonius CAD-4 diffractometer

Cu $K\alpha$ radiation $\lambda = 1.54184 \text{ \AA}$

Cell parameters from 24

reflections

 $\theta = 20.00\text{--}25.00^\circ$ $\mu = 0.76 \text{ mm}^{-1}$ $T = 293 \text{ K}$

Long needle

 $0.35 \times 0.05 \times 0.03 \text{ mm}$

Colourless

 $R_{\text{int}} = 0.029$ $\theta_{\text{max}} = 50.00^\circ$

C7—C15	1.551 (15)	C20—O7	1.205 (13)
C8—C9	1.523 (14)	C20—O8	1.323 (13)
C8—C17	1.539 (15)	C21—O8	1.435 (16)
C8—C22	1.540 (13)	C22—O3	1.194 (13)
C9—C10	1.521 (13)	C22—O4	1.316 (13)
C10—C11	1.502 (14)	C23—O4	1.465 (13)
C2—C1—C14	112.7 (8)	C10—C11—O1	121.9 (9)
C2—C1—C18	112.8 (8)	C12—C11—O1	122.1 (10)
C2—C1—C20	107.5 (9)	C11—C12—C13	112.6 (9)
C14—C1—C18	108.4 (9)	C12—C13—C14	112.4 (9)
C14—C1—C20	110.2 (8)	C1—C14—C13	114.1 (8)
C18—C1—C20	105.0 (8)	C7—C15—C16	102.3 (8)
C1—C2—C3	113.4 (8)	C15—C16—C17	108.2 (10)
C2—C3—C4	125.7 (10)	C8—C17—C16	108.7 (9)
C3—C4—C5	124.4 (10)	C8—C17—O2	125.3 (10)
C4—C5—C6	126.5 (10)	C16—C17—O2	125.9 (11)
C5—C6—C7	128.2 (9)	C1—C18—O5	120.2 (10)
C6—C7—C8	113.9 (8)	C1—C18—O6	113.4 (10)
C8—C7—C15	105.7 (8)	O5—C18—O6	126.4 (10)
C7—C8—C9	116.0 (7)	C1—C20—O7	126.3 (9)
C7—C8—C17	102.9 (8)	C1—C20—O8	111.2 (9)
C7—C8—C22	111.1 (8)	O7—C20—O8	122.4 (10)
C9—C8—C17	112.6 (8)	C8—C22—O3	123.6 (9)
C9—C8—C22	107.8 (8)	C8—C22—O4	111.8 (9)
C17—C8—C22	105.9 (7)	O3—C22—O4	124.6 (9)
C8—C9—C10	116.3 (8)	C22—O4—C23	116.5 (9)
C9—C10—C11	112.3 (8)	C18—O6—C19	109.5 (10)
C10—C11—C12	116.0 (9)	C20—O8—C21	117.5 (9)
C15—C7—C8—C17	−30.8 (6)	C7—C15—C16—C17	−26.0 (6)
C8—C7—C15—C16	35.3 (6)	C15—C16—C17—C8	7.4 (5)
C7—C8—C17—C16	14.6 (6)	C15—C16—C17—O2	−176.1 (14)
C7—C8—C17—O2	−162.0 (12)		

For both compounds, data collection: *DATCOL NRCCAD* (Le Page, White & Gabe, 1986); cell refinement: *TRUANG NRCCAD*; data reduction: *DATRD2 NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989); program(s) used to solve structures: *SOLVER NRCVAX*. Program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993) for (I); *LSTSQ NRCVAX* for (II). For both compounds, molecular graphics: *ORTEPII* (Johnson, 1976) *NRCVAX*. Software used to prepare material for publication: *SHELXL93* for (I); *TABLES NRCVAX* for (II).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1020). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Glycyl-L-Leucyl-L-Tyrosine Dihydrate 2-Propanol Solvate

BJØRN DALHUS AND CARL HENRIK GÖRBITZ

Department of Chemistry, University of Oslo, PO Box 1033, Blindern, N-0315 Oslo, Norway. E-mail: bjordna@kjemi.uio.no

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Abstract

The asymmetric unit ($C_{17}H_{25}N_3O_5 \cdot C_3H_8O \cdot 2H_2O$) consists of two crystallographically independent peptide molecules, *A* and *B*, with different conformations, χ_2^1 being *trans* and *gauche*[−] for the Leu residues in molecules *A* and *B*, respectively. The backbone conformation of both peptide molecules resembles that of the β -pleated sheet arrangement found in proteins. Comparison with two other structures containing the tripeptide Gly-L-Leu-L-Tyr reveals almost identical molecular conformations, and in one instance also a common packing pattern.

Comment

As part of a project dealing with the X-ray structures of tripeptides with non-Gly residues in the mid-position, we have crystallized Gly-L-Leu-L-Tyr as the dihydrate 2-propanol solvate (I). The structures of Gly-L-Leu-L-Tyr 2.5-hydrate dimethyl sulfoxide (DMSO) solvate (Wu, Tinant, Declercq & Van Meerssche, 1987; Subramanian & Parthasarathy, 1987) and the copper complex of the same peptide (Franks & van der Helm, 1970) have already been described.

