$S = 0.4$
1597 reflections
174 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F) + 0.063F^2]$

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)

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

	x	y	z	U_{eq}
C1	0.7928 (2)	0.0130 (2)	0.1325 (1)	0.035 (1)
C2	0.5853 (2)	0.0134 (2)	0.1381 (1)	0.034 (1)
C3	0.5157 (3)	−0.1262 (2)	0.1848 (1)	0.049 (1)
O4	0.8580 (2)	0.1457 (2)	0.1164 (1)	0.049 (1)
O5	0.8879 (2)	−0.0910 (2)	0.1418 (2)	0.055 (1)
C6	0.5031 (2)	0.0473 (2)	0.0325 (1)	0.033 (1)
C7	0.3007 (2)	0.0820 (2)	0.0400 (2)	0.035 (1)
C8	0.2028 (3)	0.0228 (3)	−0.0513 (2)	0.053 (1)
C9	0.3549 (3)	−0.0256 (2)	−0.1219 (2)	0.046 (1)
C10	0.5101 (3)	−0.0700 (2)	−0.0502 (1)	0.040 (1)
C11	0.6906 (3)	−0.0855 (3)	−0.1029 (2)	0.062 (1)
O12	0.2310 (2)	0.1514 (2)	0.1093 (2)	0.048 (1)

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

C2—C1	1.524 (2)	O4—C1	1.338 (2)
O5—C1	1.197 (2)	C3—C2	1.523 (2)
C6—C2	1.552 (2)	C7—C6	1.523 (2)
C10—C6	1.543 (2)	C8—C7	1.507 (3)
O12—C7	1.230 (2)	C9—C8	1.522 (3)
C10—C9	1.537 (3)	C11—C10	1.503 (3)
O4—C1—C2	111.2 (2)	O5—C1—C2	125.4 (2)
O5—C1—O4	123.3 (1)	C3—C2—C1	110.6 (2)
C6—C2—C1	110.2 (1)	C6—C2—C3	114.0 (1)
C7—C6—C2	111.3 (2)	C10—C6—C2	118.8 (1)
C10—C6—C7	103.2 (1)	C8—C7—C6	109.6 (2)
O12—C7—C6	124.4 (2)	O12—C7—C8	126.0 (2)
C9—C8—C7	104.4 (1)	C10—C9—C8	104.1 (2)
C9—C10—C6	102.9 (1)	C11—C10—C6	115.1 (2)
C11—C10—C9	113.1 (2)		
O4—C1—C2—C3	−164	C10—C6—C7—O12	166
O4—C1—C2—C6	69	C2—C6—C10—C9	157
O5—C1—C2—C3	15	C2—C6—C10—C11	−79
O5—C1—C2—C6	−112	C7—C6—C10—C9	34
C1—C2—C6—C7	−167	C7—C6—C10—C11	157
C1—C2—C6—C10	73	C6—C7—C8—C9	−9
C3—C2—C6—C7	68	O12—C7—C8—C9	169
C3—C2—C6—C10	−52	C7—C8—C9—C10	30
C2—C6—C7—C8	−144	C8—C9—C10—C6	−40
C2—C6—C7—O12	38	C8—C9—C10—C11	−165
C10—C6—C7—C8	−15		

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms were placed in calculated positions, except for H(O4) which was found from difference Fourier maps. Anisotropic least-squares refinement was performed (*SHELX*76; Sheldrick, 1976); H atoms were treated isotropically with a common refined temperature factor ($U = 0.06$ Å^2).

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2,9-Dioxo-1,6,8,13-tetraoxadispiro[4.1.4.3]-tetradeca-3,10-diene, C10H8O6

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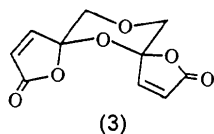69622 Villeurbanne CEDEX, France*

Abstract

The title tricyclic compound was obtained by photo-oxygenation of bis(5-formylfurfuryl) ether, followed by a dehydration reaction. The compound appears as a *trans* isomer, with respect to the relative positions of the lactonic rings to the dioxane ring, which remains chair shaped. Such a *trans* configuration has been found previously in anemonin, where the two lactonic rings are *trans* with respect to the cyclobutane ring.

Comment

It is known that the photo-oxygenation of furfural derivatives may give hydroxybutenolides (Bernasconi *et al.*, 1984; Cottier, Descotes, Nigay, Parron &

Gregoire, 1986). Recently, we were able to stabilize the tertiary alcohol function by an acetylation reaction. Therefore, when this sequence of reactions is applied to bis(5-formylfurfuryl) ether, the acetylation reaction gives the title compound (3). IR and ^1H and ^{13}C NMR spectra have not allowed the elucidation of the structure, in particular, the stereoconfiguration of the lactonic rings with respect to the dioxane ring.

(3)

The asymmetric unit includes two molecules, as shown in the stereodiagram (Fig. 2) drawn using *PLUTO* (Motherwell & Clegg, 1978). The two molecules have the same configuration (Fig. 1). The compound appears as a *trans* isomer, regarding the lactonic rings with respect to the dioxane ring, which remains chair shaped.
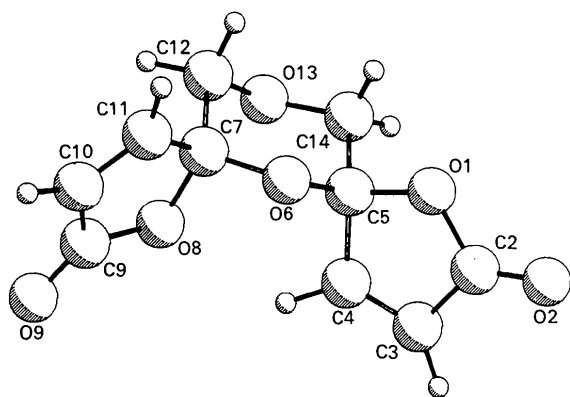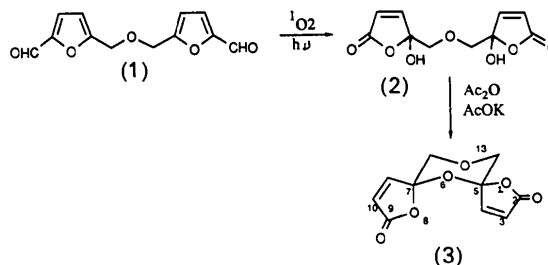
The structure of anemonin, which is a similar tricyclic compound with two lactonic rings, has been elucidated previously (Moriarty, Romain, Karle & Karle, 1965). In this compound, the two lactonic rings are also in *trans* positions with respect to the cyclobutane central ring.

Experimental

The photo-oxygenation of the furfuryl derivative (1) gave the hydroxybutenolide (2). The title compound (3) was obtained by an acetylation reaction. Colourless crystals were obtained from a hexane/ethyl acetate solution (1/4).

Fig. 1. Molecular configuration of the molecule showing the atom-numbering scheme.

Crystal data

$C_{10}H_8O_6$	$D_x = 1.51$ Mg m^{-3}
$M_r = 224.2$	Cu $K\alpha$ radiation
Triclinic	$\lambda = 1.5424$ Å
$P\bar{1}$	Cell parameters from 25
$a = 8.048$ (1) Å	reflections
$b = 8.398$ (1) Å	$\theta = 26.7$–$52.5°$
$c = 15.918$ (2) Å	$\mu = 1.06$ mm^{-1}
$\alpha = 101.54$ (1)°	$T = 291$ K
$\beta = 98.88$ (1)°	Prism
$\gamma = 106.62$ (1)°	$0.50 \times 0.45 \times 0.35$ mm
$V = 984.2$ Å^3	Colourless
$Z = 4$	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.038$
diffractometer	$\theta_{max} = 73°$
ω–$5/3\theta$ scans	$h = 0 \rightarrow 9$
Absorption correction:	$k = -10 \rightarrow 9$
empirical	$l = -19 \rightarrow 19$
$T_{min} = 0.967$, $T_{max} = 1.00$	3 standard reflections
4037 measured reflections	frequency: 60 min
3864 independent reflections	intensity variation: 5.4%
3579 observed reflections	
$[I > 3\sigma(I)]$	

Refinement

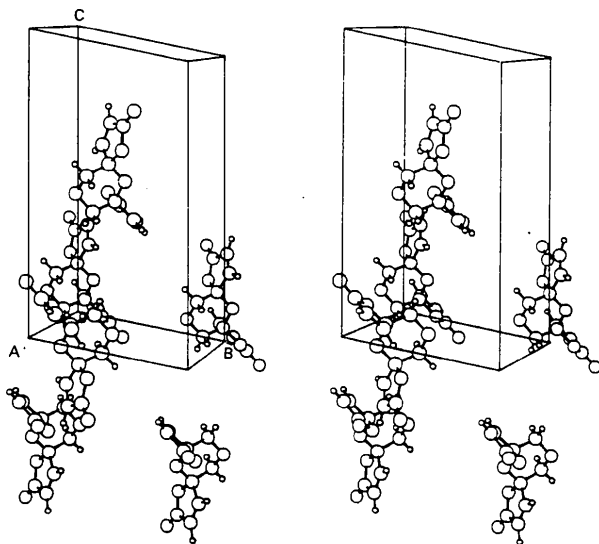|	
Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.058$	$(\Delta/\sigma)_{max} = 0.21$
$wR = 0.057$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$S = 0.61$	$\Delta\rho_{min} = -0.27$ e Å^{-3}
3579 reflections	Atomic scattering factors
337 parameters	from *SDP* (B. A. Frenz &
Only coordinates of H atoms	Associates, Inc., 1982)
refined	

Fig. 2. Stereoscopic view of the title compound.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($\mathring{A}^2$)

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O1A	0.7749 (2)	0.0674 (2)	0.0912 (1)	4.32 (4)
C2A	0.9373 (4)	0.0593 (3)	0.1283 (2)	4.34 (6)
O2A	0.9486 (3)	−0.0345 (3)	0.1741 (1)	6.20 (6)
C3A	1.0748 (4)	0.1884 (3)	0.1029 (2)	4.53 (6)
C4A	0.9972 (3)	0.2664 (3)	0.0544 (2)	3.91 (6)
C5A	0.7979 (3)	0.1977 (3)	0.0441 (2)	3.52 (5)
O6A	0.7086 (2)	0.1083 (2)	−0.0445 (1)	3.44 (4)
C7A	0.6647 (3)	0.2107 (3)	−0.0982 (2)	3.32 (5)
O8A	0.8263 (2)	0.3123 (2)	−0.1180 (1)	3.54 (4)
C9A	0.8134 (3)	0.2735 (3)	−0.2053 (2)	3.80 (5)
O9A	0.9240 (3)	0.3476 (3)	−0.2393 (1)	5.26 (5)
C10A	0.6411 (3)	0.1315 (3)	−0.2482 (2)	3.98 (6)
C11A	0.5577 (3)	0.0956 (3)	−0.1866 (2)	3.77 (6)
C12A	0.5797 (3)	0.3333 (3)	−0.0549 (2)	4.15 (6)
O13A	0.6921 (3)	0.4331 (2)	0.0282 (1)	4.63 (4)
C14A	0.7130 (4)	0.3246 (4)	0.0832 (2)	4.76 (7)
O1B	0.4665 (2)	0.4320 (2)	0.6281 (1)	3.92 (4)
C2B	0.4331 (3)	0.4318 (3)	0.7092 (2)	3.92 (6)
O2B	0.5349 (3)	0.5313 (3)	0.7743 (1)	5.90 (6)
C3B	0.2619 (3)	0.2954 (3)	0.6988 (2)	3.97 (6)
C4B	0.1974 (3)	0.2226 (3)	0.6147 (2)	3.69 (5)
C5B	0.3249 (3)	0.3007 (3)	0.5621 (2)	3.34 (5)
O6B	0.2543 (2)	0.3923 (2)	0.5083 (1)	3.46 (4)
C7B	0.1486 (3)	0.2929 (3)	0.4245 (2)	3.38 (5)
O8B	−0.0244 (2)	0.1945 (2)	0.4358 (1)	3.78 (4)
C9B	−0.1524 (3)	0.2393 (3)	0.3893 (2)	4.04 (6)
O9B	−0.3090 (2)	0.1691 (3)	0.3844 (2)	5.78 (6)
C10B	−0.0667 (4)	0.3759 (3)	0.3497 (2)	4.40 (6)
C11B	0.1064 (3)	0.4095 (3)	0.3716 (2)	4.05 (6)
C12B	0.2298 (3)	0.1682 (3)	0.3798 (2)	4.00 (6)
O13B	0.2763 (2)	0.0695 (2)	0.4358 (1)	4.30 (4)
C14B	0.4052 (4)	0.1813 (3)	0.5126 (2)	4.33 (6)

Table 2. *Selected geometric parameters* ($\mathring{A}$, °)

O1A—C2A	1.374 (4)	O1B—C2B	1.359 (3)
O1A—C5A	1.432 (3)	O1B—C5B	1.437 (2)
C2A—O2A	1.188 (4)	C2B—O2B	1.201 (3)
C2A—C3A	1.480 (4)	C2B—C3B	1.480 (3)
C3A—C4A	1.305 (4)	C3B—C4B	1.309 (3)
C4A—C5A	1.511 (3)	C4B—C5B	1.510 (4)
C5A—O6A	1.423 (3)	C5B—O6B	1.419 (3)
C5A—C14A	1.508 (4)	C5B—C14B	1.503 (4)
O6A—C7A	1.404 (3)	O6B—C7B	1.414 (2)
C7A—O8A	1.455 (3)	C7B—O8B	1.460 (3)
C7A—C11A	1.503 (3)	C7B—C11B	1.489 (4)
C7A—C12A	1.506 (4)	C7B—C12B	1.506 (4)
O8A—C9A	1.344 (3)	O8B—C9B	1.360 (3)
C9A—O9A	1.199 (3)	C9B—O9B	1.211 (3)
C9A—C10A	1.499 (3)	C9B—C10B	1.471 (4)
C10A—C11A	1.308 (4)	C10B—C11B	1.314 (4)
C12A—O13A	1.423 (3)	C12B—O13B	1.411 (4)
O13A—C14A	1.410 (4)	O13B—C14B	1.429 (3)
C2A—O1A—C5A	110.4 (2)	C9A—C10A—C11A	107.9 (2)
O1A—C2A—O2A	121.2 (3)	C7A—C11A—C10A	110.3 (2)
O1A—C2A—C3A	107.1 (2)	C7A—C12A—O13A	109.4 (2)
O2A—C2A—C3A	131.6 (3)	C12A—O13A—C14A	109.8 (2)
C2A—C3A—C4A	109.1 (2)	C5A—C14A—O13A	111.2 (2)
C3A—C4A—C5A	109.9 (2)	C7B—O8B—C9B	108.5 (2)
O1A—C5A—C4A	103.5 (2)	O8B—C9B—O9B	121.4 (3)
O1A—C5A—O6A	104.8 (2)	O8B—C9B—C10B	108.9 (2)
O1A—C5A—C14A	107.7 (2)	O9B—C9B—C10B	129.7 (3)
C4A—C5A—O6A	111.9 (2)	C9B—C10B—C11B	108.3 (3)
C4A—C5A—C14A	116.0 (2)	C2B—O1B—C5B	109.9 (2)
O6A—C5A—C14A	111.9 (2)	O1B—C2B—O2B	121.2 (2)
C5A—O6A—C7A	115.4 (2)	O1B—C2B—C3B	108.3 (2)
O6A—C7A—O8A	109.0 (2)	O2B—C2B—C3B	130.5 (3)
O6B—C7B—C11B	109.2 (2)	C2B—C3B—C4B	108.1 (2)
O6B—C7B—C12B	112.4 (2)	C3B—C4B—C5B	110.2 (2)
O8B—C7B—C11B	104.0 (2)	O1B—C5B—C4B	103.4 (2)
O8B—C7B—C12B	108.2 (2)	O1B—C5B—O6B	104.2 (2)

C11B—C7B—C12B	114.3 (2)	O1B—C5B—C14B	107.7 (2)
O6A—C7A—C11A	108.7 (2)	C4B—C5B—O6B	112.6 (2)
O6A—C7A—C12A	112.7 (2)	C4B—C5B—C14B	115.6 (2)
O8A—C7A—C11A	103.6 (2)	O6B—C5B—C14B	112.1 (2)
O8A—C7A—C12A	107.7 (2)	C5B—O6B—C7B	115.7 (2)
C11A—C7A—C12A	114.7 (2)	O6B—C7B—O8B	108.3 (2)
C7A—O8A—C9A	109.8 (2)	C7B—C11B—C10B	110.2 (2)
O8A—C9A—O9A	123.0 (2)	C7B—C12B—O13B	111.1 (2)
O8A—C9A—C10A	108.3 (2)	C12B—O13B—C14B	109.3 (2)
O9A—C9A—C10A	128.6 (2)	C5B—C14B—O13B	109.4 (2)

The structure was solved with the Enraf–Nonius *SDP* system (B. A. Frenz & Associates, Inc., 1982) using a DEC MicroVAX 3100-80 computer, at the Centre de Diffractométrie Automatique, Université Lyon I.

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An Intermediate in the Synthesis of Formoterol

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Abstract

In the title compound, 4-benzyloxy-3-nitrophenacyl bromide, $C_{15}H_{12}BrNO_4$, the two planar nitrophenyl and benzyloxy groups are inclined at a dihedral angle of 17.1 (1)°. The NO_2 group is twisted out of the