

Monoclinic

$P2_1/n$
 $a = 15.1665$ (16) Å
 $b = 6.2451$ (6) Å
 $c = 19.946$ (2) Å
 $\beta = 99.650$ (2)°
 $V = 1862.5$ (3) Å³
 $Z = 4$
 $D_x = 1.168$ Mg m⁻³
 D_m not measured

Cell parameters from 4298 reflections
 $\theta = 1.57$ – 27.50 °
 $\mu = 0.071$ mm⁻¹
 $T = 293$ (2) K
 Block
 $0.30 \times 0.14 \times 0.12$ mm
 Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: none
 10 485 measured reflections
 3221 independent reflections
 1705 reflections with
 $I > 2\sigma(I)$

$R_{int} = 0.093$
 $\theta_{max} = 25$ °
 $h = -18 \rightarrow 17$
 $k = -5 \rightarrow 7$
 $l = -23 \rightarrow 23$
 Intensity decay: negligible

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.157$
 $S = 1.036$
 3221 reflections
 227 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0606P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.24$ e Å⁻³
 $\Delta\rho_{min} = -0.24$ e Å⁻³
 Extinction correction:
SHELXTL (Sheldrick, 1997)
 Extinction coefficient:
 0.011 (2)
 Scattering factors from
International Tables for Crystallography (Vol. C)

Table 2. Selected geometric parameters (Å, °) for (II)

C1—C7	1.461 (3)	C9—N11	1.374 (3)
C7—C8	1.323 (3)	N11—C19	1.434 (3)
C8—C9	1.471 (3)	N11—C12	1.465 (3)
C9—O10	1.225 (3)	C22—C25	1.510 (4)
C8—C7—C1	128.5 (3)	C9—N11—C12	117.5 (2)
C9—N11—C19	125.2 (2)	C19—N11—C12	117.1 (2)

Table 3. Hydrogen-bonding geometry (Å, °) for (II)

D—H...A	D—H	H...A	D...A	D—H...A
C7—H7A...O10 ⁱ	0.93	2.51	3.370 (3)	154

Symmetry code: (i) $-x, 1 - y, -z$.

For both compounds, data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structures: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structures: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1216). Services for accessing these data are described at the back of the journal.

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rac-5,5'-Bis(2-acetoxypopyl)-2,2'-fural

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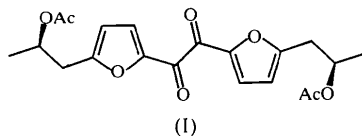
Abstract

The title compound, 2,2'-fural-5,5'-diyldi-prop-2-yl diacetate, C₂₀H₂₂O₈, lies about an inversion centre and forms a ladder-like structure wherein the furil backbone builds the steps which are then connected to each other by hydrogen-bond bridges (O...H distance 2.64 Å) between the side groups. The furan rings of neighbouring molecules are coplanar, with an interfacial distance of 3.65 Å.

Comment

The title compound, (I), was synthesized from *rac*-5-(2-acetoxypopyl)-2-furancarbaldehyde, a key intermediate in the total synthesis of the macrotretrolid Nonactin

(Schmidt *et al.*, 1976). Starting from *rac*-propylene oxide, this aldehyde can be made in three steps. Furoin condensation of the aldehyde with the help of *N*-benzene-thiazolium salt as the catalyst (Stetter *et al.*, 1976; Stetter & Dämbkes, 1977; Lee *et al.*, 1992) afforded not the furoin but the title compound, most likely through oxidation of the intermediate furoin by air. However, only one other crystal structure of a 2,2'-furyl system is known, namely that of the unsubstituted derivative (Biswas *et al.*, 1987).



The molecule of (I) possesses crystallographically imposed inversion symmetry, and the centre of the central C—C single bond represents the inversion centre. Due to this inversion symmetry, the inter-carbonyl dihedral angle amounts to 0°. The presence of a furil structure is proved by the bond lengths of the central O—C—C—O fragment [C10—C10ⁱ = 1.552 (7) Å and C10=O2 = 1.218 (4) Å; symmetry code: (i) $\frac{1}{2} - x, -\frac{1}{2} - y, 2 - z$]. The elongation of the central C^{*sp*2}—C^{*sp*2} bond is a typical feature of dicarbonyl systems and corresponds to bond distances of 1.520 (4), 1.542 (3), 1.541 (2) and 1.533 (3) Å found in α -furyl, benzil, α -naphthil and mesitil, respectively (Biswas *et al.*, 1987, and references therein).

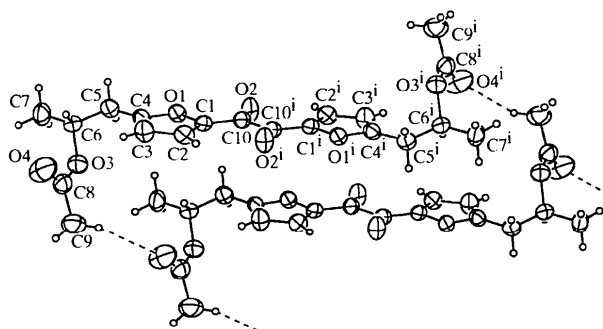


Fig. 1. The ladder-like arrangement of (I), with the atom-numbering scheme and displacement ellipsoids shown at the 50% probability level. Dashed lines indicate hydrogen-bond bridges and H atoms are drawn as spheres of arbitrary radii [symmetry code: (i) $\frac{1}{2} - x, -\frac{1}{2} - y, 2 - z$].

While all bond lengths are within the expected ranges (Table 1), the molecular arrangement is of interest. The two acetoxypropyl side groups are arranged above and below the furil plane and form hydrogen-bond bridges [O...H distance 2.64 Å; sum of the corresponding van der Waals radii 2.72 Å (Bondi, 1964)] between the acetyl O4ⁱⁱ atom and the methyl H9C atom of adjacent molecules [symmetry code: (ii) $x, y - 1, z$] (Fig. 1). The crystal packing can be described as π -stacks of the mol-

ecules along the *b* axis (Fig. 2). Within these stacks, the molecules are slipped relative to each other by about 3.60 Å. The shortest interplanar distance measured from the centre of a furan ring to the keto C10 atom of the adjacent molecule is found to be 3.65 Å. No evidence can be found for interstack interactions.

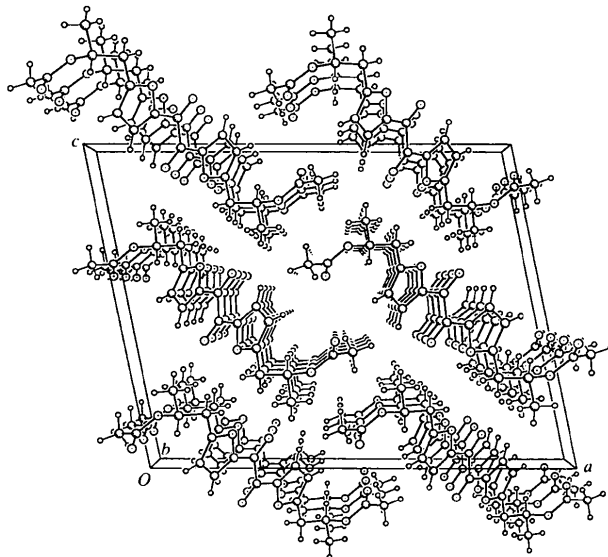


Fig. 2. View of the π -stacks in (I) along the *b* axis.

Experimental

Under an inert gas atmosphere, 3-benzyl-5-(2-hydroxyethyl)-4-methylthiazolium chloride (675 mg, 2.5 mmol) was added to freshly distilled (2-acetoxypropyl)-2-furancarbaldehyde (9.85 g, 50 mmol). The mixture was dissolved with stirring in EtOH (15 ml) and dry NEt₃ (7.5 ml), and the resulting deep-red solution was kept at room temperature overnight. During this time, the colour turned to orange. Thereafter, iced water (100 ml) was added followed by extraction with 200 ml of CHCl₃ in four portions. The organic layer was washed with saturated NaHCO₃ solution and water. After removing the solvent, the resulting dark orange oil was stirred with *n*-hexane under ice cooling to produce a yellow microcrystalline solid. This was filtered off, washed with small amounts of *n*-hexane and dried under vacuum. From the filtrate, another fraction could be isolated upon addition of a cold mixture of ethyl acetate—CCl₄ (ratio 5:3) [total yield: 1.95 g (20%) of yellow powder]. Yellow single crystals of (I) could be grown by slow evaporation of the solvent from a saturated *n*-hexane—CH₂Cl₂ solution. Elemental analysis of C₂₀H₂₂O₈ (*M*_r = 390), calculated: C 61.54, H 5.64%; found: C 60.98, H 5.78%. Spectroscopic analysis, IR (KBr, cm⁻¹): 1731 [*ss*, ν (C=O), OAc], 1637 [*ss*, ν (C=O), O=C—C=O]; ¹H NMR (CDCl₃, p.p.m.): 1.25 (*d*, CH₃, 3H), 2.00 (*s*, acetyl CH₃, 3H), 3.00 (*d*, CH₂, 2H), 5.20 (*m*, CH, 1H), 6.30 (*d*, 3-H furan, 1H), 7.55 (*d*, 4-H furan, 1H); ¹³C NMR (CDCl₃, p.p.m.): 19.9 (CH₃), 21.1 (CH₂), 34.9 (acetyl CH₃), 68.5 (CH), 111.0 (3-C furan), 126.2 (4-C furan), 149.1 (2-C furan), 160.5 (5-C furan), 170.2 (acetyl C=O), 176.5 (C=O).

Crystal data

C₂₀H₂₂O₈
M_r = 390.38
 Monoclinic
*C*2/*c*
a = 21.644 (9) Å
b = 5.398 (2) Å
c = 16.765 (5) Å
 β = 101.97 (2)°
V = 1916.1 (12) Å³
Z = 4
D_x = 1.353 Mg m⁻³
D_m not measured

Data collection

Siemens P4 diffractometer
 Profile data from ω scans
 Absorption correction: none
 2265 measured reflections
 1693 independent reflections
 923 reflections with
 $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.067
wR(*F*²) = 0.191
S = 1.026
 1693 reflections
 129 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.0846P)^2 + 0.9902P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Mo *K*α radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 28 reflections
 $\theta = 3.80\text{--}10.75^\circ$
 $\mu = 0.105$ mm⁻¹
T = 200 (2) K
 Plate
 0.80 × 0.30 × 0.08 mm
 Yellow

$\theta_{\max} = 25^\circ$
 $h = -1 \rightarrow 25$
 $k = -1 \rightarrow 6$
 $l = -19 \rightarrow 19$
 3 standard reflections
 every 100 reflections
 intensity decay: 2.42%

(Δ/σ)_{max} = 0.035
 $\Delta\rho_{\max} = 0.317$ e Å⁻³
 $\Delta\rho_{\min} = -0.201$ e Å⁻³
 Extinction correction: none
 Scattering factors from
International Tables for Crystallography (Vol. C)

Software used to prepare material for publication: *SHELXTL* and *PARST95* (Nardelli, 1995).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1337). Services for accessing these data are described at the back of the journal.

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4-Benzoyl-2-chloro-6-phenylpyrimidine

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Abstract

In the title compound, C₁₇H₁₁ClN₂O, the molecule contains an essentially planar pyrimidine ring with the 4- and 6-positions substituted by benzoyl and phenyl groups, respectively. The phenyl ring and the planar part of the benzoyl moiety form dihedral angles of 12.3 (1) and 62.1 (1)°, respectively, with the pyrimidine ring. The two phenyl rings are inclined at an angle of 130.0 (1)° with respect to one another.

Comment

The pyrimidines form a class of biologically active organic compounds important for their possible pharma-

Table 1. Selected geometric parameters (Å, °)

O1—C4	1.364 (5)	C2—C3	1.406 (6)
O1—C1	1.385 (4)	C3—C4	1.363 (5)
O2—C10	1.218 (4)	C4—C5	1.479 (5)
O3—C8	1.339 (5)	C5—C6	1.507 (6)
O3—C6	1.459 (5)	C6—C7	1.501 (6)
O4—C8	1.196 (5)	C8—C9	1.477 (6)
C1—C2	1.351 (5)	C10—C10'	1.552 (7)
C1—C10	1.445 (5)		
C3—C4—C5	134.0 (4)	O2—C10—C1	122.5 (3)
O1—C4—C5	116.9 (3)	O2—C10—C10'	120.8 (5)

Symmetry code: (i) $\frac{1}{2} - x, -\frac{1}{2} - y, 2 - z$.

Table 2. Hydrogen-bonding geometry (Å, °)

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
C9—H9C...O4 ⁱⁱ	0.96	2.64	3.395 (7)	136

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

H atoms are geometrically positioned and refined using a riding model (secondary CH₂ and aromatic CH groups) and additional rotating-group refinement (CH₃ groups) by maximizing the sum of the electron density at the three calculated H-atom positions.

Data collection: *XSCANS* (Siemens, 1996). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*.