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

C4—C2′	1.475 (2)		
C5—C4—C2′—C3′	−31.9 (10)	C9—C2—C1—C10	2.10 (5)
C5—C4—C2′—C3″	143.7 (9)		

All H atoms were refined using a riding model. In order to obtain a better data/parameter ratio for (1), 203 similarity restraints were applied to the *U* components of C atoms. For (2), a system of 301 restraints was employed. The thiophene ring in (2) is disordered in the crystal and was refined on two positions of occupancies 0.514 (3) and 0.486 (3). The final model file with details of disorder restraints is included in the deposited material.

In (1), the origin was fixed according to the method of Flack & Schwarzenbach (1988). The absolute structure cannot be determined because the anomalous scattering is too small [the Flack (1983) parameter is indeterminate]. Friedel opposites were therefore merged before refinement.

For both compounds, data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1314). Services for accessing these data are described at the back of the journal.

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A racemic strigol analogue at 100 K

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Abstract

DL-3-[(2,5-Dihydro-3-methyl-2-oxo-5-furyl)oxymethylene]-3,3a,6,6a-tetrahydro-2*H*-cyclopenta[*b*]furan-2-one, $C_{13}H_{12}O_5$, has relative configurations at the asymmetric C atoms of 3a*S*, 6a*S* and 5′*R*. The dihedral angle formed by the fused cyclopentene and furan rings is 114.81 (5)°, and these rings form dihedral angles of 119.53 (5) and 67.68 (5)°, respectively, with the methylfuranone ring.

Comment

Witchweed (*Striga* species) is an angiospermous obligate root parasite that attacks corn, sorghum, sugarcane, rice and more than 60 other important crop plants and weeds of the grass family (Worsham, 1987). These beautifully adapted parasitic weeds occur commonly in the eastern hemisphere and inflict serious crop damage in many parts of the world (Johnson *et al.*, 1976). Their life-cycle is closely coupled to their environment and to their host. It is difficult to control *Striga* species because they produce tiny seeds in vast amounts that can remain viable in the soil for as long as 20 years. The seeds will germinate only when stimulated by a chemical exuded from the roots of the host crops (Rugutt, 1996). When the *Striga* stimulant, strigol (Cook *et al.*, 1972), was isolated from cotton (*Gossypium hirsutum* L.), a non-host plant, the possibility of weed control by introduction into the soil of a synthetic stimulant to induce suicidal germination of seeds of the parasite was revived. Therefore, considerable effort has been put into the preparation of simpler but structurally related strigol analogues that retain high germination activity (MacAlphine *et al.*, 1976; Johnson *et al.*, 1976). One such strigol analogue,

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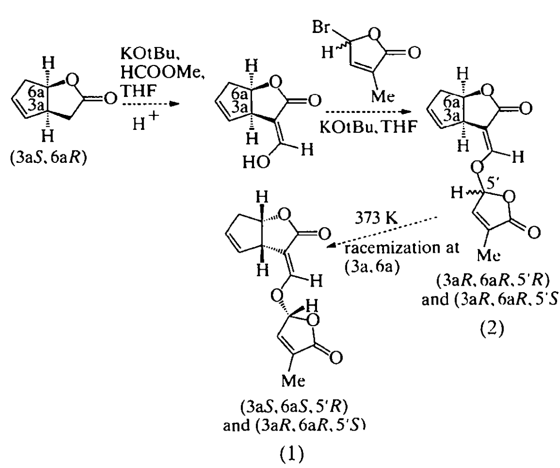
GR7, (1), was prepared by Johnson & Rosebery (1977), but not separated into its diastereomers. In (1), the *cis* fusion of the five-membered rings and the chiral center in the butenolide ring give rise to two diastereomeric pairs of enantiomers. Bosman *et al.* (1992) determined the crystal structure and absolute configuration of the (3a*R*,6a*R*,5′*R*) diastereomer, (2).



As part of an ongoing program concerned with the development of separation methodology for direct enantiomeric resolution of racemic drugs, agrochemicals, pesticides and herbicides using polymerized chiral surfactants (Shamsi & Warner, 1997, and references therein), we have synthesized the title compound [as outlined by Johnson *et al.* (1981)]. Although our NMR data of racemic (1) correlated reasonably well with those in the literature (Heather *et al.*, 1976), points of ambiguity were apparent. For this reason, and also to confirm the structures assigned to the diastereomers of (1), we undertook the crystal structure determination of what proved to be the racemic modification of the opposite diastereomer from that studied by Bosman *et al.* (1992). Our isolation of racemic crystals resulted from drying at 373 K, which was apparently not carried out by Bosman *et al.* (1992). Interestingly, the melting point of racemic (1) (407.5 K) agrees with that reported by Bosman *et al.* (1992) for resolved 3a*R*,6a*R*,5′*R* (407–408 K).

The asymmetric unit of (1) is seen in Fig. 1 to have the *S* configuration at the ring-fusion atoms C3a and C6a, and the *R* configuration at C5′. The optically pure diastereomer (2), studied by Bosman *et al.* (1992) at 293 K, also has the *R* configuration at C5′, but has the *R* configuration at positions C3a and C6a, unlike (1). The 21 C—C and C—O bond distances in (1) and (2) agree reasonably well, exhibiting a mean difference of 0.012 Å. When statistically significant differences occur, the distance in the room-temperature determination of (2) is usually slightly shorter. The dihedral angle formed by the fused cyclopentene and furan rings in (1) is 114.81 (5)°, compared with 116.9 (1)° in (2). The major difference between the conformations of (1) and (2) is in the relationship of the methylfuranone ring and the fused ring system. Our C7—O3—C5′—C4′ torsion angle is 176.5 (1)°, *versus* 155.1 (2)° in (2). Smaller differences also exist in the torsion angles about C3—C7 and C7—O3. This conformation of the connecting chain in (1) leads to the dihedral angles given in the *Abstract*.

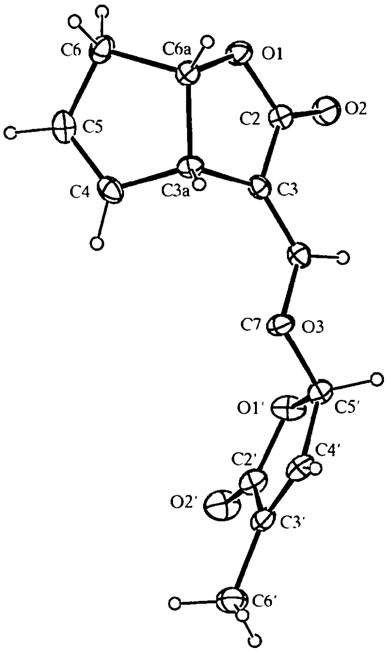


Fig. 1. The atomic numbering scheme and ellipsoids at the 50% probability level for (1), with H atoms represented with arbitrary radii.

Experimental

The title compound was prepared from the commercially available optically pure bicyclic lactone (3a*S*,6a*R*)-3,3a,6,6a-tetrahydro-2*H*-cyclopenta[*b*]furan-2-one. This starting material was purchased optically pure from Fluka and had $[\alpha]_D^{20}$ +104° (concentration 0.7 Mg m^{-3}, CH_3OH). It was formylated as shown in the Scheme. Racemic 5-bromo-3-methyl-2(5*H*)-furanone was prepared as outlined by Heather *et al.* (1976), and was immediately coupled with the formylated lactone, as described by Johnson *et al.* (1981), producing a mixture of diastereomers at C5′. Drying at 373 K for 72 h led to racemization at C3a and C6a, yielding the reported racemate shown in the Scheme, and also the racemic form of the compound reported by Bosman *et al.* (1992). Crystals of the reported racemate were grown from chloroform–diethyl ether (1:2 *v*/*v*) as needles (m.p. 407.5 K).

Crystal data

$C_{13}H_{12}O_5$
$M_r = 248.24$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1/c$
$a = 12.748\,(2)$ Å
$b = 6.110\,(3)$ Å
$c = 14.870\,(6)$ Å
$\beta = 99.51\,(2)°$
$V = 1142\,(1)$ Å^3
$Z = 4$
$D_x = 1.443$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 11–19°$
$\mu = 0.105$ mm^{-1}
$T = 100$ K
Needle fragment
0.57 × 0.55 × 0.18 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer (with Oxford Cryostream cooler)
$\theta/2\theta$ scans
Absorption correction: none
6069 measured reflections
3454 independent reflections
2926 reflections with $I > \sigma(I)$

$R_{\rm int} = 0.030$
$\theta_{\rm max} = 30.0°$
$h = -17 \rightarrow 16$
$k = 0 \rightarrow 8$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity decay: 1.8%

Refinement

Refinement on F^2
$R(F) = 0.050$
$wR(F^2) = 0.055$
$S = 1.976$
2926 reflections
212 parameters
All H atoms refined
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$

$(\Delta/\sigma)_{\rm max} = 0.026$
$\Delta\rho_{\rm max} = 0.40$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.33$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

O1′—C2′	1.373 (2)	C3a—C6a	1.555 (2)
O1′—C5′	1.436 (2)	C3—C7	1.332 (2)
O1—C2	1.351 (2)	C3′—C4′	1.313 (2)
O1—C6a	1.462 (2)	C4—C5	1.337 (2)
O2—C2	1.211 (2)		
C2—O1—C6a	111.7 (1)	C3a—C4—C5	112.4 (1)
O1—C2—C3	109.3 (1)	C4—C5—C6	112.4 (1)
C3—C3a—C6a	102.3 (1)	O1—C6a—C3a	105.9 (1)
C4—C3a—C6a	102.8 (1)	C3a—C6a—C6	107.0 (1)
C2—C3—C3a	108.8 (1)	C5—C6—C6a	104.1 (1)
C7—O3—C5′—C4′	176.5 (1)	C2—C3—C7—O3	−179.7 (1)
C5′—O3—C7—C3	170.4 (1)		

C—H distances range from 0.91 (2) to 1.00 (2) Å, while $B_{\rm iso}$ values for H atoms range from 0.9 (3) to 5.2 (6) Å^2.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: Direct methods *SIR* (Burla *et al.*, 1989). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *BTABLE PTABLE CIF IN* in *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1062). Services for accessing these data are described at the back of the journal.

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Two novel 3,4-disubstituted 1,2,4-oxadiazole-5(4*H*)-thiones

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Abstract

Compound (1), 3-(3-pyridyl)-4-*p*-tolyl-1,2,4-oxadiazole-5(4*H*)-thione, $C_{14}H_{11}N_3OS$, and compound (2), 3-(2-pyridyl)-4-propyl-1,2,4-oxadiazole-5(4*H*)-thione,