

***meso*-2,2'-Diphenyl-3,3',4,4'-tetrahydro-2,2'-bifuran-5,5'(2*H*,2'*H*)-dione**

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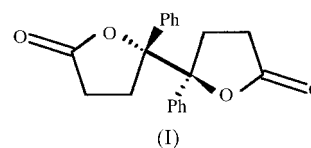
Data validation number: IUC0000269

The crystal structure of the title compound, C₂₀H₁₈O₄, contains a crystallographic inversion center. The C—C bond linking the two halves of the molecule is slightly elongated at 1.577 (3) Å.

Comment

Samarium diiodide (Girard *et al.*, 1980) has been used for some time as a highly selective one-electron reductant (Molander, 1992). Carbonyl compounds, specifically ketones and aldehydes, find themselves amongst the types of functionality reduced by this reagent. In reactions with SmI₂, these carbonyl moieties are reduced to the corresponding samarium ketyl radicals (Molander & Harris, 1996), which are subject to further reactions. In line with our interest in 1,4-dicarbonyl compounds (Williams *et al.*, 2000a), we investigated the samarium(II) iodide-mediated reactions of γ -keto esters. We anticipated that the substrate would be reduced to the corresponding ketyl radical, which might undergo pinacol dimerization or further reduction and protonation to the corresponding samarium alkoxide. Following either of these routes, we believed that the intermediate alkoxide would undergo intramolecular lactonization. We found, as the outcome of one such reaction (Williams *et al.*, 2000b), that the initially formed samarium ketyl radical underwent a pinacol dimerization coupling in the absence of a proton source, followed by lactonization to yield the substituted bis- γ -butyrolactone as both the racemate (16%) and the *meso* (16%) compound. These products were readily separable by column chromatography. The structural study was undertaken as the interpretation of the NMR data was inconclusive in distinguishing between the product as a γ -lactone or a δ -lactone.

The structural features of the near planar lactone ring present in the title molecule, (I), compare favourably with similar structure fragments retrieved from the Cambridge Structural Database (Allen & Kennard, 1993).



Experimental

To a solution of SmI₂ (2.4 equivalents, 0.096) in tetrahydrofuran (10 ml) under reflux (oil-bath temperature 348 K) was added ethyl 4-oxo-4-phenylbutanoate (0.39 mmol) in tetrahydrofuran (2 ml). The resulting solution was heated under reflux for 8 h, after which it was quenched with 0.2 ml of a 25% aqueous solution of NH₄Cl. The volatile component of the mixture was removed *in vacuo* and the residue was passed through a short filter column of silica. The products were finally purified by column chromatography (dichloromethane). The title compound, (I), was recrystallized by slow evaporation from a solution of dichloromethane and ethyl acetate, yielding colorless rectangular crystals (m.p. 511–513 K).

Crystal data

| | |
|--|---|
| C ₂₀ H ₁₈ O ₄ | $D_x = 1.333 \text{ Mg m}^{-3}$ |
| $M_r = 322.34$ | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/c$ | Cell parameters from 8662 reflections |
| $a = 9.4950 (11) \text{ \AA}$ | $\theta = 5.62\text{--}28.30^\circ$ |
| $b = 8.4241 (10) \text{ \AA}$ | $\mu = 0.092 \text{ mm}^{-1}$ |
| $c = 10.0938 (12) \text{ \AA}$ | $T = 296 (2) \text{ K}$ |
| $\beta = 95.876 (2)^\circ$ | Cleaved rectangle, colorless |
| $V = 803.13 (16) \text{ \AA}^3$ | $0.64 \times 0.46 \times 0.24 \text{ mm}$ |
| $Z = 2$ | |

Data collection

| | |
|--|--|
| Bruker SMART CCD 1K area-detector diffractometer | 1977 independent reflections |
| ω scans | 1458 reflections with $I > 2\sigma(I)$ |
| Absorption correction: empirical (SADABS; Sheldrick, 1996) | $R_{\text{int}} = 0.040$ |
| $T_{\text{min}} = 0.943$, $T_{\text{max}} = 0.978$ | $\theta_{\text{max}} = 28.3^\circ$ |
| 8662 measured reflections | $h = -12 \rightarrow 12$ |
| | $k = -11 \rightarrow 11$ |
| | $l = -13 \rightarrow 13$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | $w = 1/[\sigma^2(F_o^2) + (0.0923P)^2 + 0.0603P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.144$ | $(\Delta/\sigma)_{\text{max}} = 0.003$ |
| $S = 1.013$ | $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$ |
| 1977 reflections | $\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$ |
| 117 parameters | |
| H-atom parameters constrained | |

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-------------|-------------|--------------------|-------------|
| O1—C4 | 1.3590 (17) | C1—C5 | 1.5174 (18) |
| O1—C1 | 1.4556 (15) | C1—C2 | 1.5450 (19) |
| O2—C4 | 1.2019 (18) | C1—C1 ¹ | 1.577 (3) |
| C4—O1—C1 | 111.66 (11) | C3—C2—C1 | 104.37 (12) |
| O1—C1—C5 | 108.14 (10) | C4—C3—C2 | 105.08 (12) |
| O1—C1—C2 | 105.12 (10) | O1—C4—C3 | 110.46 (12) |
| C4—O1—C1—C2 | −13.10 (14) | C1—O1—C4—C3 | 2.55 (15) |
| O1—C1—C2—C3 | 17.93 (15) | C2—C3—C4—O1 | 9.31 (16) |
| C1—C2—C3—C4 | −16.42 (16) | O1—C1—C5—C6 | 161.76 (12) |

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

H-atom positions were calculated geometrically and subsequently allowed to refine using a riding model with a common refined isotropic displacement parameter.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SMART*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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