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#### Key indicators

Single-crystal X-ray study  
T = 180 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
R factor = 0.048  
wR factor = 0.127  
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## 2,3-Diphenyl-2,3-dihydrospiro[1,3-oxazole-5(4H),1'(3'H)-2-benzofuran]-3'-one

Two diastereoisomers of the title compound,  $\text{C}_{22}\text{H}_{17}\text{NO}_3$ , have been prepared by [3 + 2]-cycloaddition of 3-methylenephthalide with C,N-diphenylnitrone. The assigned structures of these compounds were supported by NMR data. The molecular structure of the major cycloadduct was confirmed by single-crystal X-ray diffraction.

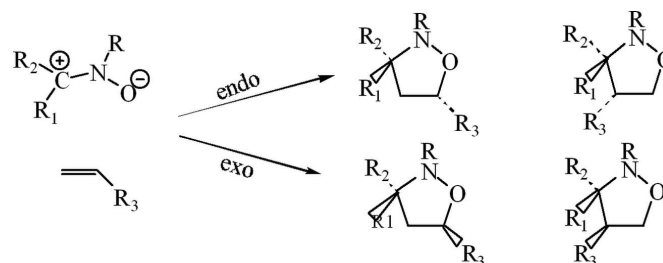
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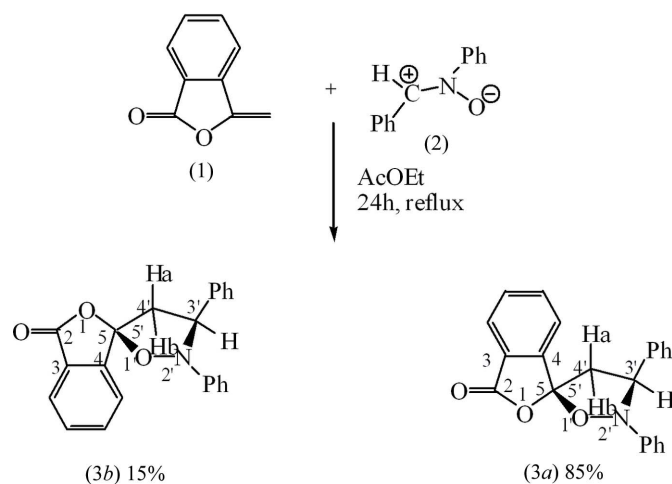
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#### Comment

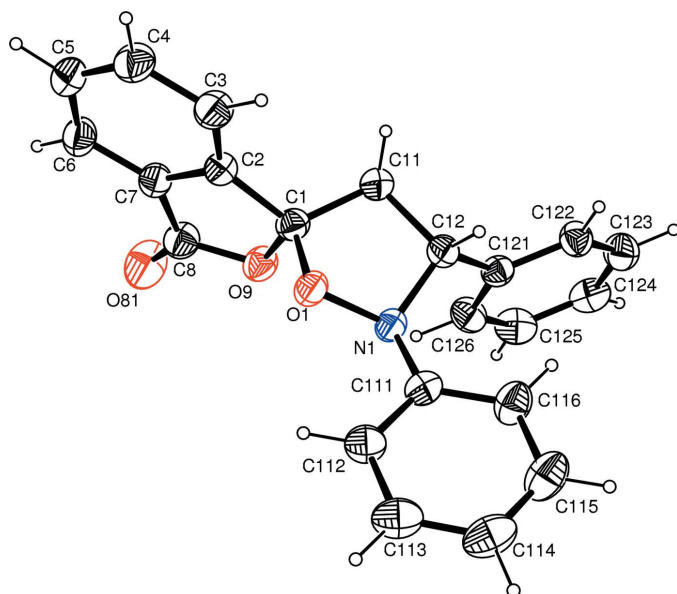
The 1,3-dipolar cycloaddition reactions between nitrones (1,3-dipoles) and alkenes (dipolarophiles) afford an extremely powerful synthetic method for the synthesis of the isoxazolidine ring system (Tufariello, 1984; Confalone & Huie, 1988; Torssell, 1988; Carruthers, 1990; Gr nanger & Vita-Finzi, 1991; Frederickson, 1997). Best regarded as a concerted but asynchronous [3 + 2] suprafacial process, the reaction enables the creation of up to two new carbon stereocenters in a single step. In such a case, the [3 + 2]-addition could provide two pairs of regioisomeric (*i.e.* resulting from different addition senses) and diastereoisomeric (*i.e.* resulting from the *endo* or the *exo* addition) products *via* four possible transition states, as depicted in the first scheme below.



As part of our research on bicyclic spirocompounds (Fihl *et al.*, 1995), we previously reported that the 1,3-dipolar cycloaddition of aromatic nitrile oxides with 3-methylenephthalide, (1), produced 3'-arylspiro[isobenzofuran-1(3H),5'(4'H)-isoxazol]-3-ones, (3), which possess good herbicidal and plant growth regulant activities (Howe & Liu, 1981). Recently, we reported the [3 + 2]-cycloaddition of methylene- $\gamma$ -butyrolactones with several nitrones (Roussel *et al.*, 2003). The reaction is highly regioselective and leads to a mixture of two diastereoisomers, the ratio of which was evaluated by <sup>1</sup>H NMR (performed on the crude reaction mixture). The structures of the spiroadducts, (3a) and (3b), were elucidated only by <sup>1</sup>H and <sup>13</sup>C NMR. Our structural proposition was based on comparison with the spectroscopic data of similar adducts for which the structures are already established (Roussel *et al.*, 2003; Cacciarini *et al.*, 2000).



To confirm unambiguously the structure assignments of (3a) and (3b), and to establish the absolute stereochemistry of each spiroheterocycle, a single-crystal X-ray study was carried out on the major spirocompound, (3a). As shown in Fig. 1, the stereochemistry of (3a) follows from the *endo* C=O approach, which favors the maximum of  $\pi$  electrons in the same space region. The molecule is built up from isobenzofuran-2-one and diphenyloxazoline groups linked by the spiro atom C1. The isobenzofuran fragment is planar, the largest deviation being 0.0295 (0) Å for atom O9, and the ketone atom, O81, lies in this plane. The oxazoline ring has a half-chair conformation with atom O1 lying above the C1/C11/C12/N1 mean plane by 0.603 (2) Å. The isobenzofuran and oxazoline groups make a dihedral angle of 72.95 (4)°. The two phenyl rings, C111–C116 and C121–C126, attached to the oxazoline, are twisted with respect to the C1/C11/C12/N1



**Figure 1**  
The molecular structure of (3a), showing the atom-labeling scheme and displacement ellipsoids drawn at the 30% probability level.

mean plane by 19.80 (9) and 80.21 (5)°, respectively. They are inclined to one another by 80.95 (4)°. The bond lengths and angles within the whole molecule are comparable to values found for related compounds (Cambridge Structural Database, Version 5.26; Allen, 2002).

## Experimental

3-Methylenephthalide, (1), and *C,N*-diphenylnitrone, (2), were synthesized according to the literature procedures (Liu & Howe, 1983; Brüning *et al.*, 1973). A solution of (2) (1.97 g, 10 mmol), (1) (1.46 g, 10 mmol) and hydroquinone (0.05 g) in ethylacetate (40 ml) was stirred and refluxed for 24 h under an atmosphere of nitrogen. The solvent was then evaporated under reduced pressure. The residue was crystallized from ethanol, leading to a mixture of diastereoisomers (3a) and (3b). They were separated and purified by chromatography on silica gel (eluant chloroform/hexane/diethyl ether 50:45:5). The spirocompounds (3a) and (3b) were finally recrystallized from ethanol.

### Crystal data

$C_{22}H_{17}NO_3$   
 $M_r = 343.37$   
 Monoclinic,  $P2_1/n$   
 $a = 5.1476$  (6) Å  
 $b = 15.3139$  (18) Å  
 $c = 21.653$  (3) Å  
 $\beta = 91.365$  (10)°  
 $V = 1706.4$  (4) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.337$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 3224 reflections  
 $\theta = 3.1$ – $28.3^\circ$   
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 180$  (2) K  
 Block, colorless  
 0.51 × 0.37 × 0.35 mm

### Data collection

Oxford Diffraction XCALIBUR diffractometer  
 $\omega$  and  $\varphi$  scans  
 Absorption correction: multi-scan (Blessing, 1995)  
 $T_{min} = 0.932$ ,  $T_{max} = 0.967$   
 13897 measured reflections

4219 independent reflections  
 2777 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.067$   
 $\theta_{max} = 28.3^\circ$   
 $h = -6 \rightarrow 6$   
 $k = -20 \rightarrow 19$   
 $l = -28 \rightarrow 27$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.127$   
 $S = 0.93$   
 4219 reflections  
 235 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0717P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.22$  e Å<sup>-3</sup>

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C–H distances equal to 0.95–1.00 Å and  $U_{iso}(H) = 1.2U_{eq}(C)$ .

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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