C8†	0.6434 (3)	0.2291 (3)	0.4812 (3)	0.0487 (5)
F8A†	0.7035 (2)	0.4299 (2)	0.6055 (2)	0.0646 (4)
F8B†	0.4656 (2)	0.1764 (2)	0.3769 (3)	0.0704 (5)
F8C†	0.6841 (2)	0.1906 (3)	0.6228 (3)	0.0761 (5)
C8'‡	0.609 (2)	0.212 (3)	0.440 (3)	0.049
F8A'‡	0.473 (3)	0.236 (4)	0.320 (4)	0.073
F8B'‡	0.548 (4)	0.087 (3)	0.463 (4)	0.073
F8C'‡	0.703 (3)	0.390 (4)	0.637 (4)	0.073
C9†	0.7027 (3)	0.3492 (3)	0.2174 (3)	0.0463 (5)
F9A†	0.5545 (2)	0.3710 (3)	0.2485 (3)	0.0730 (5)
F9B†	0.8258 (3)	0.5289 (2)	0.3679 (3)	0.0760 (5)
F9C†	0.6542 (2)	0.3049 (3)	0.0198 (2)	0.0607 (4)
C9'‡	0.752 (3)	0.388 (2)	0.270 (4)	0.046
F9A'‡	0.646 (4)	0.319 (5)	0.066 (3)	0.070
F9B'‡	0.672 (3)	0.487 (4)	0.400 (4)	0.070
F9C'‡	0.904 (3)	0.514 (4)	0.347 (5)	0.070

† Site occupancy = 0.946 (2).   ‡ Site occupancy = 0.054 (2).

Table 4. *Selected geometric parameters ($\mathring{A}$, °) for (2)*

C2—F2	1.339 (2)	F2···F3	2.631 (2)
C3—F3	1.341 (2)		
C3—C4—C5—C6	−8.4 (2)	C8—C4—C5—C9	−14.0 (3)

The H atoms in each structure were refined with independent isotropic displacement parameters. The C15 and C16 CF$_3$ groups in (1), and the C8 and C9 CF$_3$ groups in (2) were slightly disordered. In each case, the atoms of the major component of the disorder were refined with anisotropic displacement parameters. The occupancy for the major component in (1) and (2) refined to 0.887 (3) and 0.946 (2), respectively. In each case, the sum of the occupancy of the major plus minor component was constrained to be 1.0. In both structures, the minor disorder component CF$_3$ groups were treated as rigid groups; C—F 1.32 $\mathring{A}$, tetrahedral angles, $U_{iso}(C') = U_{iso}(C)$ and $U_{iso}(F') = 1.5U_{iso}(C')$. The ring–carbon distances were restrained to be the same (*i.e.* C1—C15 = C1—C15', *etc.*; s.u. = 0.01 $\mathring{A}$)

For both compounds, data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Operations Manual*; data reduction: *PROCESS* in *MolEN* (Fair, 1990); program(s) used to solve structures: *MULTAN*80 (Main *et al.*, 1980); program(s) used to refine structures: *SHELXTL* (Sheldrick, 1995); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Ethyl 3,3-Dimethyl-2-(1-oxo-3-phenylinden-2-yl)acrylate

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Abstract

The crystal structure of the title compound, C$_{22}$H$_{20}$O$_3$, was determined from intensities collected at 120 K. The dihedral angle between the phenyl ring and the indenone moiety is 124.6 (1)°. The non-bonded distance between the potentially reacting centers is 3.697 (5) $\mathring{A}$.

Comment

The title compound, (I), was synthesized (Heller, 1985) as a representative of a new potentially photochromic system that is similar to the fulgides (Stobbe & Eckert, 1905; Stobbe, 1911; Heller & Langan, 1981; Heller & Oliver, 1981). It was hoped that the photochemical ring closure from (I) to (II) would be possible.

(I)   (II)
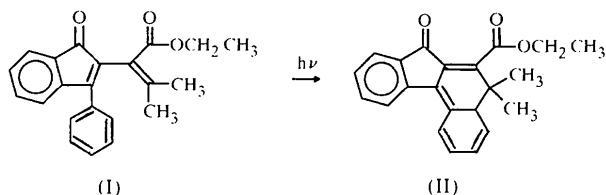
The crystal structure of (I) was determined at low temperature (120 K); the conformation of the butadiene fragment is compared with that of some related fulgides (Kaftory *et al.*, 1998).

Some of the fulgides (Kaftory *et al.*, 1998) are very rigid and because of the bulky substituents at the double bonds, the diene system in these compounds is severely skewed. In contrast, the diene system in (I) is not rigid and free rotation about the C1—C2 bond determines the conformation of the molecule. The torsion angle of the butadienyl fragment, C2—C1—C16—C17, is −63.1 (5)° in (I), while it is 55 (1)° in 2-fluorenylidene-3-adamantylidenesuccinic anhydride and 39 (1)° in 2-fluorenylidene-3-(3,4,5-trimethoxybenzylidene)succinic anhydride, as found by Kaftory *et al.* (1998).

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The dihedral angle between the phenyl ring and the cyclopentene fragment was compared with those of five similar compounds possessing the phenylindenone fragment, namely, 2,3-diphenylinden-1-one (Watson & Nagl, 1987), 3-(2-fluorophenyl)-2-phenylindenone and 6-methyl-2,3-diphenylindenone (Butler et al., 1987), 2,3-diphenyl-6-methoxyindenone (Anstead et al., 1988), and 2,3-diphenyl-1H-benzinden-1-one (Butler et al., 1990) [the refcodes in the Cambridge Structural Database (CSD; Allen et al., 1979) are COCVAR, FEKXEY, FEKXIC, GAWWAC and SIFMUP for the four compounds, respectively]. The range of the dihedral angle in these compounds is 119.2–129.2°. The dihedral angle found in the present work is 126.6 (1)°.

The geometric parameters that are relevant to the photochromic properties are compared for the following four fulgide systems that contain isopropylidene moieties: the title compound, (I), (E)-2-isopropylidene-3-[1-(2-methyl-5-phenyl-3-thienyl)ethylidene]succinic anhydride (Kaftory, 1984; CICWOA in the CSD), 1-(E)-2-isopropylidene-3-(1-naphthylmethylene)succinic anhydride (Kumar & Venkatesan, 1993; LAVLOJ in the CSD) and (E)-2,5-dimethylethylidene(isopropylidene)succinic anhydride (Yoshioka et al., 1989; VEGVIM in the CSD). The non-bonded distances between the potential reaction centers (C15 and C17 in the present work) are 3.697 (5), 3.488 (6), 3.615 (6) and 3.443 (5) Å in the four compounds, respectively. The torsion angle of the non-bonded 'interaction' described above (C16—C17···C15—C10) is another measure of the proximity of the atoms involved in the ring closure upon irradiation. The torsion angles observed in the four compounds are −84.3 (3), 68.2 (6), −65.8 (6) and 65.0 (3)°, respectively. It is interesting to note that the torsion angle at the corresponding bond formed after the ring closure was found to be 47.5 (6)° in the structure of the compound obtained by irradiation of (E)-2-isopropylidene-3-[1-(2-methyl-5-phenyl-3-thienyl)ethylidene]succinic anhydride
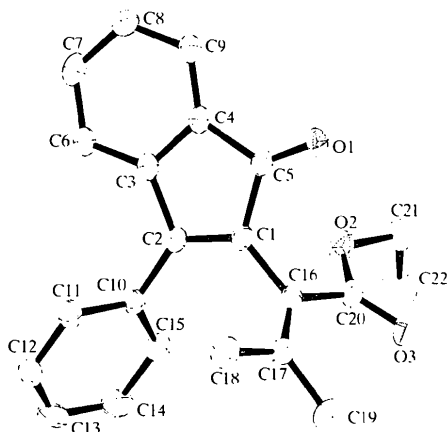
(Kaftory, 1984; CICXAN in the CSD). It is therefore expected that the ring closure of (I) will involve increased atomic motions in comparison with this compound.

Experimental

Crystal data

$C_{22}H_{20}O_3$	Mo $K\alpha$ radiation
$M_r = 332.38$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25
$Pna2_1$	reflections
$a = 7.393 (2)$ Å	$\theta = 13.0–24.0°$
$b = 13.069 (4)$ Å	$\mu = 0.084$ mm^{-1}
$c = 17.855 (5)$ Å	$T = 120 (2)$ K
$V = 1725.1 (9)$ Å^3	Plate
$Z = 4$	$0.30 \times 0.19 \times 0.10$ mm
$D_x = 1.280$ Mg m^{-3}	Yellow–orange
D_m not measured	

Data collection

Rigaku AFC-5R diffractometer	$R_{int} = 0.020$
	$\theta_{max} = 27.50°$
$\omega/2\theta$ scans	$h = -9 \rightarrow 0$
Absorption correction: none	$k = -16 \rightarrow 10$
4028 measured reflections	$l = -23 \rightarrow 23$
3950 independent reflections	3 standard reflections
(including Friedel pairs)	every 200 reflections
2960 reflections with	intensity decay: none
$I > 2\sigma(I)$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = -0.007$
$R[F^2 > 2\sigma(F^2)] = 0.063$	$\Delta\rho_{max} = 0.253$ e Å^{-3}
$wR(F^2) = 0.171$	$\Delta\rho_{min} = -0.191$ e Å^{-3}
$S = 1.069$	Extinction correction: none
3942 reflections	Scattering factors from
306 parameters	International Tables for
All H atoms refined	Crystallography (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2$	
$+ 0.6212P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structure Corporation, 1988). Cell refinement: MSC/AFC Diffractometer Control Software. Data reduction: MSC/AFC Diffractometer Control Software. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976).

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level

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Comment

1,2,4-Triazoles are very useful ligands in coordination chemistry. A series of coordination compounds containing substituted 1,2,4-triazole ligands which can form five-membered chelate rings with metal ions have been studied (Bencini *et al.*, 1985, 1987; van Koningsbruggen *et al.*, 1995). We have recently synthesized the title compound, (I), which can act as a doubly-bidentate chelating ligand and its crystal structure is reported herein.

(I)
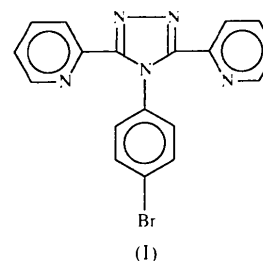
The dihedral angles that the two pyridyl rings make with the 1,2,4-triazole ring are 46.8 (2) and 28.1 (2)°, while that of the phenyl ring is 53.8 (2)°. The molecular planes perpendicular to the *b* axis are about 3.5 Å apart [$N3 \cdots N5(\frac{3}{2} - x, \frac{1}{2} + y, z)$ 3.510 (6) Å].
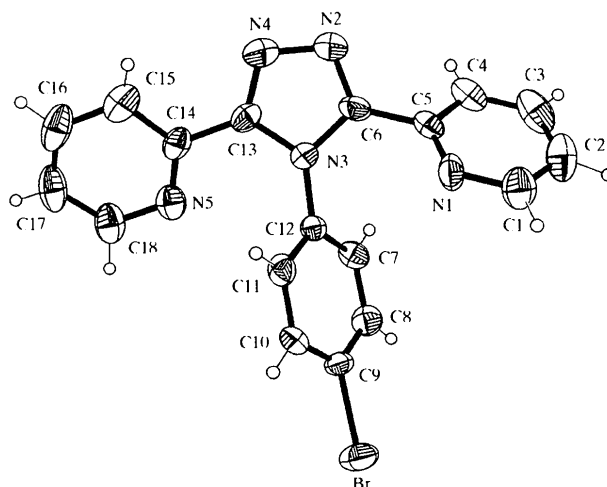
Fig. 1. *ORTEP*II (Johnson, 1976) plot at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

4-(p-Bromophenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole

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Abstract

The molecule of the title compound, $C_{18}H_{12}BrN_5$, is basically planar except that the phenyl and pyridyl rings are tilted in a propeller manner with respect to the central five-membered 1,2,4-triazole ring.

Experimental

The title compound was obtained by the reaction of equivalent amounts of 4,4'-dibromophenylphosphazoanilide and *N,N'*-dipyridoylhydrazine in *o*-dichlorobenzene at 463–473 K for 3 h (Grimmel *et al.*, 1946; Klingsberg, 1958). Single crystals suitable for X-ray diffraction were recrystallized from ethanol.

Crystal data

$C_{18}H_{12}BrN_5$	Mo *Kα* radiation
$M_r = 378.24$	$\lambda = 0.71073$ Å