O2	0.0481 (2)	0.0174 (2)	0.1843 (2)	0.0511 (8)
O3	0.0263 (2)	−0.2122 (2)	0.1783 (2)	0.0399 (7)
O4	−0.2757 (2)	−0.3778 (2)	0.0956 (2)	0.0410 (7)
O5	−0.2667 (2)	−0.2855 (2)	0.2993 (2)	0.0486 (8)
N1	−0.1284 (2)	−0.0956 (2)	0.2309 (2)	0.0346 (8)
C1	−0.1787 (2)	−0.1864 (3)	0.1277 (3)	0.0285 (9)
C2	−0.0756 (3)	−0.2343 (3)	0.0694 (3)	0.0348 (11)
C3	−0.1959 (3)	0.0036 (3)	0.2812 (3)	0.0375 (11)
C4	−0.2436 (2)	−0.2880 (3)	0.1876 (3)	0.0290 (8)
C5	−0.3348 (4)	−0.4851 (4)	0.1389 (5)	0.0598 (17)
C11	−0.2762 (2)	0.0724 (3)	0.1673 (3)	0.0335 (9)
C12	−0.3919 (3)	0.0855 (4)	0.1718 (4)	0.0524 (12)
C13	−0.4664 (4)	0.1513 (5)	0.0716 (5)	0.0705 (17)
C14	−0.4268 (4)	0.2010 (4)	−0.0374 (5)	0.0627 (15)
C15	−0.3120 (4)	0.1886 (3)	−0.0440 (4)	0.0584 (14)
C16	−0.2369 (3)	0.1250 (3)	0.0592 (4)	0.0473 (12)

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

S1—O1	1.416 (2)	C1—C2	1.532 (4)
S1—O2	1.416 (2)	C1—C4	1.513 (3)
S1—O3	1.565 (2)	C3—C11	1.508 (3)
S1—N1	1.612 (2)	C11—C12	1.377 (4)
O3—C2	1.461 (3)	C11—C16	1.370 (4)
O4—C4	1.323 (3)	C12—C13	1.376 (5)
O4—C5	1.447 (4)	C13—C14	1.368 (6)
O5—C4	1.194 (3)	C14—C15	1.371 (6)
N1—C1	1.445 (3)	C15—C16	1.386 (4)
N1—C3	1.468 (4)		
O3—S1—N1	95.2 (1)	O3—C2—C1	105.1 (2)
O2—S1—N1	112.6 (1)	N1—C3—C11	113.2 (2)
O2—S1—O3	110.9 (1)	O5—C4—C1	125.2 (3)
O1—S1—N1	111.9 (1)	O4—C4—C1	110.0 (2)
O1—S1—O3	108.2 (1)	O4—C4—O5	124.8 (3)
O1—S1—O2	116.0 (2)	C3—C11—C16	121.7 (3)
S1—O3—C2	109.6 (2)	C3—C11—C12	119.8 (3)
C4—O4—C5	116.9 (3)	C12—C11—C16	118.4 (3)
S1—N1—C3	120.8 (2)	C11—C12—C13	121.1 (4)
S1—N1—C1	113.6 (2)	C12—C13—C14	120.0 (4)
C1—N1—C3	123.4 (2)	C13—C14—C15	119.8 (4)
N1—C1—C4	111.5 (2)	C14—C15—C16	119.9 (4)
N1—C1—C2	104.4 (2)	C11—C16—C15	120.8 (4)
C2—C1—C4	114.3 (2)		

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (Frenz, 1983). Program(s) used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTON* (Spek, 1991). Software used to prepare material for publication: *PARST* (Nardelli, 1992).

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3-Phenyl-1-(2,4,6-triphenylphenyl)propane-1,3-dione

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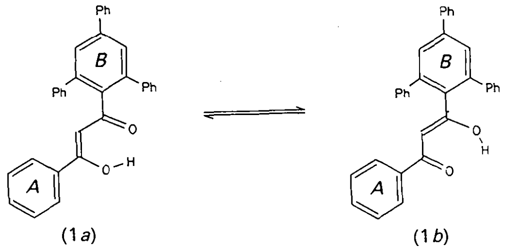
Abstract

The title compound, $C_{33}H_{24}O_2$, consists of well separated $C_{33}H_{24}O_2$ units. The bulky 1,3-diketone occurs in its enol form (as suggested by spectroscopic studies) with the hydroxy group in the neighbourhood of the unsubstituted phenyl ring. C—O bond distances

of 1.254 (3) and 1.327 (3) Å have been found. The bulky 2,4,6-triphenyl-substituted phenyl ring is bent away from the rest of the molecule which is almost planar.

Comment

Recently, a new and facile approach to substituted propane-1,3-diones from 2,4,6-triarylpyrylium salts and 1,5-diphenylpentane-1,3,5-trione was reported (Zimmermann, 1993). From IR and NMR spectroscopic studies a complete enolization of the 1,3-diketones obtained is evident. In solution, a rapid equilibrium between the two distinct enol forms (1*a*) and (1*b*) is suggested (Zimmermann, 1993), although (1*a*) should be preferred because of the formation of a conjugated π-system between the enol double bond and the neighbouring phenyl ring. This is prevented in (1*b*) by the presence of the three phenyl substituents which cause distortion of the C3—C31 bond.



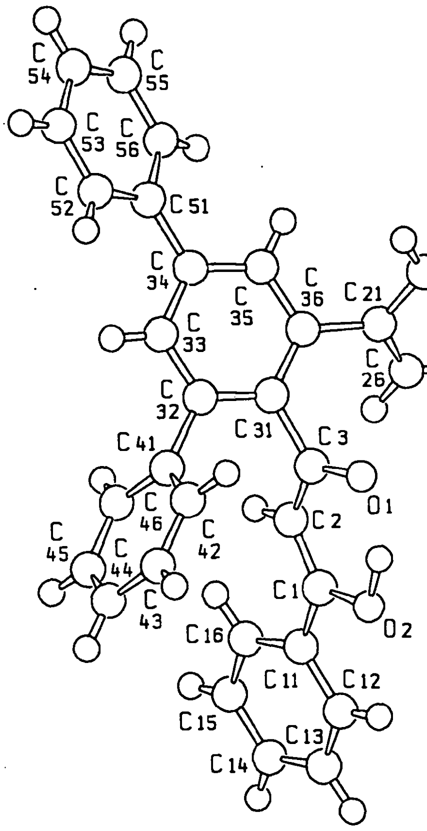


Fig. 1. *SCHAKAL* (Keller, 1989) plot of $C_{33}H_{24}O_2$ along with the atomic numbering scheme.

The spectroscopic results can be confirmed by the structure determination of 3-phenyl-1-(2,4,6-triphenylphenyl)propane-1,3-dione. A *SCHAKAL* plot (Keller, 1989) is shown in Fig. 1. Two distinct C—O bond distances can be observed [C1—O2 1.327 (3) Å and C3—O1 1.254 (3) Å] and the C1—C2 distance is shorter by 0.066 Å than the C2—C3 bond. The C1, C2, C3, O1 and O2 atoms together with phenyl ring *A* are coplanar within 0.068 (2) Å. Phenyl ring *B* is bent out of this least-square plane by 67.36 (7)°; this is easily understood when the sterical requirements of the three substituent phenyl groups are considered. The distortion explains the hindrance of π-electron delocalization from the enol bond and the preference of the equilibrium for structure (1*a*).

Experimental

Crystal data

$C_{33}H_{24}O_2$
$M_r = 452.56$
Triclinic
$P\bar{1}$
$a = 10.609\ (1)$ Å
$b = 10.865\ (1)$ Å
$c = 11.187\ (1)$ Å
$\alpha = 97.308\ (7)°$
$\beta = 94.774\ (7)°$
$\gamma = 103.166\ (6)°$
$V = 1237.0\ (2)$ Å^3
$Z = 2$

$D_x = 1.215$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 17\text{-}22°$
$\mu = 0.580$ mm^{-1}
$T = 295\ (1)$ K
Plate
0.3 × 0.25 × 0.05 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
5608 measured reflections
4278 independent reflections
2989 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.023$
$\theta_{\rm max} = 69°$
$h = -2 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$
3 standard reflections (051, 342, 421)
frequency: 200 min
intensity variation: 1.9%

Refinement

Refinement on F^2
$R(F) = 0.049$
$wR(F^2) = 0.1265$
$S = 1.041$
4278 reflections
413 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.2901P]$
where $P = (F_o^2 + 2F_c^2)/3$

$\Delta\rho_{\rm max} = 0.183$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.147$ e Å^{-3}
Extinction correction: *SHELXL* (Sheldrick, 1994)
Extinction coefficient: 0.0085 (9)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O(1)	0.2981 (2)	0.6408 (2)	0.8687 (2)	0.074 (1)
O(2)	0.4848 (2)	0.5308 (2)	0.8697 (2)	0.086 (1)
C(1)	0.5607 (2)	0.6403 (2)	0.8512 (2)	0.062 (1)
C(2)	0.5101 (2)	0.7421 (2)	0.8373 (2)	0.063 (1)
C(3)	0.3754 (2)	0.7363 (2)	0.8417 (2)	0.060 (1)
C(11)	0.6987 (2)	0.6384 (2)	0.8466 (2)	0.067 (1)
C(12)	0.7418 (3)	0.5332 (3)	0.8775 (3)	0.086 (1)
C(13)	0.8724 (4)	0.5328 (4)	0.8752 (3)	0.107 (1)
C(14)	0.9583 (4)	0.6338 (4)	0.8437 (3)	0.109 (1)
C(15)	0.9165 (3)	0.7364 (4)	0.8117 (3)	0.101 (4)
C(16)	0.7861 (3)	0.7391 (3)	0.8131 (3)	0.082 (1)
C(21)	0.2757 (2)	0.9040 (2)	1.0250 (2)	0.060 (1)
C(22)	0.1651 (3)	0.9046 (2)	1.0833 (2)	0.074 (1)
C(23)	0.1642 (3)	0.8829 (3)	1.2026 (3)	0.088 (1)
C(24)	0.2712 (4)	0.8601 (3)	1.2643 (3)	0.090 (1)
C(25)	0.3824 (3)	0.8613 (3)	1.2085 (2)	0.083 (1)
C(26)	0.3841 (3)	0.8833 (2)	1.0900 (2)	0.069 (1)
C(31)	0.3233 (2)	0.8465 (2)	0.8099 (2)	0.057 (1)
C(32)	0.3214 (2)	0.8690 (2)	0.6893 (2)	0.060 (1)
C(33)	0.2742 (2)	0.9700 (2)	0.6558 (2)	0.062 (1)
C(34)	0.2276 (2)	1.0501 (2)	0.7393 (2)	0.060 (1)
C(35)	0.2294 (2)	1.0252 (2)	0.8584 (2)	0.061 (1)
C(36)	0.2758 (2)	0.9252 (2)	0.8959 (2)	0.057 (1)
C(41)	0.3653 (2)	0.7819 (2)	0.5957 (2)	0.061 (1)
C(42)	0.2911 (3)	0.6598 (2)	0.5566 (2)	0.080 (1)
C(43)	0.3312 (4)	0.5780 (3)	0.4711 (3)	0.096 (1)
C(44)	0.4473 (4)	0.6178 (3)	0.4258 (3)	0.091 (1)
C(45)	0.5213 (3)	0.7377 (3)	0.4634 (2)	0.086 (1)
C(46)	0.4813 (3)	0.8213 (3)	0.5475 (2)	0.076 (1)
C(51)	0.1747 (2)	1.1566 (2)	0.7018 (2)	0.064 (1)
C(52)	0.1117 (3)	1.1466 (3)	0.5865 (3)	0.078 (1)
C(53)	0.0557 (3)	1.2426 (3)	0.5523 (3)	0.095 (1)
C(54)	0.0638 (3)	1.3504 (3)	0.6347 (4)	0.100 (1)
C(55)	0.1269 (3)	1.3626 (3)	0.7480 (4)	0.097 (1)
C(56)	0.1830 (3)	1.2677 (2)	0.7821 (3)	0.082 (1)

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

C(1)—C(2)	1.355 (3)	C(3)—C(31)	1.498 (3)
C(2)—C(3)	1.421 (3)	C(1)—O(2)	1.327 (3)
C(1)—C(11)	1.474 (3)	C(3)—O(1)	1.254 (3)
C(11)—C(1)—C(2)	125.1 (2)	C(1)—C(2)—C(3)	121.9 (2)
C(11)—C(1)—O(2)	114.4 (2)	C(2)—C(3)—O(1)	122.2 (2)
O(2)—C(1)—C(2)	120.5 (2)	C(31)—C(3)—O(1)	118.9 (2)

Programs used: *SHELXS*86 (Sheldrick, 1985) and *SHELXL* (Sheldrick, 1994).

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Methyl 3-(1,3-Benzothiazol-2-yldithio)-propanoate, $C_{11}H_{11}NO_2S_3$

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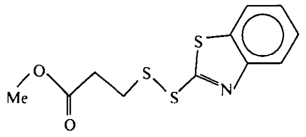
Abstract

The benzothiazole unit is similar to those found in other derivatives, *e.g.* bis(1,3-benzothiazol-2-yl) disulfide [Zingaro & Meyers (1980). *Cryst. Struct. Commun.* **9**, 1167–1172] or 2-methylthio-1,3-benzothiazole [Wheatley (1962). *J. Chem. Soc.* p. 3636]. The S atoms of the disulfide group and the condensed ring system are nearly coplanar (torsion angle τ_{NCSS} + 9.3°). The overall conformation of the title compound is almost linear.

Comment

3-(1,3-Benzothiazol-2-yldithio)propanoic acid methyl ester (1) may be prepared by condensation of 2-mercaptobenzothiazole with 2-(methoxycarbonyl)ethyl sulfenic acid, $HOS(CH_2)_2CO_2CH_3$ (Bachi, Gross & Frolow, 1982).



(1)

The structure determination of (1) was undertaken in order to analyse whether there is any interaction between the carboxyl group and the disulfide and benzothiazole units. This interaction is of interest with respect to the reactivity of (1) as an accelerator in rubber vulcanization. However, no such inter- or intramolecular interaction of the carboxyl group was found. The molecule of (1) adopts a stretched conformation. Most bond distances and valence angles are comparable to those of other benzothiazole derivatives. The ring system is planar and the two S atoms linked to the ring system are nearly coplanar with the ring plane, as with bis(1,3-benzothiazol-2-yl)

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