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(2*RS*,3*RS*)-2,3-Dimethyl-1,4-bis(3,4,5-trimethoxyphenyl)butane-1,4-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.120
Data-to-parameter ratio = 15.8

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

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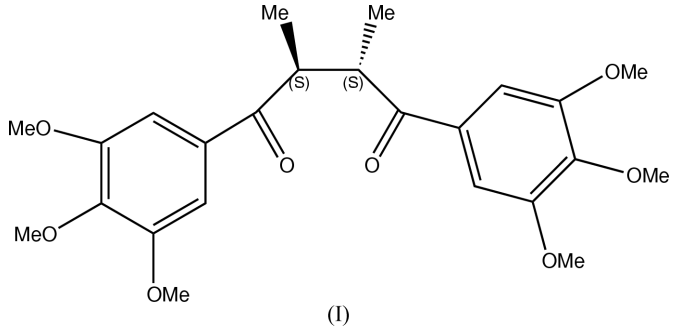
The molecule of the title compound, $C_{24}H_{30}O_8$, has two chiral centers. As it occupies a special position on the twofold axis in a centrosymmetric group, the crystal represents a racemic mixture of *RR* and *SS* enantiomers.

Comment

Diaryl-substituted heterocycles have recently attracted considerable attention (Khanna *et al.*, 1997; Penning *et al.*, 1997; Wu-wong *et al.*, 2001), due to their high biological activities (Portevin *et al.*, 2000; Szczepankiewicz *et al.*, 2001). Diaryl-substituted butane-1,4-diones are key intermediates for the synthesis of diaryl-substituted heterocycles. In view of this, we have recently focused on the preparation of diaryl-substituted butane-1,4-diones. As a part of this work, we have synthesized the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule has two chiral centers. As it occupies a special position on the twofold axis in a centrosymmetric group, the crystal

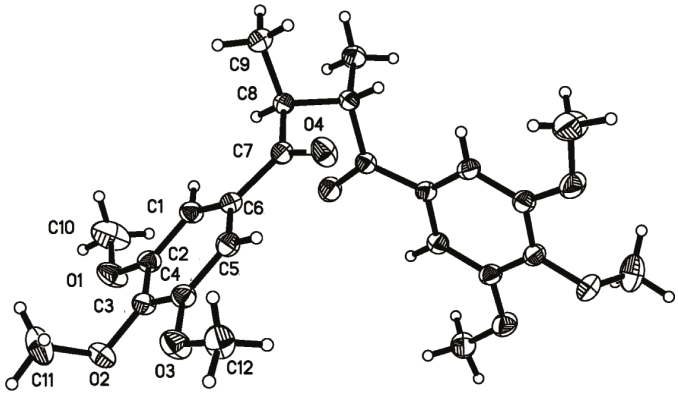


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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represents a racemic mixture of *RR* and *SS* enantiomers. The torsion angles O4—C7—C8—C8^{i}, C6—C7—C8—C8^{i} and C7—C8—C8^{i}—C7^{i} [symmetry code: (i) $\frac{3}{2}-x$, $\frac{1}{2}-y$, z] are 47.1 (2), −133.61 (18) and 58.34 (18)°, respectively.

Experimental

The title compound was synthesized (Perry *et al.*, 1972; Biftu *et al.*, 1979) as follows: $FeCl_3$ (44 mg) and Na (492 mg) were added to liquid ammonia (about 60 ml) and the mixture was stirred for about 30 min. A solution of 1-(3,4,5-trimethoxyphenyl)propan-1-one (4.0 g) in THF (20 ml) was then added slowly over a period of 30 min. After another 15 min, a solution of 2-bromo-1-(3,4,5-trimethoxyphenyl)propan-1-one (5.9 g) in THF (40 ml) was added dropwise over a period of 1 h. Stirring was continued for 30 min and NH_4Cl (4 g) was added to quench the reaction. The ammonia was allowed to evaporate and the mixture was filtered and washed with THF. The combined filtrate was concentrated and the residue was crystallized from methanol to give the product (6.4 g, 80% yield). Single crystals of the title compound, suitable for X-ray diffraction study, were obtained by slow evaporation of an ethyl acetate solution (m.p. 448–449 K).

Crystal data

$C_{24}H_{30}O_8$
M_r = 446.48
Orthorhombic, *Pccn*
a = 12.785 (4) Å
b = 14.470 (5) Å
c = 12.488 (4) Å
V = 2310.3 (13) Å^3
Z = 4
D_x = 1.284 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 804 reflections
θ = 2.7–24.0°
μ = 0.10 mm^{-1}
T = 293 (2) K
Prism, colorless
0.36 × 0.32 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.943, T_{max} = 0.973
12482 measured reflections
2355 independent reflections
1593 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.4°
$h = -15 \rightarrow 15$
$k = -18 \rightarrow 7$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.120
S = 1.03
2355 reflections
149 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.9316P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and included in the refinement in the riding-model approximation, with $U_{iso} = 1.2U_{eq}$ of the carrier atom ($U_{iso} = 1.5U_{eq}$ for methyl H atoms).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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