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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.060
 wR factor = 0.146
Data-to-parameter ratio = 8.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Rotenone–acetic acid (2/1)

The structure determination of the title compound, a 2:1
adduct of rotenone and acetic acid, $2\text{C}_{23}\text{H}_{22}\text{O}_6 \cdot \text{C}_2\text{H}_4\text{O}_2$,
confirms that the rotenone molecule has an approximately
V-shaped structure.

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Comment

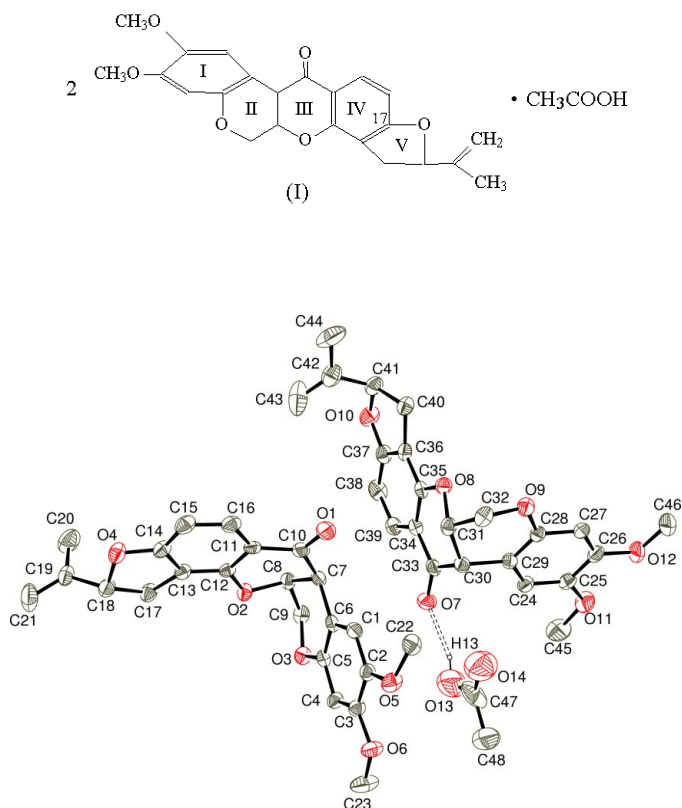
Rotenone is a naturally occurring heterocyclic compound
widely used as an insecticide. It exerts its effects mainly by
blocking oxidative phosphorylation and/or mitosis in cells
through apparently separate pathways (Loffler & Schneider,
1982). Information on the mode of action and selectivity of
rotenone is important so that the compound may be used
safely and efficiently. The three-dimensional structure of most
biologically active molecules plays a role in governing their
interactions and activities. Thus, in the course of a systematic
study of the relation between the structure and bioactivity of
rotenone, we have isolated the 2:1 adduct of rotenone and
acetic acid, (I), and report here its preparation and structure.

Figure 1

A view of (I), showing the atom-numbering scheme and the O—H...O
hydrogen bonding (as a dashed line) between one of the rotenone
molecules and the acetic acid. Displacement ellipsoids are drawn at the
50% probability level. H atoms have been omitted for clarity.

The X-ray diffraction analysis of (I) shows that there are two rotenone molecules and one acetic acid molecule in the asymmetric unit, as shown in Fig. 1. The distances and angles within the two rotenone molecules agree with those of related molecules (Begley *et al.*, 1989, 1993).

The two rotenone molecules in (I) are very similar, with two nearly flat regions (rings I and II, and rings III, IV and V). In each rotenone molecule, ring II can be regarded as having an envelope conformation, whereas ring III adopts a 1,2-diplanar conformation (Bucourt, 1974), as indicated by the torsion angles (Table 1). The torsion angles in the five-membered ring V (Table 1) indicate an envelope conformation (Bucourt, 1974), with the isopropenyl group equatorial, as proposed earlier (Büchi *et al.*, 1961; Carlson *et al.*, 1973).

Except for the isopropenyl group, the non-H atoms of (I) lie close to the plane of aromatic rings I and IV, resulting in an approximately V-shaped molecule, with dihedral angles of 74.7 (1) and 74.8 (1)° between rings I and IV in the two molecules.

The acetic acid molecule is linked through O—H...O hydrogen bonding to one of the rotenone molecules (Table 2). The packing of the molecules is governed by weak van der Waals interactions.

Experimental

Rotenone (5 g) in ethanoic acid (50 ml) was refluxed for 2 h. After cooling and filtration, the solution was allowed to stand in air at room temperature for two months, after which time a single crystal was obtained suitable for X-ray analysis.

Crystal data

$2C_{23}H_{22}O_6 \cdot C_2H_4O_2$	$D_x = 1.334 \text{ Mg m}^{-3}$
$M_r = 848.86$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 2045 reflections
$a = 9.1356 (16) \text{ \AA}$	$\theta = 5.1\text{--}39.9^\circ$
$b = 15.359 (3) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 15.082 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 92.652 (4)^\circ$	Block, colourless
$V = 2113.9 (7) \text{ \AA}^3$	$0.51 \times 0.20 \times 0.06 \text{ mm}$
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	5099 independent reflections
φ and ω scans	3945 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$R_{\text{int}} = 0.052$
$T_{\text{min}} = 0.952$, $T_{\text{max}} = 0.994$	$\theta_{\text{max}} = 28.3^\circ$
12 858 measured reflections	$h = -11 \rightarrow 12$
	$k = -20 \rightarrow 10$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2]$
$wR(F^2) = 0.146$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{\text{max}} = 0.009$
5099 reflections	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
566 parameters	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$

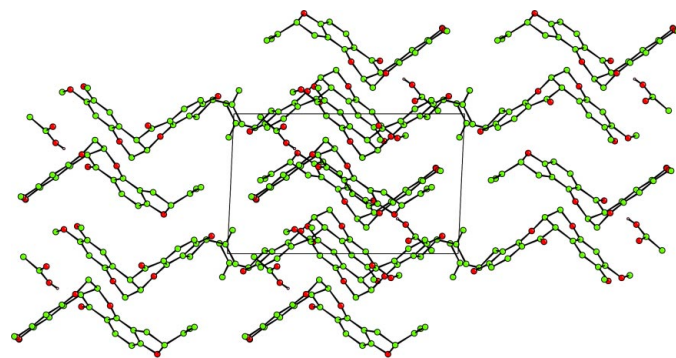


Figure 2
A view, down the b axis, of the molecular packing in (I).

Table 1
Selected torsion angles ($^\circ$).

O3—C5—C6—C7	−0.6 (6)	C32—O9—C28—C29	15.2 (6)
C5—C6—C7—C8	13.9 (6)	O8—C30—C31—C33	50.1 (5)
C6—C7—C8—C9	−41.7 (5)	C30—C31—C33—C34	−152.5 (5)
C7—C8—C9—O3	59.9 (5)	C31—C33—C34—C35	9.9 (4)
C8—C9—O3—C5	−46.6 (5)	C33—C34—C35—O8	1.7 (6)
C9—O3—C5—C6	17.1 (6)	C34—C35—O8—C30	2.1 (4)
O2—C8—C7—C10	−49.9 (5)	C35—O8—C30—C31	−139.4 (4)
C8—C7—C10—C11	27.0 (5)	O4—C13—C14—C17	−0.9 (6)
C7—C10—C11—C12	−0.4 (6)	C13—C14—C17—C18	167.0 (5)
C10—C11—C12—O2	−4.9 (6)	C14—C7—C18—O4	0.9 (2)
C11—C12—O2—C8	−18.7 (5)	C7—C18—O4—C13	5.85 (13)
C12—O2—C8—C7	46.5 (5)	C18—O4—C13—C14	−168.3 (5)
O9—C28—C29—C30	−0.6 (7)	O10—C36—C37—C40	1.0 (6)
C28—C29—C30—C31	15.1 (6)	C36—C37—C38—C39	−0.4 (8)
C29—C30—C31—C32	−42.4 (5)	C37—C38—C39—O10	0.6 (4)
C30—C31—C32—O9	58.0 (5)	C38—C39—O10—C36	−179.4 (8)
C31—C32—O9—C28	−43.5 (5)	C39—O10—C36—C37	0.7 (4)

Table 2
Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O13—H13...O7	0.82	2.01	2.776 (7)	155

Owing to the lack of atoms heavier than O, the absolute configuration of (I) could not be determined by X-ray analysis and the Friedel pairs were merged. The configuration was then assigned on the basis of the configuration of the starting rotenone (Rossi *et al.*, 1988). All H atoms were positioned geometrically and refined using a riding model.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998) and SHELXTL (Sheldrick, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and CAMERON (Watkin *et al.*, 1993); software used to prepare material for publication: SHELXTL.

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