

C4	0.2059 (5)	-0.1765 (5)	0.1925 (3)	0.0699 (11)
C5	0.1961 (5)	-0.3039 (5)	0.2821 (3)	0.0780 (12)
C6	0.1376 (6)	-0.2443 (6)	0.4548 (3)	0.0871 (13)
O7	0.0708 (2)	0.1153 (3)	0.3695 (2)	0.0555 (6)
C8	0.1922 (3)	0.1776 (4)	0.1806 (2)	0.0441 (7)
N9	0.3044 (3)	0.2517 (3)	0.2236 (2)	0.0492 (7)
O10	0.3667 (2)	0.1507 (3)	0.3065 (2)	0.0572 (6)
C11	0.1243 (4)	0.2791 (5)	0.0882 (2)	0.0527 (8)
F12	0.0112 (3)	0.3654 (5)	0.0996 (2)	0.1152 (12)
F13	0.2047 (3)	0.4091 (4)	0.0577 (2)	0.0936 (9)
F14	0.0942 (4)	0.1644 (4)	0.0121 (2)	0.1173 (12)

Table 2. Selected geometric parameters (Å, °)

N1—C2	1.327 (4)	C4—C5	1.534 (5)
N1—C5	1.450 (4)	C8—N9	1.259 (4)
N1—C6	1.462 (4)	C8—C11	1.500 (4)
C2—O7	1.222 (4)	N9—O10	1.386 (3)
C2—C3	1.527 (4)	C11—F12	1.288 (4)
C3—C8	1.508 (4)	C11—F14	1.311 (4)
C3—C4	1.515 (5)	C11—F13	1.321 (4)
C2—N1—C5	114.7 (3)	N9—C8—C11	114.0 (3)
C2—N1—C6	123.8 (3)	N9—C8—C3	127.4 (3)
C5—N1—C6	121.3 (3)	C11—C8—C3	118.6 (3)
O7—C2—N1	124.6 (3)	C8—N9—O10	112.4 (2)
O7—C2—C3	126.8 (3)	F12—C11—F14	108.2 (4)
N1—C2—C3	108.6 (3)	F12—C11—F13	105.2 (3)
C8—C3—C4	113.7 (3)	F14—C11—F13	104.7 (3)
C8—C3—C2	113.4 (2)	F12—C11—C8	113.0 (3)
C4—C3—C2	104.3 (3)	F14—C11—C8	111.4 (3)
C3—C4—C5	105.3 (3)	F13—C11—C8	113.7 (3)
N1—C5—C4	104.0 (3)		
C5—N1—C2—C3	2.2 (4)	C2—N1—C5—C4	8.8 (5)
N1—C2—C3—C4	-12.4 (3)	C3—C4—C5—N1	-15.9 (4)
C2—C3—C4—C5	17.0 (4)		

The relatively high *R* values are probably the result of poor crystal quality. The shapes of the C6, O7 and, especially, F12 and F13 displacement ellipsoids (Fig. 1) indicate high thermal agitation and unresolved disorder, which may contribute to the high *R* values. The positions of H3 and H10 were determined from a  $\Delta\rho$  map; their site coordinates were refined. All other H atoms were located in calculated positions and refined riding on the attached C atom. A common isotropic displacement parameter for all H atoms was refined [ $U = 0.121 (7) \text{ \AA}^2$ ].

Data collection: local program. Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1223). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Aversin

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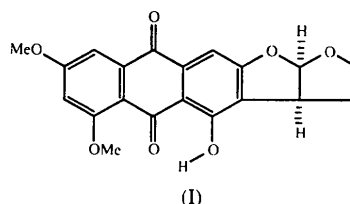
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## Abstract

The X-ray crystal structure analysis of the mould metabolite (3*aS-cis*)-2,3,3*a*,12*a*-tetrahydro-4-hydroxy-6,8-dimethoxyanthra[2,3-*b*]furo[3,2-*d*]furan-5,10-dione, which contains two C<sub>20</sub>H<sub>16</sub>O<sub>7</sub> molecules per asymmetric unit in *P*2<sub>1</sub>, reveals that the molecule contains five fused rings: a planar section of three six-membered rings and one five-membered ring, with a final *cis*-fused five-membered ring. The OH group in each molecule is involved in an intramolecular hydrogen bond [O···O 2.518 (6) and 2.547 (6) Å].

## Comment

Aversin, (I), is a metabolite isolated from a variant strain of *Aspergillus versicolor*. The structure derived previously from spectroscopic data (Bullock, Kirkaldy, Roberts & Underwood, 1963) has now been confirmed, except for the interchange of the OH and an OMe group. The two molecules in the asymmetric unit (Fig. 1) have virtually identical geometries and dimensions. The three six-membered rings of the anthraquinone skeleton and the attached five-membered ring are almost coplanar, with the final *cis*-fused five-membered ring displaced from this plane. This final five-membered ring has an envelope conformation, with C17 and C37 displaced from the respective plane of the other four atoms.



Bond lengths and angles are normal (Table 2) and the OH group is involved in an intramolecular hydrogen bond. The most significant intermolecular contacts are between OH groups, but the distances [ $O \cdots O = 3.006(4)$ ,  $H \cdots O = 2.6 \text{ \AA}$ ] and geometries suggest that these represent van der Waals interactions rather than bifurcated hydrogen bonds.

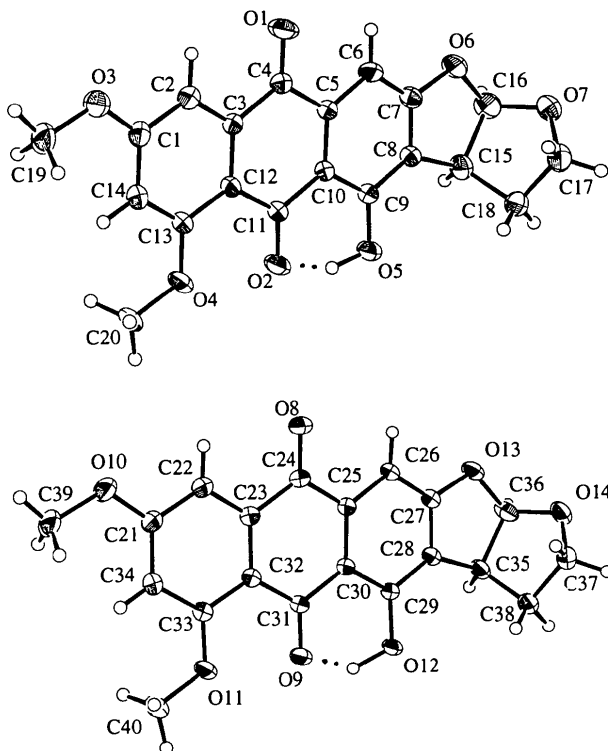


Fig. 1. View of the two molecules of aversin with 33% displacement ellipsoids.

## Experimental

Crystals from the mould were isolated more than 30 years ago (Bullock *et al.*, 1963) and were described as 'golden needles'. The problem appeared too difficult for X-ray analysis at that time and the structure analysis has been carried out only recently. The crystals are distinctly reddish in color, so that some decomposition has occurred over the 30 year storage period.

### Crystal data

$C_{20}H_{16}O_7$   
 $M_r = 368.34$   
 Monoclinic  
 $P2_1$   
 $a = 5.166(2) \text{ \AA}$   
 $b = 20.282(2) \text{ \AA}$   
 $c = 15.513(3) \text{ \AA}$   
 $\beta = 96.41(3)^\circ$

Cu  $K\alpha$  radiation  
 $\lambda = 1.5418 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 50.3\text{--}55.4^\circ$   
 $\mu = 0.99 \text{ mm}^{-1}$   
 $T = 294 \text{ K}$   
 Prism

$V = 1615.2(5) \text{ \AA}^3$   
 $Z = 4$  (2 molecules per asymmetric unit)  
 $D_x = 1.515 \text{ Mg m}^{-3}$

$0.40 \times 0.20 \times 0.20 \text{ mm}$   
 Red

### Data collection

AFC-6S diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction: none  
 3835 measured reflections  
 3432 independent reflections  
 2812 observed reflections [ $I > 3\sigma(I)$ ]  
 $R_{int} = 0.06$

$\theta_{max} = 77.6^\circ$   
 $h = 0 \rightarrow 6$   
 $k = 0 \rightarrow 25$   
 $l = -19 \rightarrow 19$   
 3 standard reflections monitored every 200 reflections  
 intensity decay: 0.6%

### Refinement

Refinement on  $F$   
 $R = 0.063$   
 $wR = 0.079$   
 $S = 1.47$   
 2812 reflections  
 487 parameters  
 H-atom parameters not refined  
 $w = 1/\sigma^2(I)$   
 $(\Delta/\sigma)_{max} = 0.01$

$\Delta\rho_{max} = 0.27 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.39 \text{ e \AA}^{-3}$   
 Extinction correction: Zachariasen (1967) type 2, Gaussian isotropic  
 Extinction coefficient:  $59(14) \times 10^{-7}$   
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^*$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
O1	0.7329 (8)	0.1713	0.9625 (3)	0.0663
O2	0.4005 (8)	0.0313 (3)	0.6774 (2)	0.0585
O3	0.0067 (8)	0.3037 (3)	0.8282 (3)	0.0656
O4	0.0395 (8)	0.1182 (3)	0.6368 (2)	0.0606
O5	0.7489 (7)	-0.0560 (3)	0.7154 (2)	0.0532
O6	1.3240 (7)	-0.0276 (3)	0.9638 (3)	0.0559
O7	1.3703 (8)	-0.1403 (3)	0.9813 (3)	0.0661
O8	-0.6185 (7)	0.1427 (3)	0.4190 (3)	0.0592
O9	-1.4549 (6)	0.2851 (3)	0.2950 (2)	0.0489
O10	-1.0661 (8)	-0.0024 (3)	0.1971 (3)	0.0614
O11	-1.5707 (7)	0.1979 (3)	0.1779 (2)	0.0558
O12	-1.3459 (6)	0.3741 (3)	0.4063 (2)	0.0430
O13	-0.6270 (6)	0.3471 (3)	0.6093 (2)	0.0508
O14	-0.5799 (6)	0.4612 (3)	0.6209 (2)	0.0559
C1	0.1050 (10)	0.2464 (3)	0.8031 (3)	0.0473
C2	0.3169 (10)	0.2214 (3)	0.8577 (3)	0.0474
C3	0.4327 (8)	0.1630 (3)	0.8369 (3)	0.0377
C4	0.6582 (9)	0.1403 (3)	0.8972 (3)	0.0430
C5	0.7867 (9)	0.0775 (3)	0.8774 (3)	0.0379
C6	0.9978 (9)	0.0567 (3)	0.9338 (3)	0.0457
C7	1.1131 (8)	-0.0012 (3)	0.9143 (3)	0.0432
C8	1.0319 (8)	-0.0390 (3)	0.8419 (3)	0.0388
C9	0.8237 (9)	-0.0182 (3)	0.7849 (3)	0.0384
C10	0.6966 (9)	0.0417 (3)	0.8020 (3)	0.0380
C11	0.4725 (9)	0.0644 (3)	0.7425 (3)	0.0403
C12	0.3429 (8)	0.1269 (3)	0.7614 (3)	0.0380
C13	0.1281 (9)	0.1525 (3)	0.7080 (3)	0.0444
C14	0.0085 (10)	0.2114 (3)	0.7294 (3)	0.0489
C15	1.1984 (10)	-0.1003 (3)	0.8429 (3)	0.0486
C16	1.3967 (10)	-0.0888 (4)	0.9246 (4)	0.0576
C17	1.1133 (12)	-0.1677 (4)	0.9613 (4)	0.0629
C18	1.0631 (11)	-0.1644 (3)	0.8645 (4)	0.0527
C19	-0.2094 (11)	0.3316 (4)	0.7759 (5)	0.0683
C20	-0.1722 (10)	0.1438 (3)	0.5805 (3)	0.0553

C21	-1.1249 (10)	0.0592 (3)	0.2225 (3)	0.0443
C22	-0.9520 (9)	0.0859 (3)	0.2898 (3)	0.0433
C23	-0.9982 (8)	0.1483 (3)	0.3198 (3)	0.0372
C24	-0.8114 (8)	0.1743 (3)	0.3916 (3)	0.0384
C25	-0.8684 (8)	0.2385 (3)	0.4302 (3)	0.0361
C26	-0.7041 (8)	0.2608 (3)	0.5022 (3)	0.0412
C27	-0.7666 (8)	0.3202 (3)	0.5387 (3)	0.0400
C28	-0.9780 (8)	0.3578 (3)	0.5070 (3)	0.0377
C29	-1.1424 (8)	0.3363 (3)	0.4360 (3)	0.0361
C30	-1.0878 (8)	0.2750 (3)	0.3962 (3)	0.0338
C31	-1.2652 (8)	0.2511 (3)	0.3228 (3)	0.0346
C32	-1.2133 (8)	0.1869 (3)	0.2847 (3)	0.0366
C33	-1.3752 (8)	0.1600 (3)	0.2143 (3)	0.0393
C34	-1.3336 (9)	0.0964 (3)	0.1845 (3)	0.0446
C35	-0.9866 (8)	0.4188 (3)	0.5616 (3)	0.0414
C36	-0.7459 (9)	0.4094 (3)	0.6290 (3)	0.0466
C37	-0.6359 (10)	0.4879 (3)	0.5346 (4)	0.0563
C38	-0.9278 (9)	0.4830 (3)	0.5157 (3)	0.0471
C39	-1.2475 (15)	-0.0328 (3)	0.1320 (4)	0.0692
C40	-1.7350 (11)	0.1734 (4)	0.1046 (4)	0.0575

Table 2. Selected bond lengths ( $\sigma \sim 0.006 \text{ \AA}$ ) and angles ( $\sigma \sim 0.4^\circ$ )

C=O	1.218, 1.221 (not hydrogen bonded)
	1.236, 1.236 (hydrogen bonded)
C—OH/OMe	1.339–1.354
C—OMe	1.420–1.438
C—C(=O)	1.462–1.487
C—C(aromatic)	1.367–1.418
C—C(5-ring)	1.496–1.555
C—O(5-ring)	1.357–1.382, 1.441–1.452
C—O—C(5-ring)	108.0–109.0
C—O—Me	116.7–119.2

Intramolecular hydrogen bonds (H atoms from difference map)

D—H...A	D—H	H...A	D...A	D—H...A
O5—H1...O2	0.99	1.65	2.547	147
O12—H12...O9	1.09	1.64	2.518	134

The structure was determined by direct methods. H atoms were placed in calculated positions, except those involved in the hydrogen bonds, which were positioned from difference syntheses. While the opposite configuration has marginally higher *R* factors (in the penultimate refinement round with a weighting scheme slightly different from that used in the final refinement cycles, *R* = 0.0651 versus 0.0649, *wR* = 0.0736 versus 0.0734), we hesitate to claim that we have established the absolute configuration. The data presented in this paper and Fig. 1 refer to the configuration with the lower pair of *R* values.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1985). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1990). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Hexaethyl (3,5-Di-*tert*-butyl-4-hydroxyphenyl)methanetrissphosphate at 173 K

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## Abstract

In contrast to the liquid phase, in the solid state three <sup>31</sup>P NMR signals were found for the title compound, C<sub>27</sub>H<sub>51</sub>O<sub>10</sub>P<sub>3</sub>. The nonequivalence of the signals can be attributed to a hydrogen bond. The molecules bonded *via* hydrogen bonds form chains.

## Comment

The hexaester (1) (Kuchar, Pasternak & Kirsanov, 1972) was the first *gem*-trissphosphate structure to be solved unambiguously. The structures of the aryl-methanetrissphosphate ester (2) (Gross, Ozegowski, & Costisella, 1990) and the unsubstituted methanetrissphosphate (3) (Gross, Costisella, Keitel & Ozegowski, 1993) were determined by <sup>13</sup>C NMR in solution. The methine C atoms of (2) and (3) showed quadruplets due to the coupling with the three equivalent P nuclei. The <sup>31</sup>P NMR spectra in CHCl<sub>3</sub> are very simple. Only one signal is observable for the equivalent P nuclei. In contrast, the <sup>31</sup>P solid-state NMR spectrum of (2) revealed three comparable signals. Two signals appear in the <sup>31</sup>P solid-state NMR spectrum of (4) (Costisella & Ramm, 1994). In the present paper we report the X-ray structure analysis of (2).