Acta Cryst. (1977). B33, 1276–1278

Colletotrichin Monohydrate Methanol Solvate

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(*Received* 22 *October* 1976; *accepted* 4 *December* 1976)

Abstract. $C_{28}H_{42}O_7.H_2O.CH_3OH$, FW 540·69; orthorhombic, $P2_12_12_1$, $a = 12{\cdot}138\ (2)$, $b = 21{\cdot}208\ (3)$, $c = 11{\cdot}817\ (2)$ Å, $Z = 4$, $V = 3042{\cdot}0\ (8)$ Å^3, $D_x = 1{\cdot}181$ g cm^{-3}; λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}90$ cm^{-1}. The structure was solved by the direct method and refined by block-diagonal least squares. The final R was 0·088 for 2464 reflexions. Each of the condensed six-membered rings is in the chair form.

Introduction. Colletotrichin is a toxic substance for higher plants isolated from *Colletotrichum nicotianae*, identical with acetylcolletotrichin isolated by Grove, Speake & Ward (1966). Prismatic crystals of the title compound were obtained from ethyl acetate–aqueous methanol–*n*-hexane solution. They are very unstable in air. A crystal 0·5 × 0·5 × 0·6 mm was sealed in a capillary tube for intensity measurement. Intensity data up to $2\theta < 55°$ were collected on a Rigaku four-circle diffractometer with Mo $K\alpha$ radiation monochromatized by graphite, an ω–2θ scan, and a scan

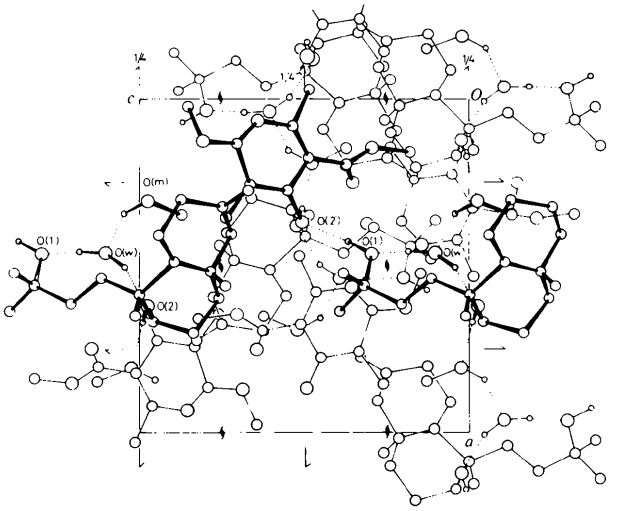

Fig. 1. The crystal structure projected along **b**, H atoms bonded to C atoms being excluded. The hydrogen bonds are represented by broken lines.

Table 1. *The positional parameters for the non-hydrogen atoms* (×10^4)

	x	*y*	*z*		*x*	*y*	*z*
C(1)	5988 (5)	4039 (3)	7602 (6)	C(2′)	1542 (6)	3646 (3)	7049 (5)
C(2)	7007 (5)	4082 (3)	8369 (6)	C(3′)	2576 (5)	3634 (3)	6659 (5)
C(3)	6689 (5)	4011 (3)	9597 (5)	C(4′)	2757 (5)	3423 (3)	5508 (5)
C(4)	5870 (5)	4516 (3)	9992 (5)	C(5′)	1761 (5)	3218 (3)	4887 (5)
C(5)	4880 (4)	4569 (3)	9146 (5)	C(6′)	785 (5)	3259 (3)	5366 (6)
C(6)	4092 (5)	5108 (3)	9426 (5)	C(7′)	1940 (6)	2945 (3)	3730 (7)
C(7)	3018 (5)	5048 (3)	8736 (5)	C(8′)	1597 (9)	3042 (4)	1793 (7)
C(8)	3231 (5)	4956 (3)	7486 (5)	C(9′)	−298 (6)	3046 (4)	4850 (7)
C(9)	4052 (5)	4445 (3)	7196 (5)	C(10′)	223 (7)	3749 (4)	8523 (8)
C(10)	5168 (5)	4568 (3)	7855 (5)	O(1)	4640 (3)	3545 (2)	12963 (3)
C(11)	5375 (5)	4354 (3)	11168 (5)	O(2)	6205 (3)	3396 (2)	9747 (4)
C(12)	6156 (5)	4070 (3)	12070 (5)	O(1′)	639 (3)	3474 (2)	6427 (4)
C(13)	5598 (5)	3925 (3)	13184 (5)	O(2′)	3688 (3)	3411 (2)	5077 (4)
C(14)	5224 (6)	4522 (3)	13812 (6)	O(3′)	2496 (5)	2499 (3)	3552 (5)
C(15)	6383 (6)	3553 (3)	13944 (6)	O(4′)	1449 (5)	3271 (2)	2955 (4)
C(16)	6509 (5)	5145 (3)	10123 (6)	O(5′)	1275 (4)	3845 (2)	8077 (4)
C(17)	2758 (6)	5309 (3)	6705 (6)	C(*M*)	3472 (10)	2219 (5)	8715 (10)
C(18)	3549 (5)	3779 (3)	7392 (5)	O(*M*)	3068 (5)	2261 (3)	9755 (6)
C(19)	5606 (5)	5197 (3)	7403 (6)	O(*W*)	4625 (5)	2777 (2)	11031 (5)

Table 2. *The positional parameters for the hydrogen atoms* ($\times 10^3$)

	x	*y*	*z*		*x*	*y*	*z*
H(1*a*)	570 (4)	357 (2)	782 (4)	H(16*b*)	597 (4)	548 (2)	1042 (4)
H(1*b*)	618 (4)	405 (2)	675 (4)	H(16*c*)	710 (4)	507 (2)	1076 (4)
H(2*a*)	744 (4)	450 (2)	826 (4)	H(17*a*)	227 (4)	564 (2)	694 (4)
H(2*b*)	751 (4)	374 (2)	824 (4)	H(17*b*)	277 (4)	519 (2)	585 (4)
H(3*a*)	743 (4)	403 (2)	1014 (4)	H(18*a*)	332 (4)	370 (2)	827 (4)
H(3*b*)	671 (6)	313 (3)	990 (5)	H(18*b*)	410 (4)	347 (2)	728 (4)
H(5)	449 (4)	417 (2)	930 (4)	H(19*a*)	585 (4)	516 (2)	656 (4)
H(6*a*)	450 (4)	556 (2)	928 (4)	H(19*b*)	510 (4)	556 (2)	753 (5)
H(6*b*)	388 (4)	511 (2)	1023 (4)	H(19*c*)	631 (4)	532 (2)	779 (4)
H(7*a*)	262 (4)	470 (2)	902 (4)	H(8′*a*)	207 (4)	335 (2)	131 (4)
H(7*b*)	260 (4)	544 (2)	884 (4)	H(8′*b*)	85 (5)	303 (3)	140 (6)
H(9)	422 (4)	446 (2)	637 (4)	H(8′*c*)	201 (6)	256 (3)	179 (6)
H(11*a*)	468 (4)	399 (2)	1102 (4)	H(9′*a*)	−71 (4)	319 (2)	538 (4)
H(11*b*)	509 (4)	474 (2)	1149 (4)	H(9′*b*)	−36 (6)	259 (3)	466 (5)
H(12*a*)	677 (4)	436 (2)	1213 (4)	H(9′*c*)	−55 (6)	327 (3)	415 (6)
H(12*b*)	651 (4)	370 (2)	1170 (4)	H(10′*a*)	−22 (5)	331 (3)	844 (5)
H(13)	427 (5)	350 (3)	1368 (5)	H(10′*b*)	−30 (6)	408 (3)	806 (6)
H(14*a*)	475 (4)	475 (2)	1338 (4)	H(10′*c*)	25 (6)	385 (3)	940 (5)
H(14*b*)	488 (4)	444 (2)	1451 (4)	H(C*Ma*)	351 (6)	176 (3)	857 (6)
H(14*c*)	581 (4)	484 (2)	1398 (5)	H(C*Mb*)	424 (5)	250 (3)	855 (6)
H(15*a*)	710 (5)	377 (2)	1393 (4)	H(C*Mc*)	284 (6)	240 (3)	817 (6)
H(15*b*)	605 (4)	351 (2)	1478 (4)	H(O*M*)	344 (5)	238 (3)	1046 (5)
H(15*c*)	656 (4)	308 (2)	1365 (4)	H(O*Wa*)	505 (4)	293 (2)	1044 (4)
H(16*a*)	685 (4)	523 (2)	943 (4)	H(O*Wb*)	458 (4)	297 (2)	1181 (4)

rate of 4° (2θ) min^{-1}. Lorentz–polarization correction was made as usual. Structure factors for 3917 reflexions were obtained, of which 1453 with $|F_o| < 3{\cdot}0\,(\sigma|F_o|)$ were considered as unobserved. The structure was solved by the direct method with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by the block-diagonal least squares with the modified *HBLS* program. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, where w is the weight as follows: $w = 0{\cdot}2$ for $|F_o| < 12{\cdot}9$ and $|F_o| > 60{\cdot}2$, and $w = (10{\cdot}5537 - 0{\cdot}4496\,|F_o| + 0{\cdot}0053\,F_o^2)^{-1}$ for $12{\cdot}9 \le |F_o| \le 60{\cdot}2$. Some H atoms were located from a difference map and some were obtained geometrically. Their positions and isotropic temperature factors were also refined. The final R was 0·088 for 2464 reflexions. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final positional parameters for non-hydrogen and H atoms are listed in Tables 1 and 2 respectively.*

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32352 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

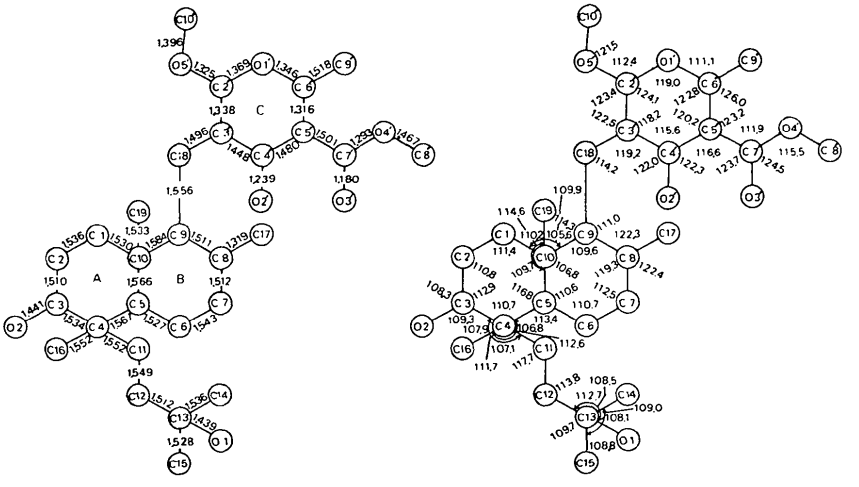

Fig. 2. Bond lengths (Å) and angles (°). Those involving H atoms are omitted. Standard errors on the bond lengths are in the range 0·007 to 0·013 Å, and on the bond angles 0·5 to 0·8°.

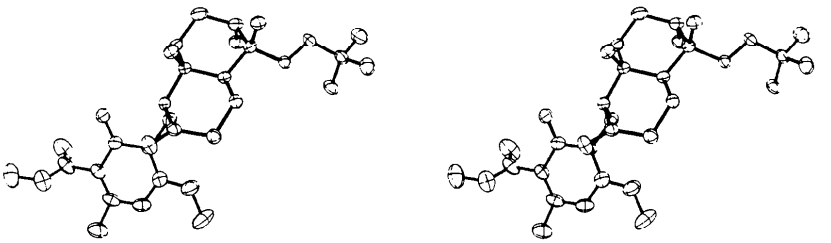

Fig. 3. Stereoscopic view with thermal ellipsoids at 30% probability.

Table 3. *Hydrogen-bond lengths* (Å) *and angles* (°)

Donor atom	Acceptor atom	Position of acceptor atom	Distance $D\cdots A$	Distance $H\cdots A$	Angle	
O(1)	O(2′)	$x, y, 1+z$	2·768 (6)	1·81 (7)	O(1)–H(13)–O(2′)	175 (7)
O(2)	O(*M*)	$\frac{1}{2}+x, \frac{1}{2}-y, 2-z$	2·720 (8)	1·89 (7)	O(2)–H(3*b*)–O(*M*)	164 (7)
O(*W*)	O(1)	x, y, z	2·805 (8)	1·83 (5)	O(*W*)–H(O*Wb*)–O(1)	162 (4)
O(*W*)	O(2)	x, y, z	2·775 (8)	1·90 (5)	O(*W*)–H(O*Wa*)–O(2)	157 (5)
O(*M*)	O(*W*)	x, y, z	2·654 (9)	1·79 (7)	O(*M*)–H(O*M*)–O(*W*)	144 (6)

Discussion. The crystal structure is shown in Fig. 1. Five intermolecular hydrogen bonds (Table 3) connect the molecules to form a two-dimensional sheet.

The bond lengths and angles (Fig. 2) are normal within experimental error. A stereoscopic drawing of the molecule is shown in Fig. 3. The two condensed rings, *A* and *B*, are in the chair form. The six atoms of the pyrone ring (*C*) are planar within experimental error and form an angle of 60·4° with the plane of the methoxycarbonyl group. This explains the UV absorption, λ_{max} = 260 nm (Suzuki, Gohbara, Kosuge, Tamura, Ohashi & Sasada, 1976). While this paper was being prepared, the molecular structure of the monoacetate of this compound was reported (Goddard, Hatton, Howard, MacMillan & Gilmore, 1976). Its structure is consistent with the present one, except for minor details.

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Acta Cryst. (1977). B**33**, 1278–1280

Iodophyllanthin

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(*Received* 14 *July* 1976; *accepted* 13 *December* 1976)

Abstract. $C_{24}H_{32}I_2O_6$, M_r = 670·3, orthorhombic, $C222_1$, a = 7·388 (4), b = 21·967 (9), c = 16·266 (8) Å, V = 2639·8 Å^3, D_m = 1·63, Z = 4, D_x = 1·686 g cm^{-3}. Mo radiation, R = 0·049 for 1174 reflexions. The molecule lies on a crystallographic twofold axis, and has normal dimensions.

Introduction. Unit-cell and intensity data (systematic absences: hkl, $h+k\neq 2n$, $00l$, $l\neq 2n$) were measured

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