2°. The two C atoms adjacent to each acetamide group deviate considerably from the mean plane of the respective group (deviations ranging from 0·14 to 0·36 Å).*

The acetamide groups are inclined to the plane through the C ring atoms by approximately the same angle (the average dihedral angle is 48° with a maximum deviation of only 2°). This geometrical configuration represents an interesting contrast to the highly distorted molecular shape of the RDX molecule in which the planes of the three nitro groups are inclined to the plane through the C ring atoms of angles ranging from 18° to 62°.

The observed C–H distances range from 1·01 to 1·08 Å for the ring C atoms and from 0·90 to 1·21 Å for the methyl groups.

Each ring C atom has short intramolecular contacts with the O and C atoms of the adjacent acetyl groups. These distances, on average, are 2·73 Å for C···O contacts and 2·93 Å for C···C contacts.

Fig. 3 shows the molecular packing in the crystal of TRAT. The shortest intermolecular contacts are C(3)···O(2) = 3·21 Å along the *a* axis and C(2)···O(1) = 3·38 Å along the direction of the unique *b* axis. All other intermolecular contacts are larger than 4·3 Å.

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* A table of the atomic distances from the best fit mean planes of the acetamide groups has been deposited with the structure factor table (see previous footnote).

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The Monoacetate of *O*-Methylasparvenone*

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Abstract. $C_{15}H_{18}O_5$, monoclinic, $P2_1$, $Z=2$, $a=5\cdot354\,(2)$, $b=11\cdot093\,(2)$, $c=11\cdot817\,(2)$ Å, $\beta=91\cdot53\,(2)$°, $D_m=1\cdot31$, $D_x=1\cdot32$ g cm^{-3}. The structure was determined by direct and Patterson methods. The final R value over 1529 independent reflections is 4·6%. The molecule forms a strong intramolecular hydrogen bond with O(1)···O(8) distance 2·550 Å. O(4) on the asymmetric carbon C(4) is axial to the cyclohexanone ring. C(4), O(4), C(11), O(11) and C(12) are coplanar and the plane through them is almost perpendicular to the least-squares plane of the cyclohexanone ring.

Introduction. 7-Ethyl-3,4-dihydro-4,8-dihydroxy-6-methoxy-1-[2*H*]-naphthalenone ($C_{13}H_{16}O_4$, *O*-methylasparvenone) is one of the phenolic constituents isolated from *Aspergillus parvulus* Smith (Chao, Schiff, Slatkin, Knapp, Chao & Rosenstein, 1975). Since this compound had not previously been isolated or synthesized, its molecular structure was unknown. The monoacetate derivative provided suitable single crystals for structure determination by X-ray analysis.

Pale brown, long needle-shaped crystals were grown by slow evaporation from ethanol solution. A crystal cut to approximate dimensions 0·2 × 0·2 × 0·3 mm was used. The precise lattice parameters and three-dimensional intensity data were derived by least-squares calculations from measurements on a four-circle computer-controlled Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation and θ–2θ scan mode with $\theta \le 75$°. Integrated intensities for 1529 independent reflections were collected, which yielded 66 weak reflections with integrated intensity less than $2\sigma(I)$. These reflections were assigned intensities of $\sigma(I)/2$ and given zero weight during the refinement. No corrections were made for absorption and extinction. All the intensity data were processed by routine methods (Shiono, 1971).

The structure was solved by a combination of direct and Patterson methods. The *MULTAN* program (Germain, Main & Woolfson, 1971) was applied to all reflections with $|E| \ge 1\cdot5$ after renormalization. The E synthesis calculated from the highest figure of merit

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showed a 'chicken wire' pattern. By checking all the peaks in this map against the E^2-1 Patterson function, the position of the fused-ring system was determined. The correct positions of the side chains were derived by repeated use of difference electron densities, checking each stage *versus* the E^2-1 Patterson function. Atomic parameters were refined by full-matrix least-squares methods using a modification of *ORFLS* (Shiono, 1970). After several cycles of refinement using isotropic thermal parameters for the non-hydrogen atoms, a difference map was obtained from which all the H atom positions were located. Anisotropic temperature factors were used for the non-hydrogen atoms in later refinement by block-diagonal least-squares methods (Shiono, 1971). The function minimized was $\sum w(|F_m|-|F_c|)^2$, where $w=(A+B|F_m|)^{-1}$. The constants A and B were adjusted during the refinement so as to make $\sum|\Delta F|^2$ approximately constant over various ranges of $|F_m|$. These values give an approximate fit to the smooth curve resulting from plotting $\sqrt{(|\Delta F|^2)_{\rm av}}$ *vs* $F_{\rm av}$ in the four ranges of F.

Two strong reflections, 001 and $10\bar{2}$, showed large ratios of F_c/F_m and were therefore given zero weight. The atomic scattering factors used were those of Cromer & Waber (1965) for O and C and of Stewart, Davidson & Simpson (1965) for H. The final $R=\sum(||F_m|-|F_c||)/\sum|F_m|$ was 0·046 and a weighted $R_w=\sum w(||F_m|-|F_c||)/\sum|F_m|$ was 0·047 for all reflections with non-zero weight.* The final atomic parameters are listed in Table 1. The molecular structure is in good agreement with the physical data such as ultraviolet, infrared and n.m.r. No attempt has been made to establish the absolute configuration.

Discussion. Interatomic distances and some valence angles, uncorrected for curvilinear thermal motion, are given in Fig. 1 and Table 2, respectively. C(1)–C(9) is the shortest bond [1·469 (2) Å] in the non-aromatic ring, which is a little less than the usual C(sp^2)–C(sp^2) single-bond distance (1·48 Å; March, 1968). Hence, there is probably some conjugation between the carbonyl group and the aromatic ring. The three bond angles of C(11) are highly distorted, but still retain the planar configuration; the sum of the bond angles is 359·9°. None of the other bond lengths and angles differ significantly from the usual values. The average C(sp^3)–C(sp^3) single-bond distance is 1·513 Å and the C(sp^3)–C(sp^2) single-bond distance is 1·501 Å. The average benzenoid C–C bond length is 1·398 Å. The aver-

Table 1. *Atomic parameters for the monoacetate of O-methylasparvenone*

Positional parameters are given as fractions of the lattice translations. Estimated standard deviations given in parentheses refer to the last digit in respective values. Anisotropic temperature factors correspond to the expression: $T=\exp[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})]$, and isotropic temperature factors to the expression: $\exp[-8\pi^2U\times\sin^2\theta/\lambda^2]$. C and O atomic positional and thermal parameters × 10^4; H atom positional and thermal parameters $[U(\text{Å}^2)]\times10^3$. An asterisk indicates a fixed parameter.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	1700 (4)	860 (2)	625 (2)	393 (9)	507 (12)	435 (10)	−7 (9)	17 (7)	−31 (9)
C(2)	1442 (4)	2040 (2)	19 (2)	533 (12)	590 (14)	466 (11)	107 (11)	−37 (9)	46 (10)
C(3)	3832 (4)	2791 (2)	63 (2)	556 (11)	392 (12)	488 (11)	136 (10)	144 (9)	141 (9)
C(4)	4837 (4)	2917 (2)	1253 (2)	392 (1)	354 (9)	574 (11)	8 (8)	159 (8)	54 (9)
C(5)	7058 (4)	1573 (2)	2642 (2)	391 (9)	370 (9)	451 (10)	−60 (8)	41 (8)	−50 (8)
C(6)	7353 (3)	462 (2)	3192 (2)	356 (9)	462 (11)	410 (9)	−2 (8)	19 (7)	−24 (8)
C(7)	5809 (5)	−523 (2)	2935 (2)	407 (9)	355 (10)	470 (11)	16 (8)	52 (8)	38 (8)
C(8)	3918 (3)	−358 (2)	2122 (2)	364 (8)	325 (9)	422 (9)	−35 (8)	49 (7)	−59 (7)
C(9)	3611 (3)	742 (0)*	1533 (2)	342 (8)	365 (9)	392 (9)	−22 (7)	55 (7)	−19 (7)
C(10)	5205 (3)	1712 (2)	1818 (2)	348 (9)	337 (9)	430 (9)	−19 (7)	107 (7)	−1 (8)
O(1)	309 (3)	2 (2)	370 (2)	555 (9)	672 (10)	572 (10)	−157 (8)	−160 (7)	−25 (9)
O(4)	3014 (3)	3566 (1)	1930 (1)	503 (7)	354 (7)	651 (9)	10 (6)	172 (6)	−27 (7)
O(6)	9115 (3)	261 (2)	4017 (1)	479 (7)	619 (10)	576 (9)	−43 (8)	−128 (6)	50 (8)
O(8)	2354 (3)	−1301 (1)	1913 (1)	512 (7)	371 (7)	654 (9)	−131 (7)	13 (6)	−52 (7)
C(11)	3352 (4)	4756 (2)	2072 (2)	530 (12)	432 (12)	758 (16)	−3 (10)	−14 (11)	−94 (11)
O(11)	5024 (4)	5303 (2)	1650 (3)	808 (13)	478 (11)	1725 (25)	−154 (11)	316 (14)	−127 (13)
C(12)	1315 (6)	5282 (3)	2754 (3)	831 (17)	597 (16)	809 (17)	202 (15)	17 (14)	−203 (15)
C(13)	10812 (4)	1201 (3)	4314 (2)	442 (11)	712 (16)	544 (12)	−41 (11)	−41 (9)	−98 (11)
C(14)	6117 (4)	−1725 (2)	3527 (2)	532 (12)	444 (12)	767 (15)	5 (10)	−37 (11)	185 (11)
C(15)	4528 (6)	−1795 (3)	4570 (3)	711 (16)	905 (23)	979 (21)	78 (17)	147 (15)	580 (19)

	x	y	z	U		x	y	z	U
H(1)	5 (4)	252 (2)	40 (2)	41 (6)	H(10)	1164 (5)	147 (3)	364 (2)	56 (7)
H(2)	80 (4)	185 (2)	−17 (2)	50 (7)	H(11)	951 (10)	177 (5)	458 (5)	165 (21)
H(3)	507 (4)	246 (2)	−42 (2)	42 (6)	H(12)	1193 (5)	82 (3)	491 (2)	69 (8)
H(4)	341 (5)	363 (3)	−27 (2)	63 (7)	H(13)	802 (6)	−186 (4)	381 (3)	87 (10)
H(5)	625 (4)	341 (3)	128 (2)	48 (6)	H(14)	562 (7)	−239 (4)	291 (3)	100 (12)
H(6)	179 (9)	618 (6)	272 (5)	140 (18)	H(15)	433 (7)	−251 (5)	479 (4)	132 (16)
H(7)	−42 (5)	524 (3)	237 (2)	75 (9)	H(16)	274 (5)	−169 (3)	424 (2)	65 (8)
H(8)	184 (8)	533 (5)	350 (4)	138 (15)	H(17)	485 (11)	−107 (5)	515 (5)	160 (17)
H(9)	825 (4)	230 (2)	284 (2)	32 (5)	H(18)	95 (8)	−100 (5)	119 (4)	155 (18)

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31316 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

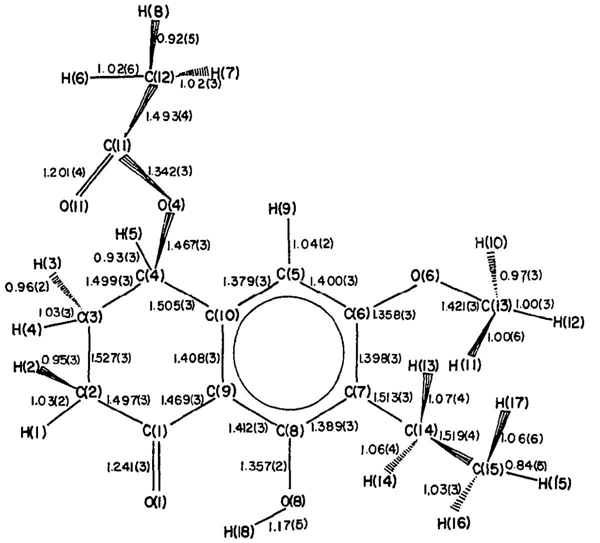

Fig. 1. Interatomic distances (Å) in the monoacetate of *O*-methylasparvenone. Estimated standard deviations are given in parentheses.

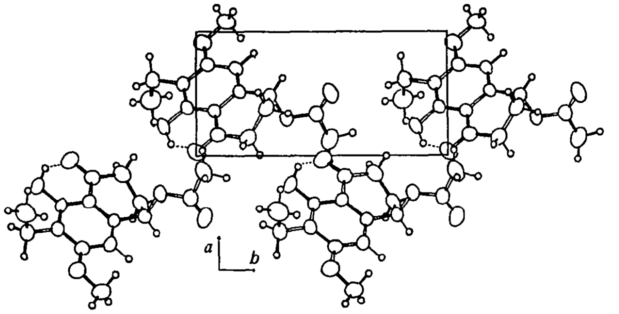

Fig. 2. The molecular packing in the crystal structure of the monoacetate of *O*-methylasparvenone, as seen in projection down **c**.

Table 2. *Bond angles (°) in the monoacetate of O-methylasparvenone*

C(2)—C(1)-C(9)	118·7 (2)	C(7)—C(8)—C(9)	121·8 (2)
C(2)—C(1)-O(1)	120·5 (2)	C(7)—C(8)—O(8)	117·3 (2)
C(9)—C(1)-O(1)	120·8 (2)	C(9)—C(8)—O(8)	120·9 (2)
C(1)—C(2)-C(3)	113·3 (2)	C(1)—C(9)—C(8)	120·4 (2)
C(2)—C(3)-C(4)	111·2 (2)	C(1)—C(9)—C(10)	120·9 (2)
C(3)—C(4)-C(10)	111·9 (2)	C(8)—C(9)—C(10)	118·7 (2)
C(3)—C(4)-O(4)	109·3 (2)	C(4)—C(10)-C(5)	119·7 (2)
C(10)-C(4)-O(4)	106·0 (2)	C(4)—C(10)-C(9)	120·1 (2)
C(6)—C(5)-C(10)	119·7 (2)	C(5)—C(10)-C(9)	120·2 (2)
C(5)—C(6)-C(7)	121·9 (2)	C(4)—O(4)—C(11)	117·5 (2)
C(5)—C(6)-O(6)	123·1 (2)	C(6)—O(6)—C(13)	118·9 (2)
C(7)—C(6)-O(6)	115·0 (2)	O(4)—C(11)-O(11)	123·0 (2)
C(6)—C(7)-C(8)	117·6 (2)	O(4)—C(11)-C(12)	110·8 (2)
C(6)—C(7)-C(14)	122·2 (2)	O(11)-C(11)-C(12)	126·2 (3)
C(8)—C(7)-C(14)	120·3 (2)	C(7)—C(14)-C(15)	111·4 (2)

age $C(sp^3)$-O and $C(sp^2)$-O single-bond distances are 1·444 and 1·352 Å, respectively.

The hydroxyl oxygen O(8) and the carbonyl oxygen O(1) form a strong intramolecular hydrogen bond with O···O distance 2·550 (3) Å and O-H···O angle 144 (4)°. For this reason, methylation and acetylation of the hydroxyl oxygen, O(8), does not easily take place and the proton chemical shift of H(18) is 12·74 p.p.m.

Table 3. *Best least-squares planes through selected atoms and the deviations of atoms from the least-squares plane*

Equations of planes $AX+BY+CZ=D$, where X, Y, Z are in Å.

Plane	*A*	*B*	*C*	*D*
1	−0·6887	0·4051	0·6195	0·2085
2	−0·6494	0·3000	0·7158	0·2981
3	0·5376	−0·1641	0·8124	2·0691

Displacements in Å from the best plane

	Plane 1	Plane 2	Plane 3
C(1)	0·008		
C(2)	0·190		
C(3)	−0·322		
C(4)	0·236		−0·005
C(5)		0·006	
C(6)		−0·002	
C(7)		−0·009	
C(8)		0·015	
C(9)	−0·085	−0·010	
C(10)	−0·028	−0·001	
O(4)			0·002
C(11)			0·019
O(11)			−0·006
C(12)			−0·009

Dihedral angle between planes 1 and 2 8·5°
1 and 3 96·5°

The best least-squares planes through the selected atoms are given in Table 3. The benzenoid ring is nearly planar and slightly bowed about the line through C(5) and C(8). The plane through C(6), O(6) and C(13) is inclined 1·9° to the benzenoid ring and the plane through C(7), C(14) and C(15) is perpendicular (89°) to this ring. C(4) is the asymmetric center. C(4), O(4), C(11), O(11) and C(12) are coplanar and this plane is almost perpendicular (86·5°) to the least-squares plane of the cyclohexanone ring. H(1), H(3) and O(4) are axial and H(2), H(4) and H(5) are equatorial to the ring.

No intermolecular hydrogen bond exists in the crystal structure. The molecular packing is shown in Fig. 2 viewed along **c**. The shortest intermolecular contacts are O(1) $[x,y,z]\cdots$H(4) $[-x,\frac{1}{2}-y,-z]=2{\cdot}51$ (3) Å and O(11) $[x,y,z]\cdots$H(7) $[1+x,y,z]=2{\cdot}57$ (3) Å. All other intermolecular contacts are larger than the sum of the van der Waals radii (Pauling, 1960).

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