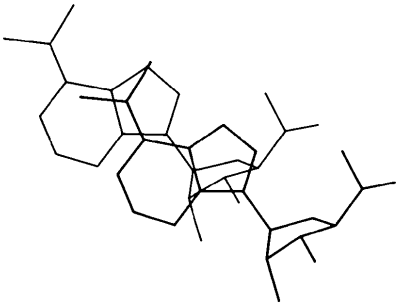

Fig. 3. A view perpendicular to the adenine base plane, indicating stacking interactions.

Molecular packing

The crystal structure is stabilized by the intermolecular hydrogen bond O(2′)—H···N(3′) [2·821 (6) Å and 172·4°]. There are no stacking interactions due to ring overlap of bases. However, the exocyclic atoms N(6) and C(10) of the base stack on top (≃3·54 Å) of the adenine base of the molecule related by the *a*-cell translation, as shown in Fig. 3.

We thank the Department of Science and Technology and Biotechnology, Government of India, for financial support.

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Dimerization Products of Substituted 1,2-Benzoquinones

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(*Received* 30 *December* 1987; *accepted* 18 *July* 1988)

Abstract. 1,4,4a,8a-Tetrahydro-2,8-dimethyl-1,4-ethanonaphthalene-5,6,9,10-tetrone (1), $C_{14}H_{12}O_4$, $M_r = 244{\cdot}25$, orthorhombic, *F*2*dd*, $a = 7{\cdot}505$ (1), $b = 14{\cdot}180$ (2), $c = 44{\cdot}548$ (5) Å, $V = 4741$ (1) Å^3, $Z = 16$, $D_x = 1{\cdot}369$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}94$ cm^{-1}, $F(000) = 2048$, $T = 293$ K, $R = 0{\cdot}0496$, 933 reflections. 1,4,4a,8a-Tetrahydro-9,9-dihydroxy-2,8-dimethyl-1,4-ethanonaphthalene-5,6,10-trione (2), $C_{14}H_{14}O_5$, $M_r = 262{\cdot}27$, monoclinic, *Cc*, $a = 9{\cdot}174$ (1), $b = 10{\cdot}799$ (2), $c = 12{\cdot}557$ (2) Å, $\beta = 103{\cdot}40$ (2)°, $V = 1210{\cdot}2$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}439$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 8{\cdot}76$ cm^{-1}, $F(000) = 552$, $T = 295$ K, $R = 0{\cdot}0320$, 819 reflections. 1,4,4a,8a-Tetrahydro-2,8-di-*tert*-butyl-1,4-ethanonaphthalene-5,6,9,10-tetrone (3), $C_{20}H_{24}O_4$, $M_r = 328{\cdot}41$, triclinic, $P\bar{1}$, $a = 8{\cdot}786$ (1), $b = 9{\cdot}112$ (1), $c = 12{\cdot}387$ (1) Å, $\alpha = 101{\cdot}24$ (1), $\beta = 92{\cdot}40$ (1), $\gamma = 114{\cdot}73$ (1)°, $V = 875{\cdot}0$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}247$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}80$ cm^{-1}, $F(000) = 352$, $T = 292$ K, $R = 0{\cdot}0499$, 1598 reflections. The bridged portions of the three molecules exhibit similar conformations although the keto function in (2) is replaced by a dihydroxy substituent. The cyclohexenedione ring in (1) is more flattened than that in (2) and (3). Bond C(1)—C(8*a*) = 1·572 (5) is significantly longer than the C(4)—C(4*a*) distance of 1·554 (1). It is suggested that this difference is inherent in the ring system and does not arise from steric factors.

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0108-2701/88/122178-05$03.00

Introduction. The pigments from some bolete fungi contain the naphtho[1,8-*bc*]pyrandione moiety as part of the structure. A possible biosynthetic pathway involving a Diels–Alder dimerization of a 1,2-benzoquinone intermediate has been suggested (Steglich, Huppertz & Steffan, 1985). In order to test this synthetic route, dimers of 4-methyl and 4-*tert*-butyl-1,2-benzoquinone were prepared by the procedure of Horner & Dürckheimer (1959) [see also Willstätter & Müller (1911)]. In the case of the methyl compound the dimerization leads to a 7:3 mixture of two isomers from which the major product (1) can be easily separated by crystallization from acetone (Steglich, Huppertz & Steffan, 1985). In the case of the *tert*-butyl compound only one isomer (3) is formed; however, the isomeric 4-*tert*-butyl formulation had been proposed earlier (Horner & Durckheimer, 1959). Compound (1) adds water to form (2) but can be regenerated by dehydration of (2). Because these molecules are of synthetic utility and have interesting steric requirements, we wish to report their investigation.

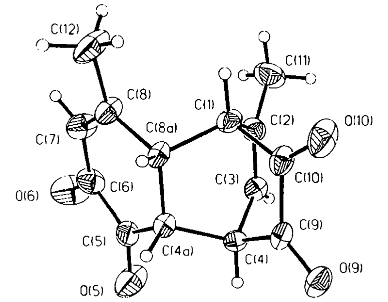


Fig. 1. Compound (1) with thermal ellipsoids drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.

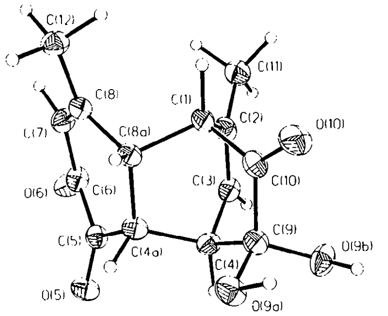


Fig. 2. Compound (2) with thermal ellipsoids drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.

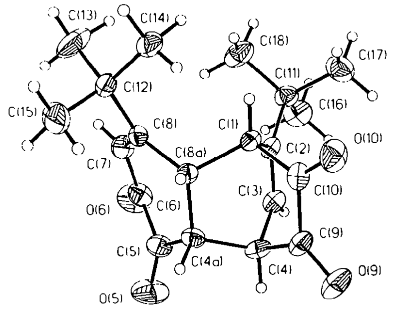
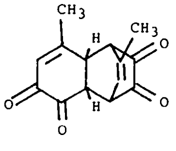
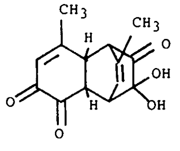


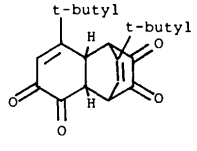
Fig. 3. Compound (3) with thermal ellipsoids drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.



(1)



(2)



(3)

Experimental. Data for compound (2) were collected on a Syntex $P2_1$ diffractometer using monochromated Cu $K\alpha$ radiation while data for compounds (1) and (3) were collected on a Nicolet $R3M/\mu$ update of a $P2_1$ diffractometer using monochromated Mo $K\alpha$ radiation. Data for (2) were collected by the θ:2θ technique while data for (1) and (3) were collected in the Wyckoff mode (2θ fixed, ω varied). Lorentz and polarization corrections were applied to all data and a ψ-scan-based empirical absorption correction was applied to the data of (1) and (3). All structures were solved by direct methods. The Nicolet program package (Nicolet Instrument Corporation, 1986) for Desktop 30 Microeclipse and Nova 4/C configuration was used for (1) and (3) while *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1970) were used for (2). Structure factors and anomalous-dispersion corrections were taken either from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) or from *International Tables for X-ray Crystallography* (1974). The function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = [\sigma^2(F_o) + gF_o^2]^{-1}$. Figs. 1, 2 and 3 are drawings of (1), (2) and (3). Tables 1–3 list atomic positional parameters while Tables 4 and 5 list interatomic distances and valence angles.*

Compound (1). Yellow transparent prismatic crystal, $0{\cdot}63 \times 0{\cdot}33 \times 0{\cdot}30$ mm, lattice parameters from 25 reflections ($24{\cdot}15 \leq 2\theta \leq 37{\cdot}34°$), data collected for $3{\cdot}0 \leq 2\theta \leq 50{\cdot}0°$, variable scan rate ($4{\cdot}0$ to $29{\cdot}3°$ min^{-1}), $h = 0,9$; $k = 0,17$; $l = 0,53$; standard reflections $(0,\overline{2},\overline{22})$ and $3\overline{3}\overline{5}$) showed only statistical

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51252 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for compound* (1)

	x	*y*	*z*	U_{eq}*
C(1)	7460 (7)	1800 (3)	4643 (1)	46 (1)
C(2)	7795 (7)	764 (3)	4564 (1)	48 (1)
C(3)	6302 (6)	274 (3)	4529 (1)	42 (1)
C(4)	4582 (6)	809 (3)	4567 (1)	39 (1)
C(4*a*)	4608 (7)	1644 (3)	4345 (1)	43 (1)
C(5)	4466 (7)	1219 (3)	4032 (1)	53 (1)
O(5)	3243 (7)	705 (4)	3973 (1)	106 (2)
C(6)	5881 (8)	1410 (4)	3795 (1)	59 (2)
O(6)	5781 (7)	988 (3)	3557 (1)	97 (2)
C(7)	7181 (7)	2091 (4)	3863 (1)	61 (2)
C(8)	7360 (7)	2529 (3)	4131 (1)	54 (1)
C(8*a*)	6265 (7)	2276 (3)	4398 (1)	42 (1)
C(9)	4657 (7)	1211 (3)	4884 (1)	41 (1)
O(9)	3575 (5)	1108 (2)	5076 (1)	56 (1)
C(10)	6413 (7)	1740 (3)	4932 (1)	47 (1)
O(10)	6842 (6)	2045 (2)	5171 (1)	72 (1)
C(11)	9646 (7)	403 (5)	4534 (1)	77 (2)
C(12)	8651 (10)	3308 (4)	4179 (1)	85 (2)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for compound* (2)

	x	*y*	*z*	U_{eq}
C(1)	4126 (5)	4224 (3)	1594 (4)	38 (2)
C(2)	4121 (6)	3819 (3)	439 (4)	41 (2)
C(3)	3104 (6)	2964 (3)	49 (4)	38 (2)
C(4)	2101 (5)	2601 (3)	799 (4)	39 (2)
C(4*a*)	1338 (6)	3800 (3)	1087 (4)	38 (2)
C(5)	269	4223 (3)	44	36 (2)
O(5)	−907 (5)	3689 (3)	−310 (3)	47 (1)
C(6)	704 (6)	5297 (3)	−583 (4)	42 (2)
O(6)	179 (5)	5400 (3)	−1566 (4)	64 (2)
C(7)	1660 (6)	6222 (3)	67 (4)	43 (2)
C(8)	2407 (5)	6024 (3)	1106 (4)	41 (2)
C(8*a*)	2540 (5)	4756 (3)	1614 (4)	36 (2)
C(9)	3089 (6)	2083 (3)	1850 (4)	41 (2)
O(9)	2238 (5)	1915 (3)	2633 (3)	52 (2)
O(9*b*)	3717 (5)	966 (3)	1599 (4)	53 (2)
C(10)	4332 (5)	3045 (3)	2274 (4)	41 (2)
O(10)	5348 (5)	2863 (3)	3055 (4)	61 (2)
C(11)	5166 (6)	4403 (5)	−162 (5)	52 (2)
C(12)	3206 (6)	7052 (4)	1795 (4)	41 (2)

Table 3. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for compound* (3)

	x	*y*	*z*	U_{eq}*
C(1)	7878 (3)	356 (3)	8345 (2)	37 (1)
C(2)	8306 (3)	1204 (3)	7392 (2)	40 (1)
C(3)	9595 (3)	1073 (3)	6941 (2)	46 (1)
C(4)	10360 (3)	99 (4)	7418 (2)	48 (1)
C(4*a*)	8941 (3)	−1667 (3)	7354 (2)	42 (1)
C(5)	8457 (4)	−2486 (4)	6130 (2)	53 (1)
O(5)	9321 (3)	−3004 (3)	5616 (2)	98 (2)
C(6)	6842 (4)	−2632 (4)	5555 (2)	55 (2)
O(6)	6696 (3)	−2698 (3)	4568 (2)	92 (2)
C(7)	5501 (3)	−2908 (4)	6229 (2)	53 (1)
C(8)	5735 (3)	−2547 (3)	7342 (2)	39 (1)
C(8*a*)	7488 (3)	−1539 (3)	7974 (2)	36 (1)
C(9)	10950 (3)	991 (3)	8612 (2)	50 (1)
O(9)	12350 (2)	1525 (3)	9111 (2)	74 (1)
C(10)	9493 (3)	1182 (3)	9161 (2)	44 (1)
O(10)	9661 (2)	1903 (3)	10111 (2)	67 (1)
C(11)	7368 (3)	2178 (4)	7112 (2)	50 (1)
C(12)	4246 (3)	−3199 (3)	7990 (2)	48 (1)
C(13)	2554 (4)	−3737 (5)	7261 (3)	98 (2)
C(14)	4320 (4)	−1953 (4)	9034 (3)	55 (2)
C(15)	4299 (4)	−4704 (4)	8333 (3)	89 (2)
C(16)	7898 (4)	2814 (4)	6075 (3)	73 (2)
C(17)	7798 (4)	3662 (4)	8082 (3)	78 (2)
C(18)	5461 (4)	1086 (4)	6903 (3)	79 (2)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 4. *Bond distances* (Å) *for compounds* (1), (2) *and* (3)

	(1)	(2)	(3)	Average
C(1)—C(2)	1·531 (6)	1·514 (7)	1·512 (4)	1·519 (9)
C(1)—C(10)	1·509 (6)	1·521 (6)	1·513 (3)	1·511 (2)*
C(1)—C(8*a*)	1·566 (6)	1·570 (7)	1·579 (4)	1·572 (5)
C(2)—C(3)	1·328 (6)	1·323 (6)	1·323 (4)	1·325 (2)
C(2)—C(11)	1·487 (7)	1·490 (8)	1·516 (5)	1·489 (2)†
C(3)—C(4)	1·506 (6)	1·513 (8)	1·503 (5)	1·507 (4)
C(4)—C(4*a*)	1·554 (5)	1·554 (6)	1·553 (3)	1·554 (1)
C(4)—C(9)	1·527 (5)	1·523 (6)	1·494 (4)	1·515 (15)
C(4*a*)—C(5)	1·521 (6)	1·514 (4)	1·508 (4)	1·514 (5)
C(4*a*)—C(8*a*)	1·551 (6)	1·543 (6)	1·551 (4)	1·548 (4)
C(5)—C(6)	1·522 (7)	1·506 (5)	1·503 (5)	1·510 (8)
C(5)—O(5)	1·201 (7)	1·213 (4)	1·195 (5)	1·203 (8)
C(6)—C(7)	1·405 (8)	1·450 (6)	1·437 (4)	1·431 (19)
C(6)—O(6)	1·218 (6)	1·221 (6)	1·211 (4)	1·217 (4)
C(7)—C(8)	1·354 (6)	1·343 (7)	1·340 (4)	1·346 (6)
C(8)—C(8*a*)	1·489 (6)	1·504 (5)	1·504 (3)	1·499 (7)
C(8)—C(12)	1·485 (8)	1·492 (6)	1·527 (4)	1·489 (4)†
C(9)—C(10)	1·531 (7)	1·543 (6)	1·531 (4)	1·531 (0)*
C(9)—O(9*a*)	1·188 (5)	1·402 (7)	1·205 (3)	1·197 (9)*
C(9)—O(9*b*)	—	1·404 (6)	—	
C(10)—O(10)	1·195 (5)	1·202 (6)	1·201 (3)	1·199 (3)

* (1) and (3) averaged.
† (1) and (2) averaged.

variations in intensities during data collection; 4965 reflections, 1128 unique ($R_{\rm int} = 0{\cdot}0067$), $933 > 3{\cdot}0\sigma(I)$; transmission factors 0·883–0·905; H atoms located in difference map but allowed to ride on attached carbon with a fixed isotropic thermal parameter, $R = 0{\cdot}0496$, $wR = 0{\cdot}0539$, 163 parameters, $R = 0{\cdot}0641$ (all data), $S = 1{\cdot}284$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}015$, $g = 0{\cdot}00129$, largest peaks in the final difference map +0·20 and −0·16 e Å^{-3}.

Compound (2). Red transparent crystal 0·80 × 0·63 × 0·50 mm, lattice parameters from 15 reflections ($12{\cdot}89 \leq 2\theta \leq 78{\cdot}99°$), data collected for $4 \leq 2\theta \leq 120°$, variable scan rate, $h = 0,8$; $k = 0,11$; $l = -13,13$; standard reflection (820) showed only statistical variations in intensity; 820 independent reflections, $819 > 3{\cdot}0\sigma(I)$; H atoms located in difference map, positional parameters refined using a single isotropic thermal parameter, $R = 0{\cdot}0320$, $wR = 0{\cdot}0465$ for 214 parameters, $S = 2{\cdot}28$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}016$, $g = 0{\cdot}000$, largest peaks in the final difference map +0·14 and −0·16 e Å^{-3}.

Compound (3). Yellow transparent crystal, 0·25 × 0·18 × 0·18 mm, lattice parameters from 25 reflec-

Table 5. *Valence angles* (°) *for compounds* (1), (2) *and* (3)

	(1)	(2)	(3)	Average
C(2)—C(1)—C(8*a*)	110·3 (3)	108·9 (4)	111·5 (2)	110·2 (10)
C(2)—C(1)—C(10)	103·1 (3)	105·5 (3)	104·7 (2)	104·4 (10)
C(8*a*)—C(1)—C(10)	108·7 (4)	107·0 (4)	106·3 (2)	107·3 (10)
C(1)—C(2)—C(3)	113·0 (4)	113·7 (5)	112·6 (3)	113·1 (5)
C(1)—C(2)—C(11)	120·3 (4)	120·2 (4)	119·7 (2)	120·1 (2)
C(3)—C(4)—C(4*a*)	107·6 (3)	107·5 (3)	108·4 (2)	107·8 (4)
C(3)—C(4)—C(9)	105·1 (3)	108·0 (4)	104·8 (3)	104·9 (2)*
C(4*a*)—C(4)—C(9)	107·9 (3)	108·3 (4)	108·7 (2)	108·3 (4)*
C(4)—C(4*a*)—C(5)	106·4 (3)	106·7 (3)	105·1 (2)	106·1 (7)
C(4)—C(4*a*)—C(8*a*)	110·8 (3)	110·0 (4)	110·0 (2)	110·3 (4)
C(5)—C(4*a*)—C(8*a*)	115·2 (4)	115·1 (3)	115·5 (2)	115·3 (2)
C(4*a*)—C(5)—C(6)	121·1 (4)	119·2 (3)	118·2 (3)	119·5 (12)
C(4*a*)—C(5)—O(5)	119·6 (4)	121·1 (3)	121·9 (3)	120·9 (10)
C(6)—C(5)—O(5)	119·3 (4)	119·7 (2)	119·9 (3)	119·6 (3)
C(5)—C(6)—C(7)	117·2 (4)	115·8 (4)	114·5 (3)	115·8 (11)
C(5)—C(6)—O(6)	118·2 (5)	120·5 (3)	119·7 (3)	119·5 (10)
C(7)—C(6)—O(6)	124·5 (5)	123·5 (4)	125·3 (3)	124·4 (7)
C(6)—C(7)—C(8)	124·9 (5)	122·9 (4)	124·3 (2)	124·0 (8)
C(7)—C(8)—C(8*a*)	122·7 (4)	122·0 (3)	120·2 (2)	121·6 (11)
C(7)—C(8)—C(12)	122·2 (4)	121·0 (4)	120·9 (2)	121·4 (6)
C(4*a*)—C(8*a*)—C(8)	117·3 (3)	116·6 (3)	115·5 (2)	116·5 (7)
C(1)—C(8*a*)—C(4*a*)	108·5 (3)	108·8 (3)	108·3 (2)	108·5 (2)
C(1)—C(8*a*)—C(8)	110·2 (4)	108·0 (4)	110·8 (2)	109·7 (12)
C(4)—C(9)—C(10)	110·0 (3)	107·2 (3)	110·2 (2)	110·1 (1)*
C(4)—C(9)—O(9*a*)	126·6 (4)	109·8 (4)	127·5 (3)	127·1 (4)*
C(4)—C(9)—O(9*b*)	—	108·3 (3)	—	
C(10)—C(9)—O(9*a*)	123·3 (4)	109·4 (4)	122·3 (3)	122·8 (5)*
C(10)—C(9)—O(9*b*)	—	110·3 (4)	—	
O(9)—C(9)—O(9*b*)	—	111·8 (4)	—	
C(1)—C(10)—C(9)	111·0 (3)	122·9 (4)	110·7 (2)	110·9 (2)*
C(1)—C(10)—O(10)	126·8 (3)	124·5 (4)	126·4 (3)	126·6 (2)*
C(9)—C(10)—O(10)	122·2 (4)	112·6 (3)	122·9 (2)	122·6 (3)*

* (1) and (3) averaged.

tions ($33{\cdot}41 \le 2\theta \le 38{\cdot}56°$), data collected for $3 \le 2\theta \le 45°$, variable scan rate (4·0 to 29·3° min^{-1}), $h = -8,8$; $k = -9,9$; $l = 0,13$; standard reflections ($\bar{3}04$ and 015) showed only statistical variations in intensities; 2282 independent reflections measured, $1598 > 3\sigma(I)$; H atoms located in difference map, positional and isotropic thermal parameters refined, $R = 0{\cdot}0499$, $wR = 0{\cdot}0430$ for 313 parameters, $S = 1{\cdot}284$, $(\Delta/\sigma)_{\max} = 0{\cdot}080$, $g = 0{\cdot}00025$, largest peaks in the final difference map +0·16 and −0·15 e Å^{-3}.

Discussion. The six-membered rings of the bicyclic systems of the three molecules exhibit similar torsion angles even when the C(9) ketone moiety in (2) is replaced by dihydroxy substituents. The bicyclic system restricts the six-membered rings to boat conformations with the torsion angle C(1)C(10)C(9)C(4) of (2) increasing to only 4·7 (5)° upon the pyramidalization of C(9). Except for the distances and angles directly associated with C(9) the structural parameters of the bicyclic systems are quite consistent for the three molecules. Molecular mechanics calculations (Allinger & Yuh, 1980) on compound (1) lead to similar torsion angles, but there are significant discrepancies in distances. The parameters of the diketone system must be adjusted to reproduce the observed values. The most interesting discrepancy involves C(1)—C(8*a*) and C(4)—C(4*a*) which are calculated to be equivalent with separations of 1·549 Å but are observed to be on average 1·572 (5) and 1·554 (1) Å, respectively. In such situations one looks for steric interactions such as those involving the C(8) and C(2) substituents.

The conformations of the six-membered fused rings composed of atoms C(4*a*) to C(8*a*) differ significantly. The six-membered rings of (2) and (3) are similar and can be described as twist or twist–boat conformations (Bucourt, 1974) with torsion angles ranging from −34·8 (4) to +21·0 (5)°. The six-membered ring in (1) has a very flattened twist conformation with angles ranging from −13·3 (7) to +9·9 (5)°. The O(5)C(5)-C(6)O(6) torsion angles are −4·3 (8), −23·3 (7) and −26·3 (5)° for (1), (2) and (3), respectively. The difference between (1) and (3) can be rationalized as due to differences in steric requirements between methyl and *tert*-butyl substituents; however, the conformational differences between (1) and (2) require additional considerations. Small changes in the torsional constants involving the diketone moiety can lead to molecular mechanics calculations giving either conformer as that of lowest energy. Since the conformational differences represent small changes in energy, intermolecular interactions may be of importance. The two hydroxyl groups of (2) are hydrogen bonded to O(5) and O(6) of an adjacent molecule ($0{\cdot}5+x$, $0{\cdot}5-y$, $0{\cdot}5+x$) with O(9*a*)···O(5′) = 2·821 (6), O(9*b*)···O(6′) = 2·802 (6) Å, O(9*a*)—H(9*a*)···O(5′) = 160 (1), O(9*b*)—H(9*b*)···O(6′) = 172 (1)°. The maximization of hydrogen bonding may be sufficient to lead to the observed conformational differences.

In (1) the shortest intramolecular interactions involving methyl H atoms are H(4*a*)···H(12*a*) = 2·10, H(4)···H(12*a*) = 2·29 and H(4)···H(11*a*) = 2·33 Å while in (2) the conformational change results in only H(1)···H(12*c*) = 2·14 Å being shorter than 2·60 Å. The larger *tert*-butyl groups lead to a number of short interactions H(1)···H(14*c*) = 2·27, H(1)···H(14*a*) = 2·26, H(8*a*)···H(14*c*) = 2·39, H(8*a*)···H(15*b*) = 2·36, H(7)···H(13*b*) = 2·07 and H(14*a*)···H(18*a*) = 2·36 Å. (Standard deviations are estimated to be 0·04 Å or less.) While there are significant interactions in (1) and (3), the interactions in (2) are insufficient to rationalize the elongation of the C(1)—C(8*a*) bond relative to C(4)—C(4*a*) as arising from steric factors. The C(1)—C(8*a*) and C(4)—C(4*a*) environments are almost identical on the C(1) and C(4) sides of the bonds but differ on the C(8*a*) and C(4*a*) sides. Atom C(8*a*) is adjacent to a double bond [1·46 (6) Å], which may be slightly elongated, while C(4*a*) is adjacent to a keto C(sp^2) carbon atom. Elongation of C(1)—C(8*a*) and possibly C(7)—C(8) may indicate an interaction of antibonding orbitals which is inherent in the molecular structure. Such differences may show up in a geometry-optimized quantum mechanical calculations rather than the normal molecular mechanics calculation.

We thank The Welch Foundation (P-074) and the National Science Foundation (CHE-8514367) for financial support.

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Structure of Dichloro(*p*-methoxyphenyl)(2-oxocyclohexyl)tellurium(IV)

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(*Received* 28 *April* 1988; *accepted* 22 *June* 1988)

Abstract. $[TeCl_2(CH_3OC_6H_4)(C_6H_9O)]$, $M_r = 402{\cdot}78$, monoclinic, $P2_1/n$, $a = 7{\cdot}435$ (3), $b = 18{\cdot}982$ (2), $c = 21{\cdot}252$ (4) Å, $\beta = 90{\cdot}66$ (2)°, $V = 2999$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}78$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}16$ mm^{-1}, $F(000) = 1568$, $T = 296$ K, final $R = 0{\cdot}068$ for 1577 observed reflections. There are two essentially identical independent molecules in the asymmetric unit. In both molecules the Te atom is bonded to the *p*-methoxyphenyl group, to the cyclohexanone and to the Cl atoms in a pseudo-trigonal-bipyramidal arrangement with axial halogens and with the lone pair at one of the equatorial positions. Coordination distances and angles: Te—Cl = 2·542 (9), 2·47 (1), Te—C(cyclohexanone) = 2·16 (3) and Te—C = 2·10 (3) Å; Cl—Te—C = 86·4 (8), 86·6 (7), 88·3 (9), 92·3 (7), Cl—Te—Cl = 174·4 (3), C—Te—C = 99 (1)° for one molecule and Te′—Cl′ = 2·536 (9), 2·487 (9), Te′—C′(cyclohexanone) = 2·23 (3), Te′—C′ = 2·10 (3) Å; Cl′—Te′—C′ = 88·7 (8), 86·7 (9), 91·0 (8), 89·5 (9), Cl′—Te—Cl′ = 176·1 (3), C′—Te′—C′ = 99 (1)° for the other. Short intramolecular Te···O contacts of 2·93 (1) Å for one molecule and 2·94 (2) Å for the other were found.

Introduction. Methods of organic synthesis based on tellurium reagents have received much attention in the last few years (Petragnani & Comasseto, 1986). α-Telluroketones are among the potential intermediates for this purpose, because of their analogy with the widely used α-selenoketones (Liotta, 1986). Recently, (dichloro)aryltelluroketones were systematically studied for the first time (Stefani, Comasseto & Petragnani, 1987). Compound (I) is one of several α-(dichloro)arytelluroketones synthesized.

To learn about the stereochemistry of this class of compounds, and more specifically about the possible

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