bromocyclopropane; the availability of relaxation of nonbonded strain is related to the stiffness of the deformation mode of the CCHal angle. A molecular-orbital interpretation of the geometrical data on the basis of the Hoffmann scheme alone is inconsistent at the moment, but it is suggested that this should be performed with due consideration of the symmetry differences of the molecules under investigation. For our part, we hope to improve the statistical significance of the bond-length effects in the bromo derivatives by neutron diffraction. Attempts to grow suitable crystals are in progress.

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Diketones from the Oxidation of Two *syn*-Sesquinorbornene Derivatives

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Abstract. Dimethyl 3,5,6,7,8,10-hexahydro-3,10:5,8-dimethanocyclodecene-4,9-dione-1,2-dioate (5), $C_{16}H_{18}O_6$, $M_r = 306{\cdot}31$, triclinic, $P\bar{1}$, $a = 12{\cdot}535\,(3)$, $b = 15{\cdot}947\,(3)$, $c = 7{\cdot}896\,(1)$ Å, $\alpha = 111{\cdot}00\,(1)$, $\beta = 92{\cdot}86\,(1)$, $\gamma = 96{\cdot}09\,(1)°$, $V = 1458{\cdot}6\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}395$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 8{\cdot}55$ cm^{-1}, $F(000) = 648$, $T = 300$ K, $R = 0{\cdot}052$ for 3807 independent reflections. 5,7,8,9,10,12-Hexahydro-5,12:7,10-dimethanobenzocyclodecene-6,11-dione (6), $C_{16}H_{16}O_2$, $M_r = 240{\cdot}30$, monoclinic, $P2_1/c$, $a = 8{\cdot}078\,(2)$, $b = 11{\cdot}814\,(2)$, $c = 13{\cdot}106\,(2)$ Å, $\beta = 96{\cdot}58\,(1)°$, $V = 1242{\cdot}5\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}284$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 6{\cdot}30$ cm^{-1}, $F(000) = 512$, $T = 300$ K, $R = 0{\cdot}049$ for 1436 reflections. The strained central double bonds of *syn*-sesquinorbornenes are easily oxidized to diketone derivatives resulting in eight-membered rings which exhibit flattened crown conformations. The ketone systems are not planar but are folded along an axis passing through the carbonyl C atoms forming interplanar angles of 161·4 (5), 163·0 (5)° for the two independent molecules of (5) and 173·5 (5)° for (6). The C(1)—C(10) double-bond systems deviate slightly from planarity.

Introduction. The reactivity of the double bond in *syn*-sesquinorbornene and its derivatives has been of considerable interest because its stereospecificity reflects the rigid relationship of the various parts of the molecule. The *syn*-sesquinorbornenes (1) and (2) are very reactive toward oxygen. Bubbling oxygen through a dichloromethane solution of (1) at room temperature for 2 d in the dark leads to a mixture of epoxide (3) (60%) and diketone (5) (40%). Similarly autoxidation of (2) has been reported to give a mixture of (4) and (6) (Paquette & Carr, 1980). We have established earlier the structure of (3) from X-ray studies of the epoxide obtained by peracid epoxidation of (1) (Subramanyam, Bartlett, Moltrasio Iglesias, Watson & Galloy, 1982). Since the conformation of the cyclooctanedione might be responsive to the special properties of the ethylene

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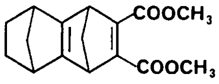
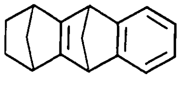
bridges, we have determined the structures of (5) and (6).



(1)



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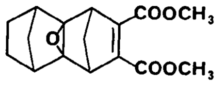

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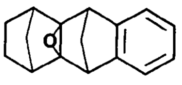

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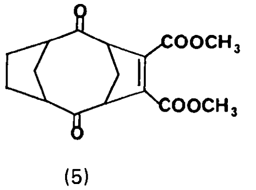

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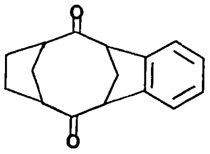

(6)

Experimental. Compound (5), colorless crystal of dimensions 0·44 × 0·37 × 0·32 mm, Nicolet $R3m/\mu$ update of $P2_1$ diffractometer, data collected in Wyckoff mode (2θ fixed, ω varied, $4 \leq 2\theta \leq 115°$), graphite-monochromated Cu $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($9{\cdot}90 \leq 2\theta \leq 32{\cdot}64°$), angles measured by a centering routine associated with the diffractometer, Laue class $\bar{1}$, lack of systematic absences and statistics consistent with space group $P\bar{1}$; two monitored reflections ($\bar{1}0\bar{2}$ and $22\bar{2}$) showed no significant changes in intensity; 4076 independent reflections measured ($0 \leq h \leq 14$, $-18 \leq k \leq 18$, $-9 \leq l \leq 9$), 3807 intensities greater than $3\sigma(I)$; Lorentz–polarization corrections applied, ψ-scan empirical absorption correction (transmission factors 0·181 to 0·280); direct methods revealed all non-H atoms, H atoms found in subsequent difference Fourier maps, block-cascade least-squares refinement with riding model for H atoms, final R of 0·052, $wR = 0{\cdot}071$, 428 parameters, 3807 reflections, $S = 2{\cdot}85$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}41$, $(\Delta/\sigma)_{\rm mean} = 0{\cdot}011$; largest peaks in final difference Fourier map of 0·42 and $-0{\cdot}28$ e Å^{-3}, $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}00076F_o^2]^{-1}$; computer programs supplied by Nicolet for Desktop 30 Microeclipse and Nova/4c configuration (Nicolet Instrument Corporation, 1986); atomic scattering factors and anomalous-dispersion corrections contained in the program package (*International Tables for X-ray Crystallography*, 1974). Table 1 lists atomic positional parameters for compound (5).

Compound (6), colorless crystal of dimensions 0·52 × 0·47 × 0·24 mm, Syntex (Nicolet) $P2_1$ diffractometer, data collected in $\theta{:}2\theta$ mode ($4 \leq 2\theta \leq 120°$), variable scan rate; graphite-monochromated Cu $K\alpha$ radiation; lattice parameters from least-squares refinement of 15 reflections ($10{\cdot}1 \leq 2\theta \leq 21{\cdot}82°$), systematic absences ($h0l$, $l = 2n + 1$, $0k0$, $k = 2n + 1$) consistent with space group $P2_1/c$, Laue class $2/m$; one monitored reflection (222) showed no significant change in intensity; 1699 independent reflections measured ($0 \leq h \leq 8$, $0 \leq k \leq 12$, $-14 \leq l \leq 14$), 1436

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2 $\times 10^3$) *for compounds* (5) *and* (6)

	x	y	z	$U_{\rm eq}$*
Compound (5)				
C(1)	2337 (2)	3268 (2)	7659 (3)	41 (1)
C(2)	1759 (2)	2443 (2)	6129 (3)	44 (1)
C(3)	2301 (2)	2153 (2)	4332 (3)	49 (1)
C(4)	3452 (2)	1938 (2)	4181 (3)	44 (1)
C(5)	4334 (2)	2735 (2)	5259 (3)	47 (1)
C(6)	4825 (2)	2530 (2)	6860 (3)	46 (1)
C(7)	4293 (2)	1588 (2)	6734 (3)	42 (1)
C(8)	3594 (2)	1572 (1)	8237 (3)	40 (1)
C(9)	2590 (2)	2049 (2)	8615 (3)	41 (1)
C(10)	2784 (2)	3054 (2)	9001 (3)	39 (1)
C(11)	3719 (2)	1163 (2)	4805 (3)	46 (1)
C(12)	1687 (2)	1725 (2)	7019 (3)	47 (1)
O(1)	1756 (2)	2039 (2)	2963 (3)	104 (1)
O(2)	3820 (2)	1146 (1)	9163 (3)	63 (1)
C(13)	3408 (2)	3667 (2)	10699 (3)	46 (1)
O(3a)	4076 (2)	3437 (2)	11513 (3)	81 (1)
O(3b)	3177 (2)	4509 (1)	11227 (3)	65 (1)
C(14)	3822 (3)	5167 (2)	12811 (5)	91 (1)
C(15)	2269 (2)	4202 (2)	7710 (3)	49 (1)
O(4a)	1456 (2)	4527 (2)	7828 (4)	86 (1)
O(4b)	3206 (2)	4641 (1)	7615 (3)	68 (1)
C(16)	3219 (3)	5583 (2)	7857 (5)	81 (2)
C(1′)	758 (2)	8006 (2)	7762 (3)	39 (1)
C(2′)	845 (2)	8957 (2)	9163 (3)	43 (1)
C(3′)	1480 (2)	9129 (2)	11000 (3)	46 (1)
C(4′)	2620 (2)	8902 (2)	11143 (3)	44 (1)
C(5′)	2793 (2)	7913 (2)	10125 (3)	47 (1)
C(6′)	3544 (2)	7893 (2)	8633 (3)	48 (1)
C(7′)	3712 (2)	8853 (2)	8581 (3)	44 (1)
C(8′)	3140 (2)	8938 (2)	6906 (3)	42 (1)
C(9′)	1915 (2)	8880 (2)	6573 (3)	43 (1)
C(10′)	1336 (2)	7970 (2)	6368 (3)	39 (1)
C(11′)	3460 (2)	9463 (2)	10467 (3)	46 (1)
C(12′)	1344 (2)	9525 (2)	8108 (3)	47 (1)
O(1′)	1078 (2)	9502 (2)	12381 (3)	78 (1)
O(2′)	3665 (2)	9019 (2)	5722 (3)	79 (1)
C(13′)	1476 (2)	7133 (2)	4784 (3)	45 (1)
O(3a′)	2052 (2)	6598 (2)	4842 (3)	111 (1)
O(3b′)	890 (2)	7061 (1)	3316 (2)	59 (1)
C(14′)	985 (3)	6283 (2)	1688 (4)	63 (1)
C(15′)	79 (2)	7265 (2)	8049 (3)	44 (1)
O(4a′)	−261 (2)	7343 (1)	9476 (3)	62 (1)
O(4b′)	−98 (2)	6513 (1)	6564 (3)	63 (1)
C(16′)	−773 (3)	5760 (2)	6727 (5)	91 (2)
Compound (6)				
C(1)	9681 (3)	2590 (2)	927 (2)	54 (2)
C(2)	8651 (4)	2306 (3)	−97 (2)	68 (2)
C(3)	7396 (4)	1365 (3)	27 (2)	72 (2)
C(4)	5884 (4)	1491 (3)	593 (3)	78 (2)
C(5)	6315 (6)	1533 (3)	1787 (3)	93 (3)
C(6)	6020 (4)	2705 (3)	2139 (2)	69 (2)
C(7)	5269 (3)	3412 (2)	1204 (2)	61 (2)
C(8)	6397 (4)	4381 (2)	973 (2)	58 (2)
C(9)	8018 (4)	4241 (2)	496 (2)	59 (2)
C(10)	9340 (3)	3678 (2)	1241 (2)	49 (1)
C(11)	4777 (4)	2532 (3)	358 (3)	70 (2)
C(12)	7973 (4)	3474 (3)	−466 (2)	69 (2)
C(13)	10134 (3)	4105 (2)	2144 (2)	61 (2)
C(14)	11273 (4)	3426 (3)	2756 (2)	70 (2)
C(15)	11588 (4)	2349 (3)	2442 (3)	77 (2)
C(16)	10804 (4)	1912 (3)	1513 (3)	72 (2)
O(1)	7666 (4)	456 (2)	−329 (3)	121 (2)
O(2)	6039 (3)	5324 (2)	1231 (2)	84 (2)

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

intensities greater than $3\sigma(I)$; Lorentz–polarization corrections applied, no absorption correction; direct methods (*MULTAN*78: Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) located all non-H atoms; H atoms located in difference Fourier map, used in structure-factor calculation but coordinates not refined, full-matrix least-squares refinement led to a final $R = 0{\cdot}047$, $wR = 0{\cdot}073$, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F_o)$ from counting statistics, $S = 3{\cdot}7$, $(\Delta/\sigma)_{\max} = 0{\cdot}10$, $(\Delta/\sigma)_{\text{avg}} = 0{\cdot}04$; largest peaks in the final difference Fourier map of 0·15 and $-0{\cdot}23$ e Å^{-3}; locally written programs for data reduction, *MULTAN*78 for direct-methods calculations and *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) for all others; atomic scattering factors for C and O from Cromer & Mann (1968), those for H from Stewart, Davidson & Simpson (1965), anomalous-dispersion corrections (*International Tables*

Table 2. *Bond lengths* (Å) *for compounds* (5) *and* (6)

	(5*a*)	(5*b*)		(6)
C(1)—C(2)	1·512 (3)	1·512 (3)		1·533 (4)
C(1)—C(10)	1·339 (4)	1·335 (3)		1·387 (4)
C(2)—C(3)	1·543 (3)	1·538 (3)		1·526 (5)
C(2)—C(12)	1·541 (4)	1·542 (4)		1·542 (5)
C(3)—C(4)	1·519 (3)	1·519 (4)		1·507 (5)
C(3)—O(1)	1·195 (3)	1·204 (3)		1·201 (4)
C(4)—C(5)	1·547 (3)	1·538 (3)		1·565 (6)
C(4)—C(11)	1·547 (4)	1·546 (4)		1·531 (5)
C(5)—C(6)	1·532 (4)	1·538 (4)		1·487 (5)
C(6)—C(7)	1·544 (4)	1·539 (4)		1·548 (4)
C(7)—C(8)	1·516 (3)	1·528 (4)		1·515 (4)
C(7)—C(11)	1·531 (3)	1·528 (3)		1·539 (4)
C(8)—C(9)	1·527 (3)	1·532 (3)		1·523 (5)
C(8)—O(2)	1·206 (4)	1·205 (3)		1·209 (4)
C(9)—C(10)	1·510 (3)	1·502 (3)		1·516 (4)
C(9)—C(12)	1·550 (3)	1·546 (3)		1·550 (4)
C(10)—C(13)	1·469 (3)	1·501 (3)	C(10)—C(13)	1·376 (4)
C(13)—O(3a)	1·196 (4)	1·186 (4)	C(13)—C(14)	1·402 (4)
C(13)—O(3b)	1·324 (3)	1·303 (3)	C(14)—C(15)	1·370 (5)
O(3b)—C(14)	1·448 (3)	1·453 (3)	C(15)—C(16)	1·405 (5)
C(1)—C(15)	1·488 (4)	1·474 (4)	C(1)—C(16)	1·377 (4)
C(15)—O(4a)	1·184 (3)	1·194 (3)		
C(15)—O(4b)	1·322 (3)	1·331 (3)		
O(4b)—C(16)	1·443 (4)	1·443 (5)		

Table 3. *Bond angles* (°) *for compounds* (5) *and* (6)

	(5*a*)	(5*b*)		(6)
C(2)C(1)C(10)	112·1 (2)	112·0 (2)		110·9 (2)
C(10)C(1)C(15)	125·7 (2)	128·6 (2)	C(10)C(1)C(16)	121·0 (2)
C(1)C(2)C(3)	115·7 (2)	116·2 (2)		111·5 (2)
C(1)C(2)C(12)	101·8 (2)	101·6 (2)		102·5 (2)
C(3)C(2)C(12)	113·5 (2)	113·8 (2)		118·0 (3)
C(2)C(3)C(4)	124·1 (2)	122·6 (2)		124·5 (3)
C(2)C(3)O(1)	117·5 (2)	118·6 (2)		117·2 (3)
C(4)C(3)O(1)	118·2 (2)	118·7 (2)		118·2 (2)
C(3)C(4)C(5)	115·4 (2)	116·8 (2)		113·3 (3)
C(3)C(4)C(11)	115·8 (2)	113·9 (2)		117·9 (3)
C(5)C(4)C(11)	103·8 (2)	104·5 (2)		103·5 (3)
C(4)C(5)C(6)	107·8 (2)	108·0 (2)		108·3 (3)
C(5)C(6)C(7)	108·3 (2)	107·4 (2)		108·6 (2)
C(6)C(7)C(8)	116·2 (2)	114·2 (2)		112·1 (2)
C(6)C(7)C(11)	104·4 (2)	104·0 (2)		104·6 (2)
C(8)C(7)C(11)	114·8 (2)	118·5 (2)		118·6 (2)
C(7)C(8)C(9)	123·4 (2)	124·0 (2)		124·4 (2)
C(7)C(8)O(2)	119·0 (2)	119·2 (2)		118·1 (3)
C(9)C(8)O(2)	117·6 (2)	116·7 (2)		117·3 (2)
C(8)C(9)C(10)	114·6 (2)	112·6 (2)		111·0 (2)
C(8)C(9)C(12)	115·3 (2)	116·8 (2)		117·1 (2)
C(10)C(9)C(12)	101·6 (2)	101·7 (2)		102·5 (2)
C(1)C(10)C(9)	112·2 (2)	112·5 (2)		111·4 (2)
C(1)C(10)C(13)	128·0 (2)	126·4 (2)		120·4 (2)
C(4)C(11)C(7)	108·1 (2)	107·6 (2)		107·5 (3)
C(2)C(12)C(9)	106·7 (2)	106·6 (2)		107·1 (2)
C(1)C(15)O(4a)	123·6 (2)	122·6 (2)	C(10)C(13)C(14)	119·6 (2)
C(1)C(15)O(4b)	113·6 (2)	112·7 (2)	C(13)C(14)C(15)	119·5 (2)
C(10)C(13)O(3a)	123·6 (2)	124·6 (2)	C(14)C(15)C(16)	121·4 (3)
C(10)C(13)O(3b)	113·5 (2)	111·9 (2)	C(1)C(16)C(15)	118·2 (3)
C(13)O(3b)C(14)	116·5 (2)	116·4 (2)		
C(15)O(4b)C(16)	116·5 (2)	116·0 (2)		

Table 4. *Interplanar angles* (°) *for compounds* (5) *and* (6)

E.s.d.'s 0·2–0·8°.

Planes*	(5*a*)	(5*b*)	(6)
1–2	108·0	107·0	103·7
1–3	156·2	156·1	156·1
2–3	95·8	96·8	100·2
2–4	161·4	163·0	173·5
4–5	110·4	110·0	106·4
4–6	97·0	98·2	100·6
5–6	152·6	151·7	152·9
1–7	175·0	178·3	178·2
1–8	67·3	14·4	—
1–9	21·6	78·7	—
8–9	60·9	76·0	—

* 1 = C(1)C(2)C(9)C(10); 2 = C(2)C(3)C(8)C(9); 3 = C(2)C(9)C(12); 4 = C(3)C(4)C(7)C(8); 5 = C(4)C(5)C(6)C(7); 6 = C(4)C(11)C(7); 7 = C(1)C(10)C(13)C(15) or C(1)C(10)C(13)C(16); 8 = C(1)C(15)-O(4a)O(4b); 9 = C(10)O(4a)O(4b)C(13).

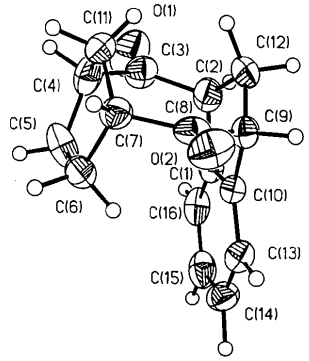

Fig. 1. Drawing of compound (5*a*). Thermal ellipsoids are drawn at the 35% probability level and H atoms are represented by spheres of arbitrary size.

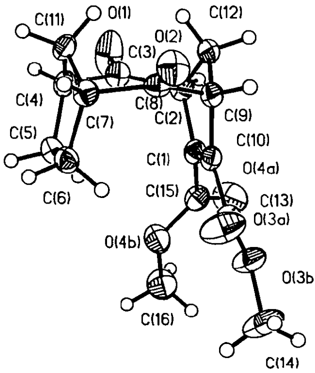

Fig. 2. Drawing of compound (6). Thermal ellipsoids are drawn at the 35% probability level and H atoms are represented by spheres of arbitrary size.

for X-ray Crystallography, 1974). Table 1 lists atomic positional parameters for compound (6). Tables 2–4 list bond lengths, valence angles and interplanar angles, respectively.*

Discussion. The structures of compounds (5) and (6) are shown in Figs. 1 and 2. The corresponding distances around the ring systems in the two independent molecules of (5) are statistically equivalent. The orientations of the $-CO_2CH_3$ groups differ in the two molecules; this leads to different degrees of conjugation with the C(1)—C(10) double bond and to variations in the side-chain bond lengths. The distances in compound (6) are not as internally consistent, which may reflect the neglect of absorption. The benzene ring imparts different geometrical constraints at C(1)—C(10) leading to some difference between (5) and (6). The C(1)—C(10) double-bond systems deviate slightly from planarity, with interplanar angles of 175·0 (5), 178·3 (5) and 178·2 (5)° in (5*a*), (5*b*) and (6), respectively; however, molecular-mechanics calculations (Allinger & Yuh, 1980) predict deviations of less than 1°. The central double-bond system in *syn*-sesquinorborenes deviates from planarity by 16–22° (Watson, 1983) and is readily oxidized to the diketone. The eight-membered ring of the diketone exhibits a flattened crown conformation which maintains the general shape of the parent compound, *i.e.* the interplanar angles between C(2)C(3)C(8)C(9) and C(3)C(4)C(7)C(8) are 161·4 (5), 163·0 (5) and 173·5 (5)° for (5*a*), (5*b*) and (6), respectively. The H(11)···H(12) separations between methano bridge H atoms range from 1·91 to 2·00 Å for the three compounds. These short contacts prevent the carbonyl system from becoming planar. The methano bridges might be expected to bend away from each other to minimize this interaction and allow the central portion of the eight-membered ring to flatten; however, the interplanar angles 2–3 and 4–6 (see Table 4) range from 95·8 to 100·6° while the angles 1–3 and 5–6 range from 151·2 to 156·2°. The 1–3 angles are consistently larger than the 5–6 angles indicating possible repulsion by the π system of the C(1)—C(10) bond (the ethylene bridge) or torsional interactions *via* substituents on C(1)—C(10) (Watson, 1983). The calculated values for 2–3 and 4–6 are 96·1 and 97·7° while those for 1–3 and 5–6 are 156·2 and 151·5°, respectively.

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* Lists of structure factors, H-atom parameters and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43744 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3-Methyladenine Hydrochloride

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Abstract. $C_6H_8N_5^+.Cl^-$, $M_r = 185·62$, monoclinic, $P2_1$, $a = 8·656$ (5), $b = 5·321$ (1), $c = 9·500$ (4) Å, $\beta = 108·13$ (4)°, $V = 415·9$ (3) Å^3, $Z = 2$, $D_m = 1·475$ (5), $D_x = 1·482$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 3·74$ mm^{-1}, $F(000) = 192$, $T = 298$ K, $R = 0·034$ for 689 observed reflections. The 3-methyladenine molecule

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