in a flattened synperiplanar conformation. The average N(1)—C bond length, 1·438 (3) Å, is shorter than the average N(2)—C bond length, 1·507 (3) Å, and the average C—N(1)—C angle, 117·5 (2)°, is larger than the average C—N(2)—C angle, 112·7 (2)°. N(1) appears to be hybridized between sp^2 and sp^3 as a result of the competition between the stability that would be achieved by participation of the N(1) lone pair in the aromatic π system and the strain imposed on the hetero ring by the long S—C bonds which force the ring to exist in a boat conformation. This phenomenon has been observed in many neuroleptic drug molecules (Martin, Svensson, Bates & Ortega, 1985).

The angle between the benzo ring planes is 134·7° which compares favorably to the fold angle in other structurally similar compounds [*e.g.* promethazine, 141° (Marsau & Busetta, 1973); thiazinamium methyl sulfate, 136° (Marsau & Cam, 1973); diethazine, 136° (Marsau, 1971); isothiazine, 140° (Marsau & Calas, 1971) and 10-[di(*n*-propyl)aminoethyl]phenothiazine, 139·1° (Southall, Malmstrom & Klein, 1987)].

This research was partially supported by the National Institutes of Health, MBRS program (RR08008) and the donors of the Petroleum Research Fund administered by the American Chemical Society. We thank Dr E. D. Stevens and the University of New Orleans for allowing us to collect data on their instrument and Lisa C. Southall for her technical assistance.

References

FRENZ, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
KLEIN, C. L. & CONRAD, J. M. (1986). *Acta Cryst.* C**42**, 1083–1085.
KLEIN, C. L., CONRAD, J. M. & MORRIS, S. A. (1985). *Acta Cryst.* C**41**, 1202–1204.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
MARSAU, P. (1971). *Acta Cryst.* B**27**, 42–51.
MARSAU, P. & BUSETTA, B. (1973). *Acta Cryst.* B**29**, 986–991.
MARSAU, P. & CALAS, M.-R. (1971). *Acta Cryst.* B**27**, 2058–2062.
MARSAU, P. & CAM, Y. (1973). *Acta Cryst.* B**29**, 980–986.
MARTIN, A. R., SVENSSON, A., BATES, R. B. & ORTEGA, R. B. (1985). *Acta Cryst.* C**41**, 1804–1807.
SOUTHALL, L., MALMSTROM, M. C. & KLEIN, C. L. (1987). *Acta Cryst.* C**43**, 62–64.
TOLLENAERE, J. P., MOEREELS, H. & RAYMAEKERS, L. A. (1979). *Atlas of the Three-Dimensional Structure of Drugs*. New York: Elsevier/North-Holland Biomedical Press.

Acta Cryst. (1987). C**43**, 1739–1742

Structure of 1-Acetoxy-1,2-dihydrothiazolo[3,2-*a*]quinolinium Perchlorate

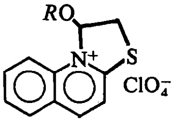
BY CHARLES L. BARNES, OSVALDO COX, LORNA RAMIREZ AND LUIS A. BERNARD

Department of Chemistry, University of Puerto Rico, Rio Piedras, Puerto Rico 00931, *USA*

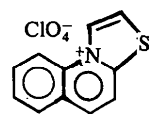
(*Received* 24 *November* 1986; *accepted* 26 *March* 1987)

Abstract. $C_{13}H_{12}NO_2S^+.ClO_4^-$, $M_r = 345·8$, monoclinic, $P2_1/c$, $a = 11·108$ (5), $b = 8·236$ (4), $c = 15·723$ (4) Å, $\beta = 94·59$ (3)°, $V = 1433$ (2) Å^3, $Z = 4$, $D_x = 1·603$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 4·3$ cm^{-1}, $F(000) = 712$, $T = 298$ K, $R = 0·051$ for 1996 observed reflections. The thiazolium ring is a distorted envelope, with bond lengths indicating delocalization through the N—C—S system. The ten atoms of the two fused six-membered rings are coplanar to within 0·015 Å. There is no possibility of hydrogen bonding, and stacking interactions of the aromatic fused ring systems dominate the packing motif.

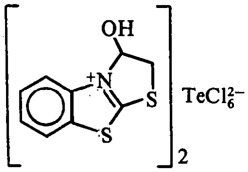
Introduction. 1-Acetoxy-1,2-dihydrothiazolo[3,2-*a*]quinolinium perchlorate (1*a*) was prepared as part of our continuing interest in the synthesis and biological activity of benzothiazolo[3,2-*a*]quinolinium salts (Cox, Jackson, Vargas, Baez, Colon, Gonzalez & de Leon, 1982; Cox, Jackson, Rivera & Ramirez, 1985). Preliminary biological studies (Ramirez, Morin, Cox & Escalona, 1984) on the effect of (1*b*) on muscular contraction were conducted using isolated frog skeletal muscle (*rectus abdominus*). The muscle response in the presence of (1) was compared with that of 10 n*M* acetylcholine (Ach). Experiments with the selective Ach blocker α-bungarotoxin indicate that (1*b*) effects a strong muscular contraction in the presence of this toxin. Therefore it is concluded that (1*b*) does not activate Ach receptors. In order to complete a structure–activity study of this series we had to prepare thiazolo[3,2-*a*]quinolinium perchlorate (2). The reported synthesis of (2) (Bradsher & Lohr, 1967) involves the dehydration of the 1,2-dihydro-1-hydroxythiazolo[3,2-*a*]quinolinium ion derivative (1*b*), prepared by the *in situ* syntheses and acid-catalyzed hydrolysis of α-(2-quinolylthio)acetaldehyde ethyl acetal. A reinvestigation of the mechanistic aspects of this synthetic sequence led us to isolate cations (1*b*) and (1*c*) as their perchlorate salts. In view of the interesting biological properties displayed by (1*b*) (Ramirez *et al.*, 1984) we decided to determine the single-crystal X-ray structure of its acetate derivative (1*a*).



(1*a*), *R* = Ac
(*b*), *R* = H
(*c*), *R* = Et



(2)



(3)

Experimental. The title compound was prepared by the acetylation of (1*b*). A solution of 150 mg (0·5 mmol) of (1*b*) in 0·5 ml of acetic anhydride and 3 drops of dry pyridine was stirred at room temperature for 2 h. The precipitated white solid was collected by vacuum filtration and recrystallized from acetonitrile to afford (1*a*) as white crystals, m.p. 503–505 K. Analysis calcd for $C_{13}H_{12}NO_6SCl$: C 45·15, H 3·47, N 4·05%; found: C 45·23, H 3·51, N 4·02%. IR (KBr): 3040·0, 2990·2, 1763·6, 1616·4, 1595·1, 1571·2, 1510·5, 1457·4, 1436·4, 1385·9, 1372·5, 1326·6, 1209·8, 1158·3, 1075·9, 1015·9, 983·2, 941·8, 832·1, 765·8, 621·9, 600·8, 458·8 cm^{-1}. ^{1}H NMR (90 MHz, Me_2SO-d_6): 9·10 (*m*, 6H), 4·32–4·06 (*m*, 3H), 2·11 (*s*, 3H). ^{13}C NMR (22·5 MHz, Me_2SO-d_6): 168·6 (*s*), 168·0 (*s*), 146·9 (*s*), 136·3 (*d*), 135·7 (*d*), 130·7 (*d*), 128·7 (*d*), 126·2 (*s*), 118·8 (*d*), 117·5 (*d*), 87·7 (*d*), 35·8 (*t*), 20·5 (*q*). Crystals grown by slow evaporation from acetonitrile; pale-yellow plate, 0·1 × 0·3 × 0·5 mm; Enraf–Nonius CAD-4 diffractometer; 24 reflections with $10 < \theta < 15°$, Mo $K\bar{\alpha}$, used to refine cell constants; systematic absences: $0k0$, $k = 2n+1$, $h0l$, $l = 2n+1$; no absorption correction applied; $2\theta_{max} = 53°$; $0 < h < 13$, $-10 < k < 0$, $-19 < l < 19$; three standard reflections measured after every 3600 s of X-ray exposure showed no deterioration; 2962 unique reflections, 1996 observed $[I > 2\sigma(I)]$; structure solved by direct methods with *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982); function minimized during refinement was $w(|F_o| - |F_c|)^2$, with $w = 1/\sigma(F_o)^2$; H atoms placed at calculated positions with fixed temperature factors, updated after each cycle of refinement but not refined; non-H atoms refined anisotropically. $R = 0·051$, $wR = 0·057$, $S = 1·76$; $(\Delta/\sigma)_{max}$ in final cycle 5%, av. 3%; final difference-map max., min. = 0·3 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); all calculations performed with *SDP* (Frenz, 1985).

Table 1. *Positional parameters and their e.s.d.'s*

	x	*y*	*z*	B_{eq}(Å^2)
Cl	0·20076 (9)	0·4327 (1)	0·29067 (6)	4·27 (2)
S(3)	0·69228 (8)	0·3713 (1)	0·23111 (6)	3·80 (2)
O(1)	0·8015 (2)	0·3567 (3)	0·0371 (1)	3·21 (5)
O(2)	1·0000 (2)	0·3385 (4)	0·0473 (2)	7·28 (9)
O(3)	0·3253 (3)	0·3929 (4)	0·3015 (2)	7·94 (9)
O(4)	0·1825 (3)	0·5341 (4)	0·2180 (2)	7·32 (9)
O(5)	0·1359 (3)	0·2865 (4)	0·2797 (3)	8·6 (1)
O(6)	0·1673 (4)	0·5131 (5)	0·3630 (2)	12·1 (1)
N(10)	0·6911 (2)	0·5529 (3)	0·0980 (2)	2·57 (5)
C(1)	0·8122 (3)	0·4788 (4)	0·1020 (2)	2·98 (7)
C(2)	0·8377 (3)	0·4097 (4)	0·1908 (2)	3·64 (7)
C(3A)	0·6220 (3)	0·5028 (4)	0·1589 (2)	2·79 (6)
C(4)	0·5012 (3)	0·5530 (4)	0·1588 (2)	3·17 (7)
C(5A)	0·5268 (3)	0·7072 (4)	0·0308 (2)	2·70 (6)
C(5)	0·4561 (3)	0·6523 (4)	0·0964 (2)	3·11 (7)
C(6)	0·4816 (3)	0·8100 (4)	−0·0357 (2)	3·38 (7)
C(7)	0·5534 (3)	0·8564 (4)	−0·0978 (2)	3·80 (8)
C(8)	0·6718 (3)	0·8017 (4)	−0·0960 (2)	3·79 (7)
C(9A)	0·6482 (3)	0·6551 (4)	0·0318 (2)	2·58 (6)
C(9)	0·7201 (3)	0·7023 (4)	−0·0323 (2)	3·40 (7)
C(11)	0·9068 (3)	0·2943 (5)	0·0139 (2)	3·82 (8)
C(12)	0·8895 (4)	0·1748 (5)	−0·0557 (3)	5·3 (1)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Discussion. Final positional parameters are given in Table 1.* Bond distances and angles are listed in Table 2. The lengths of bonds to N(10) and S(3) are similar to those seen in bis(2,3-dihydro-3-hydroxythiazolo[2,3-*b*]benzothiazolium) hexachlorotellurate(IV) (3) (von Deuten, Schnabel & Klar, 1980), which is one of very few other dihydrothiazolium structures with a tertiary N atom. In that structure, the bonds to the N atom equivalent to those to C(1), C(3*a*) and C(9*a*) averaged 1·486, 1·337 and 1·401 Å for the three independent organic cations, while the bonds to S equivalent to those to C(2) and C(3*a*) averaged 1·837 and 1·685 Å. The shortening of the S(3)—C(3*a*) bond relative to the S(3)—C(2) bond indicates delocalization through the N—C—S system and has been observed in the structure cited above as well as in 2-amino-4,5-dihydro-3*H*$^+$-1,3-thiazolium trichlorocadmate(II) (Kubiak, Głowiak & Kozłowski, 1983) and bis(2-amino-4,5-dihydro-3*H*$^+$-

---

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, thermal and positional parameters for H atoms, and least-squares-plane tables have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43927 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

Cl	O(3)	1·419 (3)	C(1)	C(2)	1·514 (5)
Cl	O(4)	1·416 (3)	C(3A)	C(4)	1·404 (4)
Cl	O(5)	1·406 (3)	C(4)	C(5)	1·344 (4)
Cl	O(6)	1·391 (4)	C(5A)	C(5)	1·419 (4)
S(3)	C(2)	1·810 (4)	C(5A)	C(6)	1·406 (4)
S(3)	C(3A)	1·712 (3)	C(5A)	C(9A)	1·414 (4)
O(1)	C(1)	1·430 (4)	C(6)	C(7)	1·365 (5)
O(1)	C(11)	1·354 (4)	C(7)	C(8)	1·388 (5)
O(2)	C(11)	1·181 (4)	C(8)	C(9)	1·369 (5)
N(10)	C(1)	1·475 (4)	C(9A)	C(9)	1·392 (5)
N(10)	C(3A)	1·338 (4)	C(11)	C(12)	1·473 (5)
N(10)	C(9A)	1·393 (4)			

O(3)	Cl	O(4)	108·0 (2)	N(10)	C(3A)	C(4)	120·8 (3)
O(3)	Cl	O(5)	107·6 (2)	C(3A)	C(4)	C(5)	118·7 (3)
O(3)	Cl	O(6)	109·5 (2)	C(5)	C(5A)	C(6)	123·1 (3)
O(4)	Cl	O(5)	111·5 (2)	C(5)	C(5A)	C(9A)	118·5 (3)
O(4)	Cl	O(6)	110·4 (2)	C(6)	C(5A)	C(9A)	118·3 (3)
O(5)	Cl	O(6)	109·9 (2)	C(4)	C(5)	C(5A)	122·0 (3)
C(2)	S(3)	C(3A)	91·7 (2)	C(5A)	C(6)	C(7)	120·4 (3)
C(1)	O(1)	C(11)	115·7 (2)	C(6)	C(7)	C(8)	120·1 (3)
C(1)	N(10)	C(3A)	114·6 (2)	C(7)	C(8)	C(9)	121·6 (3)
C(1)	N(10)	C(9A)	122·5 (2)	N(10)	C(9A)	C(5A)	117·4 (3)
C(3A)	N(10)	C(9A)	122·6 (2)	N(10)	C(9A)	C(9)	121·9 (3)
O(1)	C(1)	N(10)	103·7 (2)	C(5A)	C(9A)	C(9)	120·6 (3)
O(1)	C(1)	C(2)	113·1 (3)	C(8)	C(9)	C(9A)	118·9 (3)
N(10)	C(1)	C(2)	107·3 (3)	O(1)	C(11)	O(2)	120·6 (3)
S(3)	C(2)	C(1)	106·4 (2)	O(1)	C(11)	C(12)	112·9 (3)
S(3)	C(3A)	N(10)	114·3 (2)	O(2)	C(11)	C(12)	126·5 (3)
S(3)	C(3A)	C(4)	124·9 (2)				

C(3A)	S(3)	C(2)	C(1)	−20·2 (3)	C(1)	N(10)	C(3A)	S(3)	3·1 (3)
C(2)	S(3)	C(3A)	N(10)	10·6 (3)	N(10)	C(1)	C(2)	S(3)	24·4 (3)
C(3A)	N(10)	C(1)	C(2)	−18·5 (3)	C(3A)	N(10)	C(1)	O(1)	101·4 (3)

Numbers in parentheses are e.s.d.'s in the least-significant digits.

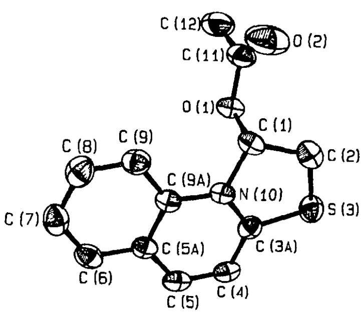


Fig. 1. Perspective view (*ORTEP*, Johnson, 1965) of the organic cation giving the numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

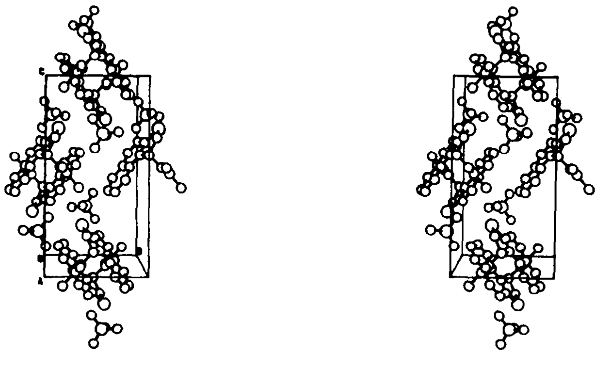


Fig. 2. Stereoview (*PLUTO*, Motherwell, 1976) of the packing down the *a* axis.

1,3-thiazolium) tetrachlorozincate(II) (Kubiac & Głowiak, 1984) and in the uncharged 2,3,6,7-tetrahydro-4*H*-thiazolo[3,2-*a*]*s*-triazin-2-on-4-thione (Flippen, 1974).

A perspective view of the molecule is given in Fig. 1. The ten atoms of the two fused six-membered rings are coplanar to within 0·015 Å.* The thiazolium ring is a distorted envelope with C(2) at the flap, as indicated by the torsion angles given in Table 2. The acetoxy side chain is quasi-axial and *trans* with respect to the displacement of C(2) in the five-membered ring.

Fig. 2 gives a stereoview of the packing. There is no possibility of hydrogen bonding, and stacking interactions of the aromatic fused ring systems dominate the packing motif. A number of close contacts result from this stacking, those less than van der Waals radii sums being: O(2)···C(9), 3·154 (5) Å (molecule at $2-x$, $1-y$, $-z$); C(3*a*)···C(6), 3·370 (5) Å and C(5)···C(9A), 3·382 (5) Å (molecule at $1-x$, $1-y$, $-z$); C(6)···C(6), 3·339 (5) Å (molecule at $1-x$, $2-y$, $-z$).

There is no clear disorder of the perchlorate anion, though the thermal parameters of the O atoms are larger than those of the other atoms of the structure. There is a weak attractive interaction between O(4) of the perchlorate anion and S(3) of the cation (at $1+x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$). The approach distance is 3·179 (4) Å, while the sum of the van der Waals radii is 3·30 Å (Taylor & Kennard, 1982). Other contacts between the perchlorate and the cation less than the sum of the van der Waals radii are: O(5)···C(1), 3·170 (5) Å and O(5)···C(2), 3·148 (5) Å (at $1-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$) and O(6)···C(11), 3·170 (5) Å (at $1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$).

The authors wish to acknowledge support from NIH grant #RR08102–15 and from the FIPI program of the University of Puerto Rico.

References

Bradsher, C. K. & Lohr, D. F. Jr (1967). *J. Heterocycl. Chem.* **4**, 71–74.

Cox, O., Jackson, H., Rivera, L. A. & Ramirez, L. (1985). 10th Int. Congr. Heterocycl. Chem. Univ. of Waterloo, Ontario, Canada, Poster P5–131.

Cox, O., Jackson, H., Vargas, V. A., Baez, A., Colon, J. I., Gonzalez, B. C. & de Leon, M. (1982). *J. Med. Chem.* **25**, 1378–1381.

Deuten, K. von, Schnabel, W. & Klar, G. (1980). *Cryst. Struct. Commun.* **9**, 761–770.

---

* See previous footnote.

Flippen, J. L. (1974). *Acta Cryst.* B**30**, 1123–1125.

Frenz, B. A. (1985). *Enraf–Nonius Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, Holland.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Kubiak, M. & Głowiak, T. (1984). *Acta Cryst.* C**40**, 2039–2041.

Kubiak, M., Głowiak, T. & Kozłowski, H. (1983). *Acta Cryst.* C**39**, 1637–1639.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

Motherwell, W. D. S. (1976). *PLUTO.* Program for plotting crystal and molecular structures. Univ. of Cambridge, England.

Ramirez, L., Morin, S., Cox, O. & Escalona, G. (1984). Abstr. Papers 19th Am. Chem. Soc. Junior Tech. Meet., Univ. of Puerto Rico, Rio Piedras, Puerto Rico, p. 88.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Acta Cryst. (1987). C**43**, 1742–1745

The Relative Configuration of 8-[(2,2-Dimethyl-3-oxocyclohexyl)methoxymethyl]-1-methylbicyclo[4.2.0]octan-7-one

By Elmer O. Schlemper

Department of Chemistry, University of Missouri–Columbia, Columbia, Missouri 65211, *USA*

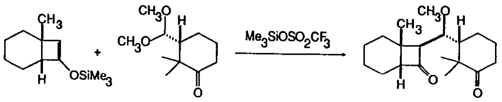
and Madelene M. Nikaido and Glenn R. Clark

Department of Chemistry, Reed College, Portland, Oregon 97202, *USA*

(*Received* 26 *July* 1986; *accepted* 7 *April* 1987)

Abstract. $C_{19}H_{30}O_3$, $M_r = 306{\cdot}45$, triclinic, $P\bar{1}$, $a = 10{\cdot}902\,(2)$, $b = 11{\cdot}251\,(2)$, $c = 7{\cdot}924\,(2)$ Å, $\alpha = 105{\cdot}69\,(2)$, $\beta = 106{\cdot}63\,(2)$, $\gamma = 75{\cdot}70\,(2)^\circ$, $V = 881{\cdot}2\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}155\,(1)$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}7107$ Å), $\mu = 0{\cdot}71$ cm^{-1}, $F(000) = 336$, $T = 295\,(1)$ K, final discrepancy factor $R_F = 3{\cdot}9\%$ for 1550 unique reflections above $2\sigma(I)$ in the range $4 < 2\theta < 45^\circ$. The title compound is the methyl ether of a *threo* aldol; the methyl group of the 8-methylbicyclo[4.2.0]octan-6-one ring system and the chain attached to C3 of the 2,2-dimethylcyclohexan-1-one are equatorial to their respective cyclohexane rings. The puckering of the cyclobutanone ring is indicated by the dihedral angle of 29·9 (2)° between the planes defined by the atoms C5—C6—C7 and C5—C8—C7 of the 8-methylbicyclo[4.2.0]octan-6-one ring system. The bond angles and distances are normal.

Introduction. The single-crystal X-ray diffraction study reported herein demonstrates that the principal crystalline product of the trimethylsilyl triflate-mediated condensation (Murata, Suzuki & Noyori, 1980) of the cyclobutanone silyl enol ether (2) with the aldehyde dimethyl acetal (3) results from mutual kinetic resolution in the *same* sense, *via* a transition state of the *same* relative topicity (Prelog & Helmchen, 1982; Seebach & Prelog, 1982; Seebach & Golinski, 1981), as that observed in the reaction of the enolate with the aldehyde in the presence of zinc chloride (Fair, Clark & Nikaido, 1985).



(2) (3) (1)

The cyclobutanone silyl enol ether (2) was prepared by trapping the corresponding enolate with chlorotrimethylsilane. The starting material was the known 7,7-dichloro-1-methylbicyclo[4.2.0]octan-6-one (Krepski & Hassner, 1978; Bak & Brady, 1979), which was reductively monodechlorinated using zinc in acetic acid following the procedure of Fleming (Fleming & Au-Yeung, 1981) to provide the α-chlorocyclobutanone 7-chloro-1-methylbicyclo[4.2.0]octan-6-one. Reduction of the α-chlorocyclobutanone with dimethyl(copper)lithium in ether (Clark, Lin & Nikaido, 1984) followed by trapping with chlorotrimethylsilane (Stork & Hudrlik, 1968; House, Czuba, Gall & Olmstead, 1969) in the presence of hexamethylphosphoric triamide produced the silyl enol ether (2) (74%).

The dimethyl acetal (3) was prepared from the corresponding aldehyde. Thus, the copper-catalyzed 1,4-addition of vinylmagnesium bromide (House, Chu, Phillips, Sayer & Yau, 1977) to 2-methyl-2-cyclo-