Table I. ESR Spectral Data of Boronitroxides $RN(O\cdot)BH_3^-M^+$

reducing agent	nitroso compd[a]	a_N	a_B	$a_{H(D)}$	g value
$NaBH_4$[b]	MNP	14.1[c]	5.7[c]	12.7[c]	2.0057
KBH_4	MNP	13.7	5.6	12.6	2.0057
$NaBD_4$	MNP	14.0	5.7	1.9	2.0057
KBH_4[d]	MNP	13.9	5.5[e]	12.6	2.0059
			1.8		
$NaBH_4$	ND	13.4	5.9	12.6	2.0057
KBH_4	ND	13.1	5.9	12.4	2.0058
$NaBD_4$	ND	13.4	5.9	1.9	2.0059
$NaBH_4$	2,3,4,5,6-MeNB	13.4	6.1	13.0	2.0057
$NaBH_4$	NB-d_5	9.4	4.8	9.9	2.0054
$NaBH_4$	2,3,4,5,6-OMeNB	12.6	5.7	12.1	2.0057
$NaBD_4$	2,3,4,5,6-OMeNB	12.6	5.7	1.8	2.0059
$NaBH_4$	t-Bu_3NB-d_2	12.7	6.1	11.6	2.0057
$NaBD_4$	t-Bu_3NB-d_2	12.7	6.1	1.8	2.0059

[a] MNP, 2-methyl-2-nitrosopropane; ND, nitrosodurene; 2,3,4,5,6-MeNB; 2,3,4,5,6-pentamethylnitrosobenzene; NB-d_5; 2,3,4,5,6-pentadeuterionitrosobenzene; 2,3,4,5,6-OMeNB; 2,3,4,5,6-pentamethoxynitrosobenzene; t-Bu_3NB-d_2; 2,4,6-tri-*tert*-butyl-3,5-dideuterionitrosobenzene. [b] Unless otherwise noted, all reactions were carried out at 25 °C in DMF with RNO (0.1 M) + MBH_4 (0.2-0.8 M). Similar results are observed for aromatic nitroso compounds in DME, Me_2SO, HMPA-THF (1:1), acetonitrile, pyridine, and ethylenediamine. [c] Hyperfine splitting constants are given in gauss. [d] MNP (0.1 M), KBH_4 (0.2 M), and dicyclohexyl-18-crown-6 (0.2 M) in benzene. [e] Hfs constants for ^{11}B and ^{10}B.

of paramagnetic "ate" complexes.

Notes Added in Proof: During the submission of our manuscript, the reaction of sodium borohydride with alcohols in the presence of nitrosodurene has been described: D. Rehorek, R. Herzschuh, and H. Hennig, *Inorg. Chim. Acta*, **44**, 75 (1980). In our opinion, the ESR spectrum observed and assigned to $C_6H(CH_3)_4N(O\cdot)BH_2$ (a_N = 22.67 G, $a_{H(2H)}$ = 15.63 G, and a_{11B} = 7.08 G) has been misinterpreted. We observed the same kind of spectrum in our experiments by using a mixture of DMF-EtOH. This spectrum has been computer simulated and the following hfs constants give a very good fit with the experimental curve (a_N = 14.39 G, $a_{H(3H)}$ = 14.39 G, and a_{11B} = 6.67 G), thus this spectrum is attributable to $C_6H(CH_3)_4N(O\cdot)BH_3^-Na^+$. We will comment on Rehorek's study in a forthcoming paper.

Acknowledgments. We thank Professors Nathan Kornblum and Jean-Marie Surzur for helpful discussions. This work was supported by the Centre National de la Recherche Scientifique through Grants LA 109 and AI 32538.

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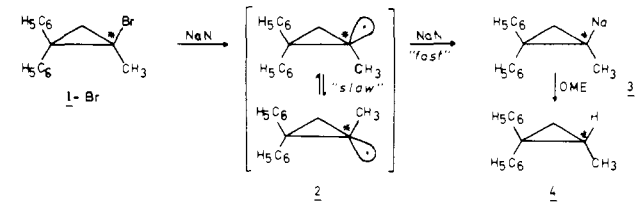
Alternative Pathways in the Reactions of Cyclopropyl Halides with Alkali Metal Naphthalenes

Sir:

From the reaction of sodium naphthalene (NaN) with optically active 1-methyl-2,2-diphenylcyclopropyl bromide (**1**-Br) in dimethoxyethane (DME), leading to 1,1-diphenyl-2-methylcyclopropane (**4**)[1] with 29% net retention of configuration, Jacobus and Pensak concluded[2] that single electron transfer (SET) from a naphthalene radical anion (MN) to a cyclopropyl radical like **2** competes successfully with the ring inversion of **2** (Scheme I).

Scheme I



Scheme II

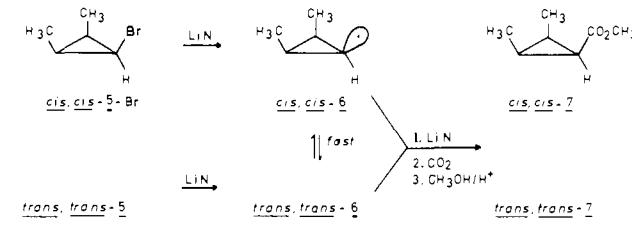


Table I. Ratios of *cis*-**11**/*trans*-**11** from the Reactions of 10-Hal with MN in THF at Room Temperature[a]

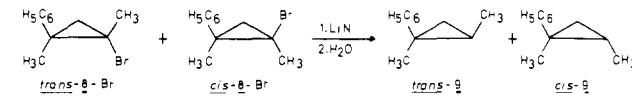
MN	*cis*-**10**-Cl	*cis*-**10**-Cl/*trans*-**10**-Cl 40:60	*cis*-**10**-Br	*cis*-**10**-Br/*trans*-**10**-Br 68:32
LiN	45:55	45:55	45:55	45:55
NaN	40:60	40:60	40:60	40:60
KN	39:61	39:61	39:61	39:61

[a] Ratios were determined by VPC; estimated error ±3%. *Normal addition:* MN (0.9 M) in THF is added dropwise to **10**-Hal (1.0 M) in THF, followed by hydrolysis after 1 min; yields of **11** were 78 ± 14%.[6]

In contrast to these findings, we have reported[3] that 1-bromo-*cis*,*cis*-2,3-dimethylcyclopropane (*cis*,*cis*-**5**-Br) and its isomer *trans*,*trans*-**5**-Br, respectively, reacted with LiN in tetrahydrofuran (THF) after carboxylation and methylation to the identical 21:79 mixture of the carboxy methylates *cis*,*cis*- and *trans*,*trans*-**7**. Consequently, in the case of the secondary cyclopropyl radicals *cis*,*cis*- and *trans*,*trans*-**6** ring inversions are much faster than SET from MN (Scheme II).[4]

The object of this communication is to demonstrate two things: (1) Inversions of free secondary and tertiary cyclopropyl radicals are always faster than bimolecular SET reactions with MN; net retention is not observed. (2) Net retention as observed in the case of **1**-Br does *not* result from a sequence as outlined in Scheme I.[2] In order to show this, we performed the following experiments.

Reaction of 78:22 and 25:75 mixtures, respectively, of *r*-1-bromo-1-methyl-*c*-2-methyl-*t*-2-phenylcyclopropane (*trans*-**8**-Br) and its isomer *cis*-**8**-Br with LiN in THF at 20 °C led after hydrolysis to the identical 45:55 mixture of the cyclopropanes *trans*- and *cis*-**9**.[5] This demonstrates that the equilibration of



(1) The precursor of **4** is the configurationally stable **3**, which is immediately protonated with retention of configuration in solvents like DME and THF: Walborsky, H. M.; Impastato, F. J.; Young, A. E. *J. Am. Chem. Soc.* **1964**, *86*, 3283; and see ref 7.

(2) Jacobus, J.; Pensak, D. *J. Chem. Soc. D* **1969**, 400.

(3) Boche, G.; Schneider, D. R. *Tetrahedron Lett.* **1978**, 2327. A rate constant $k = 1.6 \times 10^9$ L·mol^{-1}·s^{-1} was assumed for the SET reactions of MN with cyclopropyl radicals.[10]

(4) Identical reactions with *cis*- and *trans*-1-bromo-2-phenylcyclopropane, respectively, confirmed this result.[3]

(5) *trans*- and *cis*-**8**-Br were prepared in analogy to Kitaotani, K.; Hiyama, T.; Nozaki, H. *Bull. Chem. Soc. Jpn.* **1977**, *50*, 3288: bp 42-44 °C (10^{-2} torr); ^1H NMR (CCl_4) δ 1.43 (s, CH_3 at C^1 of *trans*-**8**-Br); 1.95 (s, CH_3 at C^1 of *cis*-**8**-Br). The isomer ratio was determined by transformation with *n*-butyllithium into *trans*- and *cis*-**9** (VPC analysis): bp 64-68 °C (10 torr); ^1H NMR (CCl_4) δ 0.74 (br s, CH_3 at C^2 of *trans*-**9**), 1.18 (br s, CH_3 at C^2 of *cis*-**9**).

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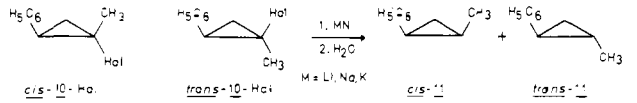
Table II. Percent Net Retention of Configuration of Cyclopropanes 4[8] Formed in the Reactions of 1-Cl, 1-Br, and 1-I (0.9 M) with KN (0.9 M) in THF at 20 °C Followed by Hydrolysis after 1 min

1-Hal	normal addition	inverse addition	25-fold dilution[a]
1-Cl	2.9	2.8	
1-Br	52.8	53.2	58.9
1-I	41.0	40.5	

[a] KN (0.36 M) and 1-Br (0.1 M), normal addition (see Table I). Yields of 4: 73 ± 16%.

the corresponding tertiary cyclopropyl radicals is fast compared to SET from MN, exactly as in the case of the secondary cyclopropyl radicals **6** (Scheme II).

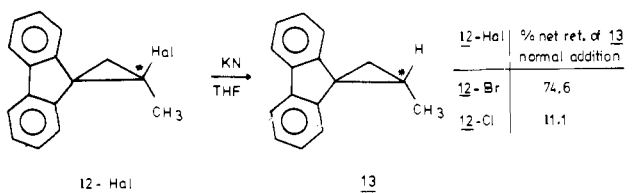
In agreement with the intermediate formation of *free* cyclopropyl radicals in the reactions of **5** and **8** with MN, the amount of cyclopropane isomers **11** formed from **10**-Cl and **10**-Br is *independent of the nature of the halide* (Table I). It is also



apparent from Table I that different gegenions do not influence markedly the rate ratios of the SET reactions of the isomeric cyclopropyl radicals. From these experiments, therefore, one can draw the conclusion that *the rates of inversion of cyclopropyl radicals are generally faster* ($k_{inv} \geq 5 \times 10^9\ s^{-1}$) *than their bimolecular SET reactions with MN.*[3]

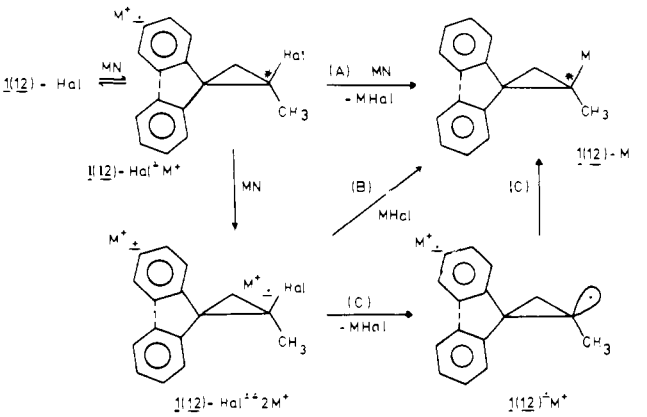
An entirely different situation emerges in the case of cyclopropyl halides like **1**-Br with *good electron-acceptor substituents*. Table II discloses that the net retention of **4** formed in the reactions of **1**-Hal with KN is *strongly dependent on the nature of the halogen.*[7] The dependence of halogen is not in agreement with the free cyclopropyl radical **2** as the intermediate being trapped by KN! Furthermore, inverse addition of reactants as well as the reaction of diluted solutions disproves the assumption of Jacobus and Pensak:[2] if bimolecular SET from KN to **2** indeed would compete successfully with the monomolecular inversion of **2**, increasing concentrations of KN should lead to increasing amounts of net retentions of **4**.[10] Since one can see from Table II that this is not the case, one can even exclude the partitioning of the mechanism outlined in Scheme I.[2]

Reactions of 1-bromo- and 1-chloro-1-methyl-2,2-biphenylenecyclopropane (**12**-Br and **12**-Cl, respectively, 0.1 M)[11] with KN (0.4 M) in THF at 20 °C support the findings with



12-Hal	% net ret. of 13 normal addition
12-Br	74.6
12-Cl	11.1

Scheme III



1-Hal: the resultant 1,1-biphenylene-2-methylcyclopropane (**13**) shows net retention of configuration, and the amount of retention is dependent on the nature of the halogen.[12]

Thus, while the cyclopropyl halides **5**-Br, **8**-Br, and **10**-Hal react with MN via the normal route for naphthalene radical anion reductions of alkyl halides[10b] (Scheme II), **1**- and **12**-Hal must react in a different manner. According to our observations, we propose the following mechanistic alternative (Scheme III). Because of the presence of good (and apparently sufficient[13]) electron-accepting substituents in **1**(**12**)-Hal, **1**(**12**)-Hal$^{\cdot-}$M$^+$ with the extra electron predominantly in the aromatic part of the molecule (and not in the σ^* (C–Hal) bond!) is formed first.[14,15] The second SET could lead directly to **1**(**12**)-M [route (A)]. Alternatively, **1**(**12**)-Hal$^{\cdot-}$2M$^+$ is an intermediate. Formation of **1**(**12**)-M via route (B) corresponds to the *intra*molecular trapping of a C-Hal$^{\cdot-}$M$^+$ species.[14] Via route (C), the cyclopropyl radical **1**(**12**)$^{\cdot-}$M$^+$ would be trapped *intra*molecularly.[16]

In summary, although the exact mechanism which leads to net retention of configuration in the reactions of the cyclopropyl halides **1**-Hal and **12**-Hal with naphthalene radical anions to the cyclopropanes **4** and **13**, respectively, is unknown presently, we have demonstrated that retention of configuration is not due to the successful competition of electron transfer to and inversion of the intermediate cyclopropyl radicals, as suggested earlier.[2] Rather, *the inversion of secondary and tertiary cyclopropyl radicals is much faster* ($k \geq 5 \times 10^9\ s^{-1}$; $\Delta G^{\ddagger} \leq 3.7$ *kcal mol*$^{-1}$) *than SET from naphthalene radical anions.*

(6) *cis*- and *trans*-**10**-Br: See ref 5. *cis*- and *trans*-**10**-Cl were synthesized from *cis*- and *trans*-**10**-Br by means of (1) 2 equiv of *tert*-butyllithium and (2) tosyl chloride: bp 84–88 °C (10 mmHg); ^{1}H NMR (CCl_4) δ 1.30 (s, CH_3 of *cis*-**10**-Cl); 1.68 (s, CH_3 of *trans*-**10**-Cl). See ref 5 for the isomer ratio. *cis*- and *trans*-**11**: Freeman, J. P. *J. Org. Chem.* **1964**, *29*, 1379.

(7) A more pronounced gegenion effect compared to the one reported in Table I is also observed: NaN or LiN led to 48.9% or 29.6%, respectively, net retention in the normal addition to **1**-Br.

(8) The percent net retentions are related to $\alpha_{546} = 150.65°$ for optically pure **4**[9].

(9) Walborsky, H. M.; Aronoff, M. S. *J. Organomet. Chem.* **1973**, *51*, 55.

(10) Compare: (a) Garst, J. F.; Barton, F. E., II *J. Am. Chem. Soc.* **1974**, *96*, 523. (b) Garst, J. F. *Acc. Chem. Res.* **1971**, *4*, 400. (c) Garst, J. F.; Smith, C. D. *J. Am. Chem. Soc.* **1976**, *98*, 1520.

(11) Optically active **12**-Br: see ref 9. Optically active **12**-Cl was prepared in analogy to **12**-Br:[9] mp (CH_3OH) 85–86 °C; $[\alpha]^{29}_D$ +19.6° (*c* 2.218, acetone); ^{1}H NMR (CCl_4) δ 1.93 (s, CH_3), 1.93 and 2.16 (AB system of CH_2, J = 6.5 Hz), 6.88–7.50 (m, 6 aromatic H), 7.56–7.81 (m, 2 aromatic H).

(12) Optically active **13** was prepared in analogy to ref 9 and Walborsky, H. M.; Aronoff, M. S.; Schulman, M. F. *J. Org. Chem.* **1971**, *36*, 1036: $[\alpha]^{25}_D$ +17.8° (*c* 1.468, acetone); $[\alpha]^{23}_{365}$ +73.7° (*c* 1.468, acetone).[20]

(13) With *one* phenyl group, as in the cases of **8**-Br and **10**-Hal (see ref 4), SET is faster into the C–Hal bond.

(14) According to recent calculations (Canadell, E.; Karafiloglou, P.; Salem, L. *J. Am. Chem. Soc.* **1980**, *102*, 855), even with methyl chloride the odd electron is transferred first to a diffuse orbital on carbon, and not to the σ^* (CCl) orbital.

(15) Electron-transfer (ET) reactions of cyclopropanes containing, e.g., phenyl substituents, have been observed earlier: (a) Walborsky, H. M.; Pierce, J. B. *J. Org. Chem.* **1968**, *33*, 4102. (b) Zimmerman, H. E.; Hancock, K. G. *J. Am. Chem. Soc.* **1968**, *90*, 4892. (c) Miller, L. L.; Jacoby, J. B. *Ibid.* **1969**, *91*, 1130. (d) Staley, S. W.; Rocchio, J. J. *Ibid.* **1969**, *91*, 1565.

(16) Comparable reactions have been interpreted recently in a similar manner.[17-19]

(17) Garst, J. F.; Roberts, R. D.; Pacifici, J. A. *J. Am. Chem. Soc.* **1977**, *99*, 3528.

(18) Quirck, R. P.; Murphy, F. H. *Tetrahedron Lett.* **1979**, 301.

(19) Walborsky, H. M.; Murari, M. P. *J. Am. Chem. Soc.* **1980**, *102*, 426.

(20) Both referees pointed to the *halogen* effect (net retention Br > I ≫ Cl) which is essentially reversed if compared with the reactions of **1**-Hal with Li and Mg metal surfaces, and with Na in NH_3 (ref 9 and 15a). However, the results of the *homogeneous* reactions reported in this communication are in agreement with the results of the homogeneous reactions reported in ref 17. Further work from our laboratories (G. Boche and U. Fährmann, unpublished results) also confirms this halogen effect: In the reactions of (*E*)-3-chloro(bromo, iodo)-3-hexenes with LiN in THF at 25 °C, followed by workup with water, the iodide led to a much higher (*Z*)-3-hexene/(*E*)-3-hexene ratio (74:26) than the chloride (22:78) and the bromide (31:69). Again, the corresponding vinyl radical cannot be the common intermediate.

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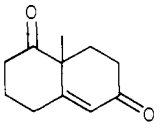
Received March 31, 1980

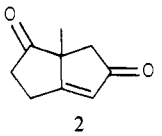
An Enantiodirected Cyclopentenone Annulation. Synthesis of a Useful Building Block for Condensed Cyclopentanoid Natural Products

Sir:

The Wieland-Miescher ketone (**1**) is well-known as a convenient building block for fused six-membered ring terpenes.[1a] Its importance is enhanced by its availability in optically active form.[1b]

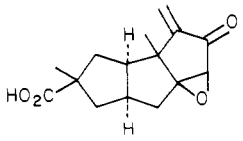


1

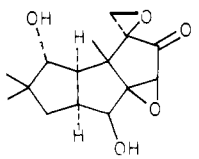


2

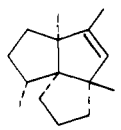
The bicyclo[3.3.0] analogue of the Wieland-Miescher ketone (**1**), enedione **2**, is a potentially versatile intermediate for the synthesis of a growing number of structurally interesting and biologically active fused five-membered ring natural products,[2] some of which are depicted below.



hirsutic acid



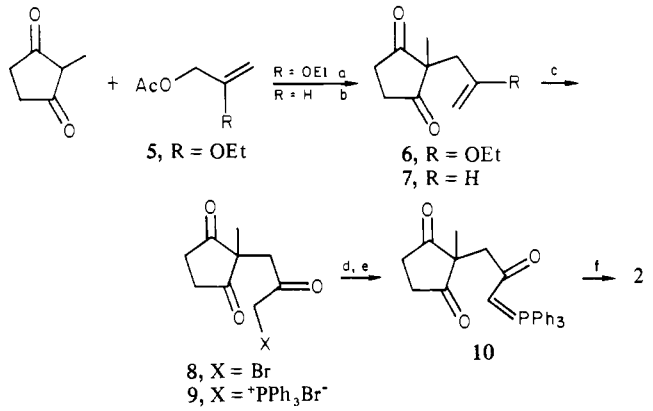
coriolin



isocomene

We now report an expedient synthesis of enedione **2** via a new cyclopentenone annulation sequence whose key features are (1) Pd(0)-directed C-alkylation of 2-methyl-1,3-cyclopentanedione, (2) intramolecular Wittig cyclization,[3b,4] and (3) the adaptability of this route for asymmetric synthesis, an important feature for the synthesis of natural products.

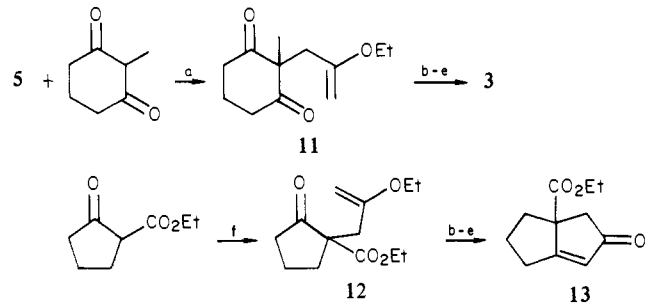
Scheme I



5, R = OEt
6, R = OEt
7, R = H
8, X = Br
9, X = $^{+}PPh_3Br^{-}$
10

[a] $Pd(Ph_3P)_4$ 1-10%, DBU, toluene, 80 °C. [b] $Pd(Ph_3P)_4$ 1%, THF, 25 °C. [c] NBS (2 equiv), H_2O (2 equiv), Me_2SO, 15-25 °C. [d] Ph_3P, benzene, 80 °C. [e] Aqueous K_2CO_3. [f] 40 °C.

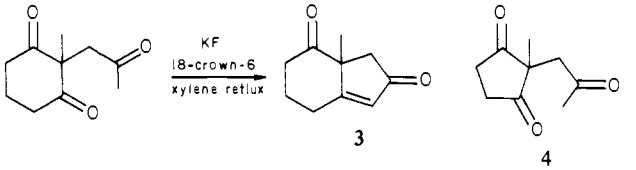
Scheme II



11
12
13

[a] $(Ph_3P)_4Pd$ (7.5%), DBU, THF, 3 days 25 °C. [b] NBS (2 equiv), H_2O (2 equiv), Me_2SO, 15-25 °C. [c] Ph_3P, benzene, 80 °C. [d] aqueous K_2CO_3. [e] 40 °C. [f] $(Ph_3P)_4Pd$ (5%), DBU, THF, 66 °C, 12 h.

Previous methods of cyclopentenone annulation[3a,b] have generally not been used for the synthesis of bicyclo[3.3.0] compounds, and most are not applicable to the synthesis of **2** because of the known tendencies of 2,2-disubstituted-1,3-cyclopentanediones to undergo deacylation reactions.[5] For example, in the more favorable cyclohexanedione series, formation of the cyclopentenone ring was not possible by using a number of standard aldol conditions; however, low yields of enone **3** were available by using fluoride ion catalyzed cyclization.[6] Triketone **4** did not give enone **2** under a variety of aldol conditions and was recovered unchanged from the above fluoride conditions.



3
4

Palladium(0)-catalyzed[7] reaction of 2-methyl-1,3-cyclopentanedione with 2-ethoxy-3-acetoxy-1-propene[8] (**5**) produced

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