Experimental Section⁴

Preparation of Spiro[2,3-benzonorbornadiene-7,1'-cyclopropane].-To a stirred solution of 6.50 g (55 mmol) of isoamvl nitrite in 80 ml of methylene chloride was added 4.6 g (50 mmol) of spiro[4,2-hepta-1,3-diene].⁵ After bringing the solution to reflux, a solution of 7.55 g (55 mmol) of anthranilic acid in 40 ml of anhydrous acetone was added dropwise over 1.5 hr. The reflux was maintained an additional 15 min and then the solvents removed in vacuo. The residue was dissolved in pentane and washed with 10% aqueous sodium hydroxide solution. After drying and removal of solvent, an oil remained whose nmr indicated it was virtually pure. Distillation at 45° (0.1 mm) produced a slightly yellow solid, 5.25 g (63% yield), mp 32-35°. Recrystallization from methanol gave colorless needles, mp 36-39°. A sample collected from vpc⁶ had mp 40-42°. The nmr spectrum showed a multiplet at 6.70-7.20 ppm (6 H), a triplet (J = 1.5 Hz) for 2 H at 3.18 ppm, and a multiplet for 4 H centered at 0.51 ppm. Anal. Calcd for C₁₈H₁₂: mol wt, 168.0938. Found: mol wt, 168.0933.

Irradiation of Spiro[2,3-benzonorbornadiene-7,1'-cyclopropane].—A solution of 200 mg (1.12 mmol) of I in 20 ml of dry degassed ether containing 2.0 mg of acetophenone was irradiated in a Srinivasan–Griffin photochemical reactor fitted with 3500-Å lamps. Following the reaction by thin layer' and vpc⁶ revealed about 45% reaction after 24 hr and 65% reaction after 48 hr. After 48 hr, the irradiation was discontinued, solvent evaporated, and the product isolated after three successive elutions with Skelly B on 1-mm preparative thin layer plates. In this way 60 mg (30% recovery) of starting material and 110 mg (55% yield) of photoproduct II were obtained. The nmr and mass spectra have already been reported (vide supra). Anal. Calcd for C₁₃H₁₂: mol wt, 168.0938. Found: mol wt, 168.0929.

Registry No.---I, 22003-58-3; II, 22003-59-4.

Acknowledgment.—We wish to express our thanks to the National Institutes of Health and the Alfred P. Sloan Foundation for support of this work. We wish to thank R. W. LaRochelle for a sample of spiro-[4.2-hepta-1,3-diene].

(4) All melting points are corrected and all boiling points are uncorrected. Unless otherwise stated magnesium sulfate was employed as a drying agent. The infrared spectra were determined with a Beckmann IR 8 infrared recording spectrophotometer fitted with a grating. The ultraviolet spectra were determined with a Cary recording spectrophotometer, Model 15. The nmr spectra were determined at 60 Mc with a Varian, Model A-60A nmr spectrometer. The mass spectra were obtained with a AEI MS-9 mass spectrometer.

(5) C. F. Wilcox, Jr., and R. B. Craig, J.Amer. Chem. Soc., 53, 3866 (1961).
(6) Vpc analysis performed on 8 ft × 0.25 in. 20% silicone oil 710 on Chromosorb P column.

(7) Thin layer chromatographic analyses and separations employed silica gel PF 254.

Structure and Reactivity in Intramolecular Aldol Condensations of *cis*-Disubstituted Cyclopropanes

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Received February 24, 1969

Our interest in intramolecular aldol condensations originated with the observation of the facile cyclization of 1,2,3,3-tetrachlorocyclopropane-*cis*-1,2-diacetaldehyde (I) to 2,3,4-trichlorobenzaldehyde¹ (III). In-

(1) J. K. Hecht, Tetrahedron Lett., 3503 (1968).

termediate II was not isolated but is assumed to be a logical step on the way to the aromatic system, especially in view of the reported isolation of a similar con-



densation product, V, from the nonchlorinated analog, cyclopropane-cis-1,2-diacetaldehyde² (IV).



The two side chains on these cyclopropanes are cisand thus are in favorable position for aldol condensation. Dialdehyde I could not be obtained in crystalline form, so the analogous dimethyl ketone was synthesized and obtained as a crystalline product. It was found to undergo a similar rearrangement to 2,3,4-trichloro-6-methylacetophenone, and its crystal structure was determined to learn the extent of interaction between the acetone side chains in the ground state. The X-ray crystal structure, which is mentioned later in this paper, will be discussed in detail elsewhere.³

Oxidation of 1,6,7,7-tetrachloro-*cis*-bicyclo [4.1.0]-hept-3-ene (VI) with osmium tetroxide-pyridine complex gave the *cis*-diol VII, which was cleaved with periodic acid to dialdehyde I. Rearrangement of I to aldehyde III was complete in 0.5 hr in 82% overall yield from glycol VII.



Synthesis of the diketone IX was accomplished as shown by oxidation of the Diels-Alder adduct (VIII) of tetrachlorocyclopropene and 2,3-dimethylbutadiene, using the method of Pappas, $et al.^4$



⁽²⁾ F. Serratosa and E. Solé, Anales Real Soc. Espan. Fis. Quim. (Madrid), 63, 865 (1967).

⁽³⁾ F. P. Boer, J. J. Flynn, Jr., and J. K. Hecht, to be published.

⁽⁴⁾ J. J. Pappas, W. P. Keaveney, E. Gancher, and M. Berger, Tetrahedron Lett., 4273 (1966).



(b)

CI

Figure 1.—(a) Possible transition state leading to the fivemembered ring aldol intermediate. The enolic double bond is on the left, while the protonated carbonyl is on the right chain. (b) Possible transition state leading to the seven-membered ring aldol product.

CI

On acid-catalyzed rearrangement of the diketone $(16 \text{ hr at } 100^\circ)$, the major product was the expected 2,-3,4-trichloro-6-methylacetophenone (X), which resulted from aldol condensation followed by aromatization.

A reverse Friedel-Crafts reaction could account for 3,4,5-trichlorotoluene (XI). Such reactions are well known in crowded aromatic systems.⁵ The unsaturated ketone XII was unexpected, but can be readily accounted for by condensation of a methyl group instead of a methylene. Because this ketone would not go to an aromatic system, there was presumably insufficient driving force for opening of the three-membered ring. Our conception of the geometries of reaction of enol with protonated carbonyl is shown in Figure 1a for five-membered ring aldol condensation leading to X and in Figure 1b for the seven-membered ring type

leading to XII. Nucleophilic displacement of chlorine by water would explain the phenolic product XIII.



This compound was assigned the structure shown because its infrared spectrum ($\nu_{max}^{\rm CCL}$ 3540 cm⁻¹) showed the hydroxyl group to be unassociated. The *o*-hydroxyacetophenone produced by displacement of the *ortho* chlorine atom would be expected to have a strongly internally hydrogen bonded hydroxyl. Possibly some diphenols were formed, but these would have been lost in aqueous extractions.

A thermal-ellipsoid type plot of the molecular structure of the diketone as determined by X-ray crystallography is shown in Figure 2.³ A close approach (3.166 Å) was found between a methylene group on one chain and a carbonyl group on the other. This does not appear to be a instance of C-H···O hydrogen bonding. In any case, C-H···O hydrogen bonding has been ruled out by Donohue⁶ in similar situations. Even if there is no attractive interaction between the methylene and carbonyl groups, the crystal structure does show that the ground state of the molecule prefers a conformation which is close to that of a possible transition state for five-membered ring aldol condensation (see Figure 1a).

Experimental Section

1,6,7,7-Tetrachloro-cis-bicyclo[4.1.0]hept-3-ene (VI).⁷—Two capped polymer pressure tubes were each charged with a solution of 20 g (0.11 mol) of tetrachlorocyclopropene, 20 ml of butadiene, and 0.4 g of 2,6-di-t-butyl-p-cresol in 30 ml of benzene and heated in an oil bath at 80° for 118 hr. The tubes were cooled until frozen solid, opened, combined, and concentrated under reduced pressure. The crude product was recrystallized from petroleum ether (bp 30-60°), yielding 38 g (82%) of adduct, mp 51-52°.

1,6,7,7-Tetrachloro-cis-bicyclo [4.1.0] heptane-endo-cis-3,4-diol. —A solution of 0.91 g (0.0039 mol) of 1,6,7,7-tetrachlorocis-bicyclo [4.1.0] hept-3-ene in 15 ml of ether was cooled to 0°. To this solution was added 1.0 g of osmium tetroxide, 0.7 ml of pyridine, and 5 ml of ether. After the mixture was stirred for 2 hr, the temperature rose to about 25° and hydrogen sulfide was bubbled through the solution for 10 min. This mixture stood for 16 hr and was filtered and concentrated. The residue was re-

(6) J. Donohue in "Structural Chemistry and Molecular Biology," A. Rich and N. Davidson, Ed., W. H. Freeman & Co., San Francisco, Calif., 1968, pp 443-465.

(7) The procedure used here is a modification of that described by D. C. F. Law and S. W. Tobey, J. Amer. Chem. Soc., 90, 2376 (1968).

⁽⁵⁾ P. H. Gore in "Friedel-Crafts and Related Reactions," Vol. III, G. A. Olah, Ed., Interscience Publishers, New York, N. Y., 1964, pp 6-7.

crystallized from methylene chloride-hexane, yielding 0.51 g (48%) of glycol, mp 137-139°. An analytical sample, mp 137-139° , was obtained by sublimation at 1 mm with a pot temperature of 120°.

Anal. Caled for $C_7H_8Cl_4O_2$: C, 31.61; H, 3.03; Cl, 53.33. Found: C, 31.51; H, 3.04; Cl, 53.57.

 $1,2,3,3-Tetrachlorocyclopropane-{\it cis-1,2-diacetaldehyde.} - A$ solution of 0.14 g (0.00053 mol) of 1,6,7,7-tetrachloro-cis-bicyclo-[4.1.0] heptane-endo-cis-3,4-diol in 15 ml of ether was added to a solution of 0.12 g (0.00053 mol) of periodic acid ($H_{5}IO_{6}$) in 200 ml of ether. After the mixture had stirred at room temperature for 45 min, it was dried over magnesium sulfate, filtered, and concentrated under reduced pressure, yielding 0.15 g (ca. 100%) of liquid dialdehyde, ν_{max}^{CCli} 1730 cm⁻¹. The nmr spectrum of the dialdehyde showed a doublet at 3.16 ppm (J = 2 cps) and a triplet at 9.78 ppm (J = 2 cps) in the ratio of 2:1. The bis(2,4dinitrophenylhydrazone) (from a similar sample of dialdehyde) had mp 186-188° after two recrystallizations from ethanol-ethyl acetate.

Calcd for C19H14Cl4N8O8: C, 36.56; H, 2.26; Cl, Anal. 22.72; N, 17.96. Found: C, 37.70; H, 3.00; Cl, 22.41; N, 17.46

2,3,4-Trichlorobenzaldehyde.---A solution of 0.15 g of 1,2,3,3tetrachlorocyclopropane-cis-1,2-diacetaldehyde (synthesized from 0.14 g of bicyclic glycol) in 5 ml of a saturated solution of sodium bisulfate in glacial acetic acid was heated at 100° for 0.5 hr. The mixture was quenched with water and extracted with ether. The ether solution was washed with saturated sodium bicarbonate solution until neutral, dried over magnesium sulfate, and concentrated, yielding 0.09 g of 2,3,4-trichlorobenzaldehyde, mp $63-77^{\circ}$. The infrared spectrum of the product was essentially identical with that of an analytically pure sample, mp 84-85° (lit.⁸ mp 86°). The overall yield of 2,3,4-trichlorobenzaldehyde from the bicyclic glycol was 82%.

1,6,7,7-Tetrachloro-3,4-dimethyl-cis-bicyclo[4.1.0]hept-3-ene (VIII).-Two capped polymer pressure tubes were each charged with a solution of 10 g (0.056 mol) of tetrachlorocyclopropene, 10ml of 2,3-dimethylbutadiene, and 0.2 g of 2,6-di-t-butyl-p-cresol in 15 ml of benzene and heated in an oil bath at 80° for 66 hr. The tubes were cooled until frozen solid, opened, combined, and concentrated under reduced pressure. The crude product was recrystallized from pentane, yielding 24.3 g (83%) of adduct, mp 69-71°. An analytical sample, mp 74-75°, was obtained by a second recrystallization from pentane.

Anal. Calcd for $C_{9}H_{10}Cl_{4}$: C, 41.57; H, 3.88; Cl, 54.55. Found: C, 41.34; H, 3.86; Cl, 54.90.

1,2,3,3-Tetrachlorocyclopropane-cis-1,2-diacetone (IX).--A solution of 10.0 g (0.042 mol) of 1,6,7,7-tetrachloro-3,4-dimethylcis-bicyclo[4.1.0] hept-3-ene in a mixture of 170 ml of methylene chloride and 80 ml of methanol was ozonized at 0° for 70 min.9 After dissolved ozone was removed by flushing with nitrogen, 15 ml (0.24 mol) of dimethyl sulfide was added and the mixture was stirred at room temperature for 40 min. The solvents were removed under reduced pressure. The residue was dissolved in methylene chloride and this solution was washed with six 100-ml portions of water and dried over magnesium sulfate. Concentration under reduced pressure and recrystallization of the residue from methylene chloride-hexane gave 7.18 g (64%) of diketone, mp 127-129°, $\nu_{max}^{CHCl_3}$ 1710 cm⁻¹. Anal. Calcd for C₉H₁₀Cl₄O₂: C, 37.02; H, 3.45; Cl, 48.57. Found: C, 36.97; H, 3.46; Cl, 48.81. Rearrangement of 1,2,3,3-Tetrachlorocyclopropane-cis-1,2-di-centers - A solution of 3.0 g (0.0103 mol) of the diketone in 75

acetone — A solution of 3.0 g (0.0103 mol) of the diketone in 75 ml of a saturated solution (ca. 0.7%) of sodium bisulfate in glacial acetic acid was heated in an oil bath at 100° for 16 hr. After cooling, the reaction mixture was dissolved in 400 ml of ether. The ether solution was washed with saturated aqueous sodium bicarbonate solution until neutral, dried over magnesium sulfate, and concentrated, yielding 1.60 g of crude product mix-This mixture was added to 100 ml of pentane, dissolving ture. entirely except for 0.20 g of starting material and a small amount of brown tar. The pentane-soluble fraction (1.27 g) was chromatographed through 40 g of silica gel (packed with benzene), eluted with 0.5% ether in benzene, and collected in 14 50ml fractions. The earliest fractions, having a combined weight



Figure 2.-View of a molecule of 1,2,3,3-tetrachlorocyclopropane-cis-1,2-diacetone. Ellipsoids are used to represent the anisotropic thermal motion of the heavier atoms, while hydrogens are drawn as 0.2-Å spheres. The methyl hydrogens were not are utawin as 0.2-A spheres. The metalyi hydrogens were not actually located in this study. The distances (Å), with standard errors in parentheses, are: C(1)-C(2), 1.528 (11); C(2)-C(3), 1.522 (10); C(3)-C(4), 1.532 (10); C(4)-C(5), 1.486 (10); C(5)-C(6), 1.498 (10); C(4)-C(6), 1.516 (10); C(6)-C(7), 1.511 (10); C(7)-C(8), 1.507 (10); C(8)-C(9), 1.493 (11); C(4)-C(4), 1.740 (4): C(5)-C(4), 1.709 (4): C(5)-C(4), 1.501 (4): C(4)-C(4)1.749 (8); C(5)-Cl(5A), 1.728 (8); C(5)-Cl(5B), 1.761 (8); C(6)-Cl(6), 1.756 (7); C(2)-O(2), 1.200 (9); C(8)-O(8), 1.215 (9); C(7)-O(2), 3.166 (10).

of 1.01 g, contained 84% of 2,3,4-trichloro-6-methylacetophenone (X) (34.8% yield) and 16% of 3,4,5-trichlorotoluene (XI) (8.0% yield). These compounds were separated by gas chromatography on a 2-ft QF-1 column at 150°. Later fractions of the silica gel chromatogram gave 0.08 g (2.7%) of 1,7,8,8-tetrachloro-5-methyl-cis-bicyclo[5.1.0]oct-4-en-3-one (XII) and 0.01 (0.04%) of 2,3-dichloro-4-hydroxy-6-methylacetophenone (XIII). Subsequent runs gave similar yields of these products.

Identification of the Rearrangement Products. 2,3,4-Trichloro-6-methylacetophenone (X) was isolated as a liquid and was purified by gas chromatography as described above. Its infrared spectrum showed a carbonyl band at 1710 cm⁻¹ (CCl₄ solution). Its nmr spectrum (CCl₄) contained singlets at 2.19 (CH₃ attached to phenyl), 2.45 (acetyl CH₃), and 7.22 ppm (aromatic H), in the ratio of 3:3:1.

Anal. Calcd for C₈H₇Cl₈O: C, 45.51; H, 2.97; Cl, 44.79. Found: C, 45.42; H, 3.27; Cl, 44.53.

3,4,5-Trichlorotoluene (XI), mp 44-45° (lit.10 mp 44.5-45.5°), showed singlets in the nmr (CCl₄) at 2.30 (CH₃) and 7.16 ppm (aromatic H) in the ratio of 3:2.

1,7,8,8-Tetrachloro-5-methyl-cis-bicyclo [5.1.0] oct-4-en-3-one (XII), mp 119-120°, was purified by recrystallization from cyclopentane. Its infrared spectrum (in CCl₄) showed a carbonyl band at 1670 cm⁻¹. The nmr spectrum (CDCl_s) had broad peaks at 2.17 (CH₃), 3.32 (allylic CH₂), and 6.10 ppm (vinyl H) in the ratio 3:2:1. The methylene group next to the ketone appeared as a quartet centered at 3.17 ppm, $J_{AB} = 17$ cps (geminal coupling), with an area corresponding to two protons. The ultraviolet spectrum exhibited a maximum absorption at 232 m μ (ϵ 13,000) in methylcyclohexane solution.

Anal. Calcd for C₉H₈Cl₄O: C, 39.45; H, 2.94; Cl, 51.76. Found: C, 39.24; H, 3.31; Cl, 51.63.

⁽⁸⁾ H. C. Brimelow, R. L. Jones, and T. P. Metcalfe, J. Chem. Soc., 1208 (1951).

⁽⁹⁾ Ozonizations were conducted with a Welsbach Model T-23 laboratory ozonator, using oxygen at a rate of about 600 ml/min.

⁽¹⁰⁾ J. B. Cohen and H. D. Dakin, J. Chem. Soc., 1324 (1902).

2,3-Dichloro-4-hydroxy-6-methylacetophenone (XIII), crystallized from methylene chloride-hexane, mp 107-108° showed a strong unassociated hydroxyl absorption in the infrared at 3540 cm⁻¹ (CCl₄). In the nmr (CDCl₃), it showed singlets at 2.20 (acetyl CH₃), 5.68 (phenolic OH), and 6.79 ppm (aromatic H) in the ratio of 3:3:1:1. The hydroxyl proton resonance was

extremely broad (about 1 ppm). Anal. Calcd for $C_{9}H_{8}Cl_{2}O_{2}$: C, 49.34; H, 3.68; Cl, 32.37. Found: C, 49.20; H, 4.25; Cl, 32.18.

X-Ray Structure Determination.³—A needle-shaped crystal of 1,2,3,3-tetrachlorocyclopropane-cis-1,2-diacetone was found to belong to the monoclinic space group $P2_1/c$, with lattice parameters $a = 13.458 \pm 0.010$ Å, $b = 10.932 \pm 0.011$ Å, $c = 9.417 \pm 0.009$ Å, $\beta = 117.20 \pm 0.06^{\circ}$, Z = 4. Intensity data were collected on a Picker automatic diffractometer using Cu Ka radiation, and the structure was refined by full matrix least squares to a conventional reliability index of 6.5% for 1150 reflections above background.

Registry No.-VI, 21473-07-4; VIII, 21473-08-5; IX, 21473-09-6; X, 21472-85-5; XI, 21472-86-6; XII, 21473-10-9; XIII, 21472-87-7; VII, 21473-11-0; 1,2,-3,3 - tetrachlorocyclopropane - cis - 1,2 - diacetaldehyde, 19427-04-4; 1,2,3,3-tetrachlorocyclopropane-cis-1,2-diacetaldehyde bis(2,4-dinitrophenylhydrazone), 19427-06-6.

The Oxygenation of Enamines. Ketonization at the β Position to Give α-Amino Ketones

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Received May 14, 1969

Within the last year, several communications have appeared concerning the reactions of enamines with oxygen. Foote and Huber¹ have reported the quantitative cleavage of the enamine double bond [eq 1

(both R and R' alkyl or aryl groups)] in a dye-sensitized photooxygenation. The reaction of oxygen with enamines from α,β -unsaturated bicyclic ketones produces ene-diones in 65-85% yields.² More recently, the copper-catalyzed oxygenation of enamines has been reported to give cleavage of the double bond in a manner similar to the dye-sensitized process.³ We have studied the oxygenation of enamines containing a vinvl hydrogen at the β position and have found α -amino ketones, products not previously reported in the oxidation of an enamine.

The reaction of 1-di-n-butylamino-1-butene (1) with oxygen in benzene at 25 or 80° gave a mixture of di-nbutylamine, N,N-di-n-butylformamide, and 1-N,N-din-butylamino-2-butanone (2). The latter two account

$$1^{1-Bu}$$
 $(n-Bu)_2NCH_2CCH_2CH_3$ $(n-Bu)_2NCH_2COCH_2CH_3$

(1

for a ca. 55% yield. The products and yields as determined by gas-liquid chromatography are listed in Table I. Di-n-butylamine is generated very likely by hydrolysis of the enamine. The most probable source of the N,N-di-n-butylformamide is cleavage of the enamine double bond, the same type of process found in the sensitized photooxygenation¹ and the copper-catalyzed oxygenation.³

TABLE	Т
TUDDU	T

	25°	80°
Product	$(40.5 hr)^a$	(8 hr) ^a
Di-n-butylamine	10.1	7.2
N,N-Di-n-butylformamide	19.0	29.4
1-N,N-Di-n-butylamino-2-		
butanone (2)	34.3	26.0
Reaction time.		

The di-n-butylamine and the formamide were identified by trapping each from the gas-liquid chromatogram and comparing their infrared spectra with those of authentic materials. Compound 2 was also isolated by trapping and it exhibited an infrared band at 1720 cm^{-1} . A mass spectrogram yielded a molecular ion at m/e 199 and a large peak at m/e 142 indicative of the cleavage shown in eq 2. This type of cleavage has

 $(n-\mathrm{Bu})_2 \mathrm{NCH}_2 \mathrm{COCH}_2 \mathrm{CH}_3 \longrightarrow$

$$(n-\mathrm{Bu})_2 \vec{\mathrm{N}} = \mathrm{CH}_2 + \cdot \mathrm{COCH}_2 \mathrm{CH}_3$$
 (2)
 $m/e \ 142$

been found for α -dimethylaminoacetone.⁴ These data suggested structure 2 and the initial assignment was confirmed by comparison of the infrared spectrum with that of an authentic sample synthesized by the method of Bailey and Keller.⁵

The oxidation of 1-pyrrolidinocyclohexene (3) at 25° in benzene or ethyl acetate proceeded rapidly and with a mild exotherm to give α -pyrrolidinocyclohexanone (4) and 6-oxo-1-pyrrolidinocyclohexene (5) as the only identifiable products. However, some gum formation occurred which made isolation of 4 and 5 difficult. Early in the reaction, before any gum could be observed, a gas-liquid chromatogram revealed the presence of only 3, 4, 5, and the hydrolysis products of 3, pyrrolidine, and cyclohexanone. The products were isolated by trapping from the chromatogram and comparing their infrared spectra with authentic samples. Distillation of the crude dark product and analysis of the distillate by glpc indicated that the yield for 4 and 5 was 9.4 and 7.8%, respectively. A large dark pot residue remained. No products from cleavage of the enamine double bond could be detected in contrast to the cleav-



⁽⁴⁾ F. R. Stermitz and K. D. McMurtrey, J. Org. Chem., 33, 1140 (1968). See also H. Budzikiewicz, C. Djerassi, and D. H. Williams, "Mass Spectrom-etry of Organic Compounds," Holden-Day, Inc., San Francisco, Calif., 1967, p 11.

^{(1) (}a) C. S. Foote and J. Wei-Ping Lin, Tetrahedron Lett., 3267 (1968): (b) J. E. Huber, ibid., 3271 (1968).

⁽²⁾ S. K. Malhotra, J. J. Hostynek, and A. F. Lundin, J. Amer. Chem. (2) S. H. Malmona, S. S. Hossyner, and E. T. E.
Soc., 90, 6565 (1968).
(3) V. Van Rheenen, Chem. Commun., 314 (1969).

⁽⁵⁾ P. S. Bailey and J. E. Keller, J. Org. Chem., 33, 2680 (1968).