243° dec. (lit.[23] m.p. 266° dec.), $\nu_{C=O}^{KBr}$ 1704 cm.$^{-1}$. Since our melting point did not agree with the literature value of this compound which had been prepared by a different method, analytical data were obtained.

Anal. Calcd. for $C_9H_6N_2O_2$: C, 62.07; H, 3.47; N, 16.08. Found: C, 61.99; H, 3.50; N, 15.89.

2,3-Dimethyl-5-quinoxalinecarboxylic Acid (V).—3-Nitro-2-aminobenzoic acid (5.0 g., 0.0274 mole) was suspended in absolute ethanol (150 ml.) and reduced in a Parr hydrogenator using 10% palladium on charcoal (0.5 g.) as the catalyst. The catalyst was removed by filtration and the solution was treated immediately with 200 ml. of 10% aqueous potassium carbonate and diacetyl (6 ml., 0.070 mole). The resulting solution was stirred at room temperature with a magnetic stirrer for 15 min. and then poured into 400 ml. of ice–water. The yield of bronze-colored solid was 65%, m.p. 186–187°, $\nu_{C=O}^{KBr}$ 1698 cm.$^{-1}$.

Anal. Calcd. for $C_{11}H_{10}N_2O_2$: C, 65.34; H, 4.98; N, 13.85. Found: C, 65.23; H, 4.97; N, 14.02.

2,3-Quinoxalinedicarboxylic Acid (II).—This compound was prepared by the procedure described by Hinsberg and König[24]: yield 53%, m.p. 188–189° dec. (lit.[24] m.p. 190°), $\nu_{C=O}^{KBr}$ 1719 cm.$^{-1}$.

Ethyl 2-Quinoxalinecarboxylate (VIII).—This compound was prepared according to the method of Maurer and Boettger[21]: yield 86%, m.p. 83.5–85° (lit.[21] m.p. 85°).

Ethyl 6-Quinoxalinecarboxylate (XII).—This compound was prepared by the method of Birkofer and Widmann[23]: yield 72%, m.p. 70–71° (lit.[23] m.p. 66°).

Ethyl 5-Quinoxalinecarboxylate (X).—5-Quinoxalinecarboxylic acid (1.7 g., 0.01 mole) was dissolved in 75 ml. of anhydrous benzene in a 250-ml., three-necked, round-bottomed flask fitted with a mechanical stirrer, reflux condenser, and silica gel drying tube. Purified thionyl chloride (1.6 g., 0.013 mole) was added to the solution and the reaction mixture was stirred and heated for 12 hr. Absolute ethanol (25 ml.) was added to the solution and heating was continued for 12 hr. The solution was then evaporated to dryness and the residue was extracted with two 100-ml. portions of a 50% mixture of cyclohexane and anhydrous ether. The extracts were treated with anhydrous potassium carbonate and then with anhydrous magnesium sulfate. After the drying agents were removed, the filtrate was reduced to a volume of 20 ml. and 20 ml. of Skellysolve H[25] was added to it. A solid was formed which could not be identified but was definitely not the desired product.

The original residue was extracted again with two 150-ml. portions of anhydrous ether and the extracts were treated with potassium carbonate. The drying agent was removed and the filtrate was reduced in volume until a precipitate formed. This solid was recrystallized from cyclohexane to yield 1% of the desired product, m.p. 215–216°.

Anal. Calcd. for $C_{11}H_{10}N_2O_2$: C, 65.34; H, 4.98; N, 13.85. Found: C, 65.50; H, 4.85; N, 14.04.

Methyl 5-Quinoxalinecarboxylate (XI).—5-Quinoxalinecarboxylic acid (1.0 g., 0.0057 mole) was suspended in 1,2-dimethoxyethane (100 ml.) in a 250-ml. erlenmeyer flask fitted with a magnetic stirrer. The solution was cooled in an ice–water bath and stirred magnetically while diazomethane (0.96 g. 0.023 mole) was added to it. The acid dissolved and the solution was allowed to stand overnight. Decolorizing carbon was added to the reaction mixture. The mixture was heated and the carbon was removed by filtration. The filtrate was concentrated to 20 ml. and treated with Skellysolve H[25] (50 ml.) and evaporation was continued until a solid formed. This product (63%) was recrystallized from cyclohexane, m.p. 73–74°.

Anal. Calcd. for $C_{10}H_8N_2O_2$: C, 63.83; H, 4.28; N, 14.88. Found: C, 63.96; H, 4.42; N, 14.80.

Diethyl 2,3-Quinoxalinedicarboxylate (IX).—This compound was prepared by the method of Chattaway and Humphrey[26]: yield 85%, m.p. 82–83.5° (lit.[26] m.p. 83°).

Ethyl 2-Pyrazinecarboxylate (VI).—This compound was prepared according to the procedure of Shindo[12]: yield 87%, m.p. 48.5–51° (lit.[21] m.p. 52–53°).

Methyl 2-Pyrazinecarboxylate (VII).—The method of Hall and Spoerri[27] was used: yield 72%, m.p. 58.5–61° (lit.[27] m.p. 62°).

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[23] L. Birkofer and A. Widmann, *Ber.*, **86**, 1295 (1953).

[24] O. Hinsberg and F. König, *ibid.*, **27**, 2185 (1894).

[25] Skellysolve H is a petroleum fraction (b.p. 70–74°) obtained from Skelly Oil Co., Kansas City, Mo.

[26] F. D. Chattaway and W. G. Humphrey, *J. Chem. Soc.*, 645 (1929).

[27] S. A. Hall and P. E. Spoerri, *J. Am. Chem. Soc.*, **62**, 664 (1940).

Diimide Reductions Using Potassium Azodicarboxylate

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Use of reagents which generate diimide—at least formally—in the reduction of olefinic bonds is a relatively recent synthetic innovation which would appear to hold much promise in its scope.[2a] Except for a recent communication by van Tamelen,[2b] no limitations to this reaction have been described. To the best of our knowledge no extensive description of the experimental aspects of these reductions using potassium azodicarboxylate (PADA) has yet appeared. In this paper we describe the effects of several experimental variables on the course of the reduction and furnish adequate descriptions of the experimental methods employed. It will become apparent that this paper constitutes a phenomenological description of the behavior of one type of diimide reduction. Because we have not been able to formulate an integrated, self-consistent mechanistic description of this reaction we have chosen to limit our remarks on this latter aspect of the reaction. We also hope to indicate clearly that conclusions which might be drawn from yield data[2b] might need to be modified in light of our results.

Results

Although reductions were routinely performed in a nitrogen atmosphere, expt. 13–15 (Table I) indicate that air has no deleterious effects on the reaction. However, because the reaction is extraordinarily sensitive to the adverse effects of water (*vide infra*), suitable precautions for a dry atmosphere are indicated.

Solvents have an important influence on the reaction. Our work suggests solvents are less effective in the order pyridine > dioxane > dimethyl sulfoxide (DMSO) ~ methanol < ethanol ~ butanol (expt. 1–3, 5, and 13–20). As might be anticipated, the magnitude of the differences in solvent effects increases as the reactivity of the substrate decreases. Thus, azobenzene is reduced virtually quantitatively in the less effective solvents DMSO and methanol; the less

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[1] NASA Predoctoral Fellow, 1963–1965.

[2] (a) See C. E. Miller, *J. Chem. Educ.*, **42**, 254 (1965), for a recent review; (b) E. E. van Tamelen, M. Davis, and M. F. Deem, *Chem. Commun.*, 71 (1965).

TABLE I

REDUCTIONS WITH POTASSIUM AZODICARBOXYLATE[a]

Expt. no.[b]	Substrate	Moles of PADA/ mole of substrate	Solvent	Temp., °C.	Time, hr.[c]	Acid (moles/ mole of PADA)	% yield[d]	Comments
1	$C_6H_5N{=}NC_6H_5$	2.0	DMSO	23	20	HOAc (2.0)	75	Yield based on isolated diacetate
2		2.3	DMSO	23	1.5	HOAc (2.0)	∽100	Based on isolated hydrazobenzene
3		1.4	CH_3OH	23	2.5	HOAc (3.0)	∽100	
4	$C_6H_5CH{=}CHCO_2CH_3$	2.0	DMSO	23	19	HOAc (2.0)	60	
5A		2.5	DMSO	23	20	HOAc (1.6)	73	
5B		2.5	CH_3OH	23	4.5	HOAc (0.81)	80	
6A		2.1	Dioxane	23	19	HOAc (1.6)	93	
6B		2.1	Pyridine	23	34	HOAc (1.6)	96	
7A		2.1	Dioxane	23	14	HOAc (1.6)	84	Acid added in two portions separated by 5.5 hr.
7B		2.1	Dioxane	23	14	HOAc (1.6)	65	Acid added in single portion
8A		2.5	DMSO	23	12	HOAc (1.6)	74	
8B		2.5	DMSO	23	5	H_2O (8.3)	13	PADA was completely consumed
9A		2.1	CH_3OH	23	6	HOAc (1.8)	60	
9B		2.1	CH_3OH	23	6	H_2O (6.0)	57	
10A		2.1	Pyridine	23	16	HOAc (1.6)	>90	
10B		2.1	4% H_2O–pyridine	23	16	HOAc (1.6)	28	H_2O:PADA = 2.6
11A		2.1	Pyridine	23	24	H_2O (5.6)	2	
11B		2.1	Dioxane	23	49	H_2O (7.4) HOAc (0.83)	7	PADA was not completely decomposed for 18 hr. after addition of HOAc, itself added 31 hr. after initiation of reaction
12A		2.1	Dioxane	23	0.5	HOAc (0.8) H_2O (2.8)	1.4	Aqueous acetic acid was added; PADA decomposed before addition was complete
12B		2.1	Dioxane	23	18	HOAc (1.6) H_2O (1.3)	59	
13A	*trans*-$C_6H_5CH{=}CHBr$	2.8	Dioxane	15–18	21	HOAc (2.8)	49	
13B		2.8	Dioxane	15–18	21	HOAc (2.8)	57	Performed in air; vessel protected with drying tube
14		2.8	Dioxane	15–18	18	HOAc (3.3)	41	Performed in air; vessel protected with drying tube
15		2.8	Dioxane	15–18	18	HOAc (3.3)	49	
16		1.4	CH_3OH	0	18	HOAc (3.3)	39	
17		1.4	CH_3OH	23	0.8	HOAc (3.3)	28	Performed in air
18		1.4	DMSO	18	18	HOAc (1.8)	0–2	
19		1.6	95% EtOH	15–18	132	HOAc (1.8)	63	
20		1.4	*n*-Butyl alcohol	0	18	HOAc (3.3)	46	
21		2.8	5% H_2O–dioxane	60	0.25		0	
22		2.8	5% H_2O–dioxane	40	18		5	
23		2.8	5% H_2O–dioxane	23	18		0	
24		1.9	2% H_2O–CH_3OH	15–18	116	HOAc (2.2)	66	
25	*cis*-$C_6H_5CH{=}CHBr$	2.8	Dioxane	10	18	HOAc (2.8)	22	By ultraviolet analysis
26	$C_6H_5CH{=}CHOCOCH_3$	3.7	Dioxane	23	18	DOAc (2.8)	20	
27		3.7	Dioxane	23	18	HOAc (2.8)	50	

[a] Reductions were performed in a N_2 atmosphere unless otherwise indicated. [b] Reactions with an A,B label (*e.g.*, 5A, 5B) were run concurrently. [c] Refers to time at which reaction was halted, reaction mixture was analyzed, or yellow color disappeared, whichever was shortest. [d] By v.p.c. analysis unless otherwise indicated.

reactive methyl cinnamate clearly differentiates between dioxane and methanol, although reduced in good yield in both cases; and the still less reactive β-bromostyrene is reduced in 50–60% yield in dioxane, in only 30–40% yield in methanol, and virtually not at all in DMSO.[3]

Perhaps most impressive is the adverse effect on the reaction of relatively small amounts of water. Whereas reduction of methyl cinnamate in pyridine is virtually quantitative, the yield in 4% aqueous pyridine (mole ratio water:PADA = 2.6) is 28% (expt. 10). Addition of water as the proton source (mole ratio water: PADA = 5.6) instead of acetic acid to a pyridine solution effects decomposition of PADA, but the yield of reduced product is only 2%! (expt. 11A). Dioxane is similarly sensitive to the presence of water (expt. 11B and 21–23), although fair yields of reduction product are possible when aqueous acetic acid containing only a small amount of water is added (expt. 12). A single experiment (8) also demonstrates the adverse affect of water in DMSO. Since in at least one experiment (11B) the PADA in dioxane was *not* decomposed by addition of water, and subsequent addition of acetic acid gave a poor yield of reduced product, it would appear that water might act upon the effective reducing agent (N_2H_2?) rather than its precursor. However, this view seems negated by successful use of aqueous media for other diimide-forming agents, *e.g.*, hydrazine or arylsulfonylhydrazines. Alternately, water might primarily affect the rate of decomposition of PADA by increasing the concentration of H_3O^+ (or an equivalent species), known to be an exceedingly effective acid catalyst for this reaction.[4] Another possibility is that water affects the ratio of *syn–anti* forms of diimide. Further data are necessary to clarify this point.

Quite interestingly, *water has no adverse effect on reductions in hydroxylic solvents.* Experiments (19 and 24) in aqueous methanol and ethanol showed none of the deleterious effects noted above, and water can be used quite conveniently as a proton source in these solvents. This suggests that water and acetic acid are about equally effective as general acids in the decomposition of PADA for diimide reductions, although King has shown[4] that the catalytic rate constant of PADA decomposition is several orders of magnitude larger for HOAc than for water. Should the previously noted effect of water be on the actual reducing agent, then it would seem that alcohols effectively compete with water for this species but, for reasons not at all clear, do not inhibit its reaction with olefins.

Yields of reduced product under similar experimental conditions are significantly greater for methyl cinnamate than β-bromostyrene or β-acetoxystyrene. This is consonant with the earlier report that electronegative atoms on the olefinic bond increase difficulty of reduction.[2b] The difference in yield between the *cis* and *trans* olefinic bromides (expt. 13–15 and 25) is in agreement with earlier work.[2a] van Tamelen has based his conclusions, regarding the effect of an olefinic heteroatom on diimide reductions of the double bond, on yield data obtained for allyl, $CH_2{=}CH{-}CH_2X$, and the corresponding vinyl system, $CH_3CH{=}CHX$. Because steric effects bias the reduction in favor of the former system,[5] and because yield data are unreliable criteria of relative reactivity even under favorable conditions,[7] we performed a competitive reduction of $C_6H_5CH{=}CHBr$ *vs.* $C_6H_5CH{=}CHCH_3$. In methanol at about 24° the latter reacts 2.8 ± 0.2 times as fast as the bromide. Thus the heteroatom does diminish reactivity, but to a much lesser extent than the yield data[2b] implied. In fact, it suggests that the reaction is relatively insensitive to inductive effects. (*Cf.* σ^* values of $BrCH_2{-}$ *vs.* $CH_3CH_2{-}$ of $+1.00$ *vs.* -0.10. Since the difference in inductive effects of Br *vs.* CH_3 directly bonded to an olefinic carbon should be *at least* as great as that measured by this difference in σ^*, the observed relative rates imply a low value of ρ^*, ~ 0.3.) This conclusion was voiced earlier by Hunig and Muller.[6]

We might summarize by mentioning the things we know about the reaction, based on our results, and those we do not yet know. We know that the nature of the solvent significantly affects the reaction. We know that water is a very powerful inhibitor of reductions in nonhydroxylic solvents but is virtually without effect in alcohols. We know that electronegative heteroatoms on an olefinic carbon decrease somewhat the reactivity of the double bond toward reduction. However, we do not yet know why these changes occur, *i.e.*, we cannot yet sufficiently describe the mechanism of these reductions to rationalize adequately all our observations in one self-consistent hypothesis.

Further competitive reductions using diimide are in progress.

Experimental Section

Potassium Azodicarboxylate (PADA).—This salt was prepared according to Thiele[8] with the exception that the crude salt was slurried with methanol prior to drying and purification by reprecipitation from aqueous ethanol. PADA was stored *in vacuo* at room temperature or in a N_2 atmosphere at 0°.

Reproducibility of reductions was poor when comparisons were made between reactions run with different batches of PADA or between reactions run with the same batch of PADA but separated by a long (over 3 weeks) time interval. Such lack of reproducibility suggests that other variables which we have not identified (carbonate content, water content?) may be as important in determining the course of reaction as some of those mentioned above.

Methods.—To minimize the effect of variability of PADA, experiments differing in only one variable were conducted simultaneously. Dioxane was distilled from sodium prior to use (PADA frequently decomposed spontaneously in crude dioxane), whereas other solvents were not purified. Except for reductions of azobenzene, yields were determined by v.p.c. analysis (Aerograph A-600 with flame-ionization detector) conducted, usually, directly on the reaction mixture after removal of solids by filtration or centrifugation. Corrections to differences in detector sensitivity were applied to observed peak areas by running chromatograms on mixtures of known composition. Ester and bro-

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[3] The results in DMSO are based on a single experiment. Because results are variable (*e.g.*, reductions of β-bromostyrene in methanol gave reduced products in yields from 9 to 39%), the observation should be accepted with reservation.

[4] C. King, *J. Am. Chem. Soc.*, **62**, 379 (1940).

[5] E. W. Garbisch, Jr. (Abstracts, 149th National Meeting of the American Chemical Society, Detroit, Mich., April 1965, p. 13P) has noted that substitution of methyl for olefinic hydrogen leads to a reduction in rate of 0.13, although this was not attributed to steric effects. An even larger effect is noted in ref. 6.

[6] S. Hunig and H. R. Muller, *Angew. Chem. Intern. Ed. Engl.*, **1**, 213 (1962).

[7] As we mention in the experimental section we have had considerable difficulty in reproducing data obtained at widely different times using different batches of PADA.

[8] J. Thiele, *Ann.*, **271**, 127 (1892).

mide analyses were performed using either 10% diethylene glycol succinate or 20% silicone oil on Chromosorb W, 80–100 mesh. For reductions of methyl cinnamate, most reactions utilized 2.0 g. of ester in 50 ml. of solvent. For reductions of β-bromostyrene, most reactions utilized about 1 g. of bromide in 20–60 ml. of solvent. All reactions were stirred continuously, either magnetically or mechanically. It was found that yields increased as the addition time of acid was lengthened. The following is a description of a typical reduction of methyl cinnamate. Into a mechanically stirred slurry of 5.0 g. of PADA in 50 ml. of dioxane in a N_2 atmosphere was dissolved 2.0 g. of ester. A solution of 1.3 g. of acetic acid in 5 ml. of dioxane was added over a period of about 30 min. After 9 hr. an equal amount of acid was slowly added. No yellow solids were noted after a total elapsed time of 19 hr. Analysis of the reaction mixture by v.p.c. showed only two peaks, identical in retention time with hydrocinnamate and cinnamate, whose areas, after correcting for differences in detector sensitivity, corresponded to 93% saturated and 7% unsaturated product.

Commercial β-bromostyrene contained 8% of the *cis* isomer[9] according to v.p.c. examination. This was corroborated by n.m.r. examination which showed parts of two vinylic AB patterns in a ratio close to 92:8, $J_{AB} = 14$ and 8 c.p.s., respectively. The bromide was distilled, recrystallized four times from methanol, freeze dried, and redistilled (b.p. 42° at 0.9 mm.) to afford $\geqslant$99.4% *trans*-β-bromostyrene. Commercial propenylbenzene was distilled with only a small forerun being collected. A vapor phase chromatogram (10% diethylene glycol succinate, 135°) showed only two components in the ratio 97:3. The major component was assigned the *trans* configuration on the basis of a strong (C–H bending) absorption at 965 cm.$^{-1}$. Infrared spectra in the series $C_6H_5CH{=}CHX$ (X = Br, CH_3, OAc) indicate that the *trans* isomers have a ring C–H bending frequency at 730–750 cm.$^{-1}$, shifted to 770–790 cm.$^{-1}$ in the *cis* isomers.

Competitive reductions were performed in methanol using an equimolar mixture of β-bromostyrene and propenylbenzene and excess PADA. After about 25% of total olefin had been reduced, the mixture was analyzed by v.p.c., using calibration curves which had been prepared for both olefins and their saturated counterparts. The relative rate constants, k/k', for reduction of the bromides were taken as being equal to the ratio of reduced products.

Acknowledgment.—This investigation was supported in part by Public Health Service Research Grant No. GM12113-01 from the National Institutes of Health and in part by the University of Connecticut Research Foundation.

[9] T. Yoshino, Y. Manabe, and Y. Kibiucki, *J. Am. Chem. Soc.*, **86**, 4673 (1964); E. Grovenstein, *ibid.*, **75**, 2639 (1953); S. J. Cristol and W. K. Norris, *ibid.*, **75**, 2645 (1953).

Reductive Cleavage of Aryloxysilanes

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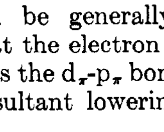
Hydrolysis in the presence of acid or base is the usual method for the removal of the trimethylsilyl group from aryloxysilanes.[1] However, in the course of our work it became necessary to remove the trimethylsilyl group from a number of compounds containing the aryloxytrimethylsilane grouping by an essentially nonhydrolytic means. A literature search failed to reveal any previous attempt to do this. Speier[2] subjected *p*-trimethylsilylphenoxytrimethylsilane to the action of hydrogen in the presence of a Raney nickel catalyst under pressure and at elevated temperatures, but the reaction resulted in the desired hydrogenation of the aromatic ring with the Si–O bond remaining intact.

The use of lithium aluminum hydride for the purpose of cleaving alkoxysilanes to silanes and alcohols has been reported on numerous occasions,[3–6] and there was no reason (except the lack of information) to suppose that aryloxysilanes would behave differently. This assumption was indeed correct, and we would like to report that the bond in question undergoes a facile cleavage in the presence of lithium aluminum hydride with almost quantitative yields. The reaction appears to proceed more readily than the reductive cleavage of alkoxysilane bonds since varying degrees of difficulty have been experienced[4,5b,7] with the latter cleavages. More difficult aryloxysilane cleavages can be accomplished at higher temperatures in *p*-dioxane.

In the examples given below trimethylsilane is produced as a side product, and this was trapped and identified. The aluminum complex formed during the reduction was destroyed with ice and dilute acid, since trimethylphenoxysilane and other silicon–carbon ethers not mentioned in this paper can be recovered unchanged from this mild treatment. Other techniques[8] could of course be used to recover the required products if even cold dilute acid cannot be tolerated.

It is not the purpose of this particular paper to comment in detail on the mechanism of this reaction.[9] However, it is interesting to note that 2,4,6-trichlorophenoxysilane undergoes rapid reductive cleavage of the silicon–oxygen bond at room temperature while trimethylphenoxysilane reacts at a much reduced rate, and hence the reaction appears to be facilitated by electron-withdrawing substituents in the aromatic ring. Sommer and his co-workers[4,5b] have postulated an SNi-Si quasi-cyclic mechanism for such reactions in nonpolar media which proceed *via* a four-center transition state. The complex so formed accommodates a negative charge within its structure. Thus,



if R′ is a phenyl group, it is to be expected that the complex formed during the rate-determining transition state would be stabilized (especially if the aromatic group contains electron-withdrawing substituents) and the reaction would be generally facilitated. Also, it can be argued that the electron withdrawal into the aromatic ring reduces the d_π-p_π bonding between silicon and oxygen. A resultant lowering of the activation

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[1] C. Eaborn, "Organosilicon Compounds," Butterworth and Co. (Publishers) Ltd., London, 1960, p. 301.

[2] J. L. Speier, *J. Am. Chem. Soc.*, **74**, 1003 (1952).

[3] See ref. 1, p. 196.

[4] L. H. Sommer, "Stereochemistry, Mechanism and Silicon," McGraw-Hill Series in Advanced Chemistry, McGraw-Hill Book Co., Inc., New York, N. Y., 1965, p. 51.

[5] (a) L. H. Sommer, C. L. Frye, G. A. Parker, and K. W. Michael, *J. Am. Chem. Soc.*, **86**, 3271 (1964); (b) L. H. Sommer, C. L. Frye, and G. A. Parker, *ibid.*, **86**, 3276 (1964).

[6] H. J. Emeleus and L. E. Smythe, *J. Chem. Soc.*, 609 (1958).

[7] H. Westermark, *Acta Chem. Scand.*, **8**, 1830 (1954).

[8] N. G. Gaylord, "Reduction with Complex Metal Hydride," Interscience Publishers, Inc., New York, N. Y., 1956, p. 1010.

[9] A more detailed paper will be published at a future date.