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TETRAHEDRON PRIZE FOR CREATIVITY IN ORGANIC CHEMISTRY

The Executive Board of Editors for Tetrahedron Publications is pleased to announce that the 1991 Tetrahedron Prize for Creativity in Organic Chemistry has been awarded to Professor W. S. Johnson of Stanford University, California for his development of methods for the synthesis of complex natural products by biomimetic pathways. There follows a summary of his accomplishments, a Curriculum Vitae, a list of his collaborators and a list of publications.

Fifty Years of Research

A Tribute to My Co-workers

by William S. Johnson

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In 1944, when I was a young assistant professor at the University of Wisconsin, a very promising new graduate student, C. David Gutsche, chose me as his thesis advisor. At some point during his stellar performance, which resulted in his completing his Ph.D. work in a record time of two years and five months, I was bragging to Professor Sam McElvain, one of my senior colleagues, about this sensational student of mine. Sam, a man of great wisdom, then said to me, "If you are lucky enough to attract several students of Gutsche's caliber to your research group, these people are likely to make you famous." That sage remark permanently oriented my attitude concerning the matter of where the credit belongs. Accordingly I submit that the efforts of over 100 predoctoral and 200 postdoctoral co-workers over a period of 50 years have been largely responsible for the results which are described in the publications listed at the end of this document. It is now my pleasure to set forth selected highlights of these accomplishments and in this way to pay tribute to my co-workers who are named in the accompanying list.

Adolf Windaus, in his 1928 Nobel Lecture[1] on steroids, said, "The synthesis of such a substance appears to the chemist particularly difficult, and up till now I have not dared to attempt it." At that time and for the following 10-15 years the tools of organic synthesis were greatly limited, and the art for the diastereoselective production of molecules with several asymmetric centers was essentially non-existent. Sir Robert Robinson and Werner Bachmann who were the foremost pioneers of steroid synthesis at the time never reached the point of trying to cope with the stereochemical problems. I remember Robinson saying that he felt that there was an intrinsic tendency for chemical reactions to lead to products with natural configuration, hence he was not inclined to worry about the problem. Be that as it may, in 1951 he and Cornforth, *et al.*[2] completed the first synthesis of a non-aromatic steroid, *epi*-androsterone, which has seven chiral centers (64 possible diastereoisomers). This was an amazing tour de force considering that, at a number of stages, separation of

diastereoisomers was required. The Woodward steroid synthesis,[3] which was completed at about the same time, was the first to even consider the problems of diastereoselectivity and represented a real advance in the way of thinking about synthesis.

The hydrochrysene synthesis of steroids. Our first (1952)[4,5] synthesis of a non-aromatic steroid is outlined in Figure 1. The hydrochrysene derivative **4** was easily produced by the sequence of two Robinson annulation reactions (**2** → **3** and **3** → **4**). Diastereoselective Birch reduction of the enone system and the styrene double bond of **4** proceeded readily, but the aromatic nucleus was totally resistant to all variations we tried. We had about given up when Brian Bannister, one of Robinson's last Ph.D. students, arrived on the scene and volunteered to try some forcing conditions he had heard about. He saved the day and was able to obtain a fair yield of the mixture of α,β and β,γ ketones **5** which on hydrogenation gave a single diastereoisomer **6**.

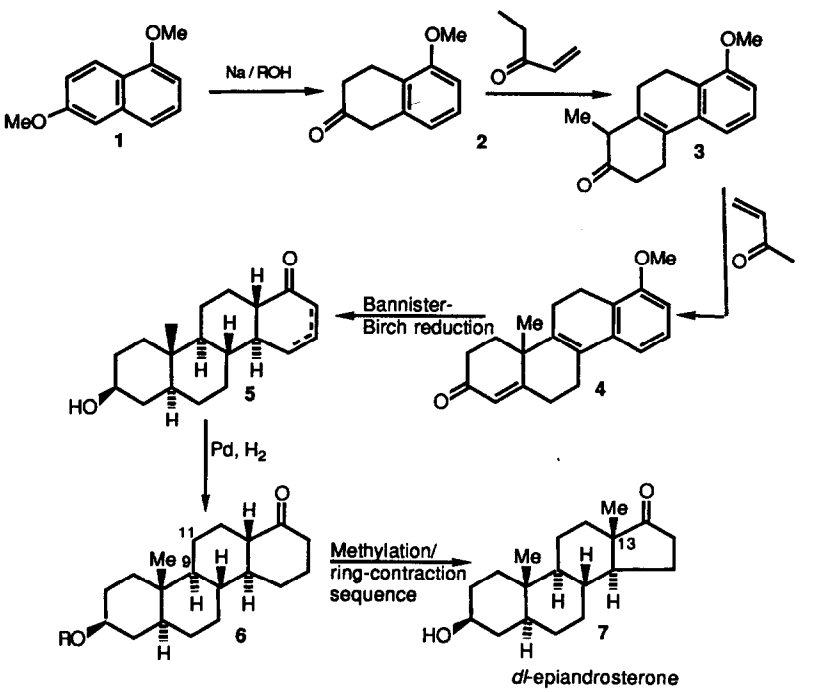

Figure 1. The first Wisconsin total synthesis of a nonaromatic steroid.

Thus step **4** → **5** introduced *six* new asymmetric centers diastereospecifically to give, after hydrogenation, a single diastereoisomer **6** having natural steroid configuration as proved by its conversion into racemic

epi-androsterone. As in the case of model methodology, developed by my own hands (see bibliography), the angular methylation-ring contraction sequence **6** → **7** was not diastereoselective, requiring an easy chromatographic separation of **13**α and **13**β epimers. Nevertheless the total results represented a major step forward in achieving diastereoselectivity.[6] Also this route was quite short making it possible to achieve a synthesis that was completely total while the earlier ones were "formal" total syntheses. This term was coined by Cornforth and Robinson for a synthesis that depends on one or more "relays" through intermediates which are obtained (for the further steps) by degradation of readily available natural products.

It is noteworthy that the aforementioned studies were performed without the aid of NMR spectroscopy. The story of the 1957 visit to Wisconsin by John D. Roberts, the foremost pioneer in the application of NMR to organic chemistry, of how he taught me to use our newly acquired 40 MHz spectrometer and to interpret the results, and of how he and I became involved in some collaborative research on diazomethane reactions is told in some detail elsewhere.[7]

Eventually we prepared intermediate **4** in kilogram quantities which made possible the completely total synthesis of many of the important steroids including aldosterone.[8] In this last case the process was completely diastereoselective. It was during the early stages of this synthesis that I went to Harvard for a year as Visiting Professor, and Raphael Pappo was made a temporary faculty member at Wisconsin in order to act as a most inspiring adviser to my research group. Throughout his three years at Wisconsin he made many innovative as well as practical contributions to our research program. Our adaptation of the above hydrochrysene approach to the synthesis of 11-hydroxy corticoid types[9] would not have been realized without the help of Gilbert Stork who has always been strongly supportive of my work as described elsewhere.[7] The synthesis of conessine[10] deserves special mention because the novel stereoselective strategy used for the development of ring D was conceived as well as performed by Jim Marshall. The names of other co-workers who made this program possible may be found in the publications listed under "Hydrochrysene Approach" in the bibliography.

Conformational Studies. Just as chemists of the Robinson generation worked without concern for stereochemical factors so we, in the early days, were working in ignorance of conformational considerations until Derek Barton showed us the light in 1950.[11] My colleague, Al Wilds, and I had the privilege of hearing him give a private discourse on his classical revelations before they were published. That occasion suddenly lifted a heavy veil of mystery that enshrouded much of the chemistry in which we were all involved, and I immediately became a Barton disciple, spreading the gospel at home and abroad. We were inspired to perform a number of studies in this area, which resulted in ten publications, the most significant of which involved the

first experimental determination of the difference in enthalpy (5.5 kcal/mole) between the chair and boat forms of cyclohexane. This was accomplished by combustion calorimetry (performed by my colleague John Margrave) on a pair of fused-ring lactones (formulas **4** and **5** (Figure 2) that differed only in that the central rings are in the chair and boat form respectively).[12] The synthetic work was deftly executed by my predoctoral student, Victor Bauer, who obtained the boat isomer **5** by lactonization of the diaxial hydroxy acid **2** under forcing conditions. The diequatorial isomer **3**, in contrast, underwent facile lactonization to give **4**. It was in the full paper[12b] that the term "twist" boat was coined.

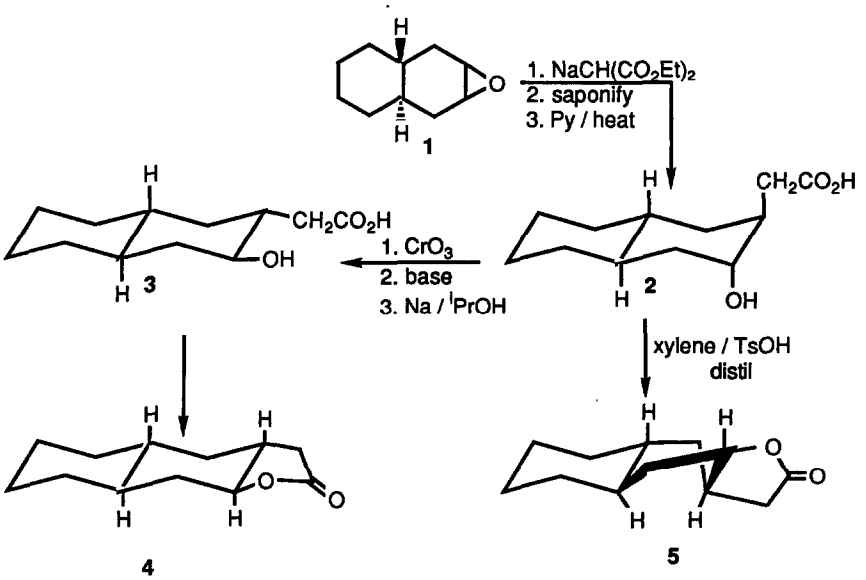

Figure 2. Synthesis of chair and boat cyclohexane derivatives.

Biomimetic Polyene Cyclizations. Inspired by the classical work of Bloch, *et al.*, and Woodward[13] who demonstrated that the open-chain polyene, squalene, is the key biogenetic precursor of lanosterol, and by the theoretical concepts of Stork, *et al.*[14] and Eschenmoser, *et al.*[15] regarding the mechanism of the biocyclization of squalene to give polycyclic triterpenoids, we began in 1960 an investigation of the non-enzymic cyclization of polyenes which we are still studying. Early experiments to effect tricyclizations were so unsuccessful that by 1963 realization of the objective was generally regarded as hopeless.[16,17] I was about to give up our studies in the area when we discovered that certain acetal functions in the presence of Lewis acids served as

excellent initiators of polyene cyclizations. At about the same time we found that appropriately substituted allylic cations (generated by the action of acid on the corresponding alcohols) also proved to be good initiators. An early example of the latter type of initiation is the cyclization **1** → **2**, shown in Figure 3. Marty Semmelhack performed the exploratory experiments on the scheme shown in Figure 3 and realized the first steroid synthesis via biomimetic polyene cyclization methodology.[18] It is noteworthy that even though rings A and D of compound **2** are opened by ozonolysis, the stereochemical integrity of the five asymmetric centers, generated in the cyclization step, is still maintained through to the final product **4**. Refinement of this scheme and the realization of the 66% cyclization yield was due mainly to Chuck Harbert's efforts.[19]

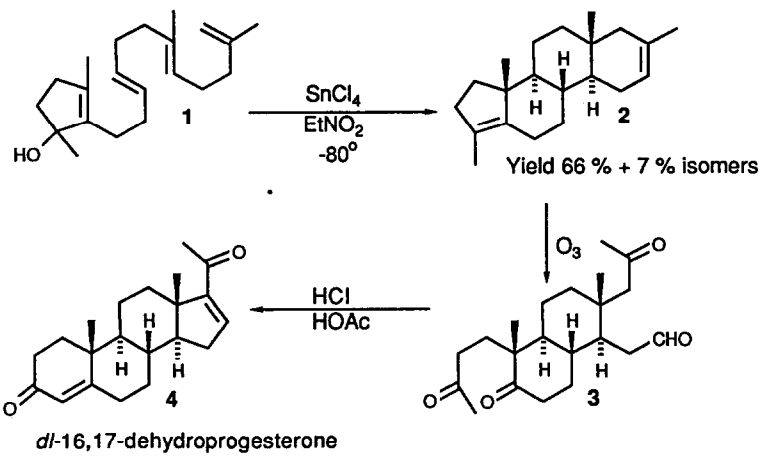

Figure 3. First steroid synthesis via biomimetic polyene cyclization methodology.

This type of tricyclization was extensively exploited. When our asymmetric synthesis of hydrocortisone (see Figure 4) was announced,[20] it was, by considerable measure, more efficient than any of the previous total syntheses. With further improvements (unpublished) our polyene cyclization approach, yielding cortisone,[21] was on the verge of being commercially feasible; however, at about that time the major market for antiinflammatory agents was being taken over by new, very effective, non-steroidal drugs.

From an academic point of view, the synthesis of corticoids is one of the most widely studied areas which has yielded a wealth of new synthetic methodology. Many of my co-workers played important roles in our synthesis. Brian Metcalf designed and developed an elegant method for making the cyclization substrate **2** in its racemic form. Ray Brinkmeyer later led the studies that resulted in the asymmetric synthesis of **2** via the

reduction of the corresponding ketone with Mosher's reagent (LAH/Darvon alcohol) to give the enantiomers of 2 in the ratio of *R*:*S* = 92:8. The cyclization step 2 → 3, strikingly retarded by the 11-hydroxyl group, was our most difficult problem; indeed it failed completely until Sina Escher discovered conditions involving the "magic" reaction solvent, trifluoroethanol, which gave 3 in yields of 29-35%. After considerable effort by many co-workers this yield was improved to 43% at best.

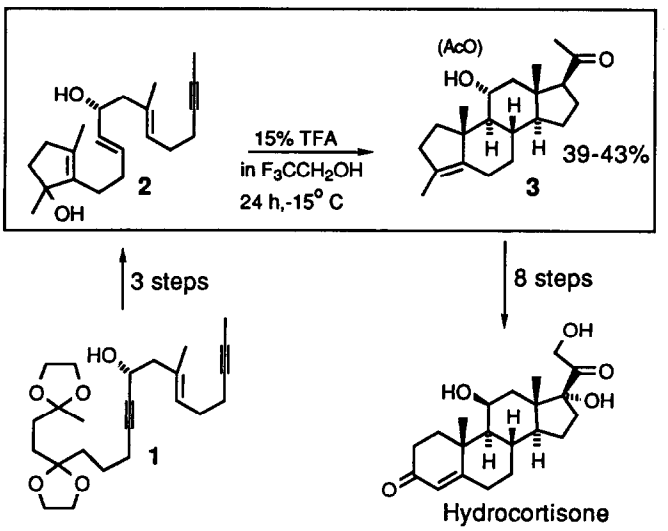

Figure 4. The Stanford total asymmetric synthesis of a corticoid.

A number of other steroids and related natural products have also been synthesized via a cyclopentenol-initiated biomimetic polyene cyclization. In three of these instances especially significant contributions were made by my co-workers as follows. In connection with the synthesis of progesterone, Mike Gravestock was largely responsible for developing the methodology for utilizing the methylacetylenic function as a cyclization terminator in conjunction with the cyclopentenol initiator. The estrone synthesis was completely conceived as well as reduced to practice by Paul Bartlett while a graduate student, and in the case of longifolene, Bob Volkmann perceived how an unexpected product of his cyclization could be converted into this natural product, and he proceeded to complete the synthesis.

Until recently attempts to realize non-enzymatic *tetra*cyclizations have been abortive. Only one type of substrate, involving the acetal initiator, gave some promise: in a procedure involving a two-phase reaction

mixture, which was discovered by Koen Wiedhaup, the tetracyclic product, consisting mainly of compound **2** (Figure 5) and its C-4 epimer, was formed in about 30% yield. For 20 years this was the world record yield for a non-enzymic polyene tetracyclization.

In 1985 a possible way for improving such cyclizations occurred to me. This new concept employs a polyene substrate having a cation-stabilizing (C-S) substituent appended to one (or more) of those carbons that are destined to develop positive character in the cyclization transition state, thus lowering the activation energy of the process. A beneficial effect of a C-S function can alternatively be envisaged for a step-wise cyclization mechanism via stabilization of the intermediate (partially cyclized) carbocations. To test this idea the acetal **3**

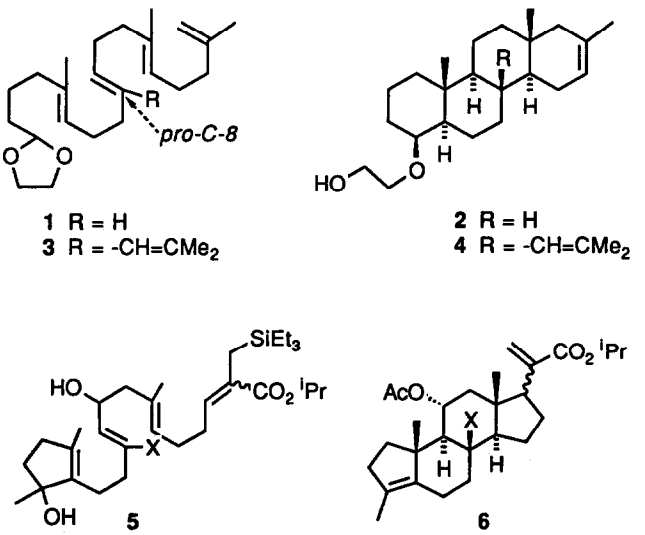

Figure 5. Enhancement of polyene cyclizations by a cation-stabilizing auxiliary.

(Figure 5), having the cation-stabilizing isobutenyl group in place of H at *pro*-C-8 (steroid numbering) was ingeniously prepared by Stephen Telfer and Soan Cheng. They also carefully studied the cyclization of this substrate which gave mainly substance **4** along with some isomers also having the all trans configuration of the ring fusions, in a combined yield of 77%.[22] Thus the effect of the C-S auxiliary at *pro*-C-8 was to more than double the yield of tetracyclic product.

An even more dramatic effect was observed in another case[23] studied by Steve Lindell and John Steele as follows. The rate of TFA-catalyzed cyclization of the substrate **5** (X = H) is strongly attenuated by the hydroxyl

group at *pro*-C-11; therefore, side-reactions become a significant factor and the optimized yield of product **6** (X = H) was only 20% after a reaction time of 24 h (Cf. the example in Figure 4). In striking contrast the cyclization of the substrate **5** (X = -CH=CMe_2), having the isobutenyl C-S auxiliary at *pro*-C-8, was complete in less than 1 min and the product **6** (X = -CH=CMe_2) was isolated in 80-83% yield.

On the basis of the foregoing observations I have proposed[24] a mechanism for the action of oxidosqualene cyclases whereby negative point charges are delivered so as to form ion pairs with and therefore stabilize the positive centers that develop in the cyclization transition states (see Figure 6). This model has the advantage that, in contrast to previously entertained postulates, no particular conformational control is required by the enzyme, not even for the non-Markovnikov closure of ring C. Thus, the bicyclic cation species at *pro*-C-8, that is stabilized by point charge b, may be regarded as forming a π complex with the 13,14 olefinic bond which now has partial positive charge at *pro*-C-13 as well as -14. Since point charge c is available to *pro*-C-13 but not -14, the six-membered ring C is formed in the non-Markovnikov manner. The involvement of point charge complexes in enzymic processes is well established, *e.g.*, the well documented Phillips mechanism for the action of lysozyme involves a negative point charge (from Aspartate 52) interacting electrostatically with a carbonium ion intermediate.[25]

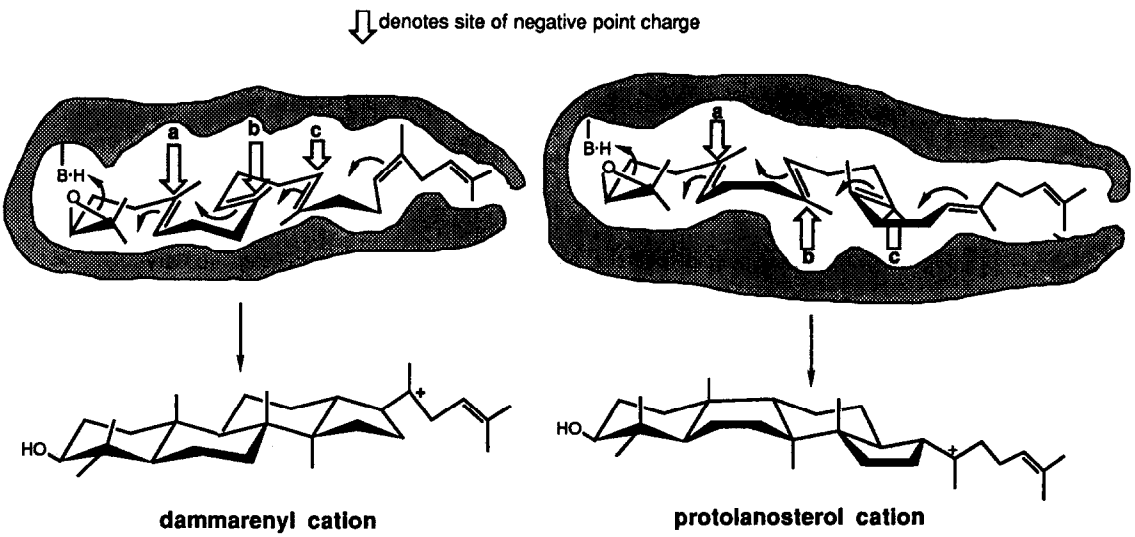

Figure 6. Proposed models for 2,3 oxidosqualene cyclases.

Recently we have been studying the use of the fluorine atom as a C-S auxiliary in polyene cyclizations. After considerable exploratory work this methodology has been applied to the total synthesis of β-amyrin (Figure 7), the key step being the cyclization **1** → **2**. The fluorine atom at *pro*-C-13 not only enhanced the cyclization, but controlled the regiochemistry so as to give the six-membered ring C. The olefinic bond involved in the formation of ring D has the Z-configuration (see formula **1**) so that, according to the Stork-Eschenmoser principle the closure gives the D/E syn-cis configuration shown in formula **2**. That the major product of the cyclization of **1** was indeed compound **2** was shown by its conversion to racemic β-amyrin which was identified unequivocally with the natural product. The principle co-workers in the fluorine studies were Balan Chenera, Vernon Fletcher, Vuligonda Vidyasagar (exploratory studies, formation and structure proof of tetracyclic products), Bob Buchanan (basic studies directed toward pentacyclic triterpenoid types), and Mark Plummer, Singham Pulla Reddy (β-amyrin synthesis).

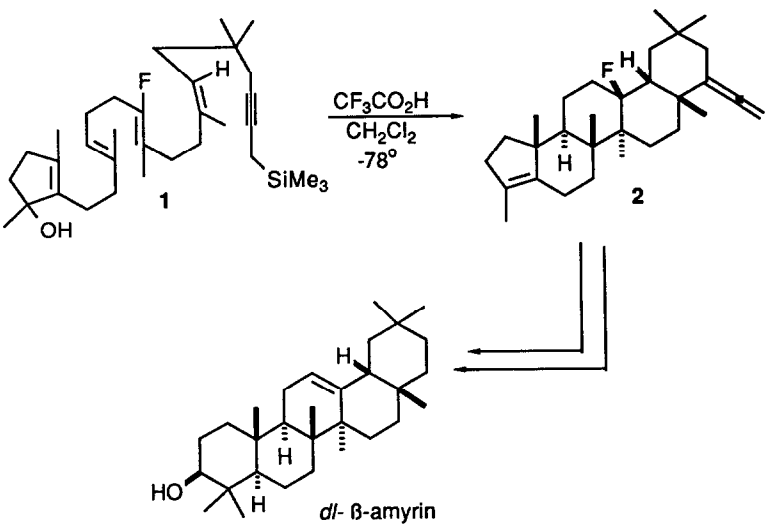

Figure 7. The fluorine atom as a C-S auxiliary in the total synthesis of β-amyrin.

Before leaving the subject of polyene cyclizations, I want to emphasize that the major part of this 31-year effort, involving 163 co-workers, was concerned with the steroselective synthesis of the polyenic substrates. In the early stages of our work there were very few stereoselective, high-yield methods for producing trisubstituted olefinic structures, so we became involved in developing new methodology. Two of these methods are noteworthy because they have been used extensively.

Julia's method for converting cyclopropylcarbinols into homoallylic bromides[27] gave very poor stereoselectivity when the olefinic bond in the product was trisubstituted. My student Steve Brady carried out a scholarly analysis of the problems involved in this type of rearrangement and, after making an important structural change in the substrate, developed a procedure, based on the work of J. D. Roberts,[28] that gave fairly good yields of >97% E-olefinic products. This method which I call the "Brady-Julia olefin synthesis" has been widely used in the synthesis of polyenic substrates as well a variety of natural products, *e.g.*, insect hormones, listed in the bibliography.

When I suggested to my co-workers that they might try using triethyl orthoacetate directly with an allylic alcohol to induce a Claisen rearrangement that would generate a tri-substituted olefinic function, it had not occurred to me that this process might show any special stereoselectivity; indeed, I was completely (and delightfully) surprised when the E-product was formed almost exclusively and in high yield. After the fact, my co-workers Fred Li and John Faulkner suggested a reasonable rationalization which is mentioned in the publication announcing the ortho-ester Claisen reaction.[29] This paper also discloses the 3-methoxyisoprene reaction

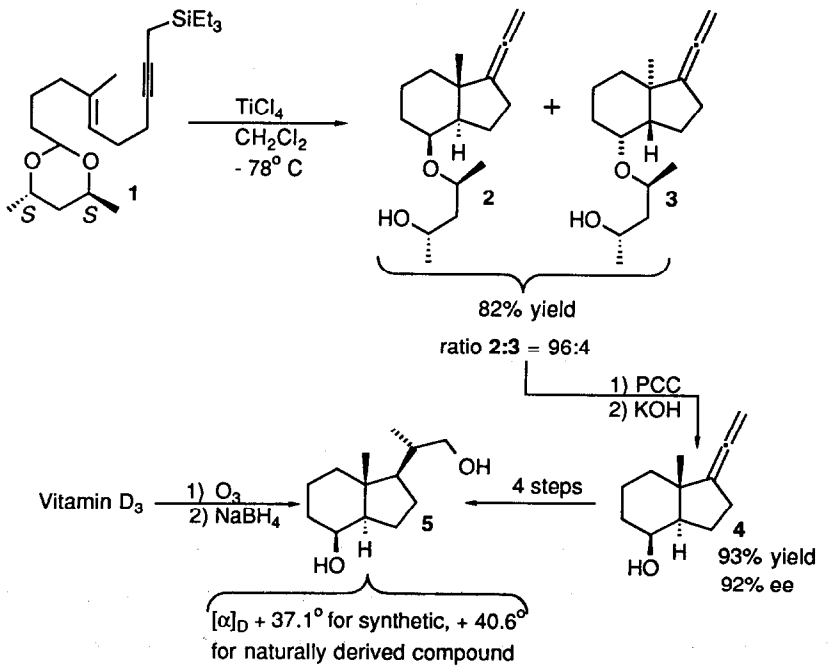

Figure 8. An asymmetric cyclization induced by a homochiral acetal.

with allylic alcohols in the Claisen rearrangement and its application to the synthesis of squalene which was beautifully developed by Lucius Werthemann. Lucius' studies were well advanced when we learned that John Faulkner, after moving to Scripps Institute of Oceanography, had also become involved in this approach to squalene; so I invited him and his student Michael Petersen to join us as coauthors of our first Claisen communication.

Asymmetric Syntheses Mediated by Chiral Acetals. My idea of testing this concept by cyclizing an acetal derived from *R,R*-butane-2,3-diol was elegantly reduced to practice by Chuck Harbert in 1968.[30] Although the bicyclic products were formed in a surprisingly high (83-84%) ee, we were discouraged from pursuing this methodology further because removal of the chiral auxiliary from the product was a low yield process. It was not until 15 years later when John Elliott had the idea of using the acetals from enantio-pure pentane-2,4-diols that exploitation of the methodology became practical because of the ease of removing the chiral auxiliary as shown in Figure 8. Moreover when the cyclization was performed at -78° with $TiCl_4$ the resulting enantiomeric excess was much improved.[31]

During the above development it dawned on me that the intermolecular version, as shown in Figure 9, had great potential; hence it was rapidly exploited by us as well as others, some of whom have generated useful

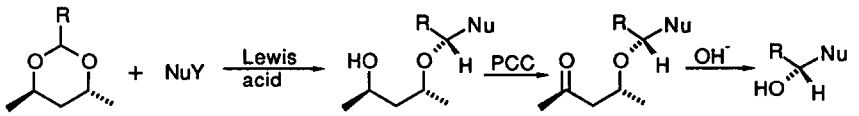

Figure 9. Generalized intermolecular reaction of homochiral acetals with nucleophiles.

modifications of the chiral auxiliary. A number of reviews of the field have appeared, the latest being the most thorough.[32] Our own studies involved the examination of a variety of nucleophiles NuY (see under "Asymmetric Reactions of Chiral Acetals" in the bibliography) which led to enantiorich intermediates for preparing products of biological interest. John Elliott made numerous, significant contributions in the planning and execution of this program. Mechanistic contributions were largely due to Paul Bartlett who is a coauthor of our first paper on the intermolecular process.[33] Others involved in this program are named in the bibliography.

Conclusion. While I am truly grateful for this opportunity to pay tribute to my co-workers, at the same time I have found this exercise frustrating because the limitations in space have necessitated my omitting

the work of a very large number of these people who have made truly significant contributions and are deserving of, but did not receive, special mention. Their names and their areas of involvement in research may be found in the bibliography.

My appreciation extends well beyond those who have worked in my laboratory. Special mention goes to Derek Barton, Konrad Bloch, Albert Eschenmoser, Jack Roberts and Gilbert Stork who are special cases because their work and teachings have greatly influenced my own in more ways than the few examples mentioned above. These five people were my choice when my Stanford colleagues decided to establish an annual symposium bearing my name, and asked me to select the speakers for the first event.

Finally, I wish to express my gratitude to the National Institutes of Health, the National Science Foundation, the American Chemical Society Petroleum Research Fund and the Wisconsin Alumni Research Foundation for research grants. In addition, special thanks are due the following industrial organizations for generous, unrestricted gifts in support of our research: Hoffman-La Roche, Pfizer, G. D. Searle, SmithKline Beecham, Sterling-Winthrop Research Institute, and Upjohn.

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Biographical Summary

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B.A., Amherst College, 1936
M.A., Harvard University, 1938
Ph.D., Harvard University, 1940

Appointments:
Research Chemist, Eastman Kodak Co., summers, 1936-39
Instructor, Amherst College, 1936-37
Instructor, University of Wisconsin, 1940-42
Assistant Professor, University of Wisconsin, 1942-44
Associate Professor, University of Wisconsin, 1944-46
Professor, University of Wisconsin, 1946-60
Visiting Professor, Harvard University, 1954-55
Homer Adkins Professor of Chemistry, University of Wisconsin, 1960-69
Professor and Executive Head, Department of Chemistry, Stanford University, 1960-69
Professor, Stanford University, 1970-75
Jackson-Wood Professor of Chemistry, Stanford University, 1975-78
Jackson-Wood Professor of Chemistry, Emeritus, Stanford University, 1978-

Awards and Other Recognitions:
National Academy of Sciences, 1954-
Honorary Doctor of Science, Amherst College, 1956
ACS Award for Creative Work in Synthetic Organic Chemistry, 1958
American Academy of Arts and Sciences, 1963-
Synthetic Organic Chemical Manufacturers Award for Creative Research in Organic Chemistry, 1963
Honorary Doctor of Science, Long Island University, 1968
Nichols Medal Award, 1968
Roussel Prize, 1970
Roger Adams Award, 1977
National Medal of Science, 1987
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Collaborators who worked in the Johnson laboratories either at the University of Wisconsin (1940-60) or at Stanford University (1960-Present)

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BOOTS, Sharon G., Managing Editor, *Analytical Chemistry*, Ph.D. 1964; Postdoc 1978-81, Stanford.
BRADY, Stephen F., Sr. Res. Fellow, Merck, Ph.D. 1967, Stanford.
BRANDSTADTER, Stephan, Postdoc 1989-Present, Stanford.
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BROT, Frederick E., Biochemist, Sigma Chemical Co., Ph.D. 1966, Stanford.
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BUELL, Bennett G., Retired (Sr. Res. Chemist, American Cyanamid), Ph.D. 1950, Wisconsin.
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BUNES, Leonard A., Riedel Environmental Services, Ph.D.1974, Stanford.
BURCKHARDT, Urs, Ciba Geigy, Postdoc 1965, Stanford.
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CHAN, Ming Fai, Abbott, Postdoc 1984-85, Stanford.
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KATNER, Allen S., Deceased (formerly with Eli Lilly), Postdoc 1964-66, Stanford.
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KEMP, A. D., Deceased (formerly Prof., U. College of Rhodesia & Nyasaland, Postdoc 1951-52, Wisconsin.
KERTESZ, Denis J., Syntex, M.S. 1960, Wisconsin.
KIKUCHI, Katsuo, Prof., Yamagata U., Postdoc 1962-63, Stanford.
KINNEL, Robin B., Prof., Hamilton College, Postdoc 1964-66, Stanford.
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SCHNEIDER, William P., Retired (Upjohn), Ph.D. 1950, Wisconsin.
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SHAHAK, Israel, Prof., Hebrew U., Postdoc 1961-63, Stanford.
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VREDENBURGH, Walter A., Pa. Industrial Chem. Corp., Ph.D. 1959, Wisconsin.
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