

The Contributions of Solomon F. Acree (1875–1957) and the Centennial Anniversary of the Discovery of the Acree–Curtin–Hammett Principle

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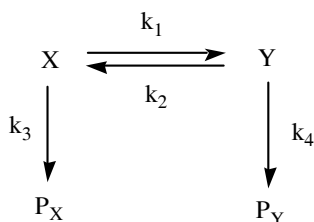
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Abstract: This paper describes the forgotten contributions of Solomon F. Acree to the understanding of the dynamics of kinetic systems that lead to multiple products. His seminal paper published in 1907 precedes those of Curtin, Hammett, Winstein, and Holness by nearly 50 years and is the true origin of the well known Curtin–Hammett principle and the Winstein–Holness equation. Acree's contribution was for products arising from equilibrating tautomers, whereas, that of Curtin, Hammett, Winstein, and Holness was for products arising from equilibrating conformers. In both cases the same principle applies. Exact solutions to the original kinetic scheme are presented with respect to product ratios at initial and final reaction times, including a new kinetic plasticity index which describes how pliable the kinetic system is to producing one product over the other. A brief biographical sketch of Acree is given including a listing of his contributions to various areas of chemical science.

Introduction

The Curtin–Hammett principle is a fundamental concept in mechanistic chemistry that has been used to approximate under simplifying conditions the magnitude of the final product ratio for a pair of products originating from a pair of equilibrating starting materials as shown in Scheme 1. The starting materials may be equilibrating conformers, tautomers, or stereoisomers. It is a concept that is well discussed in several textbooks of mechanistic organic chemistry [1–3], a review [4], and in a previous pedagogical article [5].



Scheme 1

Simply stated, the final product ratio $[P_X]:[P_Y]$ is equal to the ratio of the product forming rate constants, $k_3:k_4$, if the magnitudes of the rate constants k_1 and k_2 involved in the equilibration between X and Y are similar and are very large compared to those of the product forming steps. This independence of the final product ratio on the dynamics of the preceding equilibrium and the amounts of starting materials defines the Curtin–Hammett condition. When this condition is not met an exact analytical solution to the above kinetic scheme becomes necessary to determine the form of the final product ratio. Equation 1 yields the complete dependence of the final product ratio on all four rate constants and the starting amounts of X and Y [6].

$$\frac{[PX]_{\infty}}{[PY]_{\infty}} = \left(\frac{k_3}{k_4}\right) \left[\frac{(a+b)k_2 + ak_4}{(a+b)k_1 + bk_3} \right] \quad (1)$$

where a and b represent the initial amounts of X and Y at time zero. It is easy to verify that the right-hand side of eq 1 reduces to k_3/k_4 when (i) k_1 and $k_2 \gg k_3$ and k_4 and (ii) $k_1 = k_2$. Note that if only condition (i) holds, then the final product ratio will have a dependence on both the ratio k_3/k_4 and the equilibrium constant $K = k_1/k_2$ according to

$$\frac{[PX]_{\infty}}{[PY]_{\infty}} = \left(\frac{k_3}{k_4}\right) \left(\frac{1}{K}\right) \quad (2)$$

We now give a brief account of the works of Curtin and Hammett on this principle in the 1950s and juxtapose these against Acree's earlier work of 1907 which also predicted what would be later known as the Winstein–Holness equation. Following this, a review of recent work on the intrinsic dynamics of Scheme 1 is presented including key applications in the study of dynamic kinetic resolution. Finally, we close by giving a brief biographical sketch of Solomon Acree highlighting some of his other important contributions to chemical science that have also escaped attention.

Curtin Statement (1954)

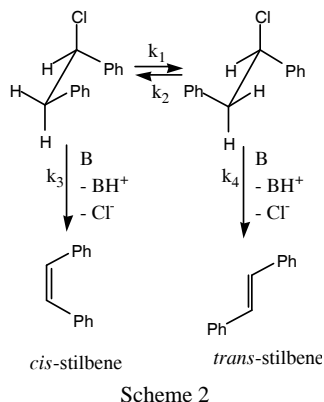
David Curtin in his 1954 paper [7], published in a journal that no longer exists and is difficult to access, refers to a private communication from Hammett in 1950 attributing that the idea originated from Hammett in his comment:

It was pointed out by Professor L.P. Hammett in 1950 that if the transition state theory is accepted and if rotation between the various possible conformations of the starting material is very rapid compared to the rate of the reaction, the relative amounts of products formed from the two critical conformations are completely independent of the relative populations of the conformations and depend only upon the difference in free energy of the transition states.

In a footnote, he continues:

It is obvious that if these conditions fail to hold -- that is, if the barriers to rotation are large compared to reaction -- the product composition will be determined solely by the relative numbers of molecules in the conformations leading to each of the products.

The chemical reaction referred to in Curtin's work is the E2 base catalyzed elimination of halide from two conformational rotomers of 1,2-diphenyl-1-chloroethane to yield *cis*- and *trans*-stilbene shown in Scheme 2.



Curtin does not give any mathematical relationships in his paper beyond the statement above.

Hammett Statement (1950)

Louis Hammett in his book [8] refers to the principle as the "Curtin principle" but makes the following footnote: "Because Curtin is very generous in attributing credit, this is sometimes referred to as the Curtin-Hammett principle."

In contrast to Curtin's general statement describing the phenomenon, Hammett does give a mathematical relationship for the relative rates for production of *trans* and *cis*-stilbene as shown in eq 3.

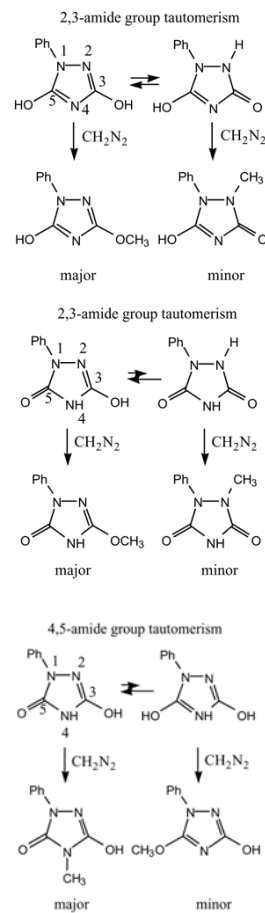
$$\frac{d[\text{trans-stilbene}]/dt}{d[\text{cis-stilbene}]/dt} = \frac{q_{\text{trans}}^{\#}}{q_{\text{cis}}^{\#}} \quad (3)$$

where the q_s refer to transition state partition functions for the elimination steps which in turn are related to the corresponding rate constants.

Acree Statement (1907)

As early as 1902 Solomon Acree studied the constitution of phenylurazoles [9–12] and the phenomenon of tautomerism [13–27]. In a paper published in 1907 [14] in the American Chemical Journal on work carried out at Johns Hopkins University he examined oxygen and nitrogen alkylations of tautomeric mixtures of 1-phenyl urazoles with diazomethane in ether solution as shown in Scheme 3. On page 3 of that paper he reasoned that "it is perfectly obvious that such reactions...do not give us decisive evidence in regard to the relative amounts of the enol and keto forms in any given amide group in which the change from one tautomeric form to the other is very rapid in comparison with the reactions between the two forms and the alkylating reagents." This wording is perfectly in line with Curtin's statement though at the time Acree would have been

unaware of transition states as this concept was formulated in 1935 by Henry Eyring [28–31].



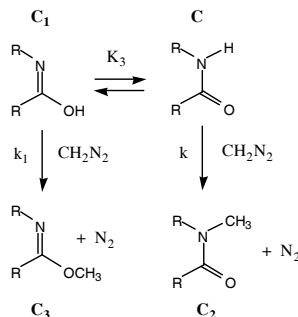
Furthermore, on page 5 Acree derived an expression for the observed second-order rate constant for the appearance of total product as a function of the relative populations of the urazole enol and keto forms. Based on his general scheme shown in Scheme 4 with the definitions of variables indicated and under the condition that the total initial concentration of diazomethane used is equal to the sum of the concentrations of the enol and keto forms of the urazole he wrote

$$\frac{d(C_2 + C_3)}{dt} = \frac{kK_3 + k_1}{1 + K_3} (C + C_1 - C_2 - C_3)^2 \quad (4)$$

where $C + C_1 - C_2 - C_3$ represents the concentration of diazomethane or urazole after time t . He deduced that the population of urazole in the keto form is $kK_3/(1 + K_3)$ and that in the enol form is $1/(1 + K_3)$. He gave the product ratio C_2/C_3 as a constant equal to kK_3/k_1 which implied that he assumed that the product forming steps were slower than the equilibration between tautomers. Note that this relationship is identical to eq 2. Also, we note that the expression

$$k_{\text{obs}} = \frac{kK_3 + k_1}{1 + K_3}$$

is exactly the Winstein–Holness equation [32] which relates the observed first-order rate constant for the total disappearance of both tautomers, or the total appearance of both alkylated products, to the respective populations of starting tautomers at equilibrium. Acree realized that it was “impossible to determine the value of k , k_1 , or K_3 from these two equations alone.”

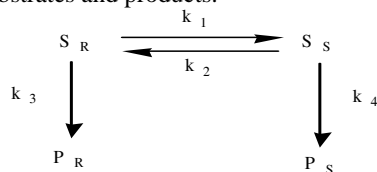


Scheme 4

The kinetic analysis of Scheme 4 and conclusions given by Acree are clearly perfectly in line with the simplifying Curtin–Hammett condition. All of these observations and conclusions predate the work of Curtin, Hammett, and Winstein by 47 and 48 years, respectively; however, the papers of Curtin, Hammett, and Winstein do not mention Acree’s work. We may speculate that this oversight may be due to the fact that the American Chemical Journal became a defunct journal by 1914 when it was absorbed by the Journal of the American Chemical Society and hence Acree’s work simply missed their notice.

Dynamic Kinetic Resolution

In addition to applying to equilibrating conformers or tautomers, the kinetic scheme shown in Scheme 1 is applicable to the technique of dynamic kinetic resolution [32–52] used in synthetic organic chemistry to optimize the synthesis of stereoisomeric products from corresponding enantiomeric or diastereomeric starting materials that are in equilibrium. This is arguably the most important practical application of the Curtin–Hammett principle. Scheme 5 shows an analogous kinetic system with appropriate stereochemical descriptors R and S for substrates and products.



Scheme 5

If a chemical reaction produces enantiomeric or diastereomeric products and it is desired to carry out a subsequent transformation on one of them, then one is faced with the prospect of first separating or resolving the product mixture from the first reaction, usually racemic, before carrying out the second. Resolution of racemic mixtures by standard methods results in an automatic loss of half the starting material assuming that the resolution procedure goes to completion. This means that if the individual stereoisomers in the starting racemic mixture are not interconvertible then the maximum amount of desired product formed in the subsequent

step is exactly half that of the starting material and so the maximum yield for this second step is 50%. This situation is designated as anti-Curtin–Hammett behavior and corresponds to kinetic resolution. If, on the other hand, there is a possibility to have a rapid interchange between starting materials to an extent that the Curtin–Hammett condition applies, then in principle it is possible to shunt all of the starting material toward the product of interest in the subsequent reaction. The more dynamic the equilibration is between S_R and S_S, the more likely this outcome will occur. This situation is designated as Curtin–Hammett behavior and corresponds to dynamic kinetic resolution.

In a recent report in 2003 the complete dynamics of Scheme 5 was explored [6] including a full kinetic analysis of the dependence of the initial and final product ratios on all four rate constants and starting amounts of S_R and S_S. The method used to derive expressions for these ratios shown in eqs 5 and 6,

$$\frac{[P_R]_0}{[P_S]_0} = \left(\frac{a}{b}\right) \left(\frac{k_3}{k_4}\right) \quad (5)$$

$$\frac{[P_R]_\infty}{[P_S]_\infty} = \left(\frac{k_3}{k_4}\right) \left(\frac{(a+b)k_2 + ak_4}{(a+b)k_1 + bk_3}\right) \quad (6)$$

where a and b are the initial amounts of S_R and S_S, respectively, was the method of Laplace transforms [53]. It is important to point out that though there are no products at time zero their ratio extrapolated to zero time is a finite quantity. These two expressions pointed to a new kind of experiment that could be carried out that would quantitatively determine the intrinsic efficiency or plasticity of dynamic resolution which depended only on key rate constant ratios. This parameter was shown to be able to account for all experimentally observed cases between and including the limits of complete dynamic kinetic resolution (Curtin–Hammett conditions) and complete kinetic resolution (anti-Curtin–Hammett conditions).

The crux of the new experiment is to determine initial and final product ratios for a set of initial substrate ratios covering the full range of possible optical purities of starting materials beyond the simple racemic condition. Hence, for a given starting substrate ratio, a/b , one records time dependent product progress curves and from these the initial and final product ratios are determined by extrapolation to zero and infinite time. A plot of $[P_R]_0/[P_S]_0$ versus a/b yields a slope equal to $r = k_3/k_4$. A plot of final product excess with respect to P_R, pe_∞ , versus initial substrate excess with respect to S_R, $(a - b)/(a + b)$, according to eq 7

$$pe_\infty = \frac{[P_R]_\infty - [P_S]_\infty}{[P_R]_\infty + [P_S]_\infty} = \frac{\frac{[P_R]_\infty}{[P_S]_\infty} - 1}{\frac{[P_R]_\infty}{[P_S]_\infty} + 1} = \frac{1}{rv + u + 1} \left(\frac{a - b}{a + b}\right) + \frac{rv - u}{rv + u + 1} \quad (7)$$

yields a slope equal to

$$\frac{1}{rv + u + 1}$$

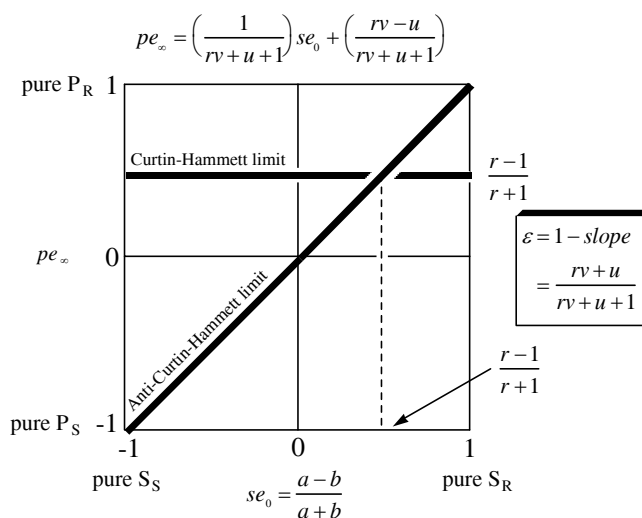


Figure 1. Relationship between final product excess and initial substrate excess according to eq 7 showing the two limiting slopes corresponding to the Curtin–Hammett condition (zero slope, 100 % kinetic plasticity) and anti-Curtin–Hammett condition (unit slope, 0 % kinetic plasticity).

and an intercept equal to

$$\frac{rv-u}{rv+u+1}$$

where $r = k_3/k_4$, $u = k_1/k_3$, and $v = k_2/k_3$. The rate constant ratios u and v may be determined directly from the slope and intercept of such a plot using the relations

$$u = \frac{1}{2} \left[\frac{1 - \text{intercept}}{\text{slope}} - 1 \right] \quad (8)$$

and

$$v = \frac{1}{2r} \left[\frac{1 + \text{intercept}}{\text{slope}} - 1 \right] \quad (9)$$

An expression for the intrinsic efficiency or kinetic plasticity of dynamic kinetic resolution, ε_{DKR} , may be obtained by examining the full range of possible values for the slope of this plot. In the Curtin–Hammett limit where $u = v \rightarrow \infty$ so that $k_1 = k_2$ and $k_1, k_2 \gg k_3, k_4$, the slope reaches a minimum value of zero. This result indicates that the final product ratio is independent of the starting amounts of S_R and S_S . In the anti-Curtin–Hammett limit where $u = v = 0$ so that $k_1 = k_2$ and $k_1, k_2 \ll k_3, k_4$ the slope reaches a maximum value of one. This case indicates that the final product ratio mirrors the initial starting material ratio. From these two boundary conditions we set $\varepsilon_{DKR} = 1$ to represent the Curtin–Hammett case of 100 % efficiency or kinetic plasticity and $\varepsilon_{DKR} = 0$ to represent the anti-Curtin–Hammett case of 0 % efficiency or complete kinetic rigidity. Hence, we may define the intrinsic efficiency or performance of dynamic kinetic resolution as in eq 10.

$$\varepsilon_{DKR} = 1 - \text{slope} = 1 - \frac{1}{rv+u+1} = \frac{rv+u}{rv+u+1} \quad (10)$$

Figure 1 illustrates graphically the two limiting cases described above. Equation 10 is applicable to any kinetic scheme that looks like Scheme 1 or 5 regardless of the isomeric relationship of the starting materials. In the case of isomeric substrates that cannot be easily separated and their initial ratio manipulated as described above separate kinetic experiments are required to determine individual rate constants. To date there has not been any experimental verification of this new idea reported in the literature; however, the 2003 report did survey a number of published chemical systems where enough experimental detail was given so that minimum estimates of ε_{DKR} could be made and thus confirmed in principle the validity of eq 10. In that work several variant kinetic schemes were examined and their properties and corresponding exact initial and final product ratio expressions were derived. The important point is that all prior work on such kinetic systems *assumed* from the outset that the Curtin–Hammett condition was valid and that Winstein–Holness kinetic behavior was applicable. These claims were then verified by carrying out *independent* theoretical calculations on energy barriers pertaining to the relevant transition states. The present analysis shows that the Curtin–Hammett condition may be verified directly from experimentally acquired data. Moreover, even if it turns out that this special condition may not be valid for a given chemical system it is still possible to quantify the actual extent of meeting this condition according to the parameter defined in eq 10.

Brief Biographical Sketch

My discovery of Acree's work came unexpectedly from reading Tarbell and Tarbell's book on the history of organic chemistry in the United States [54]. In the literature this reference and another two [6, 55] are the only ones that mention Acree's seminal work. A photograph of him appears in Tarbell and Tarbell's book on page 72. Further research revealed that very little has been written about Acree's contributions to chemistry. Two very short anonymous obituary notices appeared in the scientific literature in 1957 [56–57] and Tarbell and Tarbell presented an oral presentation at the 182nd ACS meeting held in New York City August 23–28, 1981 on Acree's contributions to reaction mechanisms between 1904 and 1916 [58]. His name is also entered in four compilations of scientists [59–62]. No full biography has been published and the National Institute of Standards and Technology (NIST) where he spent most of his career kept no biographical records; however, the librarian there indicated two obituary notices did appear in the *Washington Post* (October 26, 1957 page B2) and in *The New York Times* (October 30, 1957 page 29).

In the next section a summary of Acree's family history, education, and contributions to chemical science is presented based on these few sources and a thorough literature search of his scientific papers. It is evident from this compilation that Acree made significant contributions to fundamental analytical, physical, and mechanistic chemistry, and was well ahead of his time in addressing the renewable energy problem by examining the utility of agricultural waste as a useful biofeedstock to produce sugars from wood, in particular.

Solomon Farley Acree (December 18, 1875–October 23, 1957) was born in McGregor, Texas the first child of George Wren Acree (1852–1884) and Elizabeth Virginia Acree (née

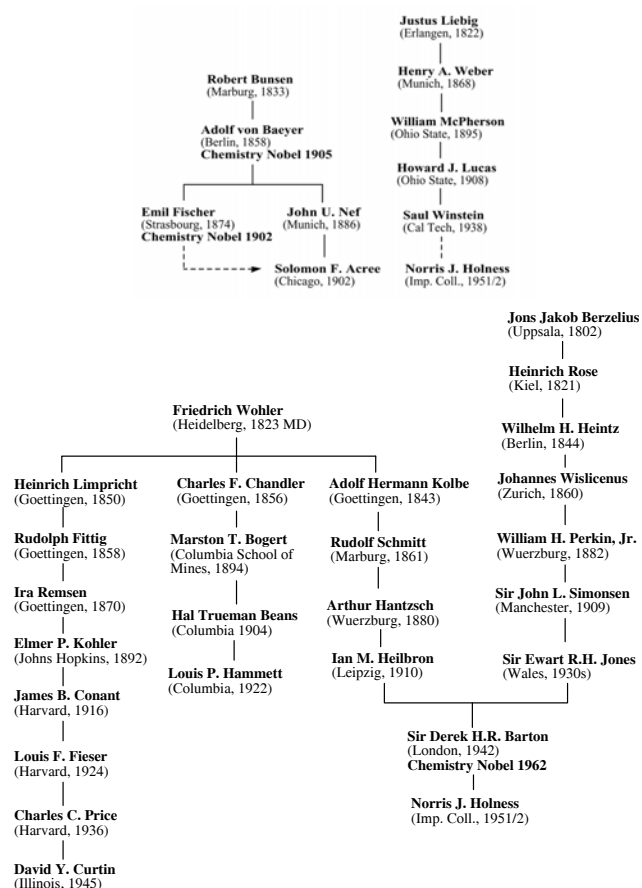


Figure 2. Scientific genealogies of Solomon F. Acree, David Y. Curtin, Louis P. Hammett, Saul Winstein, and Norris J. Holness.

Grimes). The genealogy of the Acree name in Texas can be traced to his paternal grandfather Robert Acree, the son of a tailor, who immigrated at 18 years of age from England to Virginia. After periods in Harrisburg (Mississippi), Augusta (Texas), and Boston (Massachusetts) he settled as a farmer in Flatonia, Texas. The county of Grimes in Texas bears the maiden name of Solomon's mother. The lineage can be further traced back to Sir Christopher Wren (1632–1723), a great English architect of the 17th century who helped found the Royal Society and designed well known structures as the chapel at Pembroke College and the library at Trinity College at Cambridge University, Sheldonian Theatre in Oxford, and after the Great Fire of London of 1666 the new St. Paul's Cathedral, the Royal Exchange, Custom House, Temple Bar, the College of Physicians, Greenwich Observatory, Chelsea Hospital, the Ashmolean Museum at Oxford, Hampton Court, Greenwich Hospital, Buckingham House, Marlborough House, and the western towers and north transept of Westminster Abbey. Another lineage through Elizabeth Acree, a sister of Solomon's father, by marriage to Robert R. Crockett links the Acree family tree to that of David (Davy) Crockett (1786–1836), the Alamo hero, frontiersman, and politician known colloquially as the "king of the wild frontier." This makes Solomon Acree a great nephew of Davy Crockett!

Solomon's father was born in Harrisburg, Mississippi; moved first to Augusta, Texas; and then settled in Flatonia. The Acrees owned land but gave up farming and went into the mercantile business in 1878. When his father died Solomon was 9 years old and his mother worked as a school teacher to

raise him and his two younger brothers Robert Frederick Acree and John Harrison Acree who were 7 and 3 years of age respectively at the time. Solomon married Ruby Jarvis Tiller and had a son George Wren Acree and a daughter Ruby J. Acree Clement.

Solomon obtained his B.S. and M.S. degrees from the University of Texas in 1896 and 1897 respectively and then obtained his Ph.D. from the University of Chicago in 1902 under John U. Nef with a thesis "On Sodium Phenyl and the Action of Sodium on Ketones." In 1903 he did post-doctoral work at the University of Berlin in the laboratory of Emil Fischer who was a student of Adolf von Baeyer who in turn was Nef's doctoral advisor. A scientific genealogy tree of Acree, Curtin, Hammett, Winstein, and Holness is shown in Figure 2. Acree's studies on tautomerism of urazoles was probably inspired by earlier contributions to the discovery of tautomeric structures by several workers including Baeyer. Acree was associate professor at the University of Utah (1901–1904), Johnston scholar (1905–1906), and associate professor (1907–1914) at Johns Hopkins University. After this time he changed positions frequently. Between 1914 and 1917 he was a chemist at Forest Products Labs and professor of forestry chemistry at the University of Wisconsin. From 1917 to 1919 he was at SUNY at Syracuse in the College of Environmental Science and Forestry. From 1919 to 1920 he was a chemist at the Hahemann Hospital in Rochester, New York. From 1920 to 1924 he was technical director and Vice President of Graham Chemical Company and International Chemical Products Company. From 1924 to 1926 he returned to academia as a professor at George Washington University. From 1927 to his retirement he then settled as senior chemist at the National Bureau of Standards, now the National Institute of Standards and Technology (NIST). There he pioneered along with Roger G. Bates the accurate determination and standardization of acidity constants for organic acids and bases using various electrodes in the Hydrogen Ion Measurements Section. He also headed the Fiber Structures Section where his work on biofeedstocks and renewable energy sources was done. Over his extensive experience in academia and industry he made contributions in many areas of pure and applied chemistry beyond the already cited topics of tautomerism and the forerunner work of the Curtin–Hammett principle.

During his time at Berlin he made contributions on triphenylcarbinols and triphenylmethanes [63], 1-naphthylmagnesium bromide [64], tolyldiphenylcarbinol [65], phenyl sodium and alkylmagnesium bromides [66], and the esterification of benzoic acid and mandelic acid [67]. Other areas in which he made significant contributions include enzymes [68–69], formaldehyde color test for proteins often called the Acree color test [70], catalysis [71–90], the pinacol-pinacolone rearrangement [91], electrolytes [92–95], electrical conductivity of solutions [96–101], dilatometry [102], the use of dyestuffs as pH indicators and the quinone-phenolate theory [102, 103–115], hydrogen and calomel electrodes [117–127], ethylene electrode [128] lead electrode [129], glass electrode [130–131], ionization constant measurements for acids and bases [132–153], buffers [154–166], carbohydrates [169–181], inversion of menthone [182–183], the chemical constituents of poison ivy [184–185], oxidation and reduction reactions [186], determination of diazoalkylenes by titration [187], semicarbazides [188], dipole moment measurements [189–190], UV spectrophotometry [191–192], activity coefficient measurements [193–194], determination of ascorbic acid

concentrations in urine and blood [195], potentiometric titration [196], the industrial utilization of farm and wood waste to retrieve monomeric sugars such as mucic acid, xylose, pentoses, and aldoses [197–207], and wood and creosote chemistry [208–214].

Conclusion

It is important for students, teachers, and researchers of chemistry to know their subject well. This means knowing both the content as well as being able to trace that knowledge back to its origin so that a properly connected network of ideas is built up in the mind. This serves a number of important purposes. One becomes a better researcher in science and a better teacher of science. One is able to get a “big picture” view of their subject which allows one to connect the dots between past ideas to come up with the next good idea and to identify gaps in the knowledge database thereby making contributions to fill in the missing connections between concepts. This is particularly powerful when the connected dots are from disparate fields. One is also able to appreciate better their subject and their place in it. It gives students especially the confidence to forge ahead with their own fresh ideas and to build constructively on what others have done before them while being guided by a thorough investigation of the literature. A good discussion of these points is given in a recent account of the development of physical organic chemistry [56, 215]. In light of the findings presented here it is fair now to recognize Solomon Acree’s contribution to the Curtin–Hammett principle especially that 100 years have elapsed since his seminal work. Future editions of textbooks on organic chemistry should be revised to mention the origin of this principle and amend its name to the Acree–Curtin–Hammett principle. The introduction of a new kinetic plasticity index both demonstrates that this principle can be applied generally to any pair of equilibrating structures regardless of their structural relationship and quantifies succinctly the degree to which this special condition is met in such chemical systems.

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