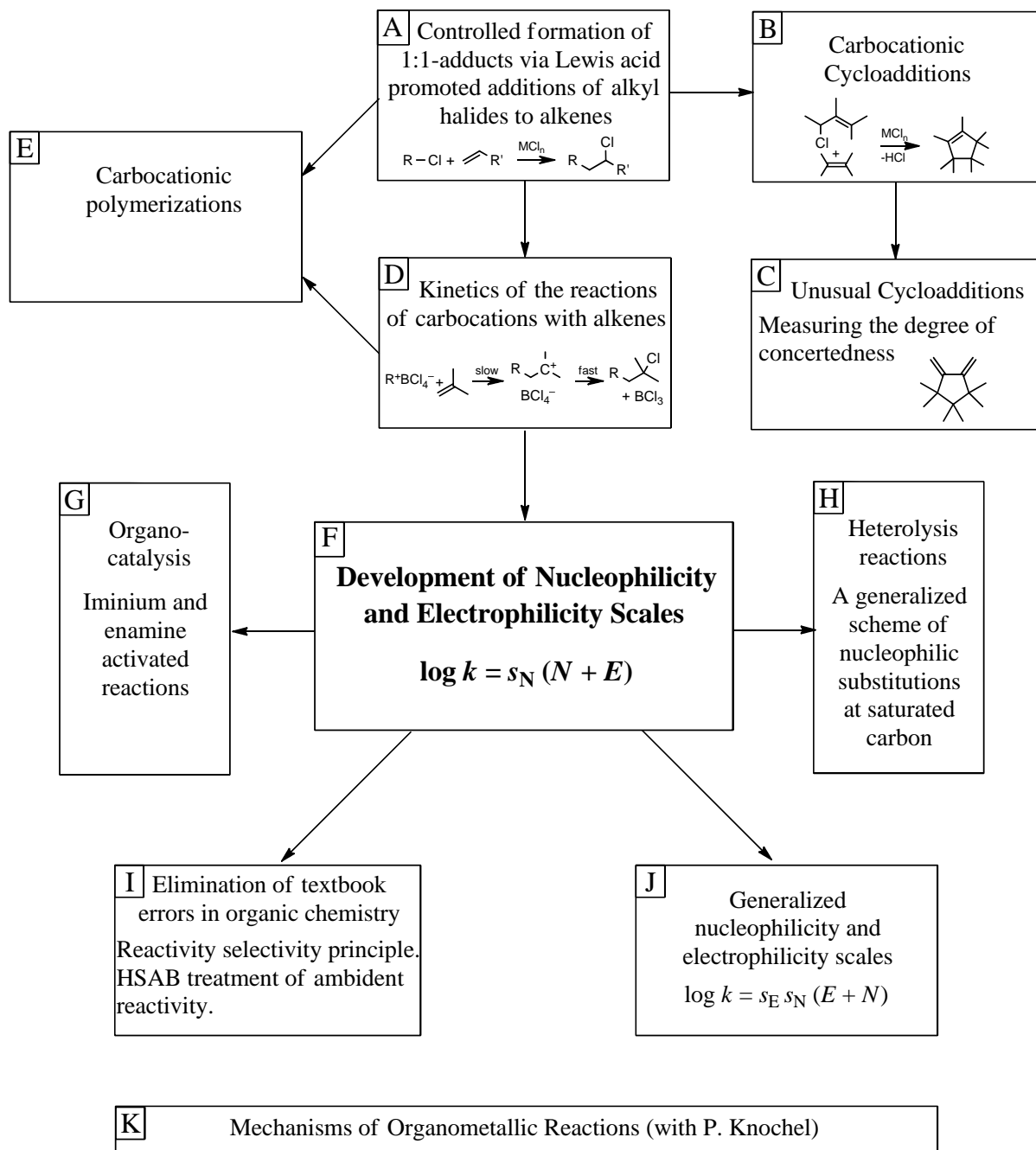


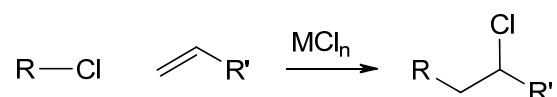
Research Activities in the Mayr Group

(past and present, Aug. 2011)



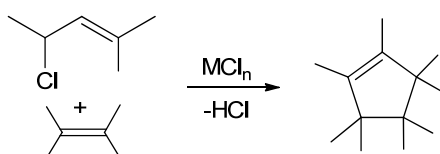
A. Aliphatic Friedel-Crafts Reactions

Lewis acid induced reactions of alkyl halides with alkenes may either yield 1:1 adducts or lead to polymers. In the early 1980s, rules were developed how to selectively produce [1:1]-adducts and avoid the formation of polymers (#19, #34): When catalytic amounts of Lewis acids are employed, the reactant RX must ionize more readily than the product R-C-C-X. With stoichiometric quantities of strong Lewis acids, this rule must be reversed (#100, #136)



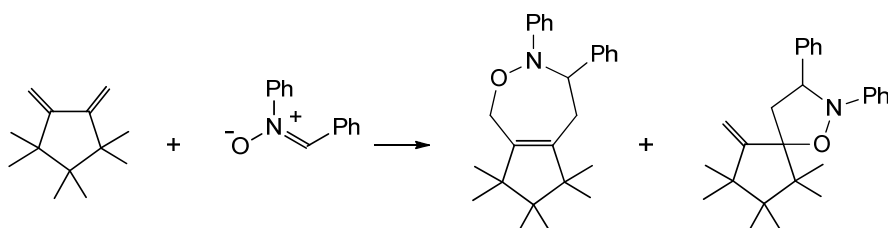
B. Carbocationic Cycloadditions

The rules developed in A were employed to realize carbocationic cycloadditions. Selective formations of 4-, 5-, 6-, and 7-membered carbocycles were achieved by Lewis acid catalyzed reactions of propargyl- and allyl halides with alkenes and 1,3-dienes (#17, #18, #20, #29-#33)



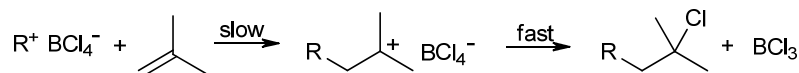
C. Unusual Cycloadditions

Octamethylcyclopentene, synthesized as described in B was transformed into a *s-cis* fixed 1,3-diene with sterically shielded 2- and 3-positions. The investigation of unusual cycloadditions of this diene (review: #106), e.g. [4+3]-cycloadditions of nitrones (#65, #94), [4+1]-cycloadditions of carbenes (# 43), [4+2]-cycloadditions of ketenes (#69), [4+3]-cycloadditions of 2-azaallyl anions (# 117), and noncatalyzed [4+4]-cycloadditions of 1,4-dienes (#93) gave insight in the “concertedness” of various cycloadditions.



D. Kinetics of the Reactions of Carbocations with Alkenes

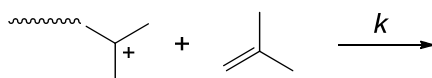
The rules developed in A were used to develop a kinetic method for determining the rates of attack of carbocations at CC-double bonds.



Though this type of reaction represents the key-step of many transformations in organic, macromolecular, and biochemistry, prior to our work, rate constants for such processes were essentially unknown. Three communications in *Angewandte Chemie* (#47, #56, #57) and three back to back full papers in *J. Am. Chem. Soc.* (#95, #96, #97) changed this situation abruptly. Nowadays, hundreds of rate constants, mostly by us, are available, and electrophilic alkylations of alkenes are now among the best understood CC-bond forming reactions (#100).

E. Carbocationic Polymerizations

The methods developed in A and D were used in Macromolecular Chemistry for designing initiators, coinitiators, and comonomers in carbocationic polymerizations (#137, #152, #157, #186, #206). As reviewed by Puskas (*J. Polymer Science: Part A: Polymer Chem.* **2005**, 43, 5394) our work changed the previously accepted value for the propagation rate constant of cationic isobutylene polymerization by several orders of magnitude. The diffusion-clock method, a well-established tool in Physical Organic Chemistry, was introduced into Macromolecular Chemistry (#140) and has been used for determining numerous propagation rate constants (Faust et al.).



F. Nucleophilicity and Electrophilicity Scales.

The kinetic methods developed in Part D were analogously employed to determine rate constants for the attack of carbocations and Michael acceptors at various C-nucleophiles (allylsilanes, allylstannanes, carbocyclic and heterocyclic arenes, enol ethers, enamines, diazoalkanes, metal- π -complexes, carbanions, isonitriles, ylides), hydride donors, halide and pseudohalide anions, N-nucleophiles (amines, pyridines,

amide anions, azoles), as well as P-, O-, and S-nucleophiles. By defining benzhydrylium ions, quinone methides, and benzylidene malonates as reference electrophiles, which cover a reactivity range of more than 30 orders of magnitude, it has become possible to generate the most comprehensive nucleophilicity scale, presently available (#124, #191, #219, #250, #262).

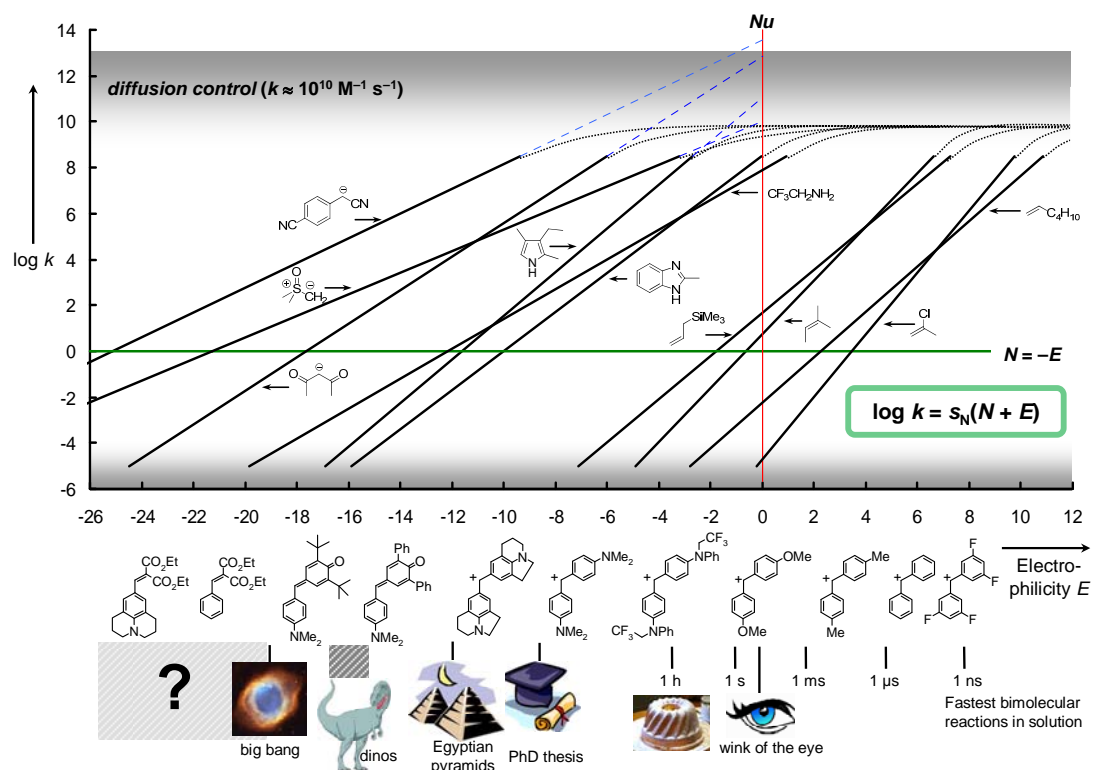


Figure 1. Rate constants for Electrophile-Nucleophile Combinations

It has been shown that equation 1, where electrophiles are characterized by one parameter (E) while nucleophiles are characterized by the nucleophilicity parameter N and the nucleophile-specific sensitivity parameter s_N can be used to predict absolute rate constants with an accuracy of factor 10 to 100 in an overall reactivity range of 40 orders of magnitude.

$$\log k_{20^\circ\text{C}} = s_N(N + E) \quad (1)$$

For qualitative analyses, the sensitivity factor s_N can be neglected, and as a rule of thumb one can expect electrophile-nucleophile combinations to take place at room temperature if $(N + E) > -5$. Since diffusion limit is reached at $k = 10^9 - 10^{10} \text{ M}^{-1}\text{s}^{-1}$, chemo-, regio- and stereoselectivity often break down when $(N + E) > 10$. As a consequence, most synthetically used reactions are found in the green corridor of Figure 2.

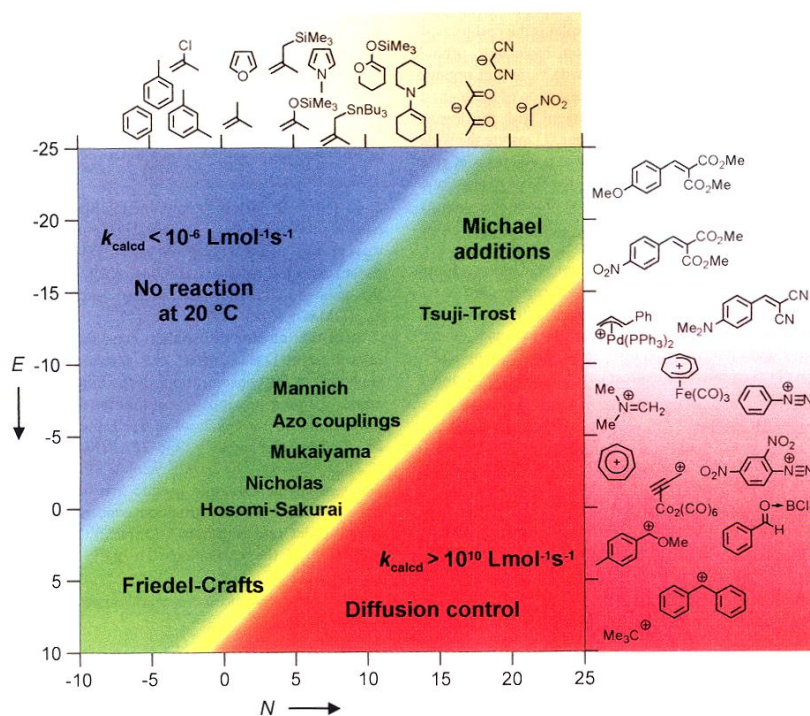


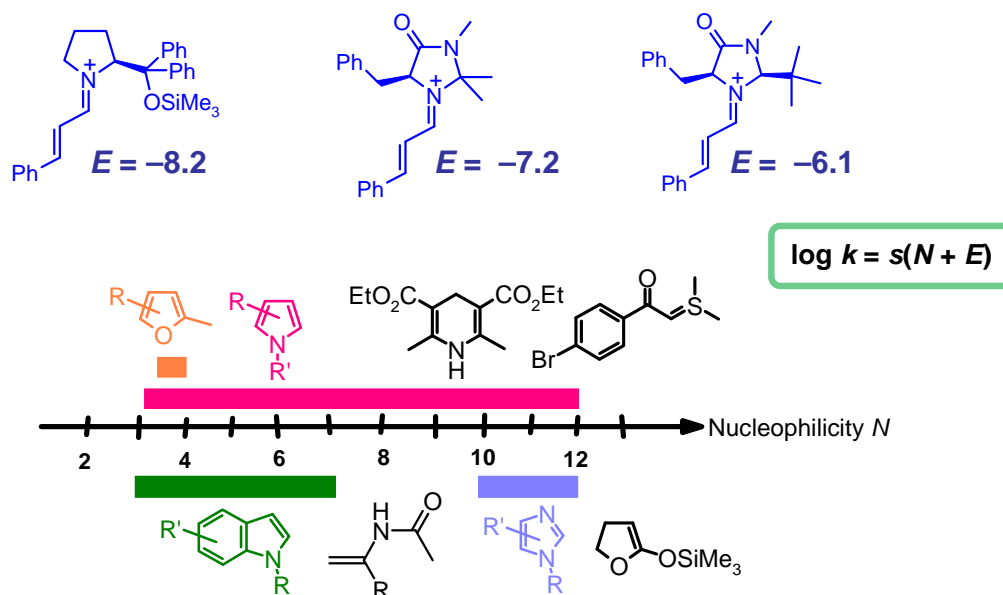
Figure 2. Where to find synthetically useful reactions

Open access to our database of electrophilicity and nucleophilicity parameters is provided at <http://www.cup.uni-muenchen.de/oc/mayr/DBintro.html>.

Poster presentations of our reactivity scales which can be used for synthesis planning can be downloaded (<http://www.cup.lmu.de/oc/mayr/ReactScalesPoster.pdf>).

G. Nucleophilic Organocatalysis

The key-steps in many organocatalytic cycles are electrophile-nucleophile combinations as investigated in part F. Benzhydrylium ions and structurally related quinone methides have been employed to compare nucleophilicities (k) and Lewis basicities of tertiary amines (#239, #275, #287), pyridines (#235, #302), amidines (#255), and phosphines (#214), i.e., nucleophilic organocatalysts. We have determined reactivity parameters of intermediates, e. g. iminium ions (#260) or enamines (#195), as well as of potential substrates of iminium and enamine activated reactions (#270, #284, #295). As nucleophiles which can be employed for iminium activated reactions should be active enough to react fast with iminium ions but not too active in order to avoid reactions with the precursor carbonyl compounds, nucleophiles with $3 < N < 12$ appear to be the most suitable substrates (#290).



Nucleophiles must be strong enough to react with iminium ions and weak enough not to react with the precursor carbonyl compounds.

Figure 3. Nucleophiles suitable for iminium activated reactions

H. Heterolytic Cleavages: The S_N1 – S_N2 -Spectrum

The method which allowed us to develop the comprehensive nucleophilicity and electrophilicity scales in section F has analogously been employed to develop nucleofugality scales (leaving group abilities) (#300).

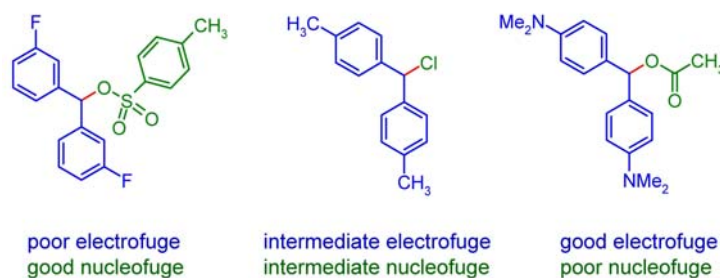


Figure 4. Electrofuge-nucleofuge combinations which dissociate with measurable rates

Solvolysis rate constants of combinations of poor electrofuges with good nucleofuges as well as of good electrofuges with poor nucleofuges (Figure 4) have been determined to provide a semiquantitative scheme of heterolysis rates (Figure 5).

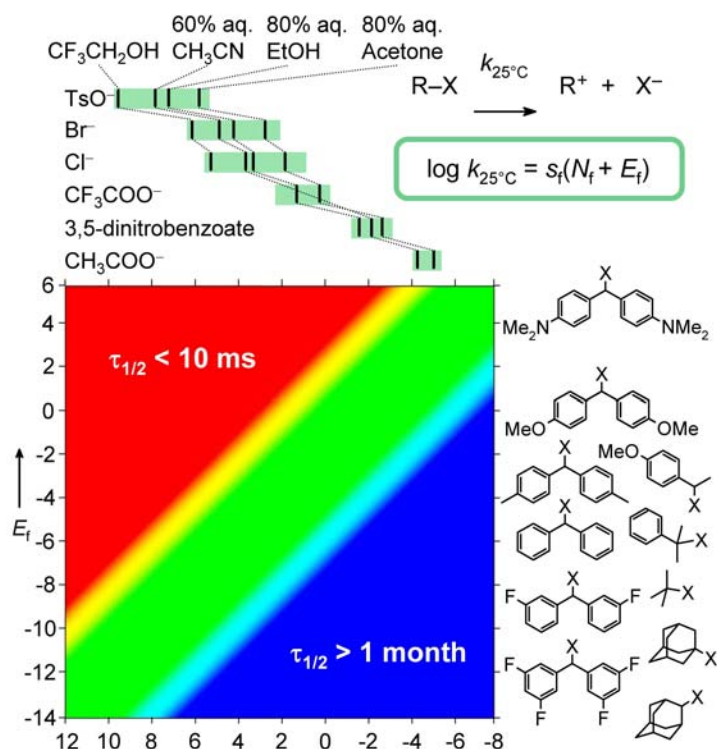
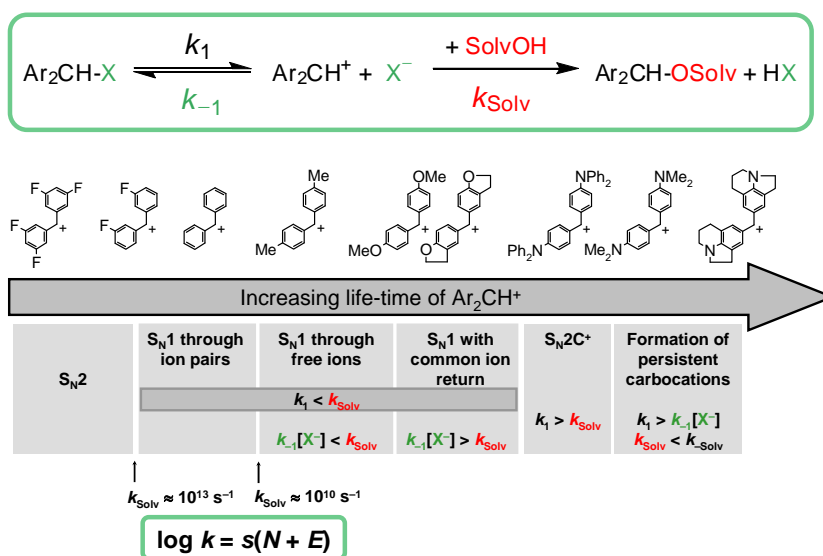


Figure 5. A practical guide for estimating rates of heterolysis reactions

By developing a stopped-flow technique, which allows the investigation of solvolysis rate constants in the millisecond time scale (#248), the green corridor of Figure 5, which indicates the experimentally accessible range has almost been doubled (#248). As illustrated in Figure 6, it has become possible to quantitatively predict the mechanistic change from S_N2 reactions over ordinary S_N1 reactions with and without ion return to heterolytic cleavages with formation of persistent carbocations (#268, #272)



I. General Concepts of Organic Chemistry.

The reactivity scales developed in Section F have been employed for a rigorous examination of common concepts of Organic Chemistry. Though the reactivity selectivity principle has been abandoned as a general rule more than 30 years ago, many chemists still believe that it is generally applicable, violated just by a few exceptions. In a review we have shown, why it is impossible that selectivity generally decreases with increasing reactivity (#228).

Detailed experimental studies on the ambident reactivities of SCN^- (#204), CN^- (#215), NO_2^- (#221), OCN^- (#256), PhSO_2^- (#283), and the pyridine anions (#297) showed that the "principle of hard and soft acids and bases" or the related Klopman-Salem concept of "frontier- and charge controlled reactions" does not even correctly describe the regioselectivities of the prototypes of ambident nucleophiles. In a quantum chemical investigation we have demonstrated that a consistent description of activation-controlled can be based on Marcus theory (#288).

An extensive review on ambident reactivity provides a consistent analysis of ambident reactivity on the basis of Figure 7 and demonstrates that the application of PMO theory on reactivities of ambident anions toward carbocations often implies extrapolations to transition states of reactions, which do not have a transition state as they undergo activation-less combinations (#305).

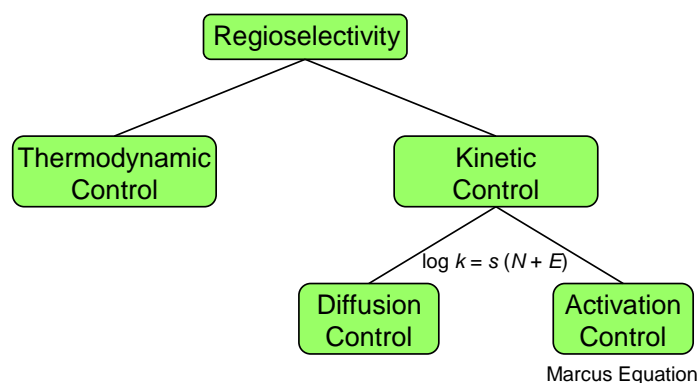


Figure 7. A systematic analysis of ambident reactivity.

J. Relationships Between Different Reactivity Scales.

Investigations of the rates of the reactions of the S-methyldibenzothiophenium ion with large variety of nucleophiles gave an excellent linear correlation of $(\lg k)/s_N$ vs N . The slope of this correlation which ranged from $1 < N < 18$ was 0.61, indicating that equation 1 has to be extended by an electrophile-specific sensitivity parameter s_E in order to include also S_N2 type reaction (#231). As shown in Figure 8, the

general equation (2) includes equation 1, the Swain-Scott and Ritchie equation as special cases.

$$\log k = s_E s_N (E + N) \quad (2)$$

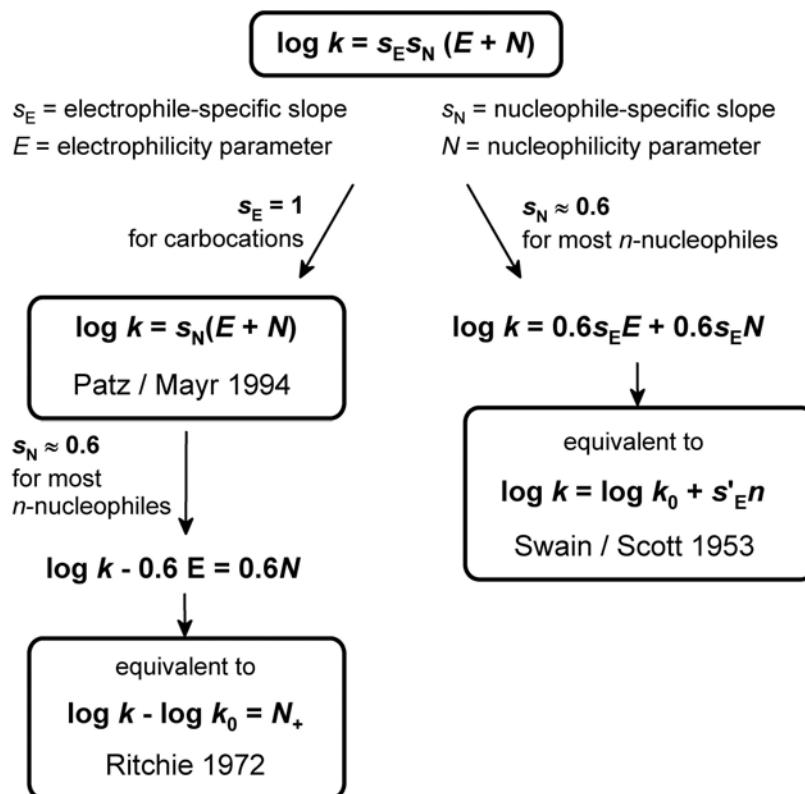


Figure 8. Relationships between various correlation equations.

We are presently investigating the physical basis of these correlations.

K. Mechanisms of Organometallic Reactions

In collaboration with the Knochel group, we are investigating structure-reactivity relationships in organometallic reactions. Competition experiments on magnesium/halide exchange reactions in haloarenes with $i\text{PrMgCl}\cdot\text{LiCl}$ (#246, #269, #274) and in Negishi cross-couplings (#279) have revealed the different transition states in the corresponding metallation steps (Figure 9).

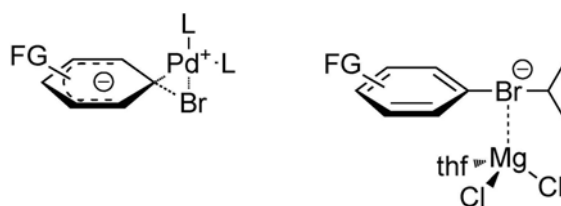


Figure 9. Comparison of the transition states of the oxidative addition of PdL_2 and of the Br-Mg exchange with $i\text{PrMgCl}\cdot\text{LiCl}$.

Prof. Dr. Herbert Mayr

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(last update: 11.8.2011)

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