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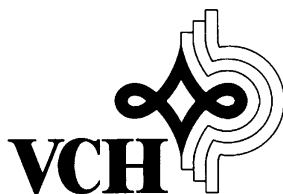
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Sextettumlagerungen als Solvens-Polaritätssonde

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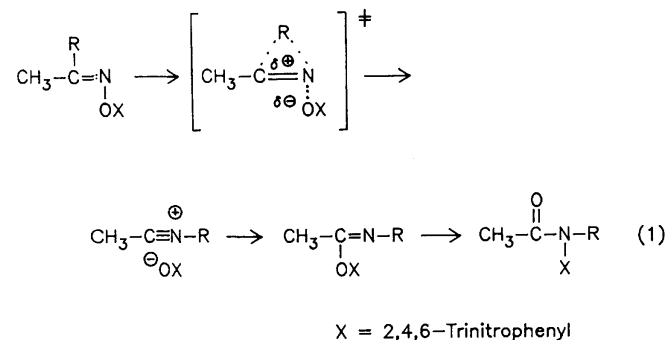
Sextett Rearrangements as Solvent Polarity Probes

The rate constant of the Chapman variant of the Beckmann rearrangement is used as a solvent polarity probe and is

mainly influenced by the polarizability of the solvent. Analogies to S_N reaction are discussed.

Die von Winstein und Grunwald¹⁾ entwickelte empirische γ -Solvenspolaritätsskala basiert auf der Solvens-Abhängigkeit der Geschwindigkeitskonstante einer chemischen Reaktion – der Solvolysereaktion von *tert*-Butylchlorid (Übersicht Lit.^{2,3)}). Dieses Meßprinzip lieferte eine für praktische Probleme zunächst sehr erfolgreiche Polaritätsskala. Es erwies sich aber im Nachhinein als problematisch, da es auf Medien großer Polarität beschränkt ist und zudem Unsicherheiten durch das Auftreten verschiedener Ionenpaare, Reversibilität der Einzelschritte und zum Teil eine spezifische Solvatation des sich bildenden Kations und Anions aufweist. Dies führte schließlich zur Bevorzugung sekundärer Polaritätsskalen auf spektroskopischer Basis²⁾. Eine breit anwendbare und unproblematische Polaritätsskala auf der Basis einer chemischen Reaktion wäre eine wertvolle Ergänzung der bereits vorhandenen Skalen. Die Bersonsche ω -Skala⁴⁾, die auf der Lösungsmittelabhängigkeit einer Diels-Alder-Reaktion beruht, erfüllt dies nur bedingt, da sie zum einen als bimolekulare Reaktion prinzipiell störanfällig gegen Aggregationsphänomene⁵⁾ ist und zum anderen bei ihr im geschwindigkeitsbestimmenden Schritt keine Ionisation erfolgt.

Die Geschwindigkeitskonstante der Beckmann-Umlagerung⁶⁾ ist stark solvensabhängig und soll hier als Polaritätsmaß verwendet werden, weil sie sich als Meßsonde besonders eignet. Von der Beckmann-Umlagerung ist der Mechanismus der Chapman-Variante⁷⁾, die Umlagerung von Ketoxim-pikraten, eingehend untersucht worden⁸⁻¹⁴⁾. Für *anti*-Methylketoxim-pikrate, die synthetisch gut zugänglich sind¹⁰⁻¹⁴⁾, folgt die Reaktion dem Weg nach Gl. (1).



Der geschwindigkeitsbestimmende Schritt der Reaktion ist die Wanderung des Restes R unter Bildung eines Nitrilium-Ions¹⁶⁾. Als schnelle Folgereaktionen schließen sich die Rekombination des gebildeten Ionenpaares und schließlich die Bildung des *N*-Pikrylacetamids an.

Wir halten die Geschwindigkeitskonstante der Chapman-Variante der Beckmann-Umlagerung als Solvenspolaritätssonde für besonders geeignet, die auch von Fischer¹⁵⁾ zur Untersuchung von Solvens-Effekten bereits verwendet wurde, da nach den bisherigen Kenntnissen der geschwindigkeitsbestimmende Schritt der Reaktion, die Ionisation, irreversibel erfolgt (vgl. Lit.^{6,17)}). Hierfür spricht auch die starke negative Reaktionsenthalpie von ca. -80 kcal/mol¹⁸⁾, die bei einer Aktivierungsenthalpie von ca. 30 kcal/mol^{13,14)} für die Alkyl-Wanderung einen reversiblen Schritt nicht zulassen sollte. Ein weiterer Vorteil ist die räumliche Trennung zwischen der wandernden Gruppe R, die im Übergangszustand einen großen Teil der entstehenden positiven Partialladung trägt¹⁴⁾, und der Abgangsgruppe $\ominus\text{OX}$. Spezifische Solvatationen des entstehenden Ionenpaares sollten damit weniger wahrscheinlich werden. Schließlich ist noch der große molare Extinktionskoeffizient der Pikrate wichtig, der eine präzise UV-spektroskopische Messung der Konzentration auch bei starker Verdünnung zuläßt, wodurch das zu untersuchende Medium nur wenig gestört wird.

Als wandernde Gruppen R kommen Alkyl- oder Aryl-Reste in Frage. Bei Aryl-Resten besteht grundsätzlich die Möglichkeit der Bildung von π -Komplexen. Um diese mögliche Komplikation auszuschließen, bevorzugen wir Alkyl-Reste. Eine schließlich noch denkbare spezifische Rückseitensolvatation von R wird durch die Verwendung solcher Brückenkopfrete unmöglich gemacht, bei denen dieser Bereich abgeschirmt ist. Als Brückenkopfrete R werden die Strukturen 1–4 eingesetzt. Über einen Vergleich der Solvens-effekte bei diesen strukturell sehr unterschiedlichen Substraten lassen sich spezifische Solvatationen erkennen und ausschließen.



Als zu untersuchende Lösungsmittel werden Methanol (1), Ethanol (2), 1-Butanol (3), DMSO (4), DMF (5), CH_2Cl_2 (6), CHCl_3 (7) und THF (8) verwendet, die sich in der Art ihres Solvatationsvermögens stark unterscheiden^{2,18,20)}. Die Methylketoxim-pikrate mit den Resten 1–4 lagern in diesen Lösungsmitteln nach 1. Ordnung um (in THF bis ca. 63% Umsatz). Die Geschwindigkeitskonstanten der Reaktionen sind in Tab. 1 angegeben. Ein Vergleich der Werte liefert zunächst das wichtige Ergebnis, daß bei allen verwendeten Solvenzen die Verhältnisse der Geschwindigkeitskonstanten bei den Resten 1–4 etwa gleich sind – dies spiegelt sich auch in Abb.

1 (oben) wieder. Hieraus muß geschlossen werden, daß spezifische Solvatationen der verschiedenen Strukturen nur klein sein können und daß mit der Meßmethode im wesentlichen die globalen Solvenseffekte erfaßt werden. Stärkere Abweichungen werden lediglich bei den Lösungsmitteln CH_2Cl_2 und CHCl_3 beobachtet.

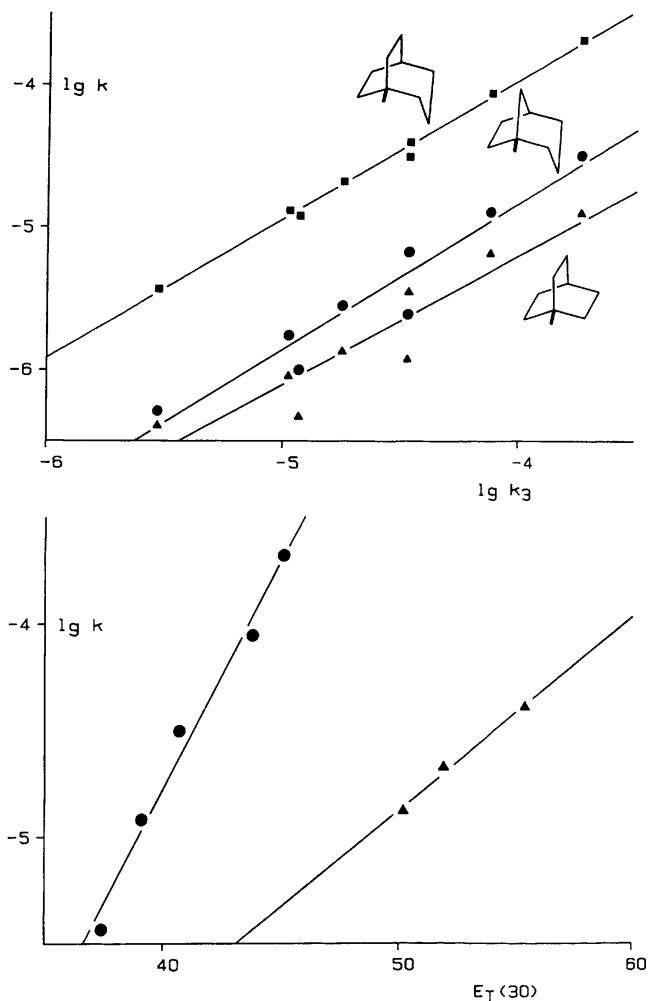


Abb. 1. (oben): Lineare Korrelation zwischen $\lg k$ von 3 und $\lg k$ von 1 (■), 2 (●) und 4 (▲) in verschiedenen reinen Lösungsmitteln (Numerierung siehe Tab. 1); (unten): lineare Korrelation zwischen den $\lg k$ -Werten der Beckmann/Chapman-Umlagerung von 3 in Tab. 1 und den $E_T(30)$ -Werten nach Dimroth und Reichardt. — ▲: Alkohole [$\lg k = 9.09 \cdot E_T(30) - 9.4$, $r = 0.994$]. ●: sonstige Lösungsmittel [$\lg k = 0.21 \cdot E_T(30) - 13.3$, $r = 0.990$]

Vergleicht man von Tab. 1 ausgehend die $\lg k$ -Werte mit den $E_T(30)$ -Solvens-Polaritätswerten nach Dimroth und Reichardt^{21,22}, so findet man, wie in Abb. 1 (unten) dargestellt, zwei lineare Korrelationen — eine für Alkohole und eine für die übrigen Lösungsmittel (für die Korrelation sind die gemessenen $E_T(30)$ -Werte der verwendeten Lösungsmittelchargen aufgetragen). Es fällt insbesondere auf, daß die Umlagerung in DMF und DMSO [Nr. (4) und (5)] sehr schnell erfolgt. In vorangegangenen Arbeiten^{19,23} wurde gezeigt, daß diese Lösungsmittel über ihre besonders große mikroskopische Polarisierbarkeit solvatisierend wirken. Ein brauchbares Maß für diese Effekte ist die χ_R -Skala nach Brooker²⁴, die ebenfalls linear mit $\lg k$ korreliert.

Damit ist offensichtlich, daß eine Solvatation über die Polarisierbarkeit des Lösungsmittels einen wichtigen Beitrag bei der Sextettumlagerung liefert. Andererseits sind aber auch Orientierungs-

phänomene der Solvenzien wichtig, wie dies von der linearen Korrelation mit der $E_T(30)$ -Skala widergespiegelt wird. Die letzteren Effekte nehmen aber bei Solvolysereaktionen eine zentrale Stellung ein — vgl. z. B. die verhältnismäßig gute Korrelation zwischen den $E_T(30)$ - und Y -Werten²¹. Wir sind der Meinung, daß diese Unterschiede auf der unterschiedlichen Ladungsverteilung in den beiden Systemen beruhen. Bei den Solvolysereaktionen entsteht eine punktförmige Ladung — das Carbenium-Ion (hierbei muß allerdings auch noch die Solvatation des Edukts berücksichtigt werden²⁵), das gut durch eine Umorientierung des Lösungsmittels und auch speziell von den Wasserstoff-Brückenassoziaten der Alkohole solvatisiert werden kann. Bei der Beckmann/Chapman-Umlagerung sind dagegen die entstehenden Ladungen über viele Atome verteilt. Eine Orientierung des Lösungsmittels und der Wasserstoff-Brückenassoziate können sich bei der geringen Ladungsdichte viel weniger auswirken — daher die zweite, versetzte Gerade für Alkohole mit einer geringeren Steigung —, stark dagegen wirkt sich die Polarisierbarkeit des Lösungsmittels aus. Dies kann auch bei anderen Reaktionen, z. B. nucleophilen Substitutionen, von Bedeutung sein und könnte auch die starke Reaktionsbeschleunigung²⁶ erklären, die in einigen Fällen auftritt, wenn auch nur kleine Anteile DMSO den polar-protischen Lösungsmitteln zugesetzt werden.

Schließlich ist zu fragen, ob der reaktionsbeschleunigende Effekt von DMSO und DMF nicht etwa auf einer Komplexbildung beruhen könnte. Um dies auszuschließen, wurde die Geschwindigkeit der Umlagerung des Oxim-pikrats mit $R = 4$ im binären Gemisch $\text{CH}_2\text{Cl}_2/\text{DMSO}$ als Funktion von dessen Zusammensetzung untersucht. Es konnte dabei gezeigt werden, daß $\lg k$ quantitativ von der Zwei-Parameter-Gl. (2) beschrieben wird, die allgemein für die Polarität binärer Gemische als Funktion ihrer Zusammensetzung gilt^{19,27}.

$$\lg \frac{k}{k^0} = E_D \cdot \ln(c_p/c^* + 1) \quad (2)$$

c_p in Gl. (2) ist die molare Konzentration der stärker polaren Komponente, hier DMSO, $\lg k$ gilt für das Gemisch und $\lg k^0$ für die reine, weniger polare Komponente, hier CH_2Cl_2 . E_D und c^* sind die Parameter der Gleichung.

Eine Auftragung von $\lg k/k^0$ gegen $\ln(c_p/c^* + 1)$ ist in Abb. 2 angegeben.

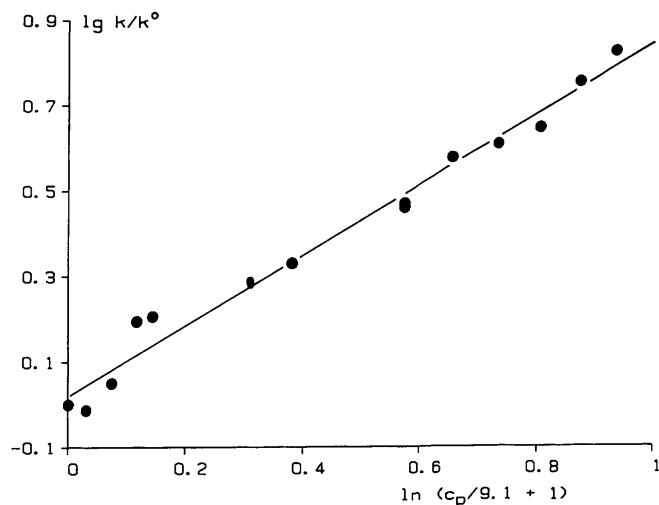


Abb. 2. Lineare Beziehung zwischen $\lg(k/k^0)$ und $\ln(c_p/c^* + 1)$ nach Gl. (2) für das Gemisch $\text{CH}_2\text{Cl}_2/\text{DMSO}$ (Oxim-pikrat mit $R = 4$)

Die lineare Beziehung ($E_D = 0.863 \text{ kcal} \cdot \text{mol}^{-1}$, $c^* = 9.73 \text{ mol} \cdot \text{l}^{-1}$, $r = 0.991$ bei 14 Meßwerten) belegt, daß eine normale Solvation durch das DMSO erfolgt. Der c^* -Wert, der die Wechselwirkung der stärker polaren Komponente widerspiegelt, liegt im mittleren bis großen Bereich (vgl. Lit.¹⁹). — Dies spricht ebenfalls für ein normales Solvationsverhalten von DMSO.

Tab. 1. Solvensabhängigkeit der Reaktionsgeschwindigkeit ($k \cdot 10^7$ [s^{-1}]) von Methylketoxim-pikraten bei 25°C

Solvens	Rest R			
	1-Bicyclo- [2.2.2]octyl (1)	1-Bicyclo- [3.2.1]octyl (2)	1-Ada- mantyl (3)	1-Bicyclo- [3.2.2]nonyl (4)
Methanol (1)	35.2	67.5	338	397
Ethanol (2)	13.4	28.3	178	210
1-Butanol (3)	9.03	17.5	106	131
DMSO (4)	126	322	1830	2080
DMF (5)	65.3	128	750	878
CH ₂ Cl ₂ (6)	11.9	24.7	337	313
CHCl ₃ (7)	4.67	10.0	117	120
THF (8)	4.05	5.15	29.5	36.8

Experimenteller Teil

Die verwendeten Oxim-pikrate wurden nach Literaturangaben hergestellt^{13,14}. Die Umlagerung der Pikrate wurde UV-spektroskopisch^{13,14} bei 25°C verfolgt (Spektrometer LAMBDA 3 von Perkin-Elmer) und gehorchte streng dem Zeitgesetz 1. Ordnung. Sie wurde routinemäßig bis zu Umsätzen von 85% verfolgt. In THF wurden die Meßwerte bis zu einem Umsatz von 63% verwendet, da bei höherem Umsatz Abweichungen von der 1. Ordnung auftraten. Die Bestimmung der Geschwindigkeitskonstanten beruht auf 2 bis 3 Meßreihen zu je 10 bis 15 Meßpunkten. Die Genauigkeit der Geschwindigkeitskonstanten beträgt typisch 2%.

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