of χ_D. In obtaining these values, small corrections in Φ_{rad} have been made for the extent to which $^2\tau$ is different in each solution. Now, $\Phi_{rad} = {}^4\eta_{isc}({}^2k_{rad}/{}^2k_0)$ where $^4\eta_{isc}$ is the efficiency of forming ^2E via intersystem crossing from $^4T_2$. Although radiative transition probabilities ($^2k_{rad}$ in this case) are proportional to the square of the index of refraction of the medium,[17] the virtual identity of $n_D{}^{20}$ for H_2O (1.3330) and D_2O (1.3283) and the independence of Φ_{rad} with χ_D for $Cr(phen)^{3+}$ rule out any variation of $^2k_{rad}$. We must conclude that *$^4\eta_{isc}$ for $Cr(bpy)_3{}^{3+}$ decreases with increasing χ_D* and, in contrast, *$^4\eta_{isc}$ for $Cr(phen)_3{}^{3+}$ remains constant*. Thus, Figure 1 shows the dependence of $^4\eta_{isc}$ (relative to its value in H_2O) as a function of χ_D.

If $^4\eta_{isc}$ for $Cr(bpy)_3{}^{3+}$ is taken[18] as ~ 1 in H_2O, Figure 1 shows that $^4\eta_{isc}$ varies with χ_D to ~ 0.2 in D_2O. Inasmuch as $^4\eta_{isc} = {}^4k_{isc}/({}^4k_{isc} + {}^4k_{nr})$, $^4k_{isc} \gg {}^4k_{nr}$ in H_2O but $^4k_{isc} < {}^4k_{nr}$ in D_2O. *The transition from H_2O to D_2O results in a change in the relative values of $^4k_{isc}$ and $^4k_{nr}$ in the two solvents.* Designating the rate contants in pure H_2O as $^4k_{isc}{}^H$ and $^4k_{nr}{}^H$ and those in pure D_2O as $^4k_{isc}{}^D$ and $^4k_{nr}{}^D$, we see from Figure 1 that $^4k_{nr}{}^D/{}^4k_{isc}{}^D \sim 4$; inasmuch as $^4\eta_{isc}{}^H \sim 1$, $^4k_{nr}{}^H/{}^4k_{isc}{}^H \leq 0.1$. If the intersystem crossing process is not sensitive (or at most, only mildly sensitive)[19] to the change in the isotopic composition of the solvent, so that $^4k_{isc}{}^H \sim {}^4k_{isc}{}^D$, then it is readily seen that $^4k_{nr}{}^D \gg {}^4k_{nr}{}^H$. For values of $^4k_{nr}{}^H/{}^4k_{isc}{}^H$ in the range from 0.1 to 0.01, representing $^4\eta_{isc}{}^H$ values of 0.91–0.99, $^4k_{nr}{}^D/k_{nr}{}^H$ has values ranging from ~ 10 to ~ 100.

In order to account for this very large deuterium-favored solvent isotope effect on the nonradiative decay of $^4T_2$ to $^4A_2$, it should be noted that $^4T_2$, having $t_{2g}{}^2e_g{}^1$ orbital population, is distorted (increased Cr–N bond distance) relative to $^4A_2$ ($t_{2g}{}^3$ orbital population). We view $^4T_2$ as having solvent molecules within the opened interligand pockets exhibiting a weak electronic interaction between lone-pairs and the vacant t_{2g} orbital that points into the pockets; the interligand solvent molecules represent the isotopic composition of the bulk solvent. The direction and magnitude of the solvent isotope effect argues against the coupling of the vibrational levels of $^4T_2$ and $^4A_2$ by the solvent; because of the lower vibrational frequencies of D_2O relative to H_2O, D_2O is less efficient in its coupling and would favor a modest hydrogen-favored effect. The solvent involvement in the nonradiative process could arise from the microstructure of the solvent in the immediate vicinity of the complex ion; D_2O is a more structured solvent than is H_2O.[20] If the nonradiative process is entropically more favorable in D_2O than in H_2O with little or no enthalpic difference, then $^4k_{nr}{}^H < {}^4k_{nr}{}^D$. That the entropic effect is significant is supported by our observation that $^4\eta_{isc}$ in D_2O is *increased* in the presence of salts in a manner that parallels the ability of these salts to modify the solvent structure.[20]

Our results with $Cr(phen)_3{}^{3+}$ show an invariance of $^4\eta_{isc}$ toward χ_D. Inasmuch as $^4\eta_{isc}{}^H \sim 1$,[21] $^4k_{isc} \gg {}^4k_{nr}$ in both H_2O and D_2O suggesting that $^4k_{nr}$ here is less sensitive to solvent isotope substitution than in $Cr(bpy)_3{}^{3+}$. If $(^4T_2)Cr(phen)_3{}^{3+}$ were somewhat less distorted than is $(^4T_2)Cr(bpy)_3{}^{3+}$ due to the greater rigidity of the phen ligands, the interligand solvent molecules would be less strongly bound. This would result in a lessened contribution of the entropic effect in the nonradiative mode.

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(19) If it is assumed that variations in the overall values of $^4k_{isc}$ and $^4k_{nr}$ reflect the composition of bulk solvent in a linear way ($^4k_{isc} = {}^4k_{isc}{}^H\chi_H + {}^4k_{isc}{}^D\chi_D$ and $^4k_{nr} = {}^4k_{nr}{}^H\chi_H + {}^4k_{nr}{}^D\chi_D$ where $\chi_H + \chi_D = 1$), the use of the values of $^4\eta_{isc}$ as a function of χ_D leads to the result that $^4k_{isc}{}^H/{}^4k_{isc}{}^D \sim 4$. The direction and magnitude of this solvent isotope effect suggests that intersystem crossing may involve vibronic coupling to the solvent.

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(21) An earlier study[18] determined $^4\eta_{isc}$ for $Cr(phen)_3{}^{3+}$ in 1:3 v/v H_2O:DMF to be ~ 0.2. A recent reexamination[22] of the system in the absence of organic solvents indicates that $^4\eta_{isc} \sim 1$.

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Reluctant Azoalkanes: Short-Wavelength (185 nm) Liquid-Phase Photolysis and High-Temperature (400–1000 °C) Gas-Phase Pyrolysis of 3,3,5,5-Tetramethylpyrazolin-4-one

Waldemar Adam,*† Andreas Fuss, Francois P. Mazenod, and Helmut Quast

Department of Chemistry, University of Puerto Rico
Rio Piedras, Puerto Rico 00931, and Institute of Organic
Chemistry, University of Würzburg, Am Hubland
D-8700 Würzburg (BRD), Germany

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Recently we demonstrated[1] that reluctant azoalkanes, such as those in which the azo linkage is contained in a six-membered ring,[2] undergo facile liquid-phase denitrogenation on irradiation with 185-nm light. It was, therefore, of interest to explore whether such short-wavelength photodenitrogenation was a general phenomenon of reluctant azoalkanes. For this purpose we chose 3,3,5,5-tetramethylpyrazolin-4-one (**1**), which represents one of the few reluctant substrates in which the azo linkage is contained in a five-membered ring. For example, this potential precursor to tetramethylcyclopropanone could not be efficiently denitrogenated ($\Phi = 0.012$ at 313 nm in benzene) via conventional photolysis, i.e., excitation of the n_-,π^* chromophore.[3] However, elevated temperatures or nonpolar solvents promoted nitrogen elimination under conventional photolysis conditions (≥300 nm).[4] We now report that short-wavelength (185 nm) radiation is effective in extruding nitrogen from pyrazolinone **1**. For comparison, we present also the results of its high-temperature (400–1000 °C) thermolysis.

Table I. Photolysis Conditions, Percent Consumption,[a] and Quantum Yields of the Photodenitrogenation of 3,3,5,5-Tetramethylpyrazolin-4-one (1)[b]

photolysis conditions		pyrazolinone	
λ, nm	$t \times 10^{-3}$, s	consumption, %	quantum yield, %[c]
185[d]	0.6	75	63 ± 5[h] (56 ± 5)[i]
254[e]	54.0	5	0.05 ± 0.01
300[f]	27.0	14	0.24 ± 0.05
350[g]	28.8	6	0.11 ± 0.01

[a] Disappearance of 1 was monitored by GC on a 5 ft × 1/8 in. stainless steel column, packed with 5% SE-30 on Chromosorb P, using column and injector temperatures of 105 and 130 °C, respectively, and a N_2 flow of 29 mL/min; $R_f = 173$ s for 1 under these conditions. [b] $\lambda_{max} = 356$ nm (ϵ 164) in hexane.[3d] [c] Quantum yields were determined by using 2,3-diazabicyclo[2.2.1]heptene (DBH) as actinometer, for which the photodenitrogenation efficiency is 100% at 185 and 350 nm.[1] [d] 50-W hot cathode mercury resonance lamp with relative intensities at 185 and 254 nm of 20% and 80%, respectively; solutions were not degassed. [e] RPR-2537-Å lamps with relative intensities at 185, 254, 313, and 365.4 nm of 0.8%, 100%, 1.9%, and 1.6%, respectively. [f] RPR-3000-Å lamps with relative intensities at 300 and 312 nm of 100% and 77%, respectively. [g] RPR-3500-Å lamps with relative intensities at 350–354 and 356 nm of 100% and 97%, respectively. [h] The cis–trans isomerization of cyclooctene was also used as actinometer, for which the isomerization efficiency is 35% at 185 nm.[6] [i] This quantum yield represents product formation (cf. Table II).

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*To whom correspondence may be addressed at the University of Würzburg.

† NIH Career Development Awardee (1975–80).

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Table II. Conditions, Percent Consumption, and Absolute Product Yields of the Denitrogenation of 3,3,5,5-Tetramethylpyrazolin-4-one (1)

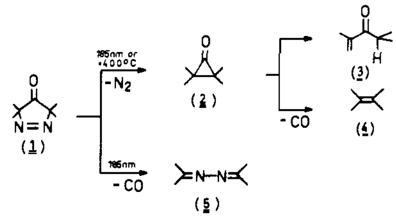
			relative product composition, %		
reaction conditions	pyrazolinone consumption, %	total products, %	α,β-enone (3)[a]	tetramethylethylene (4)[b]	ketazine (5)[c]
185 nm[d]	68	61[e]	48[f]	29	15[g]
400 °C[h]	100	90[i]	73[j]	27	
420 °C[k]	100		57	43	
1000 °C[l]	50–80		35 ± 10	65 ± 10	
250–400 nm[m]	52	100	traces	63	37

[a] α,β-Enone 3 was identical by IR and ^{1}H NMR with an authentic sample prepared according to: House, H.O.; Frank, G. A. *J. Org. Chem.* 1965, *30*, 2948; it was quantitated by GC using the same column conditions as specified in Table I. [b] Tetramethylethylene (4) was identified by comparison of its retention time (GC) and ^{1}H NMR spectrum with the authentic material; it was quantitated by GC using the same column as specified in Table I but operated at injector and column temperatures of 120 and 32 °C, respectively, and a nitrogen flow of 30 mL/min. [c] Ketazine 5 was identical by comparison of its retention time (GC) and ^{1}H NMR spectrum with the authentic material; it was quantitated by GC using a 10 ft × 8 in. stainless steel column packed with 10% Carbowax 20M on Chromosorb W and operated at injector and column temperatures of 120 and 90 °C, respectively, and a nitrogen flow of 31 mL/min. [d] Same conditions as specified in Table I (cf. footnote *d*). [e] Includes ca. 3% (relative) of tetramethylcyclopropanone (2), which was detected by IR (characteristic 1838- and 1820-cm^{-1} carbonyl bands) and characterized by comparison with an authentic sample prepared by photodecarbonylation of tetramethyl-1,3-cyclobutanedione (ref 8a); ca. 5% (relative) of acetone, which was identical by comparison of retention time (GC) and ^{1}H NMR with the authentic material and quantitated by GC under the same column conditions as specified in footnote *c*; ca. 4% unidentified low molecular weight products. [f] α,β-Enone 3 is photolabile, undergoing decarbonylation to the extent of ca. 20% on 185-nm irradiation for 600 s, but it is stable toward pyrolysis. [g] Stable toward 185-nm irradiation (only traces of acetonitrile were formed) and high-temperature pyrolysis. [h] Vacuum flash pyrolysis (VFP) by volatilizing the pyrazolinone 1 at 90 °C and 5.5 torr through a 40-cm quartz tube which was heated at 400 °C; the effluent was collected in a dry-ice trap. [i] Traces of dimethylketene were detected by IR (2257-cm^{-1} carbonyl band). [j] Stable toward thermolysis conditions. [k] Direct on-column injection of a decaline solution of 1 at GC injector temperature of 420 °C using column conditions as described in footnote *b* but column temperature of 140 °C. [l] Curie point pyrolysis (Bühler, Ch.; Simon, W. *J. Chromatogr. Sci.* 1970, *8*, 23) using a 10 ft × 1/8 in. stainless steel column packed with 10% SE-30 on Chromosorb AW-DMCS and operated at column and injector temperatures of 140 and 150 °C, respectively. [m] Irradiated in [D$_{12}$]-cyclohexane under rigorous exclusion of oxygen with the full arc (unfiltered) of a 1000-W Hg/Xe high-pressure lamp (Canrad Hanovia 977 B0010) in a quartz vessel at 10 °C (cf. ref 4).

The photolysis of 3,3,5,5-tetramethylpyrazolin-4-one (**1**) at 185 nm was conducted in Photrex (J. T. Baker) *n*-pentane (ca. 0.01 M, not degassed) by using a 50-W hot cathode mercury resonance lamp. The latter was accommodated in a Suprasil immersion well.[5] Since most (ca. 80%) of the 50-W hot cathode mercury resonance lamp light output is at 254 nm, it was necessary to check the efficiency of photodenitrogenation of **1** at this wavelength. Furthermore, since the 254-nm lamp also puts out appreciable amounts of 313- (ca. 1.9%) and 365.4-nm (ca. 1.6%) radiation, we also assessed the quantum yields of denitrogenation of the pyrazolinone at these wavelengths. The latter photolyses were performed in a Rayonet photoreactor under the same conditions as the 185-nm irradiation, except that at 254 nm a Vycor vessel was used and at 310 and 350 nm Pyrex vessels were used. The quantum yields at these various wavelengths are collected in Table I. Depending on the particular wavelength, the pyrazolinone **1** loses nitrogen by ca. 500- to 1000-fold[3d] more efficiently at 185 nm. This clearly establishes the utility of such high-energy radiation (185 nm or 155 kcal/mol) for the photodenitrogenation of reluctant azoalkanes.[1]

Since the pyrazolinone **1** represents a bichromophoric substrate,[7] i.e., it contains the azo and carbonyl groups for excitation, it was of interest to determine the product composition in the 185-nm photolysis. At 185 nm the azo chromophore should absorb preferentially, and we would expect that denitrogenation should outweigh decarbonylation. This expectation is indeed borne out by our product data (Table II), since the total yield of denitrogenated products (**2**–**4**) is ca. sixfold larger than that of the decarbonylated product (**5**). However, the conventional long-wavelength (≥300 nm) photolysis also led preferentially to denitrogenation, as determined recently.[4] In fact, the ratio of denitrogenated vs. decarbonylated products of the long-wavelength irradiation matches that of the 185-nm photolysis. While in the long-wavelength photolysis the denitrogenated product is tetramethylethylene (presumably formed via photodecarbonylation of tetramethylcyclopropanone[8a]), at 185 nm the major product is α,β-enone **3** (presumably formed via rearrangement of tetramethylcyclopropanone[8c]). Thus, both 185-nm and ≥300-nm wavelength denitrogenations presumably afford tetramethylcyclopropanone, but its subsequent fate differs considerably at these two wavelengths.

Scheme I



Since in the 185-nm photolysis vibrationally excited cyclopropanone might have formed on denitrogenation and this "hot" cyclopropanone rearranged into α,β-enone **3**, it was of mechanistic interest to examine the thermolysis of the pyrazolinone **1**. It was especially significant to determine the relative proportion of denitrogenation vs. decarbonylation products and compare these results with those of the photolysis. In view of the high thermal stability of **1**,[3d] it was necessary to apply drastic pyrolytic conditions (Table II). Vacuum flash pyrolysis (VFP) at 400 °C, direct column injection by VPC at 420 °C, or Curie point pyrolysis at 1000 °C gave α,β-enone **3** and alkene **4** as the major products. Denitrogenation was the exclusive course of decomposition under these pyrolytic conditions. Furthermore, α,β-enone **3** was the major product and presumably arose from rearrangement of the intermediary cyclopropanone.

The photolysis and pyrolysis results are rationalized in Scheme I in which we only attempt to account for the major products. The principal process of the 185-nm photolysis and the only reaction of the pyrolysis of pyrazolinone **1** are denitrogenation, affording tetramethylcyclopropanone (**2**). Subsequently, the cyclopropanone rearranges into α,β-enone **3** or decarbonylates into tetramethylethylene (**4**); both reactions have precedents in the literature[8] and have been confirmed by control experiments (Table II). In the case of the 185-nm photolysis, a small amount of the cyclopropanone survives, allowing infrared detection. It is im-

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portant to emphasize that decarbonylation of the pyrazolinone **1** takes place on 185-nm photolysis. Furthermore, conventional photolysis of **1**, i.e., long-wavelength ($\geq$300 nm) irradiation, also leads to these products and in similar amounts;[4] however, the efficiency of denitrogenation is significantly (ca. 500-fold) enhanced at 185 nm (Table I). Whether this enhanced photoreactivity of **1** derives from n_+,π^* or π,π^* excitation of the azo linkage is not known at this point because the necessary spectroscopic data is lacking. We conclude that 185-nm radiation is quite effective for the extrusion of nitrogen from reluctant azoalkanes. Work is in progress to explore the photolytic behavior of reluctant acyclic and aromatic azo compounds.

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Book Reviews

IUPAC Compendium of Analytical Nomenclature. Definitive Rules 1977. Edited by H. M. N. H. Irving, H. Freiser, and T. S. West. Pergamon Press, Oxford, England. 1978. viii + 222 pp. $19.50.

It has become a cliché to say of a book that it belongs on every reference shelf, but some books are so fundamental that it must be said. Many scientists tend to forget the importance of a standard terminology, but there is no doubt that early agreement upon a system of names, symbols, and rules for naming new compounds was an important factor in the rapid advance of chemistry as a science. Chemistry has thus been spared some of the arguments over terminology that have caused confusion in other disciplines. Those chemists who doubt the importance of standard nomenclature should recall their earliest lessons in chemistry and the emphasis that was placed on correct names of compounds.

The terminology of classical analytical chemistry is well defined, but during the last 3 decades or so, the analytical arts have advanced so rapidly that the language of analytical chemistry is somewhat in disarray. Each innovator has tended to make up his own terms as he went along, and so fast is our current system of information transfer that many imprecise and misleading terms have become entrenched.

Under these circumstances, it is gratifying that an organization of the prestige and influence of the International Union of Pure and Applied Chemistry has taken a hand in attempting to bring order out of the situation. As explained in the preamble to "Compendium of Analytical Nomenclature", various IUPAC commissions over 20 years have reported their recommendations on nomenclature in the various branches of analytical chemistry. These reports have been published in "Pure and Applied Chemistry" and have been brought together in the present volume. Because of the variety of subjects treated and changes in the composition of the commissions, it has been difficult to achieve a uniform treatment, although certain guiding principles, which are stated in the preamble, have been observed in all cases.

Obviously not every analytical chemist will find every chapter equally useful, and some readers will not agree with some of the recommendations. The first three chapters, on presentation of results, precision balances, and scale of work (sample size), and the appendix on usage of "equivalent" and "normal" comprise a useful review of basics, along with Chapter 8 on titrimetry. Other chapters vary in their appeal. Chapters have been presented in the order of original separate publication; this probably was the most convenient way for the editors to work, but the consequence is that subjects are not always grouped logically. For example, automatic analysis and mass spectrometry precede such fundamental considerations as pH and titrimetry; the chapter on gas chromatography precedes the discussion of chromatography in general. Fortunately, there is a substantial index to help the reader locate a particular subject, and most users will be looking for rules on a specific technique, rather than reading the book straight through.

Every reader will have a list of candidate subjects that should have been included. This type of work necessarily proceeds slowly and could only be current if analytical chemistry were to remain static for a time, a most undesirable alternative. However, IUPAC is to be commended for undertaking this important and useful work and for making its results available at a reasonable cost.

Helen L. Reynolds, *Food and Drug Administration*

Advances in Biochemical Engineering. Volume 14. Microbial Metabolism. Edited by A. Fiechter (Eidgenössische Technische Hochschule, Hönggerberg, Zürich). Springer-Verlag, Berlin-Heidelberg-New York. 1980. 162 pp. $41.20.

Although this series is directed toward biochemical engineers and biotechnologists, the chapter on ergot alkaloids and their biosynthesis will also be of interest to pharmaceutical chemists. The chapter on large scale cultivation of animal cells in monolayers should be of interest to a variety of investigators. Bioconversions of nitriles and their applications, induction of xenobiotic monooxygenases, and chemical and physical features of cellulosic materials as substrates for enzymatic hydrolysis complete this volume. References are included.

M. C. W. Smith, *Ann Arbor, Michigan*

Advances in Biochemical Engineering. Volume 15. New Technological Concepts. Edited by A. Fiechter (Eidgenössische Technische Hochschule, Hönggerberg, Zurich). Springer-Verlag, Berlin-Heidelberg-New York. 1980. 129 pp. $46.50.

Two chapters in this volume should be of interest to workers other than biochemical engineers. One describing the use of lactase in milk systems has application in the dairy industry, while analytical chemists will be interested in the chapter concerned with enzymes in analytical chemistry. The two remaining chapters are devoted to the loop reactor for cultivating yeast on *n*-paraffin substrate and the production of extracellular microbial polysaccharides. References are included.

M. C. W. Smith, *Ann Arbor, Michigan*

How to Obtain Abundant Clean Energy. By Linda Baine McGown (California State University, Long Beach) and John O'M. Bockris (Texas A&M University, College Station, Texas). Plenum Press, New York and London. 1980. xii + 262 pp. $14.95.

This book is an introduction to the subject of energy and will be of interest to the general reader. It includes discussions of energy, how it is used, where it comes from, etc. Atomic energy, solar energy, and energy from geothermal sources, tides, hydrogen, and wind are included. The final chapters are concerned with the politics of survival and answers to questions. A glossary and index are included.

M. C. W. Smith, *Ann Arbor, Michigan*

Food, Energy and Society. By David and Marcia Pimental (Cornell University). John Wiley and Sons, New York. 1979. viii + 165 pp. $10.95.

This book, for the general reader, presents extensive analyses of the interdependence of food, energy, and society. The first five chapters are devoted to the history of food production, energy use, and developing societies. The next six chapters present data on the use of energy for food production, processing, packing, and transport. For example, it is estimated that 87 kcal of fossil energy are spent to transport 1 kcal of strawberries from California to New York. The final chapter discusses future food needs, strategies for meeting food needs, energy needs in food production, land constraints, water constraints, climate, environmental pollution, and the future with a question mark. Numerous tables, graphs, three appendices, references, and an index are included.

M. C. W. Smith, *Ann Arbor, Michigan*