A Specialist Periodical Report

Photochemistry

Volume 1

A Review of the Literature Published
between July 1968 and June 1969

Senior Reporter

D. Bryce-Smith,

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Introduction

This volume inaugurates what is hoped will be an annual series of comprehensive reports on the progress of photochemistry. In compiling the work, the authors have adopted as a guiding principle the need to present as unified a treatment as possible; and although various Chapters stress, say, the more 'physical' or 'organic' aspects of the subject, in reflection of the emphasis in the original papers, the aim has been to produce a work which as a whole will be of value to both physical and organic chemists, and even, with the inclusion of a section on Inorganic Photochemistry, to inorganic and organometallic chemists. Only the more biochemical aspects have been omitted, though with reluctance, partly because of the need to keep the work to a manageable length; but consideration will be given to the inclusion of a section covering the more chemical aspects of photobiology in future reports. It is hoped that the section on photopolymerisation will be of special use to the growing number of chemists interested in the industrial applications of photochemistry, although the authors are aware that many interesting developments lie hidden in the growing patent literature on the subject, which they have not found it feasible to cover. References to work in borderline areas will sometimes be found in more than one Chapter.

This report is concerned with the twelve months July 1968 to June 1969, inclusive. The authors have attempted to achieve both a report and a critical commentary, as far as possible; but the aim to have an edited manuscript at the printers only two and a half months after the end of the period under review has inevitably restricted the desirable element of commentary. The name of the author principally responsible for compilation is given for each Chapter, although the authors accept Cabinet responsibility for the work as a whole.

Until about ten years ago, photochemistry was largely a branch of physical chemistry, and organic chemists relied almost wholly on the Bunsen burner or its equivalent, and/or catalysts, for making and breaking bonds. The advent of gas-chromatography and the development of new spectroscopic tools made it feasible for organic chemists to study the often small quantities of compounds which are formed by chemical transformations of electronically excited molecules, and it soon came to be realised that a whole new branch of chemistry was easily accessible, having its own rules, and often providing structurally interesting molecules. The good news has also reached the inorganic and organometallic chemists, but is only just beginning to make an impact. There are numerous signs of industrial interest in photochemistry, most evidently in Japan, the United States, and Germany.

The year under review has seen solid progress in many areas, and a lot of enthusiastic 'wildcatting' (to borrow a term from the petroleum industry) in others. Quite often one finds that apparently arbitrarily chosen complicated molecules are studied long before their structural parents have been looked at. Such approaches are probably relatively inefficient in promoting a greater understanding of the subject as a whole.

A greater awareness of the importance of chemical quenching processes for luminescence phenomena appears to be growing, and it is no longer expected that the quantum yields for photophysical processes such as fluorescence and phosphorescence should total unity. Kasha, Jortner, and Ting have independently developed some stimulating new ideas relevant to 'radiationless' transitions between electronically excited states. The whole basis of earlier theories has been questioned. It had previously been thought that such transitions are time-dependent and require some perturbation influence possibly connected with failure of the Born–Oppenheimer approximation. Kasha in particular has now argued that the transitions may be time-independent, and that intermolecular perturbation may occur prior to interaction with the external field. This approach would require replacement of the hallowed term 'intersystem crossing' with a new term 'intersystem mixing'. Future work may clarify the degree of difference implied by this distinction. The related new ideas developed by Jortner are specially relevant to the mechanism of bond-breaking processes which occur consequent upon initial electronic excitation.

Until recently, upper excited singlet and triplet states were thought to cascade down so rapidly to the lowest excited states (S_1 and T_1) that, except in the special case of azulene, all luminescence emission phenomena, energy transfer processes, and chemical transformations must proceed from these lowest excited states. This viewpoint is being modified in the light of clear evidence for energy transfer from T_2 anthracene, and indications that some photoadditions of olefins to cyclopentenone proceed *via* the T_2 state of the enone. It seems possible that some phenomena now attributed to 'non-vertical' energy transfer may actually involve higher excited states. Cases of $T \rightarrow T$ fluorescence in arylcarbenes have been claimed, and a valuable new technique involving simultaneous monitoring of an e.s.r. signal has made it possible to use $T \rightarrow T$ absorption spectra to provide a quantitative measure of triplet state populations.

Contrary to previous belief, the position of λ_{max} for fluorescence is not necessarily independent of the wavelength of the exciting radiation, for example in the case of quinine which is often used as a reference standard.

Energy-transfer phenomena in photochemistry have previously been thought largely to involve triplet species, but cases of singlet energy transfer are now being reported, sometimes for systems hitherto supposed to involve energy transfer between triplets. The formation of singlet and triplet excimers and exciplexes in fluid solutions continues to attract interest, although the precise nature of the bonding and geometry in such complexes is still less clear than one would like.

The fallacious assumption is still occasionally being made that the ability of triplet sensitisers such as benzophenone to sensitise a process implies that the unsensitised process proceeds *via* triplet intermediates. It is still much easier to establish a triplet pathway for a reaction than to exclude the possibility of a contribution from excited singlet intermediates. Triplet processes are often thought to be non-stereospecific, but one or two substantially stereospecific examples are beginning to appear. More reliable and unambiguous diagnostic tests for triplet intermediates have long been needed: there is some hope that lanthanide ions may provide sensitive probes for such intermediates, particularly those of $\pi\pi^*$ type.

Studies of photochemical transformations are generally most conveniently carried out in fluid media; but more cases are becoming known where photoreactions follow substantially different courses in the gas and liquid phases. Several workers have pointed out the importance of temperature effects in photoreactions, but temperatures are still frequently unspecified in publications. There is increasing interest in the effects of acid media, since it is now recognised that molecules have acid–base characteristics in excited states different from those in the electronic ground state, and that proton-transfer can be rapid in comparison with excited state lifetimes. Protonation of excited states can produce carbonium ions and lead thereby to isomerisation and other reactions typical of these species. Solvent effects are often loosely attributed to the solvent 'polarity', but it is rarely made clear whether the property referred to is related to the dielectric constant or to the solvating (*e.g.* hydrogen bonding) character.

Previous discrepancies concerning the quantum yield for photodecomposition of water at 1849 Å have been largely resolved, and evidence has been obtained for the formation of solvated electrons at shorter wavelengths. Studies of inorganic phosphors have been stimulated by the ability of such systems to show laser action, but much remains to be done here, and also on the use of inorganic oxide-type semiconductors as photosensitisers. There is growing interest in the photochemistry of co-ordination compounds in solution.

Many may feel that work published during the year in the more organic regions of photochemistry has been impressive for its quantity rather than its overall quality; but significant advances have certainly been made in several areas. The Woodward–Hoffmann orbital symmetry rules continue to be valuable in rationalising many varieties of photochemical behaviour, but have occasionally been applied without analysis to systems, *e.g.* anthracene, where their relevance is uncertain.

Among numerous new photoisomerisations, Zimmermann's transformation of cyclo-octatetraene into semibullvalene is specially interesting, as is Anet's report that bond-inversion in cyclo-octatetraenes is promoted by light. White and his co-workers have described an extraordinary mono-*trans* isomer said to be formed from the irradiation of 1,2,4,7-tetraphenylcyclo-octatetraene.

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Interest in enone and dienone photochemistry continues at a high level: this rich seam shows few signs of being exhausted. The mechanism of the preparatively-useful isomerisation of $\alpha\beta$-enones to the $\beta\gamma$-isomers has been considerably clarified.

Mechanistic studies on the photochemical *cis–trans* isomerisation of ethylenes have suggested that both singlet and triplet processes can occur. The direct isomerisation of stilbenes increasingly appears to be a singlet process, although doubts persist in the cases of certain substituted stilbenes. There may even be some small contribution from singlet intermediates in the benzene-sensitised isomerisation of but-2-ene, especially at high olefin concentrations, although the process is often used to measure the triplet yields of aromatic hydrocarbons.

Work continues on the photochemistry of benzene and its derivatives. The photoisomerisation of *o*- to *m*-xylene has been shown to be a singlet process, in accordance with previous predictions based on orbital and state symmetry considerations. It has been suggested that the unusually short lifetimes of aromatic triplet species may be due in part to the formation of triplet excimers. Past discrepancies concerning the solid-state photodimerisation of anthracene have been resolved, and attributed to a hitherto-unrecognised wavelength dependence.

The photo-Fries reaction has received continued attention. Previous studies had identified free-radical intermediates, but it now seems possible that the *ortho*-Fries rearrangement in *para*-blocked phenyl esters can be viewed as a concerted process. Some interesting effects of solvent viscosity and 'polarity' have been observed for this reaction.

Applications of photochemical procedures continue to be made in the synthesis of natural products, *e.g.* thyroxine, and interesting reports have appeared on the photochemistry of purines and pyrimidines, a topic of importance in connection with the effects of u.v. radiation on nucleic acids.

D. B-S.

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1

Spectroscopic and Theoretical Aspects

This chapter is devoted to those spectroscopic and theoretical aspects of the subject which will be of immediate interest to photochemists. Since absorption of light by a molecule necessarily precedes the photochemical and photophysical processes of interest, mention will be made of the absorptive act where this provides useful information. Theoretical treatments of the factors influencing the photophysical properties of excited states, including luminescence and radiationless transitions, and attempts to account for observed behaviour of excited molecules are considered here in detail. The rationalisation of the mechanisms of both photochemical and thermal reactions by orbital symmetry considerations will be considered only briefly in this chapter. Discussion of these rules with reference to particular organic molecules is to be found in those chapters dealing with the photochemistry of the relevant compounds.

1 Absorption and Energy Level Calculations

Absorption spectroscopy can provide much useful information concerning the nature of the excited states studied. In general, molecular orbital calculations have not been precise enough to predict exact energies of excited electronic states of most complex molecules, but the results are often useful in the characterisation of the states. All mono-olefins exhibit a single strong optical transition whose absorption maximum falls between 7·6 and 6·2 ev, with ε_{max} of *ca.* 10^4. Many substituted olefins show a lower-energy transition with ε_{max} of *ca.* 1000, which is incompletely characterised. S.C.F. calculations have been performed on ethylene and *trans*-cyclo-octene in an attempt to determine which orbitals are involved in these transitions.[1] The conclusions reached are that the strong absorption observed is due to the $\pi_z \rightarrow \pi_z^*$ transition, as was known before, but in the case of *trans*-cyclo-octene, another transition, the $\pi_z \rightarrow \pi_y^*$ must also be available in the 5—7 ev region to account for the observed rotational strength of the transition. The transition with $\varepsilon_{max} \sim 1000$ can only be assigned to the $\pi_z \rightarrow 4a_g$ $(3s\sigma^*)$ transition. Thus at least three excited singlet states of simple olefins lie in the 5—7 ev region. Data on the electronic spectra of a large number of conjugated compounds in the u.v. and

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[1] M. Yaris, A. Moscowitz, and R. S. Berry, *J. Chem. Phys.*, 1968, **49**, 3150.

visible region of the spectrum have been compared with calculations of energy levels by use of Hückel molecular orbitals in a recent review.[2] Pariser–Parr–Pople calculations on the excited states of some aromatic molecules yield results for the energies of singlet states in good agreement with experiment.[3, 4] An interesting fact arising from the calculations is that the non-bonding electrons on the fluorine atom in fluorobenzene have no effect on the ring in the lower energy transitions. Appreciable effect would only be noticed on the E_{2g} state of benzene, transformation to which is in an inaccessible part of the spectrum and has not been observed experimentally.

The electronic absorption spectra of carbonyl compounds have been the subject of several studies.[5–7] While there is no doubt that the lowest lying excited singlet state of carbonyl compounds is the $n\pi^*$ state, controversy exists over the assignation of the second singlet. In aromatic ketones, there seems little doubt that this arises from a $\pi \rightarrow \pi^*$ transition, and a similar transition has also been assumed in the case of aliphatic ketones. However, a polarisation study of crystalline heptadecan-9-one and the changes observed upon melting of the sample seem to indicate unequivocally that the second singlet in this compound arises from the $n \rightarrow \sigma^*$ transition.[5] Since the second singlet in acetone has been assigned to this transition by some authors, the implication is that in aliphatic ketones the second excited singlet state is of a nature different from that in aromatic compounds. Though the second singlet is considered to be of the $\pi\pi^*$ configuration, extended Hückel molecular orbital calculations were performed on a variety of substituted alkyl and aryl carbonyl compounds, and a rationale obtained for the observed red and blue shifts of transitions to both first and second excited states.[6] Substitution clearly affects the $n \rightarrow \pi^*$ transition more than the transition to the second excited state.

The forbidden $S_0 \rightarrow T_1$ transition in benzophenone can be seen in absorption in the crystalline state. A polarised high-field Zeeman study reveals that the triplet state reached by such absorption is the symmetry analogue of the $^3A_2(C_{2v})$ state of formaldehyde.[7] The transition gains its intensity *via* spin–orbit coupling with the $^1A_1$ and $^1B_2$ states. Since the $\rangle C{=}O$ and aryl moieties are non-planar, there is the possibility of strong vibronic coupling between the $^1\pi\pi^*$ states and carbonyl $^1\sigma \rightarrow \pi^*$ transitions. Since the $^1\sigma\pi^*$ states are already strongly spin–orbit coupled to the $n\pi^*$ states, the net effect of twisting is to intensify the $S \rightarrow T$ transition. The second triplet state of benzophenone[8] has never been located experimentally, although it has been convenient to assume that it lies in energy between the lowest singlet $^1n\pi^*$ state and the lowest triplet $^3n\pi^*$. In this position the $\pi\pi^*$ triplet can

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[2] R. Zahradnik, *Fortschr. Chem. Forsch.*, 1968, **10**, 1.
[3] P. Yvan and O. Chalvert, *Compt. rend.*, 1968, **266**, *C*, 1351.
[4] C. Leibovici, *Compt. rend.*, 1968, **267**, *C*, 1121.
[5] W. C. Johnson jun., and W. T. Simpson, *J. Chem. Phys.*, 1968, **48**, 2168.
[6] K. Yates, S. L. Klemenko, and I. C. Csizmadia, *Spectrochim. Acta*, 1969, **25**, *A*, 765.
[7] S. Dym, R. M. Hochstrasser, and M. Schafer, *J. Chem. Phys.*, 1968, **48**, 646.
[8] M. Batley and D. R. Kearns, *Chem. Phys. Letters*, 1968, **2**, 423.

be used to account for the observed high rates of intersystem crossing from the $^1n\pi^*$ to $^3n\pi$ states. Theory shows that transitions between these states should be of much lower probability than transitions between states of different configurations. However, if it is assumed that the $^3\pi\pi^*$ state lies just lower in energy than the $^1n\pi^*$, ready intersystem crossing between these levels could occur, followed by internal conversion to the $^3n\pi^*$ state.

There is evidence now, however, that this explanation is unsatisfactory, since a search for singlet–second triplet absorption in crystals of benzophenone and 4,4′-di-iodobenzophenone has failed to show the existence of this state in the expected energy region. Experimentally, the technique of phosphorescence excitation[9, 10] was used to overcome the difficulties of detecting the very weak absorption. Both compounds exhibited singlet–triplet absorption attributable to the $S \rightarrow T_{n\pi^*}$ transition, but neither showed any absorption between this wavelength region and the onset of the strong singlet–singlet absorption. The conclusions to be drawn from this study are that the $^3\pi\pi^*$ state in these compounds lies higher in energy than the $^1n\pi^*$ singlet, or at least is nearly degenerate with it. This inference, if correct, has the important implication that the rapid intersystem crossing observed in benzophenone between the $^1n\pi^*$ and $^3n\pi^*$ states occurs without the intermediacy of the $^3\pi\pi^*$ state, despite theoretical predictions to the contrary.[11] However, an indirect involvement of this state *via* second-order vibronic mixing cannot be excluded.

Calculations using a modified version of the Pariser–Parr–Pople method on the electronic spectra of unsaturated ketones yield results in good agreement with experiment for the π–π^* transition, but results of varying success when the method is applied to n–π^* transitions.[12]

Detailed calculations of Franck–Condon factors and their effect upon the rates of radiationless processes in molecules are considered in the next section. Brief mention will be made here only of calculations pertaining to small molecules. Calculations on Lyman bands of hydrogen have shown that hitherto neglected vibration–rotation interaction effects are appreciably large for a number of the bands.[13] For the Schumann–Runge bands in the $^{16}O_2$ and $^{18}O_2$ isotopes, and the Lyman H_2 bands, Franck–Condon factors were also found to vary considerably with rotation quantum number.[14] A simple method of obtaining structural data from vibrational intensities has been applied to molecules in which there is a large change in shape in going from the ground state to the excited state.[15] The transitions considered are the 1500 Å system of ammonia, the 1600 Å system of PD_3, the α-X system of HCN, the 2400 Å system of acetylene, and the 3500 Å

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[9] D. R. Kearns and W. A. Case, *J. Amer. Chem. Soc.*, 1966, **88**, 5087.

[10] A. Marchetti and D. R. Kearns, *J. Amer. Chem. Soc.*, 1967, **89**, 768.

[11] M. A. El-Sayed, *J. Chem. Phys.*, 1963, **38**, 2834.

[12] N. L. Allinger, T. W. Stuart, and J. C. Tai, *J. Amer. Chem. Soc.*, 1968, **90**, 2809.

[13] M. Halmann and I. Laulicht, *J. Quant. Spectroscopy Radiative Transfer*, 1968, **8**, 935.

[14] D. Villarejo, R. Stockbauer, and M. Inghram, *Chem. Phys. Letters*, 1968, **2**, 11.

[15] W. L. Smith and P. A. Warsop, *Trans. Faraday Soc.*, 1968, **64**, 1165.

system of formaldehyde. For a transition from a linear ground state to a bent excited state it is possible to decide whether the transition correlates with an allowed or forbidden transition in the linear–linear configuration.

Absorption spectra can provide data on the physical properties of excited states also. Thus a special technique has been developed to measure the change in dipole moment of molecules on going from the ground to excited states, using a strong electric field.[16] For a mixed crystal of azulene in naphthalene, the dipole moments of azulene in its first two excited states have been measured. The direction of the dipole changes in both states from that in the ground state. The changes in dipole moment of the singlet state of naphthalene and substituted naphthalenes in different solvents have been measured.[17] From an extension of the theory of solvent shifts based on the Onsager model of dielectrics, it is possible to derive equations which include the change of polarisability, $\Delta\alpha$, from the ground state to an excited state.[18] For some aromatic molecules $\Delta\alpha$ in the excited state is calculated to be appreciably smaller than in the ground state. However, solvent shift methods have failed to detect any measurable polarisability change. The reason for this failure may lie in the fact that in the presence of the very large field due to the solvent shell, the assumption that the magnitude of the induced dipole moment is still proportional to the field may not be valid, owing to a saturation effect.

2 Radiationless Transitions

The term radiationless transition refers to processes in which molecules change from one electronic state to another without emission of radiation. If no change in multiplicity is involved, the process is termed an internal conversion, whereas transitions between states of different multiplicity are referred to as intersystem crossings. If the final state is dissociative, the transition is deemed predissociation. Any successful theoretical treatment of radiationless processes must be able to explain the vast amount of experimental data which has been accumulated in the literature and which may be summarised into the following general observations.

(*a*) With the exception of azulene and its derivatives, only the lowest electronically excited levels of polyatomic organic molecules emit radiation, implying rapid radiationless transitions between higher excited states.

(*b*) Radiationless transitions are first-order processes, as far as can be determined experimentally.

(*c*) As the energy gap between the electronic terms of the initial and final states increases, the rates of radiationless processes decrease.

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[16] R. M. Hochstrasser and L. J. Noe, *J. Chem. Phys.*, 1969, **50**, 1684.

[17] B. M. Uzhinov, A. I. Kozachenko, and M. G. Kuzmin, *Zhur. priklad Spektroscopii*, 1968, **9**, 1041.

[18] P. Suppan, *Spectrochim. Acta*, 1968, **24**, *A*, 1161.

(*d*) Deuteriation of molecules containing C—H bonds greatly reduces the rates of radiationless transitions, leading to an increase in observed radiative lifetimes.

(*e*) Generally, as the temperature of a system is raised, radiationless transitions become more important, or at least, radiative processes are quenched.

(*f*) Molecules of high enough complexity (see Chapters 2 and 3) appear to undergo radiationless transitions in the absence of perturbing collisions, *i.e.* the phenomenon is a strictly intramolecular one. For small molecules this is not the case.

A number of theoretical approaches,[19–39] have appeared in the literature attempting to explain the above experimental observations, and some of these have been briefly reviewed in an article purporting to show cases of transitions which do not fall into the categories above.[40] More recently, there has appeared an excellent and critical evaluation of the main theories to date,[33] and it is worthwhile summarising the salient features of the most important here.

Gouterman.[19, 20]—This treatment will be considered first, since it differs from all subsequent theories in that a perturbation external to the molecule undergoing the radiationless transition is essential to the mechanism. The case of a solute molecule embedded in a crystalline host is treated by postulating interactions of the molecule with the crystal phonon field. By analogy with photon-induced radiative transitions, an expression is obtained for the spontaneous radiationless transition rate $A^s_{i\rightarrow f}$

$$A^s_{i\rightarrow f} = \frac{4\omega^3}{3\hbar^2 c_s{}^2} | M^s_{if} |^2 \tag{1}$$

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[19] M. Gouterman, *J. Chem. Phys.*, 1962, **36**, 2846.

[20] P. Seybould and M. Gouterman, *Chem. Rev.*, 1965, **65**, 413.

[21] H. Sponer, *Radiation Res.*, 1959, **1**, 658.

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[24] J. P. Byrne, E. F. McCoy, and I. G. Ross, *Austral. J. Chem.*, 1965, **18**, 1589.

[25] G. W. Robinson and R. P. Frosch, *J. Chem. Phys.*, 1962, **37**, 1962; G. W. Robinson and R. P. Frosch, *ibid.*, 1963, **38**, 1187.

[26] G. W. Robinson, *J. Chem. Phys.*, 1967, **47**, 1967.

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[28] W. Siebrand and D. F. Williams, *J. Chem. Phys.*, 1968, **49**, 1860; B. R. Henry and W. Siebrand, *ibid.*, 1968, **49**, 5369; W. Siebrand, *Chem. Phys. Letters*, 1968, **2**, 94.

[29] M. Y. Ovchinnikova, *Teor. i Eksp. Khim.*, 1968, **4**, 575.

[30] T. E. Martin and A. H. Kalantar, *J. Chem. Phys.*, 1968, **49**, 235.

[31] S. H. Lin, *J. Chem. Phys.*, 1966, **44**, 3759.

[32] S. H. Lin, *Theor. Chim. Acta.*, 1968, **10**, 301.

[33] B. R. Henry and M. Kasha, *Ann. Rev. Phys. Chem.*, 1968, **19**, 161.

[34] M. Bixon and J. Jortner, *J. Chem. Phys.*, 1968, **48**, 715.

[35] J. Jortner and R. S. Berry, *J. Chem. Phys.*, 1968, **48**, 2757.

[36] D. P. Chock, J. Jortner, and S. A. Rice, *J. Chem. Phys.*, 1968, **49**, 610.

[37] S. A. Rice, I. McLaughlin, and J. Jortner, *J. Chem. Phys.*, 1968, **49**, 2756.

[38] W. Rhodes, *J. Chem. Phys.*, 1969, **50**, 2885.

[39] C. H. Ting, *Photochem. and Photobiol.*, 1969, **9**, 17.

[40] D. Phillips, J. Lemaire, C. S. Burton, and W. A. Noyes jun., *Adv. Photochem.*, 1968, **5**, 329.

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