Ramamurthy thanks J. Saltiel for a copy of the article that appeared in IAPS Newsletter

PHOTOCHEMISTRY BECOMES MORE COMPLEX: A SYMPOSIUM HONORING GEORGE S. HAMMOND ON HIS 80TH BIRTHDAY

Organized by **Carl C. Wamser** (Portland State University) and sponsored by the organic and physical chemistry divisions, this symposium was held on April 2 and 3 in San Diego, as part of the 221st National Meeting of the American Chemical Society. It was a personally and scientifically rewarding, heart-warming affair that brought to this attendee particularly strong nostalgia for the Caltech days in George Hammond's laboratory. A large appreciative audience included a fine representation of those of us who had the good fortune to be touched by George as he blazed through the different stages of his career. The Iowa State years, the Caltech years, the UC, Santa Cruz years, the Allied Chemical years, and, after his retirement, the Bowling Green, Georgetown University and now Portland State University years were all represented.

Carl Wamser (Ph. D., Caltech) opened the first afternoon session of the symposium with biographical comments about George's early years, running the family dairy farm in Auburn, Maine at 13 after his father's death and then attending nearby Bates College. The first speaker, **Jay Kochi** (University of Houston, Ph. D., Iowa State), prefaced his scientific lecture with a tribute to George, and his seminal contributions to physical organic chemistry and photochemistry in particular, and his ability to influence his colleagues and students at all levels. Jay spoke of George's gift of imagination, his generous intellect and his superb ability for zeroing in on the essential. A quote from **Orville Chapman** "*George never met an idea he did not like*" struck a cord with all of us who knew him. Jay knew George before his photochemistry years. He met him first as a UCLA undergraduate some 53 years ago, when George gave a substitute lecture for **Saul Winstein**, with whom he was a postdoctoral fellow after earning his Ph. D. with **Paul Bartlett** at Harvard. He was so impressed that he followed George to Iowa State, where George had a profound effect in helping assemble a dynamic group of faculty that included **Ernie Wenkert**, **Chuck Depuy** and **Orville Chapman**. Jay spoke of the Chinese tradition of revering, honoring, and respecting longevity, and presented George with a classical Chinese poem, that George is to have translated in time for his 100th birthday celebration. Jay then announced the permanent establishment of the *George S. Hammond Lectureship at Bates College*. Former Iowa State colleagues accomplished this through contributions to George's alma mater, a wonderful gesture that touched George deeply.

The scientific part of Jay's lecture (*Charge transfer photochemistry via electron donor/acceptor organizations*) began with examples illustrating the ubiquitous, alas unheralded, involvement of colored charge transfer complexes in "garden variety" organic reactions, such as bromine addition to alkenes, electrophilic aromatic substitution and the Diels-Alder reaction. Contact ion radical pair (CIRP) intermediates, although very common, are not mentioned in Organic Chemistry textbooks. Jay reviewed incisive contributions from his own research, concentrating on those for which CIRP formation is induced by light absorption into the CT band. He emphasized the importance of including solvation energies in correlations of reaction rate constants with the electronic transition energy ($h\nu_{CT}$) of the CT band. For instance, rate constants for oxymercuration of aromatic hydrocarbons show random correlation with corresponding $h\nu_{CT}$s but yield to a dramatically linear correlation when solvation energies are included.

Nick Turro (Columbia University, Ph. D. Caltech) was the second speaker. Nick was my classmate at Caltech having arrived there in the summer of 1960. He spoke of the decisively shaping influence of the late **Peter Leermakers** (Ph. D. Caltech) in bringing him to George and to Caltech, and how, on being labeled by George an "anti-photochemist" when, in a public lecture, he demonstrated for him the luminol chemiluminescence reaction, he rose to the challenge by changing research directions. Nick turned from seeking entry into triplet states thermally via transition metal ion catalysis to photochemistry and triplet photosensitization. With Peter's participation this transition immediately bore fruit in the benzophenone-photosensitized photolysis of ethyl pyruvate and launched Nick's remarkable career in photochemistry. Nick credits George for having given us the tools to unravel photochemical mechanisms by showing how triplet sensitization could be applied generally in the unambiguous study of triplet state reactivity. Then, in the early 60s, at the dawn of modern molecular photochemistry, when each breakthrough came close on the heels of another, it was a heady atmosphere in the Hammond research group and with George's encouragement his co-workers would long maintain "photochemical highs".

Apparently, not entirely satisfied with the title of his lecture "*Supramolecular and magnetic control effects in organic photochemistry*", Nick tried "Super duper molecular photochemistry" and "Better things and better living through complexity". Supramolecular photochemistry, the study of photochemical reactions in the confining media of zeolites, cyclodextrins, micelles, and complex fluids, brings into play noncovalent interactions between substrate and environment that have a controlling influence on the outcome of photochemical reactions. Nick described the photolysis of symmetrical (ACOA) and unsymmetrical (ACOB) dibenzyl ketones in the super cage environments of zeolites. Because of different size restrictions, substrates in different locations in the zeolite (framework, holes, internal pores) behave as isomers with different relative mobility, as revealed by ^{2}H NMR measurements on strategically deuterated substrates. Conditions were described that allow control of cage products on photolysis of ACOB from 100% cage effect, CE, (AB product only) to 0% CE (statistical AA/AB/BB ratio) and to –100% CE (AA and BB products only). Enormous effects on radical lifetimes were established by transient EPR measurements (compare microseconds outside with seconds and hours within supracages). Depending on radical size, supracage size restrictions dramatically inhibit radical dimerization and favor disproportionation. In zeolites providing larger cages, cage reactions of the primary radical pairs (e.g., A/COB) could be studied and the effect of ^{13}C in the carbonyl carbon on triplet → singlet radical pair conversion and of external magnetic fields were described. At the end of this spectacular lecture Nick expressed our gratitude to George: "George we all thank you and love you for your mentoring insights, dedication, and your ability to imprint your scientific attributes on all your students and collaborators".

After a break and a chance to greet old friends, it was **Nate S. Lewis**' turn to speak. Perhaps Nate illustrates best the intricate scientific ties between many of the Symposium participants. Now a professor at Caltech, he had started out as an undergraduate there. Encouraged in 1976 by **Kent Mann**, a graduate student, he was "sucked into" the research collaboration between the **Harry Gray** and **George Hammond** groups, without knowing at the time that that decision would have a profound effect on his future. It led him to MIT for his Ph. D. studies with **Mark Wrighton** (Florida State B. S., Caltech Ph. D.), who was the first to set the Hammond/Gray collaboration on fire, and then back to Caltech.

The title of Nate's lecture was "*Nanocrystalline titanium dioxide solar cells sensitized with ruthenium or osmium polypyridyl complexes: Photoelectrochemical studies and electron transfer dynamics*". He described the use of osmium polypyridyl complexes as sensitizers of nanoporous TiO_2 electrodes to solar illumination. Spectral responses of TiO_2 solar cells are extended to longer wavelengths with the use of $Os^{II}(H_2L)_2(CN)_2$, (where L is 4,4'-dicarboxylato-2,2'-bipyridine) or $Os^{II}(H_2L)_3^{2+}$ than with the use of $Ru^{II}(H_2L)_2(NCS)_2$ and all produce very high external quantum yields for photocurrent flow. Singlet and triplet MLCT states of the ruthenium and osmium bipyridyl complexes $Ru^{II}(H_2L)_2(CN)_2$, $Os^{II}(H_2L)_2(CN)_2$, $Ru^{II}(H_2L)_2(NCS)_2$ and $Os^{II}(H_2L)_2(NCS)_2$ inject electrons into the semiconductor with rate constants in the 10^{10} - 10^{13} s^{-1} range. The much slower rates of charge recombination decrease with increasing driving force, indicating that they fall in the Marcus inverted region. Solar cell efficiencies over 5% were achieved. Higher efficiencies may result by designing sensitizers in nanocrystalline solar cells that take advantage of the Marcus inverted region behavior of charge recombination. This augurs well, because an efficiency increase to 15% is likely to lead to commercially feasible applications.

Tom Penner (Caltech Ph. D.) was the first industrial speaker of the Symposium (Imaging Materials and Media, Eastman Kodak Company). In his Ph. D. studies during the late 1960s, Tom had inherited **David Whitten**'s (Caltech postdoctoral) research project concerning relationships between photochemistry and radiation chemistry. The fact that Tom's presentation was co-authored by Dave Whitten is testimony to the lasting nature of research collaborations between George's co-workers. Tom spoke of George's view that the teaching of chemistry in terms of the traditional divisions presents an impediment to learning the subject. In his talk, Jay Kochi had also mentioned the Hammond/Gray paradigm of organizing the teaching of chemistry according to function: Synthesis, analysis, structure, and “the heart of chemistry” dynamics.

Tom’s topic, “*Photoprocesses in ordered thin films*”, concerned the design of materials for optical and photonic applications. Achieving desired performance depends, in part, on including functionality to control intermolecular interactions for large-scale organization. Chromophores that interact with light in a specified way are essential components. Systems consisting of deliberately interactive multiple components offer opportunities and challenges for optimizing conditions to maximize performance. Energy migration in dye-polymer/clay films was described. Formation of *J*-aggregates by cyanines and other dyes incorporated in polymers allows layer-by-layer fabrication of films on anionic clays. Efficient energy transfer from dye to dye and from layer to layer produces an antenna function for harvesting light energy. The effect of increasing the number of layers is not completely understood. For instance, a case was described for which energy migration is very efficient for the first six dye layers, but further layers (up to ten total) are non-contributing. This antenna effect has practical applications in silver halide imaging. An effective absorbance of 0.15 for a monolayer of dye coating on a silver halide crystal leads to a large fraction of wasted incident light. The antenna effect of multilayer dye assemblies markedly increases light absorption efficiency in photographic films. The ordered antenna dye on the silver halide grain acts as a template for the formation of *J*-aggregates with their characteristic large absorbances.

Dick Weiss (Caltech postdoctoral) presented the last talk of the first session of the symposium. As a graduate student with Eugene Snyder at the University of Connecticut in the late sixties, the Woodward and Hoffmann concept of orbital symmetry conservation in electrocyclic reactions had intrigued Dick, who knew that its application to photochemical reactions was problematic. The draw of California and photochemistry, in that order, brought Dick to Caltech, armed with Eugene’s advice to bet George that he could not stop smoking. Dick has enjoyed a close association with George that extended well beyond the initial two postdoctoral years. It continued through Dick’s three-year appointment as an NAS Overseas Fellow at the Universidade de Sao Paulo in Brazil, a program in which George was intimately involved, and more recently, through George’s highly positive association with Dick’s research group at Georgetown University. He finished the introduction to his talk with two GSH quotations. The first was George’s enthusiastic “Gee that’s great” reaction to new results which had an exhilarating effect on students, and the second was from George’s reflective side “Any scientific (or other) discipline when put into a small box will surely shrivel and die”. Dick has been studying the control of photochemical reactions by polymer media. He took comfort in the fact that although the photochemistry was confined to small boxes (cages), they have flexible walls.

The title of Dick's contribution was *"Extracting fundamental (simple) photochemical and photophysical information from reactions of guest molecules in complex polymeric media"*. Results were presented for the photo-Fries reactions of 1-naphthyl esters in unstretched and stretched polyethylene. Stretching the polymer leads to a transition in structure from spheralytic organization of the crystalline portion to aligned microcrystallites and causes a decrease of mean free 'hole' volumes of 10-20 Å^3. The free volume change influences the fate of caged singlet radical pairs. For instance, the ortho/para ratio of the acetylnaphthol products formed on photolysis of 1-naphthyl acetate changes from 4.2 in unstretched to 4.9 in stretched polyethylene. For the bulkier myristate, only ortho product is observed in either unstretched or stretched polyethylene. The confinement of the polymer environment prevents the relative reorientation of the initial radical pair that is required for para coupling. Decarbonylation of the initial alkanoyl radical in the cage gives a new radical pair whose fate is also influenced by the medium. The second part of the talk concerned covalent attachment of aromatic hydrocarbons to the polymer backbone via proton bombardment. According to Dick, Marshall McLuhan's 1964 aphorism, "The medium is the message", although made in a different context, aptly describes the controlling influence of polymeric media on photochemical processes, with the caveat that "To understand the message, first understand the medium" (Richard Weiss, 1984 unpublished).

Harry Gray (Caltech) was the first speaker of the second afternoon session on Tuesday, April 3. Harry spoke of George's enormous influence on his life, on his research career and on the inorganic and organometallic chemical community, generally. "I was a decent inorganic chemist, and then I met George", he joked. George was responsible for Harry's conversion to photochemistry and for his move from Columbia to Caltech, but when Harry turned to George and said "Thank you" he was speaking for all of us.

The title of Harry's contribution was *"Probing enzymes with photosensitizers linked to substrates"*. He spoke first of the enormous barrier to electron transfer between metal ions in water due to the extremely high water reorganization energy and how the low reorganization energy of the protein medium leads to a reduced, friendly to life, barrier. For instance, electron transfer between Fe^{2+} and Fe^{3+}, an identity reaction, occurs within 100 ms in water at a separation of 6.5 Å but would require 10^{16} years at a separation of 20 Å. In a protein environment electron transfer between Fe^{2+} and Ru^{2+} at a separation of 20 Å occurs within 0.5 µs! A series of cytochrome $P450_{cam}$ (P450) substrates tethered to luminescent Ru sensitizers bind the enzyme with comparable or greater affinities than those of the unmodified substrates. Ru-Fe(heme) energy transfer in the bound complex dramatically accelerates excited state decay. Analyses of luminescence decay kinetics yield dissociation constants for P-450/Ru-substrate complexes and similar Ru/Fe distances are extracted for a variety of complexes from the energy transfer parameters. The similarity of the distances suggests a common substrate binding access channel. Confirmation of the validity of the calculated distances was obtained by x-ray crystallography of the crystal of one complex. Selective binding of the modified substrates to P450 was inferred from the luminescence kinetics in the presence of other heme proteins, indicating that such substrates could form the basis for a new class of optically detected in situ biosensors. The use of fluorinated biphenyl linkers instead of methylene chains enhances binding 100-fold.

The second speaker, **Doug Neckers** (Bowling Green) started with a tribute to George: “We honor George Hammond, the man who brought light to an organic chemistry laboratory”. As a graduate student with Earl Huyser at the University of Kansas, Doug had studied thermally and photochemically induced free radical reductions of ketones. Influenced by fellow postdoctoral fellow Nick Turro in Paul Bartlett’s laboratory at Harvard, he settled on photochemistry as a career. Doug was the driving force that created The Center of Photochemical Sciences at Bowling Green in 1985 and brought George there as a Senior McMaster Institute Research Fellow in 1988. Through George’s frequent visits to the center, Doug has maintained a close relationship with him.

Doug spoke of his photopolymerization studies that evolved into three-dimensional imaging and photo-stereolithography. His 3-D photochemistry has had a significant impact on paleontology through the remarkable achievement of producing a 3-D polymer copy of the skull of a 2800-year old Egyptian mummy from the CT scan. Doug’s formal contribution was on *“Photoreactions studied by step-scan transient IR spectroscopy”*. He described how this technique has been applied to elucidate long-standing mechanistic questions in photochemistry. For instance, dating back to Doug’s Ph.D. studies, intramolecular γ-H abstraction in the triplet state of alkyl phenylglyoxylates had been postulated to yield a biradical that dissociates to a ketone, derived from the alkoxy group of the ester, and 1-hydroxy-1-phenylketene as a reactive intermediate. Step-scan transient IR spectroscopy provides IR spectra in a time-window of 20 ns to 5 µs following excitation and allows direct detection of ketene formation (400 ns rise-time) from the triplet state (389 ns decay time). The short lifetime of the biradical intermediate (15 ns) precludes its observation in the IR spectra. The ketene is trapped as the methyl mandelate when benzene or cyclohexane solutions of the phenylglyoxylates are irradiated in the presence of methanol. In another example, dating back to work carried out in collaboration with Nick Turro in Paul Bartlett’s laboratory, Doug described the IR detection of tetramethylcyclopropanone formed as a transient on photolysis of tetramethyl-1,4-cyclobutanedione.

Next was **Dave Eaton** (DuPont iTechnologies) who was George's last Caltech Ph.D. as a full Caltech resident, before George moved to Santa Cruz. In his Ph.D. work he had examined pyrazoline precursors to possible diradical intermediates that could potentially be involved in diene photorearrangements and photoisomerization, partly with **Bob Bergman** as a collaborator. After joining DuPont, Dave was able to observe George in his role as a consultant. George contributed intellectually as well as in his traditional role as cheerleader to the many technical developments involved in commercialization of photopolymer films for electronics and printing applications and in the process has gained many friends at DuPont. In fact, it was early collaborations among **Carl Wamser**, George and two DuPonters (**Cathy Chang** and **Charlie Baylor**) that led to an understanding of the exciplex nature of the interactions between benzophenone and Michler's ketone and to the commercial use of this "synergistic" photoinitiator. It was Dave's pleasure to convey DuPont's salute to George on his 80th birthday!

In his talk, *"Photochemical industry becomes complex, too"* Dave elaborated on the historical aspects of photopolymer technology, from its invention at DuPont by Louis Plambeck in the 1950's through its introduction into commercial use in the 60's and 70's. During this time the use of photopolymers revolutionized the ways that the printing process was accomplished and led, along with many other innovations, to the microelectronics revolution we have experienced over the last 40 years. Photopolymer printing plates and photoresists for the manufacture of printed wiring boards are now commonplace. New commercial laser-based systems take advantage of the digital data streams that are the backbone of our information exchange processes today. Dave concluded with predictions of uses of photopolymer materials in future optical communication network applications, as waveguide materials and optical components. The message: though photopolymers are now 50 years old, they continue to show promise for important new innovations for a long time to come. In this respect, they are just like George who continues to amaze us all after all these years.

Jim Yardley (Columbia Radiation Laboratory) spoke on *"Laser photochemistry: 25 years toward a fulfilled promise?"* In 1977, the lure of laser chemistry prompted Jim's move from the University of Illinois to Allied Chemical. He was there in 1978 to witness George's move to AlliedSignal, Inc. and to experience the remarkable research reorganization under his leadership.

Jim pointed out that initial attempts to develop laser activated "synthons", starting for instance, with iron pentacarbonyl and seeking through photolysis at increasing laser intensities to form tetra- and tricarbonyl reactive intermediates, failed to yield the desired selectivity. Such initial failures, coupled with the high cost of laser photochemistry, led the scientific world to turn to "high value chemistry": isotopic separation, pharmaceuticals, and catalysis, but again commercial viability proved elusive. Early successes were realized in the realm of semiconductor processing, including the development of new photoresist materials. Another area of laser photochemistry in which George played a crucial role at Allied concerned the development of guided wave optical devices. Through photochemistry, it is possible to define the spatial variation of refractive index within a polymeric medium and thus direct and control the propagation of light within that medium. This required the development of chromophore/polymer combinations that were not opaque in the UV. Photochemical transformations in the chromophore have yielded enormous changes in refractive index. Looking into the future, laser photochemistry holds the key for development of the next several generations of semiconductor devices through short wavelength laser lithography. Fabrication of low cost optical devices for telecommunications and for a host of data communication applications is also likely to depend on laser photochemistry. Jim concluded that the future of laser photochemistry is bright in ways not widely appreciated 25 years ago.

Fittingly, George’s wife, **Eve L. Menger** (Portland State University) gave the last scheduled talk of the Symposium. Eve’s talk “*Leitmotif from the life of a photochemist*”, liberally sprinkled with quotations from George’s talks, was a retrospective of George’s career. George’s scientific and teaching genius and his philosophy were revealed to the audience in his own words. When George left Caltech for UC, Santa Cruz in 1972, many of his friends thought he had taken leave of his senses. George had been drawn by the new challenges and the predictions that Santa Cruz was destined soon to become a UCLA or a Berkeley. As Vice Chancellor of all sciences, he had the opportunity and authority to implement his new ideas in the less rigid environment of a new place and, thus to work against artificial divisions in science and to emphasize instead its unity. After an exciting first 18 months in which several of George’s ideas were brought to fruition (some, e.g., a technical writing program, have blossomed and are still in existence), growth at Santa Cruz, and the flexibility it allowed, came to an abrupt halt. His appointment as Foreign Secretary for the National Academy of Sciences provided new opportunities, which George whole-heartedly embraced without sacrificing his teaching commitments. George managed to keep everything going under circumstances where the “urgent takes priority over the important”. In his classes at Santa Cruz, the students’ admiration of the way he “ordered molecules around” earned him the nickname General George. Unfortunately, among the consequences of the mistaken demographic projections were the inability to continue hiring young faculty, and a continuous struggle to get existing research laboratories properly outfitted. On top of this, some colleagues were opposed to the creation of a Photochemistry Center, viewing it as an unnecessary dilution of diminished resources. When George left Santa Cruz for Allied Chemical in 1978, friends viewed the decision as beyond the pale, but the years at Allied proved exciting and productive.

George, the thinker/philosopher was revealed in excerpts from a series of remarkable speeches on topics ranging from chemical education, to chemistry in general, and to research management. According to George, the way chemistry was generally taught left much to be desired. Freshman chemistry delivered a hodgepodge of topics and organic chemistry placed a premium on the memorization of a bunch of name reactions. He advocated the restructuring of chemistry along functional lines: structural chemistry, dynamics and chemical synthesis with sensible examples of the contributions of chemistry to other sciences. This would involve the integration of, for instance, organic and inorganic chemistry and would avoid existing divisional lines with their terribly stylized traditional courses and their devastating effect on the learning of chemistry. He called for careful scrutiny of what was taught and described our current divisional system as artificial, incoherent, redundant and harmful to its most important element, the students. In a 1971 speech to the American Institute of American Engineers, George criticized the notion that teachers should motivate students. According to George, motivation comes from within. He attacked still another artificial teaching boundary by stressing that chemical science and engineering form a continuum. He called for a more inclusive, open style of teaching, while recognizing limitations imposed on teachers by their own experiences: “My own experience of open style teaching is limited—I am a product of my own experience”. In another talk he cautioned that the rapid growth of science is not reflected in what is taught. He advised that the fact that a particular topic had been included in a course for a long time was not necessarily an indication that it was basic! Furthermore, if a topic were included in a course because it was 'basic', one should be prepared to say to what it is basic.

In 1968, talking about chemistry’s role in society, George spoke of the severe challenges faced by that science that call for its honest review. Prophetically, he saw the largest advances in biochemistry and in materials science, not in new compounds but in new properties. “My own field, Chemical Dynamics, what use is it?” he asked. If it provides the ability to use chemical reactions in systems, then the automobile combustion engine is a shame for chemists.

In 1971 George addressed the uneasy relationship between academia and industry and what each wants from the other. Industry needs graduates and universities need money, but professors view students headed to industry as unfortunate fellows destined for tedium. In 1984, to the question “Why terminate a project?” George gave a multiple-choice response:

1) It never should have been started.
2) The original goal has been accomplished.
3) The original goal is not important.
4) Even if the boss is stupid enough to think that it is good for something, there must be a subtle way to bail out.

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At the end of Eve's talk, **George Hammond** took the stage for a few moments. The whole thing, symposium/banquet, he said was just great, an incredible pleasure. He was very stimulated by this terrific symposium, but its title "Photochemistry Becomes More Complex" was probably not right, because photochemistry has always been complex. It is true, however, that we are working with more and more complexity. We have made progress and must use what we have learned and keep moving. Progress has been enormous especially in the tools that we have at our disposal. It is incredible what we can now do from transient x-ray diffraction to transient IR to **Ahmed Zewail**'s direct observation of transition states. We have clearly not been standing still, but nonetheless, George feels impatience with the rate of change.

George finished with a special thought about photochemistry. He addressed the question: Is photochemistry to go its way, fade in the background like algebra? For example, 40 years ago solvolysis studies were enormously important, but now those who do solvolysis in the course of their research hardly talk about it. To survive as a science, there must be the continuous realization that photochemistry, like other sciences, is not self-contained. One must avoid rigid definitions that separate what is, from what is not photochemistry. Similarly, George finds it irritating when called an "organic chemist". New developments at the periphery are most exciting. Photochemistry will survive in good health as a science as long as its borders remain fuzzy and expanding.

This account would not be complete without mention of the banquet that was held in George's honor on Monday, April 2. It was a gathering of some 65 friends, colleagues, and former research associates and students that joined George and Eve on this festive occasion. We each had the good fortune of having interacted with George at various stages in his career, and after dinner, we each took the microphone to say a few words to George and about George. Through those words, vignettes scattered over more than half a century, emerged the George we experienced, and taken together, wove a tapestry of his remarkable life and qualities: A warm, generous, gregarious and approving human being with an enormous appetite for science and the infectious ability to advance it. George, many happy returns!

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