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MORRIS SELIG KHARASCH

1895—1957

A Biographical Memoir by
FRANK H. WESTHEIMER

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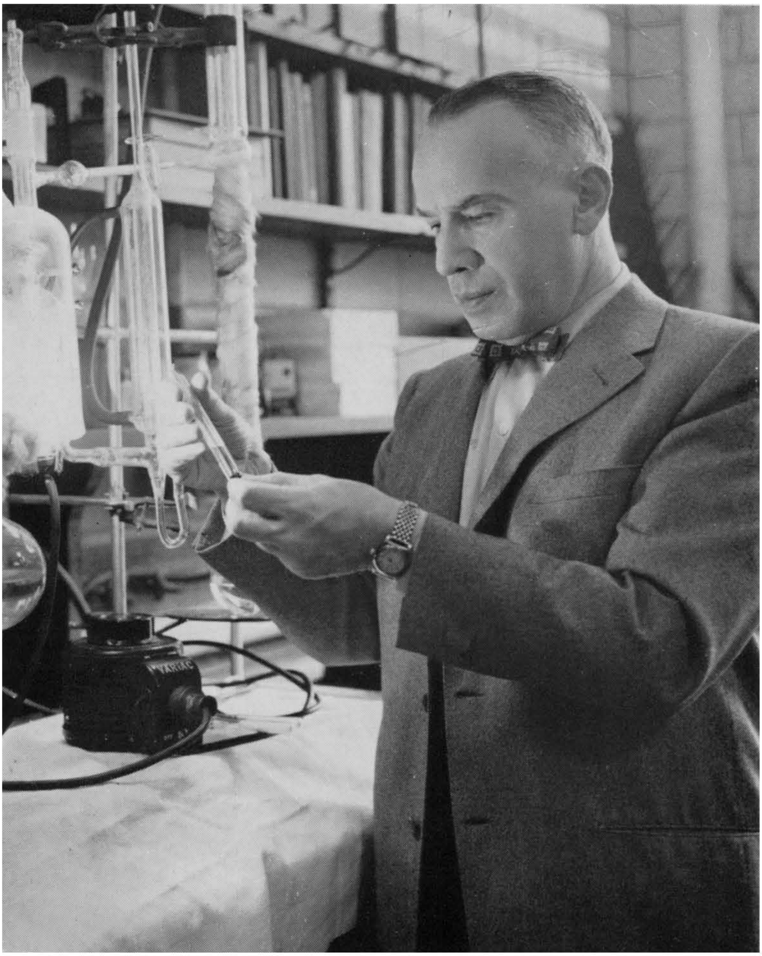

M S Kharasch

MORRIS SELIG KHARASCH

August 24, 1895–October 9, 1957

BY FRANK H. WESTHEIMER

THE TRANSFORMATIONS of organic chemistry can be roughly divided into two categories: polar reactions and free radical reactions. Most of the processes of nineteenth-century organic chemistry exemplify the former type; the recent growth of free radical chemistry is in large part the result of the highly original investigations of Morris Kharasch. He discovered—or perhaps it would be better to say invented and explained—many new reactions. In most instances, these discoveries were the result of the application of a new set of principles, which postulated free radicals as transitory, unstable intermediates in chemical processes. He thoroughly enjoyed his work, and took real pleasure, not only in his accomplishments, but in the day-to-day challenge of the laboratory. To the end of his career, he was seeking new and novel chemical reactions, and his most recent publications as well as his earlier ones bear the stamp of his originality.

Morris Selig Kharasch was born in Kremenetz, in the Ukraine, on August 24, 1895. His family was comfortably situated, but he and his brothers left Russia to take advantage of the education and opportunities which were available in the United States. Morris Kharasch came to the United States when he was thirteen; an older brother had preceded him, and helped take care of him here. He graduated from the Crane High School in Chicago in 1913 and continued his education at the University of Chicago. He received his B.S. degree in 1917 and his Ph.D. in 1919, despite the fact that he spent part of the intervening years in the U.S. Army, in the Chemical Warfare Service. After he received his doctorate, he held a National Research Council Fellowship at the University of Chicago until 1922, when he accepted an Associate Professorship at the University of Maryland. In 1924 he was appointed Professor at Maryland, but returned to the University of Chicago as Associate Professor of Chemistry (1928) and Professor (1930); from 1935 to 1953 he held the chair endowed by Carl William Eisendrath; from 1953 until his death he held the Gustafus F. and Ann M. Swift Distinguished Service Professorship. In 1956 the University of Chicago honored him by creating an Institute of Organic Chemistry under his direction, but the new Institute was scarcely under way when, on October 9, 1957, he died suddenly in Copenhagen, while carrying out an assignment for the United States government.

He was married, on June 24, 1923, to Ethel May Nelson, and had two children: a son, Robert, who is a lawyer in Washington, and a daughter, Elizabeth (Mrs. A. David Pearson; Dr. Pearson is an inorganic chemist with the Bell Laboratories).

Kharasch's life was largely devoted to his researches. For many years he took no vacation; when finally he could be persuaded to leave his work for a few weeks, he quickly became restless and usually returned to Chicago ahead of schedule. He enjoyed relaxation so long as it did not take him far from the laboratory, and took pleasure in an occasional game of bridge, chess, or billiards. He played these games hard, and in a daring, unconventional, and often spectacularly successful fashion. In some ways his technique at the bridge table was a reflection of his approach to research; he was always looking for opportunities which the usual player would not think of. Although Kharasch was not a highly social person, he enjoyed the company of his friends, and especially of his intimate family. He was entirely happy in his home, proud of and devoted to his children. Other children, in addition to his own, interested him, and they in turn were tremendously attracted to him. He felt a deep loyalty to his graduate students. During the depression, when jobs for chemists were hard to find, he worked diligently to place the graduates in chemistry from the University of Chicago. Since he himself was a highly successful consultant, several large industrial companies were willing even in the worst economic times to accommodate the men he recommended; but it is not now clear how he managed to create jobs around the University for some of his former students (and for other chemists), and perhaps one may guess that on occasion these jobs existed, temporarily, at his own expense. Although he enjoyed his personal and scientific families, he did not like large public meetings, and could only seldom be induced to present a paper at a symposium. When he did, however, the paper was crammed with new chemistry, and laced with stories. He was a storehouse of stories—some quite humorous, some only worthy of television, and a few which he repeated until they became almost trademarks of the man. On one occasion, he was induced to attend a conference on reaction mechansims. A night session went on and on, aimlessly and interminably, until Kharasch rose and told a joke which was both short and very much to the point; the session broke up cleanly in laughter. But although his presentations of his work were as interesting as they were rare, the real story of his life is published in the chemical journals, and it is in his chemistry that his biography must be written.

When he was at the University of Maryland, he carried out theoretical studies on thermochemistry and valuable experimental research on the chemistry of compounds of mercury. However, his important work on free radicals was begun when, at the age of thirty-three, he returned to the University of Chicago. In order to appreciate his contributions, a few words of background are needed.

The chemistry of free radicals in solution had begun with the brilliant work of Moses Gomberg, at the University of Michigan, in 1900. But the compounds which Gomberg had found were of a rare type, and his discovery had not altered the main stream of chemical discovery, or modified the way in which chemists thought about ordinary preparative processes. A few other papers on free radicals in solution had appeared; H. Staudinger had suggested that free radicals might be intermediates in vinyl polymerization, and Bäckstrom had made a convincing case for such radicals in the autoxidation of benzaldehyde. But the concept was strange to organic chemists, generally unproved, and little understood. Morris Kharasch was led to a consideration of free radicals, and to work which strongly influenced the thinking about the mechanisms of organic chemical processes, by a roundabout path. In 1930 the literature on the reaction between hydrogen bromide and olefins was in a state of confusion. Some chemists reported that the product of the reaction with allyl bromide was 1,2-dibromopropane, some that it was 1,3-dibromopropane, and some that it was a mixture. An eminent chemist had offered a complex theoretical explanation of the data, which may have satisfied some, and thereby delayed experimental reexamination of the difficulty, but Kharasch was not impressed by it. At first glance, this problem might seem an unpromising one for launching a new career. The chemical literature abounds with contradictions, and anyone can make a dull program correcting the minor errors of others; only on rare occasions do such contradictions lie at the base of real innovations in science. However, Kharasch had correctly grasped that this was one of those exceptional cases.

The graduate student chosen to carry on this first investigation was Frank Mayo. His subsequent career has shown him a brilliant investigator in his own right; Kharasch chose his collaborator with skill. Mayo studied the reaction between hydrogen bromide and allyl bromide, and found quite variable results. But Kharasch's careful review of the data showed that the results depended on the age of the sample of allyl bromide which had been used as starting material. Freshly distilled samples gave mostly the 1,2-dibromide; older samples gave more of the 1,3-dibromide. From these facts, Kharasch surmised that oxygen from the air had attacked the older samples, and that the cause of the discrepancy was the presence of a peroxide in the allyl bromide. Once the correct hypothesis had been formulated, the solution of the problem was quick and sure. Samples of allyl bromide which were strictly oxygen-free gave exclusively 1,2-dibromopropane, those which contained much peroxide gave exclusively the 1,3-isomer.

$$BrCH_2CH{=}CH_2+HBr \begin{array}{l} \xrightarrow{\text{No } O_2} BrCH_2CHBrCH_3 \qquad (1) \\ \xrightarrow[\text{peroxide}]{} BrCH_2CH_2CH_2Br \qquad (2) \end{array}$$

This second reaction (eq. 2) has become known, in the chemical literature, as "abnormal addition" of HBr, and as the "peroxide effect"; the phenomenon is of course associated with Kharasch's name. Kharasch and Mayo published their experimental findings in the *Journal of the American Chemical Society* in 1933; the paper foreshadowed an important advance—almost a revolution—in organic chemistry, and the publication has been accorded a high place in the annals of chemical discovery. The original paper gives the facts, but no theory; Kharasch reserved his statement of theory, his explanation of the "peroxide effect," until 1936. At that time not only he, but other chemists, and in particular Hey and Waters in England, advanced the concept of free radicals as reactive intermediates in organic chemistry.

For the particular case discussed above, Kharasch suggested that the reaction proceeds by way of the following steps:

$$BrCH_2CH{=}CH_2+Br^{\bullet} \rightarrow BrCH_2-\underset{\bullet}{C}H-CH_2Br \qquad (3)$$

$$BrCH_3-\underset{\bullet}{C}H-CH_2Br+HBr \rightarrow BrCH_2CH_2CH_2Br+Br^{\bullet} \qquad (4)$$

The bromine atom ($Br^{\bullet}$) is consumed in the first step and regenerated in the second. The free radical, $BrCH_2-\underset{\bullet}{C}H-CH_2Br$, is postulated as an intermediate in the process.* Such radicals are too reactive to be isolated; nevertheless, much chemistry can be rationalized on the basis of the assumption that they exist and participate as in-

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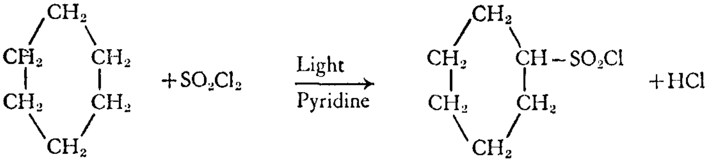
* The "dot" marks an "unpaired electron." Ordinary organic compounds have even numbers of electrons, and these electrons are paired. Those compounds with an odd number of electrons must necessarily have at least one unpaired electron. They are paramagnetic, and are called "free radicals."

termediates in chemical reactions. Their chemistry can be elucidated despite the fact that they cannot be kept in a bottle.

The sum of reactions (3) and (4) is of course reaction (2); further, the process is formulated as a "chain"; a few bromine atoms are sufficient to effect the conversion of a large quantity of allyl bromide to 1,3-dibromopropane. The peroxide serves to produce the bromine atoms and initiate the "chain" of equations (3) and (4). Some "chain terminating" step must eventually destroy the radicals. In the absence of peroxide no bromine atoms are formed, and the reaction takes an alternate (polar) course.

Kharasch postponed presenting an hypothesis for the "peroxide effect" until he had carried out many experiments on problems related to "abnormal addition." To explain his position, he repeatedly told one of his inevitable—and most probably apocryphal—stories. He liked to recall that, among the four Marx brothers, one of them was less skilled than the others, and eventually dropped out of the act. According to the story, when the next contract was to be signed, the Marx brothers were offered a contract at only three fourths of their former rate. But the remaining brothers are supposed to have asked for a raise, on the ground that the three of them were better without the fourth. So Kharasch felt that an experimental paper was better without theory, until that theory was well established.

In the long run, it was the theory of the role of free radicals in organic chemistry which opened a new field of exploration, and it is for the theory as well as for the new chemistry that Kharasch will be remembered. With a new field open before him, Kharasch exploited to the full the opportunity he had created. In the course of the next few years he invented a free-radical sulfonation reaction (with Alice Read; eq. 5) and a new reagent for chlorination (with H. C. Brown; eq. 6); he discovered (with H. C. Brown; eq. 7) a way to carboxylate paraffin hydrocarbons and (with E. Jensen and W. H. Urry; eq. 8) the addition of carbon tetrachloride to olefins, the addition of bromotrichloromethane to olefins, and the addition of chloro and bromo esters and of aldehydes to olefins. Many of these reactions would have been considered impossible only a few years earlier, had anyone bothered to consider them at all. But that is because chemists had been thinking in terms of polar, rather than



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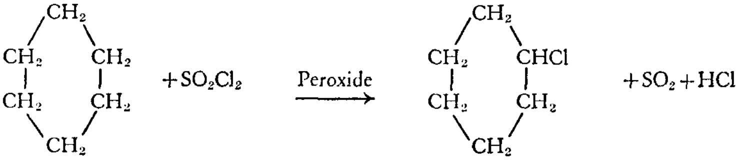

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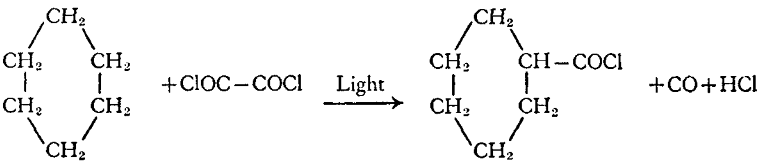
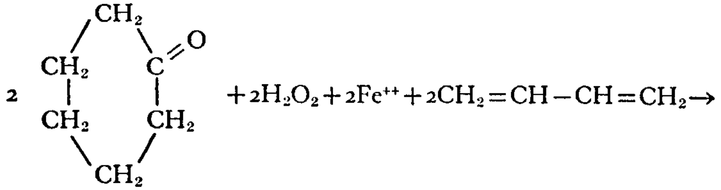


(7)

$$C_6H_5CH{=}CH_2 + BrCCl_3 \longrightarrow C_6H_5CHBr-CH_2-CCl_3 \qquad (8)$$

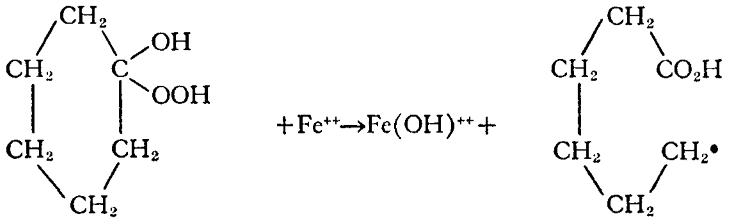
in terms of free radical, reactions. Kharasch (with Gladstone) invented a new preparation of substituted succinic acids by a free radical reaction path, discovered (with S. Kleiger and P. Tawney) the effect of trace metals on the course of Grignard reactions, and (with Sternfield and Mayo) discovered the effect of oxygen on certain molecular rearrangements. He found (with A. Fono and W. Nudenberg) a free radical cleavage of cumyl peroxide initiated by ferrous ion and exploited (with W. Nudenberg; eq. 9) similar reactions in a highly original and unconventional preparation of long-chain fatty acids from cyclic ketones, butadiene, hydrogen peroxide, and ferrous ion.



$$+2H_2O_2+2Fe^{++}+2CH_2{=}CH-CH{=}CH_2\rightarrow$$

$$HO_2C-(CH_2)_5-(CH_2-CH{=}CH-CH_2)_2-(CH_2)_5-CO_2H \quad (9)$$
$$+2Fe^{+++}$$

Presumably this reaction proceeds by way of the formation of an organic peroxide, which is reduced by ferrous ion in a one-electron step to yield an unstable free radical.



$$+Fe^{++}\rightarrow Fe(OH)^{++}+$$

(10)

This radical then adds to butadiene, to form an allylic radical which

$$HO_2C-(CH_2)_5-CH_2-CH{=}CH-CH_2\bullet$$

dimerizes to give the final reaction product.

This complicated reaction sequence illustrates the advances in radical chemistry between the discovery of the peroxide effect in 1932 and the design of synthetic procedures two decades later. In the intervening years, the stabilities and reactivities of radicals were compared. Some proved too unstable to be detected at room temperature; some could be observed by physical means (e.g., electron spin resonance spectroscopy) but rapidly reacted with other molecules, such as olefins; others were relatively inert, and could only be trapped by special reagents. The quantitative studies by numerous independent investigators added considerably to Kharasch's essentially qualitative discoveries. But reaction (9) combines much knowledge into a single unconventional synthetic procedure.

During the same period in which Kharasch was discovering free radical reactions of preparative importance to organic chemistry, the theory of polymerization was developed. Polymerizations, like other chemical reactions, are divided into free radical and polar processes. The developments in the field of free radical chemistry had an important impact on the studies of polymerization, and conversely, considerable information about the properties of free radicals was obtained from studies of polymerization. Kharasch was of course invited to participate in the synthetic rubber program of the United States, and here he employed his understanding of free radicals to good advantage. For example, by applying principles he had discovered, he found out how to protect the rubber factories from a highly undesirable side reaction, the formation of "popcorn" polymer. He had an important part in proving the mechanism by which long-chain mercaptans and other chain-transfer agents can control the molecular weight of synthetic rubber. But his major contribution to the field of polymerization was indirect; it lay in his demonstration of the properties of aliphatic free radicals.

In addition to his activities as professor, Kharasch proved highly successful as an industrial consultant, and was at times associated with the du Pont Company, Eli Lilly, the U.S. Rubber Company, and others. In general, his industrial discoveries grew out of his academic ones. His invention of merthiolate arose from his early interest in the chemistry of organic compounds of mercury; a commercial method for the preparation of the anesthetic cyclopropane was a consequence of the work with Mayo on abnormal addition; and so on. Kharasch believed that he could test his theories as well by showing an industrial application of them as he could with his academic researches, so that his consulting work strengthened and did not detract from his University activities.

In addition to his scientific accomplishments, he was an excellent teacher. When he taught advanced organic chemistry, he worked hard over each lecture, and succeeded in imparting the spirit as well as the content of chemistry to his students. A small part of his encyclopedic knowledge is preserved in a monumental compendium of the reaction of Grignard reagents which he and O. Reinmuth prepared; regrettably, he did not codify most of his work on radicals. But one of his former students, Professor Cheves Walling, has incorporated much of Kharasch's work into his monograph on "Free Radicals in Solution." Toward the end of his career, Kharasch gave up formal lecturing, but continued to train graduate students. The names of some of those he trained have been mentioned here in connection with their contributions. Most of them have continued the tradition of free radical chemistry, and many of them now enjoy outstanding careers of their own.

Kharasch was of course honored for his accomplishments. He received the Presidential Merit Award in 1948 for his services to the Chemical Warfare Service during the Second World War, the Scott Award of the Franklin Institute in 1949, and the Richards Medal of the Northeastern Section of the American Chemical Society in 1952; he was one of the founders of the *Journal of Organic Chemistry* and the American editor for *Tetrahedron*. In 1946 he was elected to membership in the National Academy of Sciences. This certainly is a distinguished list of honors. But his accomplishments and the impact of his scientific innovations were more highly regarded, and are today more highly regarded, than this list would indicate. If he received more recognition from the cognoscenti than from the prize committees, this must serve as a commentary on the latter. And perhaps a man gets, to some extent at least, what he wants most. Kharasch was always more interested in a new chemical reaction than in a new honor. He got enough of the latter to satisfy him; he probably discovered (or invented) more new chemical reactions than any other chemist of our time. He will be remembered because he made free radicals a familiar and useful part of the working equipment of organic chemists, and so changed the way in which they look at their science.

KEY TO ABBREVIATIONS

Am. J. Obst. and Gyn. = American Journal of Obstetrics and Gynecology
Bur. Stand. J. Res. = U.S. National Bureau of Standards Journal of Research
Chem. and Ind. = Chemistry and Industry
Chem. Rev. = Chemical Reviews
Ind. and Eng. Chem. = Industrial and Engineering Chemistry
J. Am. Chem. Soc. = Journal of the American Chemical Society
J. Bact. = Journal of Bacteriology
J. Biol. Chem. = Journal of Biological Chemistry
J. Chem. Ed. = Journal of Chemical Education
J. Chem. Phys. = Journal of Chemical Physics
J. Org. Chem. = Journal of Organic Chemistry
J. Phys. Chem. = Journal of Physical and Colloid Chemistry
J. Soc. Chem. Ind. = Journal of the Society of Chemical Industry (London)

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