

those taken up at this conference. He also encouraged Latin-American scientists to participate in these efforts.

The major sponsor of the symposium in Lima was Universidad Peruana de Ciencias Médicas y Biológicas, and in Cali, Universidad del Valle. Pablo Mori-Chavez was general secretary for the organizing committee.

Substantial financial assistance was also provided by the following organizations: Pan American Union, Damon Runyon Memorial Fund, Rockefeller Foundation, Anna Fuller Fund, The Jane Coffin Childs Memorial Fund for Medical Research, the United States Atomic Energy Commission, National Cancer Institute, National Science Foundation, International Atomic Energy Agency, the British Council, and numerous commercial firms in Lima and Cali.

The U.S. National Academy of Sciences-National Research Council also sponsored the meeting and encouraged the development of this series of symposia in Latin America. The first one was held in Santiago, Chile, in 1961 on "Tissue transplantation"; the second, in 1962, took place in São Paulo and Rio de Janeiro with two programs, "Mammalian tissue culture and cytology" and "Specific topics in radiobiology"; and a fourth symposium is being organized in Buenos Aires in 1964 on "Genes and chromosomes—structure and function." Publication of the proceedings of this year's conference, as a monograph from the National Cancer Institute, is anticipated.

CHARLES C GONGDON
Biology Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Free Radicals

Free radicals, particularly when trapped in solid materials at low temperatures, were the topics of discussion at the sixth international symposium on free radicals which took place 2-5 July 1963 at Cambridge, England. The total attendance was about 260, with the host (United Kingdom) delegation numbering almost as many as the representatives from all other countries combined.

R. G. W. Norrish (University of Cambridge), organizer of the symposium, gave an introductory lecture which dealt with studies of free radicals in the gaseous state and showed the transient nature of such radicals as



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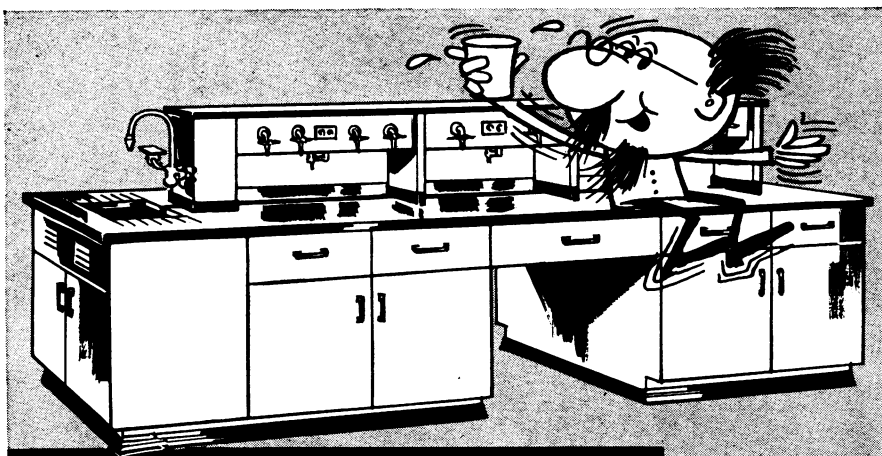
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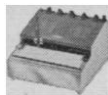
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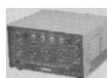
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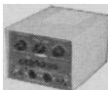
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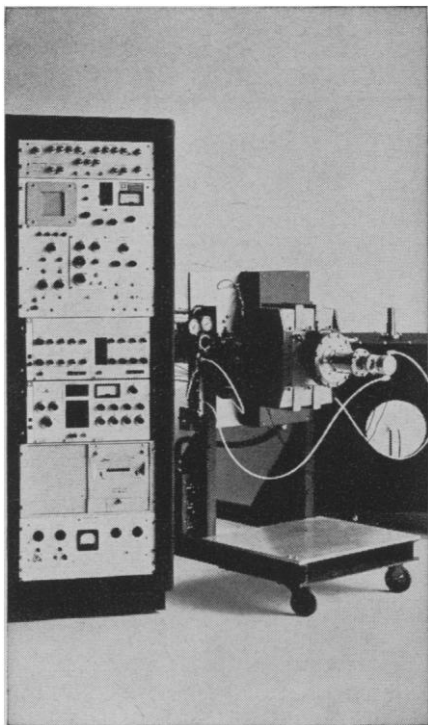
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intermediate species and the ubiquitous role of chain reactions. Under these conditions, free radicals bore the status of some postulated entities necessary for the logic of reaction kinetics but were hardly "real" enough for direct and on-the-spot inspection. It was G. N. Lewis who in 1942 first achieved stabilization of the radicals by trapping them in the frozen state, thus obviating difficulties due to very short life times. But the principal job of direct and specific inspection of trapped species had to wait for the introduction, after the end of the Second World War, of a technique known as electron spin resonance (ESR). Since then, the rapid adoption of the ESR technique for the study of free radicals has been most phenomenal. Whereas there was only one paper on ESR in the first international symposium on free radicals (Quebec, Canada, 1956) 27 of the 39 papers presented at this symposium dealt with this method.

Free radicals can be produced and trapped in solids in a variety of ways. In some experiments radicals were generated in an electric discharge and were condensed, along with other discharge products, on a cold target. In a majority of cases, however, they were generated by irradiating the solid sample at a low temperature with ultraviolet light or γ -rays (occasionally x-rays). Results obtained by using electron beam bombardment were not materially different from those using x- or γ -rays because radical production was most probably accomplished by secondary electrons in either case.

The stabilization of free radicals in solid media or on solid surfaces is not very well understood. It is hard to understand, for instance, that hydrogen atoms are not stable in irradiated ice at liquid nitrogen temperature whereas they are quite stable in certain irradiated frozen acids at the same temperature. J. Weiss (New Castle) raised questions of this kind and speculated about the nature of the trapping sites. If, according to one suggestion, the hydrogen atoms could only be stabilized at certain trapping sites, then with a limited site density the radical concentration ought to reach a saturation value after prolonged irradiation. There is as yet no experimental verification of this suggestion. In another direction, V. B. Kasanskii and G. B. Pariiskii (Moscow) studied the problem of stabilization of free radicals on solid surfaces. They found, for instance, that



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hydrogen atoms could be stabilized on the surface of a silica gel (an insulator) up to about 170°K. On the other hand, hydrogen atoms or any other free radicals could not be stabilized at all on the surface of a semiconductor or conductor. Theories postulating a one-electron bond between a radical and an insulator or a two-electron bond between a radical and a semiconductor (or conductor) may well explain the above-mentioned phenomena but they are so far only qualitative.

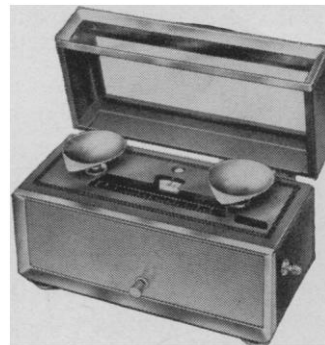
In the field of radical identification by ESR technique, many successful examples exist. However, there is also a case of considerable confusion that is concerned with the problem of identifying free radical species in a solid hydrogen-oxygen system (H₂O, H₂O₂, or mixture) when one of several radical production methods (frozen discharge product, uv, x-ray, γ-ray, or electron bombardment) is used. For a number of years various groups of workers all over the world (American, Russian, English, and French) have put forth their claims of identification (such as OH, HO₂, and so forth) with rather different kinds of ESR data and quite dissimilar interpretations. More recently, Siegel, Baum, Sholnik, and Flournoy (Aerojet Corp., California) observed a doublet ESR spectrum in γ-irradiated ice and interpreted this as due to the OH radical. The results of Kroh, Green, and Spinks (University of Saskatchewan) with partially tritiated ice strongly supported this identification.

However, one would hardly get this feeling of surety if he had listened to some of the researchers who dealt with this same general subject. After studying radicals produced in frozen H₂O₂-H₂O solutions by ultraviolet and ionizing radiations, S. J. Wyard and R. C. Smith (Guy's Hospital, London) concluded that one of five observed spectra could most probably be assigned to HO₂ but none could reasonably be assigned to OH. Other experiments with ultraviolet-irradiated, frozen H₂O₂-H₂O solution led to the belief that the observed spectra were due to a mixture of OH and HO₂ radicals, according to B. C. Green and J. W. T. Spinks (University of Saskatchewan).

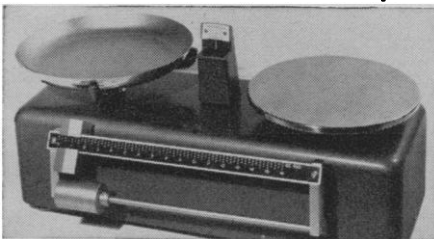
R. Marx, S. Leach, and M. Horani (University of Paris) studied the condensed product (at 77°K) of water vapor bombarded by low energy electrons and interpreted their observed spectra as due to HO₂ and one other complicated radical. Experiments by R. Livingston (Oak Ridge National

3

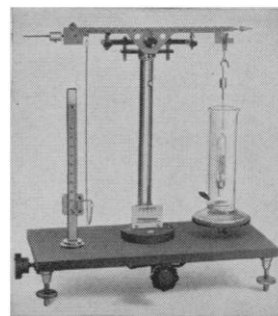
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Laboratory) with frozen discharge products of water vapor and γ -irradiated ice and solid hydrogen peroxide (both single- and polycrystalline) revealed five different types of ESR spectra, two of which could be assigned as triplet state systems. Of the three remaining spectra, two were regarded as unknown and the last one may possibly be assigned to OH. Livingston further conjectured that the triplet state species may be thought of as two OH molecules coupled in a hydrogen-oxygen complex. In summary, the problem of identifying free radicals in a hydrogen-oxygen system is still unresolved.

The electronic structure of free radicals by ESR analysis was investigated by F. J. Adrian, E. L. Cochran, and V. A. Bowers (Applied Physics Laboratory, Johns Hopkins University) in their studies of the HC=R type of free radicals. Here the unpaired electron occupies a σ -orbital which, unlike a π -orbital, has very rarely been treated experimentally or theoretically. They observed the hyperfine splittings for formyl (HC=O) and vinyl (HC=CH₂) radicals and were able to assign the observed splittings for the vinyl radical to its α and two β protons. Their theoretical investigations on the basis of a valence bond model gave results which were in good agreement with the experimental values and, in addition, spelled out the specific splittings for the two non-equivalent β protons. In another work, R. J. Cook, J. R. Rowlands, and D. H. Whiffen (National Physical Laboratory, Teddington) observed the ESR spectrum of frozen furoic acid (C₅H₄OCOOH) after x-ray irradiation. Free radicals were known to be formed by the addition of a hydrogen atom to the aromatic molecule. The problem for these investigations was to determine at which position around the aromatic ring the hydrogen atom was added. With a molecular orbital approach they concluded, from calculations, that the hydrogen atom was added at position 5, with oxygen at position 1, and COOH at position 2.

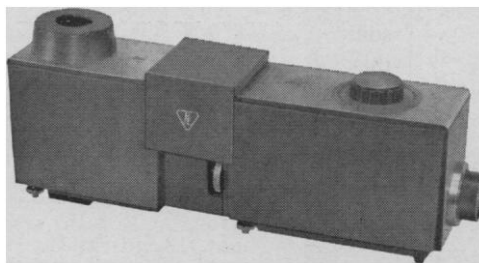
A different type of electronic structure problem is that of a triplet state molecule. This area of research, which was initiated several years ago by C. A. Hutchison, Jr. (University of Chicago), was discussed by W. A. Yager, R. W. Murray, G. Smolinsky, A. M. Trozzolo, and E. Wasserman (Bell Telephone Laboratories, Murray Hill). They observed a number of stable triplet state molecules in rigid glasses when certain organic compounds were decomposed



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by ultraviolet light. They found that a divalent carbon species (methylenes) and a monovalent nitrogen species (nitrenes) represented two classes of triplet state molecules which had the stable characteristics of being in the ground state.

Some ingenious experimental methods were presented for the study of radical reactions and intermediate radical species. A new technique was introduced by J. E. Bennett and A. Thomas ("Shell" Research, England) who used a rotating cryostat for direct measurement of rates of radical-molecule reactions. The rotating cryostat served as a "conveyor belt" on which radicals were first frozen and then bombarded by molecules for specific reactions. The reaction products were then examined by an ESR spectrometer. Another new technique was initiated by P. L. Kolker, T. J. Stone, and W. A. Waters (Oxford University) for the study of transient free radicals involved in oxidation and reduction processes. By appropriately injecting the reactants, they were able to observe intermediate radical species when the reaction products passed through the ESR spectrometer at a very high flow rate. It was possible to establish whether the observed species were the primary or secondary products of a reaction sequence. The information on the identity of transient radicals should throw light on the mechanism of chemical reactions and the nature of electron transfer for oxidation and reduction processes.

C. K. JEN

*Applied Physics Laboratory,
Johns Hopkins University,
Silver Spring, Maryland*

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The initial extramural activity of the new Instituto Di Ricerche Farmacologiche "Mario Negri" in Milan, Italy, was the organization of lectures and demonstrations on spectrophotofluorometric techniques in biology, given by an invited international staff and 110 participants from 19 countries. A NATO grant aided in the financing, and the institute provided the fine facilities of its laboratories and lecture hall now being completed in Milan. The institute, directed by Silvio Garrattini, was founded by a bequest of Mario Negri, a Milanese philanthropist.

The program was organized as a survey course. Practical experience and

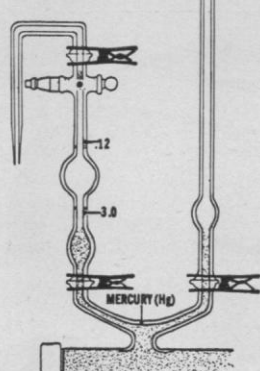
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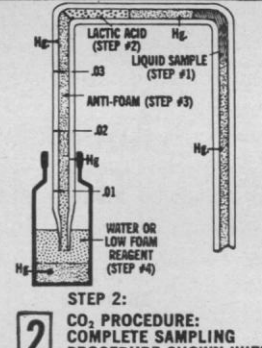
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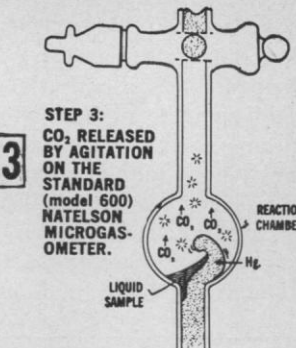
1 STEP 1:
SYSTEM UNDER
REDUCED PRESSURE
TO REMOVE ALL AIR
PRIOR TO SAMPLING.



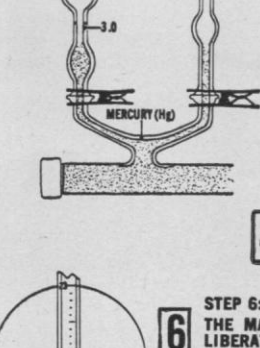
4 STEP 4:
CO₂ RELEASED BY AGITATION
ON THE MOTORIZED (model
650) NATELSON MICROGAS-
OMETER.



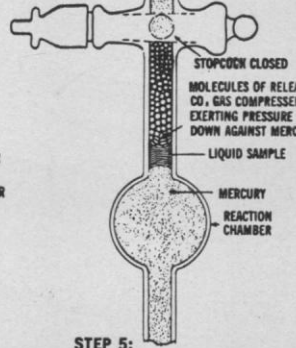
6 STEP 6:
THE MANOMETRIC PRESSURE (P₁) AS A RESULT OF COMPRESSING THE
LIBERATED CO₂ IN THE REACTION CHAMBER. THROUGH SELECTIVE
ABSORPTION WITH 3N NaOH, P₂ IS OBTAINED.



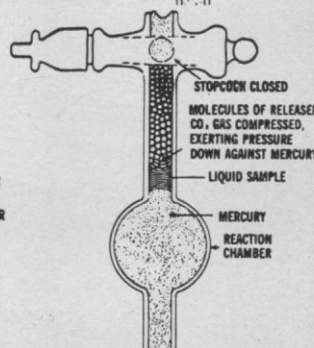
2 STEP 2:
CO₂ PROCEDURE:
COMPLETE SAMPLING
PROCEDURE SHOWN WITH
REAGENTS AND MERCURY
SEALS IN THE PIPETTE.



3 STEP 3:
CO₂ RELEASED
BY AGITATION
ON THE
STANDARD
(model 600)
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OMETER.

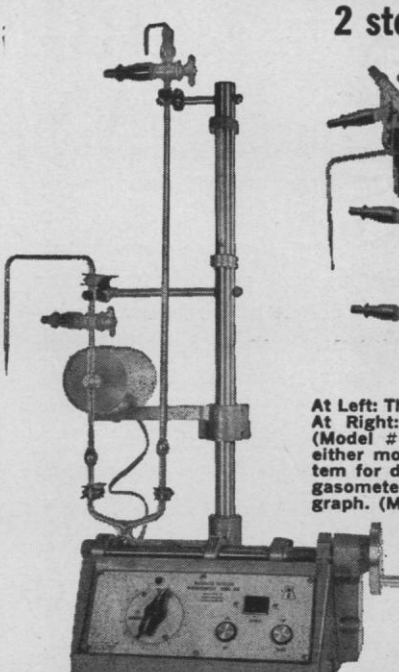


5 STEP 5:
THE MOLECULES OF RE-
LEASED CO₂ GAS COM-
PRESSED SHOWN EXERTING
PRESSURE DOWNWARD ON
THE MERCURY.

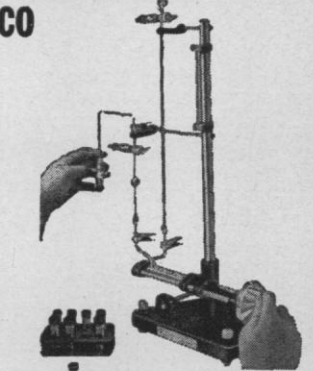


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