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SOME HISTORY OF LIQUID SCINTILLATOR DEVELOPMENT AT LOS ALAMOS¹

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I. INTRODUCTION

Liquid scintillation counting, as we know it today, is the result of continuing work in numerous laboratories by a large number of investigators, many of whom are attending this conference. Development of apparatus and methods has depended on input from almost all fields of science and technology-- physics, chemistry, mathematics, biology, engineering, medicine, and many more. A fortunate combination of talents, technology, knowledge, inspiration, and need all seemed to come together at the right time to provide for the rapid development of liquid scintillation counting in the early 1950s.

At the Los Alamos Scientific Laboratory, the quiet competence of E. Newton Hayes formed the basis of a program that continued formally throughout the decade and encompassed essentially all aspects of the field--synthesis of new solutes; characterization and evaluation of solutes, solvents, quenchers, optics, and electronics; chemistry and physics of the scintillation process; development and practical applications of both small-volume internal-sample and large-volume external-sample counters; and enthusiasm for collaboration with other investigators from many and varied disciplines. This article will review the early developments made in

¹This work was performed under the auspices of the U. S. Department of Energy.

this project which, in essence, is a summary of Dr. Hayes' work during that period.²

In 1950, when Hayes arrived in Los Alamos, liquid scintillators were being used in physics research and particularly in the nuclear weapons testing program. The liquids recently had been supplanting the large sodium iodide and naphthalene crystal systems since, in addition to their large-volume capabilities, oscilloscopes had then become fast enough to make use of them. Hayes, however, knew nothing about scintillators at that time; his position in the Biomedical Research Group was primarily that of organic synthesis and labeled compounds. The group did carry out some development and service work for the Test Division on purification of terphenyl, quaterphenyl, naphthalene, and other materials, and it was through these activities, as well as by personal contacts and friendships, that Hayes soon became aware of scintillators (Fullman, 1979; Harrison, 1979; Jackson, 1979; Herbert, 1979; Langham, 1967; Schuch, 1979; Williams, 1979). It was apparent to him that certain compounds with which he had been involved in his graduate research at Northwestern University, such as 2,5-diphenyloxazole and 2-phenyl-5-(4-biphenylyl)oxazole (Hayes, King, and Peterson, 1952a), were closely related to the best solution scintillators known at the time, p-terphenyl and p-quaterphenyl. Replacement of a phenyl ring by a heterocyclic aromatic ring in the linear aromatic systems should give structures that would scintillate and perhaps offer some advantage, at least with respect to solubility. He was encouraged to pursue his ideas by Wright Langham, his Group Leader, who had long been interested in developing better counting methods for carbon-14 and tritium in samples from biochemical and metabolic tracer studies.

So it was late in the summer of 1950 that Hayes prepared his first liquid scintillator solutions and tested them using a relative anode current device with cobalt-60 excitation in the Weapons Test Division Laboratories (Fullman, 1979; Jackson, 1979). It was immediately found that 2-phenyl-5-(4-biphenylyl)oxazole (PBO) was a better scintillator than terphenyl and that the analogous 2-phenyl-5-(4-biphenylyl)-thiazole was greatly inferior (Hayes, 1953). From these first correlations of chemical structure with properties began a

²1. Newton Hayes succumbed to amyotrophic lateral sclerosis on May 24, 1977. His physical activities necessarily had to decrease over the several years preceding; however, his mental activities were maintained at their usual high level, finally at home with the help of his wife Catherine and a computer terminal. H's absence is sorely felt by all who knew him, both personally and professionally.

program that would synthesize and evaluate hundreds of compounds as solutes, investigate the other components and the many aspects of the scintillation process, and develop practical counters that would be useful in many and diverse applications.

11. SYNTHESIS AND CHEMISTRY

For a systematic study of the relationship of chemical structure to scintillation properties so that better scintillators could be developed and also a better understanding of the scintillation process, it was necessary to synthesize a large number of compounds (Hayes, Kerr, Ott, Hansbury, and Rogers, 1958; Ott, 1958). Two major classes of compounds showed the most promise of providing the best scintillators--2,5-diaryloxazoles and -1,2,4-oxadiazoles--and were the most extensively investigated by preparation of a large number of derivatives (Hayes, 1953; Hayes, Rogers, and Ott, 1955a; Ott, Hayes, and Kerr, 1956a). Other heterocyclic systems that were studied included 2,5-diarylthiazoles (Kerr, Hayes, Ott, and Hansbury, 1959a), diaryltetrazenes (Wiley, Jarboe, and Hayes, 1957a), various aza-aromatics (Gilman, Weipert, Soddy, and Hayes, 1957), diarylpyrones (Wiley, Jarboe, and Hayes, 1957b), phenanthridines and benzoquinolines (Wiley, Jarboe, and Hayes, 1958a), oxygen-containing heterocycles (Gilman, Weipert, Dietrich, and Hayes, 1958a), N-arylated heterocycles (Gilman, Weipert, Rieck, and Hayes, 1958b), triarylpyrazolines (Wiley, Jarboe, Hayes, Hansbury, Nielsen, Callahan, and Sellars, 1958b), oxazole and thiazole quaternary salts (Kerr, Ott, and Hayes, 1960; Ott, Hayes, and Kerr, 1956a), 2-fluorenyl-substituted oxazoles and oxadiazoles (Barnett, Daub, Hayes, and Ott, 1960), dioxabridged biphenyls and binaphthyls (Simpson, Daub, and Hayes, 1973a), and dioxamethylene-bridged quaterphenyls (Simpson, Daub, and Hayes, 1973b). The listing also includes aromatic hydrocarbons such as arylphenanthrenes and dihydrophenanthrenes (Birkeland, Daub, Hayes, and Ott, 1960, 1961), diarylethylenes (Daub, Hayes, Holty, Ionescu, and Schornick, 1968; Lehman, Daub, Hayes, Yguerabide, 1971), polyaryl derivatives of metals and metalloids (Gilman, Weipert, and Hayes, 1958c), biphenyl derivatives (Gilman, Weipert, and Hayes, 1958d), and arylfluorenes (Barnett, Daub, Hayes, and Ott, 1959). Some investigations dealt mainly with the effect of chemical structure on performance, which included the effects of a dialkylamino group (Kerr, Hayes, Ott, Lier, and Hansbury, 1959b), steric inhibition of resonance (Taber, Daub, Hayes, and Ott, 1965), and a stereochemical approach to self-quenching in some quaterphenyls

(Whaley, Daub, and Hayes, 1971). It was recognized early that certain materials, for various reasons, caused quenching, and certain specific studies were made of oxygen quenching (Ott, Hayes, Hammel, and Kephart, 1955) and of photo-oxidation products of PPO (Ackerman, Daub, and Hayes, 1971).

III. EVALUATION AND TESTING

The performance of a particular compound in a scintillation counter depends on how its properties fit in with the total system. No one characteristic alone determines whether a scintillator component is good or not. The combination of its characteristics determines whether it is the material of choice for the particular application, and a material that is best for one purpose may not be the one to use in another. Its efficiency, spectral properties, solubility, cost, stability, compatibility, and other factors all need to be considered. Thus, characterization, testing, evaluation, and comparisons must be made of all the system components--solute (primary and secondary), solvents, diluents, reflectors, detectors--and their interactions. Such activities comprised a large part of the program from the beginning (Hayes, 1953) and included relative pulse height and relative current comparisons of primary (Hayes, Ott, Kerr, and Rogers, 1955c) and secondary solutes (Hayes, Ott, and Kerr, 1956c), as well as absorption, fluorescence, and scintillation spectra (Ott, Hayes, Kerr, and Benz, 1956b; Ott, Hayes, Hansbury, and Kerr, 1957; Ott, Kerr, Hayes, and Hansbury, 1960). The importance of solvent (Hayes, Rogers, and Sanders, 1955b), background vs efficiency (Domer and Hayes, 1960), quench monitoring and efficiency calibration (Hayes, 1966), spectral effects, reflectors and photomultipliers (Hayes, 1954; Swank, Buck, Hayes, and Ott, 1958) were noted and examined. The chemistry of the counting sample (Hayes, 1958) was considered, and a reliable carbon-14 standard (Williams, Hayes, Kandel, and Rogers, 1956) was prepared. Properties and comparisons also were made with studies more concerned with instrumentation and specific applications.

IV. COUNTER DEVELOPMENT AND APPLICATIONS

Development of the small-volume internal-sample liquid scintillation counters occurred essentially in parallel with that of the large-volume external-sample counters. Many of the requirements and components for the two types are the

same, but the longer path the emitted photons must travel causes certain elements, such as reflectors and solvents, to have a much greater effect on overall efficiency in large-volume detectors. On the other hand, refrigeration of the phototubes in an internal-sample counter necessitates solutes with high solubility, such as PPO, and solvents compatible with the sample. As the capabilities of these systems became apparent, applications rapidly developed, and new systems were adapted or developed to meet additional needs (Langham, 1958).

A. Internal-Sample Counters

Utilization of coincidence and fast discriminator circuitry, refrigeration of the phototubes, and use of PPO as solute were made in the first practical counter at Los Alamos (Hayes, Hiebert, and Schuch, 1952b; Hiebert and Watts, 1953; Hayes and Ott, 1957; Hiebert and Hayes, 1958). Many applications of the system (and improved versions) were soon made, including counting of tritium-labeled water and organic compounds (Hayes and Gould, 1953; Langham, Eversole, Hayes, and Trujillo, 1956), suspensions (Hayes, Rogers, and Langham, 1956d; Ott, Richmond, Trujillo, and Foreman, 1959), and various samples encountered in biology and medicine (Hayes, Anderson, and Langham, 1956a; Hayes, 1956; Langham, 1958). The early observation during solvent studies that natural carbon-14 in p-cymene (from natural-product sources) could be readily determined (Hayes, Williams, and Rogers, 1953) led to the interesting series on contemporary carbon-14 levels (Hayes, Anderson, and Arnold, 1956b; Hayes, Hansbury, and Kerr, 1960a; Hayes, Hansbury, Kerr, and Williams, 1960b; Kerr, Williams, Hansbury, and Hayes, 1962b).

B. External-Sample Counters

For experiments to detect the free neutrino, large-volume counters were needed that not only were efficient but had special requirements for the solution as to elemental composition [that is, proton and boron or cadmium densities (Cowan, Reines, Harrison, Anderson, and Hayes, 1953)]. The counter, 30 inches in diameter and 30 inches in height, with a toluene-terphenyl- α NPO solution, also became the first whole-body counter for determining the radioactivity content of several humans and dogs by incorporating a cylindrical insert to form a well counter (Reines, Schuch, Cowan, Harrison, Anderson, and Hayes, 1953). Larger whole-body (human) counters soon followed (Anderson, 1958; Anderson, Schuch, and Kerr, 1961; Anderson, Hayes, and Hiebert, 1958a,b; Kerr, Hayes, and

Anderson, 1962a), as well as a smaller version for small animals (Langham, 1958) and giant detectors for other purposes (Reines, 1958, 1960). Although not of large volume, specialized liquid scintillation rate meters for gamma and fast neutron doses were developed that employed hexafluorobenzene as a hydrogen-less solvent (Williams, 1960).

V. CONCLUSION

In the early 1960s, the scintillator program (as such) in the Biomedical Research Group was phased out, and Hayes (and others) shifted efforts to molecular biology and biological information transfer studies. However, interests in liquid scintillators naturally continued, and a number of collaborative projects were informally maintained.

Hayes received the E. O. Lawrence Memorial Award for 1969 in recognition of his fundamental contributions to the development of liquid scintillation counting. This award, which consists of a citation, gold medal, and cash, is made annually to five young scientists for meritorious contributions in atomic energy research. It was particularly fitting that he be so honored, since his research was a fulfillment of the Atomic Energy Commission's mandate to make the peaceful uses of nuclear energy benefit all mankind (Anderson, 1968). Hayes was modest and retiring by nature and could easily have been passed over by persons not intimately familiar with his great productivity coupled with a propensity for sidestepping the scientific spotlight. His array of publications is impressive not only for its numbers but also for content and his willingness to share the credit with those who have worked with him (Dunham, 1968).

Dr. Hayes would have been extremely pleased to see the program of this conference with its many subjects and topics that were of such importance and interest to him and to make the observation that there are still many important and interesting questions to be answered in the field of liquid scintillation counting.

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