III. ELECTRON MAGNETIC RESONANCE*

Academic and Research Staff

Prof. K. W. Bowers

Graduate Students

Nancy H. Kolodny	A. C. Nelson	YM. Wong
C. Mazza	R. S. Sheinson	B. S. Yamanashi
	S. N. Suchard	

A. RADICALS IN THE GAS PHASE

Paramagnetic species in the gas phase are of special interest, because of their unusual physical and chemical properties. In the past few years, detection of a few simple radicals has been reported $(NO^1, OH^2, CIO^3, BrO^3)$. These successes make us considerably optimistic about the detectability of more complex but much less stable radicals, such as CH_3 and C_2H_5 . We hope that study of these species in the gas phase may lead to an understanding of their internal molecular dynamics. In the past few months, two methods have been explored for generating ethyl radicals. In both cases, hydrogen atoms were produced by a microwave discharge of hydrogen gas at ~100- μ pressure. In one case, ethylene was added to the stream of hydrogen atoms; in the other, ethyl iodide was used as reactant. The choice of ethyl iodide was suggested by the fact that the activation energy of the reaction

 $C_2H_5I + H \longrightarrow HI + C_2H_5$

is very small. Present results favor the use of ethyl iodide.

Production and detection of the radicals is carried out in a flow system through an Electron Magnetic Resonance spectrometer. Throughout the process, the system is maintained at constant pressure between 100μ and 300μ . Numerous lines have been observed, but at very poor signal-to-noise ratios. Current work is directed toward improving the signal-to-noise ratio with improved flow systems and better resonant-cavity design. Also, a small mass spectrometer is being constructed to monitor the concentrations of the various species that are formed. Work is also under way to produce enough CH to work with (by different means).

K. W. Bowers

References

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^{*}This work was supported by the Joint Services Electronics Programs (U.S. Army, U.S. Navy, and U.S. Air Force) under Contract DA 28-043-AMC-02536(E).

(III. ELECTRON MAGNETIC RESONANCE)

B. CONSTRUCTION OF A RAPID-SCANNING EPR SPECTROMETER COUPLED WITH A FLASH-DISCHARGE SOURCE

The use of high-intensity, short-duration flash-discharge lamps for the production and study of transient species and excited states of molecules by optical means is well known. The power of magnetic resonance methods has not yet been applied to such methods, because of a large number of difficulties that we now seem to have overcome. Our system behaves as follows: A 1-mfd, 3000-volt capacitor bank is discharged through a specially designed Xenon flash tube that is focused on the sample of interest in the resonant cavity of an EPR spectrometer; the flash triggers a series of pulses, which (i) triggers a ramp used for field sweep, (ii) initiates the recording of the spectrum by a small digital computer (1000 points at 25 μ sec per point), (iii) shuts off the computer, (iv) shuts off the ramp, (v) recharges the capacitor bank, and (vi) begins the cycle over again, adding the new signal to the old for a predetermined number of cycles; the initial magnetic field is shifted and the whole process is repeated. At present, we are involved in the optimization of the various components of this apparatus, and we hope to present a complete description in the next progress report.

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