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HALOGEN ATOM REACTIONS ACTIVATED BY RADIATIVE
NEUTRON CAPTURE AND ISOMERIC TRANSITION

Progress Report No. 7

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PROJECT ABSTRACT

By employing radiogas chromatography, solvent extraction technique, the freeze-thaw method and other techniques for determining total organic and individual product yields induced by radiative neutron capture (n,γ), isomeric transition (IT), and other nuclear processes, "hot atom" reactions of chlorine, bromine and iodine are being studied extensively in various gaseous, liquid, and solid halothanes, hydrocarbons and other organic systems. The study of the systematics of hot atom reactions in the gaseous state will be continued. The reactions of ^{38}Cl , $^{80}\text{Br}^m$, ^{80}Br , $^{82}\text{Br}^m + ^{82}\text{Br}$, $^{82}\text{Br}_2$, ^{128}I , ^{130}I , $^{130}\text{I}^m + ^{130}\text{I}$ activated by radiative neutron capture or isomeric transition in halo substituted alkanes will be further studied. Of importance will be calculations of kinetic energy spectra of hot atoms activated by radiative neutron capture, where possible, using computer techniques. The gas to liquid to solid transitions in halogen hot atom chemistry, involving the various isotopes of chlorine, bromine and iodine activated by the (n,γ) and (IT) processes will be investigated in more detail. Attention will be given to the high pressure gaseous reactions and those occurring in the critical temperature region. The reactions of iodine with olefins will be continued. Reactions of ^{123}I , ^{128}I , ^{130}I , and $^{130}\text{I}^m + ^{130}\text{I}$ with various gaseous, liquid and solid olefins will be studied employing iodine, rare-gas moderators or molecular additives in an attempt to learn more about the systematics and mechanisms of complex hot and thermal reactions. The stereochemistry of energetic $^{34}\text{Cl}^m$, ^{38}Cl , ^{80}Br and $^{80}\text{Br}^m$ substitution reactions involving organic molecules containing one asymmetric

carbon will be continued employing the facilities of Brookhaven National Laboratory. The general applicability of kinetic theories to heavy halogen gaseous systems will be studied.

PREFACE

The following is a report of the work completed during the period February 15, 1972 to February 14, 1973. These studies were supported by the U.S. Atomic Energy Commission, Division of Research, Contract No. AT(11-1)-1617, the Department of Chemistry of the University of Nebraska, and the University Research Council. Most of the material for this report has been accepted, submitted, or will be submitted for journal publication.

FACILITIES

A. Triga Mark II Reactor

All neutron irradiations were made in the triga Mark II "swimming pool" nuclear reactor at the Veteran's Administration Hospital at Omaha, Nebraska. A flux of 1×10^{17} thermal neutrons $\text{cm}^{-2} \text{sec}^{-1}$ was present at an operating power of 15.5 kilowatts. Various positions in the "lazy susan" sample holder were employed and the assembly was rotated to ensure that all samples received the same neutron flux and radiation dose. For very short irradiations the assembly was not rotated and samples were irradiated in the same position. The radiation dose was approximately 3×10^{17} e.v.g. min^{-1} using Fricke dosimetry.

The Brookhaven National Laboratory 60" cyclotron was used for production of $^{34}\text{Cl}^m$ whose hot atom chemistry and retention of optical configuration studies were performed in the laboratory of Dr. A. P. Wolf.

This past year the nuclear reactor operated routinely for at least four eight-hour days a week at 15.5 kilowatts. The radiochemistry group used for the period from August 1, 1970, to July 31, 1971, 302 hours (integrated time in reactor) at 15.5 kilowatts power, making 2,100 irradiations.

B. Radiochemistry Laboratory

All sample preparation and much of the radioassaying were performed in a well-equipped, specially-designed radiochemistry

laboratory in the new Hamilton Hall Chemistry building. The two hot laboratories contain four six foot "Oak Ridge-Type" hoods, several radioactive waste sinks, radiation safety equipment, seven separate vacuum lines for preparing gas, high pressure gas, liquid and solid state systems, and the usual laboratory facilities. The counting room is equipped with two radiogas chromatographs, three single-channel analyzers with several 2" x 2" NaI crystals, four GM counting stations, four gas-flow counters, and a Nuclear-Data 128-Channel analyzer with a 3" x 3" NaI crystal specially housed in a concrete, cadmium and lead cave.

A small portable radiogas chromatograph is presently being built to be located in the radiochemistry room of the Omaha V. A. Hospital reactor, allowing us to analyze routinely many more ^{128}I and ^{80}Br containing samples. This is quite necessary for the study of ^{128}I reactions activated by the (n,γ) process in the various isomers of butylene in both the gas and condensed states, precluding the necessity of making corrections for radiation damage and thermal reactions occurring during lengthy neutron irradiations.

If and when we get a neutron generator we have a specially-constructed room for its housing. This room is presently used for equipment storage.

For most of our ^{80}Br , ^{38}Cl and ^{128}I organic yield experiments, we used the radiochemistry facilities and radioassaying equipment of the Omaha VA Hospital. The use of their RIDL 400-channel analyzer with 3" x 3" NaI crystal, 3" x 5 mm NaI crystal, and Ge(Li) detectors was most helpful in many of our studies.

SUMMARY OF PRESENT PROGRAM

Since the discovery of the short-lived isomers $^{82}\text{Br}^m$ and $^{130}\text{I}^m$ it is now possible to do extensive studies of radiative neutron capture and isomeric transition activated reactions of bromine and iodine in organic media. ^{1,2,3} Willard in a review ⁴ stated that dominant among the questions to which answers are being sought are: (1) the relative importance of kinetic energy and of charge in determining the chemical fate of the atoms studied, (2) the effect of phase on the mechanisms which lead to chemical stabilization.

In our attempt to answer these questions in chlorine, bromine and iodine hot atoms chemistry we are concentrating our efforts in five areas:

- (1) Systematics of halogen hot atom reactions.
- (2) Gas to liquid to solid transitions in halogen hot atom chemistry.
- (3) Reactions of iodine with olefins.
- (4) Stereochemistry of chlorine, bromine and iodine atoms with molecules containing one asymmetric carbon.
- (5) General applicability of kinetic theories to heavy halogen gaseous systems.

Summarized below is our main progress during the report period:

1. Systematics of Halogen Hot Atom Reactions

It was found in a systematic study ⁽⁵⁾ of ^{128}I reactions activated by radiative neutron capture in various gaseous halomethanes that the formation of ^{128}I -labeled organic products proceeds entirely by hot (requiring excess kinetic energy to occur) reactions. This is unlike the reactions of ^{128}I

with CH_4 where the organic product $\text{CH}_3^{128}\text{I}$ was formed not only by hot ^{128}I atoms but by thermal ion-molecule reactions involving I^+ in the $^1\text{D}_2$, $^3\text{P}_0$, $^3\text{P}_1$, and $^3\text{P}_2$ states. In the various halomethane systems only two ^{128}I -labeled organic products were found, those resulting from halogen and hydrogen substitution. The limiting ^{128}I organic yields in gaseous CH_3F , CH_3Cl , CH_3Br , and CH_3I were 11.2, 4.2, 0.67, and 0.20% respectively. The kinetic energy spectra for (n, γ)-activated ^{128}I atoms or ions were calculated and the results showed that an appreciable fraction of the ^{128}I species are born with low kinetic energies, in or near the reactive zone. The only physical or chemical parameter that explained the trend in organic yields was the energy degradation factor of the halomethane system, correlating well with the kinetic energy spectrum of ^{128}I .

The reactions of (n, γ)-activated and (I.T.)-induced ^{130}I in gaseous CH_4 and CD_4 were determined and discussed.⁶ The results suggested that the reactions of ^{130}I were complex involving not only iodine atoms or ions possessing excess kinetic energy but also translationally thermalized and electronically excited iodine ions in $^1\text{D}_2$, $^3\text{P}_0$, and $^3\text{P}_1$ states. The hot organic yields of (n, γ)-activated $^{130}\text{I} + ^{130}\text{I}^{\text{m}}$ were consistently higher than (I.T.)-activated ^{130}I yields in the gaseous system studied. This may suggest that (n, γ)-activated $^{130}\text{I} + ^{130}\text{I}^{\text{m}}$ hot reactions occur predominantly as the result of kinetic energy imparted to the recoil iodine atoms or ions following gamma-ray cascades while the isomeric transition induced hot reactions which have lower organic yields occur predominantly as a result of kinetic energy acquired following coulombic repulsions.

The reactions⁷ of ^{82}Br activated by isomeric transition in CH_4 were studied in regard to the effect of rare gas concentration on the individual product yields. Two ^{82}Br labeled products, $\text{CH}_3^{82}\text{Br}$ and $\text{CH}_2^{82}\text{Br Br}$ were identified. Extrapolation of the individual product yields to zero mole fraction CH_4 gives $0.9 \pm 0.2\%$ and $0.8 \pm 0.9\%$ for $\text{CH}_2^{82}\text{Br Br}$ and $\text{CH}_3^{82}\text{Br}$ respectively. An important observation was that the only ^{82}Br labeled product affected by the argon sensitized radiation damage is $\text{CH}_2^{82}\text{Br Br}$, which is formed exclusively by thermal (non-kinetic energy activated) ion-molecule reactions. No radiation damage was associated with the $\text{CH}_3^{82}\text{Br}$ product.

Information regarding the absorption of a slow neutron by an iodine or bromine nucleus which results in the prompt emission of a gamma cascade is in the literature. We have attempted using these data to obtain a kinetic energy spectrum for hot iodine atoms⁵ and will attempt to do the same for other hot atoms, where there is sufficient gamma-ray data in the literature. Our purpose in evaluating the kinetic energy spectra of ^{128}I was to obtain data regarding the distribution of recoil energy imparted to an iodine atom as a result of cascade gamma emission. For our calculation we employed an IBM-360 Model 651H computer. These results will be of fundamental importance in developing an understanding of (n,γ) -activation and the applicability of kinetic theory to hot atoms produced by these activations.

2. Gas to Liquid to Solid Transitions in Hot Atom Chemistry

In our estimation one of the most potentially significant experiment for the condensed state in recent years is the gas to condensed phase

transition experiment of ^{18}F in CH_3F by Richardson and Wolfgang.⁸ For one thing it ties in work done in low pressure, high pressure, and condensed state systems in terms of a density parameter. These authors found that the two labeled products, methyl fluoride and methylene fluoride show sharp rises in their product yields from the lower densities leveling off at 0.2 g/cm^3 .³ This is characteristic of improving collisional deexcitation of primary products formed with such high internal energy that they would otherwise undergo secondary decomposition.

A second rise in yields is observed at higher densities. The authors¹⁰ feel that it is implausible to ascribe this phenomenon to collisional stabilization of excited molecules. The most natural explanation of the final rise in yields is that it is due to caging effects.

Hot fluorine atoms can break bonds forming radicals such as CH_2F and CH_3 .

The ^{18}F after losing its excess energy can undergo recombination as shown below:

While Franck-Rabiowitsch caging has long been postulated in condensed phase hot atom chemistry, there has never been any direct evidence for it.

It is of great importance for condensed state studies to perform similar experiments for ^{38}Cl , $^{80}\text{Br}^m$, ^{80}Br , ^{82}Br , $^{82}\text{Br}^m + ^{82}\text{Br}$ and for ^{128}I , ^{130}I , $^{130}\text{I}^m + ^{130}\text{I}$ atoms activated by radiative neutron capture or isomeric transition. We have initiated a program where we are

studying the effects of increasing density and phase change on the individual product yields of chlorine, bromine and iodine isotopes activated by the (n,γ) and (I.T.) process.

At the present time we have data, though some of it is preliminary, on the following systems:

No attempt is made to analyze our data at this time. A preliminary observation we would like to report,⁹ however, is that the bromine organic yields as a function of density are similar to those reported by Richardson and Wolfgang.³⁰

3. Reactions of Iodine with Olefins

We are continuing progress on this series of studies. Paper No. III

of this series reports¹⁰ on the reactions of (n,γ)-activated ^{128}I with gaseous ethylene and propylene. The total organic product yields were found to be $18 \pm 1\%$ and $24 \pm 2\%$, respectively. In both studies $\text{CH}_3^{128}\text{I}$ was found to be the predominant product. Gas additive studies have shown that $\text{CH}_3^{128}\text{I}$ is stabilized in the order $\text{Xe} > \text{Kr} > \text{He}$. This trend, exactly opposite to that expected of kinetic energy moderation, is attributed to the ion-molecule reaction $\text{I}^+(\text{}^3\text{P}_0 \text{ or } \text{}^3\text{P}_1) + \text{C}_2\text{H}_4 \rightarrow \text{C}_2\text{H}_4\text{I}^+$ as the initiating step in $\text{CH}_3^{128}\text{I}$ formation. Additional labeled products from ethylene are $\text{C}_2\text{H}_3^{128}\text{I}$ and $\text{C}_2\text{H}_5^{128}\text{I}$. The formation of $\text{C}_2\text{H}_3^{128}\text{I}$ is dependent upon the presence of molecular iodine and appears to be due to the reaction $^{128}\text{I}_2^+ + \text{C}_2\text{H}_4 \rightarrow \text{C}_2\text{H}_4^{128}\text{I} + \text{HI}^+$. The results suggest that $\text{C}_2\text{H}_5^{128}\text{I}$ is formed via the activated monoiodoethyl radical. $\text{CH}_3\text{C}^{128}\text{I} = \text{CH}_2$ from propylene is believed to be produced in an analogous way to that of $\text{C}_2\text{H}_3^{128}\text{I}$ from ethylene.

We are currently studying the reactions of Iodine-128 with the various isomers of butylene in both the gas and liquid states.

4. The Stereochemistry of Energetic Halogen Substitution Reactions at Asymmetric Carbon Atoms

At the present time, Mr. R. Pettijohn of our research group is working in Dr. A. P. Wolf's laboratory at Brookhaven National Laboratory. This is a collaboration effort with Dr. Wolf. The following is a report by him of his activities at Brookhaven National Laboratory.

A complete understanding of the substitution process at asymmetric carbon atoms by an energetic halogen is of fundamental importance to the recoil chemistry of the halogens.¹¹⁻¹³ Previous studies¹⁴⁻¹⁹ of the substitution of recoil halogen for halogen, eq. 1., have shown that the replacement occurs primarily with retention of configuration.

The almost complete retention of optical configuration in the gas phase¹⁵⁻¹⁸ has been explained by the impact model to be due to the unimolecular decomposition of those species undergoing the highly vibrationally excited inversion process.²⁰

In contrast, less stereospecificity is observed in the condensed phases.^{14-15,19} Whether this is due to a true inversion process followed by solvent stabilization or radical-radical cage combination reactions leading to racemization of the intermediate radical is still unclear.

All previous stereochemical studies of halogen substitution processes have used diastereomers as target molecules. Recent evidence has shown that the primary substitution event is being over-shadowed by strong conformational steric effects in these species.¹⁹ Our study, using enantiomeric molecules in which these effects are not present, should provide a sterically-unhindered and conformationally-independent pathway for the substitution of activated fluorine, chlorine or bromine atoms.

EXPERIMENTAL - CURRENT AND PROPOSED

Figure 1, diagrams the synthetic routes used in the preparation of D- and L-2-chloropropionic acid,²¹⁻²² D and L-2-chloropropionyl chloride,²³⁻²⁴

and D and L-2-chloropropionol.²² The above compounds were prepared by the method used in the corresponding reference(s) with only minor changes in procedure. Optical purity was measured to be $\geq 95\%$ in all cases.

In each of these compounds the single chlorine atom is attached to an asymmetric carbon atoms and is in an environment of minimum steric hindrance to a substituting halogen approach. A substitution taking place with retention of configuration on a molecule with an L optical configuration will result in a labelled products of L configuration. A substitution taking place with inversion of configuration on a molecule with L optical configuration will result in a labelled product of D configuration.

Since both reaction modes are possible it is necessary to be able to resolve the mixture of D and L isomers into its components after activation of the initially optically pure target molecule. This separation has been accomplished for both 2-chloropropionic acid and 2-chloropropionyl chloride by formation of diastereomers with L-Valine methyl ester.¹⁵⁻¹⁶ The techniques being used in determining the degree of inversion or retention of optical configuration during the chlorine for chlorine substitution on optically active L-2-chloropropionyl chloride and L-2-chloropropionic acid are shown in Figs. 2 and 3 respectively. The chromatographic separation is shown in Fig. 4.

Due to the high boiling points of the diastereomeric amides the conventional Wolf-type flow through counter could not be used. A heated flow through glass cell has been designed and fabricated and is now being tested. It will provide for the efficient and quantitative flow counting of high boiling compounds without the need of trapping the peaks on

activated charcoal. Two 3 x 4 inch Harshaw NaI crystals are linked in a coincidence circuit to lower the background to virtually zero for counting position annihilation gammas from ^{34m}Cl and ^{18}F . For non-position emitting isotopes the signals from the two crystals are added. The crystals are kept at room temperature by use of efficient forced air cooling foils between the heated flow through section and the crystals.

A technique for the resolution of a mixed solution of D and L isomers of 2-chloropropanol is presently being developed.

Optically active chlorofluoroacetic acid with chlorine and fluorine at a single asymmetric center has been synthesized as shown in Fig. 5.²⁷⁻²⁸ The resolution has been achieved by formation of the dehydroabietylamine salts.²⁹⁻³⁰ Optical active chlorofluoroacetyl chloride and chlorofluoroethanol are currently being prepared. The resolution of the chlorofluoroacetic acid, acid chloride and alcohol will be carried out in an analogous way to that of the 2-chloro series.

Brookhaven National Laboratory has a wide range of facilities for the activation of halogen isotopes. Presently, use is being made of the 60-inch cyclotron (12-40-MeV p, 10-20-MeV d, 30-40 MeV ^4He , and 10-57-MeV ^3He) to produce ^{34m}Cl and ^{18}F recoil species. Future plans include the investigation of charged particle produced ^{38}Cl , ^{39}Cl , ^{80}Br and ^{80m}Br . The Medical Research Reactor (BMRR) with a thermal neutron flux of $\approx 10^{13}$ n/cm² sec makes possible the additional complementary study of (n, γ)-activated ^{38}Cl , ^{80}Br , ^{80m}Br , and ^{82}Br . See Table I. Studying the substitution process by the use of this wide variety of recoiling isotopes will facilitate the determination of any

isotope as well as activation energy effect. In addition, the effect of changes of phase, temperature, solvent, and scavenger on the retention to inversion ratio will make possible a clear interpretation of the actual recoil induced substitution process.

Figure 1. Synthesized optically active halocarbons with chlorine at the asymmetric center.

*These compounds have also been prepared in the D-configuration from D(-)-Alanine.

Figure 2. Schematic diagram of the technique being used in determining the degree of inversion or retention of optical configuration during the chlorine for chlorine substitution on optically active L-2-chloropropionyl chloride.*

Figure 3. Schematic diagram of the technique being used in determining the degree of inversion or retention of optical configuration L-2-chloropropionic acid.*

*Also D-2-chloropropionic acid.

Figure 4. Resolution of the LD and LL diastereomers following the reaction of the above compounds with L-Valine methyl ester. Column - 1/4" x 25' carbowax 20M on Anakrom 80/90 Mesh.

*Impurity due to trace amounts of D-Valine methyl ester.

Figure 5. Synthesized optically active halocarbons with chlorine and fluorine at the asymmetric center.

TABLE I. Recoil halogen isotopes produced at Brookhaven National Laboratory to be used for the energetic halogen substitution experiments.

Production	Isotope	Reaction	Half-life
Cyclotron	^{19}F	$^{19}\text{F}(\text{p},\text{pn})^{18}\text{F}$	109.7 min.
Cyclotron	$^{34\text{m}}\text{Cl}$	$^{35}\text{Cl}(\text{p},\text{pn})^{34\text{m}}\text{Cl}$	32.0 min.
Cyclotron	^{38}Cl	$^{40}\text{Ar}(\text{d},\alpha)^{38}\text{Cl}$	37.2 min.
Cyclotron	^{39}Cl	$^{40}\text{Ar}(\text{d},\alpha)^{39}\text{Cl}$	55.5 min.
Cyclotron	^{80}Br	$^{81}\text{Br}(\text{p},\text{pn})^{80}\text{Br}$	17.6 min.
Cyclotron	$^{80\text{m}}\text{Br}$	$^{81}\text{Br}(\text{p},\text{pn})^{80\text{m}}\text{Br}$	4.4 h.
Reactor	^{38}Cl	$^{37}\text{Cl}(\text{n},\gamma)^{38}\text{Cl}$	37.2 min.
Reactor	^{80}Br	$^{79}\text{Br}(\text{n},\gamma)^{80}\text{Br}$	17.6 min.
Reactor	$^{80\text{m}}\text{Br}$	$^{79}\text{Br}(\text{n},\gamma)^{80\text{m}}\text{Br}$	4.4 h.
Reactor	^{82}Br	$^{81}\text{Br}(\text{n},\gamma)^{82}\text{Br}$	35.4 h.

5. Kinetic Theory Applications to Hot Halogen Atom Reactions Activated by Radiative Neutron Capture

Our present progress is summarized in the Ph.D. thesis of Dr. M. Young of our group. Briefly, the more recent kinetic theories are considered in their relevance to heavy halogen atom reactions activated by radiative neutron capture, and the validities of these theories are tested for (n,γ) activated ^{128}I and $^{130}\text{I}^m + ^{130}\text{I}$ reactions.

PERSONNEL, PUBLICATIONS, TALKS AND MEETINGS

A. PERSONNEL LISTING:

1. Project Director
E. P. Rack
2. Postdoctoral Associate
R. Helton
3. Graduate Students
O. Gadeken (graduated with M.S., 1972)
R. Helton (graduated with Ph.D., 1972)
D. Oates (graduated with M.S., 1972)
R. Pettijohn
K. Schwartz (graduated with M.S., 1972)
M. Yoong (graduated with Ph.D., 1972)
4. Undergraduate Students
Douglas Duven
5. Technician
W. M. Grauer

B. PUBLICATIONS:

1. M. Yoong, Y. C. Pao and E. P. Rack, Systematics of (n, γ)-Activated Hot ^{128}I Reactions in Gaseous Methane and Halomethanes. The Energy Degradation Factor; J. Phys. Chem., 76, 2685 (1972).
2. R. R. Pettitjohn and E. P. Rack, Reactions of Iodine with Olefins. III. Radiative Neutron Capture Induced Reactions of Iodine-128 with Gaseous Ethylene and Propylene, J. Phys. Chem., 76, 3342 (1972).
3. J. B. Nicholas, M. Yoong, and E. P. Rack, Reactions of ^{130}I Activated by Radiative Neutron Capture and Isomeric Transition in Gaseous CH_4 and CD_4 , Radiochim. Acta. (In press).
4. R. W. Helton, W. M. Grauer, and E. P. Rack, The Gas to Liquid to Solid Transitions in Halogen Hot Atom Chemistry I. Reactions of (n, γ)-Activated Bromine-80 in Methyl Fluoride, Radiochim. Acta., (In press).
5. R. W. Helton, D. W. Oates, and E. P. Rack, Reactions of ^{82}Br Activated by Isomeric Transition in CH_4 : A Re-Evaluation, Bull. Chem. Soc., Japan (In press).
6. A. J. Blotcky, L. J. Arsenault and E. P. Rack, An Optimum Procedure for the Determination of Selenium in Biological Specimens Using $^{77\text{m}}\text{Se}$ Neutron Activation, Anal. Chem. (In press).

C. TALKS

R. R. Pettijohn (with E. P. Rack, "Reactions of Iodine with Olefins. III. Radiative Neutron Capture Induced Reactions of Iodine-128 with Gaseous Ethylene and Propylene. New York, New York, August, 1972. Division of Nuclear Chemistry and Technology.) American Chemical Society National Meeting.

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