

**BORON IN NUCLEAR MEDICINE: NEW SYNTHETIC APPROACHES TO
PET AND SPECT**

Final Report

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George W. Kabalka

Department of Chemistry
The University of Tennessee
Knoxville, Tennessee 37996-1600

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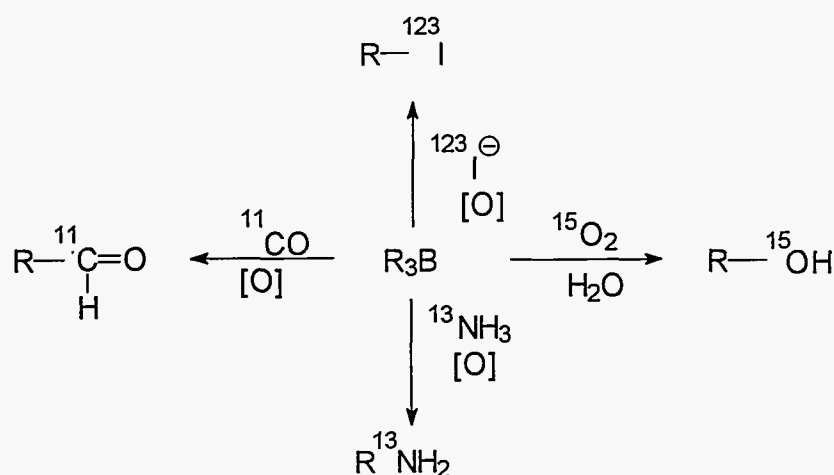
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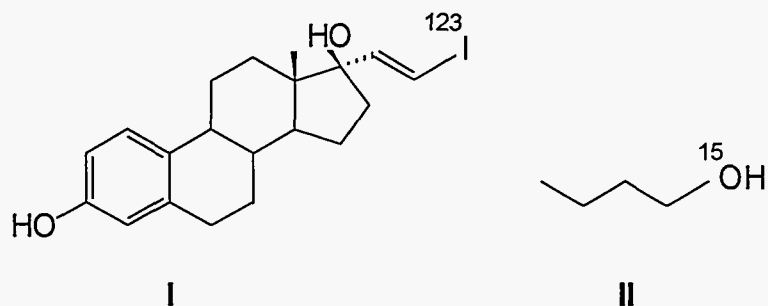
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1. New Isotope Incorporation Reactions

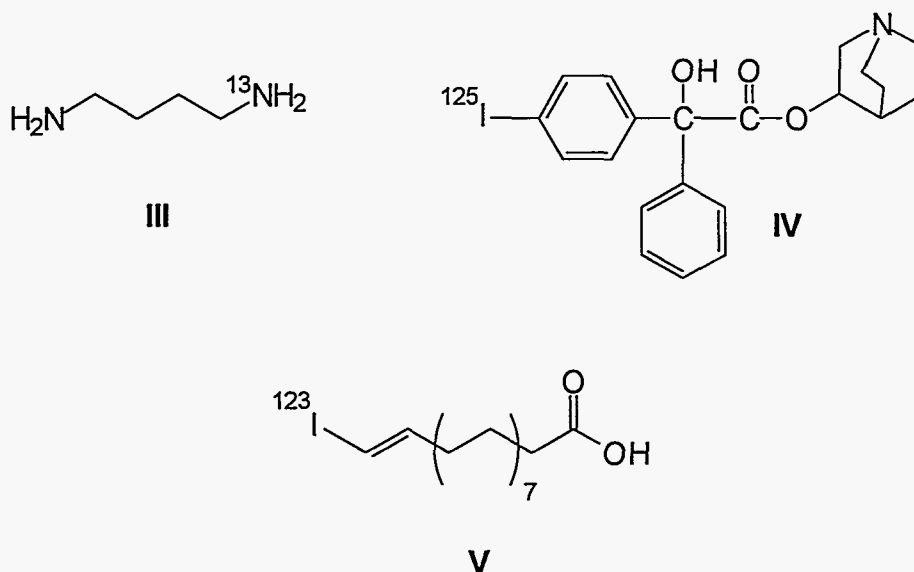
The major thrust of the D.O.E. program at The University of Tennessee is the development of new synthetic methodology for use in the rapid incorporation of short-lived isotopes. The program has been a productive one; one hundred and thirty five journal articles were published and one hundred and ninety one refereed abstracts appeared in print. In the initial stages of the program, efforts were directed at the use of functionalized organometallic reagents which would rapidly react with radiolabeled agents generated directly in the cyclotron or reactor targets. These included $^{18}\text{F}^-$, $^{15}\text{O}_2$, $^{13}\text{NH}_3$ and ^{11}CO for positron tomography, and $^{123}\text{I}^-$ for single photon tomography. The initial studies involved the use of simple organoborane precursors which led to the successful development of extensive, new isotope incorporation chemistry.^{1,2} The early studies validated the



feasibility of using reactive intermediates, such as the organoboranes, and led others to investigate organometallic agents based on mercury, tin, and silicon.³ In addition, the early boron studies led to the first syntheses of new agents which have since proven to be clinically important; these include (E)-17 α -[^{123}I] iodovinylestradiol, I,^{4,5} and [^{15}O]-butanol,



Other agents prepared via organoborane chemistry included [^{13}N]-putrescine, III,³ [^{125}I]-QNB, IV,⁶ and a wide variety of fatty acids such as V.⁷

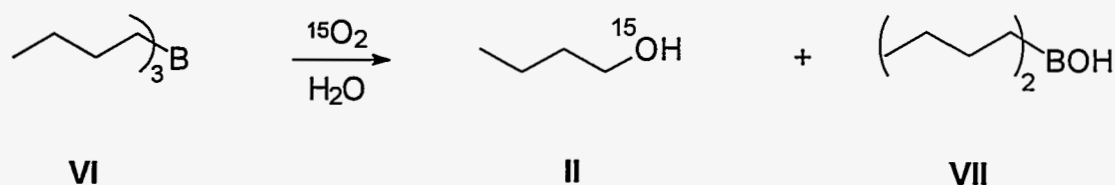


During the course of this research, it became apparent that the organometallic chemistry we, and others, had been developing would be even more useful in the clinical environment if the radiolabeled products could be readily separated from the undesired byproducts. We began to develop reactive organometallic reagents in which the metal

(often boron) was attached to a nonreactive organic or inorganic matrix such as polystyrene, silica or alumina.

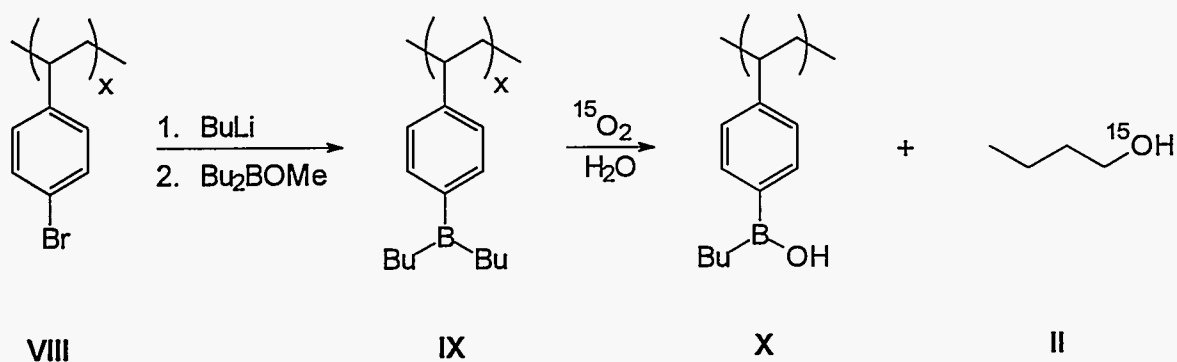
(a) Oxygen-15

We developed the synthesis of oxygen-15 labeled butanol, **II**, early in this D.O.E. research effort.⁸ [¹⁵O]-Butanol has since been found to be a valuable blood flow agent

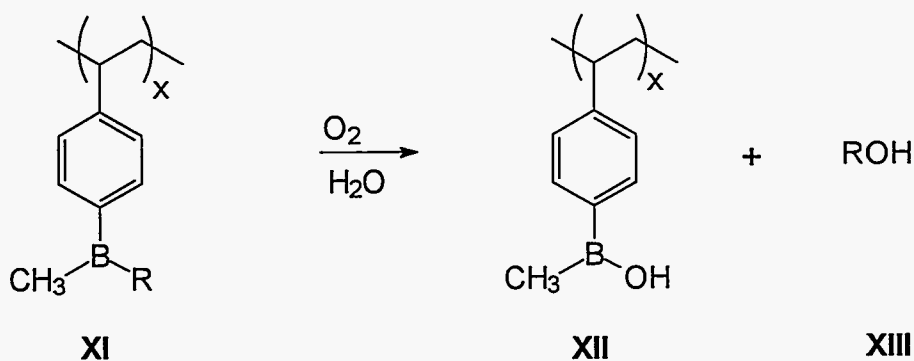


in humans.⁹ The synthesis of **II** in the clinic is hampered by the concurrent formation of the borinic acid byproduct **VII** which arises because only one of the three butyl groups in the original tributylborane is generally utilized. To obviate this problem, we and others have used silica gel,¹⁰ alumina,¹¹ and other solid surfaces¹² to trap the reaction byproducts.

We also developed a series of boronated organic and inorganic matrices in an effort to enhance the clinical utility of the oxygen-15 incorporation reaction. In the first approach, a dibutylboronated polystyrene, **IX**, was prepared as shown on the next page and its reactions with oxygen-15 gas were evaluated.¹³

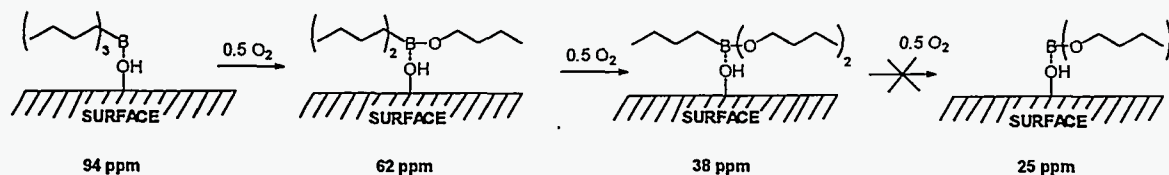


The yields of [^{15}O]-butanol using **IX** consistently exceeded 50% when corrected for decay, and the synthesis was complete in less than two minutes. We also prepared the methylboronated polystyrene derivative **XI** to take advantage of the fact that methyl groups have been shown to be less reactive than larger alkyl moieties.¹⁴ The yields of alcohol **XIII** approached 80%!¹⁵ The application of this chemistry to the synthesis of oxygen-15 labeled butanol is currently being evaluated.



As noted, a variety of inorganic matrices can also be utilized to adsorb the byproducts of the borane oxidation reaction. We have examined the ability of a series of hydroxylated surfaces including alumina, silica gel, cotton, cellulose acetate, celite, and polyvinyl alcohol to adsorb tributylborane and retain the byproducts of the oxidation

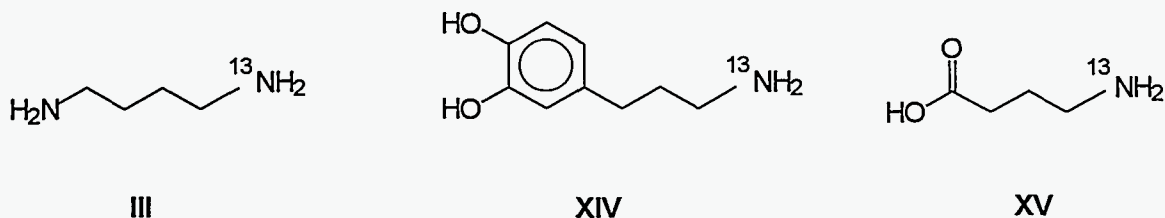
reaction. Alumina, silica gel, cotton, and celite all exhibited satisfactory behavior in that isolated yields of pure [^{15}O]-butanol routinely exceed 40% (corrected for decay). Through the use of boron-11 solid state NMR (SSNMR), we were able to verify that oxidation of tributylborane adsorbed to surfaces is incomplete even in the presence of excess oxygen.



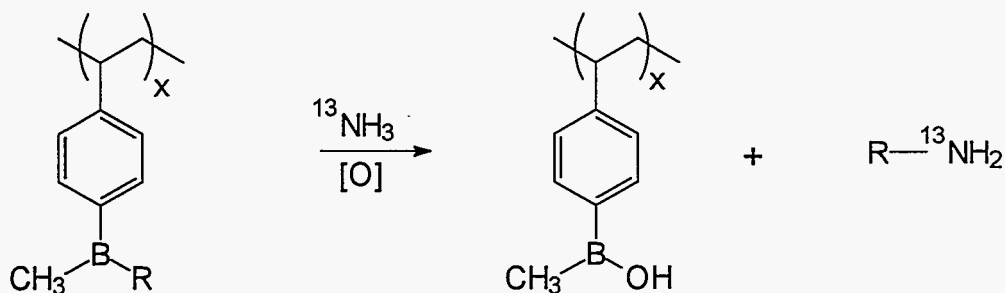
These results contrast those obtained in solution where it has been found that the tributylborane is completely oxidized.¹⁶

(b) Nitrogen-13

We also developed a series of polymeric borane derivatives which were used to prepare nitrogen-13 labeled amines of potential value in diagnostic Nuclear Medicine. Nitrogen-13 labeled putrescine, **III**, dopamine, **XIV**, and γ -aminobutyric acid, **XV**, were each prepared using modified borane polymers.



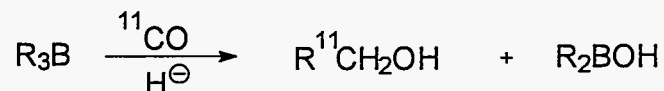
The polymers consisted of methylboronated polystyrene derivatives which were synthesized using chemistry which we developed in an effort to maximize reaction of the desired alkyl group.² The isolated yields of the nitrogen-13 labeled reagents, corrected



To EOB, ranged from 60 - 80%^{17,18} which surpassed the yields obtained in our initial solution studies.¹⁹

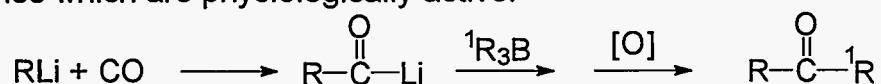
(c) Carbon-11

In the initial stages of this study, we demonstrated that organoboranes could be utilized to incorporate carbon-11 via reaction of trialkylboranes with carbon monoxide.²⁰



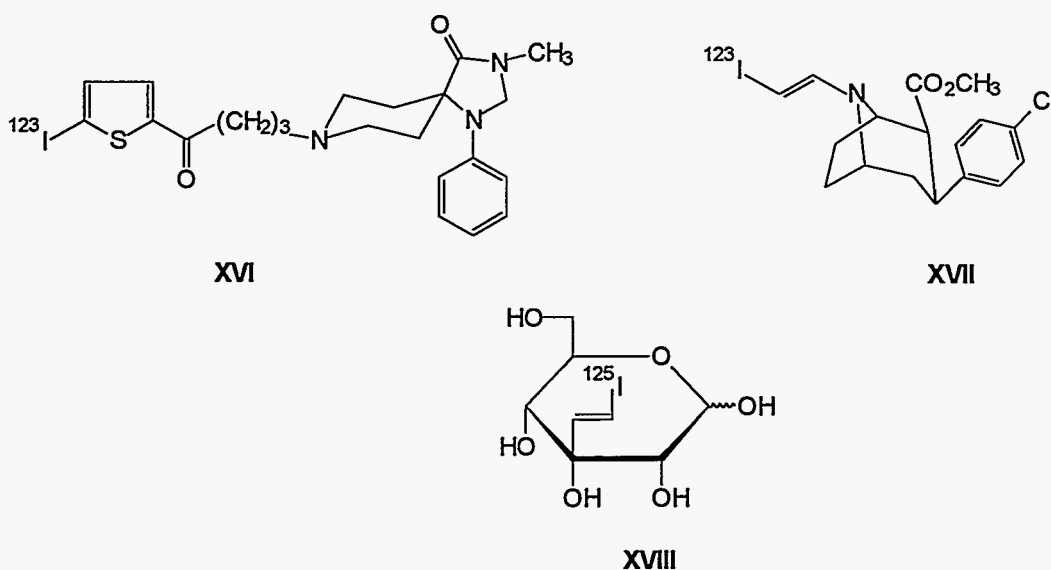
We used the reaction to prepare ketones and secondary alcohols but found it to be somewhat limited by the difficulty inherent in preparing unsymmetrical trialkylboranes.²¹

We are in the process of evaluating a new carbonylation reaction as a route to carbon-11 labeled ketones which are physiologically active.²²



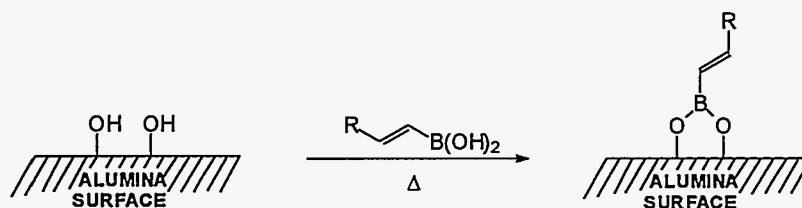
(d) Radiohalogenation

Our early studies on the incorporation of radiohalogens via borane chemistry^{23,24,25,26,27,28} provided a means for regiospecifically incorporating radiohalogens and sparked an interest in the use of organometallic reagents in nuclear medicine. This was especially true for our development of the vinyl iodide moiety as a means of stabilizing radioiodine *in vivo*.²⁹ During the current phase of our research, we prepared a number of radioiodinated reagents via organoborane and organotin reactions. The agents include iodine-123 labeled 3-*N*-methyl-5-iodo-2-thienyl-butyrophenone, **XVI**,³⁰ *N*-(3-iodopropen-2-yl)-2 β -carbomethoxy-3 β -(4-chlorophenyl)tropane, **XVII**, and 4-*O*-(*E*)-3-iodopropen-2-ylallose, **XVIII**.³¹

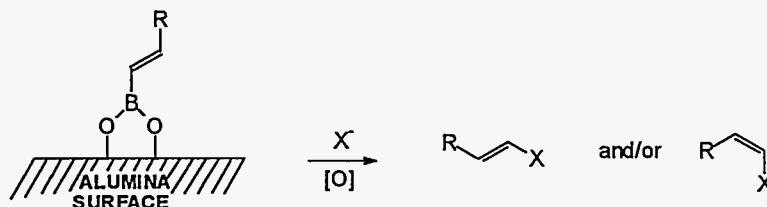


Two of these agents have proven to be physiologically active and are currently undergoing evaluation in animal models by our colleagues at Emory University (**XVII**) and the Australian National Science and Technology Organization (**XVI**). We are now focusing our attention on enhancing the utility of the isotope incorporation reactions through the use of reactive solid state matrices.

We are, for example, investigating the preparation of boronic ester derivatives of alumina which are prepared by dehydrating mixtures of boronic acids and aluminas. The studies are based on our earlier observations involving silica gel as the solid support.³²

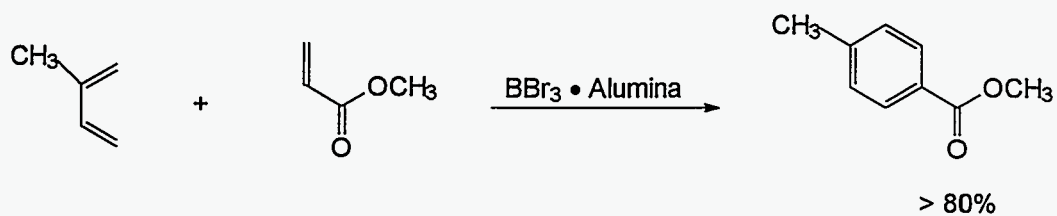


Some of the new materials have been halogenated to produce vinyl halogen derivatives.^{33,34,35,36} Interestingly, we have found that the regiochemistry of the vinyl halides appears to be dependent on the surface. A fact that could be significant in

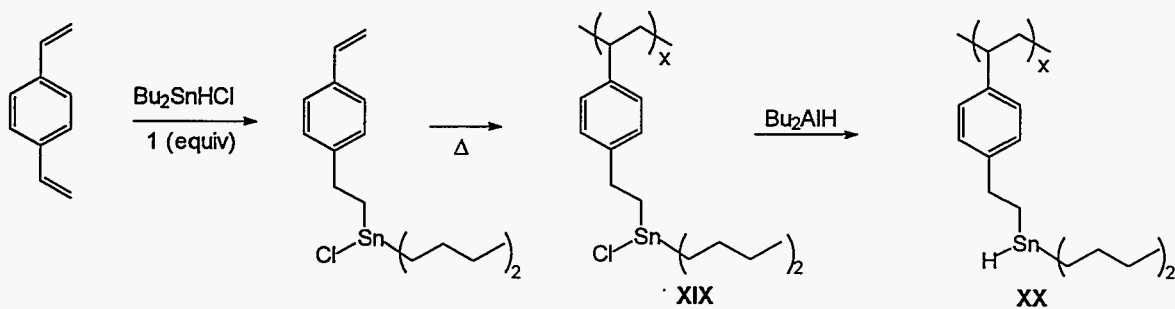


light of recent studies which indicate that the (*Z*)-vinyl iodides often demonstrate enhanced physiological activity.^{37,38} As anticipated, the boronic acid byproduct is retained on the alumina which simplifies isolation and purification of the desired products.

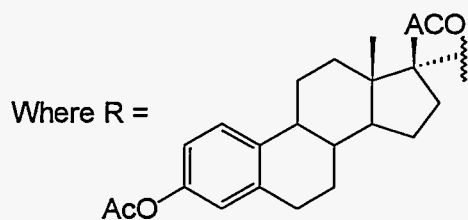
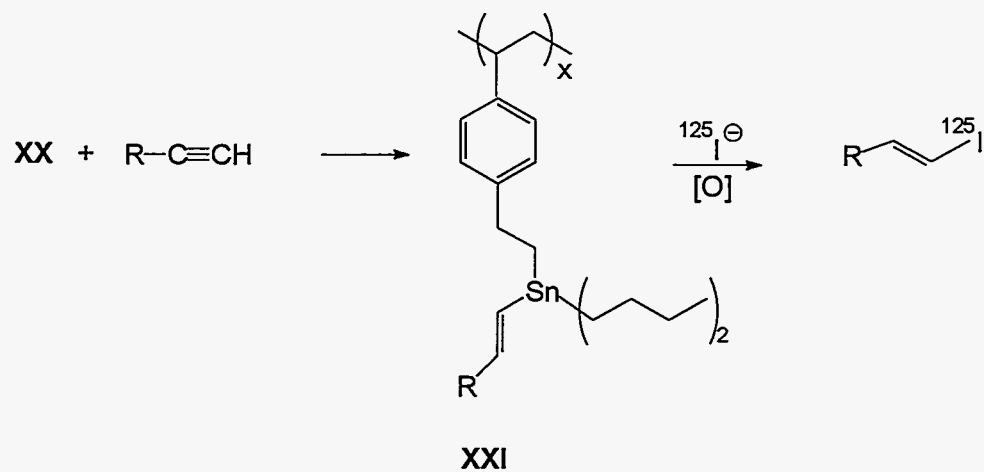
The coating of alumina with boron derivatives has led to some exciting discoveries. In addition to the change in regiochemistry of the vinyl iodide noted earlier, we have found that a number of the boronated aluminas have significant regiochemical effects on reactions such as the Diels-Alder reaction.³⁹ These results, shown on the next page, could prove valuable in our syntheses of radiopharmaceutical precursors.



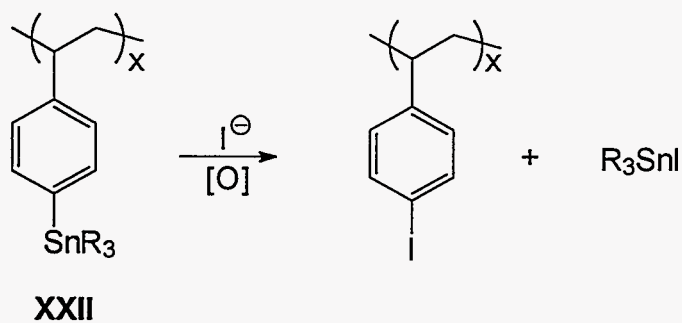
Our investigation of new iodination reactions also includes the preparation of polystyrene derivatives of tin,⁴⁰ **XX**. This chemistry often compliments that of boron.



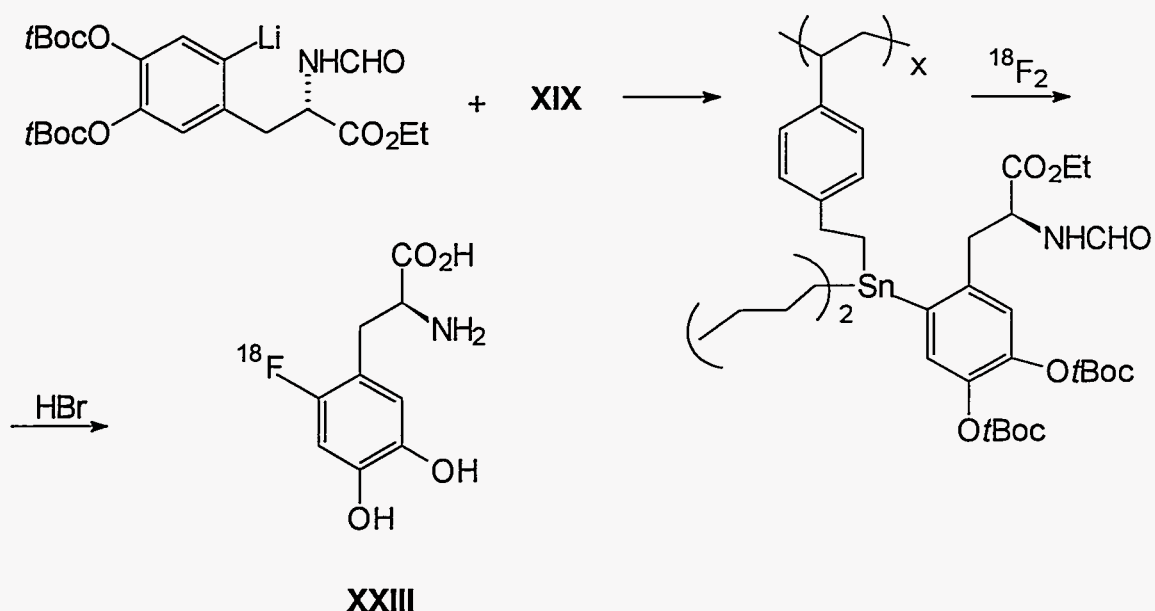
Polymer **XXI** (next page) was used to prepare a radioiodinated estradiol in excellent (47%) radiochemical yield⁴¹ via a stannylation-iodination sequence.



In earlier studies, Culbert and Hunter⁴² used simple stannylated polystyrenes to prepare aryl iodides but they discovered that destannylation of the polystyrene backbone occurs when the tin is directly attached to the polymer, as it is in **XXII**.



Our new polymers, prepared from **XIX**, obviate the problem. We believe that the new technology could be applied to an in-line automated synthesis of fluorine-18 labeled DOPA, **XXIII**, for use in our automated synthesis unit.⁴³



In summary, many of the goals outlined in the original proposal have been accomplished. New isotope incorporation routes for nitrogen-13, carbon-11, and oxygen-15 have been developed and are being used to prepare clinically useful radiopharmaceuticals. An investigation into new radiohalogen incorporation routes has been initiated but further studies are required before they are applicable to the clinical environment. These studies form the basis for our Renewal Proposal, **Section D**.

2. Students Trained

An important goal of the D.O.E. Nuclear Medicine Program at The University of Tennessee is to provide training for students (predoctoral and postdoctoral) in the scientific aspects of Nuclear Medicine. The academic nature of the program helps to insure the continued availability of scientists dedicated to the advancement of nuclear

medicine. Since 1993, ten postdoctoral, twenty graduate and ten undergraduate students have received support and training under the auspices of the D.O.E. program at The University of Tennessee:

(a) Postdoctoral

POSTDOC	AREA OF RESEARCH	TENURE
Murthy, Akula	Alzheimer Pharmaceuticals	6/94 - 12/96
Narayana, Chatla	Organic Reactions	6/1/89 - 5/31/96
Zhang, Zheng-Yu	Carbon-11 Synthesis	1/1/94 - 12/1/95
Collier, T. Lee	Automation of Radiopharmaceutical Synthesis	8/29/91 - 5/3/93
Shoup, Timothy	Oxidations Using Sodium Perborate	1991
Guindi, Laila H.	Boron Chemistry	1987 - 1990
Meng, Xian-Jun	Radiohalogenated Imaging Agents	1989 - 1990
Matur, Shyam	Radiolabeling	1985 - 1988
Varma, Rajender S.	Construction of Pharmaceutical Precursors	1986 - 1987
Goudgaon, Naganna	Boron-Prepharmaceuticals	1987

(b) Graduate Students

GRADUATE STUDENT	DEGREE/YEAR	TITLE OF THESIS
Moss, Thomas H.	M.S. -- 1986	Preparation of a Modified Gd-DTPA Complex: A Potential Contrast Agent for Magnetic Resonance Imaging
McCollum, Gary W.	Ph.D. -- 1986	The Synthesis of Alkylamines and Dialkylamines Via Organoboranes
Edwards, Robbie M.	M.S. -- 1986	Monophenylborane: Synthesis and Use in Hydroborations
Fabrikiewicz, Ann S.	Ph.D. -- 1987	"Reactions of Boranes With Carbonyl Compounds: An Investigation

GRADUATE STUDENT	DEGREE/YEAR	TITLE OF THESIS
Steward, Jay	M.S. -- 1987	The Addition of HI to UnSaturated Hydrocarbons Using I ₂ and Al ₂ O ₃
Bierer, Donald E.	Ph.D. -- 1988	Synthetic Approaches to Iodovinyl Amino Acids
Wang, Zhe	Ph.D. -- 1990	Synthesis of Amines Via Organoboranes
Davis, Mark A.	M.S. -- 1990	Magnetic Resonance In Aging: The Development and Evaluation of Several Gd-DTPA Derivatives for Use as MRI Contrast Enhancements Agents and the Application of Boron-11 Spectroscopy and Imaging For the Detection and Localization of a BNCT Agent <i>In-Vivo</i>
Sponholtz, William	M.S. -- 1991	The Addition of Iodine to Vinyl Boronic Acid Intermediates in the Presence of Alumina
Zippi, Elizabeth	Ph.D. -- 1991	Polymer Based Substrate for Potential Generation of Nitrogen-13 for Positron Emission Tomography
Lee, Lay C.	Ph.D. -- 1992	Studies of Organic Reactions
Waterhouse, Rikki	Ph.D. -- 1993	"Radioiodination Via Organometallics"
Bowers, Karla	M.S. -- 1993	"Polymer-Based Organoboranes"
Lambert, Steve	Ph.D. -- 1994	"Iodination Via Iodine Monochloride"
Green, James F.	Ph.D. -- 1994	"Polymer-Based Systems for Incorporating Short-Lived Isotopes"
Pace, R. David	Ph.D. -- 1994	"Crotylboration Reactions"
Vagle, Kurt	M.S. -- 1995	"Surface Reactions"
Maddox, John	Ph.D. -- 1995	"New Carbon-Carbon Bond Forming Reactions Via Organoboranes"
Marks, Ronald C.	Ph.D. -- 1995	"Thiophene-based Polymers"
Gotsick, Timothy	Ph.D. -- 1996	"Radiolabeled Opiate-Receptor Agents"

3. Bibliography of Publications Resulting from Research Sponsored by DOE: 1993-1995

(a) Journal Articles

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5. Kabalka, G. W.; Varma, R. S.; Gai, Y.-Z.; Baldwin, R. M. "A New Route to Iodine-Labeled N-Isopropyl Iodoamphetamine Via Organoboranes," *Tetrahedron Lett.* **1986**, *27*, 3843.
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(b) Published Abstracts

191 Abstracts have been published from 1986 to 1996.

(c) Invited Presentations

134 Invited presentations have been presented from 1986 to 1996.

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