UNTANGLING THE ENERGETICS AND DYNAMICS OF ELEMENTARY ATOMIC BORON REACTIONS

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The primary objectives of this proposal are to elucidate the energetics and dynamics of elementary reactions of ground state boron atoms (B) with
simple di-,tri-, and tetra atomic, nitrogen- and oxygen-bearing molecules. These closed shell molecules serve as prototype reaction partners
accessing the H_xBC_yN (x=0,1,2,3; y=0,1) and $H_xBC_yO_z$ (x=0,1,2; y=0,1; z=1,2) systems which are important in the fields of basic physical
chemistry (reaction dynamics), combustion chemistry, material sciences, chemical propulsion systems, physical organic chemistry, and chemical
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Untangling the Energetics and Dynamics of Elementary Atomic Boron Reactions

Final Report - AFOSR Grant FA9550-09-1-0177

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Table of Contents

1. Objectives

Probing elementary chemical processes in extreme environments presents a valuable approach to experimentally access the potential energy surfaces (PESs) of reaction intermediates and short lived species. Here, we focus on elementary processes involved in two of these extreme environments, i.e. organo boron chemistry and the interaction of ionizing radiation with (surface coated) polymers.

Regarding the boron chemistry, the objectives of this proposal were to elucidate the energetics and dynamics of elementary reactions of ground state boron atoms ($B(^2P_j)$) with simple nitrogen- and oxygen-bearing molecules by focusing on the key systems ammonia (NH_3) and hydrogen cyanide (HCN) as well as water (H_2O), oxygen (O_2), and carbon dioxide (CO_2), respectively. The closed shell molecules serve as prototype reaction partners to access the H_xBC_yN (x=0,1,2,3; y=0,1) and $H_xBC_yO_z$ (x=0,1,2; y=0,1; z=1,2) potential energy surfaces which are important in the fields of basic physical chemistry (reaction dynamics), combustion chemistry, material sciences, chemical propulsion systems, physical organic chemistry, and chemical vapor deposition processes (boron-nitride films, ternary BCN compounds). The experiments are pooled together with electronic structure calculations to verify the elucidated reaction mechanisms theoretically; this will ultimately bridge the understanding of reactive scattering processes involving small boron-bearing systems via quantum chemical methods and experiments.

The polymer add-on project investigated the effects of ionizing radiation with (surface coated) polymers (Kapton, Teflon, PE, PMMA) utilizing a surface scattering machine. These experiments helped to untangle the stability of polymers together with their coatings toward space weathering. The latter can originate from multiple components: galactic cosmic ray particles (GCRs), solar wind particles (mainly 1 keV H^+, 4 keV He^+, and ~10 keV O^+), and mono energetic UV/VUV photons up to 10.2 eV (Lyman Alpha). Recall that the effect of space weathering is important to untangle the stability of polymers in space missions, light materials to be utilized for permanent settlements on the Moon and Mars, and also to elucidate protective coating layers of these polymers in Low Earth Orbit (LEO) spacecrafts. These experiments were conducted in collaboration Steven J. Sibener (U Chicago); SJS prepared thin polymer films (100 – 200 nm) on highly polished silver wafers via spin coating. Calculations utilizing the TRIM and CASINO codes suggest that although Al_2O_3 coatings of a few nm thickness protect polymers from hyperthermal oxygen atoms (a damaging component of non-coated polymers in LEO spacecrafts), these coatings can be penetrated easily by GCRs and solar wind particles.

2. Relevance to AFOSR Sponsored Research

This proposed research is in line with the strategic plan of the AFOSR. Our research fits within the *Molecular Dynamics* program. Here, the objectives of this program are to understand, predict, and control the chemical reactivity and flow of energy in molecules. These data can be utilized than to understand weakly ionized plasmas, propulsion systems, and the elementary steps involved in the synthesis of materials related to boron-nitride films and ternary BCN compounds on the molecular level (cross links with the *Ceramic and Nonmetallic Material* program). This provides solid scientific background for current and future Air Force-related applications of ceramics, ceramic-matrix composites (CMCs), and carbon-based composites in hypersonic aircrafts and space structures. Due to the collaborations with theoreticians (Mebel/Newhouse), our research holds also strong links to the *Theoretical Chemistry* program. Further, one of the prime interests of the AFOSR program is to sponsor combustion related research which provides data and procedures to enable the

development of reliable chemical propulsion systems. The latter should be able to quantitatively predict the performance of combustion and propellant systems to minimize the emission of unwanted by-products and to maximize the propulsion efficiency. However, all combustion models require crucial input parameters such as the knowledge of rate constants of the chemical reactions over a wide temperature and pressure range, the identification of reaction intermediates, which either form products, are stabilized via three body reactions, react with other molecules, or decay back to the reactants, and the assignment of the primary reaction products together with branching ratios. At present this includes AFOSR sponsored research to understand, predict, and to control rocket propulsion systems and the reactivity, energy flow in molecular systems, an also the interest in steady state plasmas. Here, our research initiative connects to the *Combustion and Diagnostics* and *Space Power and Propulsion* programs aimed to untangle the chemistry of reacting flows of chemical propulsion systems in, for example, chemical rockets and ramjet engines. Based on these data, the ultimate goal is to predict and to eliminate combustion instabilities, to minimize the emission of unwanted by-products, and to optimize the efficiency of chemically-based propulsion systems.

3.   Accomplishments

Our research program has been very productive. During the AFOSR project FA9550-09-1-0177, the PI has focused on two extreme environments: i) elementary reactions of boron-bearing species and implications to combustion chemistry, CVD processes, and rocket propulsion systems (2.1) and ii) the interaction of ionizing radiation in form of energetic electrons with polymers used in space crafts (2.2). This has resulted in ten publications so far (**P1**-**P9**) with publications **P2** and **P7** showcasing our AFOSR-sponsored research on cover pages of Chem. Rev. and Phys. Chem. Chem. Phys..

3.1.   Crossed Molecular Beams Studies
3.1.1. Crossed Molecular Beams Machine Modifications

To characterize the spin-orbit and rovibrational states of boron-bearing reactants, we incorporated laser induced fluorescence (LIF) detection within our crossed beams machine. We would like to emphasize that the LIF setup can be operated in concert with the TOF quadrupole mass spectrometer of the detector of the crossed beams machine. This enables us to simultaneously characterize the peak velocity and speed ratio (velocity spread) [TOF quadrupole mass spectrometer] and also the spin-orbit / rovibrational states of distinct segments of the pulsed supersonic beam, which contains the boron-bearing open shell reactants. This setup was operated successfully for supersonic beams of ground state boron atoms, $^{11}B(^2P)$, and boron monoxide radicals, $^{11}BO(X^2\Sigma^+)$.

For example, a section corresponding to a peak velocity of 2200 ± 20 ms^{-1} of the supersonic beam was characterized via laser induced fluorescence (LIF) to evaluate the population of the two spin-orbit states in ground state boron: $J = 1/2$ ($^2P_{1/2}$) and $J = 3/2$ ($^2P_{3/2}$, 15.254 cm^{-1}). The 5 µJ frequency doubled output of Lambda Physik Scanmate dye laser saturated the $^2P - {}^2S$ transitions near 249.75 nm and 249.85 nm for $J = 1/2$ and $J = 3/2$, respectively. The interference filter centered at 250 nm with a 10 nm bandwidth placed in front of the photomultiplier tube discriminated against fluorescence on the surfaces inside the chamber. In saturation mode the ratio of state populations is inferred directly from the ratio of the line intensities. Statistics on the ratios for the four individual scans yield an average J = 1/2 to J = 3/2 ratio of 1.6 ± 0.2 corresponding to a temperature $T = 18.5 \pm 2.0$ K.

4

With respect to boron monoxide, the electronic ground state of boron monoxide was probed via the $A\,^2\Pi - X\,^2\Sigma^+$ (0,0) transition at ~425 nm by the pulsed 10 μJ output of a Lambda Physik Scanmate dye laser pumped by the third harmonic of an integrated Nd:YAG laser operating at 10 Hz with an output power of 50 mJ per pulse. The fluorescence was detected by a photomultiplier tube (PMT) filtered by a Schott color glass long-pass GG-495 filter for (2,0) fluorescence detection and scattered detection laser light suppression. The signal was then amplified and filtered by a high pass filter prior to feeding into a digital oscilloscope interfaced to a computer for data collecting and processing. Note that boron monoxide radicals are exclusively in the ground electronic state, $X^2\Sigma^+$, by the time they reach the interaction region. The radiative lifetime of the first excited electronic state, $A\,^2\Pi_{\pm 1/2}$, of boron monoxide of 1.8 μs is shorter than the flight time from the ablation region to intersection point of a few tens of μs. For instance, the section of the boron monoxide radical beam corresponding to a peak velocity of 1162 ± 12 ms^{-1} was characterized by a rotational temperature of 250 ± 40 K which yields an upper limit of internal energy of the supersonic boron monoxide beam of about 2.0 kJmol^{-1}. No vibrationally excited of boron monoxide radicals were detected.

3.1.2. Reaction Dynamics of Atomic Boron (B(^{2}P$_j$)) with Hydrocarbons

We completed our research on elementary reactions of ground state boron atoms with unsaturated hydrocarbons under single collision conditions with the reaction of ground state boron atoms with diacetylene (C$_4$H$_2$(X$^1\Sigma_g^+$)) (**P1**). This lead to the very first identification of a cyclic ^{11}BC$_4$H(X^{1}A$^{'}$) isomer; electronic structure calculations suggested that a linear HCCBCC(X$^1\Sigma^+$) isomer should also be formed. We also compiled the generalized concepts on the chemical dynamics of boron atom reactions with unsaturated hydrocarbons under single collision conditions in a high-impact review paper in Chem. Rev. (**P2**). Briefly, our studies concluded that: i) the reactions of boron atoms with unsaturated hydrocarbons proceeded via indirect scattering dynamics through complex formation and were initiated by barrier-less additions of atomic boron to the π-electron density of the hydrocarbon reactants (in case of the boron-D6-benzene system, we were able to observe the ^{11}BC$_6$D$_6$ adduct); ii) no insertion processes into carbon-hydrogen single bonds were observed; iii) the initial collision complexes isomerized via successive ring closure/ring opening and/or hydrogen migration processes; iv) the doublet reaction intermediates fragmented via atomic hydrogen loss pathways forming exclusively singlet closed shell reaction products; v) the overall reaction energies to form organo-boron molecules from the separated reactants were exoergic by up to 140 kJmol^{-1}; vi) no molecular hydrogen elimination channel was open; vii) with the exception of the formation of c-BC$_2$H(X^{2}A') and HBCC(X$^1\Sigma^+$) (boron-acetylene reaction), the exit transition states were found to be rather tight and located up to 30 kJmol^{-1} above the separated products.

3.1.3. Reaction Dynamics of Atomic Boron (B(^{2}P$_j$)) with Nitrogen-bearing Molecules

We investigated the energetics and dynamics of elementary reactions of ground state boron atoms (B(^{2}P$_j$)) with simple nitrogen-bearing molecules under single collision conditions by focusing on the key systems ammonia (NH$_3$) (**P3**) and hydrogen cyanide (HCN) (**P4**). The closed shell molecules serve as prototype reaction partners to access the H$_x$BC$_y$N (x=0,1,2,3; y=0,1) potential energy surfaces which is important in the fields of reaction dynamics, combustion chemistry, material sciences, chemical propulsion systems, physical organic chemistry, and chemical vapor deposition processes (boron-nitride films, ternary BCN compounds). Electronic structure calculations on these systems were conducted in collaboration with the groups of Bartlett (University of Florida), Mebel (Florida International University), and Chang (Taiwan).

5

Our studies suggested that the reaction of atomic boron with ammonia was initiated by a barrier-less addition of the boron atom to the non-bonding electron pair of the nitrogen atom forming a weakly bound BNH_3 collision complex. This intermediate underwent a hydrogen shift to a doublet $HBNH_2$ radical which decomposed via atomic hydrogen loss to form the imidoborane ($HBNH(X^1\Sigma^+)$) molecule – an isoelectronic species of acetylene ($HCCH(X^1\Sigma_g^+)$) (Figure 1). These data show that crossed beam studies can be applied not only to study the formation of unstable transient species such as the imidoborane (HBNH) molecule, but also that the underlying mechanism can be utilized to predict the chemical reactivity of isoelectronic systems as elucidated in our paper for the carbon – methyl radical – two open shell species – which are very difficult to study experimentally (**P3**).

Considering the reaction of boron atoms with hydrogen cyanide, we identified the linear boronisocyanide species, ($BNC(X^1\Sigma^+)$) (Figure 1). This molecule presents the simplest tri-atomic molecule with three distinct, neighboring main group atoms of the second row of the periodic table of the elements: boron, carbon, and nitrogen. This makes boronisocyanide a crucial benchmark system to understand the chemical bonding and the electronic structure of small molecules, in particular when compared to the isoelectronic tricarbon molecule, ($CCC(X^1\Sigma_g^+)$). We also showed computationally that higher energy isomers like the hitherto unnoticed, ring-strained cyclic $BNC(X^3A')$ structure, which is isoelectronic to the triplet, cyclic tricarbon molecule, ($C_3(X^3A_2')$), do exist as local minima. Our studies presents the first directed synthesis and observation of gas phase boronisocyanide providing a doorway for further fundamental studies on one of the simplest triatomic molecules composed solely of group III – V elements (**P4**).
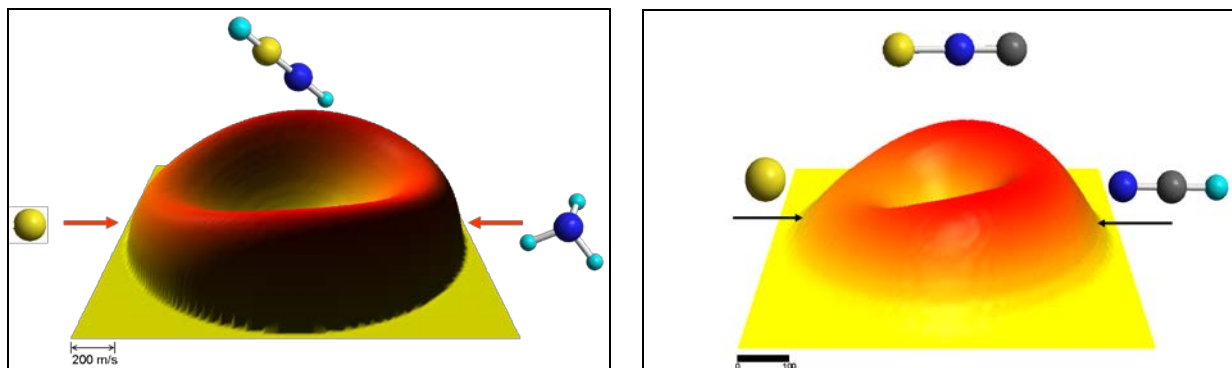

Figure 1. Flux contour map of the imidoborane molecule (HNBH) (left) and boronisocyanide (BNC) (right) formed in the reaction of atomic boron, $^{11}B(^2P_j)$, with ammonia, $NH_3(X^1A_1)$, and hydrogen cyanide ($HCN(X^1\Sigma^+)$) at collision energies of 20.5 kJmol^{-1} and 29.2 kJmol^{-1}, respectively.

3.1.4. Reaction Dynamics of Boron Monoxide Radicals (^{11}BO; $X^2\Sigma^+$) with Hydrocarbons

In preparation to set up the crossed beams machine for the reactions of ground state boron the boron monoxide radical (BO; $X^2\Sigma^+$), we switched our detection system to laser induced fluorescence. Here, the boron monoxide radical (BO; $X^2\Sigma^+$) was generated via laser ablation of boron atoms utilizing oxygen (O_2), carbon dioxide (CO_2), methanol (CH_3OH), and water (H_2O) as seeding and reactant gases (**P5**). As a matter of fact, the boron monoxide radical (^{11}BO; $X^2\Sigma^+$) beam was found to be of sufficient high intensity to conduct a crossed molecular beams reaction with acetylene (C_2H_2, $X^1\Sigma_g^+$) at a collision energy of 13 kJmol^{-1} leading via indirect scattering dynamics to the hitherto unobserved linear $HCC^{11}BO(X^1\Sigma^+)$ product. The overall reaction to form $HCC^{11}BO(X^1\Sigma^+)$ plus atomic hydrogen from the separated reactants was determined to be exoergic by of 62 ± 8 kJmol^{-1} (**P6**).

6

3.2. Polymers

Polymer films, commonly used in spacecraft design to coat exterior surfaces and sensitive equipment, were selected to perform laboratory simulation experiments on the interaction of polymers with galactic cosmic ray particles (**P7**, **P8, P9**).

Mechanisms of the electron-induced degradation of polymers utilized in aerospace applications (polyethylene (PE), polytetrafluoroethylene (PTFE), polystyrene (PS), PMMA, Kapton) were examined over a temperature range of 10 K to 300 K at ultra-high vacuum conditions (~ 10^{-11} Torr) (**P7**, **P8**). These processes simulate the interaction of energetic secondary electrons generated in the track of galactic cosmic ray particles in the near-Earth space environment with polymer material. The chemical alterations were monitored quantitatively by Fourier-transform infrared spectroscopy, mass spectrometry, and *via* UV-VIS spectroscopy. These data yielded important information on the temperature dependent kinetics on the formation of, for instance, benzene (C_6H_6) production in PS, fluorinated *trans*-vinylene (–CF=CF–) and terminal vinyl (–CF=CF$_2$) groups in PTFE together with molecular hydrogen release in PE and PS (Figure 2). Our degradation yields were found to be typically two to three orders of magnitude less than those previously reported in the literature.

We also finally, we have finalized the experimental studies of the interaction of spin-coated polyethylene with energetic oxygen ions as present in the magnetospheres of Earth, Jupiter, and Saturn (**P9**) in collaboration with Prof Sibener (Chicago) (Figure 2).
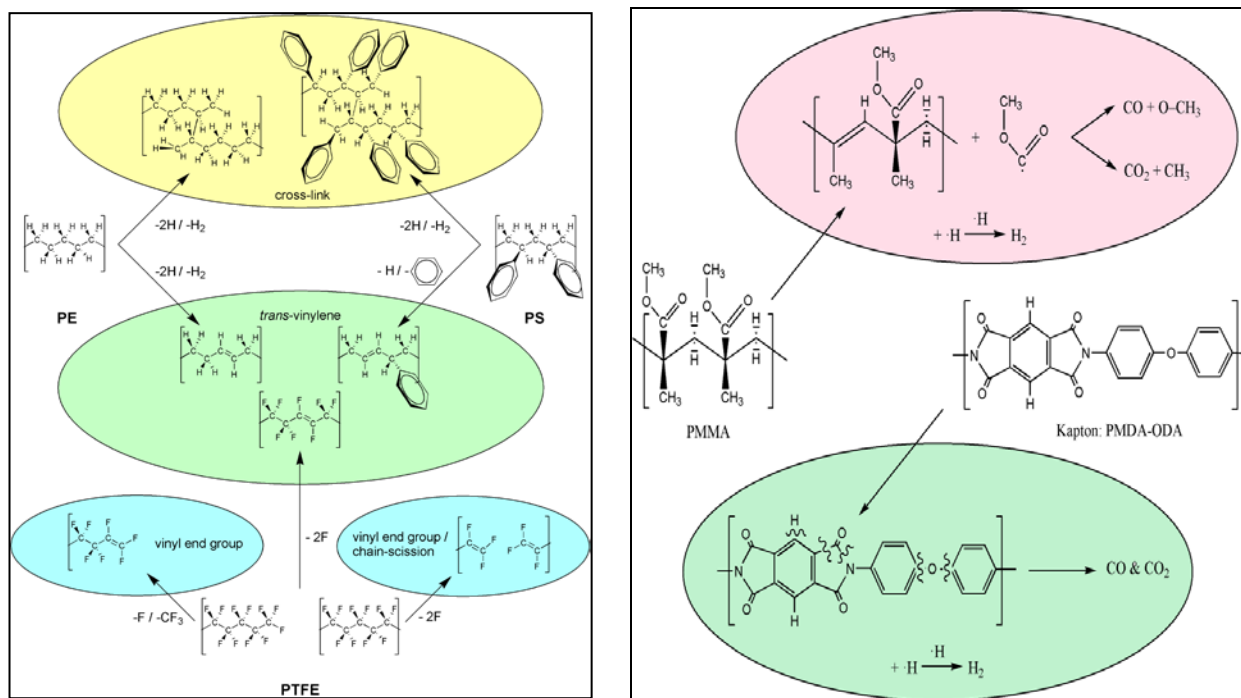

Figure 2. Summary of the degradation processes in the interaction of energetic electrons with five polymers under ultra-high vacuum conditions.

4. Personnel Associated with the Research Effort

During this reporting period, postdoctoral fellows *Drs Maksyutenko* and *Ennis* were supported on the AFOSR grant. Partial support was also given to Dr. Zhang. Finally, *Edwin Kawamura* (Staff Scientist, University of Hawaii) was available for this project to build electronic equipment and also to maintain pumps and home-built electronic equipment

5. Publications

P1: P. Maksyutenko, F. Zhang, R.I. Kaiser, Untangling the Chemical Dynamics of the Reaction of Boron Atoms, $^{11}B(^2P_j)$, with Diacetylene, $C_4H_2(X^1\Sigma_g^+)$ – A Crossed Molecular Beams and Ab Initio Study. JPCA 114, 10936-10943 (2010).

P2: N. Balucani, F. Zhang, R. I. Kaiser, Elementary Reactions of Boron Atoms with Hydrocarbons - Toward the Formation of Organo-Boron Compounds. Chem. Rev. 110, 5107-5127 (2010).

P3: F. Zhang, P. Maksyutenko, R.I. Kaiser, A.M. Mebel, A. Gregusova, S. Ajith Perera, R.J. Bartlett Gas Phase Synthesis of the Imidoborane Molecule (HNBH) – A Combined Crossed Beam and Theoretical Study. JPCA 114, 12148–12154 (2010).

P4: B. Jones, P. Matsyutenko, A.H.H. Chang, R.I. Kaiser, Crossed Molecular Beam Study on the Ground State Reaction of Atomic Boron $[B(^2P_j)]$ with Hydrogen Cyanide $[HCN(X^1\Sigma^+)]$. The Journal of Physical Chemistry A 114, 8999-9006 (2010).

P5: P. Maksyutenko, D.S.N. Parker, F. Zhang, R.I. Kaiser, An LIF Characterization of Supersonic BO and CN Radical Sources for Crossed Beam Studies. Reviews of Scientific Instruments 82, 083107/1-7 (2011).

P6: D.S.N. Parker, F. Zhang, P. Maksyutenko, R. I. Kaiser, A crossed beam and ab initio investigation of the reaction between boron monoxide $(^{11}BO;X\ ^2\Sigma^+)$ and acetylene $(C_2H_2;X\ ^1\Sigma_g^+)$. Physical Chemistry Chemical Physics 13, 8560-8570 (2011).

P7: C.P. Ennis, R.I. Kaiser, Mechanistical Studies on the Electron-Induced Degradation of Polymers: Polyethylene, Teflon, and Polystyrene. Physical Chemistry Chemical Physics 12, 14884-14901 (2010).

P8: C.P. Ennis, R.I. Kaiser, Mechanistical Studies on the Electron-Induced Degradation of Poly-methylmethacrylate and Kapton. Physical Chemistry Chemical Physics 12, 14902-14915 (2010).

P9: C. Ennis, H. Yuan, S. Sibener, R.I. Kaiser, On the Chemical Processing of Hydrocarbon Surfaces by Fast Oxygen Ions. Physical Chemistry Chemical Physics 13, 17870-17884 (2011).
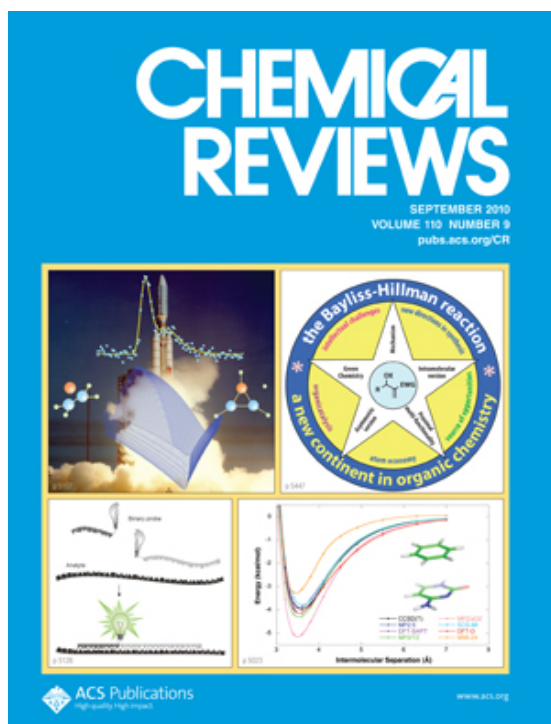
6. Invention Disclosures and Patents Granted

There is nothing to report here.

7. Honors and Awards

In 2011, Ralf I. Kaiser was elected Fellow of the Royal Society of Chemistry (UK).

In 2012, Ralf I. Kaiser was elected Fellow of the American Physical Society "*For pioneering experimental investigations of the chemical evolution of the Solar System and the Interstellar medium using crossed molecular beams and surface scattering to probe the underlying phenomena on the most fundamental, microscopic level*"

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Ralf I Kaiser

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Abstract

The primary objectives of this proposal are to elucidate the energetics and dynamics of elementary reactions of ground state boron atoms (B) with simple di-,tri-, and tetra atomic, nitrogen- and oxygen-bearing molecules. These closed shell molecules serve as prototype reaction partners accessing the HxBCyN (x=0,1,2,3; y=0,1) and HxBCyOz (x=0,1,2; y=0,1; z=1,2) systems which are important in the fields of basic physical chemistry (reaction dynamics), combustion chemistry, material sciences, chemical propulsion systems, physical organic chemistry, and chemical vapor deposition processes (boron-nitride films, ternary BCN compounds).

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Program Officer

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