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New Insights into Catalytic Sites: Characterization of Spectroscopy and Reactivity of Metal Oxide Clusters with Anion Slow Electron Velocity-Map Imaging

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14. ABSTRACT

One of the outstanding problems in all of physical chemistry is to understand reactive sites in heterogeneous catalysts, many of which comprise complex transition metal oxides. Size-selected transition metal oxide clusters can serve as tractable model systems for the analogous bulk catalyst. This research program has focused on developing novel spectroscopic probes of such clusters. During the last grant period, slow electron velocity-map imaging of cryogenically cooled anions (cryo-SEVI) was implemented and shown to be a qualitative advance in the spectroscopic investigation of transition metal oxide clusters, yielding high resolution (~2cm^{-}1) photoelectron spectra from which one can elucidate structural and bonding motifs of the anions and the neutrals generated by photodetachment. There has been a particular focus on polymetallic clusters such as Ti2O4, Zr2O4, and Fe3O,since these species have largely been spectroscopically intractable using more conventional methods. Additional experiments based on infrared multiple-photon dissociation of cryo-cooled anions have focused on complex hydrated clusters such as $NO3^{-}$(HNO3)m(H2O)n and $HSO4^{-}$(H2SO4)m(H2O)n that are important in atmospheric chemistry and aerosol formation.

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2. OBJECTIVES

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3. STATUS OF EFFORT

Research during the previous grant period can be roughly divided into four components. Initially, the spectroscopy and energetics of several radicals, clusters, and transition state species were investigated using anion slow-electron velocity-map imaging (SEVI). This was followed by a major modification to the instrument, in which ion trapping and cryogenic cooling techniques were incorporated. These modifications enabled the acquisition of well-resolved cryo-SEVI spectra for a series of transition metal oxide clusters comprising up to three transition metal atoms, work that provides the foundation for the primary proposed research direction in this proposal. Finally, our very productive collaboration with Prof. Knut Asmis on the infrared spectroscopy of hydrated anions has continued. Key results are summarized below.

4. ACCOMPLISHMENTS/NEW FINDINGS

1) Spectroscopy of radicals, clusters, and transition states

SEVI is a powerful tool for investigating the spectroscopy and dynamics of radicals, clusters, and transition states.[1] Over the years, in our group and elsewhere, it has been demonstrated that photodetachment of mass-selected negative ions provides an elegant means for investigating these reactive and transient species.[2-4] Even before ion trapping and cooling was incorporated into the instrument, the additional resolution provided by SEVI compared to conventional photoelectron spectroscopy enabled us to uncover new spectroscopic and dynamical features associated with these species. Specifically, we characterized the *n*-methylvinoxy,[5] phenoxy and thiophenoxy,[6] and propadienylidene[7] radicals through SEVI spectroscopy of the corresponding anions. In addition, we measured SEVI spectra of the open-shell complexes RgS^-,[8] and characterized the transition state region of the F + H_2 and F + CH_4 reactions via SEVI of the appropriate precursor anions.[9]

The SEVI spectrum of the *n*-methylvinoxide anion probed the effects of multiple isomers and vibronic coupling between close-lying states of the *n*-methylvinoxy radical.[5] Transitions between the $\tilde{X}\,^1A'$ anion ground electronic state and the radical $\tilde{X}\,^2A''$ and $\tilde{A}\,^2A'$ states were observed. The major features in the spectra were attributed to transitions involving the lower energy *cis* conformers of the anion and neutral, while the higher energy *trans* conformers contribute only a single small peak. Franck-Condon simulations of the $\tilde{X}\,^2A'' \leftarrow \tilde{X}\,^1A'$ and $\tilde{A}\,^2A' \leftarrow \tilde{X}\,^1A'$ transitions were performed to assign vibrational structure in the spectrum, and to aid in identifying peaks in the *cis*-*n*-methylvinoxy $\tilde{X}\,^2A''$ band that occur only through vibronic coupling. Our SEVI spectra of the phenoxide and thiophenoxide anions revealed vibrationally-resolved transitions to the $\tilde{X}\,^2B_1$ state of each radical species.[6] Photodetachment to the $\tilde{A}\,^2B_2$ excited state of the thiophenoxy radical, a fully allowed transition in photoelectron spectroscopy, was also well-resolved, and yielded the first accurate term energy for this state.

Clusters comprising open-shell atoms or molecules pose a particular challenge to both theory and experiment because multiple close-lying electronic states associated with the open-shell species are typically coupled and split within the cluster. Photodetachment of the corresponding anion has also proved to be a powerful spectroscopic probe of both the anionic and neutral electronic and vibrational levels of these systems.[10, 11] In this vein, we have measured SEVI spectra of RgS^- (Rg = Ne, Ar, Kr) anions.[8] The NeS^- results represent the first observation and characterization of an anionic Ne complex. Numerous well-resolved transitions were observed for each species, showing an increasing perturbation of the fine-structure associated with S^- photodetachment with increasing mass of the Rg atom. New interaction potentials for the RgS anion and neutral complexes were calculated that allowed us to simulate the SEVI spectra with high accuracy and to assign its resolved features.

The $F + H_2 \rightarrow FH + H$ reaction and its various isotopologues have been extensively studied as the quintessential bimolecular reaction. The $F + CH_4 \rightarrow FH + CH_3$ reaction has evolved into a benchmark polyatomic reaction and provides insight into how additional degrees of vibrational freedom affect chemical reaction dynamics. Negative ion photoelectron spectroscopy complements scattering experiments on these reactions by directly accessing the transition state region via photodetachment of an anion with appropriate geometry,[12] in this case FH_2^- and $F^-(CH_4)$ anions. We have measured SEVI spectra of both anions to obtain significantly improved results for both systems compared to previously published work.[13, 14] A small peak in the para-FH_2^- spectrum was identified, matching simulations of a product resonance associated with HF(v'=3). SEVI spectra of the $^2P_{3/2}$ bands of FCH_4^- and FCD_4^- show extended fine structure from transitions to the entrance valley van der Waals region and the reactant side of the $F + CH_4$ transition state region. Much of this structure is attributed to bending or hindered rotation of the methane moiety and may be a spectroscopic signature of reactive resonances.

2) Modifications of the SEVI instrument

For atomic systems, the energy resolution of SEVI is about 2.5 cm^{-1}, a considerable improvement over anion PES in which the resolution is generally 100 cm^{-1} at best. However, peak widths in the SEVI spectra of molecular anions and clusters have typically been 20-25 cm^{-1},[15] considerably broader than the spectra of atomic systems. In the original version of the SEVI experiment,[16] ions were produced by expanding an appropriate gas mixture into vacuum with a pulsed solenoid valve. Anions were created from this expansion by using either an electric discharge or by electron attachment from a pulsed ring anode. In this production scheme, ions are cooled in the free jet expansion after the source, but the efficacy of this cooling, particularly for vibrational degrees of freedom, varies considerably between systems. Incomplete vibrational and rotational cooling results in spectral congestion that limits the effective resolution of SEVI through a combination of unresolved rotational contours, hot bands originating from excited anion vibrational levels, and unresolved sequence bands.

To eliminate this problem, the SEVI instrument has been extensively modified,[17] and now includes radio-frequency (rf) ion guides and an ion trap with buffer gas cooling so that the anions can be cooled to the greatest extent possible prior to spectroscopic investigation. The new version of the apparatus is shown in Fig. 1. Negative ions are generated in a pulsed source via ionization or laser ablation. They pass through an rf ion guide and are mass-selected in a quadrupole mass spectrometer. They are then injected into an rf-octupole trap, and cooled by collisions with a low pressure buffer gas in contact with a cryostat held at 5 K. Best results were found for a mixture of 80% He/20% H_2, which gave significantly better vibrational cooling than pure He at the same pressure and temperature.[18] The anions are extracted from the trap into a time-of-flight mass spectrometer and photodetached. The resulting photoelectrons are energy-analyzed using SEVI.

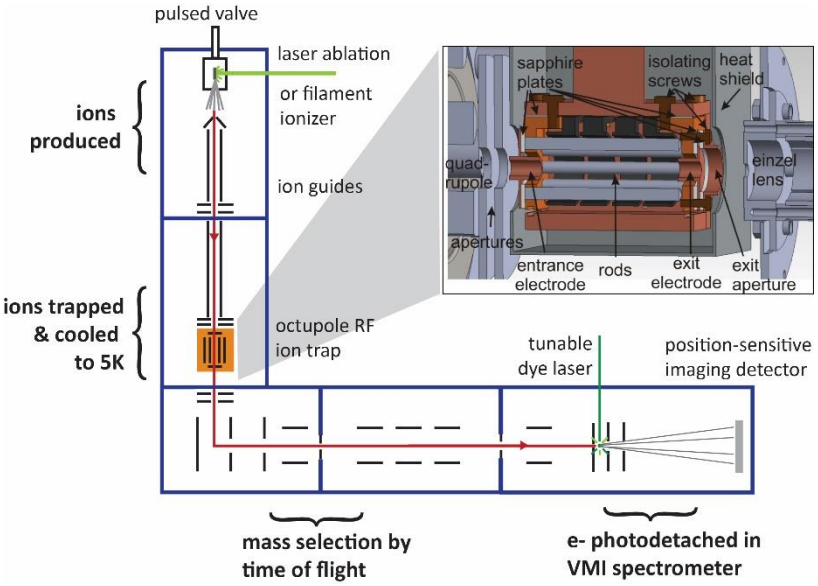

Figure 1. Schematic of cryo-SEVI instrument

As a first test of the new instrument, we measured the cryo-SEVI spectrum of the C_5^- anion and compared it to the SEVI spectrum of anions produced in free jet expansion with no additional cooling.[17, 19] The results are shown in Fig. 2. The two peaks arise from the two spin-orbit levels of C_5^-, which has a $^2\Pi$ ground state, and are split by only 25 cm^{-1}. Clearly, the population of the spin-orbit excited state (peak 2) is much lower in the spectrum of the cooled anions; from the relative peak heights, we extracted a temperature of 10 K for those anions, as opposed to 60 K for anions with no cryo-cooling. In addition, the peaks are substantially narrower for the cooled anions: 4-6 cm^{-1} compared to 20-25 cm^{-1}. Hence, trapping and cooling results in an internal temperature only slightly warmer than the cryostat temperature, and sub-meV peak widths as a result of reduced spectral congestion. We have thus achieved our

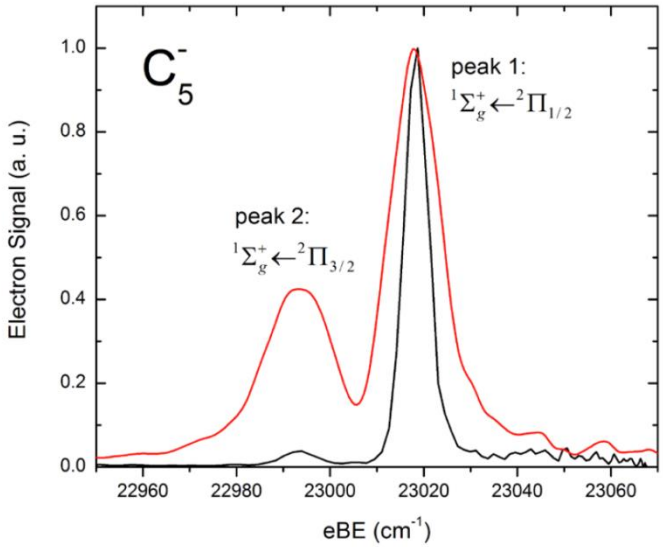

Figure 2. SEVI spectra of C_5^- with (black) and without (red) cryo-cooling

goal of obtaining spectral resolution comparable to that seen for atomic systems, enabling high resolution SEVI spectra of complex clusters and other species as outlined in the next section.

3) A key thrust of our research effort is to unravel the photoelectron spectra of transition metal oxide clusters, since these are tractable model systems for reactive sites in catalysis. These species present significant spectroscopic challenges, including multiple low-lying structural isomers, a dense manifold of electronic states, and very low vibrational frequencies. As a result, photoelectron spectra of transition metal oxide cluster anions generally show little or no vibrational structure, especially if there are two or more transition metal atoms. With this in mind, we have measured cryo-SEVI spectra of a series of transition metal oxide cluster anions, starting with the triatomic dioxide species TiO_2^-, ZrO_2^-, HfO_2^-, and VO_2^-, and followed by work on the more complex clusters $Ti_2O_4^-$, $Zr_2O_4^-$, Fe_3O^-, and Co_3O^-.

The group 4 transition metal (Ti, Zr, Hf) oxides are an important class of materials, with extensive applications as catalysts, catalyst supports, photocatalysts, dielectric materials, and corrosion resistant materials. Vanadium oxide catalysts play a key role in the oxidative dehydrogenation of propane and the oxidation of methanol, two processes of considerable industrial importance. Our first set of experiments was performed on triatomic group 4 transition metal di-oxide clusters.[20] The triatomic MO_2^- anions are closed-shell species with $^1A_1$ electronic states, while the neutrals have $^2A_1$ ground states that are well separated from higher-lying states. Hence, the electronic spectroscopy of these clusters is relatively simple, making them good test systems for the new instrument. The SEVI spectra of the three anions, shown in Fig. 3, exhibit fully-resolved vibrational progressions in all three vibrational modes (including the ν_3 mode, shown in the insets, which presumably is seen through vibronic coupling), yielding accurate vibrational frequencies and electron affinities.

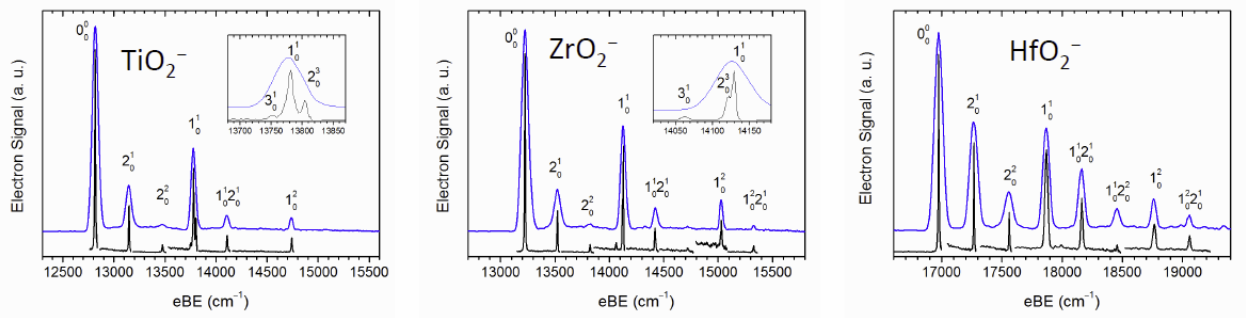

Figure 3. SEVI spectra of TiO_2^-, ZrO_2^-, and HfO_2^-. Overview spectra are shown in blue, and high resolution composite spectra are shown in black.

The electronic structure of VO_2 and VO_2^- is considerably more complicated.[21-23] The anion has two nearly degenerate triplet states, the $^3A_1$ and $^3B_1$ states, while neutral VO_2 has a $^2A_1$ ground state and low-lying $^2B_1$ and $^2A_1$ excited states. As shown in Fig. 4,[24] we observe fully vibrationally-resolved transitions from the anion ground state to the first three electronic states of neutral VO2. The anion ground state is identified as the $^3B_1$ state with the aid of Franck-Condon simulations and electronic structure calculations. The improved resolution and ion cooling

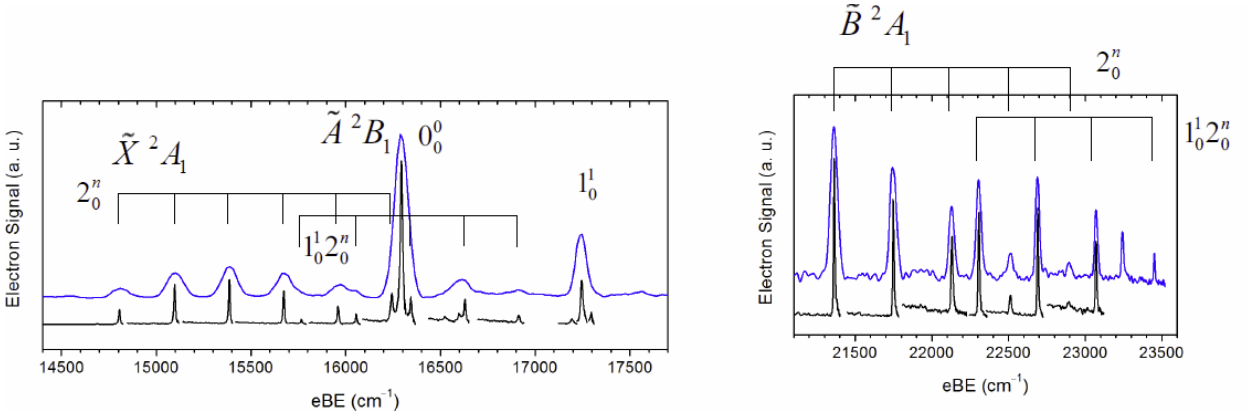

Figure 4. SEVI spectra of VO_2^-, showing transitions to the X, A, and B states of VO_2.

allows us to obtain accurate vibrational frequencies, electron affinities, and term energies. We also reassign bands observed in the previously reported PE spectrum of VO_2^- [25] to be consistent with our interpretation.

The Ti_2O_4 and Zr_2O_4 clusters exhibit an additional level of complexity, since electronic structure calculations find three closely-lying structural isomers for the anionic and neutral clusters,[26] as shown in Fig 5. The energy ordering depends on the charge state and predict differing ground state structures depending on the theoretical method used. While there is experimental evidence that both neutral clusters have C_{2h} ground states,[27] the energy ordering for the anions is considerably less settled.

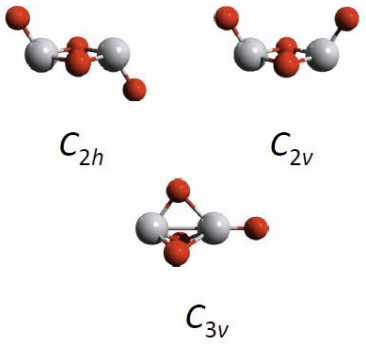

Figure 5 Structural isomers of Ti_2O_4, Zr_2O_4, and their anions.

SEVI spectra of $Ti_2O_4^-$ and $Zr_2O_4^-$ are shown in Figs. 6 and 7.[28] The spectra show extensive vibrational structure and, more importantly, are totally different. The $Ti_2O_4^-$ spectrum shows two extended bands, a strong band B with a characteristic peak spacing of 95 cm^{-1} and a bi-modal intensity distribution, and a weaker band A with a peak spacing of 180 cm^{-1}. The $Zr_2O_4^-$ spectrum shows a strong vibrational origin (peak A) and irregular peaks characteristic of multiple active vibrational modes. There is also a very peak progression (see inset) with a peak spacing of 95 cm^{-1}. By comparing the experimental results with Franck-Condon simulations and calculated vibrational frequencies, we assign the dominant feature (band B) in

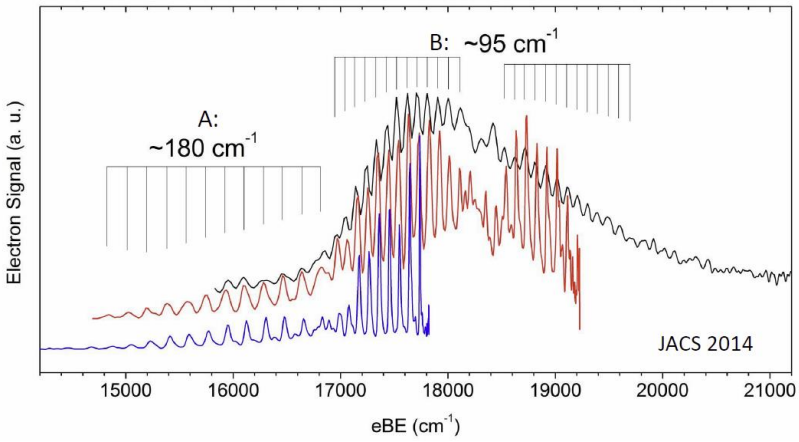

Figure 6. SEVI spectrum of $Ti_2O_4^-$. Bands B and A originate from anion C_{2v} and C_{2h} isomers, respectively.

the $Ti_2O_4^-$ PE spectra to photodetachment from the C_{2v} isomer, and band A to the higher-lying (and hence less populated) C_{2h} isomer. The lowest-energy isomer of $Zr_2O_4^-$ is the C_{3v} isomer, but a small amount of the C_{2v} isomer accounts for the weak band in the inset.

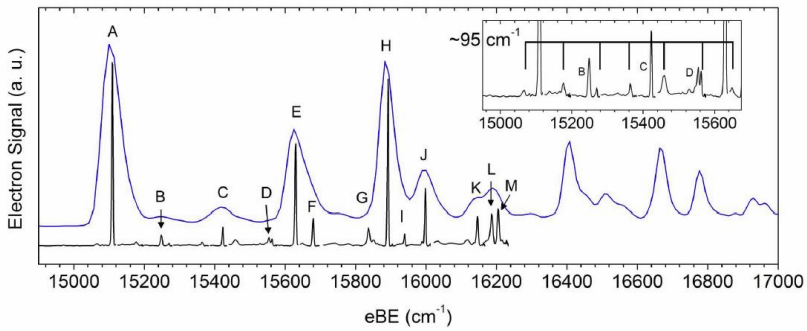

Figure 7. SEVI spectrum of $Zr_2O_4^-$. Main and weak bands (inset) are from C_{3v} and C_{2v} isomers, respectively.

Most recently, we have obtained high-resolution SEVI spectra of the transition metal suboxide clusters Fe_3O^- and Co_3O^-. As shown in Fig. 8,[29] the spectra are very well-resolved. Several vibrational frequencies of the neutral ground state Fe_3O and Co_3O clusters are assigned for the first time, and a low-lying excited state of Fe_3O is observed. The experimental results are compared with density functional electronic structure calculations and Franck-Condon spectral simulations, enabling identification of the structural isomer and electronic states. As has been found in photoelectron spectra of other trimetal oxo species, $Fe_3O^{0/-}$ and $Co_3O^{0/-}$ are assigned to a μ_2-oxo isomer with planar C_{2v} symmetry. We identify the ground states of Fe_3O^- and Co_3O^- as $^{12}A_1$ and $^9B_2$ states, respectively. From these states we observe photodetachment to the $^{11}B_2$ and $^{13}A_1$ ground and excited states of Fe_3O, as well as the $^8A_1$ ground state of Co_3O.

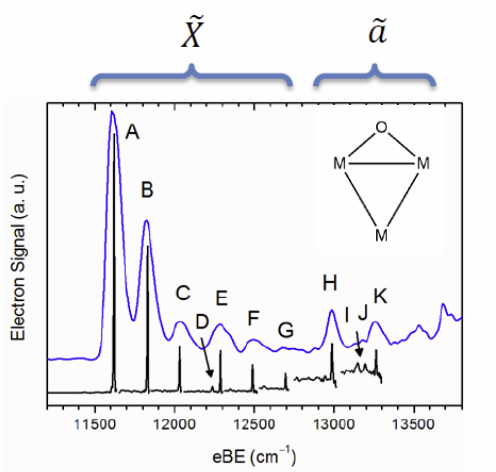

Figure 8. SEVI spectrum of Fe_3O^-.

Cryo-SEVI has also been applied to the study of free radicals and bimolecular transition states. We obtained highly-resolved spectra of the polycyclic aromatic hydrocarbon radicals indenyl (C_9H_7) and fluorenyl ($C_{13}H_9$) via SEVI of the corresponding anions, both of which are aromatic.[30] Detailed analysis showed that the fluorenyl spectrum could be interpreted in terms of normal Franck-Condon considerations, but that the vibrational structure in the indenyl spectrum was dominated by nominally forbidden transitions that were allowed only through vibronic coupling between the radical ground state and a low-lying excited state. In our study of the CH_2CN radical via SEVI of CH_2CN^-, we demonstrated how one could vary the ion trap temperature to adjust vibrational state populations in the anion, and could thus precisely control and understand which features in the spectrum originated from the ground and vibrationally excited anion states.[31]

The cyro-SEVI spectrum of $F^-(CH_4)$ ions reveals dynamics in the entrance channel and transition state region of the benchmark polyatomic reaction $F + CH_4 \rightarrow HF + CH_3$.[32] The experimental spectra show extended, low frequency progressions with characteristic spacings of

15-20 cm^{-1}. Comparison with quantum scattering calculations shows that these features result from complex vibrational motion on the neutral reactive surface involving both $F\cdot\cdot CH_4$ stretching and rotational motion of the F atom relative to the CH_4. Recent unpublished cryo-SEVI experiments on FH_2^- and FD_2^- show well-resolved peaks that, by comparison to quantum scattering calculations, clearly correspond to product resonances, marking the first spectroscopic identification of the resonances in the F + H_2 and F + D_2 reactions.

4) Infrared multiple photon dissociation of hydrated anions

In our continued collaboration with Prof. Knut Asmis (Leipzig), we have measured infrared spectra of a series of hydrated anions using the technique of infrared multiple photon dissociation, in which cryogenically cooled, mass-selected anions are dissociated by the absorption of multiple photons from a tunable infrared free electron laser.[33] This work is motivated by the importance of clusters comprising bisulfate (HSO_4^-) and nitrate (NO_3^-) anions, their conjugate acids, and water, in atmospheric chemistry and in aerosol formation. This program initially focused on $X^-(H_2O)_n$ clusters in order to probe how solvation shells form around anions of fundamental interest and to understand how solvation affects the structure and spectroscopy of the anion. We have recently expanded this program to cover more complex clusters involving anions, their conjugate acids, and water molecules, such as $NO_3^-(HNO_3)_m(H_2O)_n$ and $HSO_4^-(H_2SO_4)_m(H_2O)_n$. These studies show how the very strong hydrogen-bonding in a binary complex of an anion and its conjugate acid is affected by solvation and hydration.

Our experiments on $HSO_4^-(H_2O)_n$ clusters (n=1-16) showed extensive vibrational structure assigned to stretching and bending modes of the bisulfate core, as well as to water bending and librational modes.[34] Comparison to simulated spectra from electronic structure calculations indicates that the acidic proton in bisulfate is involved in hydrogen-bonding starting at n=1, and the water-water hydrogen bonds are present for n≥2. We have also begun investigating hydrated $H_2PO_4^-$ anions, starting with the binary $H_2PO_4^-(H_2O)$ complex.[35] It was not possible to simulate this spectrum within the harmonic approximation, and instead required ab initio molecular dynamics simulations to account for the apparent large amplitude motion of the water molecule. We are currently investigating whether simulations of these types will provide a better framework for understanding the infrared spectra of this genre of clusters.

In our work on anion/acid clusters, we first explored mixed clusters of NO_3^- and HSO_4^- with nitric and sulfuric acid molecules. We found that bisulfate was the main charge carrier for $HSO_4^-\cdot H_2SO_4\cdot HNO_3$, but not for $NO_3^-\cdot H_2SO_4\cdot HNO_3$.[36] The spectrum of the mixed dimer anion showed evidence for two isomers, $HSO_4^-\cdot HNO_3$ and $NO_3^-\cdot H_2SO_4$. In $HSO_4^-(H_2SO_4)_m(H_2O)_n$ clusters,[37] we observed the triply-hydrogen bonded $HSO_4^-(H_2SO_4)$ motif in clusters with no water molecules, while this motif was disrupted for hydrated clusters with m>1. In $NO_3^-(HNO_3)_m(H_2O)_n$ clusters, we found evidence for an equally shared proton for $NO_3^-\cdot HNO_3$ with a characteristic absorption at 877 cm^{-1}.[38] This arrangement is disrupted by the addition of at

least one more HNO_3 molecule or two more water molecules, leading to localization of the proton near one of the nitrate cores.

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5. PERSONNEL REPORTED

Principle Investigator
Daniel M. Neumark

Graduate Students
Jessalyn Devine
Jongjin Kim
Marissa Weichman
Tara Yacovitch

Volunteer
Corey-Shih

Post-Doctoral Scholar
Christian Hock

Administrative
Michelle Haskins

6. PUBLICATIONS

1. C. H. Hock, J. B. Kim, M. L. Weichman, T. I. Yacovitch, and D. M. Neumark. "Slow Photoelectron Velocity-Map Imaging Spectroscopy of Cold Negative Ions." J. Chem. Phys. 137, 244201 (2012).

2. T. I. Yacovitch, N. Heine, C. Brieger, T. Wende, C., Hock, D. M. Neumark, and K. Asmis. "Vibrational Spectroscopy of Bisulfate/Sulfuric Acid/Water Clusters: Structure, Stability and IRMPD Intensities." J. Phys. Chem. A 117, 7081 (2013).

3. J. B. Kim, M. Weichman, T. I. Yacovitch, and D. M. Neumark. "Slow Photoelectron Velocity-Map Imaging Spectroscopy of the C_9H_7 (Indenyl) and $C_{13}H_9$ (Fluorenyl) Anions." J. Chem. Phys. 139, 104301 (2013).

4. M. Weichman, J. B. Kim, and D. M. Neumark. "Vibrational Fine Structure of C_5^- via Anion Slow Photoelectron Velocity-Map Imaging." J. Chem. Phys. 139, 144314 (2013).

5. J. B. Kim, M. Weichman, and D. M. Neumark. "High-Resolution Anion Photoelectron Spectra of TiO_2^-, ZrO_2^-, and HfO_2^- Obtained by Slow Electron Velocity-Map Imaging." Phys. Chem. Chem. Phys. 15, 20973 (2013).

6. J. B. Kim, M. Weichman, and D. M. Neumark. "Vibronic Structure of VO_2 Probed By Slow Photoelctron Velocity-Map Imaging Spectroscopy." J. Chem. Phys. 140, 034307 (2014).

7. T. Westermann, J. B. Kim, M. Weichman, C. Hock, T. I. Yacovitch, J. Palma, D. M. Neumark, and U. Manthe. "Resonances in the Entrance Channel of the F + CH_4→ HF + CH_3 Reaction." Angew. Chem. Int. Ed. 53, 1122 (2014).

8. M. L. Weichman, J. B. Kim; and D. M. Neumark. "Rovibronic Structure in Slow Photoelectron Velocity-map Imaging Spectroscopy of CH_2CN^- and CD_2CN^-." J. Chem. Phys. 140, 104305 (2014).

9. J. B. Kim, M. L. Weichman, and D. M. Neumark. "Structural Isomers of Ti_2O_4 and Zr_2O_4 Anions Identified by Slow Photoelectron Velocity-Map Imaging Spectroscopy." J. Am. Chem. Soc. 136, 7159 (2014).

10. J. B. Kim, M. L. Weichman, and D. M. Neumark. "Slow Photoelectron Velocity-Map Imaging Spectroscopy of the Fe_3O^- and Co_3O^- Anions." J. Chem. Phys. 141, 174307 (2014).

11. J. B. Kim, M. L. Weichman, and D. M. Neumark. "Assignment of Electronic Bands in the Photoelectron Spectrum of VO_2^- Anion." J. Chem. Theory Comput. 10, 5235 (2014).

12. M. L. Weichman, J. B. Kim, J. A. DeVine, D. S. Levine, and D. M. Neumark. "Vibrational and Electronic Structure of the α- and β-naphthyl Radicals via Slow Photoelectron Velocity-Map Imaging." J. Am. Chem. Soc. 137, 1420 (2015).

13. M. Weichman, J. Kim, D. J. Neumark. "Slow Photoelectron Velocity-Map Imaging Spectroscopy of the *ortho*-Hyroxyphenoxide Anion." J. Phys. Chem. A, 119, 6140 (2015).

14. J. B. Kim, M. L. Weichman, and D. M. Neumark. "Low-Lying States of FeO and FeO^- by Slow Photoelectron Spectroscopy." Molecular Physics 113, 2105 (2015).

15. J. B. Kim, M. L. Weichman, T. F. Sjolander, D. M. Neumark, J. Kloss, M. H. Alexander, and D. E. Manolopoulos. "Spectroscopic Observation of Resonances in the $F+H_2$ Reaction." Science 349, 510 (2015).

7. INTERACTIONS/TRANSITIONS

July 22-27, 2012
Lewiston, ME
"Excess Electrons in Clusters and Liquid Jets"
GRC – Electron Spectroscopy and Dynamics – Bates Colleges, Lewiston, ME
Invited Lecture

August 5-10, 2012
Lewiston, ME
"Slow Electron Velocity-map Imaging of Negative Ions:Applications to Spectroscopy Dynamics"
GRC – Vibrational Spectroscopy – University of New England, Biddeford, ME
Invited Lecture

October 22-26, 2012
Paris, France
"Transition State Spectroscopy Using Slow Electron Velocity-map Imaging"
Stereodynamics of Chemical Reactions
Invited Lecture
October 28-31, 2012
Potomac, MD
AMOS DOE Meeting
Attended Meeting

December 1-3, 2012
Bangalore, India
"Making Molecular Movies: Ultrafast Lasers in Chemistry"
Indian Institute of Science, Science Camp for Young Students
Invited Lecture

January 30-February 1, 2013
Pacific Grove, CA
"Slow Electron Velocity-Map Imaging of Cold Negative Ions"
60th Annual Western Spectroscopy Association Conference
Invited Lecture

February 4-9, 2013
Goettingen and Hamburg, Germany
"Femtosecond and Attosecond Dynamics in Atoms, Molecules, and Clusters"
2 Seminars in Germany
Invited Lectures

March 18-21, 2013

Baltimore, MD
"Herbert P. Broida Prize Lecture: Probing Chemical Dynamics with Negative Ion Photodetachment"
APS 2013 Herbert P. Broida Prize
Award Recipient

March 30-April 6, 2013
Hong Kong, China
"Probing Chemical Dynamics with Negative Ions"
University of Hong Kong
Invited Lecture

April 15-17, 2013
England, United Kingdom
2 Lectures
(1)"Time-Resolved Studies of Electron-Induced Dynamics in DNA Bases"
(2)"Probing Chemical Dynamics with Negative Ions"
University of Nottingham, Faraday Lecture
Invited Lecture

June 3-5, 2013
Quebec City, Quebec, Canada
"Herbert P. Broida Prize Lecture: Probing Chemical Dynamics with Negative Ion Photodetachment"
APS Conference
Invited Lecture

June 10-13, 2013
Brussels, Belgium
ERC Meeting
Attended Meeting

June 16-19, 2013
Regensburg, Germany
"Quantum Clusters"
Biannual Meetings on Quantum Fluid Clusters
Invited Lecture

July 7-12, 2013
Lake Tahoe
"Probing Chemical Dynamics with Negative Ions"
DMC Meeting
Invited Lecture

August 7-8, 2013

Arlington, VA
DARPA/PULSE Meeting
Attended Meeting

September 9-12, 2013
Indianapolis, IN
"Dynamics of Excess Electrons in Clusters and Liquid Microjets"
246th Annual ACS Meeting
Invited Lecture

October 27-30, 2013
Washington, D.C.
AMOS DOE Contractors Meeting
Attended Meeting

November 3-6, 2013
Atlanta, GA
"Interaction of Excess Electrons with Water and Nucleobases."
Emory University
Invited Lecture

February 11-14, 2014
Berlin, Germany
Fritz Haber Institute
Attended Meeting

February 23-25, 2014
Austin, TX
DARPA Meeting
Attended Meeting

February 25-28, 2014
Galveston, TX
"Time-Resolved Photoelectron Spectroscopy of Clusters and Liquid Jets"
Gordon Research Conference
Invited Lecture

March 2-4, 2014
Boulder, CO
JILA Advisory Committee Meeting
Attended Meeting

March 16-20, 2014
Dallas, TX
"Spectroscopy of Cryogenically-Cooled Anions by Slow Electron Velocity-Map Imaging"
ACS

Invited Lecture

April 26-May 2, 2014
Italy
“Dynamics of Excess Electrons in Clusters”
MIC (Molecular and Ionic Clusters) Gordon Research Conference
Invited Lecture

May 2-13, 2014
England
Royal Society of Chemistry
Invited Lecture

May 15-16, 2014
Berkeley, CA
LBNL UXSL/AMOS Review
Attended Meeting

May 19-21, 2014
Arlington, VA
AFOSR Meeting
Attended Meeting

May 27-30, 2014
Potomac, MD
DOE 34th Annual Combustion Research Meeting
Attended Meeting

July 12, 2014
Easton, MA
“Time-Resolved Radiation Chemistry: Interaction of Excess Electrons with Water and Nucleobases”
Gordon Research Seminar (GRS)
Invited Lecture (Keynote Speaker)

July 13-18, 2014
Easton, MA
"Slow-Electron Velocity-Map Imaging of Cryogenically Cooled Anions"
Gordon Research Conference (GRC)
Invited Lecture

July 24-28, 2014
Kauai, Hawaii
“Spectroscopy of Cryogenically Cooled Anions by Slow Electron Velocity-map Imaging”
ICCB, International Conference on Chemical Bonding
Invited Lecture

August 10-14, 2014
San Francisco, CA
"Spectroscopy of Cryogenically Cooled Anions via Slow Electron-Map Imaging"
ACS
Invited Lecture

September 7-14, 2014
Beijing, CHINA
"Time Resolved Radiation Chemistry: Interaction of Excess Electrons with Water and Nucleobases"
Zhang Dayu Lecture
Invited Lecture

September 27, 2014-December 13, 2014
Oxford, England
"Ultrafast X-Ray Science in the Femtosecond and Attosecond Regime"
Oxford University
Sabbatical/ Invited Lecture/Various Lectures/Research

October 26-31, 2014
Oaxaca, Mexico
"Time-Resolved Radiation Chemistry: Interaction of Low Energy Electrons with Nucleobases"
8th International Conference on Photodynamics and Related Aspects
Invited Lecture

November 17-18, 2014
Berkeley, CA
Attosecond MURI
MURI Kickoff Meeting at UC Berkeley
Invited Lecture and Attended Meeting

November 2014
Durham, England
University of Durham
Invited Lecture

November 2014
Oxford, England
"Ultrafast X-Ray Science in the Femtosecond and Attosecond Regime"
Oxford University
Invited Lecture

December 2014
Manchester, England
"Slow Electron Velocity-Map Imaging of Cryogenically Cooled Anions"

University of Manchester
Invited Lecture

January 14-16, 2015
Arlington, VA
DARPA/PULSE Review Meeting
Invited Lecture and Attended Meeting

January 23, 2015-Feburary 14, 2015
Innsbruck, Austria
"Slow Electron Velocity-Map Imaging of Cryogenically Cooled Anions"
Research at University of Innsbruck
Sabbatical

February 22-27, 2015
Galveston, TX
"Slow Electron Velocity-Map Imaging of Cryogenically Cooled Anions"
Gordon Research Conference (GRC) on Gas Phase Ions Gaseous Ions: Structures, Energetics & Reactions
Invited Lecture

March 8-12, 2015
Grindelwald, Switzerland
"Ultrafast X-Ray Science in the Femtosecond and Attosecond Regime"
Ultrafast Dynamic Imaging of Matter
Invited Lecture

April 11, 2015-May 12, 2015
Heidelberg, Germany
Research in Heidelberg
Sabbatical

April 2015
Heidelberg, Munich, Freiburg, Leipzig – Germany
"Time-Resolved Radiation Chemistry: Dynamics of Excess Electrons in Clusters and Liquid Jets"
University of Leipzig
"Ultrafast X-Ray Science in the Femtosecond and Attosecond Regime"
Max Planck Institute for Nuclear Physics, Heidelberg, April.
"Ultrafast X-Ray Sience in the Femtosecond and Attosecond Regime"
University of Freiburg
Invited Lectures

May 2015
Paris, France

"Time-Resolved Radiation Chemistry: Dynamics of Excess Electrons in Clusters and Liquid Jets"
University of Orsay
Invited Lecture

May 13-15, 2015
Berkeley, CA
DOE/LBNL - Gas Phase Chemical Physics / Condensed Phase and Interfacial Molecular Science (CPIMS) / MES and Chemical Dynamics Beamline Review
Attended Meeting

May 19-21, 2015
Albuquerque, NM
"Slow Electron Velocity-Map Imaging (SEVI) of Cryogenically Cooled Transcient Metal Oxide Cluster Anions"
AFOSR Program Review
Attended Meeting

May 26-29, 2015
Potomac, MD
"Spectroscopy and Photodisccoaition of Free Radicals."
DOE Meeting
Attended Meeting

June 28-July 3, 2015
Segovia, Spain
"Time-Resolved Radiation Chemistry: Interaction of Excess Electrons with Nucleic Acid Constituents in Clusters and in Aqueous Solution"
ISMB2015, XXVI edition of the International Symposium on Molecular Beams
Invited Lecture

July 12-17, 2015
Pacific Grove, CA
Asilomar Conference
Chaired Session

July 18-23, 2015
Taipei, Taiwan
Program Review Committee
Attended Meeting

September 28-30, 2015
Brussels, Belgium
ERC Meeting
Attended Meeting

October 11-15, 2015
Chengdu, China
“Attosecond Dynamics of Atoms, Molecules, and Solids”
COMET Conference
Invited Lecturer

October 25-28, 2015
Gaithersburg, MD
AMOS DOE
Attended Meeting

November 12-13, 2015
Orlando, FL
MURI 1
“Probing the Electronic Timescale with Tunable Attosecond Pulses”
MURI 9
“Attosecond Auger Electron Spectroscopy in Liquid Water”
Joint Attosecond MURI 1 and 9 Meetings (Host University of Central Florida)
Attended Meeting

December 15-28, 2015
Honolulu, Hawaii
(1) “Spectroscopy and Dynamics of Free Radicals Studied by Slow Electron Velocity-Map Imaging of Cryogenically Cooled Anions”
(2) “Time-Resolved Radiation Chemistry: Electron Dynamics in Iodide-Nucleobase Clusters”
Pacifichem ACS
Invited Lecture

January 15-21, 2016
Brussels, Belgium
ERC Meeting
Attended Meeting

February 28-March 4, 2015
Davos, Switzerland
“TITLE”
S3C Meeting
Invited Lecture

March 2016
Boulder, CO
JILA Scholars Program
Visiting Fellow

March 12-14, 2016
San Diego, CA

"Slow Electron Velocity-Map Imaging of Cryogenically Cooled Anions"
Invited Lecture

March 21-23, 2016
Orlando, FL
DARPA Meeting
Attended Meeting

April 29, 2016-May 3, 2016
Washington, D.C.
NAS Meeting
Award Recipient

8. NEW DISCOVERIES, INVENTIONS, OR PATENT DISCLOSURES

None

9. HONORS/AWARDS

Member, National Academy of Sciences, 2015
Fellow, Royal Society of Chemistry, 2013
RSC Chemical Dynamics Award, 2013
APS Herbert P. Broida Prize, 2013

1.

1. Report Type

Final Report

Primary Contact E-mail

Contact email if there is a problem with the report.

dneumark@berkeley.edu

Primary Contact Phone Number

Contact phone number if there is a problem with the report

510-642-3502

Organization / Institution name

University of California, Berkeley

Grant/Contract Title

The full title of the funded effort.

New Insights in Catalytic Sites: Characterization and Reactivity of Metal Oxide Clusters with Anion Slow Electron Velocity-Map Imaging

Grant/Contract Number

AFOSR assigned control number. It must begin with "FA9550" or "F49620" or "FA2386".

FA9550-12-1-0160

Principal Investigator Name

The full name of the principal investigator on the grant or contract.

Daniel M. Neumark

Program Manager

The AFOSR Program Manager currently assigned to the award

Dr. Michael R. Berman

Reporting Period Start Date

05/01/2012

Reporting Period End Date

04/30/2016

Abstract

One of the outstanding problems in all of physical chemistry is to understand reactive sites in heterogeneous catalysts, many of which comprise complex transition metal oxides. Size-selected transition metal oxide clusters can serve as tractable model systems for the analogous bulk catalyst. This research program has focused on developing novel spectroscopic probes of such clusters. During the last grant period, slow electron velocity-map imaging of cryogenically cooled anions (cryo-SEVI) was implemented and shown to be a qualitative advance in the spectroscopic investigation of transition metal oxide clusters, yielding high resolution (≈ 2 cm-1) photoelectron spectra from which one can elucidate structural and bonding motifs of the anions and the neutrals generated by photodetachment. There has been a particular focus on polymetallic clusters such as Ti2O4, Zr2O4, and Fe3O, since these species have largely been spectroscopically intractable using more conventional methods. Additional experiments based on infrared multiple-photon dissociation of cryo-cooled anions have focused on complex hydrated clusters such as $NO_3^-(HNO_3)_m(H_2O)_n$ and $HSO_4^-(H_2SO_4)_m(H_2O)_n$ that are important in atmospheric chemistry and aerosol formation.

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Archival Publications (published) during reporting period:

Changes in research objectives (if any):

Change in AFOSR Program Manager, if any:

Extensions granted or milestones slipped, if any:

AFOSR LRIR Number

LRIR Title

Reporting Period

Laboratory Task Manager

Program Officer

Research Objectives

Technical Summary

Funding Summary by Cost Category (by FY, $K)

	Starting FY	FY+1	FY+2
Salary			
Equipment/Facilities			
Supplies			
Total			

Report Document

Report Document - Text Analysis

Report Document - Text Analysis

Appendix Documents

2. Thank You

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