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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE May 1992		3. REPORT TYPE AND DATES COVERED Comprehensive Final Report November 1, 1989 - April 30, 1991	
4. TITLE AND SUBTITLE "Surface Reactions in the Space Environment"				5. FUNDING NUMBERS W112F 2303/PD	
6. AUTHOR(S) Royal G. Albridge, Carl S. Ewing, Richard F. Haglund, Jr., Joel B. Tellinghuisen and Norman H. Tolk					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Department of Physics and Astronomy Center for Atomic and Molecular Physics at Surfaces Vanderbilt University Box 1807, Station B Nashville, TN 37235-1807 AFOSR-TR- 92 04 24				8. PERFORMING ORGANIZATION REPORT NUMBER 4-20-630-4183	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Ms. Vernita Slater Administrative Contract Officer Air Force Office of Scientific Research Department of the Air Force Bolling Air Force Base, DC 20332-6448				10. SPONSORING/MONITORING AGENCY REPORT NUMBER AFOSR-90-0030	
11. SUPPLEMENTARY NOTES					
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release Distribution unlimited				12b. DISTRIBUTION CODE A D	
13. ABSTRACT (Maximum 200 words) <p>The object of this research program is to carry out experimental and theoretical studies of the detailed microscopic mechanisms by which electronic energy is absorbed, transported and dissipated in ionic solids. The ultimate aim of this program is identify and characterize essential constituent elements of comprehensive models which will quantitatively describe radiation-induced electronic phenomena. The theoretical and experimental aspects of the project has been carried out concurrently and interactively in order to realize the greatest scientific benefit from the collaboration. Throughout this project we have employed (1) experimental techniques already developed to monitor bulk and surface properties before, during, and after electron and photon irradiation and to characterize time- and energy-dependent desorption phenomena, and (2) <i>ab initio</i> quantum-theoretical approaches to develop and refine computational models for determining properties of excited electronic states of relevant localized species. This research program bears directly on a broad spectrum of questions germane to the long-term operation of platforms in space, including structural, optical and electronic degradation of materials in the ambient near-earth environment, survivability under and hardening against irradiation from directed-energy weapons, vulnerability in disturbed nuclear atmospheres, and discrimination and sensing techniques based on radiation (glow) signatures.</p>					
14. SUBJECT TERMS				15. NUMBER OF PAGES 11	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED		18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED		19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	
				20. LIMITATION OF ABSTRACT SAR	

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**Final Report**

*to the*

**Air Force Office of Scientific Research  
Directorate of Chemical and Atmospheric Sciences  
Bolling AFB, DC 20332-6448**



*for a research program titled*

**SURFACE REACTIONS IN THE SPACE ENVIRONMENT**

Grant: AFOSR 90-0030

*Research Period: November 1, 1989 through April 30, 1991*

*submitted May 13, 1992 by*

**Royal G. Albridge, Carl S. Ewig, Richard F. Haglund, Jr.,  
Joel B. Tellinghuisen and Norman H. Tolk  
Department of Physics and Astronomy, and Department of Chemistry  
Vanderbilt University, Nashville, TN 37235**

**92-13718**



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## I. Research Objectives

The object of this research program is to carry out experimental and theoretical studies of the detailed microscopic mechanisms by which electronic energy is absorbed, transported and dissipated in and near insulator solids. The ultimate aim of this program is to identify and characterize essential constituent elements of comprehensive models which will quantitatively describe radiation-induced electronic phenomena. The theoretical and experimental aspects of the project has been carried out concurrently and interactively in order to realize the greatest scientific benefit from the collaboration. Throughout this project we have employed (1) experimental techniques already developed to monitor bulk and surface properties before, during, and after electron and photon irradiation and to characterize time- and energy-dependent desorption phenomena, and (2) *ab initio* quantum-theoretical approaches to develop and refine computational models for determining properties of excited electronic states of relevant localized species. This research program bears directly on a broad spectrum of questions germane to the long-term operation of platforms in space, including structural, optical and electronic degradation of materials in the ambient near-earth environment, survivability under and hardening against irradiation from directed-energy weapons, vulnerability in disturbed nuclear atmospheres, and discrimination and sensing techniques based on radiation (glow) signatures.

Much of scientific and technological significance has been accomplished during the period covered by this AFOSR grant. In particular, we report on the following topics: (a) on the basis of new measurements taken in our laboratory we have discovered an entirely new mechanism for the desorption of excited neutral atoms from alkali halide crystal surfaces due to photon- and electron- stimulated desorption; (b) our research shows *valence-band transitions* as well as *core-level transitions* can initiate desorption of excited alkali-metal atoms; (c) significant progress has been made *onab initio* theory of impurity centers and defects in ionic lattices; and (d) work on gas-phase molecular electronic spectra produced via electronic excitation of ionic solids has stimulated important advances in spectral analysis. The facilities and experience acquired during the previous URI proved an excellent base for carrying out this program.

## II. Research Status

### 1. New Mechanism for the Desorption of *Excited Atoms* by Photon and Electron Bombardment of Alkali Halide Crystals.

Measurements taken in our laboratory suggest an entirely new mechanism for the desorption of excited neutral atoms from alkali halide crystal surfaces due to photon- and electron- stimulated desorption (ref. 1). We propose that excited alkali atoms arise from the reaction of alkali dimers with halogen atoms present on the surface. The reactants on the surface are produced dynamically due to the migration of radiation induced defects created in the near surface bulk.

The determination of the mechanism for the production of excited atoms and molecules by photon-stimulated desorption (PSD) and electron-stimulated desorption (ESD) has been a controversial issue. Two prevailing models have been invoked to account for the production of excited species. One is in analogy to the Knotek-Feibelman mechanism of the ion desorption. Following the inter-atomic Auger decay of a core hole, two holes on a single anion cause ion desorption. Neutralization of the desorbing ion into an excited state would then account for the observed excited atom yield. This neutralization model requires core hole creation. The other model assumes that the excited atoms are produced by excitation of the ground state atoms arising from ESD or PSD in the gas phase by secondary electrons. Both mechanisms have been found to be inadequate in interpreting the existing data. Recent work shows that valence excitations can cause excited alkali atom desorption, *i.e.*, core hole creation is not required for the production of excited alkali atoms (ref. 2). This work also shows that the excited alkali atom yield decreases with increasing temperature while the total electron yield goes up, thus contradicting the secondary electron excitation theory.

The new mechanism we propose is based on an exergic reaction of alkali dimers and halogen atoms. The reaction was identified first in the gas phase by the observation of the sodium D-lines when sodium vapor was mixed with chlorine, bromine, and iodine. The reaction of interest  $\text{Na}_2 + \text{Cl} \rightarrow \text{Na}^* + \text{NaCl}$  was studied directly by molecular beam experiments and later supported by theoretical calculations. Conceptually, the new model can be described through use of one-electron pseudopotential surfaces for the  $\text{Na}_2\text{Cl}$  system for colinear configuration based on calculations in Struve's paper for this reaction. The reaction goes through a postulated transition state, in which an electron from sodium dimer jumps to the chlorine atom. For small impact parameters, the reaction leads to an *excited* sodium atom. The gas phase reaction cross section is  $10 - 100 \text{ \AA}^2$  estimated by

both experiments and theory.

The majority desorbing species arising from PSD or ESD in alkali halide crystals are neutral ground state halogen atoms and alkali atoms. The desorbed ground state halogen and alkali atoms are believed to be related to *H* center and *F* center migration to the surface. The incident photon or electron creates one or several holes in occupied bands in the near surface bulk. The holes are then self-trapped forming  $V_K$  centers. The  $V_K$  centers capture electrons becoming self-trapped excitons (STE). The STE then decay either emitting light or forming an *F* center - *H* center pair. An *F* center on the surface may neutralize an alkali ion forming a neutral alkali atom. At low temperatures, alkali atoms may accumulate on the surface, resulting in a metal-rich surface. The accumulated neutral alkali atoms may then form dimers, trimers, and larger clusters on the surface. Surface metallization has been observed in many experiments at room temperature. Metal atoms have been observed to form islands instead of uniform layers on surface. The diffusion of *F* centers from the bulk to the surface is believed to be the main source of supply of *F* centers required for the metallization. On the other hand, when an *H* center diffuses to or is created on the surface it leaves a neutral halogen atom which then desorbs off the surface with either thermal or hyperthermal velocities. Therefore, photon or electron bombardment of alkali halide crystals produces the two reactants on surface necessary for the reaction mentioned above to occur. At some temperature, the most favorable metal configuration will be dimers.

In these experiments, the time dependent behavior we believe arises from the instantaneous concentration of alkali dimers which depends on the degree of metallization of the surface. At the beginning of the bombardment, there are few alkali dimers on the surface. Although the halogen yield is large at this stage, the excited atom yield is small. As the metallization increases, there is an optimized stage where many alkali dimers are formed on the surface and the halogen atom concentration remains large. At this stage, the excited alkali atom yield is at a maximum. The yield decreases in the later stage because the dominant metal configuration with greater metallization results in cluster numbers greater than two leading eventually to metal island formation and the consequent depletion of halogen atoms in the surface region. The nonzero yield at the beginning of the measurement is due to a metal rich surface even of the annealed samples.

These preliminary experiments provide strong evidence for the hypothesis that bulk defects play a key central role in desorption and other material-modification processes. In addition this technique provides an effective tool for elucidating bulk-mediated mechanisms.

## 2. Interactions of Ultraviolet Photons with Alkali Halides

The interactions of ultraviolet and vacuum ultraviolet light with dielectric surfaces is a problem of critical importance to the survivability of spacecraft in low earth orbits. During the period of the contract, we completed three critical studies in this area, showing that:

- Bulk fluorescence competes against excited-alkali-atom desorption for the incident photon energy;[ref. 3]
- Photoelectron spectroscopy combined with fluorescence spectroscopy of excited desorbing alkali atoms clearly shows that the desorption is driven by excitonic processes; [ref. 4]
- Valence-band transitions as well as core-level transitions can initiate desorption of excited alkali-metal atoms.[ref. 2]

The last cited results are particularly important in demonstrating that even relatively low-energy photons can initiate the sequence of exciton formation and decay which culminates in nuclear motion and desorption. Apart from the obvious consequences of this new mechanism for the performance of insulators in the space environment, this also establishes a critical link to the old and technologically critical problem of laser damage to materials.[ref. 5]

Photon-induced surface decomposition and desorption in alkali halides have been studied extensively for one-photon band-to-band transitions initiated by synchrotron radiation. For core-level transitions, localization of the well-screened core hole initiates desorption of both ions and neutrals by a mechanism widely agreed to be initiated by the decay of the self-trapped exciton (STE) into an *F*-center-*H*-center pair. The dissociation of the *H*-center (an  $X_2^-$  molecule) near the surface produces a desorbing  $X^0$  and a bound  $X^-$ , while the recombination of an *F*-center with a surface alkali-metal ion leads to thermal desorption of a neutral alkali atom, usually in the ground state. The role of the metallized surface in the production of *excited* alkali atoms is still an open question; progressive radiation damage may eventually extinguish emission from excited atoms by resonant ionization as the Fermi level of the metallized surface rises into the insulator bulk band gap. However, there is no reason to doubt the fundamental correctness of this picture.

One important result published during the contract period concerns the competition for incident photon energy by the various relaxation modes available in the alkali halide crystal. By measuring both the bulk fluorescence and the fluorescence from excited sodium atoms desorbed from NaCl, using ultraviolet photons on and off excitonic transition energies, we showed that bulk fluorescence is associated with non-specific excitations of the crystal while desorption and secondary electron emission are associated with excitonic band-to-band transitions.

During the period of the contract, we completed another critical experimental study in which for the first time fluorescence spectroscopy of desorbing atoms was combined with a simple measurement of the photoelectron emission to show that desorption occurs by a localized two-hole excitation *in the valence band* following the decay of the core excitation. By comparing the lineshapes of the secondary electron and excited-atom fluorescence yields obtained as a function of incident photon energy, it was possible to resolve an earlier question about the number of valence holes required for desorption to take place. The data also support a tentative conclusion that the excited atom is created by the relaxation of a highly excited quasi-molecular configuration of the halogen ion and its nearest neighbors.

In our most recent synchrotron light-source experiments, we have shown that valence-band transitions can also trigger photon-stimulated desorption of excited alkali metal atoms. This is a particularly important finding, because it is inconsistent with all known mechanisms for photon-stimulated desorption from insulators. By demonstrating that excited-atom and secondary-electron yields are uncorrelated, this experiment also dealt the death-blow to a widely accepted theory that excited-atom desorption is a simple consequence of secondary electron excitation of desorbing ground-state atoms.

We note in conclusion the interesting connection between valence-band-initiated desorption and laser-induced damage to dielectric materials. Core-level transitions are not accessible for any reasonable laser, even by means of multiphoton excitations, because the energies are simply too high. Thus, one might think that the laser-induced desorption mechanism for the halides would be fundamentally different from that inferred from the low-intensity synchrotron experiments, where the photon flux is only of order  $10^{12}$  photons/s over a larger beam spot. However, for the laser parameters in our experiments with KCl, with peak laser intensities of order  $10^{14}$  W·m<sup>-2</sup>, the local density of electronic excitation is low because the creation of the self-trapped exciton whose decay initiates the expulsion of a halogen atom requires a two-photon excitation. This connection has since

been confirmed qualitatively,[ref. 6] and further experiments are expected to elucidate this connection in quantitative fashion.

### 3. Theory and Data Analysis

In this low-budget extension of the Vanderbilt URI project, "Surface Reactions in the Space Environment," our primary aim was the completion of ongoing work on (A) *ab initio* theory of impurity centers and defects in ionic lattices, and (B) the analysis of gas-phase molecular electronic spectra produced via electronic excitation of ionic solids. Major aspects of these projects have now been completed. At the same time, most of the new work proposed in the original request for extension of the URI project was not initiated for lack of resources. This means that the work under (A) was confined mainly to completion of computations on the anionic impurity center  $O_2^-$  in point-ion lattices, and on the *F*-center in LiF. The efforts conducted under (B) centered on further development of the least-squares computational methods for extracting information from congested spectra, with application to CN  $B \rightarrow X$  emission and OH  $A \rightarrow X$  emission. In addition we applied similar techniques to some peripherally related problems in halogen spectroscopy, done in collaboration with Prof. M. C. Heaven's group at Emory University.

The *ab initio* theoretical work on  $O_2^-$  (ref. 8 below) completes our work on a triad of diatomic anions of varying polarity —  $O_2^-$ ,  $CN^-$ , and  $OH^-$ . In agreement with anticipation, the results obtained for the least polar  $O_2^-$  species showed the greatest insensitivity to its lattice environment. In fact the potential curves and spectroscopic constants obtained for the several electronic states under investigation — mainly the ground state,  $X \ 2\Pi_g$ , and the excited states,  $A \ 2\Pi_u$  and  $a \ 4\Sigma_u^-$  — varied only weakly with orientation within the lattice. In another surprise, the *X* and *A* states proved stable against "computational autodetachment" *in vacuo*, even though in some regions of internuclear distance both lie energetically above the ground state of the parent neutral molecule. Furthermore the *in vacuo* spectroscopic constants for these two states are very close to the "*in lattice*" results. Our previous experience with excited states energetically capable of autodetachment in  $CN^-$  and  $OH^-$  led us to expect the  $\Pi$  states of  $O_2^-$  to be energetically unstable with respect to variation of diffuse functions in the basis set, and at present we are unable to account for the stability of these states in our calculations. One feature of the lattice calculations which does not occur *in vacuo* is crystal field splitting of the  $\Pi$  states. However, it appears that our point-ion model can yield only a very rough picture of this effect; a more realistic "real ion" model will be required.

Most of our theoretical work on anionic molecular impurity centers has employed



the ultrasimple point-ion model but has nonetheless yielded surprisingly good agreement with experiment, where unambiguous comparisons could be made. This agreement is evidently testament to the validity of the "local" description of the bonding extant in the ground and low-lying excited electronic states of these species. On the other hand, with increasing excitation energy one or more of the valence electrons on the impurity center must move into progressively more diffuse orbitals, necessitating a more sophisticated treatment of the lattice. To probe our ability to handle this situation computationally, we have turned our attention to an age-old, clearly non-local problem — the *F*-center in LiF. For over 30 years the *F*-center has been treated with apparent success by various simple one-electron models. One might thus anticipate that its ab initio treatment would be straightforward. Yet in our recently published study of this problem (ref. 8), it was necessary to use quite large basis sets — minimal sets on the first three shells of neighboring ions (6 Li<sup>+</sup>, 12 F<sup>-</sup>, 8 Li<sup>+</sup>) plus diffuse functions on the F<sup>-</sup> ions — to achieve reasonable agreement with the experimental  $T_{1u} \leftarrow A_{1g}$  transition energy. Coincidentally, our very simplest basis set, consisting of just minimal functions on the neighboring Li<sup>+</sup>, also gave fairly good agreement with experiment. This perhaps illustrates the deceptive simplicity in the *F*-center problem.

Our work reporting the first observation of excited CN produced via ESD on alkali halides has now been published (ref. 9). At the time this work was originally prepared for publication the observations of the CN  $B \rightarrow X$  emission were preliminary in nature. These observations have now been substantiated and extended in further work by Jun Xu (refs. 10-14). In connection with the interpretation of the spectra first obtained, we devised a nonlinear least-squares procedure for extracting information about population distributions from congested spectra. (The initial CN  $B \rightarrow X$  spectra were rotationally unresolved.) In the CN case the spectral line positions were well known from earlier work, leaving only population and lineshape parameters to be obtained from the fit. However, shortly after completion of the CN study, we encountered "congested spectra" problems of a more complex nature, in which neither the line positions nor the lineshape and population parameters could be established at the outset. These spectra were obtained by Prof. M. C. Heaven's group at Emory University in studies of metastable excited electronic states of I<sub>2</sub> produced by ArF laser photolysis of I<sub>2</sub>/Ar mixtures in a free-jet expansion. Using a variation of the nonlinear routine developed for the CN problem, we have been very successful in analyzing these congested I<sub>2</sub> spectra (refs. 14, 16, 17) and are currently expanding to similar work on IBr and Br<sub>2</sub>. In related work, we have described a nonlinear least-squares procedure for reliable intensity calibration for that oldest of detectors, the photographic emulsion (ref. 15).

Work on the analysis of OH  $A \rightarrow X$  emission is being continued in conjunction with the development and testing of a promising new hybrid photodiode/CCD 1-dimensional array detector assembled by Marcus Mendenhall. Using least-squares procedures like those used to analyze CN and halogen spectra, we have been able to represent the multiline OH spectrum obtained from a Tesla discharge of H<sub>2</sub>O in Ar very well in terms of a two-temperature rotational distribution function. Yet the statistics of this fit are still far from our estimates of the statistics of the raw data from the detector, and so we are still trying to understand the sources of error in the experiment. We hope to prepare for publication a report of this work in the next few months (ref. 18).

### III. Publications Citing AFOSR-90-0030

1. "The Desorption of Excited Atoms by Photon Bombardment of Alkali Halide Crystals," D. Liu, R. G. Albridge, A. V. Barnes, P. H. Bunton, C. S. Ewig, N. H. Tolk and M. Szymanski. To be published in *Phys. Rev. Letters*.
2. "Observation of Photon-Stimulated Desorption Following Valence-Band Excitation of Alkali Halides," P.H. Bunton, R.F. Haglund, Jr., D. Liu and N. H. Tolk. *Physical Review B* 45 (9), pgs. 4566-4571 (1 March 1992)
3. "Observation of Excitonic Effects in Excited Neutral Alkali Desorption from Alkali Halides," P. H. Bunton, R. F. Haglund, Jr. and J. L. Rose, in *Desorption Induced by Electronic Transitions, DIET IV*, eds. G. Betz and P. Varga (Berlin, Heidelberg: Springer-Verlag, 1990), pp. 305-309. [Springer Series in Surface Sciences, Volume 19]
4. "Photon-stimulated desorption of excited alkali atoms from alkali halides following core-level excitation," P. H. Bunton, R. F. Haglund, Jr., D. Liu and N. H. Tolk, *Surf. Sci.* 243, 227-238 (1991)
5. "The Role of Valence-Band Excitation in Laser Ablation of KCl," R. F. Haglund, Jr., *Symposium on Laser-Induced Damage in Optical Materials*, SPIE Vol. 1441, 127-138 (1990).
6. "Ultraviolet laser ablation of halides and oxides," R. F. Haglund, Jr., M. Affatigato, J. H. Arps, K. Tang, A. Niehof and W. Heiland, *Nucl. Instrum. Meth.* B65, 206-211 (1992).
7. "Ab initio study of the electronic states of O<sub>2</sub><sup>-</sup> in vacuo and in simulated ionic solids," by Carl S. Ewig and Joel Tellinghuisen, *J. Chem. Phys.* 95, 1097 (1991).
8. "Ab initio F-Center electron topology in LiF," by Carl S. Ewig, Joel Tellinghuisen, and Marcus H. Mendenhall, *Chem. Phys. Lett.* 188, 501 (1992).
9. "CN B  $\bar{A}$  X emission in electron-stimulated desorption from alkali halides: Population analysis via nonlinear models," by Jun Xu, Marcus H. Mendenhall, and

- Joel Tellinghuisen, *J. Chem. Phys.* **931**, 5281 (1990).
10. "Generation of Excited CN Molecules Electronically Desorbed from Alkali-rich Surfaces," Jun Xu, Royal Albridge, Alan V. Barnes, Manfred Riehl-Chudoba, Akira Ueda, Norman Tolk, Dwight Russell and Paul Wang. *Surface Science* **262**, pgs. 77-87 (12 September 1991)
  11. "Optical Glow Spectra Arising from Low-energy N<sub>2</sub>, N<sub>2</sub><sup>+</sup> and Electron Bombardment of MgF<sub>2</sub> Surfaces," J. Qi, A.V. Barnes, S.L. Espy, M. Riehl-Chudoba, C-N. Sun, R.G. Albridge and N.H. Tolk. *Appl. Phys. Lett.* **59** (16), pgs. 1954-1956 (14 October 1991)
  12. "Gas-exposure Enhanced Na\* Emission from Na-rich Surfaces," Jun Xu, Royal Albridge, Alan Barnes, Xinxing Yang and Norman Tolk. *Applied Physics Letters* **60** (11), pgs. 1396-1398 (16 March 1992)
  13. "Electron- and Photon-stimulated Desorption as a Probe of the Dynamics of Molecules on Surfaces," Jun Xu, Alan Barnes and Norman Tolk. *J. Vac. Sci. Technol. A* **10** (4), pgs. 1-6 (Jul/Aug 1992)
  14. "Observation and Analysis of the  $D' \leftarrow A'$  Transition of I<sub>2</sub> in a Free-jet Expansion," by Joel Tellinghuisen, S. Fei, X. Zheng, and Michael C. Heaven, *Chem. Phys. Lett.* **176**, 373 (1991).
  15. "Characteristic curves for photographic emulsions from nonlinear fitting: A study of statistical and model error," by Joel Tellinghuisen, *Appl. Opt.* **30**, 1723 (1991).
  16. "Observation and Analysis of the  $\beta \leftarrow A$  Transition of I<sub>2</sub> in a Free-Jet Expansion," by Xiaonan Zheng, Suli Fei, Michael C. Heaven, and Joel Tellinghuisen, *J. Mol. Spectrosc.* **149**, 399 (1991).
  17. "Spectroscopy of metastable species in a free-jet expansion: The  $D' \leftarrow A'$  transition of I<sub>2</sub>," by Xiaonan Zheng, Suli Fei, Michael C. Heaven, and Joel Tellinghuisen, *J. Chem. Phys.* (in press).
  18. "Precision position and intensity spectroscopy using a 1-dimensional photodiode/CCD array detector: Application to OH  $A \rightarrow X$  emission spectra," by Marcus H. Mendenhall, Stanley W. Brown, Virginia Edwards, and Joel Tellinghuisen (to be published).

#### IV. Conference Presentations

1. "Spectroscopic Constants of O<sub>2</sub><sup>-</sup> Electronic States *in vacuo* and in ionic solids," by C. S. Ewig and J. Tellinghuisen, 45th Symposium on Molecular Spectroscopy (Columbus, Ohio), June, 1990.
2. "Least-Squares Error Propagation: Neglected Aspects," by J. Tellinghuisen, 45th Symposium on Molecular Spectroscopy (Columbus, Ohio), June, 1990.
3. "Quantum-Theoretical Studies of Defects in Ionic Solids: *F* Centers and O<sub>2</sub><sup>-</sup> Impurity Centers," by Carl Ewig and Joel Tellinghuisen, Fifth Annual AFOSR Workshop on Surface Reactions in the Space Environment (Evanston, IL), September, 1990.
4. "On the Analysis of Photometric and Spectrometric Data from Weak Light Sources," by Joel Tellinghuisen, Fifth Annual AFOSR Workshop on Surface Reactions in the Space Environment (Evanston, IL), September, 1990.

5. "New Mechansim for the Desorption of Excited Atoms by Photon Bombardmen of Alkali Halide Crystals," D. Liu, R. G. Albridge, A. V. Barnes, P. H. Bunton, C.S. Ewig, N. H. Tolk and M. Szymonski, DIET V (Taos, NM), April, 1992
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