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<b>14. ABSTRACT</b> To prepare new economical HEDM derivatives from the condensation reactions of nitric oxide with organic substrates. Once prepared these derivatives were tested for energy content, thermal stability, and product output upon decomposition. The outcome of these experiments were then used to assess which cations, and conditions, are the best to metathesize with the heavy/dense alkali metals initially isolated. We now have an excellent understanding of what the main products will be from these reactions, and the optimal conditions for their formation. This has proven to be a surprisingly variable reaction, and a variety of products can form from the addition of NO to a given substrate, but the determining variables are the base employed, substrate/base ratio, and the temperature. Nitric oxide pressure is also a likely variable as well, indeed many of the products we have characterized are new because we used ca. 3 atm of NO pressure, whereas prior researchers typically use only 1 atm. These compounds are now being tested at Thiokol for propellant characteristics after having undergone safety tests beforehand.					
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Final Progress Report -Year 3 (4/98-01)

Airforce contract number F49620-98-1-0461

DEPSCoR98: Tandem TGA/MS Studies of Single Propellants

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## Tandem TGA/MS Studies of Single Propellants

### Objectives:

To prepare new economical HEDM derivatives from the condensation reactions of nitric oxide with organic substrates. Once prepared these derivatives were tested for energy content, thermal stability, and product output upon decomposition. The outcome of these experiments were then used to assess which cations, and conditions, are the best to metathesize with the heavy/dense alkali metals initially isolated.

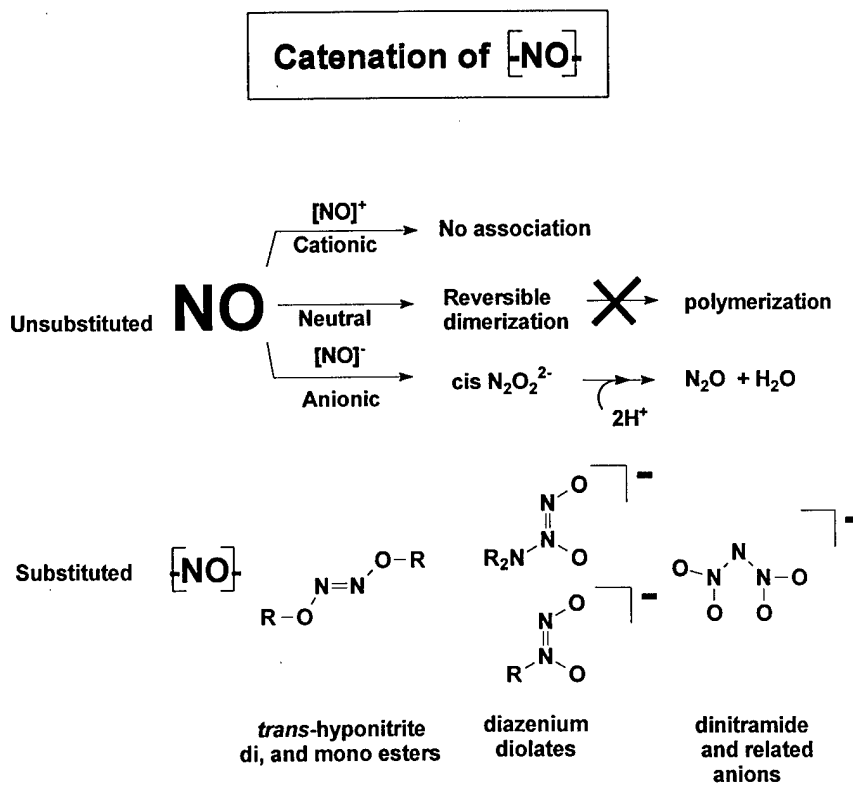
### Status of effort:

We now have an excellent understanding of what the main products will be from these reactions, and the optimal conditions for their formation. This has proven to be a surprisingly variable reaction, and a variety of products can form from the addition of NO to a given substrate, but the determining variables are the base employed, substrate/base ratio, and the temperature. Nitric oxide pressure is also a likely variable as well, indeed many of the products we have characterized are new because we used ca. 3 atm of NO pressure, whereas prior researchers typically use only 1 atm. These compounds are now being tested at Thiokol for propellant characteristics after having undergone safety tests beforehand.

### Accomplishments/New Findings:

In prior progress reports we have outlined many of our accomplishments in what has proven to be a fertile area of research. At this point I will outline in broad brush strokes where we are with our understanding of this system, and describe some recent extensions to illustrate how we can apply the general rules we have developed.

The addition of NO to organic compounds with active hydrogens results in the formation of a new nitrogen-nitrogen double bond, and the first catenizing step to generate, possibly, a polynitrogen species. General considerations for the reaction are shown on the right, and we note that the number of known

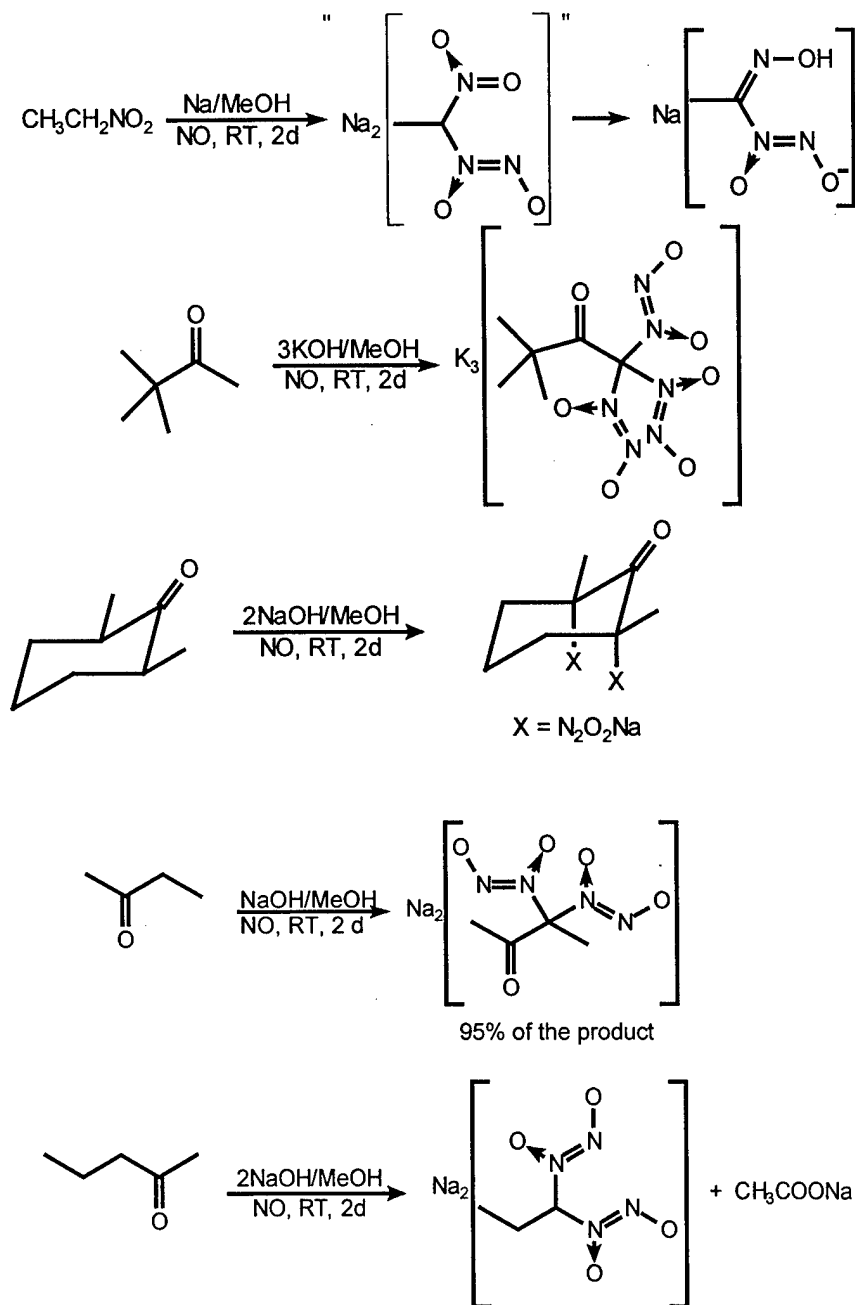


N-catenized species are somewhat small, but have surprisingly good thermal stability. A surprising feature of all of these species however is the marked differences in the stabilities of their cis/trans (Z/E) pairs. For example, there are now known diesters of cis-hyponitrite, and until recently, there were no known examples of E-diazenium diolates.

Some of the representative reactions from these diazenium diolate reactions are shown on the following page, Scheme 1. As can be seen, activating groups in the form of carbonyl groups or nitro substituents are required, and in all of these cases the resulting diazenium diolate function has a Z, or cis, configuration.

In addition, all products are isolated as air stable crystalline solids which in general are pure, even after a simple filtration step from the reaction mixture. The cations can be readily exchanged by metathesis, for example with ammonium and hydrazinium cations. In many cases these reactions are accelerated by cooling them in an ice bath. This somewhat contra intuitive observation is most likely due to the increased solubility of NO in the solvent in these conditions, and the dependence of the reaction rate on the NO concentration. The thermal properties, as determined by DSC, of some of these species have been collected in Table 1.

### Scheme 1 Selected Reactions from our Emerging PolyDiazenium Diolation Chemistry

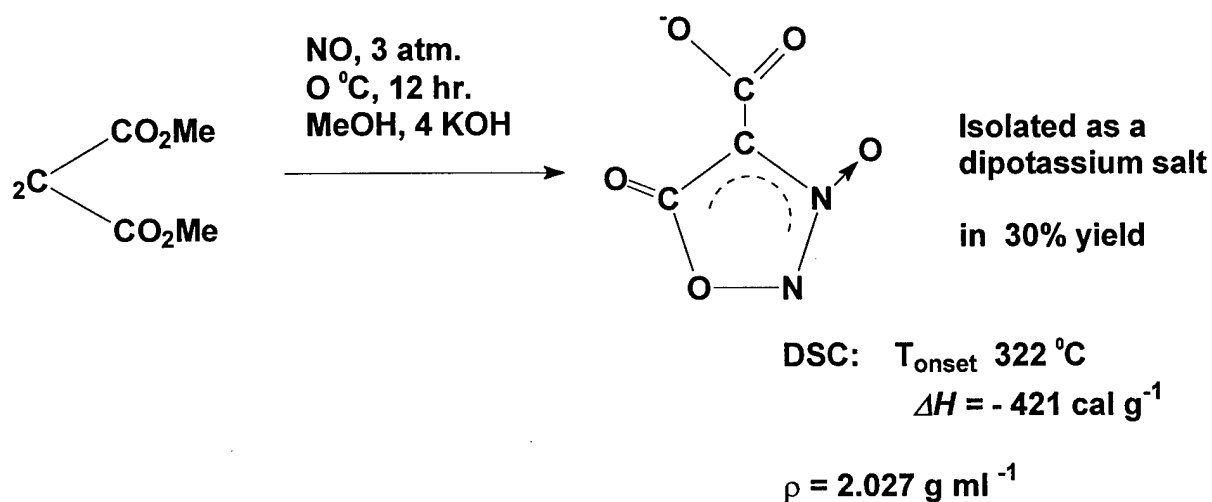


**Table 1 Thermal Decomposition Properties of Diazeniumdiolate Salts**

Compound	T <sub>onset</sub> , °C	T <sub>max</sub> , °C	ΔH, cal/g
CH(N <sub>2</sub> O <sub>2</sub> K) <sub>3</sub>	210.9	211.20	Explodes
Bu <sup>t</sup> COC(N <sub>2</sub> O <sub>2</sub> K) <sub>3</sub> ·3H <sub>2</sub> O	127.51 and 142.23	178.54 and 201.28	-339.60
CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> K) <sub>2</sub>	204.52	205.64	Explodes
CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> Na) <sub>2</sub> ·H <sub>2</sub> O	235.5	244.40	Explodes
CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> Li) <sub>2</sub> ·H <sub>2</sub> O	234.33	236.94	Explodes
[NEt <sub>4</sub> ] <sub>2</sub> [CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub> ]	172.10	177.24	-89.4
[N <sub>2</sub> H <sub>6</sub> ] <sub>2</sub> [CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub> ]	156.10	157.91	-35.6
[C(NH <sub>2</sub> ) <sub>3</sub> ] <sub>2</sub> [CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub> ]	130.00 and 196.14	149.40 and 200.88	-73.0
CH <sub>3</sub> CH(N <sub>2</sub> O <sub>2</sub> K) <sub>2</sub>	202.40	203.61	Explodes
CH <sub>3</sub> CH(N <sub>2</sub> O <sub>2</sub> Na) <sub>2</sub>	254.15	259.60	-144.1
CH <sub>3</sub> CH <sub>2</sub> CH(N <sub>2</sub> O <sub>2</sub> K) <sub>2</sub>	244.96	249.12	-341.3
CH <sub>3</sub> CH <sub>2</sub> CH(N <sub>2</sub> O <sub>2</sub> Na) <sub>2</sub>	211.29	230.32	-177.7
HOCH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH(N <sub>2</sub> O <sub>2</sub> Na) <sub>2</sub> ·2H <sub>2</sub> O	214.74	227.57	-190.4
CH <sub>3</sub> N <sub>2</sub> O <sub>2</sub> Na	294.60	332.40	-180.00
CH <sub>3</sub> CH <sub>2</sub> N <sub>2</sub> O <sub>2</sub> K	275.00	281.92	-254.2
Bu <sup>t</sup> N <sub>2</sub> O <sub>2</sub> K	275.00	284.58	-50.2
(CH <sub>3</sub> ) <sub>2</sub> C(N <sub>2</sub> O <sub>2</sub> K)(COOK)	266.96	270.65	-155.1
CH <sub>2</sub> (N <sub>2</sub> O <sub>2</sub> K)(COOK)	255.50	259.07	-442.1
CH <sub>3</sub> C(NOH)(N <sub>2</sub> O <sub>2</sub> Na)	214.10	224.69	-332.2

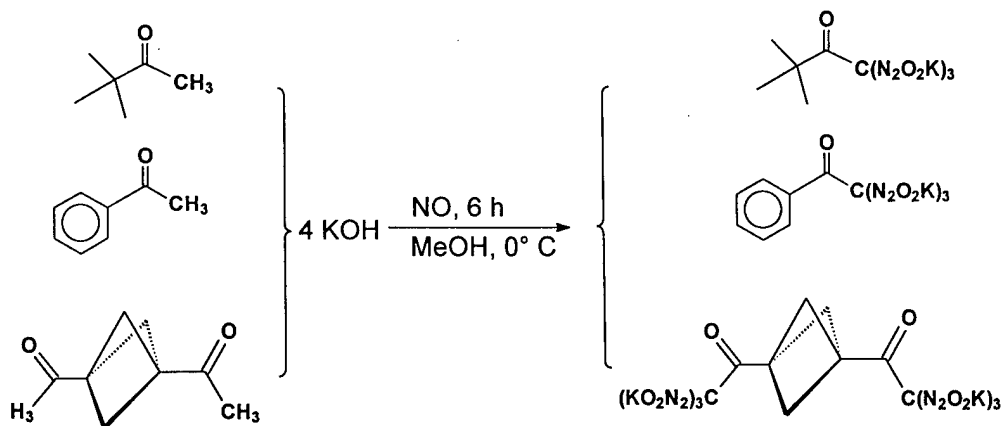
## Recent Accomplishments:

As highlighted during a recent site visit by the program director to our campus, we have some exciting recent findings which we are in the process of publishing and trying to understand. As noted above, all of the diazenium diolates we have described have a Z or cis geometry of the two oxygens. We have recently found an exception, and in this case an E, or trans, ONNO geometry is trapped out in a cyclic structure during the condensation of NO with dimethylmalonate:



A compound with these properties was briefly described by Traube in 1897, but the structure was wrong, the yield poor, and nothing further has been done with it. We find this result both fundamentally intriguing as well as practically important. Current efforts are underway to understand why in this case the Z/E isomerization is so facile; we hypothesize that this is due to stabilizing multiple bond interactions of the carbon bound NO group during the transition state. *Ab initio* theory is currently being applied to this question to examine these potential energy surfaces. On a practical level though, these cyclic structures results in very high crystal densities, and even though the value above is for a dipotassium salt, we believe that we will be able to prepare other salts by simple metathesis.

Finally, to illustrate how we now have predictive capability in these nitric oxide condensation reactions, the following new trisdiazenium diolates have been prepared:



The combination of two  $C(N_2O_2)_3$  groups into the bicyclic strained ring species is particularly significant, since these molecules have been examined as fuels for mixed propellant systems. In this case energy is stored in the ring as strain energy, and the oxidizer is potentially incorporated directly into the material, which is isolated as a stable salt.

### Summary:

We have prepared a family of new HEDM materials directly from inexpensive reagents in a single step. These compounds have excellent thermal properties and are being tested by our collaborators at Thiokol for inclusion in new propellant formulations.

### Personnel Supported:

Professor D. Scott Bohle, Principal Investigator  
Dr. Navamoney Arulsamy, Postdoctoral Associate  
Dr. Jerome Imonigie, Postdoctoral Associate  
Chris Barnes, Blaine Doletski, and Michael Smithback Graduate Students  
CJ Jones, Andrew Korich, Undergraduate summer research students

### Publications Resulting Directly from this Project: (Sept 1996-May 2001)

1. Traube's "Azoxymalonate salt" is a Sydnone with a *trans*- $N_2O_2$  structure. Navamoney Arulsamy, D. Scott Bohle *Angew. Chemie Submitted*.
2. Multiplicity Control in the Polygeminal Diazeniumdiolation of Active Hydrogen Bearing Carbons: Chemistry of a New Type of Trianionic Molecular Propeller. Navamoney Arulsamy, D. Scott Bohle, *Journal of the American Chemical Society, In Press*.
3. Chemistry of the Diazeniumdiolates 1. Structural and Spectral Characteristics of the  $[N(O)NO]^+$  Functional Group. Larry K. Keefer, Judith L. Flippen-Anderson, Clifford George, Anna P. Shanklin, Tandra M. Dunams, Danae Christodoulou, Joseph E. Saavedra, Elizabeth S. Sagan, and D. Scott Bohle, *Nitric Oxide: Biology and Chemistry*, **2001**,5,377-394.
4. Cyclohexadienone Diazeniumdiolates from Nitric Oxide Addition to Phenolates. D. Scott Bohle and Jerome A. Imonigie. *Journal of Organic Chemistry* **2000**,65,5685-5692.
5. Correlation of the Product E/Z Framework Geometry and O/O vs. O/N Regioselectivity in the Dialkylation of Hyponitrite. Navamoney Arulsamy, D. Scott Bohle, Jerome A. Imonigie, and Elizabeth S. Sagan, *Journal of the American Chemical Society*, **2000**,122,5539-5549
6. Nucleophilic Addition Reactions of Hydroxylamine, Methoxylamine, and Hydrazine with Malononitrileoxime. Navamoney Arulsamy and D. Scott Bohle, *Journal of Organic Chemistry* **2000**,65,1139-1143.

7. Radical Dinitroalkane Dianions from the Nitration of Nitroalkanes by Peroxynitrite. Ernst V. Arnold, D. Scott Bohle, Yangyan Hu. *Chemical Research in Toxicology* **2000**,*13*,963-966.
8. Synthesis and Thermal Decomposition Studies of New Nitroso- and Nitro-dicyanomethanide Salts, Navamoney Arulsamy, D. Scott Bohle, Blaine Doletski, *Inorganic Chemistry* **1999**,*38*,2709-2715.
9. Synthesis and Characterization of Alkylammonium Hyponitrites and Base Stabilized Hyponitrous Acid Salts. Navamoney Arulsamy, D. Scott Bohle, Jerome A. Imonigie, and Elizabeth S. Sagan, *Inorganic Chemistry* **1999**,*38*,2716-2725.
10. Synthesis and Thermochemistry of Stable Nitrogen Peroxides and Nitrite Pseudo-chalcogenide Salts, Navamoney Arulsamy, D. Scott Bohle, Bernhard Hansert, Patrick A. Sand, and Peter Schwerdtfeger, *Proceedings of the High Energy Density Materials Conference, 1997*. PL-TR-97-3057, ed. Pat Carrick **1998**, Air Force Research Laboratory, Edwards Air Force Base.

**Comprehensive List of Important Publications During This Project: (April 98 - May 2001)**

81. Multiplicity Control in the Polygeminal Diazeniumdiolation of Active Hydrogen Bearing Carbons: Chemistry of a New Type of Trianionic Molecular Propeller. Navamoney Arulsamy, D. Scott Bohle, *Journal of the American Chemical Society, In Press*.
80. Conformational Changes in Pediocin AcH Upon Vesicle Binding, and the Approximation of the Membrane-bound Structure in Detergent Micelles. Rachel M. Watson, Robert W. Woody, Randolph V. Lewis, D. Scott Bohle, Amy H. Andreotti, Bibek Ray, and Kurt W. Miller, *Biochemistry, In Press*.
79. Chemistry of the Diazeniumdiolates 1. Structural and Spectral Characteristics of the [N(O)NO] Functional Group. Larry K. Keefer, Judith L. Flippen-Anderson, Clifford George, Anna P. Shanklin, Tandra M. Dunams, Danae Christodoulou, Joseph E. Saavedra, Elizabeth S. Sagan, and D. Scott Bohle, *Nitric Oxide: Biology and Chemistry*, **2001**,*5*,377-394.
78. New Insights into the Metal Center of 3-Deoxy-D-arabino-heptulosonate 7-Phosphate Synthase, Peter A. Jordan, D. Scott Bohle, Cecilia A. Ramilo, and Jeremy N.S. Evans, *Biochemistry*, **2001**,*40*,8387-8396.
77. Synthesis, Structure, and Stereochemistry of Double-Chain Surfactant Co(III) Complexes. Navamoney Arulsamy, D. Scott Bohle, Patricia A. Goodson, David A. Jaeger, and Ven B. Reddy. *Inorganic Chemistry*. **2001**,*40*,836-842.

76. Functional Nickel Thiosalen Complexes: Structure and Solid State Packing of a Transition Metal Surfactant. D. Scott Bohle, Patricia A. Goodson, David a. Jaeger, and Abdullah Zafar. *Inorganic Chimica Acta* **2001**,312,210-214.
75. Salicylaldiminato Derivatives of Cyclotrimeratrylene: Flexible Strategy for New Rim-metallated CTV Complexes. *Inorganic Chemistry*, **2000**,39,5768-5770.
74. Malaria, Synchrotron Radiation, and Monte-Carlo. Peter W. Stephens, Silvina Pagola, D. Scott Bohle, and Andrew D. Kosar. *NSLS Newsletter* **2000**,7,1-4.
73. Cyclohexadienone Diazeniumdiolates from Nitric Oxide Addition to Phenolates. D. Scott Bohle and Jerome A. Imonigie. *Journal of Organic Chemistry* **2000**,65,5685-5692.
72. Correlation of the Product E/Z Framework Geometry and O/O vs. O/N Regioselectivity in the Dialkylation of Hyponitrite. Navamoney Arulsamy, D. Scott Bohle, Jerome A. Imonigie, and Elizabeth S. Sagan, *Journal of the American Chemical Society*, **2000**,122,5539-5549
71. Nucleophilic Addition Reactions of Hydroxylamine, Methoxylamine, and Hydrazine with Malononitrileoxime. Navamoney Arulsamy and D. Scott Bohle, *Journal of Organic Chemistry* **2000**,65,1139-1143.
70. The Structure of Malaria Pigment( $\beta$ -Hematin) Silvina Pagola, Peter W. Stephens, D. Scott Bohle, Andrew D. Kosar, and Sara K. Madsen, *Nature* **2000**,404,307-310.
69. Radical Dinitroalkane Dianions from the Nitration of Nitroalkanes by Peroxynitrite. Ernst V. Arnold, D. Scott Bohle, Yangyan Hu. *Chemical Research in Toxicology* **2000**,13,963-966.
68. Tetramethylammonium Superoxide and Tetramethylammonium Peroxynitrite. D. Scott Bohle and Elisabeth S. Sagan, *Inorganic Synthesis. In Press.*
67. Linear and Bent Oxo-bridged Dimers of Ruthenium Nitrosyls. D. Scott Bohle, Elisabeth S. Sagan, *European Journal of Inorganic Chemistry*, **2000**,1609-1616.
66. Synthesis and Characterization of Ni(II) S-Alkylthiosalen Complexes, D. Scott Bohle, Abdullan Zafar, Patricia A. Goodson, and David A. Jaeger, *Inorganic Chemistry*, **2000** 39,712-718.
65. Synthesis and Thermal Decomposition Studies of New Nitroso- and Nitro-dicyanomethanide Salts, Navamoney Arulsamy, D. Scott Bohle, Blaine Doletski, *Inorganic Chemistry* **1999**,38,2709-2715.

64. Theoretical, Thermal, and Coordination Chemistry of the Amphoteric Thiazate( $\text{NSO}^-$ ) Ion, Navamoney Arulsamy, D. Scott Bohle, Chen-Hsiung Hung, and Bryan D. Smith, *Inorganic Chemistry In Press*.
63. Synthesis and Characterization of Alkylammonium Hyponitrites and Base Stabilized Hyponitrous Acid Salts. Navamoney Arulsamy, D. Scott Bohle, Jerome A. Imonigie, and Elizabeth S. Sagan, *Inorganic Chemistry* **1999**,*38*,2716-2725.
62. Interrelationships between Conformational Dynamics and the Redox Chemistry of S-Nitrosothiols. Navamoney Arulsamy, D. Scott Bohle, Julia A. Butt, Peter A. Jordan, Elizabeth S. Sagan, *J. Am. Chem. Soc.* **1999**,*121*,7115-7123.
61. Cleavable Double-Chain Surfactant Co(III) Complexes. David A. Jaeger, Ven B. Reddy, and D. Scott Bohle, *Tetrahedron Letters* **1999**,*40*,649-652.
60. Reversible and Irreversible Hemichrome Generation by the Oxygenation of Nitrosylmyoglobin. Ernst V. Arnold, D. Scott Bohle, Peter A. Jordan. *Biochemistry* **1999**,*38*,4750-4756.
59. Crosstalk in the  $\text{ONOO}^-/\text{SCN}^-$  Mediated Oxidative Burst. Elizabeth S. Sagan, D. Scott Bohle, and Christopher M. Barnes, *Nitric Oxide: Biol. Chem.* **1998**,*2*,111.
58. Pathophysiological Chemistry of Nitric Oxide and its Oxygenation By-products. D. Scott Bohle, *Current Opinion Chemical Biology* **1998**,*2*,194-200.
57. Chemotactic Consequences of Nitric Oxide's Inhomogenous Diffusion Kinetics. Robert W. Atherton, Ernst V. Arnold, D. Scott Bohle, and Margaret M. Creech. *The Biology of Nitric Oxide* 6 eds. Moncada, S.; Toda, N.; Maeda, H, and Higgs, E.A.. Portland Press, London, **1998**, p. 290.
56. Enhancement of Sperm Motility by Nitric Oxide in Fathead Minnows. Margaret M. Creech, Ernst V. Arnold, Brian Boyle, Michael C. Muzinich, D. Scott Bohle, and Robert W. Atherton, *Journal of Andrology* **1998**,*19*,667-674.
55. Synthesis and Axial Ligand Substitution Chemistry of  $\text{Ru}(\text{TTP})(\text{NO})\text{X}$ ; Structures of  $\text{Ru}(\text{TTP})(\text{NO})\text{X}$ ,  $\text{X} = \text{ONO}, \text{OH}$ . D. Scott Bohle, Chen-Hsiung Hung, and Bryan D. Smith, *Inorg. Chem.* **1998**,*37*,5798-5806.
54. Aggregated heme detoxification by-products in Malarial Trophozoites:  $\beta$ -hematin and malaria pigment have a single  $S = 5/2$  iron environment in the bulk phase as determined by EPR and Magnetic M $\ddot{o}$ ssbauer Spectroscopy, D. Scott Bohle, Peter Debrunner, Peter A. Jordan, Sara K. Madsen, and Chuck Schultz. *J. Am. Chem. Soc.* **1998**,*120*,8255-8256.

53. Hydrophobic Control of Stereochemistry in the Synthesis of Double-Chain Surfactant Co(III) Complexes, David A. Jaeger, Ven B. Reddy, D. Scott Bohle, David W. Grainger, and B. Berggren, *Langmuir* **1998**,*14*,2589-2592.
52. Synthesis and Thermochemistry of Stable Nitrogen Peroxides and Nitrite Pseudo-chalcogenide Salts, Navamoney Arulsamy, D. Scott Bohle, Bernhard Hansert, Patrick A. Sand, and Peter Schwerdtfeger, *Proceedings of the High Energy Density Materials Conference, 1997*. PL-TR-97-3057, ed. Pat Carrick **1998**, Air Force Research Laboratory, Edwards Air Force Base.
51. Unusual nitrite complexes of iron(III) stabilized by the Tris(3,5-dimethylpyrazolyl)borate ligand; Synthesis and Characterization of Iron(III) and Iron(II) Nitrite Complexes, Navamoney Arulsamy, D. Scott Bohle, Bernhard Hansert, Annie K. Powell, Andrew J. Thomson, and Sigrid Wocaldo, *Inorg. Chem.* **1998**,*37*,746-750.
50. Extended rim redox active tris-metallodioxolene derivatives of cyclotricatechylene. D. Scott Bohle, and Dan A. Stasko, *Chem.Comm.* **1998**,567-568.

#### **Interactions/Transitions:**

- a. This past summer Andrew Korich, from Katherine Mondanaro's group at St. Michael's college in Vermont, spent eight weeks in our group and prepared two new trisdiazonium diolates.
- b. We have continued our collaboration with the HEDM group at Thiokol for testing the critical properties of our new salts.
- c. Results relating to this project have been presented at the HEDM conferences in Virginia(1997), Monterey(1998), Cape Caneveral(1999), and Park City(2000). In addition, these results have been presented at numerous Universities including: North Dakota State, Iowa State University, Colorado State University, and University of Utah.
- c. Transitions. It is very gratifying that Thiokol and SRI have active interests in these compounds. Dr. Thomas Highsmith visited our campus in 2000, and he gave a very well received seminar. The groups at Edwards Airforce Base have expressed an ongoing interest in our graduating students. There is a strong possibility that one of my more talented undergraduates may choose to do his Ph.D. with Dr. Christie. In many ways the training this lab provides is just as important as are our research results; where else can young American scientists get this type of training in HEDM synthesis?

## **Honors and Awards.**

### **During Grant:**

- 9/99 Early Promotion to Full Professor.
- 4/99 Extraordinary Merit for Research, University of Wyoming
- 7/97 Ed King Lecturer in Inorganic Chemistry, University of Colorado, Boulder.
- 5/96 Dreyfus Teacher Scholar Award from the Dreyfus Foundation.

### **Lifetime:**

- 9/99 Early Promotion to Full Professor.
- 4/99 Extraordinary Merit for Research, University of Wyoming
- 7/97 Ed King Lecturer in Inorganic Chemistry, University of Colorado, Boulder.
- 5/96 Dreyfus Teacher Scholar Award from the Dreyfus Foundation.
- 7/95 Humboldt Fellowship from Alexander von Humboldt Foundation(Declined).
- 5/95 Merit Award for Research, College of Arts and Science, University of Wyoming.
- 4/95 Hitchings-Elion Fellowship from the Burroughs-Wellcome Foundation.
- 4/95 Professor of the Year from the ACS Student Affiliate
- 1/95 Harmon Award for Arthritis Research
- 4/94 Cottrell Scholar Award from the Research Corporation.
- 10/89 Postdoctoral Fellowship from the National Institutes of Health.
- 7/89 Briggs Prize from the University of Auckland for Doctoral Thesis.
- 8/88 Postdoctoral Fellowship from the Universität Freiburg.
- 8/86 Student Paper Competition for the New Zealand Institute of Chemistry.
- 12/84 Postgraduate Scholarship from the University Grants Committee.
- 5/84 First Class Honours in MSc. degree examinations from the University of Auckland.