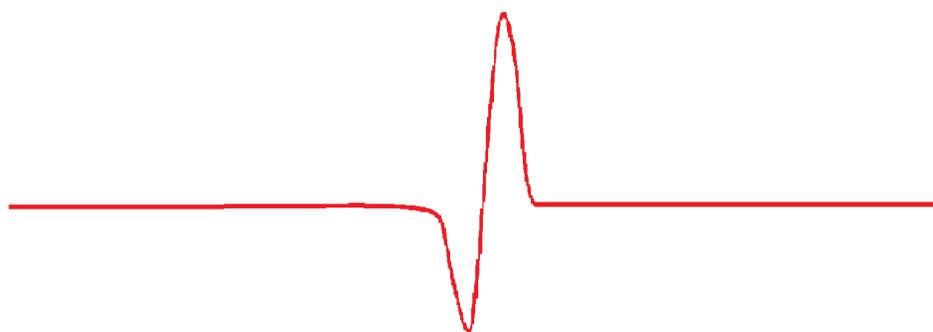


**22nd CTAS Annual Workshop
& Exhibition**



May 8-9, 2012

**Stage West Hotel
Mississauga, Ontario, Canada**



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Tuesday May 8, 2012

Training Session
Venue: Apache Room

- 8:00 – 9:00 *Registration*
- 9:00 – 10:00 **Oxidation Properties of “Solar Salt”**
Douglas Carson
INERIS, Parc Technologique Alata BP 2, F-60550 Verneuil-en-Halatte, France
- Open Forum*
- 10:00 – 10:45 **Introduction to Thermogravimetric Analysis (TGA)**
Steve Sauerbrunn,
Mettler-Toledo Inc., USA
- 10:45 – 11:15 *Exhibition, Posters & Coffee break (Seneca/Shawnee Rooms)*
- 11:15 – 12:00 **Hyphenated Thermal Analyses**
Charles Potter
TA Instruments Inc., USA
- 12:00 – 13:00 *Lunch (Muskoka Room)*
- 13:00 – 13:30 **Thermal and UV-curing Behaviour of Inks, Adhesives, and Coatings by Photo-DSC, In-situ DEA and DMA**
Peter Vichos
Netzsch Instruments North America
- 13:30 – 14:00 **Dissociation and Degradation of Supramolecular Solids: Recent Examples of Thermoanalytical Studies**
Dmitriy Soldatov
Department of Chemistry, University of Guelph



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Vendor Presentations

Venue: Apache Room

- | | |
|---------------|--|
| 14:00 – 14:15 | TA Instruments – Peter Schickel |
| 14:15 – 14:30 | Netzsch – Peter Vichos |
| 14:30 – 14:45 | Mettler-Toledo – Steve Sauerbrunn |
| 14:45 – 15:00 | C-Therm – Jarod Zhao |
| 15:00 – 16:00 | <i>Exhibition, Posters & Coffee break (Seneca/Shawnee Rooms)</i> |

Reception

Venue: *Seneca/Shawnee Rooms*

17:30 – 19:00



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Wednesday May 9, 2012

**Technical Session
Venue: Apache Room**

- 08:00 – 09:00 *Registration*
- 09:00 – 09:50 **Plenary Lecture**
Use of Calorimetric Methods to Characterize Chemical Process Hazards
Dr. Douglas Carson
INERIS, Parc Technologique ALATA, BP 2, F-60550 Verneuil-en-Halatte, France
- 09:50 – 10:00 *Open Forum*
- 10:00 – 10:25 **Optimizing DSC Performance**
Charles Potter
TA Instruments Inc., USA
- 10:25 – 10:50 *Exhibition, Posters & Coffee break (Seneca/Shawnee Rooms)*
- 10:50 – 11:15 **Synthesis of TiO₂/MWCNTs Composites Using a Supercritical CO₂ Aided Method for Photovoltaic Applications**
Donald McGillivray
University of Ontario Institute of Technology
- 11:15 – 11:40 **Physico-Chemical Properties of 2,4,6-tris (dimethylaminomethyl) Phenol in Water for the Development of CO₂ Capture Processes**
Yohann Coulier
Department of Chemistry, University of Guelph
- 11:40 – 12:00 Prize & next venue survey handout
- 12:00 – 13:00 *Lunch (Muskoka Room)*



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

- 13:00 – 13:25 **Characterization of Triacetoneperoxide**
Shanti Singh
Natural Resources Canada, Canadian Explosive Research
Laboratory, Ottawa, Ontario
- 13:25 – 13:50 **Polymer Glass Transition by RH-DMA**
Steve Sauerbrunn,
Mettler-Toledo Inc., USA
- 13:50 – 14:15 **Limiting Conductivities and Ion Association of Aqueous Sodium
Chloride Under Hydrothermal Conditions: New Experimental
Data and New Correlations**
Hugues Arcis
University of Guelph, Guelph, Ontario
- 14:15 – 14:40 **Thermochemical Production of Hydrogen: Thermal
Decomposition of Copper Oxchloride**
Liliana Trevani
University of Ontario Institute of Technology
- 14:40 – 15:05 *Exhibition, Posters & Coffee break (brief Executive meeting)*
- 15:05 – 15:20 **Transformer Liquids with Improved Thermal Conductivity
Measured by a Modified Transient Source Technique**
Adam Harris
C-Therm Technologies Ltd., Fredericton, NB, Canada
- 15:20 – 15:35 **Evolved Gas Analyses of Tires having been Heated to Explode or
Rupture**
Shanti Singh
Natural Resources Canada, Canadian Explosive Research
Laboratory, Ottawa, Ontario
- 15:35 – 16:10 *AGM / Student Awards / Announcements*



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Poster Session

A Comparison Between the Cylinder- and Capillary-Cell Nano-Differential Scanning Calorimeters for the Determination of Apparent Molar Heat Capacities of KCl

Alexander R. Lowe, J.S. Cox, and Peter Tremaine

University of Guelph, Dept Chemistry, 50 Stone Road East Guelph, Ontario, N1G 2W1

Deuterium Isotope Effects on Acid Ionization Under CANDU-6 Nuclear Reactor Conditions by Raman Spectroscopy

Michael B. Yacyshyn, Lucas M.S.G.A. Applegarth, Peter R. Tremaine

University of Guelph, 50 Stone Road East, Guelph, Ontario, N1G 2W1

Thermal Conductivity of Fruits as Revealed by the Modified Transient Plane Source Technique

Adam Harris, C-Therm Technologies Ltd., Fredericton, NB, Canada

Andre A. Levchenko, Advance AI-Thermo LLC, Princeton, NJ, U.S.A.

DSC CONSUMABLES

i n c o r p o r a t e d

High quality lab ready dsc sample pans

10% discount off of all orders placed in
2012 using promo code: CTAS

www.dscconsumables.com



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Training Session



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Oxidation Properties of “Solar Salt”

Douglas Carson and Wassila Benaissa

INERIS, Parc Technologique Alata BP 2, F-60550 Verneuil-en-Halatte, France

Solar Salt is a name sometimes given to a molten salt mixture made up of about 60% sodium nitrate (NaNO_3) and 40% potassium nitrate (KNO_3). This composition is near the eutectic point and is thermally stable until 600°C . It is popular in industrial solar energy projects and is used for storing energy in the form of heat to smooth out the peaks in electricity production. However for some technologies, combustible substances, like a thermal fluid for example, may come into contact with the molten salt. The aim of the paper is to study the oxidizing properties of the solar salt in order to estimate the energy released by a potential reaction between the salt and combustibles and estimate safety issues. In that purpose, four types of experiments were carried out: the standard UN O.1 test for solid oxidizers, the standard UN O.2 test for liquid oxidizers, differential scanning calorimetry (DSC) and isothermal calorimetry (C80). The experimental program demonstrates the oxidizing properties of the solar salt and shows that the reactivity of solar salt with other combustibles has to be taken into account in a global risk analysis of a Solar Energy Central.

Biography: Douglas Carson

Douglas Carson received his BSE in Chemical Engineering from the University of Michigan where he did undergraduate research work with Scott Fogler. His graduate work was done at the California Institute of Technology where he obtained his Ph.D. while in Bob Vaughan's research group applying solid state NMR techniques. After a post-doc in a CNRS laboratory, the "Institut de Recherches sur la Catalyse" near Lyon France, he joined the French National Coal Research Center (CERCHAR) to do research on coal gasification.

Since 1990, he has been with INERIS, the Institut National de l'Environnement Industriel et des Risques, where he formed the Thermal Stability Laboratory to characterize spontaneous combustion and dangerous properties of solids and liquids and then the Process Safety Laboratory using a wide range of calorimeters (DSC, C-80, ARC, ARSST, VSP, RC-1) to characterize chemical process hazards and to size Emergency Relief Systems for chemical reactors.

Besides the laboratory, his activities at INERIS include accident investigations, giving short courses concerning process hazards, and consulting work for industry and the French government in the areas of dust explosions and fires, chemical process safety, and the transport of dangerous goods.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Introduction to Thermogravimetric Analysis (TGA)

Steve Sauerbrunn
Mettler-Toledo Inc., USA

Thermal analysis is the measurement of a sample's physical properties as a function of temperature. This course is a beginner's guide to thermogravimetric analysis (TGA). Main topics covered will include: TGA design, effects of heating rates, effects of atmosphere, evolved gas analysis, kinetics of decomposition and controlled rate TGA to increase resolution of decompositions. Typical applications will be discussed for each technique and how to obtain optimal results. Throughout the course, 'tricks and tips' will be offered that will improve your design of TGA experiments and your evaluation of the resulting data.

Biography Steve Sauerbrunn, Ph.D.

Dr. Steve Sauerbrunn has worked in thermal analysis of polymers for about 27 years. He has authored hundreds of papers and two patents. Steve started in thermal analysis as an applications chemist with a major instrument manufacturer. After ten years, Steve moved to W.L. Gore & Associates. Steve was the Corporate Thermal Analysis Expert at Gore for seven years and specialized in the analysis of fluoropolymers. Steve has been with Mettler-Toledo for over ten years. He is the Technical Manager for the Mettler-Toledo analysis products.



CANADIAN THERMAL ANALYSIS SOCIETY
AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY
www.CTAS.org

Hyphenated Thermal Analyses

Charles Potter
TA Instruments Inc., USA

Biography Charles A. Potter, Ph.D.

Dr. Potter obtained his Ph.D. in analytical chemistry from the University of Connecticut. Since then he has worked in diverse research environments in thermal analysis and hazard analysis. He is currently a senior application scientist at TA Instruments, helping customers with TA problem solving.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Thermal and UV-curing Behaviour of Inks, Adhesives, and Coatings by Photo-DSC, In-situ DEA and DMA

Peter Vichos, Netzsch Instruments North America, Burlington, MA, USA

Dr. Stephan Knappe, NETZSCH-Gerätebau GmbH, Selb, Germany

Dr. Stefan Schmölzer, NETZSCH-Gerätebau GmbH, Selb, Germany

The main advantages of light- (normally UV-) curing systems are their fast reaction – within a few seconds at low isothermal temperature – and their absence of solvents. Often, a combination of thermal and light curing reactions is applied to dual cure adhesives or paints. Characterization of UV-curing process is needed to optimize materials and manufacturing processes. Ultraviolet (UV) light curing is a technology being applied increasingly in the fields of paints, inks, coatings, sealants, adhesives and dental composites. 1-component free radical UV systems such as acrylates can cure within some tenths of a second at room temperature. 1- or 2-component cationic epoxy resins exhibit a curing time ranging from just seconds to a few minutes. The advantages of UV resin systems are evident: Their high speed translates to high throughput, they have a low energy requirement (no heating is needed), and the lack of solvents eliminates ecological concerns. A variety of questions may arise during the UV curing process. How long does it take for the resin to begin UV curing? How high is the reactivity? How effective is the photo initiator? When is curing complete? How can the curing cycle be optimized? Is there any potential for post-curing?

The answers to questions such as these can be investigated by using thermal analysis methods as Photo Differential Scanning Calorimetry (UV-DSC), Dielectric Analysis (DEA) and Dynamical Mechanical Analysis (DMA), which are nowadays more and more used – not only in the laboratory environment, but also in-situ, i.e. in-process.

This paper gives an overview of the possibilities offered by these techniques in for paints, coating, and photovoltaic materials optimization.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Recent Examples of Thermoanalytical Studies

D. V. Soldatov

Department of Chemistry

University of Guelph, Guelph, Ontario, Canada

Supramolecular materials are based on weaker interactions such as Van der Waals forces and the hydrogen bond and therefore they dissociate upon moderate heating. This process can be accurately monitored using thermoanalytical instrumentation alone or combined with other techniques. Thermogravimetric Analysis is a very useful method to study the stoichiometry, stability and dissociation pathways of inclusion materials, where one component (guest) is much more volatile than the other (host). Differential Scanning Calorimetry is useful in detecting the corresponding phase transitions (such as incongruent melting). The reverse process of formation also can be monitored using the same instrumentation although this is a less common practice. Finally, irreversible changes can be registered to reveal more complex transformations caused by the temperature and pressure variations.

Examples of such studies where the nature of new materials created challenges in their characterization will be presented and approaches utilized or developed in our group to solve these challenges will be illustrated.

Biography Dmitriy V. Soldatov, Ph.D.

Dmitriy Soldatov was born near the city of Irkutsk in eastern part of Russia. He obtained his MSc degree in inorganic chemistry from Novosibirsk State University in 1991. His Ph.D. project, conducted at the Institute of Inorganic Chemistry (Russian Academy of Sciences, Novosibirsk) and the Institute of Physical Chemistry (Polish Academy of Sciences, Warsaw), was successfully completed in 1995. After that Dmitriy led a research group at the Institute of Inorganic Chemistry, acquired postdoctoral experience at the Steacie Institute, National Research Council Canada, and finally joined the faculty of the Department of Chemistry, University of Guelph in 2007, where he is now an Associate Professor.

Dmitriy has nearly 100 publications in refereed journals and editions (1993-2010), received several awards including Margaret Etter Award from the American Crystallographic Association (2001), serves as Executive Editor for the Journal of Structural Chemistry (since 2006) and acts as Canadian contact for Cambridge Crystallographic Data Centre (since 2008).



CANADIAN THERMAL ANALYSIS SOCIETY
AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY
www.CTAS.org

Enter Your Name Below to Participate in the Prize Draw

Write Your Full Name

- Detach This Page -



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Technical Session Abstracts



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Plenary Lecture

Use of Calorimetric Methods to Characterize Chemical Process Hazards

Dr. Douglas Carson

INERIS, Parc Technologique Alata, BP 2, F-60550 Verneuil-en-Halatte, France

Douglas.Carson@ineris.fr

Prevention in the chemical process industry is based on taking appropriate steps to put into place organizational and technical “barriers” such that the potential hazards of a process do not degenerate into industrial accidents, either by preventing dangerous situations from happening or by mitigating the effects of an uncontrolled chemical reaction. It should be noted that a chemical process includes reactors, storage units, mixing units, dryers, transportation etc. In any case, the starting point in prevention is an understanding of the process, the chemical reactions that may take place, either wanted or unwanted, and especially the thermal aspects such as heats of reaction, heat transfer, and thermodynamic energies. Calorimetric methods and thermal analyses are the cornerstones of a true understanding of the thermal aspects of a chemical reaction, whether this information is used for prevention purposes or in the case of accident investigation.

In order to understand a chemical process, a certain number of parameters must be identified to obtain a correct model of the reacting system (open or closed system, heat transfer modes within the system as well as at the boundaries, thermodynamics, and mass transfer). This argument is somewhat evident. However, what is less evident is that our calorimeters and cells are themselves reactors and obey the same laws of physics and chemistry. It is quite necessary to understand the limitations of each calorimetric method and to know what our signal actually corresponds to. In other words, we must not occult the model of our particular calorimeter. Incorrect interpretation of results can lead to incorrect conclusions concerning the industrial process. Our thermal analysis data must be correctly extrapolated to the industrial process.

This paper deals with the comparison of different calorimetric and thermal analysis methods and their basic models (Frank-Kamenetskii, Semenov, Thomas, open or closed systems ...) for several types of commercially available calorimeters: a screening calorimeter (DSC 131), a Calvet calorimeter (C80), a reaction calorimeter (RC1), and various pseudo-adiabatic calorimeters (VSP 2, ARSST, and Phi-Tec 1) as well as isothermal oven tests. Particular attention is paid to thermodynamic energies such as evaporation. An exothermic reaction was selected as a case study: the reaction of acetic anhydride by methanol, a

system which has been well studied in the literature. This reaction is studied in the six calorimeters under similar operating conditions. The objective of this series of tests is to obtain the thermo-kinetics of the reaction with each apparatus and compare the results. The goal is to determine which calorimetric method can be used (or not be used) for what purpose with respect to the reactive chemistry hazard. This approach gives also the opportunity to illustrate and summarize the potential of the experimental tools used in a complete risk assessment of a particular chemical process. The result can also be used in the education field as a simple and complete model of a case study.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Optimizing DSC Performance

Charles Potter and Els Verdonck
TA Instruments Inc., USA

Many differential scanning calorimeters are under-calibrations, and even more are over-calibrated, but few are optimally calibrated. Over-calibration wastes time, and under-calibration generates marginal data which can also waste time. So how can the calibration of a DSC be optimize to provide high quality data, and not waste time. The individual value / moving range (i.e. X/MR) control charts provide a powerful statistical and graphical tool to optimize the performance of the DSC temperature and enthalpy calibration. The basics of the individual value / moving range control chart and its application to the temperature calibration of a DSC will be discussed.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Synthesis of TiO₂/MWCNTs Composites Using a Supercritical CO₂ Aided Method for Photovoltaic Applications

Donald McGillivray, Franco Gaspari, Liliana Trevani
University of Ontario Institute of Technology, 2000 Simcoe St North

In this study we investigate the syntheses of TiO₂ thin films and TiO₂/ multiwall carbon nanotube (MWCNT) photocatalysts using supercritical carbon dioxide (SC-CO₂) as reaction media. This study exploits the advantage of SC-CO₂ over conventional sol-gel methods due to its high diffusivity and low surface tension. The high pressure flow cell designed and constructed for these studies can reach temperatures and pressures well above the critical point of SC-CO₂ (31°C, 7.38 MPa). The cell can operate under isothermal conditions or as a high-pressure cold-wall deposition cell for the preparation of thin films. Different materials have been synthesized with the aims of improving the porosity of the films, reducing the TiO₂ particle size and improving the photocatalytic activity of carbon/TiO₂ materials. Materials have been characterized using different experimental techniques, scanning electron microscopy (SEM), powder X-ray diffraction (XRD), and thermogravimetric analysis (TGA). Preliminary results will be presented.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Physico-Chemical Properties of 2,4,6-tris (dimethylaminomethyl) Phenol in Water for the Development of CO₂ Capture Processes

Yohann Coulier^{1,2}, Karine Ballerat-Busserolles¹, J.-Y. Coxam¹

¹ Institut de Chimie de Clermont-Ferrand, équipe Thermodynamique et Interactions Moléculaires, ICCF-TIM, Université Blaise Pascal
UMR CNRS 6296, 63177 Aubière, France

² Department of Chemistry, University of Guelph
50 Stone Road East, Guelph ON, N1G 2W1 Canada

The reduction of CO₂ emissions in the atmosphere is currently one of the most challenging European programs. The classical method to separate CO₂ from other gases in industrial post-combustion effluents is to use absorption-desorption cycles based on acid-base reactions. This technique is used intensively in order to purify natural gases by absorbing CO₂ in water+ethanolamine (MEA) solutions [1]. The objective of this research is to study a new class of amines that undergo liquid-liquid phase separation. This property has the potential to reduce the cost of solvent regeneration as only the phase containing dissolved CO₂ will be treated [2]. In this presentation, the thermodynamic properties of 2,4,6-tris (dimethylaminomethyl)phenol in water are reported. Densities and excess molar volumes of solutions were determined at 288.15, 298.15 and 308.15 K as a function of composition using a vibrating tube densimeter. Liquid-liquid equilibrium phase diagrams [3] and specific heat capacities were determined using a technique based on temperature scanning calorimetry. The UNIQUAC [4] and NRTL [5] models were used to correlate liquid-liquid phase equilibria and heat capacity data and to predict the excess molar enthalpies of the solutions.

[1] H. Arcis, K. Ballerat-Busserolles, L. Rodier, J.-Y. Coxam, *Journal of Chemical & Engineering Data*, 56 (2011), 3351-3362.

[2] L. Raynal, P.-A. Bouillon, A. Gomez, P. Broutin, *Chemical Engineering Journal*, 171 (2011), 742-752.

[3] Y. Coulier, K. Ballerat-Busserolles, L. Rodier, J.-Y. Coxam, *Fluid Phase Equilibria*, 296 (2010), 206-212.

[4] D.S. Abrams, J.M. Prausnitz, *Statistical thermodynamics of liquid mixtures: A new expression for the excess Gibbs energy of partly or completely miscible systems*, American Institute of Chemical Engineers, 1975, 116-128.

[5] H. Renon, J.M. Prausnitz, *Local compositions in thermodynamic excess functions for liquid mixtures*, American Institute of Chemical Engineers, 1968, 135-144.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Characterization of Triacetoneperoxide

C. Badeen, H. Feng, R. Turcotte, S. Singh

Natural Resources Canada, Canadian Explosive Research Laboratory
Ottawa, Ontario

Triacetoneperoxide (TATP) is not viable for use in conventional explosive devices due to excessive sensitivity and instability during normal manufacturing processes. For these same reasons, and with access to the Internet for synthesis details, TATP has come into favour as an ingredient in *non-conventional* explosive devices. We present data obtained at CERL detailing some aspects of this instability, especially regarding thermal properties and sensitivity of this organic peroxide.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Polymer Glass Transition by RH-DMA

Dr. Steve Sauerbrunn
Mettler Toledo Inc., USA

The use of Dynamic Mechanical Analysis (DMA) to measure the glass transition of polymers is well known. The glass transition of hydrophilic polymers (such as cellulose and polyamide) is more difficult to measure since the T_g depends on the relative humidity in the oven of the DMA. This paper will describe a new humidity generator that controls the relative humidity (RH) in the DMA oven, even as the oven temperature changes. Data presented will demonstrate how to determine the T_g of a polymer during constant RH and ramping RH whilst the temperature is held constant.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Limiting Conductivities and Ion Association of Aqueous Sodium Chloride Under Hydrothermal Conditions: New Experimental Data and New Correlations

Hugues Arcis⁽¹⁾, G.H. Zimmerman⁽²⁾, and P.R. Tremaine⁽¹⁾

⁽¹⁾ University of Guelph, Guelph, Ontario, Canada

⁽²⁾ University of Bloomsburg, Bloomsburg, Pennsylvania, USA
harcis@uoguelph.ca

Frequency-dependent electrical conductivities of solutions of aqueous sodium chloride have been measured from $T = 298$ K to $T = 623$ K at $p = 20$ MPa, over a very wide range of ionic strength ($2 \cdot 10^{-5}$ to 0.17 mol \cdot kg $^{-1}$) using a unique high-precision flow-through AC electrical conductance instrument. Experimental values for the equivalent conductivity, Λ , were used to calculate molar conductivities at infinite dilution, Λ° , using the Fuoss-Hsia-Fernandez-Prini ("FHFP") and Turq-Blum-Bernard-Kunz ("TBBK") ionic conductivity models. The resulting values for the limiting conductivity and the ion association constant of NaCl, from this work and critically evaluated literature data above 277 K, were represented to within the combined experimental uncertainties, as functions of viscosity and solvent density, respectively. New values and new correlations are reported for the limiting equivalent conductivities of the sodium ion, $\lambda^\circ(\text{Na}^+)$, and the chloride ion, $\lambda^\circ(\text{Cl}^-)$ from 277 K and 100 kPa to 1073 K and 500 MPa. The new correlation provides an updated standard system for determining single-ion transport properties from experimental electrolyte conductivity data at elevated temperatures and pressures.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Thermochemical Production of Hydrogen: Thermal Decomposition of Copper Oxychloride

Allan Nixon, Magali Ferrandon, Matthew Kaye, Liliana Trevani
University of Ontario Institute of Technology, 2000 Simcoe St North

Among the numerous thermochemical cycles for production of hydrogen by splitting of water, the copper-chlorine (Cu-Cl) cycle is one of the most promising as it can operate at temperatures below 550°C. In this work, thermal gravimetric and gas analysis of the reaction products were used to investigate the mechanism of decomposition of Cu_2OCl_2 , a key intermediate in the Cu-Cl cycle. New evidence is presented that confirms that the mechanism of decomposition of Cu_2OCl_2 in argon and air is similar and it involves several reaction steps and intermediates, including CuCl_2 , CuO , CuCl , and Cl_2 .



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Evolved Gas Analyses of Tires having been Heated to Explode or Rupture

B .Acheson, L. McCauley, P. Stanish, S. Singh, R. Turcotte
Natural Resources Canada, Canadian Explosive Research Laboratory
Ottawa, Ontario

Trials to simulate tire explosions/ruptures were performed at the Canadian Explosives Research Laboratory. Analyses of evolved gases were performed both in-situ (real-time) by quantitative FTIR, and ex-situ, after collection in canisters, by quantitative GC-MS. Neither of these techniques is ideal for determining the evolved gas behaviour of heated tires, as difficulties exist with both. To gain more information on evolved gases and tire decomposition behaviour, we cored samples of tires, after failure, and analyzed by DSC-TG-FTIR-(MS) during thermal decomposition in air.



CANADIAN THERMAL ANALYSIS SOCIETY
AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY
www.CTAS.org

Poster Session Abstracts



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

A Comparison Between the Cylinder-and Capillary-Cell Nano-Differential Scanning Calorimeters for the Determination of Apparent Molar Heat Capacities of KCl

Alexander R. Lowe, J.S. Cox, and Peter Tremaine

University of Guelph, Dept Chemistry, 50 Stone Road East Guelph, Ontario, N1G 2W1 alowe01@uoguelph.ca

Standard partial molar heat capacities of solutes, C_p° , are important thermodynamic properties for extrapolating room temperature equilibrium constants to elevated temperatures and pressures, and as a sensitive indicator of solute-solvent and solute-solute interactions. Their determination requires measurement of the specific heat capacity, relative to water, to a precision of $\pm 0.00001 \text{ J K}^{-1} \text{ mol}^{-1}$ or better. The only two commercial instruments with this sensitivity are the Sodev Picker Calorimeter, which is no longer manufactured, and the TA Nano-Differential Scanning Calorimeter. The procedure for the TA Nano-DSC, developed by Professor Earl Woolley at Brigham Young University, involves measuring the heat-capacity-density product of the solute relative to water, using the original version of the instrument which is equipped with cylindrical cells. TA also constructs a Nano-DSC equipped with tubular capillary cell whose operating principles are similar to the cylindrical-cell models, but is more robust and easier to clean. The purpose of this project was to compare the two instruments for this application over the range 5 to 120 °C, at 300 MPa, with identical solutions. The instruments were calibrated with water and a 1 molal NaCl standard solution using density and heat capacity reference values from Archer's equation of state (D.G. Archer, *J.Phys.Chem.* 1992, 21, 793-829). Values of the heat-capacity-density products of aqueous KCl (0.05 to 0.5 m) were measured and used to calculate the apparent molar heat capacities $C_{p\phi}$ for both heating and cooling experiments, and compared to the original results reported by B.A. Patterson et al. (*J.Chem.Thermo.* 2001, 33, 1237-1262). The experimental results (heat-capacity-density product) from the two instruments agreed with each other and literature values from Woolley to within $\pm 0.006 \%$. The results for $C_{p\phi}$ agreed to within $\pm 2.0 \text{ J K}^{-1} \text{ mol}^{-1}$, over the entire temperature range. We conclude that the two designs are equally suitable for apparent molar heat capacity measurements.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Deuterium Isotope Effects on Acid Ionization Under CANDU-6 Nuclear Reactor Conditions by Raman Spectroscopy

Michael B. Yacyshyn, Lucas M.S.G.A. Applegarth, Peter R. Tremaine
University of Guelph, 50 Stone Road East, Guelph, Ontario, N1G 2W1,
myacyshy@uoguelph.ca

This research reports preliminary measurements for the differences in ionization constants of weak acids and bases in light (H_2O) and heavy (D_2O) water under CANDU nuclear reactor operating conditions (250-300 °C, 10 MPa) using Raman spectroscopy. The deuterium isotope effect, $\Delta\text{pK} = \text{pKD}_2\text{O} - \text{pKH}_2\text{O}$, for bisulphate ionization has been determined for temperatures up to 200 °C by measuring the reduced isotropic Raman spectra. The integrated areas of the sulphate, SO_4^{2-} , and bisulphate, HSO_4^- , symmetric vibrational modes located at 980cm^{-1} and 1050cm^{-1} , respectively, are directly proportional to the molality (mol kg^{-1}) through the scattering coefficient, S . The corresponding D_2O vibrations occur at 980 cm^{-1} and 1060 cm^{-1} and were assumed to have identical scattering coefficients to the light water analog. The light water results for the ionization quotient, pQ , of bisulfate from $0.015 - 0.5\text{mol kg}^{-1}$ as a function of temperature were compared to potentiometric measurements and found to be within its combined experimental uncertainty (± 0.03), confirming the method. ΔpK results for the bisulphate ionization show a decrease of $0.3\ \Delta\text{pK}$ units between 25 °C and 200 °C.



CANADIAN THERMAL ANALYSIS SOCIETY

AN AFFILIATE OF THE NORTH AMERICAN THERMAL ANALYSIS SOCIETY

www.CTAS.org

Thermal conductivity of Fruits as Revealed by the Modified Transient Plain Source Technique

Adam Harris, C-Therm Technologies Ltd., Fredericton, NB, Canada
Andre A. Levchenko, Advance AI-Thermo LLC, Princeton, NJ, U.S.A.

In the paper we present the thermal conductivity data on selected foodstuffs, e.g. fruits and honey. Predictions of freezing and thawing of food products and their ingredients rely on thermal conductivity data. We demonstrate that a modified transient plain source technique is applicable in food processing industry because it is a fast and nondestructive method of measuring thermal conductivity. Other well-established techniques such as a guarded hot plate method are not suitable for food samples due to the prolonged measurement time, water migration within the sample and necessity to have large samples. Thermophysical properties of food including heat capacity and thermal conductivity change with water content and temperature, thus, can be interpreted in terms of water present.