



20th CTAS Annual Workshop & Exhibition

**May 12 & 13, 2010
Stage West Hotel
Mississauga, Ontario, Canada**



CANADIAN THERMAL ANALYSIS SOCIETY

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Wednesday May 12, 2010

Training Session Apache Room

- | | |
|---------------|---|
| 8:00 – 8:30 | <i>Registration</i> |
| 8:30 – 10:00 | Good Weighing Practice (GWP®)
Ian Ciesniewski, Mettler-Toledo Inc. |
| 10:00– 10:30 | <i>Exhibition, Posters & Coffee break (Seneca / Shawnee Rooms)</i> |
| 10:30 – 12:00 | Biological Microcalorimetry
C. A. Potter, TA Instruments |
| 12:00 – 13:00 | <i>Lunch (Muskoka Room)</i> |
| 13:00 - 14:30 | Advanced applications in non-destructive testing through "skins" of material with the modified transient plane source (MTPS) thermal conductivity technique.
A.Harris, C-Therm Technologies Ltd |
| 14:30 – 15:15 | DSC-Raman: When two great technologies finally meet!
O. Savard, Perkin Elmer LAS Inc |
| 15:15- 15-45 | <i>Coffee break (Seneca / Shawnee Rooms)</i> |



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Vendor Presentations Apache Room

15:45 – 16:05	Netsch Instruments Inc.
16:05 – 16:25	C-Therm Technologies Ltd
16:25 – 16:45	TA Instruments
16:45 – 17:05	PerkinElmer LAS Inc
17:05 – 17:25	Mettler-Toledo Inc.

Social Event:

**Reception, Exhibition and Poster Presentation
17:30–18:00**

**20th Anniversary Celebration for CTAS:
*Dinner Theatre: “The Wedding Singer”
Stage West Hotel
18:30-23:00**

***Dress Code:**

Dress shirts, slacks/dress, blouse/sweater and skirt required. No jeans, sweat suits or running shoes permitted. We ask that all patrons adhere to this policy to avoid embarrassment at the door



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Thursday May 13, 2010

**Technical Session
Apache Room**

- 08:00 – 08:30 *Registration*
- 08:30 – 09:15 **Plenary Lecture**
**The Utilization of Thermoanalytical Methods and
Equipment to Study Advanced Materials Based on Weak
Interactions**
D. V. Soldatov, University of Guelph
- 09:15 – 09:30 **Open Forum**
Moderator: D. V. Soldatov, University of Guelph
- 09:30 – 09:50 **Comparison of Non-Isothermal Crystallization Kinetics of
Molded Parts Based on Colorants by DSC**
J. Browne and C. Potter, TA Instruments
- 09:50 – 10:10 **Thermosets: How to Avoid Incomplete Curing**
B. Greil, S. Sauerbrunn and R. Riesen
Mettler Toledo Canada
- 10:10 – 10:40 *Exhibition, Posters & Coffee break (Seneca/Shawnee Rooms)*
- 10:40 – 11:00 **Zero Curve Dilemma in Computer-Aided Cooling Curve
Analysis Technique**
D. Emadi, Qatar University (Qatar)
- 11:00 – 11:20 **Effect of Some Physical Parameters on Calorimetry
Measurements of Energetic Materials**
D. Laniel¹, H. Feng², S. Singh², S. Goldthorp², and R.
Turcotte², ¹University of Ottawa, ² Natural Resources Canada



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- 11:20 – 11:40 **Interpreting Thermal Analysis Results of Energetic Materials**
H. Feng, S. Singh, and R. Turcotte, Natural Resources Canada
- 11:40 – 12:00 ***Annual General Meeting.***
- 12:00 – 13:00 *Lunch (Muskoka Room)*
- 13:00 – 13:20 **Comparison of the Reactivity of Cement Mixtures Containing SCM or Waste Glass Using Thermal Analysis**
L. M. Federico¹, S. E. Chidiac¹, and L. Raki², ¹McMaster University, ²National Research Council Canada
- 13:20 – 13:40 **Application of Thermal Analysis for the Characterisation of Nanocomposites Derived from Clay Minerals**
S. Letaief and C. Detellier, University of Ottawa
- 13:40 – 14:00 **Characterization of Gold Nanoparticles by Thermal Analysis**
M. Iqbal and S. H. Eichhorn, University of Windsor
- 14:00 – 14:30 *Exhibition, Posters & Coffee break (Seneca/Shawnee Rooms)*
- 14:30 – 14:50 **Solid-State Intramolecular Cyclization of Hydrophobic Dipeptides: Solvent-Free Synthesis in a Thermogravimetric Analyzer**
C. T. Brown and D. V. Soldatov, University of Guelph
- 14:50 – 15:10 **Clathrate Formation in the System Leu-Ala–DMSO: Phase Diagram of the System and Thermal Stability of the Leu-Ala*DMSO Inclusion Compound**
A. Yazdani, V. V. Chirmanov and D. V. Soldatov, University of Guelph



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15:10 – 15:30

**Inclusion Compounds of Leu-Ala Dipeptide:
Synthesis, Crystal Structures and Thermoanalytical
Studies**

A. Yazdani, V. V. Chirmanov and D. V. Soldatov, University of
Guelph

15:30 – 15:50

High-pressure DSC: Pushing the limits.

A.A.Levchenko, Setaram Inc.

15:50-16:10

**Thermal, UV and FTIR Spectral Studies of Metal
Cinnamates**

S. C. Mojumdar¹, G. Madhurambal², B. Ravindran³ and M.
Mariappan⁴, ¹Department of Engineering, University of New
Brunswick, ²A D M College for Women (India), ³Anjalai
Ammal-Mahalingam Engineering College (India), ⁴RVS
College of Engineering and Technology, Karaikal 609 609



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Poster Session

The New Multi-Modal Calorimeter (MMC)

P. Vickos, Netzsch Instruments Inc.

Thermal Analysis Techniques for Battery Safety and Design Battery Technology

P. Vickos, Netzsch Instruments Inc.

Determination of the Oxidation Enthalpy of Pyrite

S. C. Mojumdar, I. Bylina and V. G. Papangelakis, University of Toronto

Synthesis and Characterisation of $\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ Nanoparticles

L. R. Gonsalves¹, S. C. Mojumdar² and V. M. S. Verenkar¹, ¹Goa University, (India), ²University of New Brunswick

Synthesis and Characterization of $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$ Nanoparticles Obtained by Auto Catalytic Thermal Decomposition of Carboxylato-Hydrazinate Complex

U. B. Gawas¹, V. M. S. Verenkar¹ and S. C. Mojumdar², ¹Goa University (India), ²University of New Brunswick

Spectral and Thermal Properties of Copper(ii) Carboxylates with Fatty Acid Chains and Their Enzothiazole Adducts

M. Pajtášová¹, D. Ondrušová¹, E. Jóna¹, S. C. Mojumdar², S. L'alíková¹, T. Bazyláková¹ and M. Gregor³, ¹University of trenčín (Slovakia), ²University of New Brunswick, ³Comenius university Bratislava (Slovakia)

Thermal, Microhardness and FTIR Studies of Bisthiourea-Urea Single Crystal

G. Madhurambal¹, M. Mariappan², B. Ravindran² and S. C. Mojumdar³
¹ ADM College for Women (India), ² EGS Pillay Engineering College (India), ³University of New Brunswick

Simultaneous DSC-Spectroscopy

S. Aubuchon and C. Potter, TA Instruments.

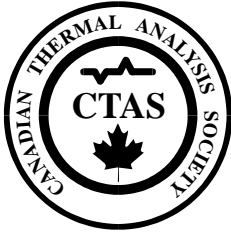


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ABSTRACTS

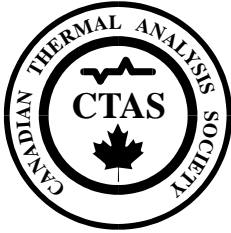


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Training Session



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Good Weighing Practice (GWP®)

Ian Ciesniewski
Mettler Toledo Inc.

A thorough approach to selection, use, calibration, and ongoing management of balances for your laboratory, department, or even entire organization.

Analytical weighing is a common and crucial part of obtaining accurate analyses.

It is a fact that while many users are well-versed in the techniques and nuances of their regular instrumentation, very few analytical balance users fully understand the process of electronic weighing, the science of metrology or the effects of sample, environment and user on the measurement uncertainty.

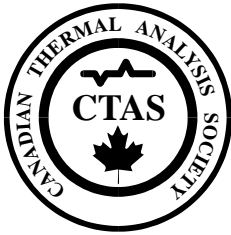
GWP is a risk-based approach to understanding how to choose the right balance, implement a scientifically correct metrology regime and minimize risk in the weighing process by identifying and dealing with sources of measurement uncertainty.

Biography

Ian Ciesniewski, Technical Director, Laboratory Weighing Division, METTLER TOLEDO Inc. has been with Mettler-Toledo since 1988. He is responsible for Technical Weighing Consulting, Education, Metrology and Technical Direction for the US Laboratory weighing market.. Ian holds a Masters' degree in Chemistry.

Ian started his career as a Research Chemist in the Chemical and Pharmaceutical industry in his native UK.

After Sales Positions with Mettler-Toledo in England, he relocated to Switzerland to run Technical Training Courses for Sales Representatives, Service Technicians and Customers, as well as being involved in new product development and developing strategies for Regulatory Compliance for Pharmaceutical and related industries. After several years in Switzerland, he moved to Mettler-Toledo Inc. in Columbus, Ohio. As Technical Director, he deals with regulatory consulting, technical development of MT's N.American Sales Force, Customer Training and MT's US Mass Metrology Program.



Biological Microcalorimetry

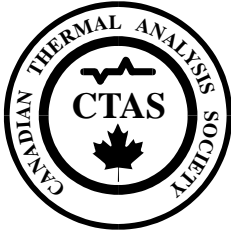
C.A..Potter

TA Instruments, New Castle Delaware, USA

Microcalorimetry encompasses several calorimetric techniques include large volume differential scanning calorimeters (DSC), isothermal titration calorimeters (ITC) and isothermal microcalorimeters (IMC) which are used in biological disciplines, medical research, food science, pharmaceutical development and many other industries. The large volume DSC is specifically designed for handling liquid samples such as proteins, nucleic acids, lipids, and enzymes. A common application is the formulation of biopharmaceutical liquid products to maximize storage stability. Isothermal titration calorimetry is used to study interactions of molecules, such as protein – ligand binding, surfactant micelle formation, and metal chelation. The energetics of intermolecular interactions, as determined by ITC, is extremely important in the biophysical study of sub-cellular functions. Isothermal microcalorimetry employs an ultra-stable thermostated bath to measure the small, nanowatt quantities of heat from microorganism growth, food spoilage, biological contamination of blood, and seed germination. A general overview of applications of biological microcalorimetry will be provided.

Biography

Dr. C. Potter obtained his PhD 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.



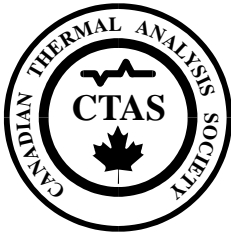
Advanced applications in non-destructive testing through "skins" of material with the modified transient plane source (MTPS) thermal conductivity technique.

A. Harris
C-Therm Technologies Ltd

In many applications it is desirable to test a material that is not directly in contact with the sensor face. Some materials to be tested are encased in a second material. Examples of such composite materials include polyurethane blown into a cavity of metal or plastic. This is the situation that arises in appliance and automotive manufacturing. In the past it has been necessary to cut the finished product, extract a foam sample and size this sample to the dimensions required for guarded hot plate testing. This is both expensive and time consuming. C-Therm's MTPS technique has been used to evaluate materials in this category. The versatile sampling parameters and adjustable test times are important features of C-Therm's technology and they can be combined with specific calibrations to blank out the outer material. The only requirement is the outer material must be deemed to be homogeneous and consistent in thickness. This is the case when testing through the metal door of a refrigerator to evaluate the polyurethane inside. To conduct any testing, the sensor must be calibrated with known materials. In this specialised application, the secondary skin material would be placed between the sensor and the calibration sample during the calibration tests. Parameters would be selected to assure the heat wave had passed through the skin and into the material being evaluated. Once the system was calibrated, it can be used online to test through the skin to evaluate the inner material.

Biography

Adam Harris, Managing Director (C-Therm Technologies Ltd.) – Adam has supported clients in the pharmaceutical, petroleum, ceramics, energetics and electronics industries over the past 8 years in evaluating the adoption of thermal instrumentation for product development and process control. Having contributed to a number of papers on application of the thermal effusivity and conductivity technologies in manufacturing and R&D environments, Adam is well respected for his business and technical acumen on the product line originally developed by Mathis Instruments. Adam graduated from the Dalhousie University School of Business Administration with a Bachelor of Commerce and has completed graduate courses at Harvard University and the University of New Brunswick.



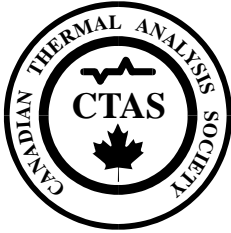
DSC-Raman: When two great technologies finally meet!

O. Savard
PerkinElmer LAS Inc.

Differential scanning calorimetry (DSC) and Raman spectroscopy are both commonly applied to the same problems, especially in looking at polymers and pharmaceutical materials. They complement each other because Raman spectra can provide qualitative information to supplement the quantitative information from DSC. For polymers, DSC and Raman spectroscopy are both used to investigate crystallinity but in rather different ways. DSC can determine the degree of crystallinity very precisely and can also follow the kinetics of crystallization by measuring the associated enthalpy changes. Raman spectra of crystalline and non-crystalline forms generally differ with the bands of the former being much narrower. Additional possibilities provided by Raman are to monitor very slow changes and also to identify situations where crystallization leads to a mixture of forms. For pharmaceutical products, simultaneous Raman and DSC measurements add a qualitative dimension to DSC data which simply measure heat flow. A typical example is the identification of polymorphs. Acetaminophen has several polymorphic forms which can interconvert with thermal treatment. A single Raman spectrum can identify a polymorph, while DSC uses a temperature scan to observe the relationship between forms that are not all stable at ambient temperatures. Simultaneous measurements are necessary to ensure that the Raman data correspond to the different stages identified by DSC.

Biography

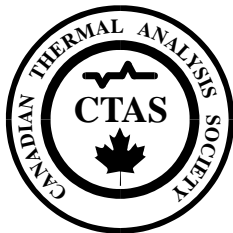
Olivier received his M.Sc. from Simon Fraser University (Burnaby, BC) in 2006 in the field of polymer chemistry after completing his B.Sc. in chemistry at Université Laval (Québec City). He then worked for a fuel cell company, as a research scientist, before joining PerkinElmer Canada in June 2007 as a product specialist for the material characterization product line (molecular and thermal analysis). In that role, his assignment is to provide technical training and support to PerkinElmer sales engineers, service engineers and customers in the related fields of thermal analysis (DSC, DMA, TGA, STA) and molecular spectroscopy (FTIR, UV/Vis, Microscopy, Imaging, Raman Spectroscopy) products throughout Canada.



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Technical Session



The Utilization of Thermoanalytical Methods and Equipment to Study Advanced Materials Based on Weak Interactions

D. V. Soldatov
University of Guelph

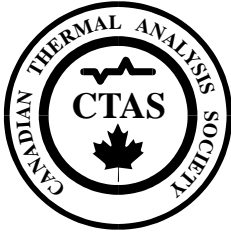
Thermal analysis is essential in the studies of inclusion materials and other supramolecular solids based on weak, intermolecular forces. Crystalline inclusion compounds comprise a lattice built by the molecules of host, and the molecules of guest included in the cavities of this host lattice. Due to the difference in volatility of the host and guest, the TG analysis is a convenient and very informative method to investigate the stoichiometry, stability and properties of inclusion compounds. On the other hand, the high organization of the materials sustained by weak interactions has a temperature limit dictated by the entropy term. Due to this fact, the DTA/DSC technique is an indispensable tool to identify this limit of stability and to provide an insight into the chemical nature, structure and dissociation mechanism of the studied materials. These studies complement preparative, thermodynamic and structural investigations and help to acquire comprehensive knowledge on the new advanced materials.

The application of the thermoanalytical methods and equipment has been an integral part of our studies on various materials conducted for over 20 years of research. The materials comprised inclusion compounds of Werner complexes, molecular and polymeric complexes based on metal bischelate units, as well as inclusion compounds and co-crystals based on oligopeptides. The studies on a number of new materials produced by our group at the University of Guelph are currently underway.

Biography

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 PhD 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.

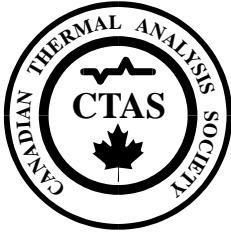
Dmitriy has nearly 100 publications in refereed journals and editions (1993-2010), received several awards including Margaret Etter Award from the American Crystallographic Association (2001), serves as Executive Editor for the Journal of Structural Chemistry (since 2006) and acts as Canadian contact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



Comparison of Non-Isothermal Crystallization Kinetics of Molded Parts Based on Colorants by DSC

J. Browne and C. Potter
TA Instruments

Injection molded parts made from polypropylene or its copolymers often contain nucleators - chemical agents designed to improve cycle time. One effect of the nucleator is to raise the crystallization temperature (T_c). Color concentrates often contain dispersing agents which will affect nucleators antagonistically and synergistically, in fact the colorants themselves can have a nucleating effect on polypropylene. In this investigation, commercial parts were purchased from a retail store and were studied non-isothermally to determine if processing differences can be expected based on the colorant. Empirical observations of Avrami parameters, activation energy (ΔE) calculated from the Avrami parameters as well as an isoconversional method are compared.



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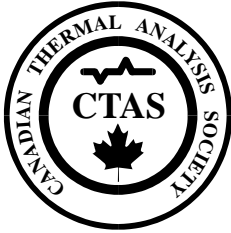
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Thermosets: How to Avoid Incomplete Curing

B. Greil, S. Sauerbrunn and R. Riesen
Mettler Toledo Canada

Oligomers and monomers that react and polymerize to form a rigid network with elevated temperatures are called thermosets. Thermosetting oligomers have been used for several decades to produce composite materials that have superior strength and reduced weight in comparison to metals. A question often heard is: “What is the fastest curing profile that I can use and still get superior polymer properties, i.e., complete cure?”

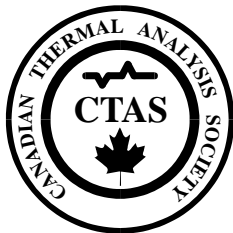
A time-temperature-transformation (TTT) diagram is useful for determining the optimum reaction conditions for a system that undergoes curing. The vitrification curve in the TTT diagram indicates the conditions under which the reaction practically stops. However, the corresponding material is not stable and can continue to cure in use. Modulated-temperature DSC is a method that allows the TTT diagram to be determined with high accuracy in a fraction of the experimental time required by conventional DSC.



Zero Curve Dilemma in Computer-Aided Cooling Curve Analysis Technique

D. Emadi
Qatar University (Qatar)

The computer aided cooling curve analysis (CA-CCA) technique has been used extensively to study solidification characteristics of metals and to predict the evolution of solid phases during solidification. The use of CA-CCA can be expanded to other areas of solidification, such as calculation of latent heat and solid fractions during solidification, if the zero curves can be calculated properly. The Newtonian and Fourier techniques of zero curve determination are described, and compared with a simple linear relationship. The latent heat calculated from different zero curve techniques were compared for an A-7%Si alloy to evaluate the accuracy of these techniques. It was concluded that a linear approach is relatively suitable for prediction of solid fraction, but it is not appropriate for latent heat predictions. The effects of test variables and sampling cup on the latent heat calculations were studied for different zero curve techniques.

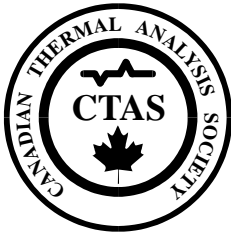


Effect of Some Physical Parameters on Calorimetry Measurements of Energetic Materials

D. Laniel¹, H. Feng², S. Singh², S. Goldthorp², and R. Turcotte²

¹University of Ottawa, ²Natural Resources Canada

To understand the thermal properties of energetic materials (EMs) it is quite often desirable to heat the sample through decomposition when using calorimetry, safeguarding the equipment from damage. Using ammonium nitrate (AN) as a test case, we evaluate results of AN decomposition when using differential scanning calorimetry (DSC), with differing degrees of sample confinement based upon the sample container. Other physical factors affecting decomposition pathway are the amount of sample (0.5-45 mg) and material of sample container. Comparison is made to the thermal decomposition of AN when in a totally confined container. Equilibria principles apply as pressure is generated, and we demonstrate that endothermic AN dissociation is a favoured decomposition pathway as the degree of venting increases. Using adiabatic calorimetry (ARC), we again explore the impact of varying sample mass and different levels of confinement on the dissociation of EMs. In addition, we show the consequences of the presence of an appropriate solvent on the activation energy (E_a) of the decomposition reaction. When studying decomposition using large sample masses in the ARC, knowledge of heat loss to the environment is desirable. Thus, sample thermal conductivity (k) and heat capacity (C_p) were obtained using a non-destructive transient plane source technique, which is a safe and convenient method to determine k for EMs. Also, results using ARC vessels having different C_p gave some insight into the thermal inertia of the system. We measure sample C_p values, at room temperature as well as above and at various water concentrations. Likewise, we demonstrate the importance of a precise k value when measuring the E_a with the adiabatic calorimeter.



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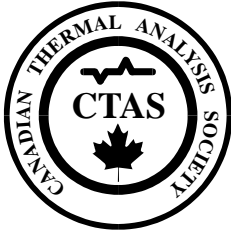
Interpreting Thermal Analysis Results of Energetic Materials

H. Feng, S. Singh, R. Turcotte

Canadian Explosives Research Laboratory, 1 Haanel Drive, Ottawa, Ontario, K1A 1M1

hongtu.feng@nrcan.gc.ca

Studies on the thermal decomposition kinetics of energetic materials often use instruments such as differential scanning calorimetry (DSC) and thermogravimetry (TG). Both these instruments may be operated under isothermal or non isothermal conditions, but an important difference is that for TG experiments the sample is required to be unconfined. It was found that the kinetic parameters of some energetic materials were quite different in closed and open systems. To illustrate this fact, we present results for the ammonium nitrate (AN) system, contained in either closed or open sample pans during the experiments. A comparison of experimental results using TG under isothermal and non isothermal conditions gave similar activation energies (E_a), as calculated by adapting ASTM E2070-08. However, when using confined samples and non-isothermal DSC data (ASTM E698-05) a different value of E_a was obtained. This result implies different thermal decomposition pathways for open and closed systems. AN was shown to endothermically dissociate after melting in an open DSC system, and exothermically decompose in a closed DSC system.



Comparison of the Reactivity of Cement Mixtures Containing SCM or Waste Glass Using Thermal Analysis

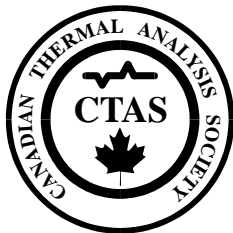
L.M. Federico*, **S.E. Chidiac****, and **L. Raki†**

*PhD Candidate, Department of Civil Engineering, McMaster University, Hamilton
federilm@mcmaster.ca, (905) 525-9140 x22044

** Department of Civil Engineering, McMaster University, Hamilton

†Institute for Research in Construction, National Research Council Canada, Ottawa

A laboratory study was undertaken to compare the performance of waste glass as a Supplementary Cementitious Material (SCM) to traditional SCMs at the same particle size and percent replacement in both high and low alkali cement paste. The results of the paste samples aged to 150 days are presented, and indicate that glass reactivity is similar to ground granulated blast furnace slag (GGBFS) and lower than silica fume (SF) at comparable particle sizes. The consumption of Ca(OH)_2 as measured by differential thermal analysis (DTA) is used as an indicator of reactivity. Alkali silica reaction (ASR) is not present for particle sizes below $100\ \mu\text{m}$, but is induced by agglomeration of the glass particles and is observed as fluorescence in optical microscopy images. Scanning Electron Microscope (SEM) imagery and Energy Dispersive Spectroscopy (EDS) is used to compare the microstructural properties of the SCMs and measure the chemical composition of the reaction product. Alkalinity of the cement influences the compounds observed by thermal analysis, and the temperatures at which their peaks occur.



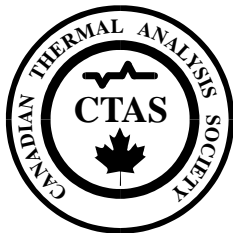
Application of Thermal Analysis for the Characterisation of Nanocomposites Derived from Clay Minerals

S. Letaief and C. Detellier

Center for Catalysis Research and Innovation and Department of Chemistry, the
University of Ottawa, Ottawa (On), K1N 6N5, Canada
Corresponding author: E-mail: sletaief@uottawa.ca

The combination of mineral matrices and organic compounds (ionic, molecular, monomer, polymer or macromolecular) exhibits multiple and varied interests. From a chemical point of view, it allows obtaining bi-functional materials, combining the chemical properties of the two partners. This combination leads to the modification of the physicochemical properties of the support mineral (properties of surface, property of adsorption, hydrophobicity, etc...) or may tend to immobilize and stabilize functional organic species. Among these nanohybrid materials, organo-clays constitute a versatile area of investigation, which is due in particular to the availability of natural clay minerals and their ability to incorporate a large variety of functional molecules, including polymers.

In this poster we present the application of thermal analysis (TGA and DTA) for a qualitative and quantitative characterization of different nanocomposites derived from Kaolinite, Sepiolite and Montmorillonite.

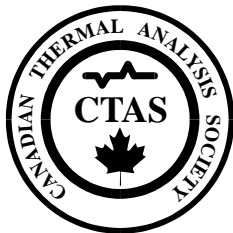


Characterization of Gold Nanoparticles by Thermal Analysis

M. Iqbal and S. H. Eichhorn

University of Windsor, Dept. of Chemistry & Biochemistry
Windsor, ON, N9B 3P4

Organic thiol ligands are most often used as a protective mono-layer on the surface of gold nanoparticles (AuNPs). Here, we report the synthesis and properties of AuNPs functionalized with alkyl selenolate, dialkyl dithiophosphate, and dialkyl dithiophosphinic acid ligands. All AuNPs are prepared by ligand exchange of dodecylamine protected AuNP precursors (2.0 ± 0.4 nm diameter) and their properties are compared to corresponding alkylthiolate protected AuNPs, which are prepared by the same method. Ligands exchange occurs without increase in size but the average core sizes of all AuNPs increases to about 3.5 nm during the purification as determined by Transmission Electron Microscopy. Differential Scanning Calorimetry is used to quantify the degree of side-chain crystallinity of the ligand layer and the thermal stability of the AuNPs. To our surprise, some AuNPs decompose in an exothermic and others in an endothermic reaction. Higher side-chain crystallinity results in higher thermal stability but, surprisingly, lower chemical stability towards decomposition with NaCN in THF solution. This result is explained with the presence of grain boundaries between crystalline domains. Preliminary results on the quantification of ligand stability by Isothermal Titration Calorimetry will also be presented.



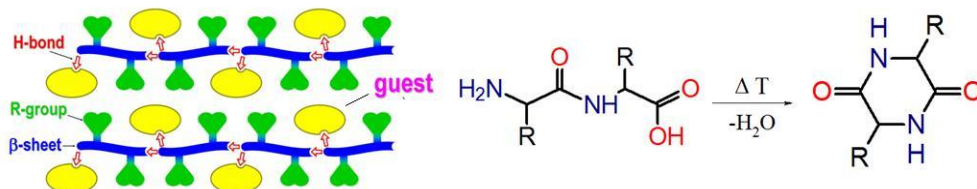
Solid-State Intramolecular Cyclization of Hydrophobic Dipeptides: Solvent-Free Synthesis in a Thermogravimetric Analyzer

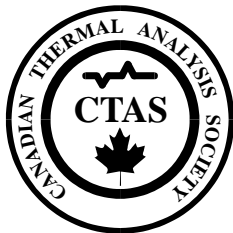
C. T. Brown and D. V. Soldatov
University of Guelph

Thermogravimetric analysis is an effective tool used in our lab to study solid dipeptides solvated with water or guest organic solvents. The solids are regarded as inclusion compounds (clathrates), where the dipeptide acts as a host building an H-bonded framework that includes the molecules of guest. The formation of inclusion compounds is driven by thermodynamics and reversible by heating. However, the heating frequently induces another, irreversible process characteristic of dipeptides: chemical reaction of intramolecular cyclization (condensation). The reaction produces one molecule of water and a volatile cyclic dipeptide that can sublime on further heating.

All these three processes can be monitored and controlled in the course of a thermogravimetric experiment. Thermal analysis of studied dipeptide inclusion compounds resulted in three distinct steps corresponding to (1) desorption of the guest molecules (mass loss reveals the amount of guest); (2) collapse of the host matrix accompanied by the intramolecular cyclization (mass loss of one mole of water); (3) complete sublimation or evaporation of the cyclic dipeptide product (total mass loss of the residue).

Therefore, the solid-state thermally induced cyclization is both a complicating factor in the dipeptide inclusions dissociation and a convenient, solvent-free synthetic route to cyclic dipeptides (2,5-deketopiperazines). The reaction was studied by preparation, thermoanalytical and spectroscopy methods.





**Clathrate Formation in the System Leu-Ala–DMSO:
Phase Diagram of the System and Thermal Stability of the
Leu-Ala*DMSO Inclusion Compound**

A. Yazdani, V. V. Chirmanov and D. V. Soldatov*
Department of Chemistry
University of Guelph, Guelph, Ontario, Canada

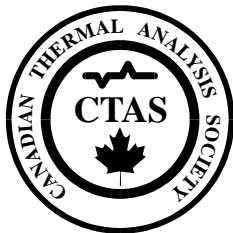
Inclusion compounds of hydrophobic dipeptides are prospective materials for pharmaceutical and biomedical applications [1], but little is known about their stability and conditions of their formation. The leucyl-alanine dipeptide (Leu-Ala) forms H-bonded layer structure that can act as a host matrix to form an inclusion compound with DMSO, where the guest DMSO molecules reside in the interlayer space [2].

Phase (x,T) diagram of the Leu-Ala – DMSO system was determined using thermoanalytical and preparation techniques in order to investigate stability, the limits of existence, and modes of decomposition of the inclusion compound. DSC was used to locate the solidus curve and TGA of liquor saturated at various temperatures was used to determine the solubility curve. In the region studied (0 to 100% Leu-Ala; -80 to 200°C), only one stable compound forms in the system which has the stoichiometry of Leu-Ala*DMSO and melts incongruently to produce liquor and solid Leu-Ala (guest-free phase). The inclusion compound was prepared independently by three different procedures and studied by DSC and TGA methods.

The phase diagram studies were complicated by irreversible cyclization of the dipeptide molecule at elevated temperatures (irreversible process), easy existence of the inclusion compound above the temperature of its incongruent melting (as a metastable phase), and abnormal reduction of the DMSO melting point by trace amounts of water.

[1] D.V. Soldatov "Soft Microporous Frameworks Based on Oligopeptides", in: Nanoporous Materials, World Scientific, NJ, 2008; pp 213-24.

[2] M. Akazome, A. Hirabayashi, K. Takaoka, A. Nomura and K. Ogura, Tetrahedron 2005; 61: 1107-13.



**Inclusion Compounds of Leu-Ala Dipeptide:
Synthesis, Crystal Structures and Thermoanalytical Studies**

A. Yazdani, V. V. Chirmanov and D. V. Soldatov*

Department of Chemistry

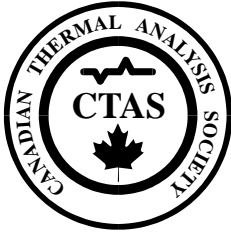
University of Guelph, Guelph, Ontario, Canada

Lower peptides may emerge as a new versatile class of host materials with future pharmaceutical, biomedical and other applications [1]. In crystals many hydrophobic dipeptides form 2D layered structures [2]. We are interested in using these clay-like structures as host matrices for guest inclusion and explore the prospects of the new materials that could be referred to as "peptide clays".

Inclusion compound with DMSO, several pyridines and other small molecules have been prepared and studied using thermal analysis techniques, X-ray crystallography and sorption experiments. Up to one mole of guest per mole of dipeptide can be accommodated in the space between the peptide layers. The conditions of formation, limits of existence and thermal dissociation properties of the inclusion compounds were investigated and the crystal structures of some of them were determined.

[1] D.V. Soldatov "Soft Microporous Frameworks Based on Oligopeptides", in: Nanoporous Materials, World Scientific, NJ, 2008; pp 213-24.

[2] C. H. Görbitz, Chem. Eur. J. 2007; 13: 1022-31.



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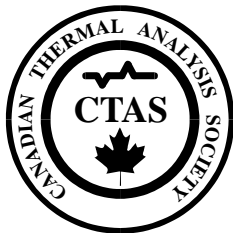
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High-pressure DSC: Pushing the limits.

A. A. Levchenko

Setaram Inc., Newark, California, U.S.A.

To mitigate the global warming, R&D is being directed towards understanding the relevant phenomena and foster innovation in the field of CO₂ capture and sequestration (CCS). Knowledge about the CO₂ sorption properties (adsorption capacity, pressure regimes, energetics and kinetics) is essential to the design of advanced materials capable of capturing CO₂ in industrial settings. Twinned Calvet calorimeters have been known for a long time, yet recent developments have opened the door to new applications under extreme conditions. This presentation covers practical aspects of high pressure Calvet calorimetry. New and published data will be presented to illustrate capabilities and advantages of the Calvet calorimeters for high pressure research with a special focus on CO₂ capture applications.



Thermal, UV and FTIR Spectral Studies of Metal Cinnamates

S. C. Mojumdar¹, G. Madhurambal², B. Ravindran³ and M. Mariappan⁴

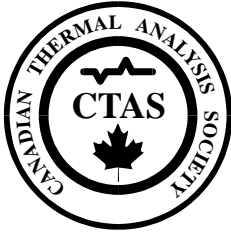
¹Department of Engineering, University of New Brunswick, Saint John, NB, E2L 4L5, Canada, E-mail: scmojumdar@yahoo.com, subhash.mojumdar@utoronto.ca

²A D M College for Women, Nagapattinam, Tamil Nadu, India

³Anjalai Ammal-Mahalingam Engineering College, Kovilvenni, Tiruvarur District, Tamil Nadu, India

⁴RVS College of Engineering and Technology, Kalikuppam, Karaikal 609 609

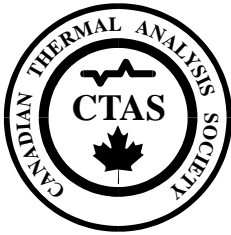
Cinnamic acid (3-phenyl-2-propenoic acid), a derivative of phenyl alanine, composes a relatively large family of organic isomers. In nature, cinnamic acid derivatives are important metabolic building blocks in the production of lignins for higher plants. Cinnamic acid possesses antibacterial, antifungal and parasite fighting abilities. Derivatives of cinnamic acid are important pharmaceuticals for high blood pressure and stroke prevention and possess antitumour activity. Cinnamic acid derivatives and flavonoids in wine create pigments and tannin agents that give each vintage its characteristic bouquet and colour. Cinnamic acid extensively studies not only due to its important biological activity, but also because of its specific structure. In the spectrum of cinnamic acid the carboxylic group is separated from the aromatic ring by a double bond. It causes conjugation between the C=C and the π electron system. Single crystals of sodium, potassium and mercury cinnamates were grown by slow evaporation of methanol solution at room temperature. The effect of metals on the electronic structure of cinnamic acid was studied. In this research many analytical methods such as FT-IR, UV, second harmonic generation (SHG) and TG-DTA were used. Incorporation of metals increases the thermal stability ensuring the suitability of material for possible non-linear optical (NLO) application up to 180°C.



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Poster Session



The New Multi-Modal Calorimeter (MMC)

P. Vickos

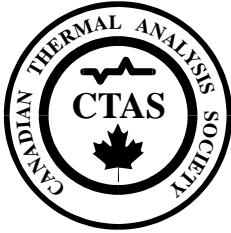
Netzsch Instruments Inc.

The Multi-Modal Calorimeter or MMC is a new bench top scanning calorimeter designed to measure the temperature, pressures, and energies related to physical and chemical changes of pure compounds and chemical mixtures quickly and efficiently.

The new calorimeter combines the characteristics of an adiabatic calorimeter with the speed and versatility found in a Differential Scanning Calorimeter (DSC.) This instrument uses a sample size larger than a DSC, about 1-3 grams, to accommodate sample addition and stirring. The larger sample size facilitates the measurement of multi-component and multi-phase sample systems. Sample injection and stirring are also possible.

Temperature and pressure are recorded as a function of time. Guard heaters are used to keep the sample and test cell in a near adiabatic environment, even at heating rates as high as 200°C/min. The power input from an immersion heater in the sample is carefully monitored and controlled. This patented method, developed initially for large adiabatic calorimeters, has been called Variphi \hat{O} . From the input energy we are able to measure endotherms, exotherms, and heat capacity, as well as temperature and pressure rates for determination of thermodynamic and kinetic parameters.

The modularity of the system allows for different calorimeter modules to be used on the same base electronics. The calorimeter can also be separated from the support systems allowing for operation of the unit in a glovebox, hood, bunker or environmental chamber.



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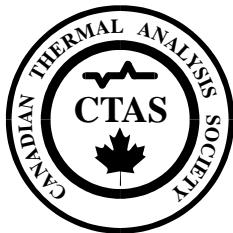
Thermal Analysis Techniques for Battery Safety and Design Battery Technology

P. Vickos

Netzsch Instruments Inc.

Recent Thermal Analysis Techniques for Battery Safety and Design Battery technologists have been very successful in creating rechargeable lithium ion batteries that have higher power density and greater performance. Although we all enjoy having a longer battery life on our portable electronic devices, these batteries are inherently more dangerous if a thermal runaway occurs. We probably have seen video of such batteries in laptop computers fail leading to fire and explosions. As the demand for the development of plug-in hybrids vehicles has turned to Li-ion cells, the need for the design and development of safe battery systems is more important than ever.

Thermal analysis techniques such as DSC, STA and adiabatic calorimetry have been used to study batteries and battery components. Testing individual material properties, the compatibility of materials used in battery cell as well as testing full battery cells has proven invaluable to the industry. There is a practical limit to using these techniques to study larger battery cells that would be use in areas such as electric cars. Other approaches, such as modeling or large scale thermal test platforms help researchers and engineers make good design choices and safer batteries. Thermal measurements outside of calorimetry also aid in the design of these cells in the ability to measure thermal transport properties and heat capacities. A review of all these techniques in battery development will be reviewed.



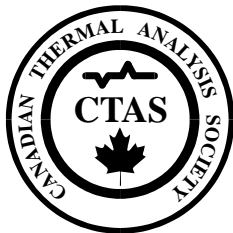
Determination of the Oxidation Enthalpy of Pyrite

S. C. Mojumdar, I. Bylina and V. G. Papangelakis

Department of Chemical Engineering and Applied Chemistry, University of Toronto,
200 College Street, Toronto, Ontario, Canada M5S 1A4, e-mail address:

subhash.mojumdar@utoronto.ca

Pyrite (FeS_2) and other sulfide minerals' oxidation data during storage in contact with air are very important for mining and metallurgical processes. Pyrite is associated with other sulphide minerals, which are major carriers of base metals such as nickel, copper and zinc. In the industrial practice, the oxidation of pyrite and its polymorph marcasite during storage under normal atmospheric conditions prior to Pressure Oxidation is also a known but not well-studied process. The purpose of this study was to evaluate the heats of oxidation of freshly ground vs. stored (for a year in contact with air) pyrite by calorimetric measurements. The present study was performed with a Setaram DSC80 equipped with a commercial mixing cell on pure pyrite mineral particles (Valdenegrillos, Spain) at 150 °C and partial oxygen pressure of 3.4 MPa. A calorimetric method to determine the enthalpy of Pressure Oxidation of sulfide minerals has been used to evaluate the enthalpies of oxidation of freshly ground and stored pyrite. Ground pyrite stored over time has long since been known to result in greater heat evolution during oxidative leaching. A likely mechanism for this phenomenon was uncovered: formation of ferrous sulfate and sulphuric acid during storage in contact with air influencing greater heat evolution at the outset of the reaction. It has been found that the heat capacity (C_p) for stored pyrite, 6.58 - 9.13 J/g K at 30 - 200 °C, is more than 10 times higher than the C_p for fresh grinded pyrite, 0.56 - 0.65 J/g K. The C_p distribution for fresh pyrite contains two exothermic peaks at 355 to 370 K. On the other hand, the C_p distribution for stored pyrite contains endothermic peaks at 355 to 420 K. The reason in the heat capacity difference is due to the presence of iron sulfate monohydrate and sulphuric acid formed during pyrite storage in contact with air. Simultaneous TG-DSC on a Q600 from TA Instruments has also been used to determine oxidation product $\text{FeSO}_4 \cdot \text{H}_2\text{O}$ and study the thermal stability of the pyrite and stored pyrite.



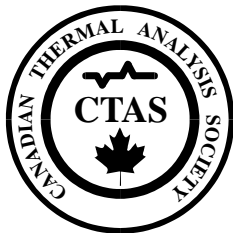
Synthesis and Characterisation of $\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ Nanoparticles

L. R. Gonsalves¹, S. C. Mojumdar² and V. M. S. Verenkar¹

¹Department of Chemistry, Goa University, Taleigao Plateau, Goa-403206, INDIA

²University of New Brunswick, Saint John, NB E2L 4L5 Canada, email:
scmojumdar@yahoo.com

Cobalt zinc ferrite ($\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$) nanoparticles have been synthesized via autocatalytic decomposition of the precursor, cobalt zinc ferrous fumarato hydrazinate. The X-ray powder diffraction of the 'as prepared' oxide confirms the formation of single phase nanocrystalline cobalt zinc ferrite nanoparticles. The thermal decomposition of the precursor has been studied by isothermal, thermogravimetric and differential thermal analysis. The precursor has also been characterized by FTIR, and chemical analysis and its chemical composition has been fixed as $\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2(\text{C}_4\text{H}_2\text{O}_4)_3 \cdot 6\text{N}_2\text{H}_4$. The Curie temperature of the 'as prepared' oxide was determined by AC susceptibility measurements.



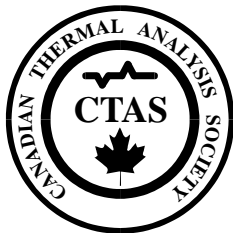
**Synthesis and Characterization of $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$ Nanoparticles
Obtained by Auto Catalytic Thermal Decomposition of Carboxylato-
Hydrazinate Complex**

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$\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$ nano-particles have been synthesized by self – propagating auto-combustion of nickel zinc ferrous fumarato-hydrazinate complex. The precursor complex has been characterized by chemical analysis, IR, AAS, thermal analysis and isothermal weight loss studies. The precursor on ignition undergoes self-propagating auto combustion to give $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$ nanoparticles. The X-ray diffraction studies confirmed the single phase formation of nanosize ‘as synthesized’ $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$. TEM observation showed the average particle size to be 20nm. Infra-red and magnetization studies were also carried out on the ‘as synthesized’ $\text{Ni}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$. The lower value of saturation magnetization and higher Curie temperature found from ac-susceptibility measurements of the ‘as synthesized’ ferrite also hints at its nano size nature.



Spectral and Thermal Properties of Copper(ii) Carboxylates with Fatty Acid Chains and Their Benzothiazole Adducts

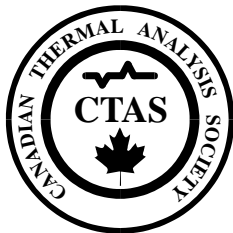
M. Pajtášová¹, D. Ondrušová¹, E. Jóna¹, S. C. Mojumdar², S. L'álíková¹, T. Bazyláková¹ and M. Gregor³

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²Department of Engineering, University of New Brunswick, Saint John, NB, E2L 4L5, Canada, E-mail: scmojumdar@yahoo.com, subhash.mojumdar@utoronto.ca

³Faculty of Natural Sciences, Comenius University Bratislava, Geological Institute, Mlynská Dolina, 84215 Bratislava, Slovakia

The carboxylates of the higher fatty acids with the metal ions have many applications in different industrial spheres as driers in paints and inks, the components of lubricating greases, stabilizers of plastics, catalysts, the additives at fuel, as well as the corrosion-preventive material, promoters of rubber-steel cord adhesion, etc. Their benzothiazole adducts can be utilized as antibacterial composites, inhibitors of endopeptidases, as the fast accelerators of sulphur vulcanization etc. The carboxylato-Cu(II) complexes of type $[\text{Cu}_2(\text{RCOO})_4]$ and their benzothiazole adducts $[\text{Cu}_2(\text{RCOO})_4\text{bt}_2]$ (bt = benzothiazole, R = $\text{CH}_3(\text{CH}_2)_{n-2}$, n = 12, 14, 16, 18) are the main object of this study. The studied carboxylato-Cu(II) complexes are formed from dimeric units to polymeric chains (chromofor CuO_5). The structural changes are due to coordination of ligand (benzothiazole), that the polymeric chains of carboxylato-Cu(II) complexes degraded to discrete centrosymmetric tetracarboxylate-bridged dimers (chromofor CuO_4N). These prepared compounds $[\text{Cu}_2(\text{RCOO})_4]$ and $[\text{Cu}_2(\text{RCOO})_4\text{L}_2]$ were studied by spectral (IR, UV-VIS) and thermal (TG, DTA, DSC) analysis.



Thermal, Microhardness and FTIR Studies of Bisthiourea-Urea Single Crystal

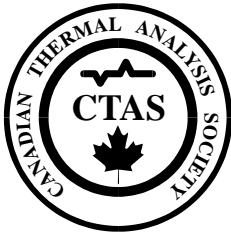
G. Madhurambal¹, M. Mariappan², B. Ravindran² and S. C. Mojumdar³

¹ ADM college for Women, Nagapattinam 611 001, Tamilnadu, India

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Nonlinear optic (NLO) frequency conversion materials have a significant impact on laser technology, optical communication and optical storage technology. The search for frequency conversion materials over the past decades has led to the discovery of many NLO materials with high nonlinear susceptibilities. However their often inadequate transparency, poor optical quality, lack of robustness, low laser damage threshold and inability to grow organic materials in practical device application. Hence, recent research is concentrated on organic materials due to their large nonlinearity, high resistance to laser induced damage, low angular sensitivity and good mechanical hardness. Recent metal complexes of thiourea and thiourea analogs have been investigated. Metal complex of thiourea, commonly called semiorganics, include the advantage of both organic and inorganic part of the complex. In this present work, mixed crystals of bisthiourea-urea (BTU) have been grown by slow evaporation technique at room temperature. The changes in the FTIR, X-ray diffraction (XRD), hardness parameters and thermal stability have been studied. Single crystals of a new nonlinear optic material, bisthiourea-urea have been grown from aqueous solution by slow evaporation technique with a period of 4 weeks. The structure of the grown crystals was confirmed by X-ray diffraction (XRD). The presence of title compound in the crystal lattice has been qualitatively determined by FTIR analysis. The mechanical properties and thermal stability of the grown crystals were evaluated by Vickers microhardness test and TG-DTA, respectively.



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Simultaneous DSC-Spectroscopy

S. Aubuchon and C. Potter
TA Instruments.

Hyphenated thermal analysis techniques with spectroscopic techniques continue to gain popularity. The thermal technique provides quantitation while complimented by the qualitative molecular information from the spectroscopic technique. For several decades, TGA/MS and TGA/IR have been widely used. DSC/Raman, DSC/NIR, and other thermal-spectroscopic techniques are now commercially available. The chemical reactions, phase changes, and polymorphic transitions are easily observed in pharmaceutical, thermoset, food, and other matrices by these hyphenated DSC techniques. A universal optical accessory for the DSC greatly facilitates implementation using NIR or Raman probes from different vendors.