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Thermochemistry and Chemical Hazard Potential

Sucrose and Sucrose Reactions

An ioMosaic® Publication

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Thermochemistry and Chemical Hazard Potential
Sucrose and Sucrose Reactions

Process Safety and Risk Management Practices

authored by

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1 Introduction

Thermochemistry is very important when exploring the hazard potential of a chemical decomposition or a chemical reaction [1, 2]. Accurate formation energies are essential for the evaluation of hazard potential and thermochemistry. Formation energies include the enthalpy of formation (ΔH_f), the Gibbs free energy of formation (ΔG_f), and the absolute entropy of formation (S_o).

Formation energies are typically reported at 25 °C (298.15 K) and 1 bara (standard conditions). Most simulation software databanks include ideal gas formation energies and some, such as SuperChems® Expert, include formation energies for liquid and solid phases as well.

There are many circumstances where thermophysical properties may not be available, especially when developing new molecules and new chemistries. When formation energies and heat capacity data are missing they can either be numerically estimated using group contribution and/or quantum chemistry methods or they can be partially measured and estimated semi-quantitatively.

This paper develops a novel method for the calculation of formation energies using a combination of quantum chemical methods, calorimetry measurements, and Gibbs free energy minimization.

2 Thermochemistry and Hazard Potential

In general, formation energies in the ideal gas phase are sufficient to evaluate hazard potential because phase change (fusion, vaporization, sublimation) energies are typically small compared to the formation energy absolute values. Public online databases such as the NIST WebBook and the NIST JANAF Tables contain formation energies for many chemical species and elements in the liquid, solid, and/or ideal gas phases.

Formation energies in the solid, liquid, and ideal gas phases are related using phase change energies (ignoring sensible heat):

$$\Delta H_{f,l} = \Delta H_{f,g} - \Delta H_{\text{vaporization}} \tag{1}$$

$$\begin{aligned}\Delta H_{f,s} &= \Delta H_{f,g} - \Delta H_{\text{vaporization}} - \Delta H_{\text{fusion}} = \Delta H_{f,l} - \Delta H_{\text{fusion}} \\ &= \Delta H_{f,g} - \Delta H_{\text{sublimation}}\end{aligned} \tag{2}$$

where $\Delta H_{f,l}$ is the enthalpy heat of formation of liquid at standard conditions in $\mathrm{J/kmol}$, $\Delta H_{f,s}$ is the enthalpy heat of formation of solid at standard conditions in $\mathrm{J/kmol}$, and $\Delta H_{f,g}$ is the enthalpy heat of formation of ideal gas at standard conditions in $\mathrm{J/kmol}$.

Numerous solids that are unstable will often decompose at temperatures close to their melting point. As a result, such solids will not have a physically meaningful heat of vaporization. Heats of reactions calculated using formation energies do not account for heat of solution and/or heats of dilution.

In general, solids that are unstable will start to decompose at temperatures close to their melting point.

One has to be careful when using published data for formation energies. Formation energy data

must be thermodynamically consistent before use in simulation software such as SuperChems® Expert Gibbs free energy minimization and constrained minimization modules. At standard conditions:

$$\Delta G_{f_{298.15K}} = \Delta H_{f_{298.15K}} - 298.15 \left[S_{o_{298.15K}} - \sum_i n_i s_{o_{i_{298.15K}}} \right] \text{ or} \quad (3)$$

$$S_{o_{298.15K}} = \frac{\Delta H_{f_{298.15K}} - \Delta G_{f_{298.15K}}}{298.15} + \sum_i n_i s_{o_{i_{298.15K}}} \text{ or} \quad (4)$$

$$\Delta H_{f_{298.15K}} = \Delta G_{f_{298.15K}} + 298.15 \left[S_{o_{298.15K}} - \sum_i n_i s_{o_{i_{298.15K}}} \right] \quad (5)$$

where $\Delta G_{f_{298.15K}}$ is the Gibbs free energy of formation in $\mathrm{J/kmol}$, $\Delta H_{f_{298.15K}}$ is the enthalpy of formation in $\mathrm{J/kmol}$, $S_{o_{298.15K}}$ is the absolute entropy of formation of the chemical or molecule in $\mathrm{J/kmol/K}$, n_i is the number of moles of element i in the molecule, and $s_{o_{i_{298.15K}}}$ is the absolute entropy of formation of element i in $\mathrm{J/kmol/K}$.

To guarantee thermodynamic consistency, ΔG_f must be calculated from ΔH_f and S_o values using the element formation energies of the simulation software or properties database. When heat capacity data is available, formation energies and thermodynamic equilibrium constants can be calculated at different temperatures.

3 Formation Energies Estimation Methods

3.1 Preferred Method

We can easily measure the Heat of Combustion of a chemical. Once the heat of combustion is measured, an absolute entropy (S_o) value is estimated from group contribution and/or quantum chemistry. The heat of combustion test is normally conducted under 400 psi of oxygen to cause complete combustion. An initial guess of the enthalpy of formation (ΔH_f) is provided. The Gibbs free energy of formation (ΔG_f) is then calculated directly from ΔH_f and S_o (see Equation 3). Interestingly enough, combustion reactions usually approach and equilibrium state. As a result, calculation of heats of formation from heat of combustion measurements using chemical equilibrium by direct minimization of the Gibbs free energy [3] is highly effective and reliable.

The heat of combustion is then calculated from direct minimization of the Gibbs free energy using simulation software such as SuperChems® Expert. The procedure is repeated until the calculated heat of combustion is equal to the measured heat of combustion. This method is highly reliable but it requires the availability of all potential combustion products and their formation elements in the software used. SuperChems® Expert contains a large database of potential combustion products for both organics and inorganics.

3.2 Alternative Methods

Adiabatic and/or differential scanning calorimetry are semi-quantitative methods that can be used to establish formation energies. Instead of measuring the heat of combustion, we measure the chemical heat of decomposition and/or heat of reaction using DSC, ARC, APTAC, or VSP2. Once the heat of decomposition or reaction is measured, an iterative procedure is used, similar to the iterative procedure that is used for heat of combustion. In this particular case we rely on constrained Gibbs free energy minimization to calculate the heat of decomposition or the heat of reaction. This method is also highly reliable but it requires thermodynamic data to exist for the all the reaction components (except the chemical in question) and/or decomposition products. Note that SuperChems® Expert enables the user to constrain the final equilibrium state to match additional measured data obtained by GC/MS or FTIR or the final pressure in the test cell after cool down. The adiabatic calorimetry test can also be simulated using SuperChems® Expert to validate the decomposition and/or reactions stoichiometries, phase behavior, and energetics.

Finally, quantum chemical estimates of formation energies and heat capacity data can be obtained directly from commercially available software such as Gaussian (see [4]) or research software such as GAMESS. Although the calculation of formation energies is somewhat cumbersome using Gaussian, both Gaussian and GAMESS can easily calculate heat capacity data and absolute entropy data that can be used with the semi-quantitative methods described earlier using heat of combustion or adiabatic calorimetry measurements.

4 Case Study: Sucrose Thermochemistry

On November 12, 2024, a reactor exploded at the Givaudan facility in Louisville, Kentucky. At the time of the incident, the reactor was in use to produce caramel coloring. The process data prior and up to the failure of the reactor [5] is shown in Figure 1. The measured rapid increase in temperature and pressure inside the reactor indicates that a runaway reaction took place starting at 2:40 PM.

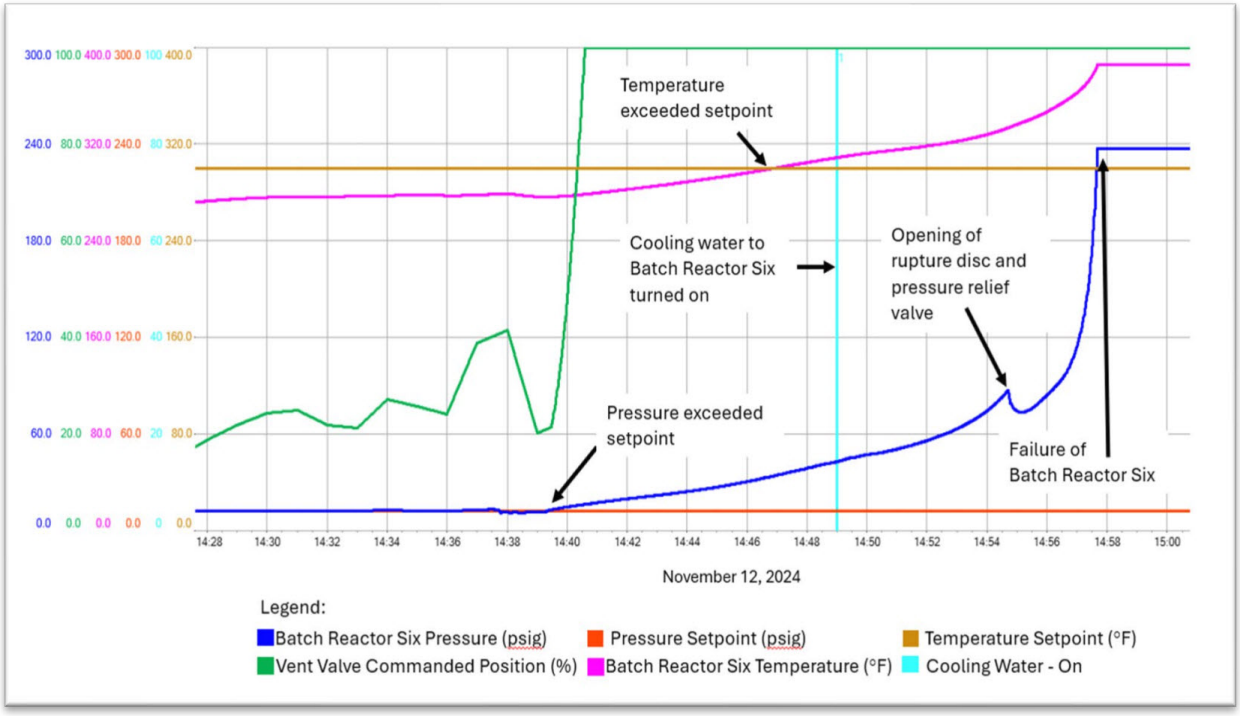
The main ingredient in reactor was “Invert Syrup” which contains sucrose and water. Sucrose is the chemical name for table sugar.

4.1 Sucrose Heat of Formation

The NIST WebBook reports a heat of combustion value of -5,638.0 $\mathrm{kJ/gmol}$ and an absolute entropy of 392.40 $\mathrm{J/gmol/K}$ for solid sucrose. ioKinetic® measured a heat of combustion of solid sucrose at -5,616 $\mathrm{kJ/gmol}$. GAMESS was used to calculate an ideal gas absolute entropy of 673.67 $\mathrm{J/gmol/K}$ at standard conditions. Using the measured heat of combustion value and the estimated absolute entropy value we calculated using SuperChems® Expert an ideal gas heat of formation of $-1,790$ $\mathrm{kJ/gmol}$ and an ideal gas free energy of formation of $-1,200$ $\mathrm{kJ/gmol}$. This is shown in Table 1. The calculated theoretical flame temperature was 2,277 K.

Since the formation energies of oxygen and nitrogen are zero, the heat of combustion of solid

Figure 1: Process data from batch reactor six leading up to the explosion



Source: CSB Investigation Report [5]

sucrose ($\Delta H_{c,s}$) can be expressed as:

$$\Delta H_{c,s} = \underbrace{\sum_i \Delta H_{f,i}}_{\text{combustion products}} - \underbrace{\Delta H_{f,s}}_{\text{sucrose}} = \underbrace{\sum_i \Delta H_{f,i}}_{\text{combustion products}} - \underbrace{\Delta H_{f,g} + \Delta H_{\text{vaporization}} + \Delta H_{\text{fusion}}}_{\text{sucrose}} \quad (6)$$

The heat of combustion of solid can be related to the heat of combustion of an ideal gas:

$$\Delta H_{c,s} = \Delta H_{c,g} + \Delta H_{\text{vaporization}} + \Delta H_{\text{fusion}} \quad (7)$$

4.2 Sucrose Hydrolysis

The NIST WebBook reports a liquid phase sucrose hydrolysis heat of reaction of -14.93 kJ/gmol (see Figure 2). The hydrolysis reaction of one mole of sucrose with one mole of water yields one mole of fructose (levulose) and one mole of glucose (dextrose) as shown below:

$$C_{12}H_{22}O_{11} + H_2O \xrightarrow{\text{slightly exothermic}} \underbrace{C_6H_{12}O_6}_{\text{Glucose}} + \underbrace{C_6H_{12}O_6}_{\text{Fructose}}$$

Using liquid formation energy for water and solid formation energies for sucrose, glucose, and fructose we calculate a heat of reaction of:

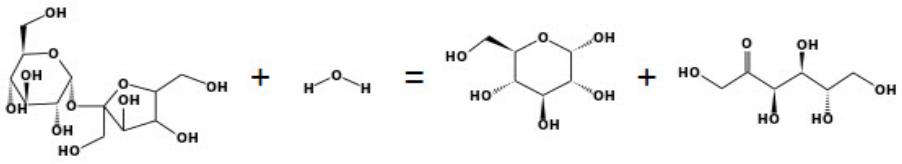
$$\Delta H_{rxn} = -1,271 - 1,265 + 2,221 + 285.8 = -29.2 \text{ kJ}$$

Table 1: Calculated combustion equilibrium data at standard conditions using SuperChems® Expert yielding -5,594 kJ/gmol of sucrose or -668 cal/g of mixture

		Hazard Rating	Heat of reaction	CART	Effective CART
** Energy and Volume Change	** Melhem Index	HIGH	-668.03 cal/g	2276.89 K	2399.85 K
Chemical Name	Formula	Phase	Initial Moles	Change	Final Moles
SUCROSE	C12H22O11	Vapor	0.0833	-0.0833	0.0000
OXYGEN	O2	Vapor	1.0000	-0.9996	0.0004
NITROGEN	N2	Vapor	3.7900	0.0000	3.7900
WATER	H2O	Vapor	0.0000	0.9163	0.9163
CARBON DIOXIDE	CO2	Vapor	0.0000	0.9996	0.9996
OTHER. < 0.0001 moles			0.0000	0.0000	0.0000
TOTALS			4.8733	0.8330	5.7063

Source: ioMosaic®SuperChems® Expert Combustion data by direct minimization of the Gibbs free energy.

Figure 2: Literature reported liquid phase hydrolysis reaction of sucrose



By formula: $C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6$

Quantity	Value	Units	Method	Reference	Comment
$\Delta_r H°$	-14.93 ± 0.16	kJ/mol	Eqk	Goldberg, Tewari, et al., 1989	liquid phase; solvent: Aqueous

Source: NIST WebBook.

Using ideal gas formation energies for all reactants and products, SuperChems® Expert calculates a hydrolysis heat of reaction of -166.6 kJ. We note that both heats of reactions using solid heats of formation and ideal gas heats of formation ignore heat of solution effects. This is consistent with the reported slightly exothermic value by NIST WebBook as shown in Figure 2. Note, adding phase change energies to the ideal gas calculated heat of reaction will increase the calculated value back to -29.2 kJ.

4.3 Dehydration of Fructose and Glucose

The dehydration reactions of fructose and glucose (dextrose) to 5-hydroxymethylfurfural (HMF) are endothermic based on literature and calculated formation energies data for HMF:

$$C_6H_{12}O_6 \xrightarrow{\text{endothermic}} \underbrace{C_6H_6O_3}_{\text{HMF}} + 3H_2O$$

The calculated SuperChems® Expert ideal gas heats of dehydration of fructose and glucose are 57.29 kJ/gmol and 12.08 kJ/gmol.

4.4 Acids Formation

HMF reacts with water to yield organic acids formation, such as Levulinic acid, Acetic acid, and Formic acid (see Table 2):

$$\underbrace{C_6H_6O_3}_{\text{HMF}} + 2H_2O \xrightarrow{\text{exothermic}} \underbrace{CH_2O_2}_{\text{Formic Acid}} + \underbrace{C_5H_8O_3}_{\text{Levulinic Acid}}$$

Table 2: Calculated acids formation equilibrium data at standard conditions using SuperChems® Expert yielding -162.90 kJ/gmol of HMF or -240.13 cal/g of mixture

		Hazard Rating	Heat of reaction	CART	Effective CART
** Energy and Volume Change	** Melhem Index	MEDIUM	-240.13 cal/g	876.30 K	876.30 K
Chemical Name	Formula	Phase	Initial Moles	Change	Final Moles
5-(HYDROXYMETHYL)FURFURAL	C6H6O3	Vapor	1.0000	-1.0000	0.0000
WATER	H2O	Vapor	2.0000	-2.0000	0.0000
FORMIC ACID	CH2O2	Vapor	0.0000	1.0000	1.0000
LEVULINIC ACID	C5H8O3	Vapor	0.0000	1.0000	1.0000
OTHER. < 0.0001 moles			0.0000	0.0000	0.0000
TOTALS			3.0000	-1.0000	2.0000

Source: ioMosaic®SuperChems® Expert Acids formation data by direct minimization of the Gibbs free energy.

4.5 Sucrose Decomposition

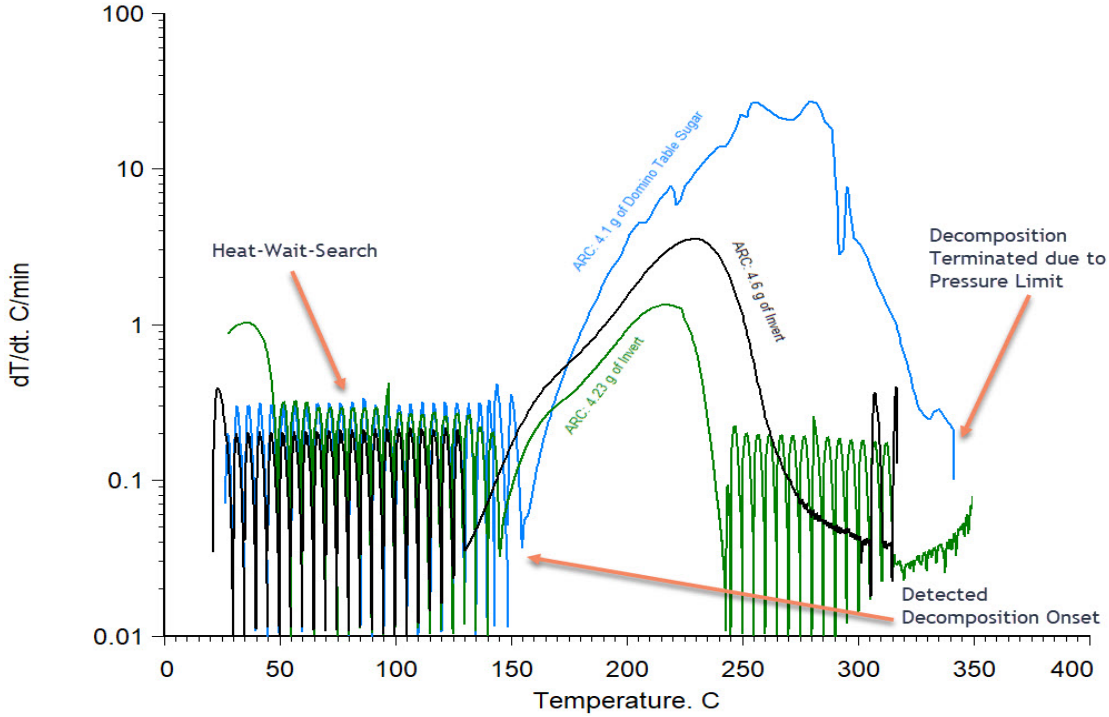
The overall equilibrium heat of decomposition is calculated by SuperChems® Expert to be -857 kJ/gmol of sucrose or -598.54 cal/g of sucrose as shown in Table 3. This calculated heat of decomposition represents an upper bound since the decomposition is predicted to proceed to a final equilibrium state with the most stable equilibrium products. This decomposition reaction does not reach the final equilibrium state as shown by adiabatic calorimetry and is rate controlled.

Table 3: Calculated decomposition equilibrium data at standard conditions using SuperChems® Expert yielding -857 kJ/gmol (-598.54 cal/g) of sucrose

		Hazard Rating	Heat of reaction	CART	Effective CART
** Energy and Volume Change	** Melhem Index	HIGH	-598.54 cal/g	986.62 K	2194.22 K
Compound Name	Formula	Phase	Initial Moles	Change	Final Moles
CARBON-REF	C	Solid	0.0000	10.3262	10.3262
SUCROSE	C12H22O11	Vapor	1.0000	-1.0000	0.0000
CARBON DIOXIDE	CO2	Vapor	0.0000	0.8369	0.8369
WATER	H2O	Vapor	0.0000	9.3262	9.3262
METHANE	CH4	Vapor	0.0000	0.8369	0.8369
OTHER. < 0.0001 moles			0.0000	0.0000	0.0000
TOTALS			1.0000	20.3261	21.3261

Source: ioMosaic®SuperChems® Expert Decomposition data by direct minimization of the Gibbs free energy.

Figure 3: Measured decomposition rates of sucrose and "Invert Syrup" using the ARC



Source: ioKinetic®.

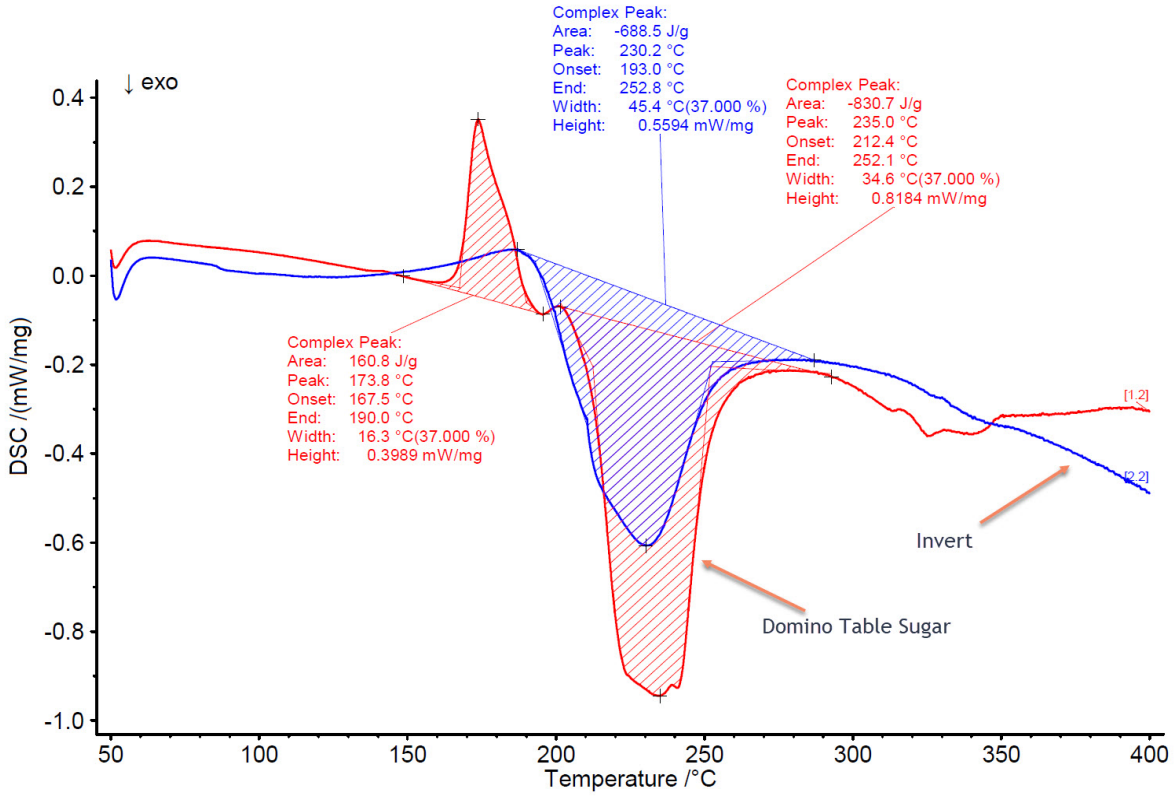
The measured rate of sucrose decomposition by adiabatic calorimetry is shown in Figure 3. We note that this measured rate is not for just sucrose (Domino table sugar) but for "Invert Syrup" as well, which is a mixture of sucrose (50 %) and glucose/fructose (50 %). "Invert Syrup" also contains 23 % water by weight according to its safety data sheet.

Using an integral heat capacity value for each ARC test shown in Figure 3 and correcting for thermal inertia of each test as well as water content we calculate the following heats of decomposition: -219 cal/g sucrose (4.1 g sample), -194 cal/g of sucrose (4.2 g sample) and -216 cal/g of sucrose (4.6 g sample).

A DSC measurement by ioKinetic® for table sugar (sucrose) (see Figure 4) shows a melting temperature from 167 to 190 °C and a heat of fusion of 161 kJ/kg. The NIST WebBook reports a melting temperature of 188.85 °C and a heat of fusion of 134.97 kJ/kg. The DSC measured heat of decomposition for table sugar (sucrose) is -199 cal/g (-831 J/g) of sucrose. The "Invert Syrup" DSC data corrected for water contents without phase change shows a heat of decomposition of -688.5/0.77 = -894 J/g (-213 cal/g of sucrose). Both DSC data sets are consistent with the ARC data sets.

DSC and ARC data yield a heat of decomposition for sucrose ranging from -194 cal/g sucrose to -219 cal/g of sucrose or an average of **-206.5 cal/g** (+/- 12.5 cal/g or +/-6 %) of sucrose. This agreement is excellent considering the data was generated using two different types of instruments with different measurement technologies. Average heat capacity data was used to calculate the adiabatic temperature rise from the ARC measurements without phase change corrections.

Figure 4: Table sugar (sucrose) heat of fusion and decomposition measurement by DSC



Source: ioKinetic®.

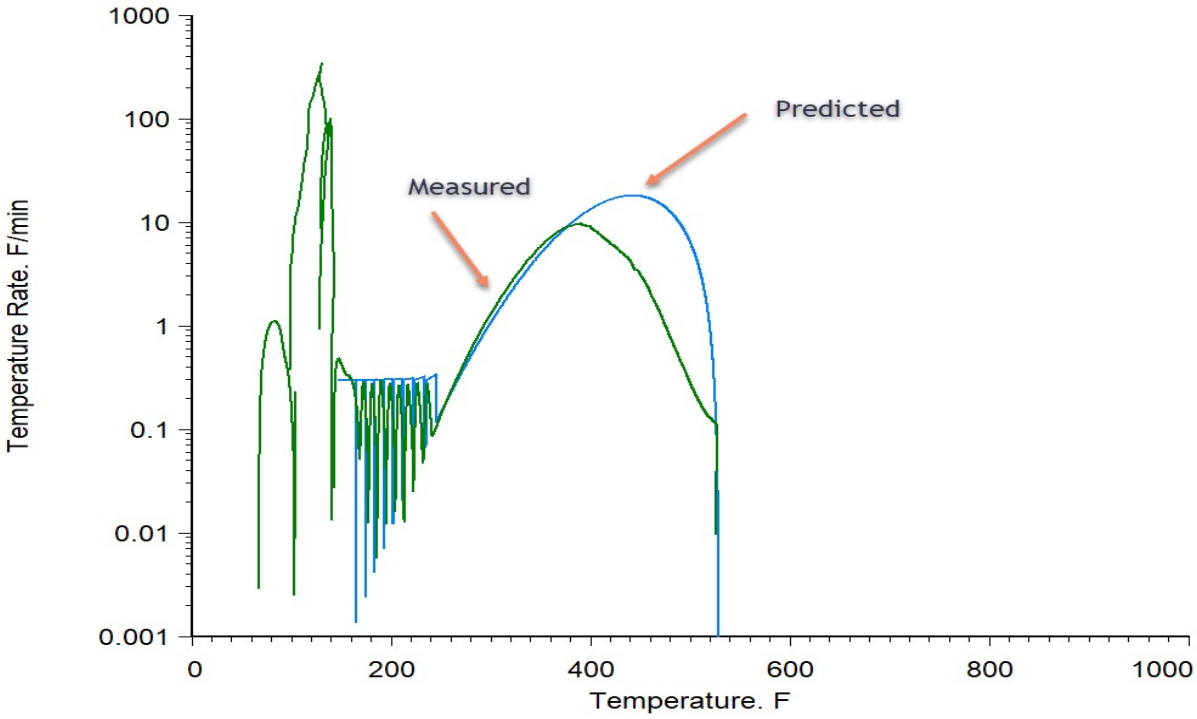
Ultimately, the best and preferred method of developing rate limited reaction stoichiometries and formation energies of complex decompositions is by dynamic simulation of the adiabatic calorimetry test with reasonable reaction stoichiometries and thermophysical properties as shown in Figure 5. The decomposition stoichiometry is developed by using constrained Gibbs free energy minimization as discussed earlier. Thermophysical properties are estimated using well established thermophysical properties estimation methods.

5 Calorimetry Data Reduction Tools

Calorimetry testing generates large volumes of data. Automated methods for calorimetry data reduction, visualization, scaleup, and analysis are essential for correct interpretation and use of such data. Process Safety Office® SuperChems® Expert includes a versatile and easy to use calorimetry data reduction, scaleup, visualization, and analysis utility. SuperChems® Expert can analyze calorimetry data from ARC, APTAC, VSP2, DSC, and TGA.

Some of the features of this utility include but are not limited to (a) the development of reaction rate models, (b) the development of isoconversion rate models, (c) time to maximum rate calculations, and (d) determination of self accelerating reaction or decomposition temperatures (SART/SADT). Site specific meteorological data sets can be used in the calculation of SART/SADT for a variety

Figure 5: Measured and predicted rate of temperature rise (dT/dt) vs. temperature (T) using the APTAC and SuperChems® Expert



Source: ioMosaic®

of vessel geometries. Both Semenov and Kamenetskii type estimates are available. Reduced data can be exported to SuperChems® Expert dynamic models for comparison, refinement, and use in runaway reaction dynamics simulations and/or the development of safe operating limits and relief requirements.

6 Conclusions

We have developed a novel method that combines Gibbs free energy minimization, quantum chemistry, and calorimetry measurements to calculate reliable formation energies.

Gibbs free energy minimization can be used with calorimetry measurements and computational quantum chemistry methods to provide valuable insights into the potential hazard of chemicals and chemical mixtures [1]. This information can be leveraged by dynamic simulation software such as Process Safety Office® SuperChems® Expert to gain additional insights into the likelihood of chemical hazards and risks, and for the development of relief requirements, safe operating limits for storage, processing, and transportation.

7 References

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About the Authors

Dr. Melhem is an internationally known pressure relief and flare systems, chemical reaction systems, process safety, and risk analysis expert. In this regard he has provided consulting, design services, expert testimony, incident investigation, and incident reconstruction for a large number of clients. Since 1988, he has conducted and participated in numerous studies focused on the risks associated with process industries fixed facilities, facility siting, business interruption, and transportation.

Prior to founding ioMosaic® Corporation, Dr. Melhem was president of Pyxsys Corporation; a technology subsidiary of Arthur D. Little Inc. Prior to Pyxsys and during his twelve years tenure at Arthur D. Little, Dr. Melhem was a vice president of Arthur D. Little and managing director of its Global Safety and Risk Management Practice and Process Safety and Reaction Engineering Laboratories.

Dr. Melhem holds a Ph.D. and an M.S. in Chemical Engineering, as well as a B.S. in Chemical Engineering with a minor in Industrial Engineering, all from Northeastern University. In addition, he has completed executive training in the areas of Finance and Strategic Sales Management at the Harvard Business School. Dr. Melhem is a Fellow of the American Institute of Chemical Engineers (AIChE) and Chair of the AIChE Design Institute for Emergency Relief Systems (DiERS).

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