

STUDY OF THE THERMAL BEHAVIOR OF HIGH-LEVEL WASTES USING THE LASER DRIVEN THERMAL REACTOR

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ABSTRACT

High-level wastes (HLW) consist of multiphase and multicomponent substances of which there is a dearth of information on their thermal behavior in a dynamic environment. A prime candidate technology for HLW treatment is thermal waste destruction, and vitrification for its disposal. The focus of this work was to study the thermal behavior, and develop an empirical model, of the thermal decomposition of an organic simulant HLW substance. Measurements were carried out using a laser driven thermal reactor (LDTR) at conditions representative of actual thermal treatment processes. The device provides reliable thermophysical and chemical kinetic data over a wide range of temperatures, heating rates, gas pressures, and environments. Unlike conventional thermal analysis techniques, which are designed to measure a selected thermal or chemical property in a nominally near-constant, equilibrium temperature environment, the LDTR measures an integrated thermal response. This integrated response represents the effects of multiple thermal and chemical properties in an environment characteristic of real-world reactors, where the temperatures are high, and rapidly changing. The LDTR was used as an off-line analytical device to characterize the thermal behavior of the organic high-level simulant waste PAS 94, under different thermal and physical conditions. The results showed a strong exothermic contribution of 2.9 kJ/g between 720 K and 870 K, which was virtually undetected in measurements obtained with a conventional differential scanning calorimeter. The thermal behavior of this waste was then used to develop a two-stage empirical model of the reaction kinetics. These data demonstrate the importance of heating rate and temperature on detection and characterization of HLW reactive behavior.

INTRODUCTION

Process-oriented technologies such as thermal treatment of chemical wastes, fuels, and propellants require data for optimization of system design and operation. These data include thermodynamic parameters and chemical kinetic rates of the processes involved. Groups of chemical reactions occurring during specific phenomenological processes can be identified as to their significance in the entire reaction scheme. These chemical reaction rates determine the course of the process. Once data on the rates of these processes are obtained, a technology can be optimized for given operating conditions. For this reason, the laser driven thermal reactor (LDTR) was developed to meet such demands, namely, to provide quantitative information for detection and characterization of the material thermal behavior of underlying physical phenomena, and optimization of thermal technologies. The LDTR can play an important role in

developing strategies to enhance chemical process efficiency, and control the formation of undesirable chemical byproducts.

Hanford high-level waste is a complex multiphase mixture of a variety of organic complexants, solvents, aging products, sodium nitrate, sodium nitrite, process additives such as iron hydrous oxides, sodium hydroxide, and a variety of other inorganic materials. The determination of the potential for radioactive release from Hanford tank wastes to the environment, as caused by a rapid release of chemical energy, is a difficult and complex problem. Recent studies have determined that a thermally accelerating runaway reaction is unlikely to occur. The remaining unresolved issue is whether a *high-energy initiator* such as via lightning, gasoline-originated fires, or the environment (e.g., melters for vitrification) can cause deflagration of HLW. How such waste mixtures will behave and react during exposure to a high-energy initiator, and what will be the chemical reaction byproducts, needs to be explored in a well-controlled environment with state-of-the-art diagnostics. The behavior of these waste substances can be dependent on several factors, such as the sample temperature and heating rate, properties of the surrounding gaseous environment, and more. The screening tool being used presently to determine the potential of the waste for such a reaction is the differential scanning calorimeter (DSC). The DSC and other conventional thermal analysis techniques (e.g., thermogravimetry) are appropriate for temperature levels up to 1000 K, heating rates up to a few degrees per minute, and small sample sizes less than 20 mg (1). The DSC, for example, is appropriate for modeling thermally accelerating reactions initiated by low energy events under equilibrium conditions. In high-temperature nonequilibrium applications (e.g., presence of a high-energy initiator), chemical processes may be completed (and reactants consumed) before the temperature level of interest is achieved with conventional approaches. An example is sodium nitrate and sodium nitrite, which decompose at 650 K and 590 K, respectively, but may explode above 810 K (2). With the DSC, the relatively slow heating rate and decomposition of these chemicals may result in their consumption before detection of their explosive behavior. Recorded DSC data will therefore not be representative of the thermal behavior of the sample at actual process conditions. Another point to note is that conventional methods do not determine directly the heat transfer (i.e., thermal loss) from tested samples, which affects significantly the accuracy of the data.

Data analyses with conventional techniques have been found to conceal thermal characteristics that occur at higher temperature, nonequilibrium situations because of material decomposition. High-energy initiators produce heating rates of several dozen to hundreds of degrees per second and temperatures in excess of 1300 K. High heating rates must therefore be used to reach these higher temperatures before completion of material decomposition and chemical reactions. The rapid attainment of high temperatures to initiate such reactions can be achieved by using a new approach, referred to as the laser driven thermal reactor, that is based on the programmable laser heating of materials. One can uncover such nonequilibrium characteristics, i.e., detect exothermic processes that are associated with reactions occurring at higher temperatures. Thus, analysis of HLW with the LDTR can be used to unravel the fundamental physical pathways associated with these processes.

Currently, no technology comparable to the LDTR is available that can perform at the required specifications. The unique features of the LDTR that surpass those of conventional thermal analysis methods are: rapid temperature sensing, programmable and rapid heating rates, quick data

measurement and analysis, simple design, robustness, controllable enclosed environment, high accuracy, and the ability to analyze from milligrams up to a few grams sample quantities. Larger samples allow greater flexibility in studying substance thermal behavior at actual process temperatures. These features also enable this technique to be used as an on-line sensing device. On-line sensing is especially important for monitoring inhomogeneous wastes with inorganic components (such as with halogenated species with low heating values), which retard the chemical reactions, or with energetic compounds that may greatly accelerate chemical reactions. Unlike conventional thermal analysis techniques, which are designed to measure a selected thermal or chemical property in a nominally constant temperature environment, the LDTR measures an integrated thermal response, representing the effects of multiple thermal and chemical properties. The LDTR makes these measurements in a dynamic environment characteristic of real-world reactors where the temperatures are high and changing rapidly.

This study demonstrates the ability of the LDTR to monitor accurately the thermal behavior of simulant HLW PAS 94. A simple empirical model of the sample chemical kinetic behavior is developed for different representative conditions.

LASER DRIVEN THERMAL REACTOR

The LDTR is an off-line analytical device that is used to determine the thermal characteristics of substances by rapid and controlled heating with laser radiation. The LDTR can be used to help optimize a waste treatment technology by providing thermophysical and chemical kinetic information that may be used to characterize waste materials and fuels for maximum destruction, combustion efficiency, or maximum energy release. The device can be used either as a stand-alone instrument in an analytical laboratory, or as an on-line process control sensor for high-temperature process equipment, such as for incinerators, pyrolyzers, and furnaces. As an on-line sensor, the LDTR can be used to monitor the time varying properties of the feed stream (3). Temperatures achievable with the LDTR range up to 2000 K and the heating rate can be as high as several hundreds of degrees per second. This integrated, total response of a substance to the dynamic, high-temperature environment is precisely the measurement required for assessing the performance of a full-scale chemical reactor.

The LDTR consists of a disk-shaped substrate upon which rests the sample. The substrate is connected to a thermocouple and sits inside a sphere-shaped copper reactor (10 - 25 mm in diameter). The assembly is mounted in a chamber and heated by an infrared laser beam of sufficient intensity to heat the spherical enclosure from opposing sides. A schematic of the LDTR is presented in the Fig. 1. A uniform substrate temperature is achieved via radiative transport from the inside surface of the sphere. The high thermal conductivity of the metallic sphere, and the presence of radiation heat transfer within the sphere, results in a uniform sample temperature. It has been determined that the nonuniformity of a millimeter-sized sample placed within the sphere is of the order of 1 K. This uniformity in sample temperature depends on the

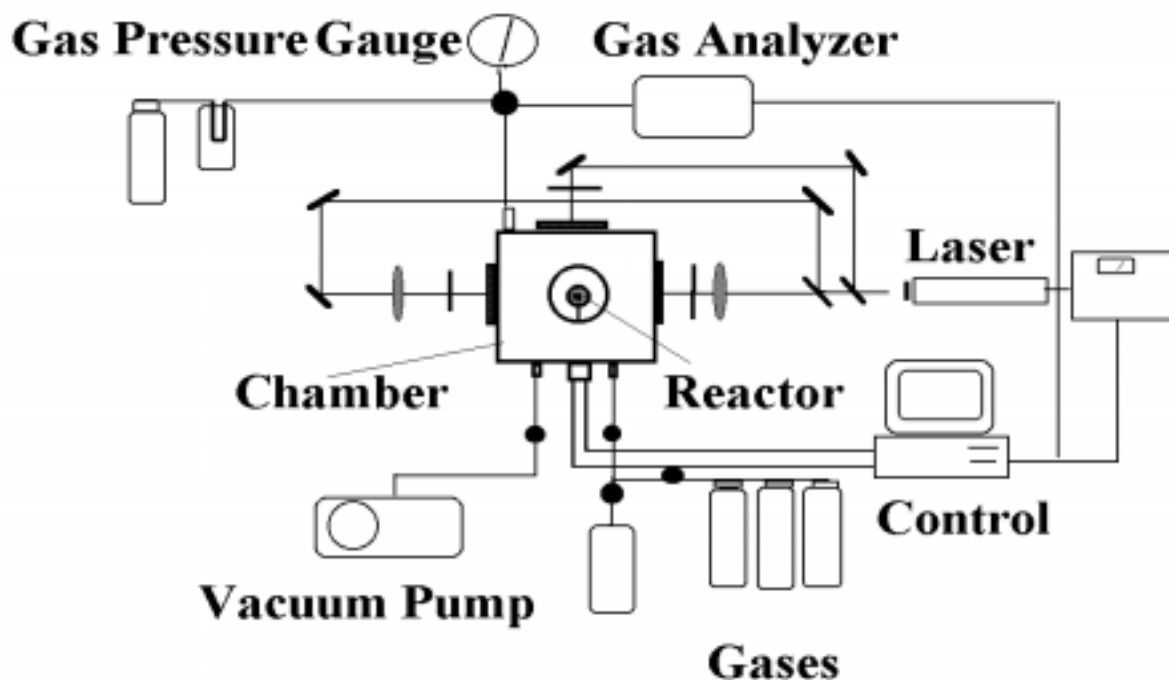


Figure 1 - Experimental arrangement for the laser driven thermal reactor.

relative dimensions of the sample and sphere, laser beam width, and sphere thermal conductivity. The temperature measured by the thermocouple is the response signature of the substance. The temperature sensing resolution is currently 10^{-1} - 10^{-2} s (one may achieve a resolution of 10^{-6} s with newly available thin-film sensing technology). Additional information on the chemical reaction can be obtained from chromatography of the gases released during the process. The LDTR has been demonstrated on several different wastes and fuels, such as simulant high-level organics, nitro-compounds, ozone absorbed by activated carbon, sulfur compounds in heavy fuel oil, and coals (4-6). Kinetic parameters (i.e., endothermic and exothermic rate constants) of the physico-chemical processes can be deduced from time-resolved temperature measurements of the sample, reactor, and surrounding medium. In addition, the laser intensity control system allows one to program the thermal field and sample heating rate. With the programmable LDTR, one can rapidly heat samples in a well-controlled fashion to initiate processes such as mass transport, high temperature decomposition, and reaction chemistry.

Calibration of the apparatus is carried out by measurement of the sample heat transfer characteristics (i.e., the thermal loss parameter) within the reactor. Theoretical assessment of the thermal loss parameter requires a model of the temperature field distribution surrounding the test sample, and knowledge of the sample surface area and shape. These requirements, however, make it virtually impossible to conduct a theoretical assessment with actual samples and under real operating conditions. Two methods that were developed to determine the thermal losses with the LDTR are presented elsewhere (7,8). In the absence of any chemical heat release effects, direct laser heating can be used to determine the thermal loss from a sample. This

approach provides a method to decouple experimentally the effects of chemical heat release, and convective and radiative heat losses. An alternative method is available for determining the thermal losses by measuring the delay in the sample temperature rise during heating of the reactor. The principal method is through direct heating of the sample with a third laser beam (through an opening in the top of the reactor) at an initial time, t_0 . After the laser beam is discontinued, t_1 , the relaxation rate of the sample temperature is measured as the sample returns to its initial equilibrium temperature, T_0 . The relaxation rate is then used to derive the thermal loss parameter for the LDTR system, and is thus referred to as the "relaxation method."

The second method, referred to as the "heating rate method", calibrates the device by carrying out measurements at different heating rates. Measurement of the thermal loss parameter is based on the time lag determination between heating of the reactor and the sample. In this case, measurements are repeated several times at different heating rates to determine accurately the thermal loss parameter. When compared with each other, the two calibration methods correlate well, which increases the accuracy of the LDTR measurement. The variation of the thermal loss parameter with temperature has been determined using both calibration methods. The data are in good agreement to within accuracy of 10 % (8).

The calibration methods have two requirements for reliability and accuracy. First, the position of the substrate inside the reactor must be the same for each experiment. Second, although the temperature varies over the reactor surface, the distribution must be the same in different heating regimes (i.e., with different laser intensities). This will provide a uniform distribution of temperature over the sample. Measurement of temperature at one point on the reactor surface is sufficient to verify the second requirement, and is used to compare the temperature evolution with and without a sample. The degree to which these requirements are violated influences the accuracy of the method.

One might argue that heating of the reactor could be carried out not only by lasers, but also by other heating methods, such as electrical heating. However, the laser-based method of heating offers significant advantages over electrical heating. The main advantages are: 1) direct heating of the reactor, by eliminating convective heating of the surrounding gases; 2) direct measurement of the total thermal loss (both thermal and chemical), due to direct heating of the sample; 3) efficient energy transfer to the sample, by matching the laser wavelength and reactor surface properties (i.e., absorption coefficient of the laser beam $\beta(T)_{1.06\mu\text{m}}$ is higher than the total reactor emissivity $\alpha(T)$: $\beta(T)_{1.06\mu\text{m}} > \alpha(T)$, see Ref. (6); and 4) controlled programming of the temperature field, due to the laser short time response (5).

Major features of the LDTR system are: 1) high laser powers (up to 260 W with a cw Nd:YAG laser operating at 1 μm) and stability; 2) high accuracy for repeated sample positioning (with linear motion control); and 3) rigorous and accuracy data processing. These features result in more accurate data due to a two-fold increase in temperature and a three-order-of-magnitude increase in heating rate over conventional techniques. In addition, a variety of materials (i.e., solids, liquids, and solid/liquid/gas mixtures) can be examined in different gaseous environments (e.g., oxidizing, inert, humid, mixtures) and pressures (from 0.01 kPa to 500 kPa).

EXPERIMENTAL STUDY OF SIMULANT HIGH-LEVEL WASTE PAS 94

PAS 94 is a simulant of Hanford HLW that consists of over 24 different compounds including a variety of organic complexants, solvents, aging products, sodium nitrate, sodium nitrite, process additives such as iron hydrous oxides, sodium hydroxide, and a variety of other inorganic materials. The recipe for preparation of the simulant is given in Ref. (1). The LDTR experiments were performed on the sample for different preparation conditions (i.e., for different degrees of drying in a vacuum oven, gas type, and pressure). The DSC analyzes were performed at DoE Pacific Northwest National Laboratory (PNNL) (9) and at NIST. The analysis of the thermal behavior of the simulant samples was also performed using the NIST laser driven thermal reactor. The LDTR measurements were carried out for different heating rates and temperature levels (exceeding those obtainable with the DSC). Comparative analysis of the LDTR and DSC data resulted in detection of features in the thermal response that were unique to the LDTR measurement. The LDTR was used to demonstrate the many fold improvement of the LDTR over the conventional DSC technique. For example, Fig. 2 presents a comparison of the time required to heat a sample of PAS 94 and measure its temperature. The DSC heating rate was 5 K/min and the maximum temperature achieved was 820 K. These results indicate that the characteristic time to complete a measurement with LDTR was 20 s - 30 s while the DSC required 6600 s.

Thermal Behavior of Simulant PAS 94 Organic Waste

Experiments were carried out with PAS 94 organic waste simulant using the LDTR at temperatures and heating rates that were representative of those for high-energy initiators. The experiments were carried out at a temperature of 900 K, heating rate of 40 K/s (adequate to resolve all phenomena), with a sample size of 15 mg, and in both argon and air environments. The PAS 94 samples were also predried in order to match the conditions and measurement of the DSC experiments by PNNL. With the DSC, moisture content results in the slow progression of endothermic processes that obscure the potential exothermic behavior of the material. With the LDTR no pretreating of the sample is needed to detect all type of processes (e.g., endothermic and exothermic). The samples were dried for two days at room temperature in a vacuum oven prior to the experiment. The DSC measurements that were carried out at PNNL are presented elsewhere (1,9). The LDTR measurements are presented in Fig. 3 for both the untreated "*actual*" and "*dried*" samples. The *actual* samples were measured in an air environment that represents real-world conditions, and the *dried* samples were measured in argon in order to simulate the PNNL predried experimental conditions. The figure presents the variation of temperature with time for the *actual* (see Fig. 3a) and *dried* (see Fig. 3b) samples. Each figure contains a sample and baseline measurement, which is obtained by first carrying out the measurement with the sample (i.e., "sample" data) and then repeating the measurement again (i.e., "baseline" data). The sample measurement is given in the figure and compared to the baseline. When the sample measurement is below the baseline curve the reaction is considered endothermic, and when it is above the curve, the reaction is exothermic. The sudden exothermic rise in the sample results indicates that there is a critical exothermal behavior at this temperature.

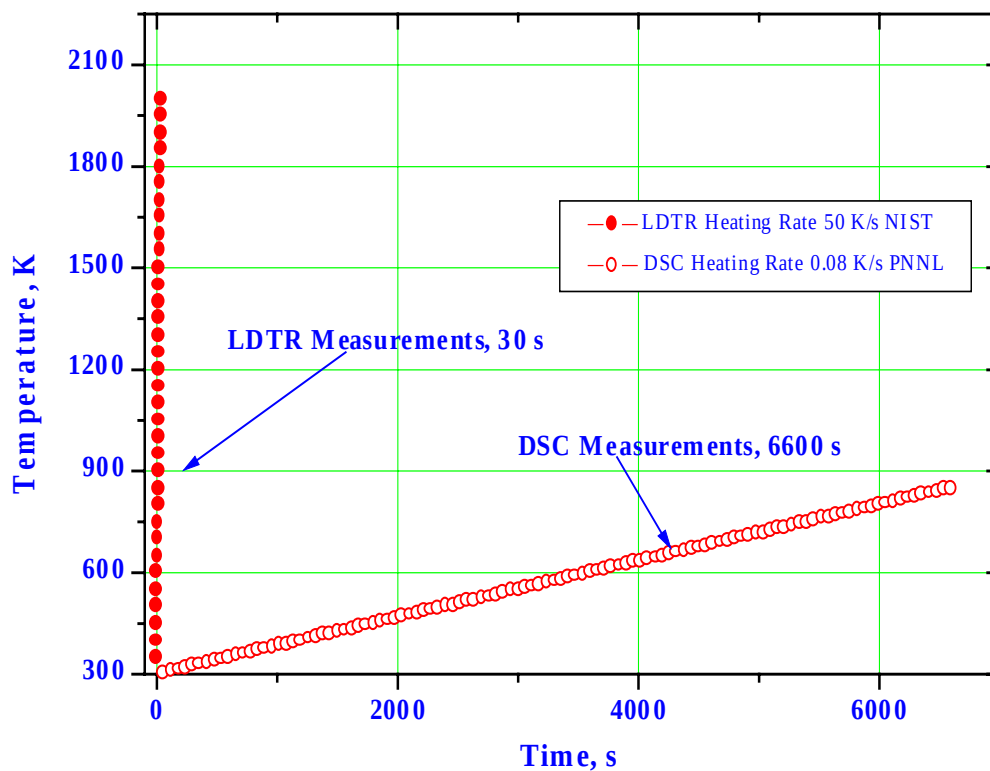


Figure 2 - Comparison of LDTR and DSC heating rates for simulant HLW PAS 94.

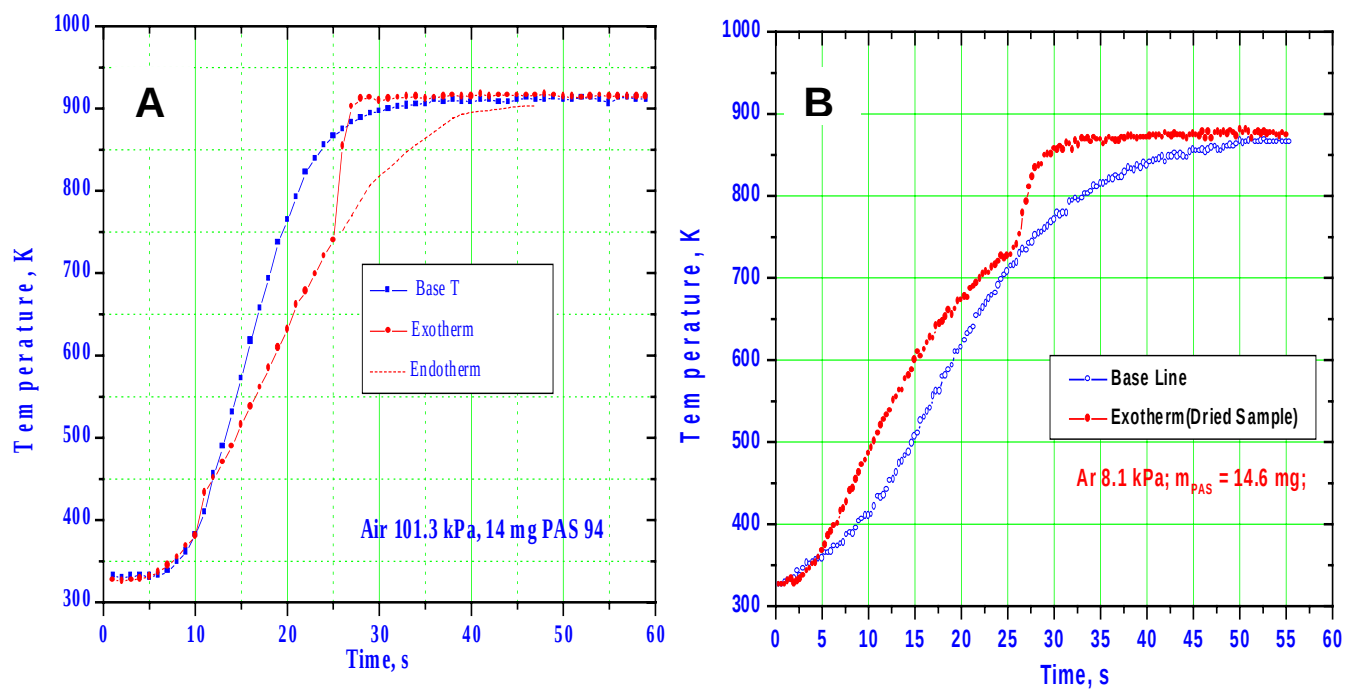


Figure 3 - Thermal behavior of (a) untreated (*actual*) and (b) predried (*dried*) simulant HLW PAS 94 using the LDTR.

The DSC results indicated that drying caused the overall exothermal energy release to decrease from 654 J/g to 443 J/g (10). However, the net energy release increased from nearly zero to 367 J/g due to the decrease in the water endotherm. An exotherm at 720 K increased significantly from 5 J/g to 84 J/g. For the LDTR measurements, the *actual* sample also indicated the presence of an intense exotherm that formed at 720 K (see Fig. 3b), with an energy release of 2957 J/g. The net release (contributions from both the endotherm and exotherm) for the entire heating process was 1.7 J/g. The LDTR results indicated the presence of a strong exothermic energy release of 1.2 - 2.9 kJ/g from 720 K - 870 K, and a net release of exothermic energy over the entire process of 1.7 kJ/g. These results indicate that even with DSC-sized samples, the LDTR was found to be more sensitive in detecting the material thermal features. Clearly the results were found to be highly dependent on heating rate.

Contribution from Sodium Nitrite and Sodium Nitrate

As mentioned earlier, sodium nitrite and sodium nitrate constitutes 20 - 25 % of the total mass fraction of the HLW PAS 94. From a safety and design point of view, it is important to have quantitative empirical data on the thermal behavior of these compounds. Thus, the thermal behavior of sodium nitrite and sodium nitrate was studied with the LDTR and compared with DSC measurements. The experiments were performed with *actual* (no predrying) chemicals in both argon and air at a pressure of 101 kPa. The results indicate that there was no effect of the environment on the thermal behavior of these chemicals. The thermal behavior of sodium nitrite was also determined with the DSC. The DSC results (not presented) indicated that in the temperature range of 550 K - 600 K there was strong endothermic process, which was attributed to the thermal decomposition and melting of NaNO_2 . The heating rate during these DSC experiments was 5 K/min. Experiments with the LDTR were carried out at characteristic heating rates of 30 - 40 K/s, which were 350 - 400 times higher than achievable with the DSC. The results indicated the presence of an endothermic process between 350 K - 650 K, and an exothermal process above 650 K. The endothermic process was attributed to water evaporation (above 350 K), and melting and thermal decomposition between 550 K and 650 K. Similar features were also obtained with DSC measurements carried out at NIST. The total heat absorption for the entire process was -1325 J/g. The rate of thermal decomposition of NaNO_2 was determined and the results (not presented) indicated that there is a linear dependency of the rate of decomposition with temperature, and the empirical relationship was:

$$q(T) = (1087 \pm 171) - (2.1 \pm 0.3) \times T, \text{ W/g},$$

in the temperature range of 550 K - 650 K. Unlike the DSC results, the LDTR detected a strong exothermal process detected above 650 K (until termination of the experiment at about 750 K). The experimental results for sodium nitrate thermal behavior also indicated a linear dependency of the rate of decomposition with temperature, and the empirical expression was:

$$q(T) = (832 \pm 79) - (1.2 \pm 0.1) \times T, \text{ W/g}.$$

Process Rates and Empirical Model

Figure 4 presents LDTR results (semi-logarithmic scale) for simulant PAS 94 kinetic parameters (i.e., endothermic and exothermic process rates) with the chamber operating at a pressure of 101 kPa in air, and 8.1 kPa in argon. Figure 4a presents the rate of the endothermic process for the *actual* sample at temperatures in the range of 370 K - 620 K. The line through the data is a best-fit estimation with the empirical expression given near the top of the figure. The star symbols indicate the melting points for sodium nitrate (544 K), sodium nitrite (581 K), and sodium hydroxide (591 K); these three chemicals represent over 54 % of the mixture (10). The LDTR results indicate that the rate of the endothermic process is flat near these melting points, because of the phase change of these three components from solid to liquid. The heat release rates of the

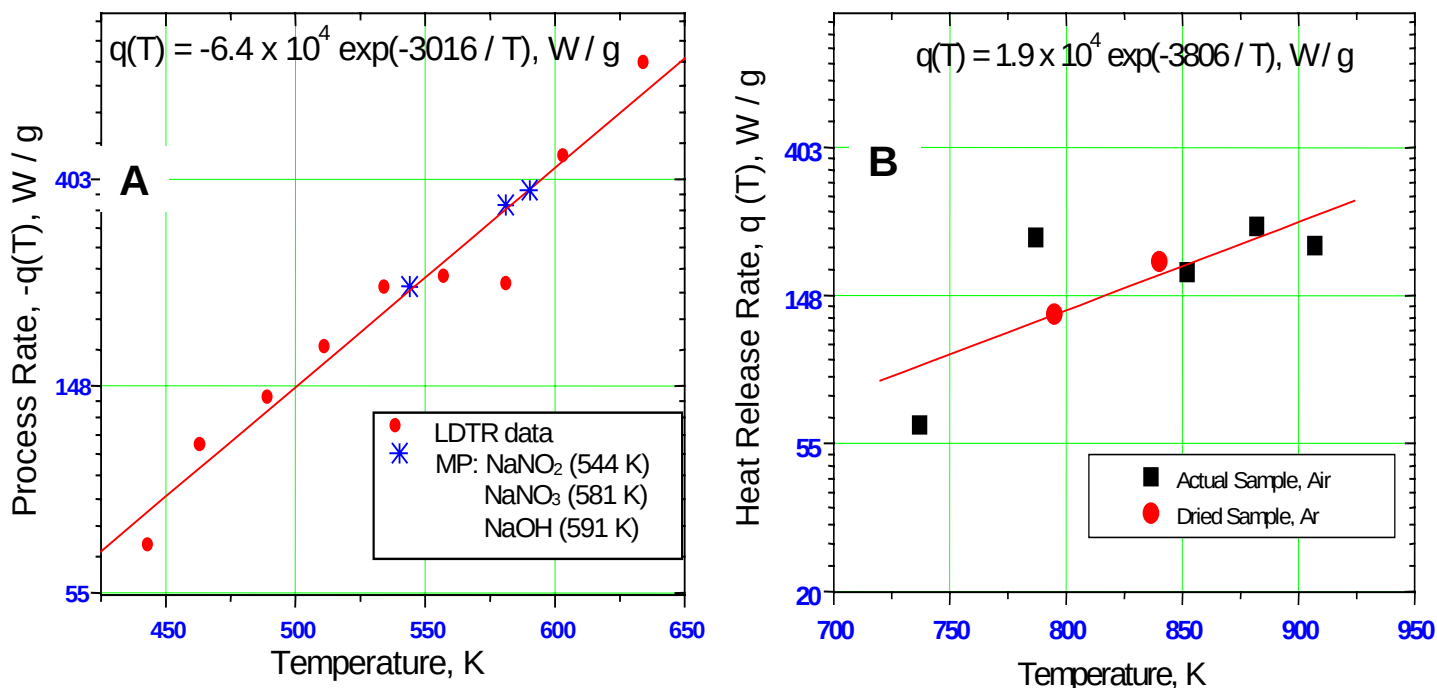


Figure 4 - Determination of the (A) rate of the endothermic process for the actual sample, and (b) rate of the exothermic process for both the *actual* and *dried* simulant HLW PAS 94.

exothermic processes for the *actual* (square symbols) and *dried* (circle symbols) samples are presented in Fig. 4b. The results indicate that the rate of the exothermic process for both the *actual* and *dried* samples are correlated and provide quantitative information on the sample heat release rate. Thus, the LDTR can enable one to assess different scenarios by determining how much and how fast thermal energy is released from the sample. The time integral from the differential heat release rate, $q(T)$, also provides the total heating value of the process. For example, the results from Fig. 3 indicate the presence of a sharp exotherm that develops at about 720 K. One can then develop an empirical model of the sample thermal behavior by representing the rates of the endo- and exothermic processes with the following Arrhenius relationship:

$$q(T) = q_0 \exp(-E_a/T), \quad (\text{Eq. 1})$$

where $q(T)$ is the heating rate, q_0 is a pre-exponential parameter, and E_a is the activation energy in units of absolute temperature (T). The total heat release (or absorption) rates are expressed by:

$$Q = \int q(T) dt, \quad (\text{Eq. 2})$$

A global two-step model of the sample thermal behavior (see Fig. 3) is presented in Eqs. 3 and 4:

I. *For the Actual Waste:*

$$\text{PAS 94} \Rightarrow \text{A} \xRightarrow{q_1^{\text{End}}, Q_1^{\text{End}}} \text{B} \xRightarrow{q_1^{\text{Exo}}, Q_1^{\text{Exo}}} \text{C}, \quad (\text{Eq. 3})$$

The kinetic rate of the endothermic stage from A to B is: $q_1^{\text{End}}(T) = -6.4 \times 10^4 \exp(-3016/T)$, W/g; in the temperature range from 400 K to 650 K, where $Q_1^{\text{End}} = -1.2$ kJ/g is the total heating value. The subscript "1" refers to the *actual* waste case. The kinetic rate of the exothermic stage from B to C is: $q_1^{\text{Exo}}(T) = 1.9 \times 10^4 \exp(-3806/T)$, W/g; in the temperature range from 650 K to 900 K, where $Q_1^{\text{Exo}} = 2.9$ kJ/g is the total heating value. The total heating value between A and C was 1.7 kJ/g.

II. *For the Dried Waste:*

$$\text{PAS 94} \Rightarrow \text{A} \xRightarrow{q_2^{\text{Exo}}, Q_2^{\text{Exo}}} \text{B} \xRightarrow{q_2^{\text{Exo}}, Q_2^{\text{Exo}}} \text{C}, \quad (\text{Eq. 4})$$

There was no endothermic stage in this case (see Fig. 3b). The kinetic rate of the exothermic stage from A to B is: $q_2^{\text{Exo}}(T) = 4.4 \times 10^3 \exp(-1895/T)$, W/g; in the temperature range from 400 K to 650 K, where $Q_2^{\text{Exo}} = 0.6$ kJ/g is the total heating value. The subscript "2" refers to the *dried* waste case. The kinetic rate of the exothermic stage from B to C is: $q_2^{\text{Exo}}(T) = 1.9 \times 10^4 \exp(-3806/T)$, W/g; in the temperature range from 650 K to 900K, where $Q_2^{\text{Exo}} = 1.2$ kJ/g is the total heating value and is a factor of two lower than the value of Q_1^{Exo} in the *actual* waste case. The total heating value between A and C was 1.8 kJ/g and is consistent with the *actual* waste case. This model points out that without pre-drying in the early stage of heating, the endothermic processes dominate because of the absorption of energy by water. Also, the reaction characteristics of the exothermic processes for the *actual* and *dried* samples are different, as evidenced by their different exothermic behavior. However, the overall kinetics appear to be similar for both cases (also see the correlated behavior in Fig. 4b). Note that the *actual* waste measurements were carried out in an oxidizing (air) and the *dried* case in inert gas (argon) environment. Thus, this result suggests that the thermal release characteristics of PAS 94 may not depend on combustion of organics in oxygen.

CONCLUSION

This study demonstrated the capability of the LDTR to elucidate the effects of heating rate and temperature on waste material reactive behavior. This information has direct relevance to hazardous situations that might occur in waste tanks, such as the potential for rapid, uncontrolled reactions. These data also can be used to provide fundamental thermophysical and chemical

kinetics data for simulant wastes such as sodium nitrate/sodium nitrite mixtures for HLW vitrification and stabilization. The LDTR measurements were used to develop an empirical model, which provides a phenomenological description of the process. The LDTR capabilities enabled detection of a much larger exotherm than observed with the conventional DSC. These results demonstrate that the LDTR may be used to provide new information on tank waste thermal behavior, under conditions representative of a high-energy initiator such as lightning and rapid heating during glass melter vitrification.

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