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# FY 1997 Progress Report on Tube Propagation Testing of Tank Waste Using the PRSST

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Abstract: The subject of this FY 1997 progress report is tube propagation tests of actual, dried tank waste to verify the contact temperature ignition (CTI) criterion for point-source ignition in the Hanford Site waste tanks. Testing is in support of the Organic Tanks Safety Project and will help resolve safety issues with waste containing organic constitutuents. In FY 1997, improvements were made to the laboratory apparatus and procedures for conducting the testing, and the final testing strategy was formulated. The strategy lays out details of the tests to be performed, samples to be tested, and modes of reporting results.



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# FY 1997 Progress Report on Tube Propagation Testing of Tank Waste Using the PRSST

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Numatec Hanford Corporation

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Assistant Secretary for Environmental Management

Project Hanford Management Contractor for the
U.S. Department of Energy under Contract DE-AC06-96RL13200

Approved for public release; distribution is unlimited

## CONTENTS

## LIST OF TERMS

| | |
|---|---|
| CTI | contact temperature ignition |
| FAI | Fauske & Associates, Inc. |
| FY | fiscal year |
| PRSST | Propagating Reactive System Screening Tool |
| TOC | total organic carbon |

# 1.0 INTRODUCTION

The determination of how some Hanford Site tank wastes should be stored safely rests in part on verifying the wastes do not possess the ability to propagate an exothermic self-decomposition, that is, they are not susceptible to a point-source ignition (Meacham et al. 1997). Fauske & Associates, Inc. (FAI) has developed a conservative criterion, the contact temperature ignition criterion (CTI), for the composition of Hanford Site tank wastes (Fauske et al. 1995). The CTI criterion is used to judge whether the the Hanford Site tank wastes could propagate. The criterion states that tank waste will not support propagation at ambient temperature where total organic carbon (TOC) content is less than $(4.5 + 0.17x_w)$ percent and where $x_w$ is the percent water content.

The activity reported here is directed at comparing the CTI criterion to the TOC concentration that supports propagation in actual tank wastes. The tube propagation test is used wherein a sample is packed into an insulated tube with thermocouples located along or within its length, and the sample is ignited at one end of the tube. If the sample succeeds in burning down the length of the tube, as evidenced by the thermocouple readings, the sample is said to have supported a propagating reaction.

Tube propagation tests are performed on samples of dried tank wastes, and on additional samples with added organic chelator salt to bring their total TOC to specified levels. This "spike test" sequence will verify the location of the CTI propagation boundary for actual tank wastes.

# 2.0 STATUS

## 2.1 TESTING STATUS

An interim letter report of the testing status was issued in Fiscal Year (FY)1996. It is attached as Appendix A. In summary, the Propagating Reactive System Screening Tool (PRSST) instrument was developed by FAI (Fauske 1996a and 1996b) to perform tube propagation tests on radioactive wastes in a hot cell. Some consultation took place with Hanford hot cell operators to develop modifications that minimized sample usage and permitted operating the instrument with remote manipulators. The instrument was checked by comparing results on a surrogate compound to those obtained by FAI. The instrumental response to propagation failures and its performance as a self-heat calorimeter also were characterized. In self-heat calorimetric mode, the instrument slowly and uniformly heats the samples to high temperatures and records if, when, and to what extent the sample self heats through exothermic decomposition. After reaching high temperatures, some samples that otherwise fail to propagate in a tube propagation test still may yield information useful in verifying the CTI.

Finally, the instrument has been installed in a hot cell and has been used to test one sample of actual waste from tank 241-U-105. Completion of the overall testing strategy will involve more tank waste samples in FY 1998.

## 2.2 INSTRUMENT AND EQUIPMENT STATUS

As a result of testing, FAI has modified the instrumental sample holder to provide more uniform sample heating. Although this modification has not been tested, it is expected to improve the self-heat calorimetric performance of the instrument but at the cost of some additional sample holder manipulation.

A software bug in the original instrument prevented working directly in metric pressure units, and testing had to be done in English units. An attempt to change this was unsuccessful; therefore, all pressure data are recorded in English units and must be converted to metric units during the post-processing of results.

Two complete instruments and one instrument without a computer provide backup and/or increased testing capacity.

Three hot cells in the 222-S building laboratories at the Hanford Site were removed from service for an extended time in FY 1997 to install regulation-mandated double-walled drains. Two are back in service, and the third is expected to be in service in October 1997.

A further result of testing is the recognition that better sample drying equipment and methods are required. Polytetrafluoroethylene vessels, which are special small-scale temperature controlled vacuum ovens, and a small-scale, oil-free vacuum pump have been procured and installed in a hot cell to provide the ability to dry two samples simultaneously at 105°C under laboratory vacuum.

## 2.3 PROCEDURE STATUS

Test strategy, sample preparation, and testing were originally governed by test plans (Bechtold 1996, Meacham 1995, and Reynolds 1996). Testing experience and shifts in program strategy required modifications to the plans. As a result,the test details were indorporated into a formal laboratory technology procedure (Bechtold 1997). The procedure incorporates recent instrument modifications, lessons learned in FY 1996, and new waste-handling measures to assure compliance with Washington State Department of Ecology regulations. The procedure was approved by laboratory management and issued in August. A required room inspection to ensure regulatory compliance in waste handling is expected in early September.

## 2.4 CTI VERIFICATION STRATEGY STATUS

The original version of the test strategy consisted of testing three dried tank wastes for propagating ability, spiking the wastes with (additional) organic chelate salt, and retesting incrementally until propagation was observed. Propagatable spiked wastes were to be moistened to 5 percent water, and the process was to be repeated until a propagatable spike again was achieved, whereupon a final propagatable spike was to be sought at 10 percent moisture. This series would demonstrate the location of the CTI boundary between propagatability and nonpropagatability to significant moisture levels.

For FY 1997, the strategy has been revised and simplified. The revised strategy calls for testing dried and spiked composites from tanks 241-C-104, 241-U-102, 241-U-105, and 241-U-106. To do a spike test, do the following.

1. Dry a batch of sample material at 105 °C under laboratory vacuum to constant dry weight.

2. Remove aliquots of the dried material and perform the following analyses:

    a. differential scanning calorimetry

    b. thermogravimetric analysis

    c. total organic carbon

    d. ion chromatograph for

        (1) inorganic anions

        (2) organic acids including oxalate, glycolate, formate, acetate, citrate, and nitrilotriacetate

3. Test an aliquot of the unspiked, dried material using the PRSST.

4. Add aqueous $Na_3HEDTA$ solution to an aliquot of the dried material so that the total organic in the aliquot is six weight percent when redried to constant weight. Ensure there are at least stoichiometric amounts of oxidizers in the sample. If the aliquot is fuel-rich, add oxidizer as necessary.

5. Test this spiked aliquot of sample using the PRSST.

6. If the spiked aliquot from step 5 fails to produce a propagating reaction, spike another aliquot of sample by an additional one weight percent organic (in the manner identified in step 4) and test using the PRSST. Repeat this step until an aliquot propagates.

7. If the spiked aliquot from step 5 produces a propagating reaction, spike another liquot of sample with one weight percent less organic (in the manner identified in step 4) and test using the PRSST. Repeat this step until an aliquot fails to propagate.

8. Determine the highest weight percent of organic which failed to propagate. Repeat the PRSST analysis once with an aliquot spiked with this weight percent organic.

In addition to the spike tests above, two tank sampling and analysis plans (Schreiber 1997a and 1997b) have been recently issued to test dried samples from tanks 241-C-201 and 241-C-202.

Finally, tests are to be performed on safety screen core samples as part of Tank Waste Remediation System tank characterization activities. Currently, one sample from tank 241-U-103 exceeds the energetics notification limit and will be tested.

## 2.5 SAMPLE STATUS

All four tank composites for the spike tests have been made and are available for testing.

The tank 241-U-103 tank sample is also available for testing.

Tanks 241-C-201 and 241-C-202 are to be sampled in September 1997. If samples are taken successfully, they should be available for testing in early October 1997.

# 3.0 PLAN TO COMPLETION

1. Secure permission to operate the LT procedure from laboratory management in early September 1997.

2. As directed by program management, prepare dried and spiked samples for tube propagation testing at double the rate originally planned because this task is currently on the critical path in resolving the organic complexant safety issue.

3. While PRSST testing prepared samples, evaluate whether it is advantageous or practical to double the testing rate. If so, set up a spare instrument, drying oven and vacuum pump in hot cell 1F and staff a second team to perform tests.

4. Report the spike testing results as directed by the LOI in January 1998.

5. Prepare, test, and report on tank 241-C-201 and 241-C-202 samples by March 31, 1998 as directed by the tank sampling and analysis plans by March 31, 1998.

6. Prepare, test, and report on the tank 241-U-103 sample by April 30, 1998.

## 4.0 CONCLUSIONS

All elements are in place to complete the CTI verification at this time except final approval to operate the approved laboratory technology procedure. Approval is expected in early September whereupon testing can begin with sample preparation at double the rate originally planned.

## 5.0 REFERENCES

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Fauske, H. K., M. Epstein, D. R. Dickinson, R. J. Cash, D. A. Turner, J. E. Meacham, 1995, *The Contact-Temperature Ignition (CTI) Criteria for Propagating Chemical Reactions Including the Effect of Moisture and Application to Hanford Waste*, WHC-SD-WM-ER-496, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Fauske & Associates, Inc., 1996a, "FAI's Propagating RSST Meets the Challenge", *FAI Process Safety News*, Vol. 3, No. 3, Fauske & Associates, Inc., Burr Ridge, Illinois.

Fauske & Associates, Inc., 1996b, "Propagating RSST: FAI Introduces Specialized RSST System for Propagation Testing," *FAI Process Safety News*, Vol. 3, No. 2, Fauske & Associates, Inc., Burr Ridge, Illinois.

Meacham, J. E., 1995, *Test Plan for Samples from Hanford Waste Tanks 241-BY-108, BY-104, BY-105, BY-106, BY-108, BY-110, TY-108, U-105, U-107, U-108, and U-109*, WHC-SD-WM-TP-378, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Meacham, J. E., A. B. Webb, N. W. Kirch, J. A. Lechelt, D. A. Reynolds, G. S. Barney, D. M. Camaioni, F. Gao, R. T. Hallen, and P. G. Heasler, 1997, *Organic Complexant Topical Report*, HNF-SD-WM-CN-058, Rev. 1, Lockheed Martin Hanford Corporation, Richland, Washington.

Reynolds, D. A., 1996, *Organic Test Plan for Selected Tanks*, WHC-SD-WM-TP-378, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

Schreiber, R. D., 1997a, *Tank 241-C-201 Push Mode Core Sampling and Analysis Plan*, HNF-SD-WM-TSAP-137, Rev. 0, Lockheed Martin Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.

Schreiber, R. D., 1997b, *Tank 241-C-202 Push Mode Core Sampling and Analysis Plan*, HNF-SD-WM-TSAP-138, Rev. 0, Lockheed Martin Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.

# APPENDIX A

# INTERIM REPORT OF RESULTS OF PRSST TEST PLAN

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**Westinghouse Hanford Company** — **Internal Memo**

From: Process Chemistry & Statistics — 75760-PCS96-067
Phone: 373-2162 T6-09
Date: June 25, 1996
Subject: INTERIM REPORT OF RESULTS OF PRSST TEST PLAN

To: J. E. Meacham S7-14

cc:

| | |
|---|---|
| R. Akita | T6-20 |
| H. Babad | S7-30 |
| T. H. Bushaw | T6-30 |
| R. J. Cash | S7-14 |
| D. A. Dodd | T6-50 |
| G. T. Dukelow | S7-14 |
| G. B. Griffin | T6-16 |
| V. W. Hall | T6-04 |
| J. R. Jewett | T6-09 |
| T. J. Kelley | S7-21 |
| A. G. King | T6-03 |
| N. W. Kirch | R2-11 |
| J. E. Meacham (5) | S7-14 |
| C. T. Narquis | T6-16 |
| A. D. Rice | T6-06 |
| D. A. Turner | S7-14 |
| DBB File/LB | |

REFERENCE: D. B. Bechtold, Laboratory Test Plan for Measuring Tank Waste Propagation or Self-Heating using the PRSST, WHC-SD-WM-TP-438 Rev. 0, dated 1996.

The Propagating Reactive System Screening Tool (PRSST) has to date been tested against inert sand, a propagatable surrogate and a dried sample of Tank U-105 sludge according to the referenced plan. Both propagation tests and self-heating tests have been performed in the PRSST. The instrument has been found to be workable as a propagation tester in the hot cell so long as the moisture content of samples is controlled. Its use as a self-heat tester is valuable for non-propagating samples that would otherwise be wasted. However, parallel tests using the Reactive System Screening Tool (RSST) would be needed to demonstrate that the PRSST could substitute for the RSST as a self heat tester. This report summarizes the test results, the progress made in operating the PRSST and the lessons learned towards effective tank waste testing in the hot cell.

The tests to be described are identified and associated with the attached figures in the following table:

| PRSST Test Identification | | | | |
|---|---|---|---|---|
| Test Identification | Figure(s) | Sample | Mode | Result |
| 960507B | 1 | Sand | Propagation Test | Inert Temperature Trajectories |
| 960509 | 2,3 | Surrogate (in cold Lab) | Propagation Test | Propagated |
| 960604 | 4 | Surrogate (in hot cell) | Propagation Test | Failed to propagate. Partial reaction at ignitor. Moisture pickup during loading. |
| 960604A | 5 | Surrogate Retest, same sample (in hot cell) | Propagation Test | Failed to propagate on re-ignition. Eventual ignitor burnout, Partial reaction at ignitor. |
| 960605 | 6,7,8 | Surrogate, same sample (in hot cell) | Self-heat Test | Self-heated, then propagated to both ends at ~ 240°C |
| 960611 | 9,10 | Surrogate (in hot cell) | Propagation Test | Propagated, due to faster loading, less moisture, but slower burn rate than perfectly dried material. |
| 960617 | 11,12 | U-105 C136S5UH, dried 7 days at 105°C, ground | Propagation Test. | Failed. Slight reaction at ignitor, eventual ignitor burnout, residual moisture. |
| 960617A | 13,14,15 | U-105 C136S5UH, same sample | Self Heat Test | Self heated, slight, partial propagation at ~ 450°C |

Here is a test-by-test description of the test results:

1. PRSST Checkout.

   A few items not covered in the manual (the Reference) were discovered that are useful for subsequent testing.

   a. The volumes of the two bombs containing test cells loaded with sand to mock actual samples, were measured by gas expansion. The values were 1007 $cm^3$ and 984 $cm^3$, within 2% of the stated bomb capacity of 1 liter.

   b. All the thermocouple leads through the glands of the cold-testing bomb had shunts of 90 KΩ to the bomb casing, while thermocouple leads 1 and 2 of the second (hot cell testing) bomb, which share a gland, both had shunts of 5 to 6 KΩ to the casing. These latter shunts were low enough that the instrument controller would report thermocouple readings from these leads without thermocouples being attached to the other ends. Fortunately, the false readings are always a few degrees higher than ambient, and once connections to actual thermocouples are made, the temperature responses are correct. Only the third thermocouple leads in the second bomb, which shares a gland with the ignitor leads, had no measurable shunt. Ordinarily, shunts like these would be avoided if possible, but they are apparently a necessary consequence of the need for a good pressure seal. The testing reported here verifies that the thermocouples apparently develop enough signal to overpower these shunts and provide accurate temperature signals to the controller.

c. The software contains a bug where selecting the planned pressure units of bar causes the controller program to crash. For now, the course of action has been to continue to use the default units of psi for data acquisition and convert to bar during data processing. Fauske & Associates, Inc. (FAI) has sent a software fix, but incorporating it has been postponed to a later time.

2. Cold Testing with Sand.

The Reference shows that the test cell is an insulated cylinder with three thermocouples placed along its outside wall, the first being at the same distance that the internal ignitor is positioned when it is immersed in the sample. Sand is inert, and permits determining the baseline instrument response to a propagation test when no reaction whatever occurs in a sample. Figure 1 confirmed that the ignitor operation is indeed verified by the temperature trajectory of thermocouple 1. Also indicated is the bleed of heat down the outside of, and/or through the sample contained in the tube. The sand test was conducted in the cold-testing bomb; note the concurrent depression of the thermocouple 3 signal while the ignitor is on. This is leakage of ignitor signal across the shunt in the shared gland. Also learned is the fact that the controller logs test data at a very rapid rate whenever the ignitor is turned on -- too rapidly, if nothing interesting happens. Data from several tests had to be winnowed down to a manageable number of values for post-processing.

3. Cold Testing with Surrogate.

The surrogate recipe is found in the Reference. As expected, the surrogate propagated during test 960509. The result compared favorably with that obtained by FAI with the same surrogate and on the same model of instrument, as shown in the following table:

| Surrogate Propagation Test Comparison | | | |
|---|---|---|---|
| Test Entity: | WHC | FAI | WHC |
| Test: | 960509, Cold Lab | -- | 960611, hot cell |
| Selected Pre-heat Temperature: | 30°C | 50°C | 50°C |
| Peak Combustion Temperatures: | 475-525°C | 580-610°C | 520-560°C |
| Average Propagation Speed: | 5.22 cm/min | 5.19 cm/min | 2.1 cm/min |
| Noncondensible Gas Yield: | 0.00431 mole/g | -- | 0.00425 mole/g |
| Noncondensible Gas Rate | 0.017 mole/cm$^2$/min | -- | 0.009 mole/cm$^2$/min |

An important contributor to the lower peak temperatures realized at Westinghouse is the fact that the selected pre-heat temperature was lower in this test. FAI has pointed out that the narrow sample tube in this design, coupled with the external thermocouples, allows heat losses to play a role in determining the peak temperatures actually detected. Consequently, the effect of a higher pre-heat temperature on peak temperatures is amplified by the exponential dependence of reaction rates on temperature.

Another lesson learned by the test is that the method of pinpointing the moment of reaction front passage should be changed. Instead of taking the intersection of the maximum slope of the thermocouple trace with its baseline as the moment of front passage, the choice was made to use the time location of the maximum slope itself. This offered a more uniform determination to be made among the thermocouples when their responses were not congruent. To use this method, an 11-point smoothing of the thermocouple trace slopes was found necessary.

4. Surrogate Testing in the Hot Cell.

A Series of four tests were conducted using surrogate in the hot cell, three of which utilized the same sample in series, while the fourth used fresh sample. The first hot cell surrogate test, 960604, was also the first occasion a sample was loaded into the PRSST while inside the hot cell, and consequently it required considerable time to do so. This allowed the sample to absorb moisture from the air. To compound the moisture absorption, the loaded test cell sat in the enclosed bomb overnight before the actual propagation test was conducted. The result was that the surrogate failed to propagate, even though the temperature trajectories and the pressure signal indicated considerable reaction on the part of the surrogate at the ignitor.

A propagation retest (960604A) of the same surrogate sample was made soon after, this time leaving the ignitor on until it burned out. Again, the surrogate did not propagate, despite further indications of a reaction at the ignitor. The only real-time indication that the ignitor burned out was the eventual decay in sample temperatures. After the test, a continuity check verified the ignitor had burned out.

The last test on that particular sample, 960605, was a self-heat test. Here, it was learned that the ends of the test cell suffer greater heat losses than the center, and consequently their temperatures lag the middle zone on heatup. The sample self-heated as expected until the second (middle) thermocouple reached 260°C, whereupon the sample undertook to propagate from the vicinity of this location in both directions, to reach both ends of the cell. Under the assumption of uniform, one dimensional propagation, the time of ignition, location of ignition, and speed of propagation could be worked out by the order and timing of the thermocouple deflections. The calculated result was that the sample ignited at a position 0.06 cm from thermocouple 2 towards thermocouple 1, at 107.04 minutes into the test, and raced towards thermocouples 1 and 3 at a speed of 20.2 cm/min.

Examining these results shows that the calculated ignition time is not consistent with the first indication of rapid pressure rise (see
5. Figure 7), but that the other results are consistent with the notions that ignition should occur where the temperature is highest and that high-temperature propagation speeds should be faster than those at tank temperatures. This points out that the externally placed thermocouples are adequate for measuring the propagation speed of a fully developed front as produced by the ignitor, but that a spontaneous, pointwise ignition can produce an inherently three-dimensional, possibly undetected and non-uniform front development until the vessel walls steer its paths in axial directions. In this case, the best estimate of the propagation speed will come from using only the deflection times from the last two thermocouples.

During a self-heat test, the PRSST was operated like the RSST. The surrogate self-heat test results in the PRSST emphasized that the thermocouples are now separated from the sample by its container, and the container is not as well protected against heat losses as in the RSST. This lessens the fidelity of the uniform, adiabatic heating assumption when working up the data and compromises the results to some extent. Furthermore, the parasitic heat capacity of the test cell is unknown in the present instrument, and consequently any calculation of the actual energy release will require not only an estimate of the sample heat capacity, but also an estimate for the test cell as well. On the other hand, the pressure *vs* temperature data suggest that in the larger PRSST bomb, better than 90% of the gas space can be viewed as being at the (ambient) bomb wall temperature, which helps when making crude estimates of real-time gas production. In all, the PRSST is less effective than the RSST as a self-heat test instrument. It can yield valuable information on an otherwise wasted sample in this mode, but should not be viewed as a substitute for the RSST without parallel RSST tests to confirm its adequacy.

The last surrogate test in the hot cell, 960611, utilized another sample of surrogate, this time loaded more quickly and not allowed to stand before conducting the test. As a consequence, less moisture was absorbed and the sample propagated from 50°C, exceeding 560°C in peak temperature, but propagating at a slower rate of 2.1 cm/min. Again, the absorption of moisture by the surrogate while being dispensed in the hot cell probably had a role, this time in producing the relatively slow propagation speed. Moisture is clearly a very effective dampener of propagation, and hot cell technique will have to be optimized as much as possible to keep samples dry. Additionally, it appears advisable to correlate sample dryness with propagation tendency by requesting follow-up moisture analysis of an unused portion of each sample tested.

6. U-105 Sludge Testing in the Hot Cell.

The laboratory result for moisture of core 136 segment 5UH from this tank averaged 45.3% by TGA. However, approximately 3% of that value can be assigned to the first stage of an interfering exothermic reaction, leaving a better estimate of 42.3%. The aliquot provided for propagation testing proved slow to dry, as indicated by Figure 11, and had reached neither 42.3% weight loss nor constant weight after 7 days at 105°C, but rather 20.2% weight loss or as much as 27.7% residual moisture. Surprisingly, the sample set up to a hard cake and was easily ground to a powder without being, or becoming, sticky in the hot cell atmosphere. This suggests that the aliquot received was perhaps drier than the original segment, and the resulting prepared test material perhaps drier than 27.7% residual moisture.

The dried, ground U-105 sample was first tested in the PRSST at 50°C by leaving the ignitor on until it burnt out. The net pressure gain between this test (960617) and the following one verified that a slight amount of sample had been pyrolized by the ignitor before the latter burned out, but otherwise the temperature trajectories of the test indicated no other significant reaction and no propagation. The ignitor energy deposit could be estimated to be between 750 and 1500 Joules, enough to initiate a propagation in a willing material. It has been demonstrated that this sample in its prepared state will not propagate, but it has not yet been shown that the waste can't propagate when it is proven "dry". This emphasizes once more the advisability of follow-up moisture analyses of samples prepared for PRSST testing.

The next and last test was a self-heat experiment of the unpropagated U-105 sample. Like the experience with the surrogate, the sample temperatures deviated considerably from each other depending on location, but that the usual kinds of self-heat information can be derived from the central zone monitored by thermocouple 2, including rate data on the initiating reaction. The U-105 sample did suffer a slight "propagation" at approximately 450°C in the central zone, as indicated in Figures 14 and 15, but this event did not carry to either end of the sample. Through both tests, the sample generated 0.0038 mole/g noncondensible gas on a prepared basis, or 0.0053 mole/g on a "dry" basis. The central zone managed a corrected self-heat of 185°C from onset, but the further correction of this value to eliminate instrument effects would depend on knowing not only the sample heat capacity, but the unknown test cell heat capacity as well. For the same reason, the energy released by the sample can be stated no more precisely than:

$$\hat{Q}\left(\frac{\text{Joule}}{\text{g dry}}\right) = 239\overline{C}_{ps}^{react} + 22.1C_{ph}W_h$$

where $C_{ph}W_h$ is the test cell heat capacity and $\overline{C}_{ps}^{react}$ is the specific heat capacity of the sample under reaction conditions.

A number of conclusions and recommendations regarding use of the PRSST on actual waste have been drawn from the testing to date:

A. The ignitor survives long enough to deposit adequate initiating energy to a sample. Its successful operation is verified by the deflection of the first thermocouple. The most reliable means to determine that at least a portion of the sample has been burned by the ignitor is the appearance of noncondensible gas.

B. Sample drying should not be rushed, and should continue at 105°C, or some other safe, incrementally higher temperature, until constant weight is achieved. This may mean more sample stirring than previously necessary to achieve this practical definition of dryness in a reasonable time. The state of sample dryness for testing should be confirmed by moisture analysis.

C. Hot cell drying and loading technique must be developed a bit further to permit loading dried, ground sample directly and expeditiously from the oven to the test cell. Minor alterations to the drying oven and appropriate sample handling tools should permit this.

D. The test cell closure and bomb seal-up times can be improved by specifying an additional 2.5 cm of thermocouple length on the test cell thermocouples when they are reordered from the manufacturer.

E. The planned method of determining a propagation front passage past a thermocouple should be changed to be defined by the time location of the maximum slope of the smoothed thermocouple trace.

F. Operating the PRSST as a self-heat tester is useful for verifying the reactivity of samples that otherwise would not propagate. In some cases, it can yield a propagation speed for the kinds of high temperature propagations that are merely indicated in RSST tests. However, the PRSST in this mode does not provide as faithful an adiabatic condition as the RSST, nor as complete an estimate of energy release until the heat capacity of the test cell can be estimated. You may wish to see comparative testing against actual RSST tests to determine if this mode of operation could replace the RSST.

G. The controller software should be updated to permit specifying pressure units of bar.

H. The gas line to the bomb should be fitted with a manipulatable bleed valve so that bomb pressure can be vented to the hot cell without loosening the bomb lid bolts.

I. Figure 6, Appendix II, pg II-19, of the Reference erroneously shows the large thermocouple prongs being closest to the bomb wall. This should be corrected to show them closest to the bomb center.

The PRSST has been shown to be able to initiate and measure a propagation in the hot cell with a propagatable material, if attention is paid to controlling the reintroduction of residual moisture. If the changes suggested above are incorporated, then the PRSST can provide a definitive test of propagation in actual waste.

Feel free to call if you have any questions on this matter.

D. B. Bechtold, Principal Scientist
Process Chemistry & Statistics

dls

Attachment

75764-PCS96-067

Attachment

Figures from PRSST Test Results

Consisting of 16 Pages including this cover page

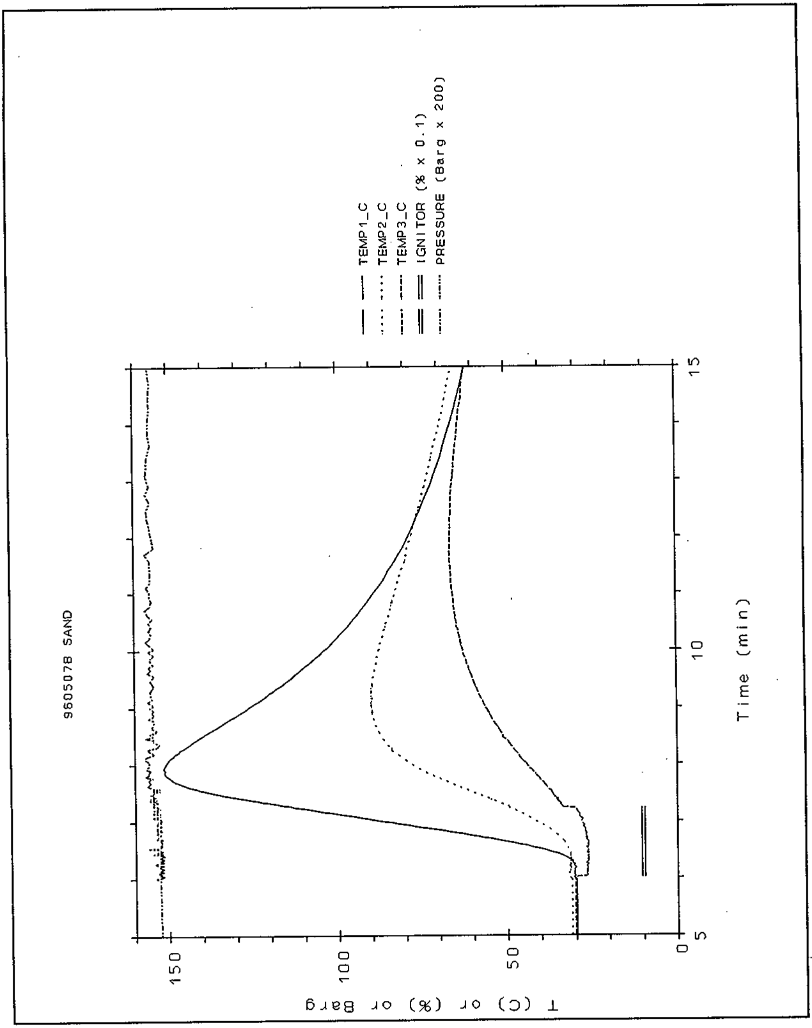
960507B SAND
TEMP1_C
TEMP2_C
TEMP3_C
IGNITOR (% x 0.1)
PRESSURE (Barg x 200)
T (C) or (%) or Barg
0
50
100
150
Time (min)
5
10
15

**Figure 1**

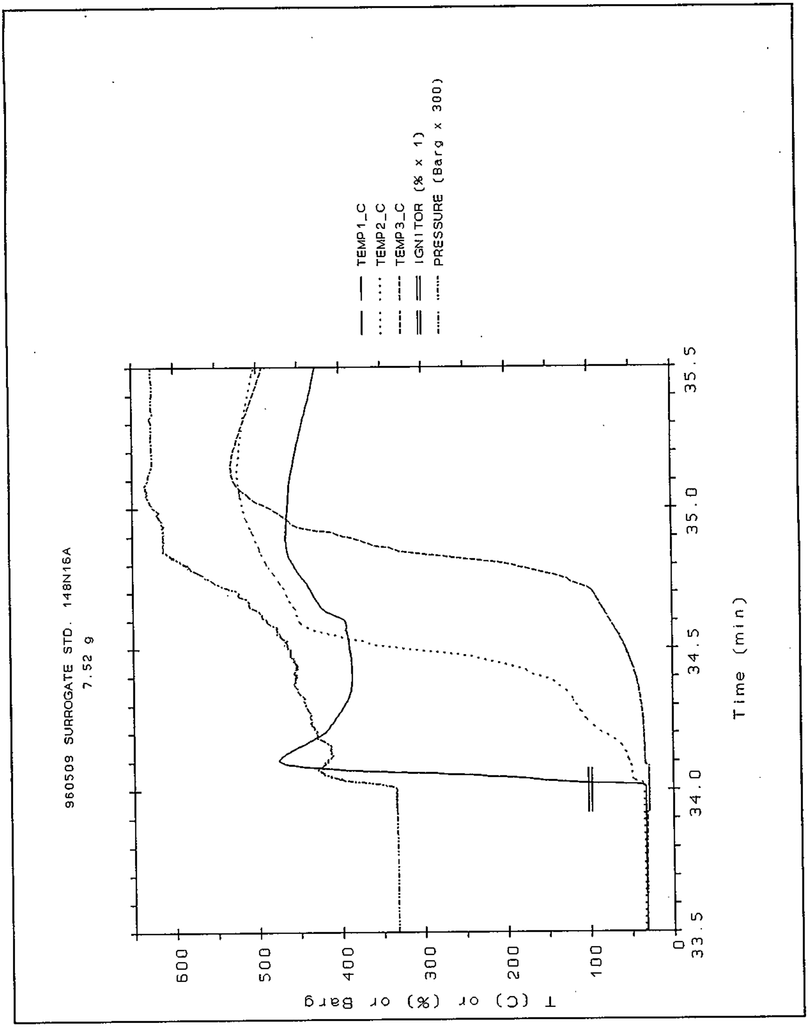
960509 SURROGATE STD. 148N16A
7.52 g
TEMP1_C
TEMP2_C
TEMP3_C
IGNITOR (% x 1)
PRESSURE (Barg x 300)
T (C) or (%) or Barg
0
100
200
300
400
500
600
33.5
34.0
34.5
35.0
35.5
Time (min)

**Figure 2**

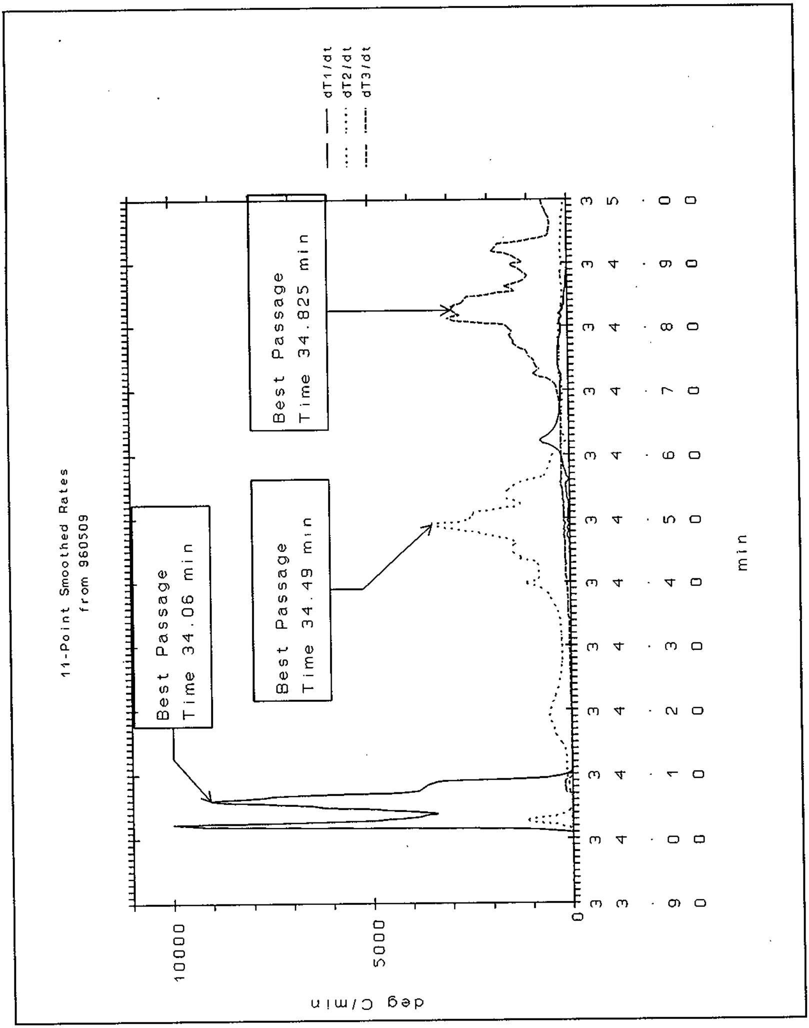
11-Point Smoothed Rates
from 960509
Best Passage
Time 34.06 min
Best Passage
Time 34.49 min
Best Passage
Time 34.825 min
dT1/dt
dT2/dt
dT3/dt
10000
5000
0
deg C/min
33.90
34.00
34.10
34.20
34.30
34.40
34.50
34.60
34.70
34.80
34.90
35.00
min

Figure 3

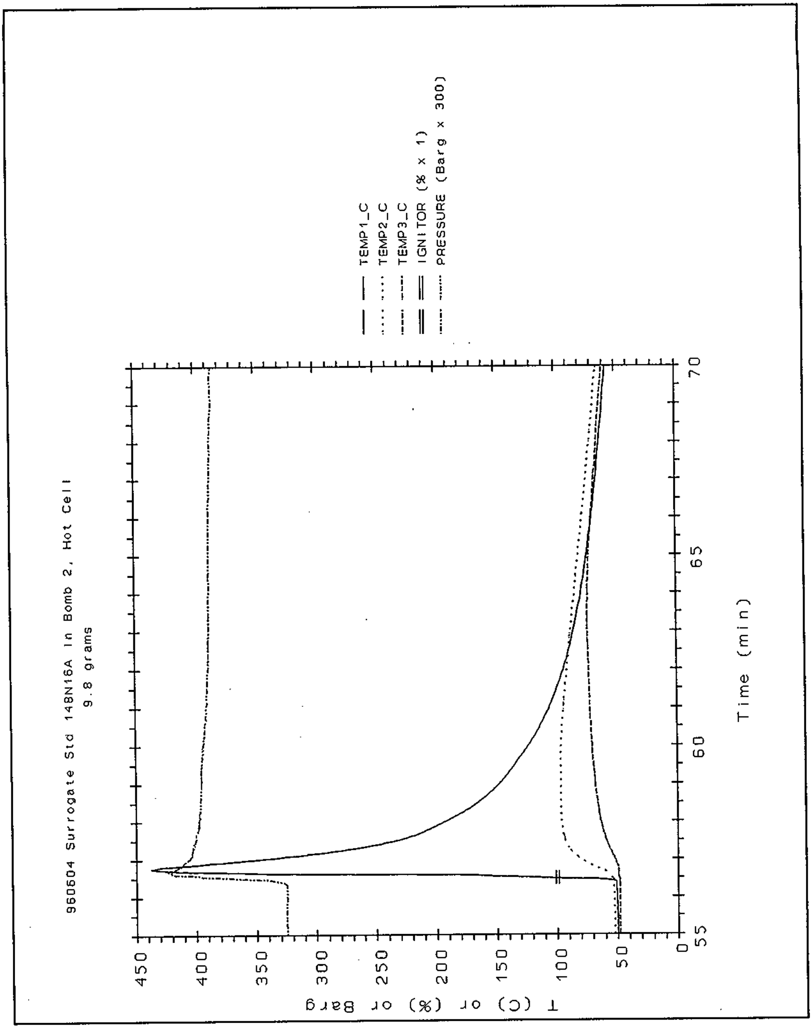
960604 Surrogate Std 148N16A In Bomb 2, Hot Cell
9.8 grams
TEMP1_C
TEMP2_C
TEMP3_C
IGNITOR (% x 1)
PRESSURE (Barg x 300)
T (C) or (%) or Barg
0
50
100
150
200
250
300
350
400
450
Time (min)
55
60
65
70

**Figure 4**

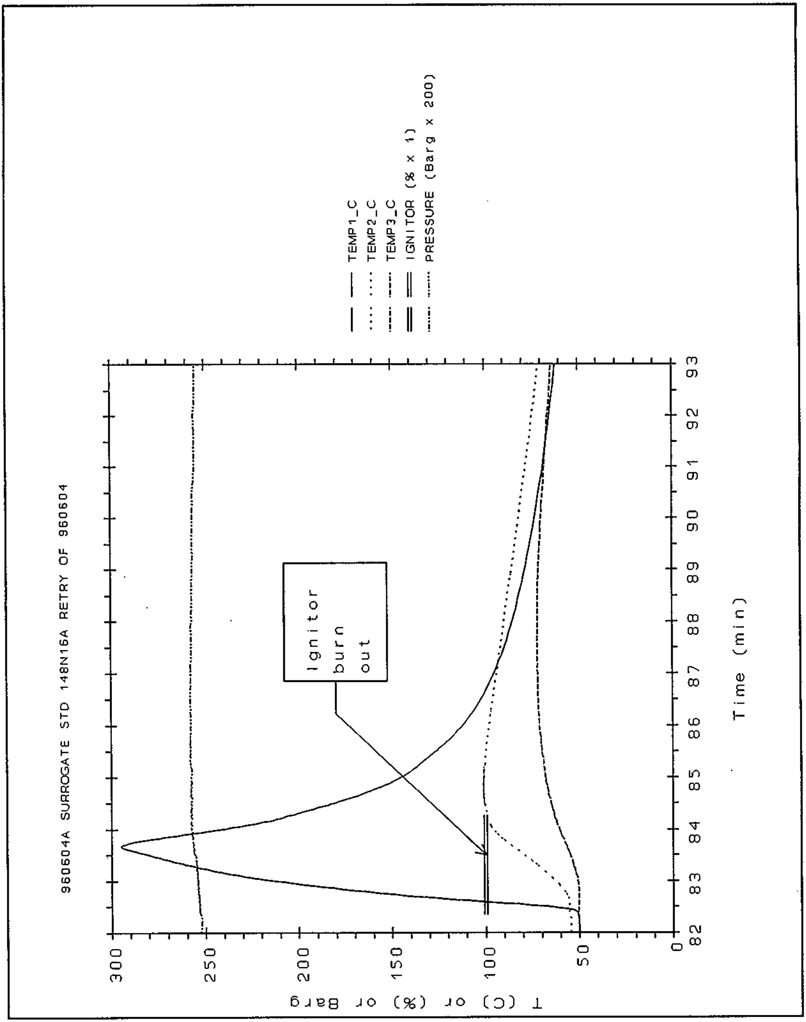
960604A SURROGATE STD 148N16A RETRY OF 960604
TEMP1_C
TEMP2_C
TEMP3_C
IGNITOR (% x 1)
PRESSURE (Barg x 200)
Ignitor burn out
T (C) or (%) or Barg
Time (min)
300
250
200
150
100
50
0
82
83
84
85
86
87
88
89
90
91
92
93

**Figure 5**

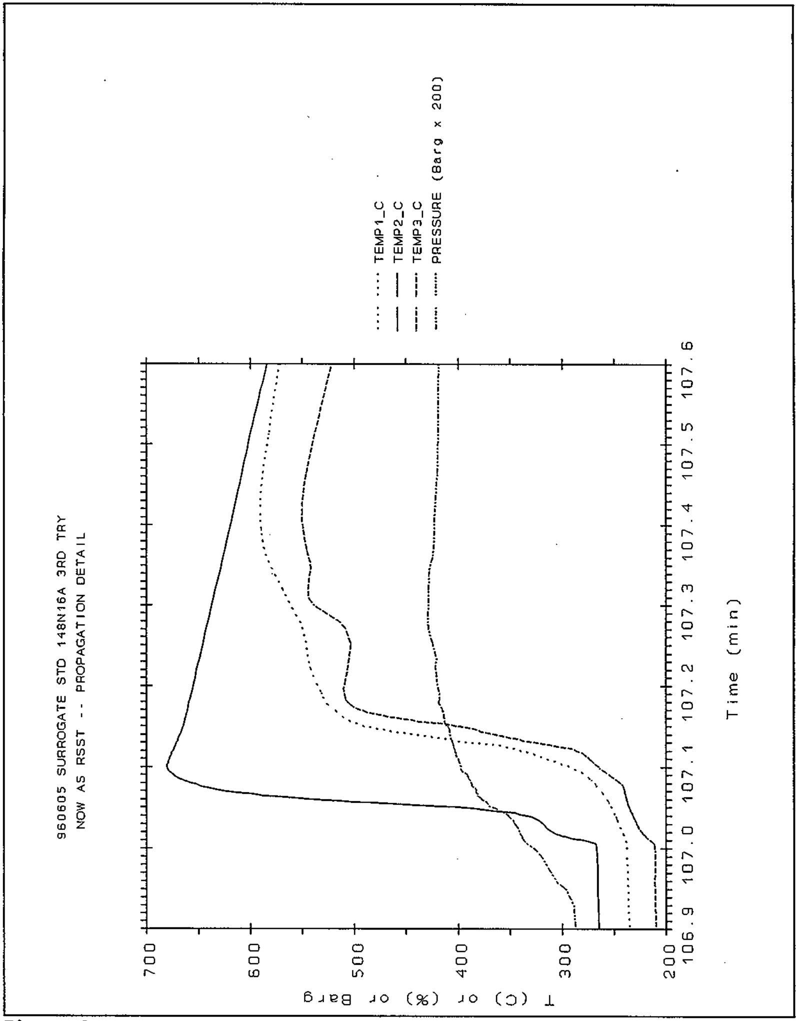
960605 SURROGATE STD 148N16A 3RD TRY
NOW AS RSST -- PROPAGATION DETAIL
TEMP1_C
TEMP2_C
TEMP3_C
PRESSURE (Barg x 200)
T (C) or (%) or Barg
Time (min)
200
300
400
500
600
700
106.9
107.0
107.1
107.2
107.3
107.4
107.5
107.6

Figure 6

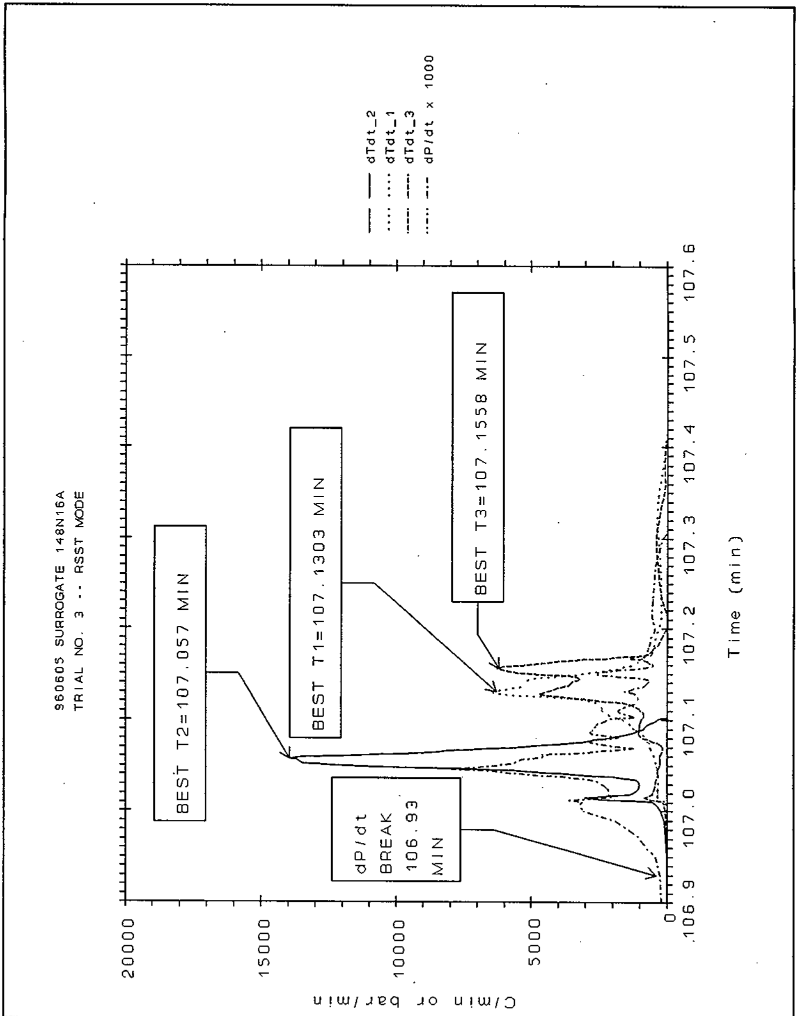
960605 SURROGATE 148N16A
TRIAL NO. 3 .. RSST MODE
dTdt_2
dTdt_1
dTdt_3
dP/dt x 1000
BEST T2=107.057 MIN
BEST T1=107.1303 MIN
BEST T3=107.1558 MIN
dP/dt BREAK 106.93 MIN
C/min or bar/min
Time (min)
20000
15000
10000
5000
0
106.9
107.0
107.1
107.2
107.3
107.4
107.5
107.6

Figure 7

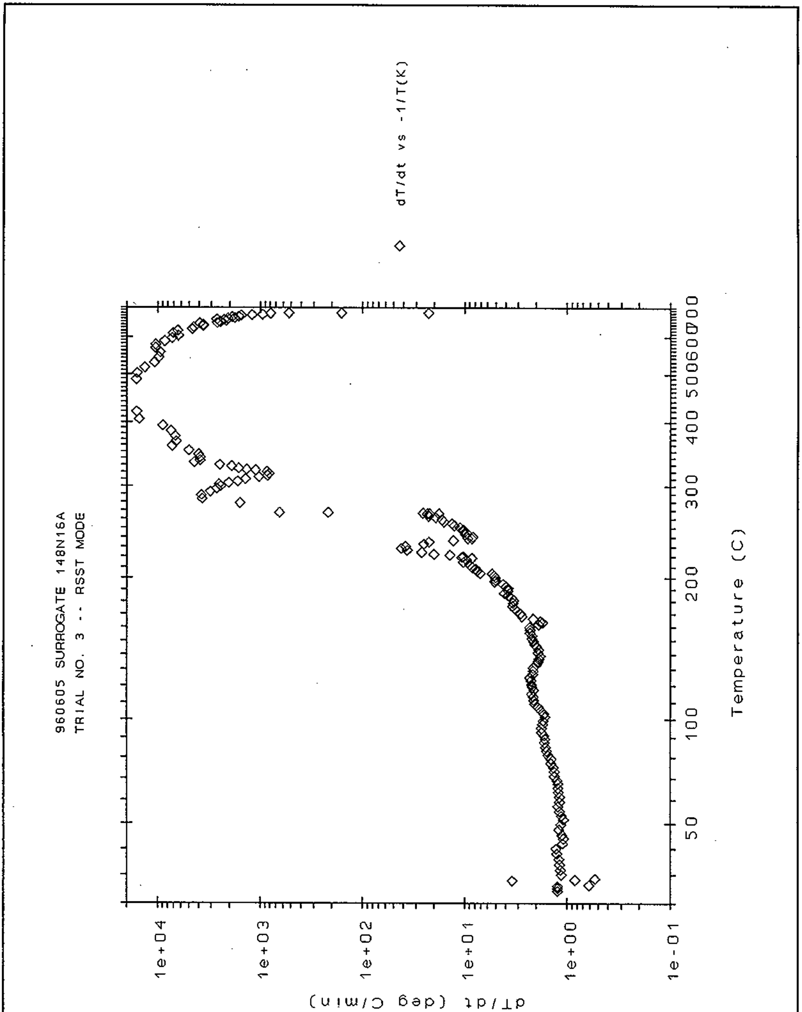
960605 SURROGATE 148N16A
TRIAL NO. 3 -- RSST MODE
dT/dt vs -1/T(K)
dT/dt (deg C/min)
1e+04
1e+03
1e+02
1e+01
1e+00
1e-01
Temperature (C)
50
100
200
300
400
500
600
700

Figure 8

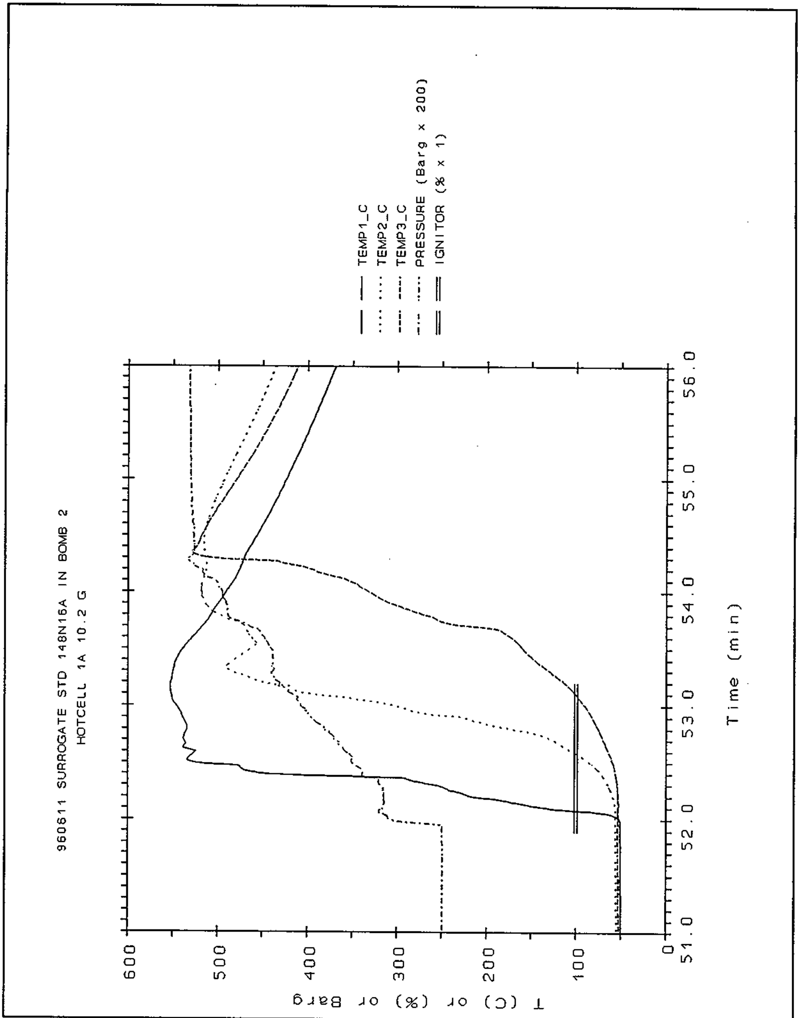
960611 SURROGATE STD 148N16A IN BOMB 2
HOTCELL 1A 10.2 G
TEMP1_C
TEMP2_C
TEMP3_C
PRESSURE (Barg x 200)
IGNITOR (% x 1)
T (C) or (%) or Barg
Time (min)
0
100
200
300
400
500
600
51.0
52.0
53.0
54.0
55.0
56.0

Figure 9

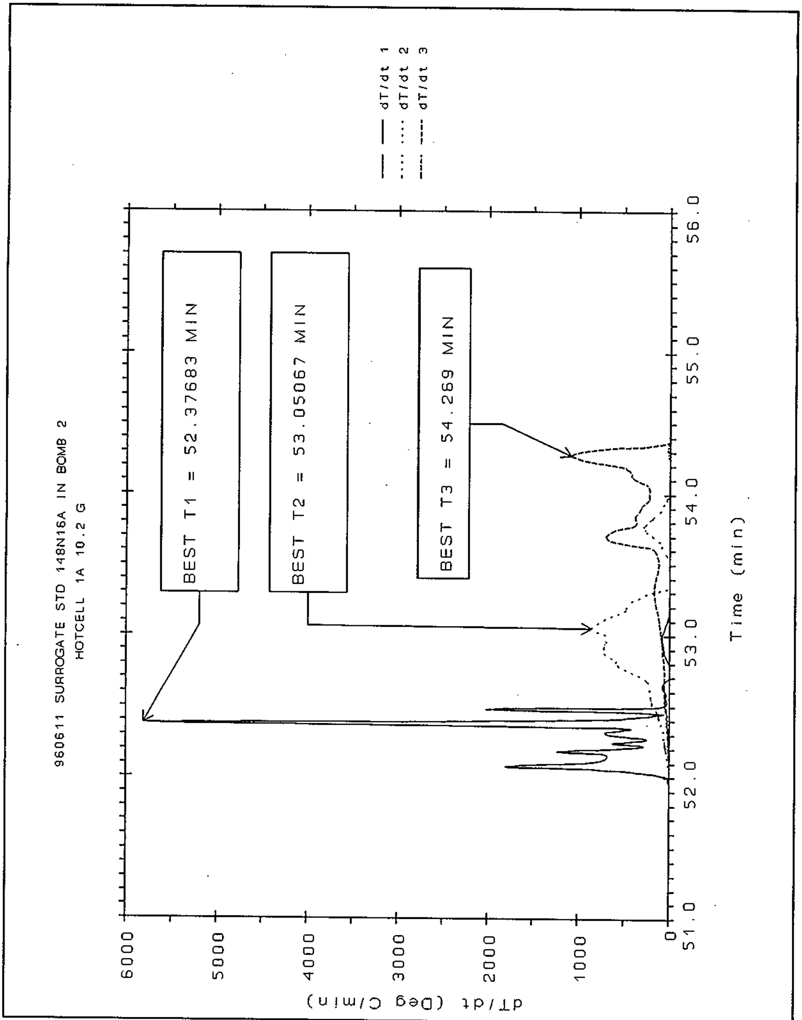
960611 SURROGATE STD 148N16A IN BOMB 2
HOTCELL 1A 10.2 G
BEST T1 = 52.37683 MIN
BEST T2 = 53.05067 MIN
BEST T3 = 54.269 MIN
dT/dt 1
dT/dt 2
dT/dt 3
dT/dt (Deg C/min)
Time (min)
0
1000
2000
3000
4000
5000
6000
51.0
52.0
53.0
54.0
55.0
56.0

Figure 10

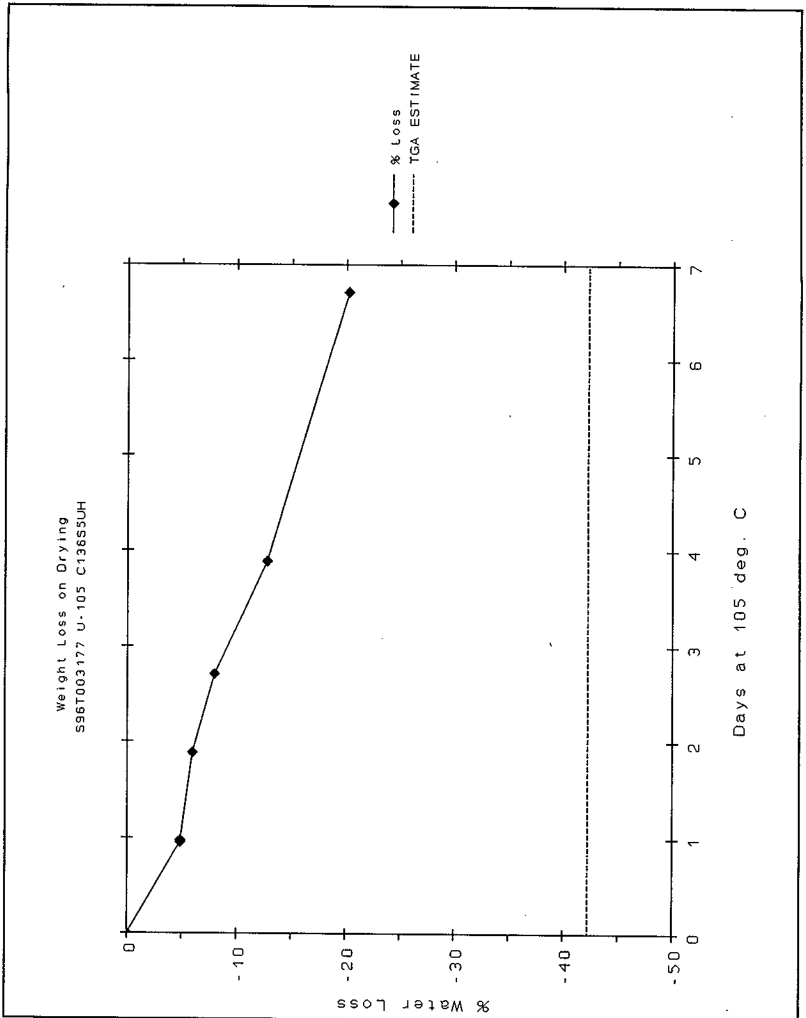
Weight Loss on Drying
S96T003177 U-105 C13655UH
% Loss
TGA ESTIMATE
% Water Loss
0
-10
-20
-30
-40
-50
Days at 105 deg. C
0
1
2
3
4
5
6
7

Figure 11

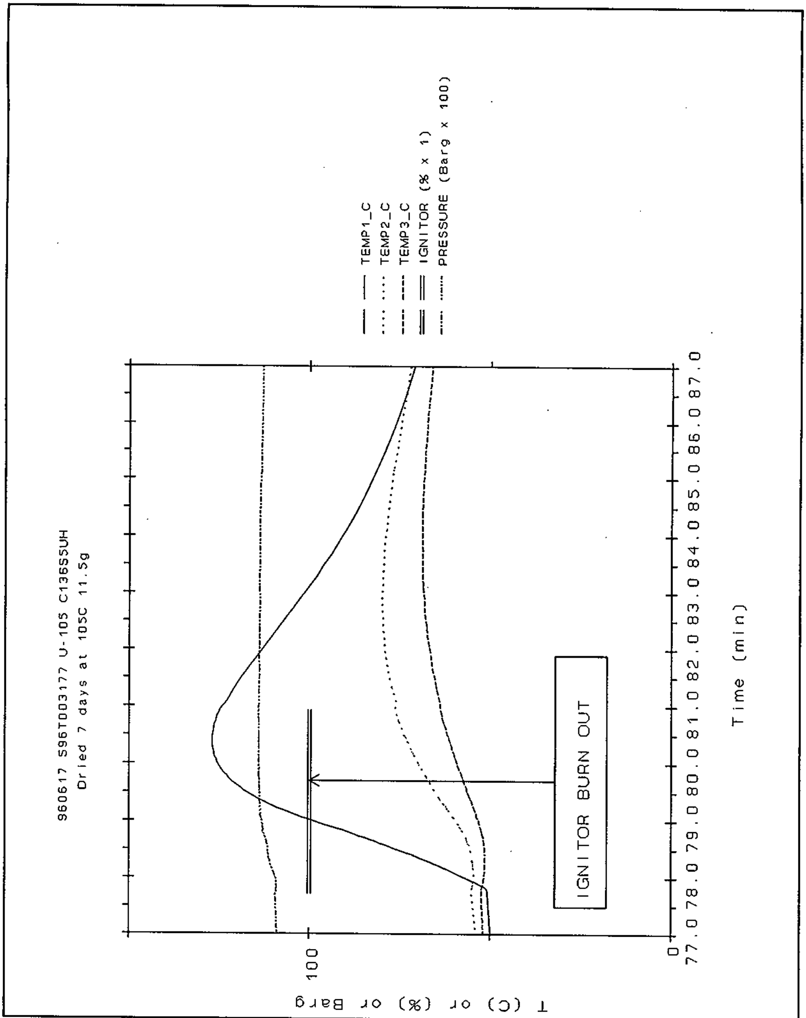
960617 S96T003177 U-105 C13655UH
Dried 7 days at 105C 11.5g
TEMP1_C
TEMP2_C
TEMP3_C
IGNITOR (% x 1)
PRESSURE (Barg x 100)
IGNITOR BURN OUT
T (C) or (%) or Barg
0
100
Time (min)
77.0 78.0 79.0 80.0 81.0 82.0 83.0 84.0 85.0 86.0 87.0

**Figure 12**

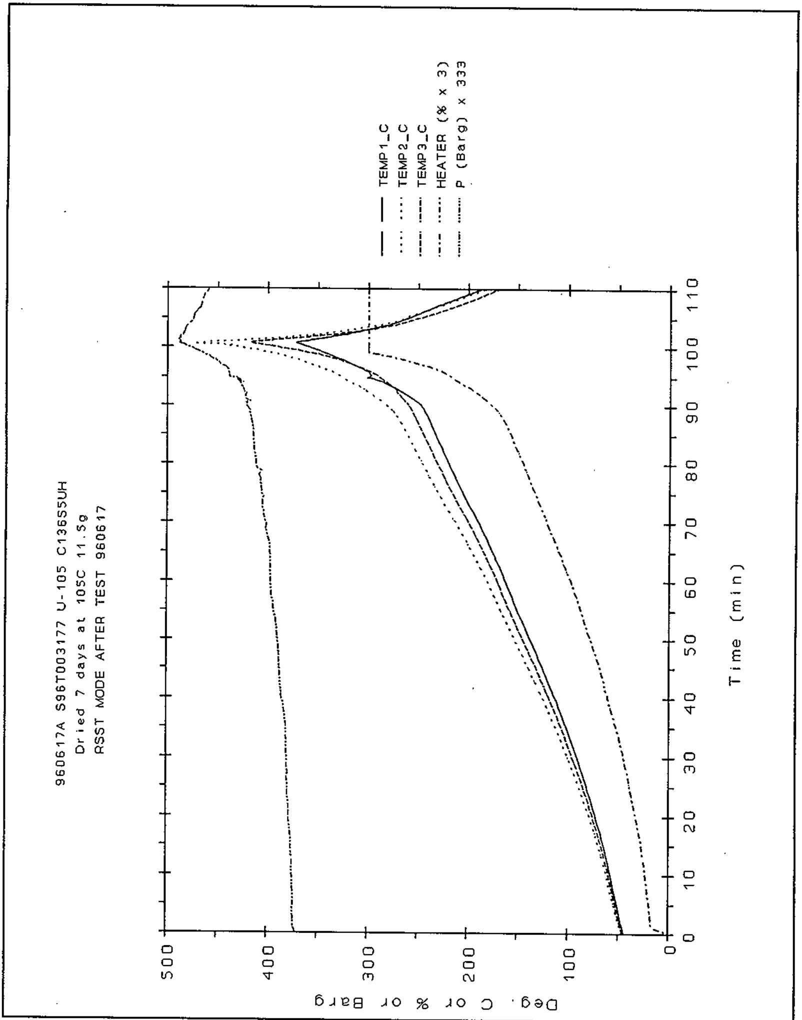
960617A S96T003177 U-105 C13655UH
Dried 7 days at 105C 11.5g
RSST MODE AFTER TEST 960617
TEMP1_C
TEMP2_C
TEMP3_C
HEATER (% x 3)
P (Barg) x 333
Deg. C or % or Barg
0
100
200
300
400
500
Time (min)
0
10
20
30
40
50
60
70
80
90
100
110

Figure 13

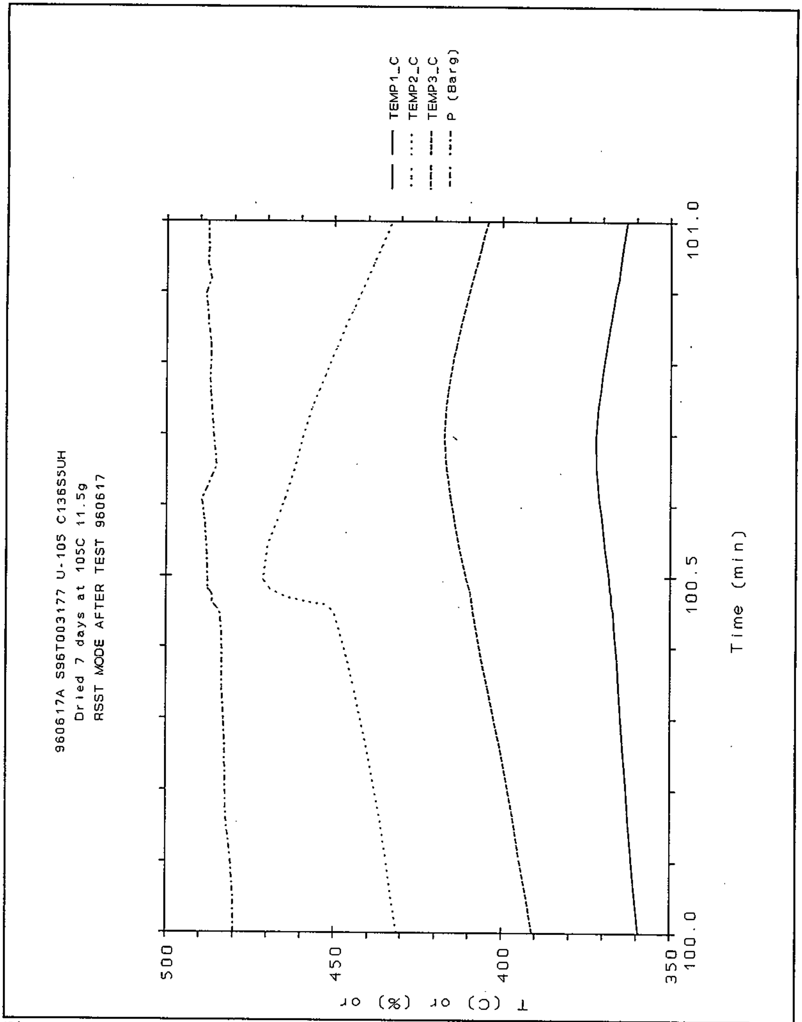
960617A S96T003177 U-105 C13655UH
Dried 7 days at 105C 11.5g
RSST MODE AFTER TEST 960617
TEMP1_C
TEMP2_C
TEMP3_C
P (Barg)
500
450
400
350
100.0
100.5
101.0
Time (min)
T (C) or (%) or

Figure 14

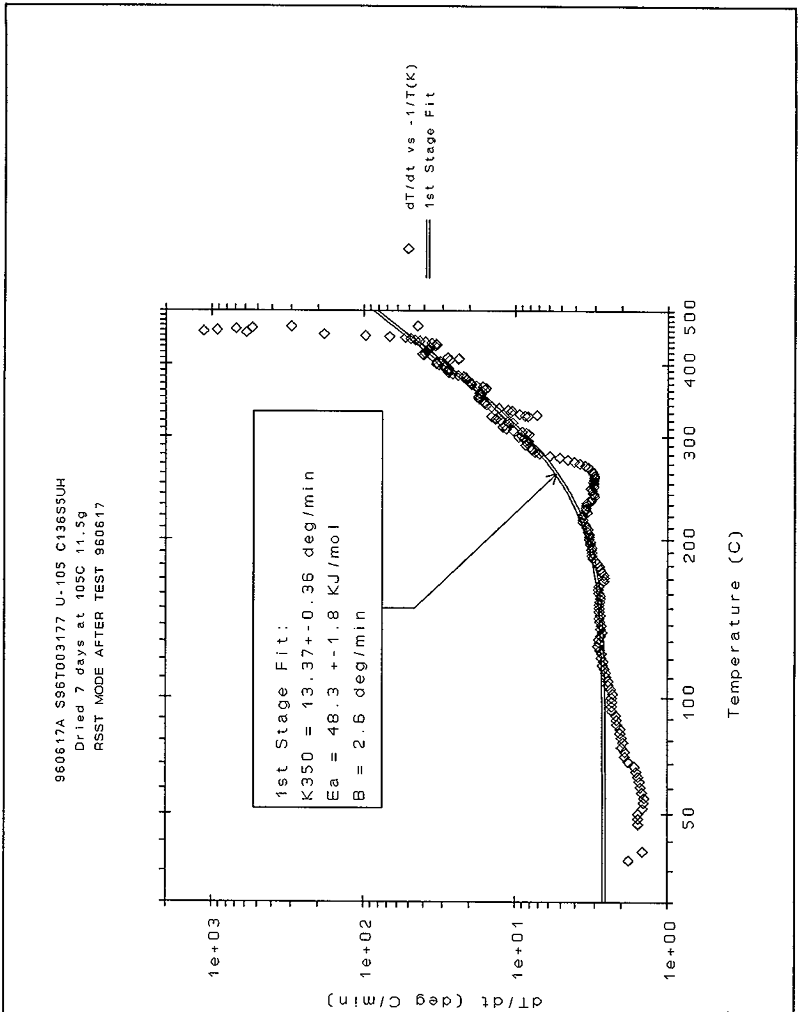
960617A S96T003177 U-105 C136S5UH
Dried 7 days at 105C 11.5g
RSST MODE AFTER TEST 960617
dT/dt vs -1/T(K)
1st Stage Fit
1st Stage Fit:
K350 = 13.37+-0.36 deg/min
Ea = 48.3 +-1.8 KJ/mol
B = 2.6 deg/min
dT/dt (deg C/min)
1e+03
1e+02
1e+01
1e+00
Temperature (C)
50
100
200
300
400
500

**Figure 15**

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