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Quarterly Report on Defense Nuclear Facilities Safety Board Recommendation 90-7

Prepared for the U.S. Department of Energy
Office of Environmental Restoration and
Waste Management

Westinghouse
Hanford Company Richland, Washington

Hanford Operations and Engineering Contractor for the
U.S. Department of Energy under Contract DE-AC06-87RL10930

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G. T. Dukelow

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QUARTERLY REPORT ON DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7

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ABSTRACT

This is the second quarterly report on the progress of activities addressing safety issues associated with Hanford Site high-level radioactive waste tanks that contain ferrocyanide compounds. An implementation plan (Cash 1991) responding to the Defense Nuclear Facilities Safety Board Recommendation 90-7 (FR 1990) was issued in March 1991 describing the activities planned and underway to address each of the six parts of the recommendation.

All of the activities listed in the implementation plan are underway, including the multifunctional instrument tree and infrared tasks which resumed in late July. Although technical difficulties and resource limitations delayed some work, noteworthy progress has been made in completing a number of ferrocyanide program milestones. Thermal modeling shows that the heat loading of tank 241-BY-104 is much lower than previously listed and that significant hot spots within the waste are highly unlikely, if not impossible. Computerized continuous temperature monitoring was installed on schedule on five of the highest interest tanks and five additional tanks will be on-line in December. Temperature readings show that the scatter previously encountered is eliminated with this new system. Accuracy checks of tank instrument trees shows most thermocouples are reading correctly and many more are usable than listed previously. Tank intrusive sampling is proceeding and the first vapor samples were obtained in mid-October. Spectral scans were

completed for twelve tanks and substantial progress was made on design of an infrared scanning system. Chemical reaction studies are underway with synthetic ferrocyanide compounds believed to be more representative of the ferrocyanide materials actually deposited in the tanks. Tests indicate that water plays a major role in impeding a possible runaway reaction. An emergency planning exercise was conducted in May, emergency procedures were updated this quarter, and validation of the procedures and a second emergency exercise is scheduled for October.

Cash, R. J., 1991, *Implementation Plan for the Defense Nuclear Facilities Safety Board Recommendation 90-7*, WHC-EP-0415, Westinghouse Hanford Company, Richland, Washington.

FR, 1990, "Implementation Plan for Recommendation 90-3 at the Department of Energy's Hanford Site, WA," *Federal Register*, DNFSB Recommendation 90-7, Vol. 55, No. 202, pp. 42243 - 42244.

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LIST OF TERMS

ASC	adiabatic scanning calorimetry
CASS	Computer Automated Surveillance System
CdTe	cadmium telluride
CHN	carbon, hydrogen, and nitrogen
CRT	cathode ray tube
CTMS	Continuous Temperature Monitoring System
DNFSB	Defense Nuclear Facilities Safety Board
DOE	U.S. Department of Energy
DOE-HQ	U.S. Department of Energy-Headquarters
DSC	differential scanning calorimetry
EA	Environmental Assessment
EDS	energy dispersive spectroscopy
EPA	U.S. Environmental Protection Agency
FONSI	Finding of No Significant Impact
FY	fiscal year
ICP	inductively coupled plasma
IR	infrared spectrometry
LANL	Los Alamos National Laboratory
LFL	lower flammability limit
LOW	liquid observation well
NEPA	National Environmental Policy Act
MIT	Multifunctional Instrument Tree
PNL	Pacific Northwest Laboratory
risers	available openings in tanks
RL	DOE Field Office, Richland
SA	Safety Assessment
SEM	scanning electron microscopy
TAP	tank advisory panel
TC	total carbon
TGA	thermogravimetric analysis
TOC	total organic carbon
TRAC	Track Radioactive Components
Tri-Party Agreement	*Hanford Federal Facility Agreement and Consent Order*
TTX	time-to-explosion
UR	uranium recovery
WHC	Westinghouse Hanford Company
XRD	x-ray diffraction

QUARTERLY REPORT ON DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7

1.0 INTRODUCTION

1.1 PURPOSE

This quarterly report* provides a status of the activities underway at the Hanford Site on the ferrocyanide safety issues as requested by the Defense Nuclear Facilities Safety Board (DNFSB) in their Recommendation 90-7 (FR 1990). In March 1991, an implementation plan (Cash 1991) was prepared and sent to the DNFSB responding to the six parts of Recommendation 90-7. All of the activities in the implementation plan are underway, and the status of each is described in Section 2.0.

1.2 QUARTERLY HIGHLIGHTS

This section highlights major accomplishments for the quarter. Highlights are summarized briefly

- Work resumed on development of the Multifunctional Instrument Tree (MIT). To expedite installation of MITs in watchlist tanks, Los Alamos National Laboratory will design and fabricate three prototype units for the Hanford Site.

- Thermocouple trees will be fabricated on site using existing tree designs already utilized in the waste tanks. Four units will be installed in fiscal year (FY) 1992.

- The infrared scanning system will deployed in one of the ferrocyanide tanks for a proof of principle test. Transient thermal modeling will start in FY 1992 to determine if waste surface temperatures will increase soon enough to detect a possible hot spot before its temperature becomes a concern.

- Thermocouple accuracy checks were completed this quarter. Many thermocouples not used previously were found to be functional and in good condition. Other elements appear to be repairable.

- Thermal modeling of Tank 241-BY-104 shows that the heat loading is less than half of that listed in the table (Appendix A). Air infiltration into the tank plays a major role in determining the heat generated in the tanks. The hot spot modeling report was completed and is being cleared for public release.

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*See Appendix B for metric-to-English and English-to-metric conversions.

- The computer-based continuous temperature monitoring and alarm system is operational and five BY Farm tanks are now being surveyed continuously. The readings are steady and free of the scatter prevalent before.

- Preparations were nearly completed during the quarter for vapor space sampling of the first ferrocyanide tank (241-BY-104). The readiness review was concluded and pre-start items are nearing final disposition. Sampling is scheduled to start by mid-October.

- Safety and environmental documentation for surface sampling and push mode core sampling were completed and forwarded to the U.S. Department of Energy for review and approval. A revised safety assessment was submitted for vapor sampling all remaining ferrocyanide tanks. Approval of this documentation is still pending.

- The readiness review for auger surface sampling was started and 75 percent complete at the end of the quarter. Preparations are in progress for push mode core sampling of tank 241-C-112 by December 1991.

- Initial characterization of synthetic ferrocyanide chemicals produced by the various flowsheets was completed. Planned work for FY 1991 was completed for chemical reaction studies, the chemical nature of iron and cyanide in wastes, and energetic studies using synthetic ferrocyanide-containing wastes. Detailed results are discussed in Section 2.5.1 of the quarterly report.

- A propagation test program was started this quarter to determine which waste properties play a key role in mitigating or inhibiting a ferrocyanide reaction. Flowsheet material representative of the uranium recovery process will not propagate even when completely dry and heated to 350 °C.

- Revised emergency preparedness procedures were developed this quarter to respond to site wide emergencies, especially at locations such as Tank Farms. The procedures will be validated with an exercise planned for the first quarter of FY 1992.

1.3 REPORT FORMAT

The quarterly report on progress of activities under each of the six parts of DNFSB Recommendation 90-7 is arranged in the same order as in the Implementation Plan (Cash 1991). The arrangement also follows the same order provided in the recommendation. To report on progress, each part of the recommendation is repeated in italics followed by one or more paragraphs explaining the scope of work on each part or subpart of the recommendation. Subheadings for each task activity report the following items of progress:

- Progress During Reporting Period
- Planned Work For Subsequent Months

- Problem Areas and Action Taken
- Milestone Status.

1.4 BACKGROUND

Radioactive wastes from defense operations have accumulated at the Hanford Site in underground waste tanks since the early 1940's. During the 1950's, additional tank storage space was required to support the defense mission. To obtain this additional storage volume within a short period of time and without constructing additional storage tanks, Hanford Site scientists developed a process to scavenge radiocesium from tank waste liquids. In implementing this process, approximately 140 metric tons of ferrocyanide were added to 24 single-shell tanks.

Ferrocyanide is a stable complex of ferrous ion and cyanide that is considered nontoxic because it does not dissociate in aqueous solutions. However, in the presence of oxidizing materials, such as nitrates/nitrites, ferrocyanide can be made to explode in the laboratory by heating it to high temperatures (above 285 °C [545 °F]) or by an electrical spark of sufficient energy. The explosive nature of ferrocyanide in the presence of an oxidizer has been known for decades, but the conditions under which the compound can undergo an uncontrolled exothermic reaction have not been thoroughly studied. Because the scavenging process involved precipitating ferrocyanide from solutions containing nitrate and nitrite, it is possible that an intimate mixture of ferrocyanides and nitrates/nitrites exists in parts of some of the single-shell tanks.

Efforts have been underway since the mid-1980's to evaluate the potential for a ferrocyanide explosion in the Hanford Site single-shell tanks (Burger 1989; Burger and Scheele 1988). In 1987, the environmental impact statement, *Final Environmental Impact Statement - Disposal of Hanford Defense High-Level, Transuranic and Tank Wastes*, (DOE 1987) was issued. The environmental impact statement projected that the bounding "worst-case" accident in a ferrocyanide tank would be an explosion resulting in a subsequent short-term radiation dose to the public of 200 mrem.

A recently completed General Accounting Office study (Peach 1990) postulates a greater "worst-case" accident with independently calculated doses one to two orders of magnitude greater than the 1987 environmental impact statement (DOE 1987). A special Hanford Site Ferrocyanide Task Team was commissioned in September 1990 to address all issues involving the ferrocyanide tanks, including the consequences of a potential accident. On October 9, 1990, Secretary of Energy Admiral James D. Watkins announced (DOE 1990) that a supplemental environmental impact statement would be prepared containing an updated analysis of safety questions for the Hanford Site single-shell tanks (including a ferrocyanide explosion).

Using process knowledge and transfer records, an evaluation process which is still ongoing, 24* tanks have been identified at the Hanford Site as containing 1,000 g-mole (465 lb) or more of ferrocyanide as the $Fe(CN)_6$ radical. In October 1990, the ferrocyanide issue was declared an Unreviewed Safety Question** because the safety envelope for these tanks may no longer be bounded by the existing safety analysis report (WHC 1986). Work in and around any of the ferrocyanide tanks requires detailed planning with the preparation of supporting safety and environmental documentation and approval by the U.S. Department of Energy (DOE) top management. These restrictions are required for safety and greatly increase the time required to complete work or install equipment in the tanks.

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*Two more tanks potentially containing ferrocyanide were identified since DOE responded to Recommendation 90-7 (FR 1990) in November 1990.

**Unreviewed Safety Question as defined by DOE Orders 5480.5 (DOE 1986) and N 5480.YY (DOE draft), "A proposed change, test or experiment shall be deemed to involve an Unreviewed Safety Question if:

1. The probability of occurrence or the consequences of an accident or malfunction of equipment important to safety, evaluated previously by safety analysis will be significantly increased, or

2. A possibility for an accident or malfunction of a different type than any evaluated previously by safety analysis will be created which could result in significant safety consequences."

2.0 IMPLEMENTATION PLAN TASK ACTIVITIES

The implementation plan (Cash 1991) addresses each task activity set up in response to the six parts of DNFSB Recommendation 90-7 (FR 1990). In this progress report, each part of the recommendation is stated, and then the progress of activities relating to that part is described.

2.1 DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7.1

"Immediate steps should be taken to add instrumentation as necessary to the single-shell tanks containing ferrocyanide that will establish whether hot spots exist or may develop in the future in the stored waste. The instrumentation should include, as a minimum, additional thermocouple trees. Trees should be introduced at several radial locations in all tanks containing substantial amounts of ferrocyanide, to measure the temperature as a function of elevation at these radii. The use of infra-red techniques to survey the surface of waste in tanks should continue to be investigated as a priority matter, and on the assumption that this method will be found valuable, monitors based on it should be installed now in the ferrocyanide bearing tanks."

2.1.1 Installation of New Multifunctional Instrument Trees

Fifty-three high-level radioactive waste tanks at the Hanford Site have been declared watchlist tanks. These include tanks containing ferrocyanide compounds, organic compounds, high-heat-producing tanks, and tanks known to produce hydrogen. These tanks require improved monitoring capabilities for the condition of the tank contents.

Many of the existing thermocouple trees in the watchlist tanks were installed when the tanks were constructed or were added later but are of similar design. In several of the tanks, the thermocouple trees are inoperative; also, some thermocouples in certain remaining trees are not functional. In most cases, the faulty thermocouples cannot be replaced. In some cases, the historical data from these thermocouple trees are suspect.

To comply with this recommendation, new thermocouple trees must be fabricated and installed. Because the number of available openings (risers) in the single-shell tanks is limited and several thermocouple trees are needed in the watchlist tanks at statistically significant locations, a multifunctional monitoring capability is being incorporated into the design.

- **Progress During Reporting Period.** Planning work on the Multifunctional Instrument Tree (MIT) was resumed in late June. It was decided to re-evaluate the overall waste tank data requirements, revise the criteria, and define the actual data needed for surveillance. The necessary surveillance data was determined to be temperature monitoring, pressure sensing, and gas sampling.

Characterization data requirements such as liquid and sludge sampling, etc., shall be obtained by other programs.

With the surveillance requirements re-defined, the MIT design requirements were reviewed in a joint effort with Los Alamos National Laboratory (LANL). This team effort proved very beneficial and resulted in revised criteria that will simplify the design and fabrication of the MIT. LANL will design and fabricate three prototype units for deployment in Hanford watchlist tanks. Of the three MITs to be obtained, one tree is to be used for test purposes, one tree is to be installed in 241-BY-104, and the remaining tree will be installed in a waste tank undetermined at this time, or possibly in flammable gas-generating tank 241-SY-101.

A strategy has been developed to provide in fiscal year (FY) 1992 the thermocouple trees necessary to monitor conditions more closely in five waste tanks of high concern. The strategy uses existing thermocouple tree designs already deployed in the waste tanks. In addition the strategy will also use two existing thermocouple trees stored on site. Slight modifications are required to these two trees to obtain adequate temperature information. This will allow installation of additional temperature measurement capabilities in the tanks earlier than projected. Implementing this strategy is considered a positive step toward achieving overall waste tank safety and remediation goals.

- **Planned Work for Subsequent Months**. The new strategy addresses the priority and basis for installing thermocouple trees and multifunctional instrument trees in the waste tanks that are judged to be of highest concern with regard to safety. Thermocouple trees will be installed into each of the tanks 241-BY-104, 241-C-112, 241-BY-110, and 241-C-109. An MIT will be installed into 241-BY-104 in addition to the thermocouple trees.

  LANL plans to develop a draft schedule and cost estimate for design and fabrication of the three MITs by the end of October.

- **Problem Areas and Action Taken**. Installation of the MITs was delayed because to the initial design criteria were too complex. This led to a costly design concept and difficult features to fabricate using normal shop fabrication methods. The deputy manager of Westinghouse Hanford Company (WHC) Tank Farm Projects was appointed to lead this task. A dedicated group of engineering personnel was also selected.

- **Milestone Status**. Because of the problems described above, the first MIT will not be installed in a ferrocyanide tank until later in FY 1992. Thermocouple trees will be installed earlier, starting in the second quarter of FY 1992.

2.1.2 Alternate Tank Monitoring Technologies

Alternate technologies for monitoring the ferrocyanide tanks include possible infrared scanning of tanks for waste surface temperature changes, spectral gamma scanning and neutron scanning in 12 tanks with liquid observation wells (LOW), upgrades to existing tank monitoring instrumentation, and hot spot thermal modeling. Each of these tasks will be discussed in this section.

2.1.2.1 Infrared Imaging System. Infrared imaging systems are commercially available from numerous vendors. Because these systems are sensitive to changes of ± 0.5 °C or less, they may prove very beneficial for mapping the surface temperature profiles in the ferrocyanide tanks. Thermal modeling performed on ferrocyanide tank 241-BY-104 shows that if hot spots with temperatures of concern are possible, surface temperature differences should be great enough to be detected by infrared mapping, assuming surface irregularities and other factors affecting sensitivity are negligible or can be accounted for during analysis of infrared tank data. However, the hot spot temperature may increase too rapidly to be detected in time at the surface of the waste. Transient modeling to predict how fast the waste surface temperature would change is to be conducted in FY 1992 (see Section 2.1.4).

One drawback of an infrared imaging system is the limited life caused by gamma radiation exposure to the semiconductor components within the scanner. Based upon an average radiation level within the single-shell tanks of 150 R/h, the useful life of an infrared scanner may be limited to approximately 100 h. Therefore, deployment for surface monitoring would have to be done periodically, perhaps monthly, unless tank anomalies dictate otherwise. Another concern is the dependence of measuring a surface temperature on the emissivity of the surface and its ability to emit infrared radiation. This property changes with variations in surface composition, texture, moisture content, and angle of incidence. Because the waste surface in most of the tanks is not uniform, accurate temperature measurement may not be possible; however, temperature mapping may be useful to detect potential hot spots and for historical comparison when one scan is compared with another taken at a different time.

- **Progress During Report Period.** Design of the deployment system which includes the enclosure, support stem, purge system, and electrical was 95 percent complete at the end of the quarter. Drawings are in the review and approval process. A formal design review committee has been selected and will begin the formal system review early next quarter. All purchase orders except for minor miscellaneous items have been submitted to the shops for estimating and scheduling purposes. An estimate of fabrication delivery dates has not yet been received, but this information will be put into the overall schedule as soon as received in October.

  A structural/seismic analysis required for all Safety Class 2 equipment has begun and will be completed in mid-October. A thermal analysis is in process to evaluate purge requirements to maintain the scanner at the vendor-specified temperature.

Additional preliminary testing, as described below, was continued during this reporting period. It is expected that further testing will confirm the results of these preliminary tests. Simulant which matches the expected composition of the actual waste in 241-BY-104 was ordered and received in late September. This material will be used to complete the planned tests. The preliminary tests were done using sugar water and urea, a simulant that was more readily available. Tests done by burying a heat source, along with thermal modeling, appear to indicate that surface temperature changes from a hot spot located any significant distance beneath the surface of the waste, using infrared technology, may not occur soon enough to allow actions to be taken to prevent the temperature of a hot spot from becoming a safety concern. In addition, photographs taken of the waste surface in the mid-1980s show pools of liquid, significant surface irregularities, and foreign material on the surface which may result in temperature variations as large or larger than those which would be expected from a hot spot near the bottom of the tank. Additional analyses to determine the effect of surface irregularities will be completed during the next reporting period.

Various other preliminary tests related to calibration, environmental conditions, and system operation were done. Results of transmission tests indicate that, in atmospheres of given humidity, the accuracy of the indicated temperatures varies as a function of distance. The higher the humidity, the greater the accuracy variation. There is a potential that this may result in difficulty relating infrared scans taken at different times and/or under varying conditions to each other. It would also mean that the accuracy could vary significantly across the wide angle field of view of individual scans.

A safety assessment (SA) for deployment and operation of the infrared system in the ferrocyanide tanks is being prepared and is expected to be transmitted to DOE by the end of October. The Environmental Assessment (EA) for the National Environmental Policy Act (NEPA) was begun. It is expected, however, that the approvals will not be completed, including all State reviews until March 1992. Current review and approval times are longer than originally scheduled. Additional time required for the NEPA review and approval process which includes the U.S. Department of Energy-Headquarters (DOE-HQ), the DOE Field Office, Richland (RL), and Washington State is projected to delay the demonstration of the system in tank 241-BY-104 until April 1992.

- **Planned Work For Subsequent Months**. Design, fabrication, analysis, and formal design review of the system as well as delivery of the pan and tilt unit will be completed during the next reporting period. The SA and the EA will be completed and transmitted to the RL to initiate their review and approval process. Final assembly and testing of the system, and approvals for installation and operation, should be completed and ready for deployment by the end of March 1992.

- **Problem Areas and Action Taken.** Preliminary testing and thermal modeling of the tank indicate that the stated purpose of the Infrared Scanning System may not be met because of the possible slow temperature response at the surface to hot spots in the tank. Increased design time is projected to cause a delay in the fabrication and assembly to early in the second quarter of 1992. However, the projected critical path is still the NEPA review.

- **Milestone Status.** Capital funds were made available early in this reporting period which allowed resumption of work. The schedule was impacted by an increased amount of time for design, and by the increased external agency review and approval time for safety assessments and NEPA requirements. Worst case estimates are that scanning of the 241-BY-104 tank may be as late as April 1992, and completion of the draft report in May 1992. An alternative plan is being developed to scan a non-watchlist tank which would not require NEPA approvals, a safety assessment, and a detailed readiness review. This alternative would have the advantage of more flexibility such as lowering thermocouples to the surface of the waste to confirm temperatures seen by the infrared camera.

2.1.2.2 Gamma and Neutron Scans in Liquid Observation Wells. Other increased monitoring now done on applicable ferrocyanide tanks at the Hanford Site includes gamma scans and neutron probe scans. These techniques can only be applied for tanks that have LOWs. The LOW is a closed-end, nonmetallic (sometimes fiberglass) tube approximately 10 cm (4 in.) in diameter that enters the tank through a riser and extends to near the tank bottom. Twelve ferrocyanide tanks have LOWs. In some cases, the LOWs are also used for measuring the waste temperature by placing a thermocouple into the well.

Routine gamma scans show gross radiation levels within a tank and neutron scans show moisture, both as a function of elevation. The liquid level and the salt cake level are both discernable in the tank. The WHC scientists are using a new state-of-the-art gamma detector, cadmium telluride (CdTe), that allows characterization of certain isotopes within the waste as a function of elevation. In addition, new signal processing and analyses of gamma and neutron scans are being applied to obtain useful information previously not available, such as percent moisture content as a function of elevation. Gamma and neutron count rates are recorded at 3 cm (0.1-ft) intervals to provide a nearly continuous scan along the height of the tank. In the past, the gross scans were used primarily for tank liquid-level measurement and to identify potential leaking tanks.

- **Progress During Reporting Period.** Collimated spectral gamma scans have been obtained for cesium concentration measurements in the twelve ferrocyanide tanks with installed LOWs. The spectral gamma scan detector was calibrated to determine cesium concentration for an assumed waste density. Tank cesium inventories and heat loads associated with cesium have been calculated for these tanks based upon these scans. The tank cesium inventories and associated heat loads are listed in the table below. The scans also show that the maximum concentration of cesium measured (275 μCi/g) was in tank 241-BY-106 near the bottom of the tank. This concentration is equivalent to 3.16 x 10 E-06 g Cs/g of waste. Some question on the

application of the LOW gamma scans still exists because of the manner in which LOWs were installed. Water lancing was used to wash a hole into the waste for insertion of the LOW. The readings obtained in the LOWs may not represent horizontal concentrations throughout the 22.9 m (75 ft) diameter waste tanks since the scans only monitor gamma emissions up to a distance of about 30 cm (12 in.) radius. Further evaluations are needed to determine the applicability of observed results. There is general agreement, however, between the cesium inventories determined by historical accounting and spectral gamma scanning.

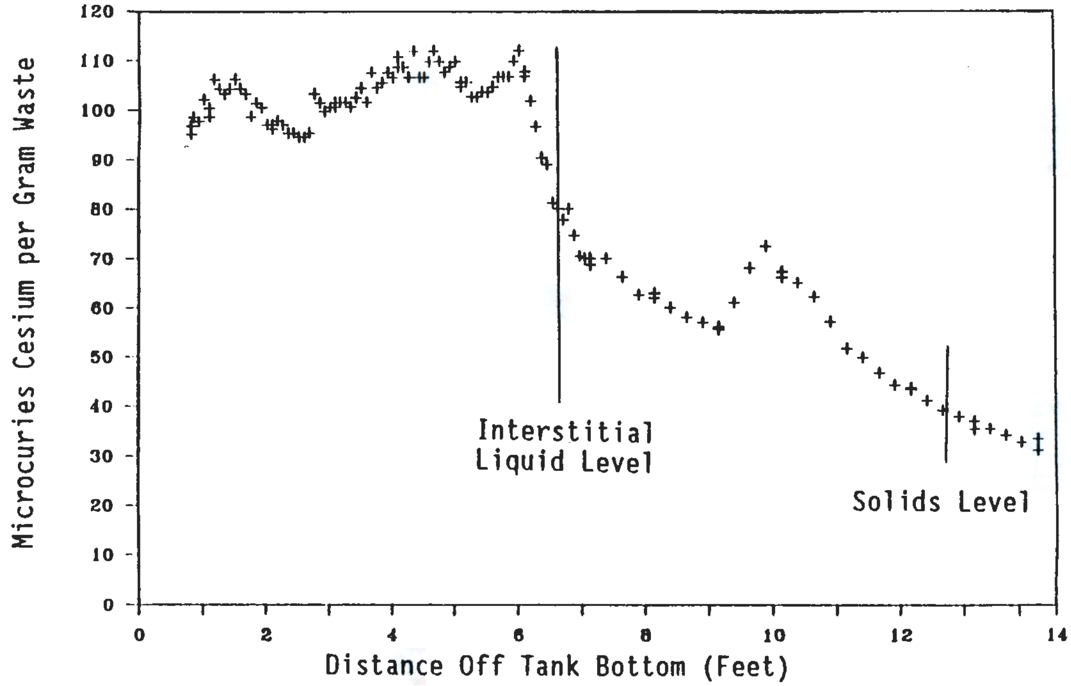
Cesium Inventories and Associated Heat Load.

Tank	Spectral scan (Ci)[1]	Associated heat load (BTU)	TRAC[2] (Ci)
241-BX-111	1.8 E+05	1370	4.4 E+05
241-BY-101	6.1 E+04	464	5.2 E+05
241-BY-103	1.8 E+05	1370	2.6 E+04
241-BY-104	2.3 E+05	1748	5.2 E+05
241-BY-105	2.0 E+05	1520	4.4 E+05
241-BY-106	4.3 E+05	3270	5.2 E+05
241-BY-107	1.9 E+05	1445	1.7 E+05
241-BY-110	2.2 E+05	1673	5.2 E+05
241-BY-111	1.6 E+05	1217	5.2 E+05
241-BY-112	1.0 E+05	760	7.8 E+04
241-TX-118	1.3 E+05	989	9.0 E+05
241-TY-103	4.6 E+04	350	1.7 E+05

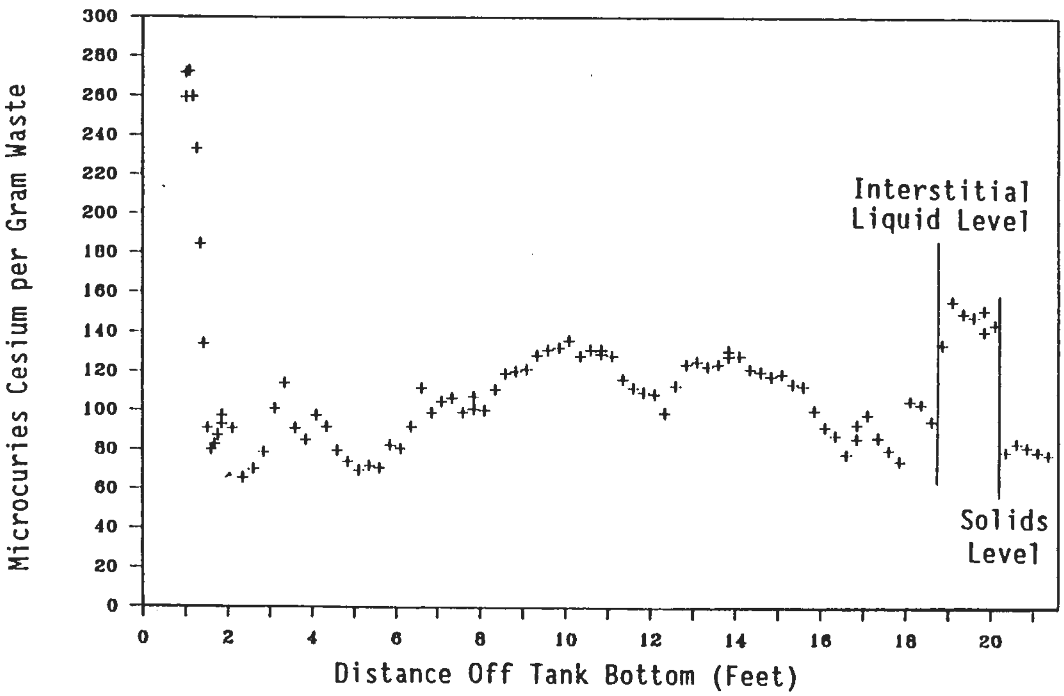
[1]Based upon original lead shielded collimators: to be revised upon issuance of report using tungsten collimators.
[2]TRAC data decayed to January 1991 (Jungfleisch, 1984).

Initial collimated spectral gamma scans have been obtained for europium and cobalt concentration measurements in two of the ferrocyanide waste tanks. The detector is in the process of being calibrated for europium and cobalt so that tank concentrations can be obtained. Initial results indicate that europium is located near the bottom of the waste tanks indicating that the transuranic elements would also be located near the bottom. Additional europium measurements are planned later for the remaining ferrocyanide tanks with LOWs. Examples of cesium and europium scans are shown in Figure 2-1.

Figure 2-1. Results of Spectral Gamma Scans for Europium 154 and Cesium 137 in Several Ferrocyanide Tanks.

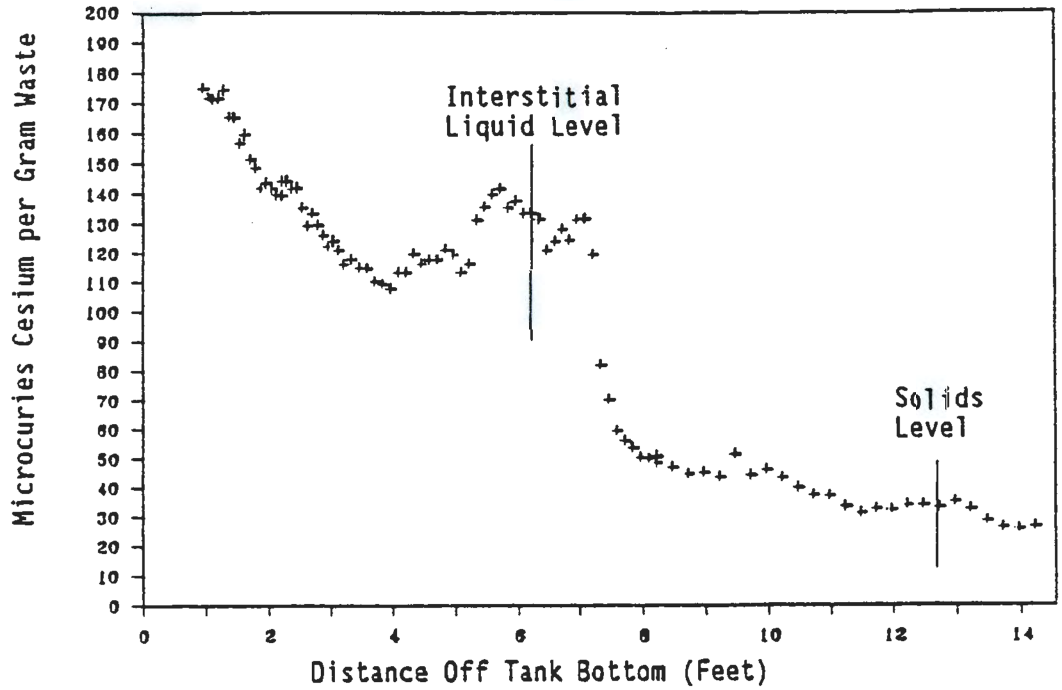


Cesium Concentration in BY-104 Waste Showing Generally Even Distribution Throughout the Liquid and Variable Distribution in the Solids Above the Liquid

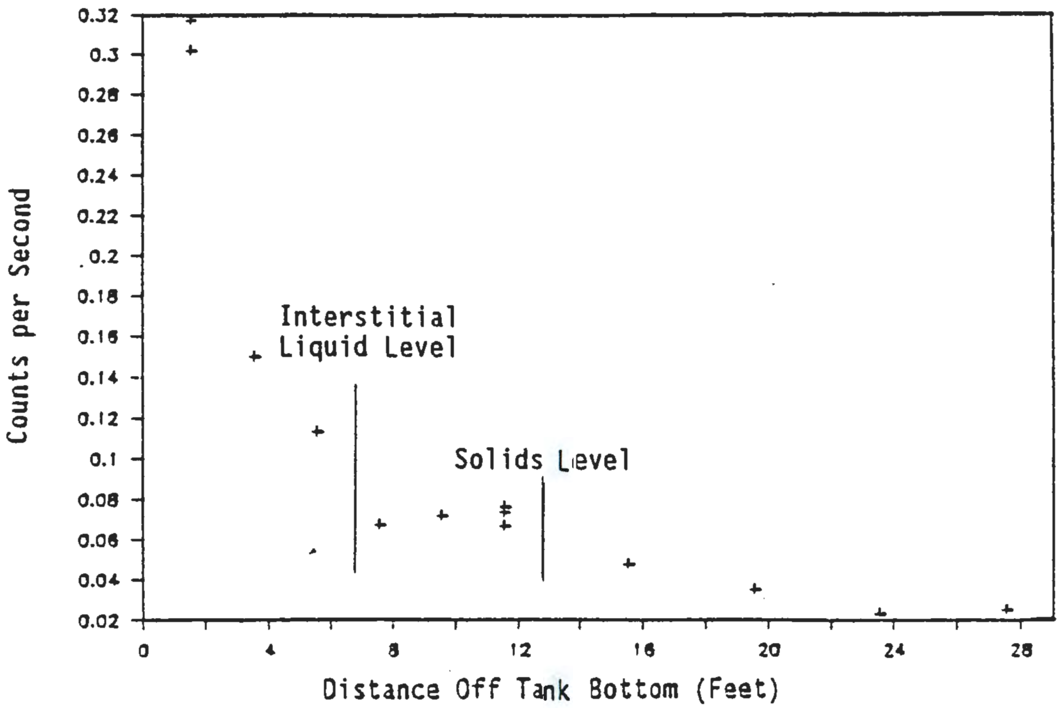


Cesium Concentration in BY-106 Waste Showing an Increased Concentration at the Bottom of the Tank

Figure 2-1. Results of Spectral Gamma Scans for Europium 154 and Cesium 137 in Several Ferrocyanide Tanks. (continued)



Cesium Concentration in BY-110 Waste Showing a Moderate Concentration Increase Near the Bottom of the Tank



Relative Europium Location in BY-104 Waste Showing an Increased Concentration at the Bottom of the Tank (Europium acts as a tracer for transuranics)

- **Planned Work For Subsequent Months.** Issue a report summarizing results of the spectral scans.

- **Problem Areas and Actions Taken.** None.

- **Milestone Status.**

  - September 30, 1991: Completed Cs-137 spectral gamma scans for ten 200 East Area tanks and for two 200 West Area tanks versus 11 planned.

  - September 30, 1991: Completed Eu-154 spectral scans for two 200 East Area tanks.

2.1.3 Upgrades to Existing Tank Temperature Monitoring Instrumentation

This task determines the operability and accuracy of presently installed thermocouples in the 24 ferrocyanide tanks at the Hanford Site. Until new multifunctional instrument trees and thermocouple trees are installed, the existing thermocouples will be used to provide temperature readings for the ferrocyanide tanks.

Field measurements have been taken on each thermocouple in the existing trees to determine the resistance and voltage across the junction and across each lead to ground. The exact condition of each thermocouple is being determined by the resistance and voltage measurements.

- **Progress During the Reporting Period.** Thermocouple field measurements have been performed on all 24 ferrocyanide tanks. A test report documenting their condition is presently being prepared and will be completed during the first month of the next reporting period. It was found that 17 tanks have no more than 1 or 2 thermocouples out of service, 5 tanks have less than 50 percent of the thermocouples functioning, and 2 tanks have no working thermocouple trees. The temperature of the latter two tanks are monitored with a single thermocouple at the bottom of the LOW. Work on repairing the thermocouple leads in the tree in tank 241-BY-108 was completed and these thermocouples are now connected to the 24-h Continuous Temperature Monitoring System (CTMS); see Section 2.2.1. Some of the non-working thermocouples in the trees may be repairable. An evaluation is in progress to determine if there should be any additional thermocouples added to the tanks as a result of the failed thermocouples found.

- **Planned Work for Subsequent Months.** The test report will be completed and reviewed during October 1991. Terminal measurements will be performed on the thermocouples in tank 241-BY-108 to better determine their condition. Worn spots in the insulation of lead wires of the thermocouples in this tree have been repaired, eliminating electrical "shorts" between the leads. Temperature profiling will be performed on the thermocouple tree in tank 241-TY-103 to determine if this technique is feasible on other trees

in ferrocyanide tanks. This task will consist of attempting to insert a new thermocouple into the tree in 241-TY-103 and lowering it to the elevations of each of the thermocouples to verify the approximate temperatures of the installed thermocouple. The thermocouple tree in 241-TY-103 has only one good thermocouple (near the top). Two of the thermocouples in this tree may be repairable. Work will continue on preparing work packages for repair and restoration of other thermocouple trees in watchlist tanks.

- **Problem Areas and Action Taken.** Preparation of the test report has taken longer than originally estimated due to the volume of measurements to review and to analyze. Overtime is being used to expedite its completion.

- **Milestone Status.**

  - September 8, 1991: Complete field work (terminal measurements) Completed ahead of schedule.

  - October 31, 1991: Complete draft test report. Work in progress.

  - December 31, 1991: Complete review and issue cleared report.

2.1.4 Hot Spot Thermal Modeling

The decay of radioactive materials in the tanks generates heat. Rapid chemical reactions within the waste containing ferrocyanide might occur if the temperatures in a tank become high enough to cause exothermic excursions. Because there is presently only one thermocouple tree per ferrocyanide tank (241-BY-105 has two trees; tanks 241-BY-111 and -112 have no trees) and the trees are not always at the same location, uneven heat generation could exist in these tanks and not be detected. This task models and analyzes the available temperature data from the ferrocyanide tanks to determine the heat load and temperatures as a function of axial and radial distance within the tank. Sensitivity and parametric analyses are included to determine the magnitude of allowable hot spots within the waste.

State-of-the-art validated computer codes are used in the modeling. They are benchmarked with existing data and employ two- and three-dimensional capabilities. Present work involves only steady-state modeling, but FY 1992 work will address transient modeling.

- **Progress During the Reporting Period.** An analysis of hot spot characteristics was completed during this period. The analysis investigated the concentrations required for thermal runaway with respect to size and shape of a postulated hot spot in ferrocyanide tank 241-BY-104. It was determined, that as the hot spot size became smaller, the concentration required became very much larger, and the temperature reached before thermal runaway became hotter. A 23 cm (9 in.) square cylinder required a concentration of over one thousand times the average with a temperature of 183 °C, (361 °F), to reach a runaway condition. A square cylinder of the height of

the waste, 2.56 m (8.4 ft), required a concentration of 5.4 times the average with a temperature of 124 °C, (255 °F), to reach a runaway condition. This requires 6.8 percent of the total tank heat load into 1.3 percent of the waste volume. A layer containing the entire heat production of the waste, radioactive decay and chemical reaction, was reduced to 7.6 cm (3 in.) in thickness without reaching a thermal runaway condition. A summary of the results obtained by thermal hydraulic analyses of tank 241-BY-104 is shown in Table 2-1.

A report documenting the results of the thermal hydraulic analyses performed for tank BY-104 was prepared. This report is currently being cleared for public release and will be available next quarter.

An analysis of the air space above the waste in the tank was conducted to determine the airflow through the tank due to leakage, and the heat loss resulting from this air flow. The analysis determined that with gaps in the penetrating piping of as little as 0.79 mm (1/32 in.) the resulting airflow was 16.5 L/s (35 CFM). The heat removed from the tank was very dependent upon the moisture evaporated. A conservative estimate of water loss of 0.4 L/h, (2.5 g/day), with the airflow of 16.5 L/s removed 535 W (1,826 BTU/h).

- **Planned Work for Subsequent Months**. The following items will be completed during the first quarter of FY 1992.

  1. Determine which ferrocyanide tank is considered to be the worst-case tank from a safety concern (no longer believed to be 241-BY-104; see Section 2.5.1 for the discussion on flowsheet results).

  2. Perform a detailed thermal analysis of this tank, including estimation of the heat load, the potential for thermal runaway due to uneven heating, and determine the estimated airflow and water loss from the tank.

  3. Model tank 241-C-112 for the effect of hot spots and for transient heat transfer to predict how soon the waste surface temperature will change with time as a function of hot spot parameters. This tank contains only 394,000 L (104,000 gal) of waste (96 cm [38 in.] deep at the side wall and 127 cm [50 in.] deep in the tank center). Heat transfer characteristics for this tank are expected to be different from 241-BY-104 which contains 1,537,000 L (406,000 gal) of waste.

  4. Issue for public release the report on thermal hydraulic analysis of single-shell tank 241-BY-104.

- **Milestone Status**.

  - August 31, 1991: A three-dimensional analysis of the dome space in tank 241-BY-104 to evaluate air temperature

Table 2-1. Summary of Thermal Hydraulic Analyses of Tank 241-BY-104.

BY-104 HOT SPOT MODEL RESULTS

Qbase BTU/hr	Chem Heat	Qhot BTU/hr	K crust	K damp	K wet	K spot	Max T °F	Heat Conct	Hot Spot Location
5500	no	500	.10		.27	.10	320	110x	9" below crust
5500	no	500	.10		.27	.27	330	110x	on bottom
5500	no	800	.10		.27	.10	350	199x	on bottom
5500	no	550	.10		.27	.10	350	120x	on bottom, dry around
8000	no	1000	.16	.21	.27	.16	350	150x	on bottom
8000	no	1000	.16	.21	.27	.16	352	150x	on bottom, dry over
8000	no	800	.16	.21	.27	.16	350	120x	on bottom, dry around
8000	yes	0	.16	.21	.27		136	1x	no hot spot, chem heat
8000	yes	260	.10	.10	.10	.10	289	39x	on bottom, completely dry

sensitivity to heat load and hot spots, and to determine air flow patterns was completed in September. A report on this work was prepared and is currently being reviewed for release as a public document.

2.2 DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7.2

"The temperature sensors referred to above [Recommendation 90-7.1] should have continuous recorded readouts and alarms that would signal at a permanently manned location any abnormally high temperatures and any failed temperature instrumentation."

2.2.1 Continuous Temperature Monitoring

This task is providing continuous monitoring of presently installed (and operable) thermocouples for the 24 ferrocyanide tanks. The new multifunctional instrument trees and thermocouple trees, as they are added to each tank, will be connected into the system. Monitoring will be continuous. All data will be collected automatically at the continuously-manned Computer Automated Surveillance System (CASS) Operator Control Station in the 2750-E Building, 200 East Area. The monitoring system will be independent of the CASS and be capable of displaying data to an operator on request. Trend data on selected points will be available for display in numeric or graphic form.

The system will have the capability to assign alarms for change in value of any temperature point. Alarms will trigger an audible annunciator and be logged immediately to hardcopy. An alarm summary display will provide a list of the most recent alarms in order of occurrence. Each alarm will be identified by point and time of occurrence. Operator acknowledgement of the alarm will silence the audible annunciator.

Signal conditioning and multiplexing will be performed locally at each tank. This eliminates the need to transmit low-level signals to the tank farm boundary and reduces cable runs. Electronic noise, extension wire corrosion, and thermal gradients are thereby reduced.

- **Progress During Reporting Period.** The CTMS was installed on tanks 104, 105, 106, 108, and 110 in BY farm and is operational. The operator interface consists of two cathode ray tube (CRT) graphics displays located in the CASS control room which is continuously manned. The system has alarms with audible annunciation for change in temperature from an established baseline. Data are logged to disk as well as hard copy. All alarms are logged to hard copy. In addition, the system has provision for colored on-line viewing of data trends as well as graphical presentation of the data showing axial and radial distribution of the temperatures relative to tank geometry.

An abandoned thermocouple tree in Tank 241-BY-105 was tested and determined to be functional during the thermocouple evaluation program. This tree was connected to the new monitoring system and data are being collected on two trees in this tank. This provides radial as well as axial temperature distributions.

The thermocouple leads at Tank 241-BY-108 were in considerable disrepair as a result of damage to the junction box several years ago. The thermocouple leads were lying on the ground and were corroded. Most of the identification markings had been lost. These thermocouples were tested during the thermocouple evaluation program and all but one was found to be operational. In addition, by using a combination of lead-to-ground and loop resistance measurements, the length of the thermocouple wire was determined and the thermocouple position (height from tank bottom) on the tree was established. A new junction box has been installed at this location and the thermocouple wires are now properly terminated in the box. This tree is also connected to the monitoring system.

The data collected to date for five BY tanks indicates the temperatures are quite stable. Typically, they vary by only about ± 1 °F over a period of several days. The monitoring system provides the capability to view any temperature value plotted versus time. Up to six selected temperatures can be placed on the same plot. See Figure 2-2 for examples of the readouts.

- **Planned Work For Subsequent Months.** The system will be expanded to include the five remaining ferrocyanide tanks in BY Farm by the end of the next quarter and design will commence for expansion to four tanks in C Farm. The system will also be connected to the four new thermocouple trees and one new multifunctional instrument tree in BY and C Farms. Further planning is required for the remainder of the ferrocyanide tanks.

- **Problem Areas and Action Taken.** Further evaluation is required to establish plans for the remainder of the ferrocyanide tanks. In addition, there may be a delay in C Farm because of fresh air respiratory requirements for entry into the tank farm.

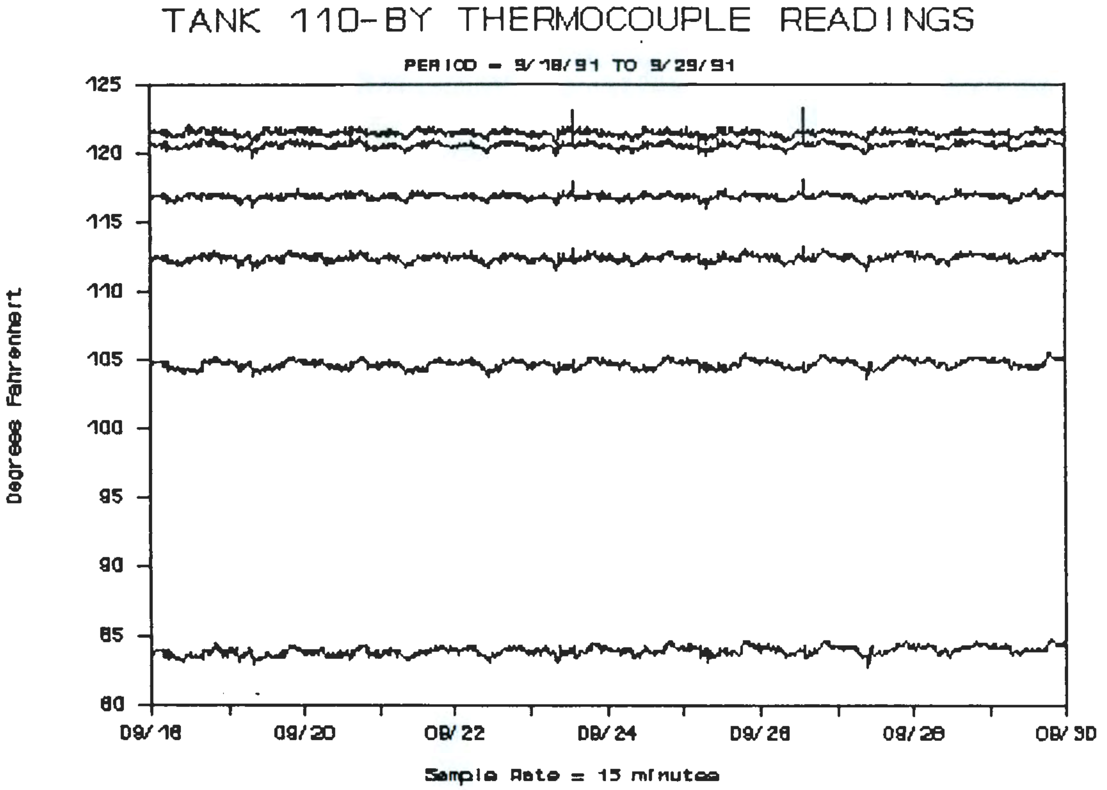
- **Milestone Status.**

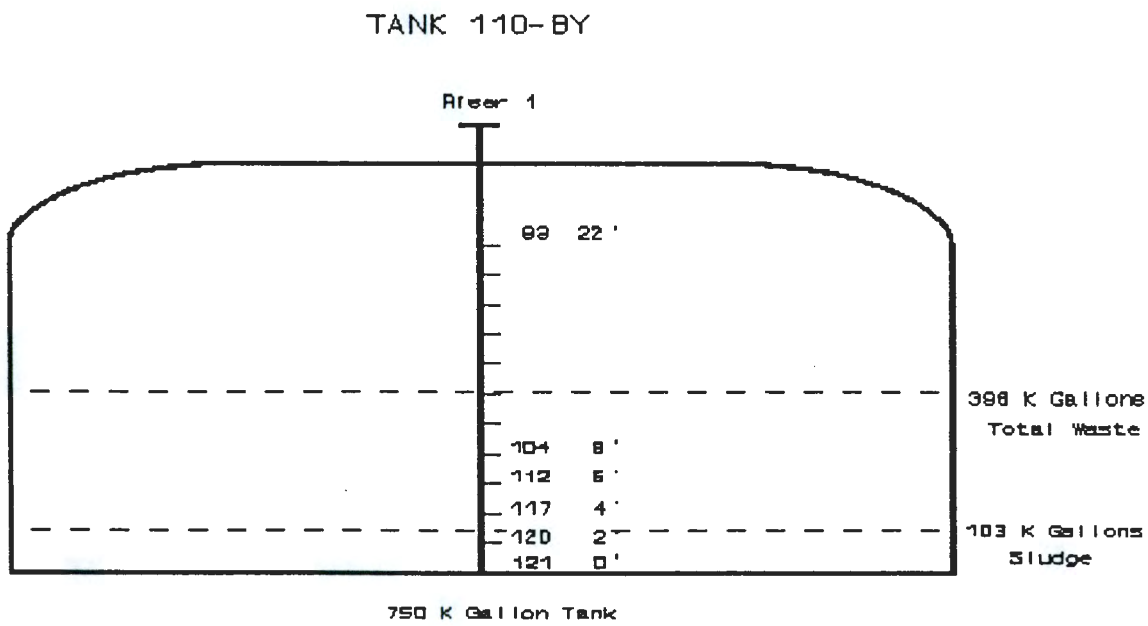
  - September 26, 1991: Completed installation of the continuous temperature monitoring system for five tanks in BY Farm.

  - December 31, 1991: Complete installation of continuous monitoring of five remaining tanks in BY Farm.

  - April 30, 1992: Complete installation of continuous monitoring of four tanks in C Farm.

Figure 2-2. Tank 241-BY-110 Thermocouple Readings.





2.3 DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7.3

"Instrumentation should also be installed to monitor the composition of cover gas in the tanks, to establish if flammable gas is present."

2.3.1 On-Line Gas Monitoring

The multifunctional instrument trees discussed in Section 2.1 are being designed to have dome space gas sampling tubes incorporated within the body of the tree. These tubes will be located within each tree and will penetrate the side of the tree at locations that allow gases to be sampled from several elevations within the tank dome space.

The dome space gas sampling tubes will exit through the top of the instrument tree and can be cleaned if vapor deposits cause plugging. Once the trees are installed and operational, the gas drawn from these tubes will be monitored for gas composition and moisture (humidity). This will be done on either a continuous basis, if warranted, or on a periodic basis depending upon trending data. It is anticipated that the frequency of gas monitoring and/or the need for continuous monitoring will be determined after a majority of the ferrocyanide tanks have been vapor sampled. A continuous gas monitoring system will be developed and possibly installed on a tank as a demonstration in FY 1992 if the quantities of gases detected during vapor sampling are a safety concern.

2.3.2 Interim Flammable Gas Monitoring

An intensive effort is now underway to conduct flammable gas monitoring and analyses in the 24 ferrocyanide tanks, starting with tank 241-BY-104. Because the safety issue associated with ferrocyanide tanks is listed as an "Unreviewed Safety Question," this activity requires Secretary of Energy approval to perform sampling activities within the tanks. Although past sampling conducted by Industrial Hygiene and Safety has indicated no flammable gas content above 6 percent of the lower flammability limit (LFL), no qualitative measurements were obtained.

All 24 ferrocyanide tanks are passively ventilated through individual high-efficiency particulate air filters. The "breathing" is dependent upon changes in barometric pressure. The pressure change causes a small volume of stagnant air to be replaced with fresh air, which helps control the concentration of chemical vapors inside the tanks.

Initially tank dome space gas samples will be taken through at least two different risers and at three elevations for the first few ferrocyanide tanks. The lowest elevation sample will be about 25 cm (1 ft) above the waste surface, with one sample in the middle and one near the top of the dome space. Gas sampling criteria have been defined and include identification of the chemicals to be monitored, detection limits, accuracy and precision of the analytical methods, and sample positions inside the tank.

- **Progress During Reporting Period**. A quality assurance plan for vapor sampling of the ferrocyanide tanks was approved and distributed. The plan identifies procedures, describes applicable quality control and quality assurance methods, and provides information used to support data that will be collected for gas sampling of the 24 ferrocyanide tanks. It was prepared for compliance to the QAMs-005/80 elements (EPA 1983) and the vapor space sampling criteria document (Klem 1991). It uses applicable features in existing laboratory quality assurance project plans. The plan covers a developmental or "proof of principal" period and is given a WHC quality assurance impact level of 3 until evaluation of the results indicate a different level is needed.

  The SA was revised to include comments from DOE and expanded to include gas sampling of all ferrocyanide tanks. Comments from DOE were resolved and incorporated into Revision 1 of the SA for gas sampling of the BY farm tanks. Revision 1 was transmitted to DOE on July 8, 1991 and has been accepted. Revision 2 of the SA incorporates other ferrocyanide tanks in addition to the BY Farm and was transmitted to DOE on August 29, 1991 for acceptance. Comments were received from DOE and are in the process of being dispositioned.

  The EA (DOE-EA/0533) and request for a "Finding of No Significant Impact" (FONSI) was transmitted to Washington State on July 9, 1991 for comment and approval. Washington State response was received on July 15, 1991, and DOE approval was given on August 2, 1991. The readiness review process was completed at WHC on August 9, 1991, and the DOE Field Office, Richland started their readiness review on August 12, 1991. Notice of completion of the WHC pre-start readiness items was transmitted to the DOE on August 15, 1991. Additional pre-start items were subsequently identified; see problem area below.

  Testing of equipment and gas sampling of non-watchlist tank 241-BX-104 was completed on August 6, 1991. Samples of cryogenic fluid were delivered to the laboratory for analysis. In-the field sample results at the six positions show no combustible gas was detected by the intrinsically safe combustible gas and oxygen meter and a trace level of hydrogen ($\leq$0.1 percent) was detected by the on-line gas chromatograph. These results were anticipated for non-watchlist tank 241-BX-104. Analysis of the cryogenic fluid by the laboratory is in progress.

- **Planned Work For Subsequent Months**. Gas sampling of the first ferrocyanide tank 241-BY-104 has been rescheduled to begin in October 1991. Tank 241-C-112 has been scheduled for gas sampling after tank 241-BY-104. Tank 241-C-109 is next, followed by tanks 241-C-111 and 241-C-108. Planning is in progress for completing gas sampling of all 24 ferrocyanide tanks.

- **Problem Areas and Action Taken**. Startup of gas sampling in tank 241-BY-104 was delayed by comments received from the Compliance Section of the DOE Readiness Review Board. Several of the comments

were prestart items. These items have been addressed and the completed WHC responses were transmitted to DOE on October 8, 1991 as the last step prior to startup of vapor space gas sampling. Gas sampling of tank 241-BY-104 is now expected to be completed by late October 1991.

There may be a delay in obtaining vapor space gas samples from C Farm because the farm has been placed on fresh air respiratory protection due to suspected organic vapors from the tanks. Although work in the tank farms is routinely done using respiratory protection, the time required to accomplish given tasks is lengthened because of the mask restrictions.

- **Milestone Status.**

  - June 30, 1991: Submit completed safety assessment to DOE for gas sampling of BY Tank Farm ferrocyanide tanks. Completed on July 8.

  - July 31, 1991: Complete vapor space sampling of ferrocyanide tank 241-BY-104. Rescheduled to October 31 due to response required to DOE comments.

  - *August 31, 1991: Complete vapor space sampling of ferrocyanide tank 241-C-112. Rescheduled to November 27 due to delay in sampling 241-BY-104.

  - *August 31, 1991: Complete vapor space sampling of tank 241-C-109. Rescheduled to December 31 because of above delays.

2.4 DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7.4

"The program of sampling the contents of these tanks should be greatly accelerated. The proposed schedule whereby analysis of two core samples from each single shell tank is to be completed by September, 1998 is seriously inadequate in light of the uncertainties as to safety of these tanks. Furthermore, additional samples are required at several radii and at a range of elevations for the tanks containing substantial amounts of ferrocyanide."

2.4.1 Ferrocyanide Tank Waste Characterization

Characterization of the ferrocyanide tanks contents is necessary to guide chemical reaction studies, to apply the study results to mitigation and/or

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*The schedule in the implementation plan assumed two ferrocyanide tanks could be sampled per month, once the first tank had been sampled. It is expected that sampling of tanks at this rate will be possible after more experience is gained with the first few tanks. Some delays may be encountered because of adverse weather conditions and possible equipment breakdowns.

remediation of these tanks, and to provide a basis for estimating the consequences of an uncontrolled ferrocyanide reaction. Knowledge of the relative position of various waste constituents is also important to determine the proximity of ferrocyanide compounds to potential reactants.

2.4.2 Ferrocyanide Tank Waste Sampling and Characterization

The important reaction materials present in the ferrocyanide tanks are fuel (ferrocyanides and reduced carbon species, such as organic complexants), oxidants (nitrates and nitrites), and inerts or diluents (phosphates, aluminates, sulfates, carbonates, oxides, and hydroxides). The location of fission products, such as ^{137}Cs and ^{90}Sr, is important because they act as heat sources that can raise and maintain the temperature of the tank contents and because they are source terms in a radiological release. The water content of the waste is very important because the high heat capacity and the heat of vaporization of water makes it an effective inerting material and prevents sustained combustion. Also, wet ferrocyanide material should not react nor propagate; it would have to be dried out first. Other materials (e.g., nickel, copper, lead and the rare earths) may be important as potential catalysts.

- **Progress During the Reporting Period.** Existing records and a spreadsheet model have been used to estimate the formation and distribution of ferrocyanide solids in the tanks. A document providing best estimates of the ferrocyanide distribution at the end of the scavenging program was prepared. Review comments were resolved and incorporated into the document. The revised document is being routed for approval and release as a cleared document.

  Activities for obtaining surface samples from tank 241-BY-104 are near completion. The design of the auger sampler used successfully to obtain samples from tank 241-SY-101 was modified slightly for application in the ferrocyanide tanks. Fabrication of two auger surface samplers has been completed and the samplers have been delivered to Operations. The sampling procedure was prepared, approved, and released. Testing of the sampler and training of the operating crew was completed. The analytical plan for analysis of the samples was issued in August. The DOE comments on the SA were incorporated and Revision 2 was submitted to DOE on October 10, 1991. The EA was revised to incorporate DOE comments and resubmitted to DOE on September 16, 1991. The draft FONSI document was prepared, approved by WHC, and submitted to DOE on September 30, 1991.

  The push-mode technique is presently the only viable Hanford Site method for waste tank core sampling until it is demonstrated that rotary core sampling will not produce excessive temperatures in the waste. Three ferrocyanide tanks suitable for push-mode core sampling have been placed on the listing of the next 10 single-shell tanks to be core sampled. The tanks are included in the current revision of the Single-Shell Tank Waste Characterization Plan (WHC 1991a) to be released in October 1991. This plan provides the

sample handling and analysis protocol for the core samples. The SA for push-mode core sampling of ferrocyanide tanks was submitted to DOE on September 27, 1991. The EA was transmitted to DOE on September 9, 1991, and formal comments have been received and are being incorporated. The draft FONSI has been prepared and approved by WHC and is ready for transmittal to DOE.

Development and design work required to demonstrate that rotary core sampling of a "harder" surface can be done without producing unacceptably high bit temperatures began in July. Tests were completed in August to determine the optimum bit design for push-mode sampling with the new disposable sludge samplers. Testing of the disposable sludge sampler with various bits designed for rotary-mode sampling were unsuccessful. New samplers for rotary-mode testing were developed.

- **Planned Work For Subsequent Months**. The document assessing the inventories of the ferrocyanide tanks at the end of the scavenging program (program ended in 1957) will be issued.

  Auger surface sampling of tank 241-BY-104 is now scheduled for completion in the first quarter of FY 1992 (vapor space sampling of the tank must be completed first). Future surface sampling will depend upon analytical results of the first samples. It is not necessary to obtain surface samples of the waste in all ferrocyanide tanks because the same information can be obtained from core samples.

  Push-mode core sampling of the first ferrocyanide tank (241-C-112) is scheduled for the first quarter of FY 1992. The second ferrocyanide tank (241-C-109) will be sampled in the second quarter of FY 1992. Core sampling of ferrocyanide tanks also meets the *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) (Ecology et al. 1990) commitment to sample single-shell tanks.

  Testing of new rotary-mode core sampler designs will continue. Testing to develop a sampling envelope which enables the safe sampling of watchlist tanks will begin. Modifications for rotary-mode core sampling are scheduled to be completed and approved for field use by the first quarter of FY 1993. Core sampling of ferrocyanide tanks with saltcake will be deferred until the rotary-mode core sample truck is available for field use.

- **Problem Areas and Actions Taken**. It is expected that the majority of the ferrocyanide tanks, particularly those with a salt cake surface, will require rotary drilling to obtain core samples. The work required to demonstrate that rotary drilling of a "harder" solids surface will not produce unacceptable high temperatures was described above. Sampling of the C Farm tanks may take longer than other ferrocyanide tanks because all work in the farm presently requires the use of fresh air for respiratory protection. Planning for sampling of the tanks vapor space is well underway.

- **Milestone Status.**

  - September 30, 1991: Complete surface sampling of tank 241-BY-104. This milestone has been delayed until December 1991 because of the added time required to complete safety and readiness review documentation.

  - October 31, 1991: Obtain first push-mode core sample from a ferrocyanide tank (241-C-112). This sampling will be delayed until December 1991 for the same reasons.

2.4.3 Synthetic Flowsheet Sludge Characterization

A program activity was started this quarter to prepare synthetic ferrocyanide materials representative of the waste believed to be in the 24 ferrocyanide tanks. To prepare this material, the flowsheets used for the actual waste scavenging campaigns in the mid 1950s are being duplicated in the WHC Chemical Engineering Laboratory and at PNL, but without radioactive species. Three flowsheets were used for the scavenging runs. To the maximum extent possible these flowsheets are being utilized to produce synthetic ferrocyanide waste precipitates; minor constituents are not being duplicated.

- **Progress During the Reporting Period.** Synthetic sodium nickel ferrocyanide sludge was produced this quarter by the Hanford in-plant* (WHC-1) flowsheet on a pilot-plant scale for characterization and reactivity testing. After precipitation, this material was allowed to settle. Samples of the precipitated solids were then centrifuged to 30 equivalent gravity years and characterized. The results showed the centrifuged bottoms consisted of 66 weight percent (wt%) water and about 2 wt% ferrocyanide. Initial viscosity of the centrifuged solids was measured at greater than 10,000 cp. After exceeding the yield stress, the measured apparent viscosity was 200 - 300 cp. Additional chemical and physical analysis of this material is proceeding.

  Synthetic sodium nickel ferrocyanide sludge will also be produced on a pilot-plant scale using the Hanford in-farm** (Batch 2)

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*The in-plant flowsheet represents ferrocyanide-bearing material which resulted from scavenging the uranium recovery waste from the Hanford Site U-Plant. This flowsheet contained considerable high-level waste solids material in addition to the ferrocyanide precipitates. This material was transferred to 10 tanks in the BY Farm.

**Uranium recovery (UR) of high-level tank waste started before the ferrocyanide scavenging process was developed. Later, the supernate from the UR waste was transferred from single-shell tanks to a stainless steel vault where the pH was adjusted and the ferrocyanide process was applied. This in-farm flowsheet contained a smaller percentage of solids and a higher concentration of ferrocyanide than the in-plant flowsheet. The in-farm ferrocyanide-bearing wastes were transferred to the C Farm.

flowsheet. This flowsheet is expected to produce a sludge with the highest ferrocyanide concentration used during the 1950s for scavenging cesium from the high-level waste at Hanford. This material will be used for characterization and reactivity testing at WHC, Pacific Northwest Laboratory (PNL), LANL, and Fauske and Associates.

- **Planned Work For Subsequent Months.** Production of the synthetic in-farm flowsheet sludge will start in October. After its production and centrifugation, it will be characterized by chemical content analysis and physical properties determination (see Section 2.5.1). The in-farm flowsheet material will be used for chemical and physical properties characterization and reactivity studies. A third flowsheet was used for ferrocyanide scavenged waste from T-Farm and U-Farm (Batch 3) tanks. Synthetic material will be prepared in December using this flowsheet. The material will be centrifuged and characterized in the same manner as for the in-plant flowsheet (Section 2.5.1). The concentration of ferrocyanide in this material is expected to be lower than that using the in-farm flowsheet.

- **Problem Areas and Actions Taken.** None.

- **Milestone Status.**

  - July 31, 1991: Completed first meeting of tank waste science panel on ferrocyanide issues.

  - September 30, 1991: Completed status report of ferrocyanide characterization studies.

  - September 30, 1991: Completed status report of ferrocyanide characterization studies.

  - September 30, 1991: Completed status report of chemical nature of iron and cyanide in wastes.

  - September 30, 1991: Completed screening and scoping studies initial set for catalysts, initiators, and diluents.

  - November 30, 1991: Annual report of FY 1991 - On Schedule

2.5 DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7.5

"The schedule for the program on study of the chemical properties and explosive behavior of the waste in these tanks is indefinite and does not reflect the urgent need for a comprehensive and definitive assessment of the probability of a violent chemical reaction. The study should be extended to other metallic compounds of ferrocyanide that are known or believed to be present in the tanks, so that conclusions can be generalized as to the range of temperature and other properties needed for a rapid chemical reaction with sodium nitrate."

2.5.1 Chemical Reaction Studies

Work during the quarter on chemical reaction studies involved the following: (1) chemical nature of iron and cyanide in wastes, (2) catalyst/initiator and diluent studies, (3) sensitivity testing preparations at LANL, and (4) reactivity studies.

The most significant finding was the low concentration of ferrocyanide contained in wastes produced by simulating the in-plant* flowsheet. The concentration of ferrocyanide is so low that this material would not explode in time-to-explosion (TTX) testing. Differential scanning calorimetry (DSC) also indicated only a very weak exotherm. The behavior of this material is very different than that prepared by simulating the in-farm flowsheet. The ferrocyanide prepared by simulating the in-farm flowsheet explodes at temperatures of 300 °C and shows strong exotherms by DSC at temperatures starting at 240 °C.

- **Progress During the Reporting Period.**

  Chemical Nature of Iron and Cyanide in Wastes--Activities continued this quarter on the preparation and characterization of various ferro- and ferricyanide compounds. Different ferrocyanides were prepared and characterized at PNL simulating the in-farm flow sheet. A ferrocyanide prepared by WHC, WHC-1, simulating the in-plant flowsheet was characterized by PNL. WHC also provided PNL a sample of the commercially prepared ferrocyanide, WHC-2, for characterization. Chemical analyses generally consisted of x-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), carbon, hydrogen, and nitrogen (CHN) elemental determinations, inductively coupled plasma (ICP), total organic carbon (TOC), total carbon (TC), and IR. The thermal behavior of the various ferrocyanide containing materials was characterized by TTX tests, DSC, thermogravimetric analysis (TGA), and pyrolysis-mass spectrometry.

  The chemical analysis data has been used to estimate the composition of the PNL, WHC, and commercially prepared ferrocyanides. Qualitative elemental composition were determined by the SEM and EDS. Quantitative data from CHN was used predominately to estimate the molecular compositions. The actual molecular species present were also indicated by XRD and IR. Nominal compositions of the samples consistent with the analytical data are given below.

  The WHC-1 material (in-plant flowsheet) was shown by CHN, IR, and XRD to contains mostly $NaNO_3$ and very little ferrocyanide. The combined TC and TOC analyses indicate WHC-1 has a sodium nickel ferrocyanide content of 1.7 ± 0.88 wt% at the 95 percent confidence level (7 measurements). The PNL in-farm flowsheet materials contained substantially more ferrocyanide and also contained nitrite. Both flowsheet-prepared materials contained sulfate as

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*See Section 2.4.3.

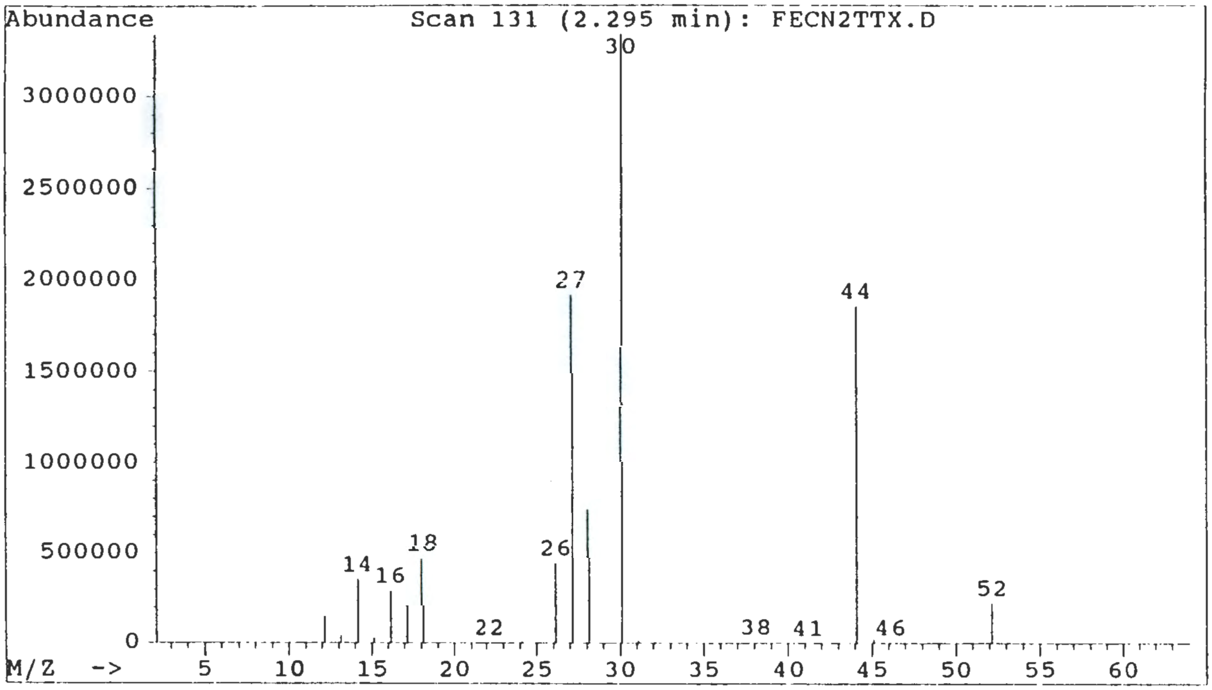
indicated by absorptions in the infrared at 1110, 1150, and 610 cm^{-1}. SEM/EDS indicated that WHC-1 also contained phosphate. Nitrate was found in FECN-28b, which was washed extensively with water.

Sample number/identification	Composition
FECN-20: (K-Na)$NiFe(CN)_6$	$Na_2NiFe(CN)_6 \cdot 5H_2O \cdot 1.7NaNO_3$
FECN-21: flowsheet $Na_2NiFe(CN)_6$	$0.25Na_2NiFe(CN)_6 \cdot 1.4H_2O \cdot NaNO_3 \cdot NaNO_2$ (+small amount of $NiSO_4$)
FECN-22: $K_2NiFe(CN)_6$	$K_2NiFe(CN)_6 \cdot 2H_2O \cdot 0.5KNO_3$
FECN-23: $NaNiFe(CN)_6$	$NaNiFe(CN)_6 \cdot 7H_2O \cdot 1.4NaNO_3$
FECN-28b: $Na_2NiFe(CN)_6$	$Na_2NiFe(CN)_6 \cdot 2.4H_2O \cdot 0.5NaNO_3$
WHC-1: in-plant flowsheet	$0.002Na_2NiFe(CN)_6 \cdot 0.3H_2O \cdot NaNO_3 \cdot 0.2NiSO_4$
WHC-2: commercial	$Na_2NiFe(CN)_6 \cdot Na_2SO_4 \cdot 4.5\ H_2O$

The mole ratios of ferrocyanide to nitrate were also estimated using IR. A series of sodium nitrate and dried potassium ferrocyanide samples were prepared in molar ratios ranging from 0.1 to 2.0 and used as standard materials for the IR analysis. The ratio of the nitrate and cyanide areas for the unknowns was compared to the area ratios obtained using the standards in order to determine molar ratios. Samples containing nitrite could not be determined reliably because the nitrite IR absorption band overlaps that of nitrate. Good agreement is generally obtained using the standards and those determined by CHN in combination with IR, SEM, and XRD analyses. Infrared indicates that FeCN-28b may contain somewhat less than the 0.5 equivalents of nitrate suggested by the best fit to CHN data. (FECN-28b was washed repeatedly to remove excess nitrate for the Catalyst and Initiator Studies.)

Sample ID	NO3/FECN by CHN	NO3/FeCN by IR
Batch 1	1.5	1.3
Batch 2	1	0.9
Batch 3	2	1.6
Ni-4	0.5	0.3
FeCN-14	0.5	0.5
FeCN-20	1.7	1.6
FeCN-22	0.5	0.7
FeCN-28b	0.5	0.2

Figure 2-3. Mass Spectrum and Relative Abundance of Ions from the Decomposition of Pacific Northwest Laboratory Flowsheet Ferrocyanide at 430 °C.



Scan 131 (2.295 min): FECN2TTX.D
Na2NiFe(CN)6 B2S1, 3.533 mg in pyroprobe

m/z	abund.	m/z	abund.	m/z	abund.
12.15	151936	26.15	446528	44.10	1860608
13.15	41016	27.15	1927168	45.10	23120
14.15	357632	28.15	751104	46.10	7805
15.15	29528	30.10	3339264	52.15	226880
16.15	295872	31.10	14715	53.15	8497
17.15	225664	32.10	8829	54.15	989
18.15	477824	38.10	9637		
19.15	2289	39.10	1500		
20.05	1531	40.10	1823		
22.15	9099	41.20	3109		
24.15	13522	42.30	1839		

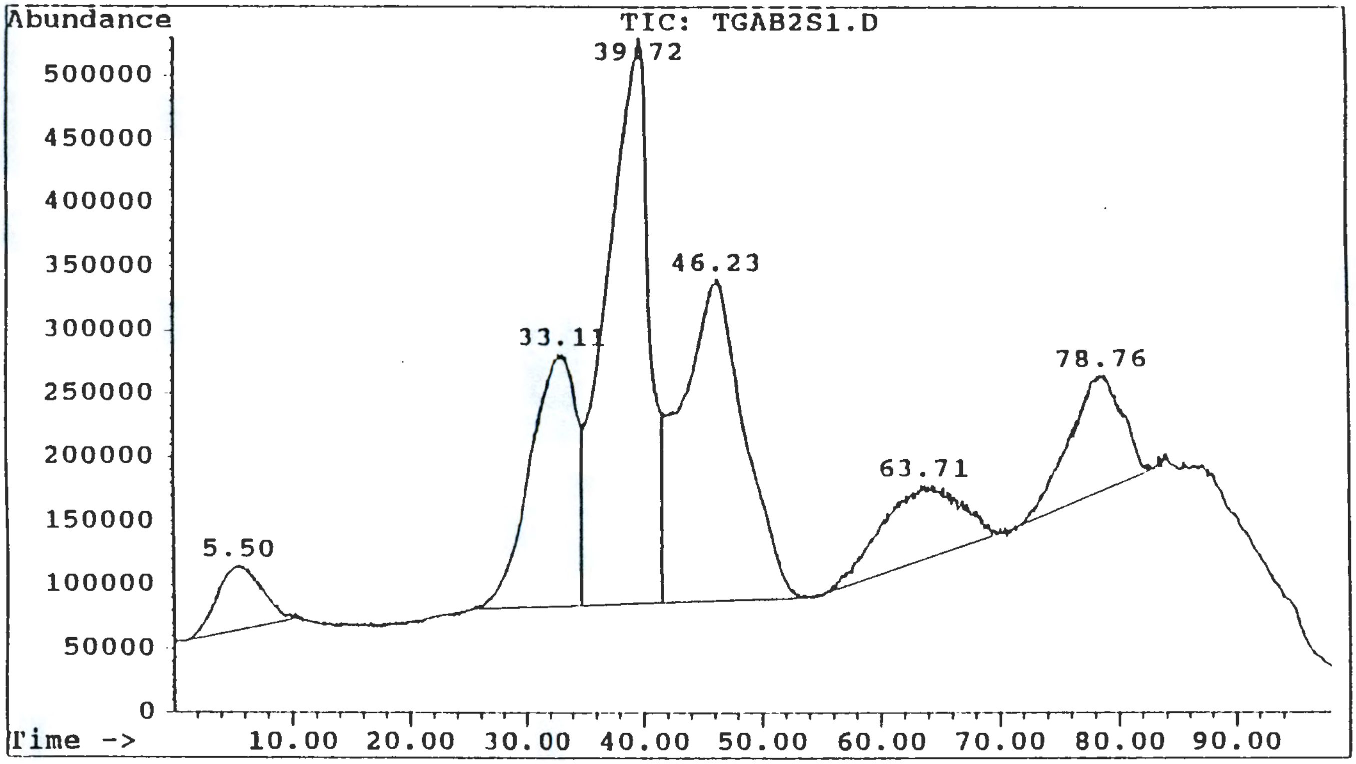
Decomposition products from the thermal decomposition of a variety of the flowsheet-prepared disodium nickel ferrocyanide have been examined using a pyroprobe-mass spectrometer system. A mass spectrum and the relative abundance of ions is shown in Figure 2-3 for the thermal decomposition at 430 °C of PNL flowsheet ferrocyanide, Batch 2 Solid 1. A similar experiment was run on WHC-1. Mass 27 (from HCN), and mass 26 (the fragment CN) are present in both materials, but peak 52 [$(CN)_2$)] was absent in WHC-1. The WHC prepared material released more H_2O (mass 18) relative to other gases upon heating than did the PNL material. The difference in behavior is probably due to the very low concentration of ferrocyanide. Weight loss over the same temperature range is typically an order of magnitude less for WHC-1 also.

Fabrication and interface to the mass spectrometer of the temperature controller/sample thermolysis assembly has been completed. This apparatus simulates the temperature profiles used during the thermal analyses experiments (DSC and TGA), whereas the pyroprobe simulates a single burst of heat more like the TTX tests. The mass spectrometer is used to continuously monitor the product gases during the temperature ramp. The total gaseous products (referred to as total ion count or summation of all ions detected) from the temperature programmed decomposition of PNL's flowsheet ferrocyanide (Batch 2 Solid 1) is shown as function of time in Figure 2-4. The temperature program is from 50 to 500 °C with a heating rate of 5 °C/min. Time zero equates to 50 °C, time 10 equates to 100 °C, and time 90 is equal to 500 °C. The extracted ion traces of the major components: water (mass 18), nitrogen and carbon monoxide (mass 28), nitric oxide (mass 30) and nitrous oxide and carbon dioxide (mass 44) are shown in Figure 2-5. By examining the extracted ion traces for mass 12 (carbon), mass 14 (nitrogen), and mass 16 (oxygen) shown in Figure 2-6, the mass 44 peak at time 34 (temperature 220 °C) contains no carbon dioxide and at time 40 (temperature 250 °C) is predominately carbon dioxide. The extracted ion traces for mass 26 (CN fragment), mass 27 (HCN), and mass 52 [$(CN)_2$] are shown in Figure 2-7. The HCN concentration is the greatest at time 38 (temperature 240 °C) and disappears as the carbon dioxide peak centered at time 40 grows.

PNL flowsheet material, Batch 2 Solid 1, was analyzed a second time in order to compare it to previously obtained results. Figure 2-8 shows the results from the first experiment (top), and results of the most recent analysis (bottom). The sample size was varied from 4.45 mg (top trace) to 7.66 mg (bottom trace) and the corresponding total weight loss after heat up 500 °C was 41.56 percent and 41.38 percent, respectively. Besides the differences in abundance caused by different sample sizes, the two traces are identical except for the water loss at 78 °C. This is because the second sample was purged with dry helium for approximately 3 h prior to thermolysis which removed excess moisture.

Three additional flowsheet samples, PNL flowsheet FECN-21 and FECN-28b plus nitrate/nitrite and WHC-1 flowsheet, were also analyzed. Figure 2-9 shows the traces from top to bottom, Batch 2

Figure 2-4. Total Ion Count for the Thermal Decomposition* of Pacific Northwest Laboratory Flowsheet Ferrocyanide (Batch 2 Solid 1).



*Temperature programmed from 50 °C to 500 °C at a rate of 5 °C/min. Temperature at any time is calculated by multiplying the time by 5 and adding 50.

Figure 2-5. Extracted Ion Traces for the Major Mass Ions (18, 28, 30 and 44) for the Thermal Decomposition of PNL Flowsheet Ferrocyanide.

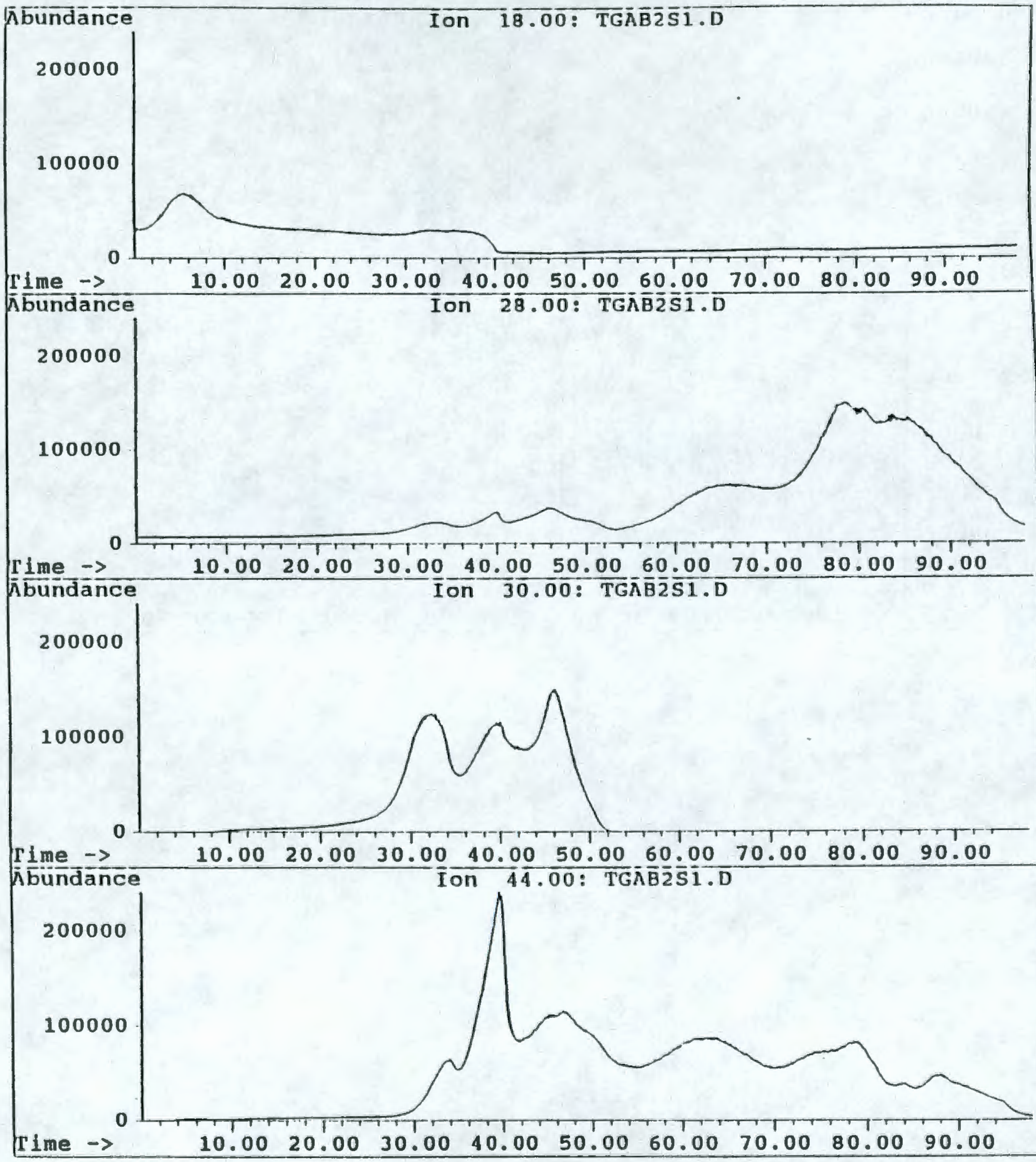


Figure 2-6. Extracted Ion Traces for Carbon (Mass 12), Nitrogen (Mass 14) an Oxygen (Mass 16) from the Thermal Decomposition of Pacific Northwest Laboratory Flowsheet Ferrocyanide.

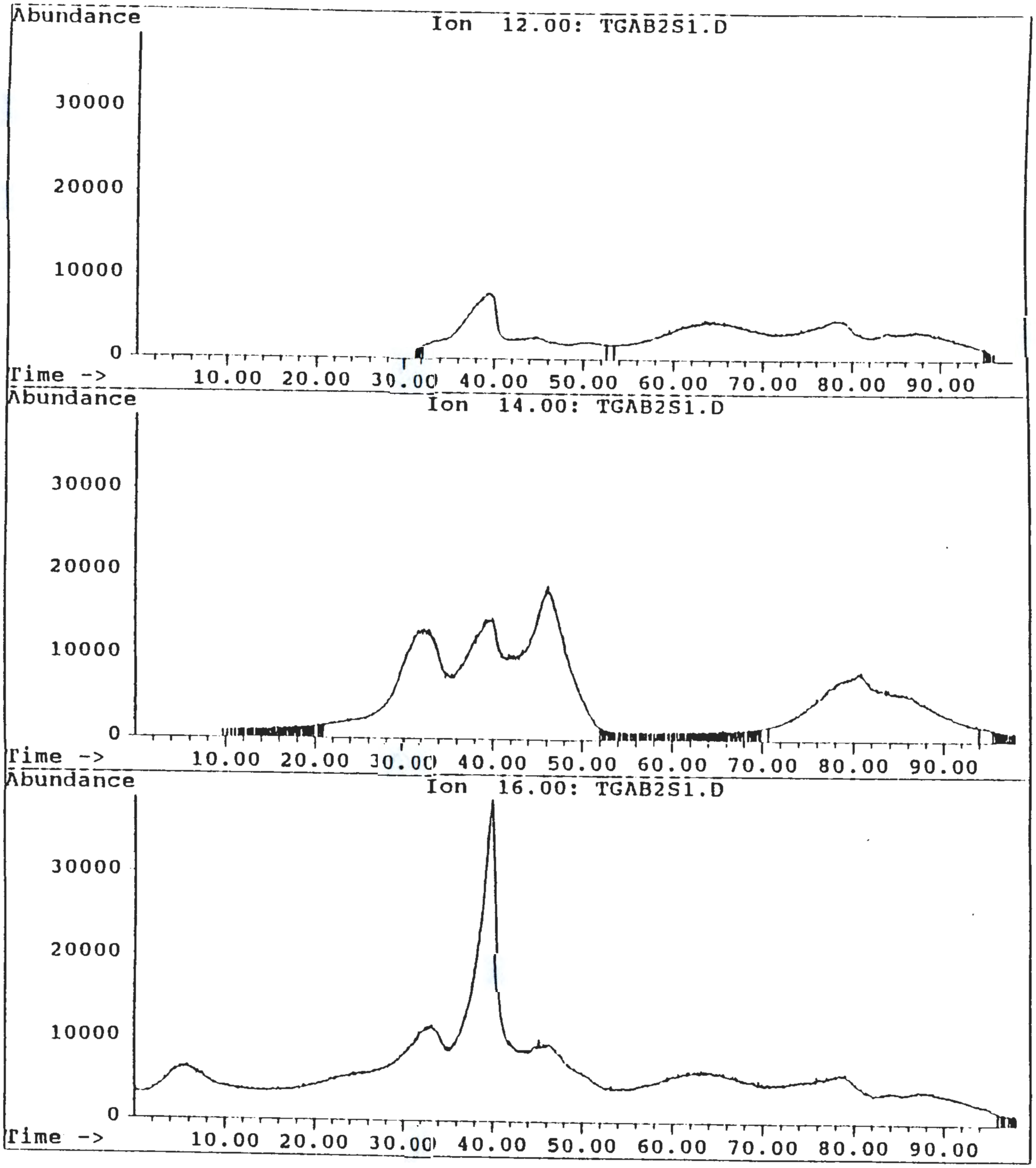


Figure 2-7. Extracted Ion Traces for Mass Ions 26, 27 and 52 from the Thermal Decomposition of Pacific Northwest Laboratory Flowsheet Ferrocyanide.

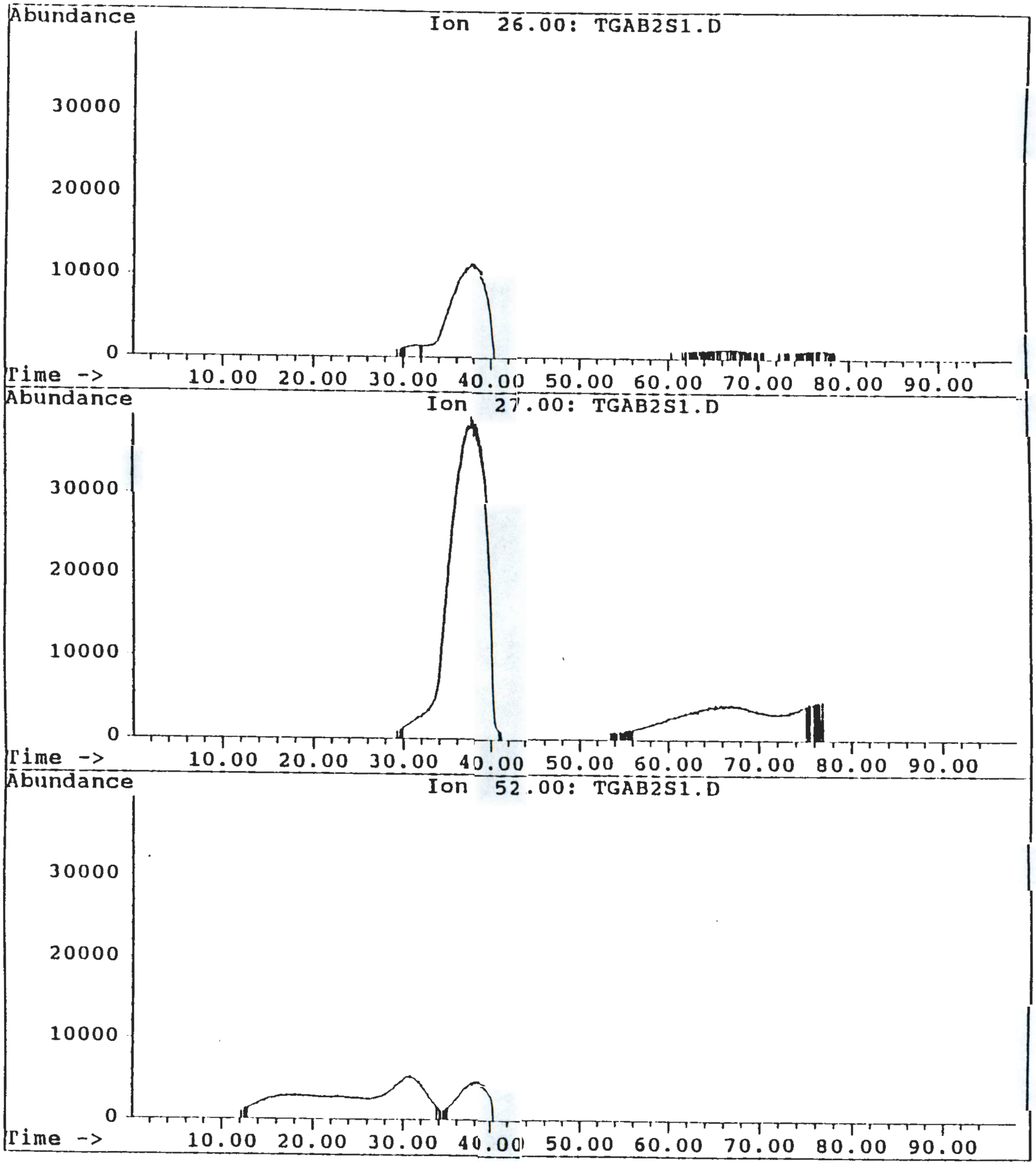


Figure 2-8. Duplicate Experiments of Different Sample Size Showing Total Ion Traces for the Thermal Decomposition of Pacific Northwest Laboratory Flowsheet Ferrocyanide (Batch 2 Solid 1).

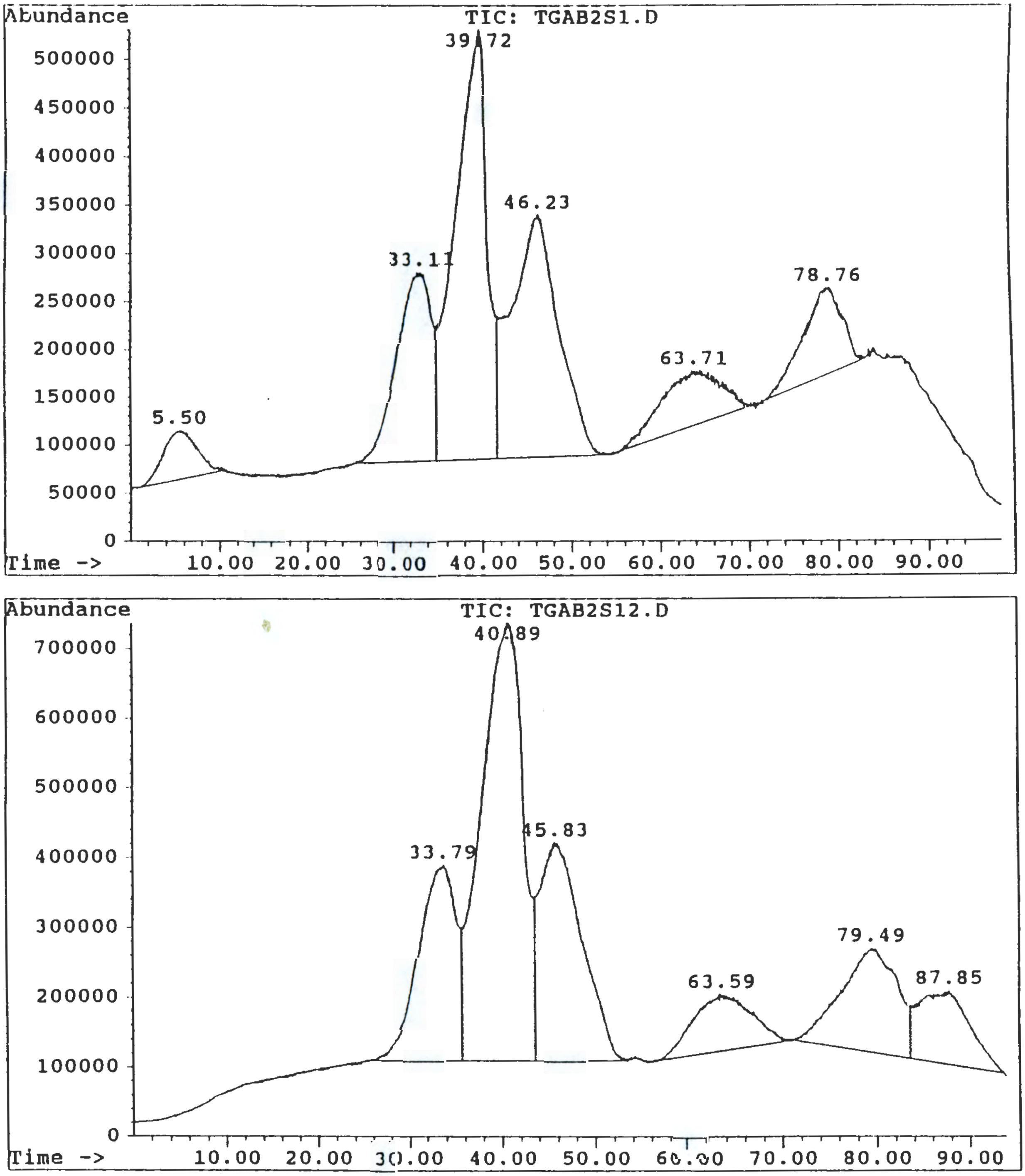
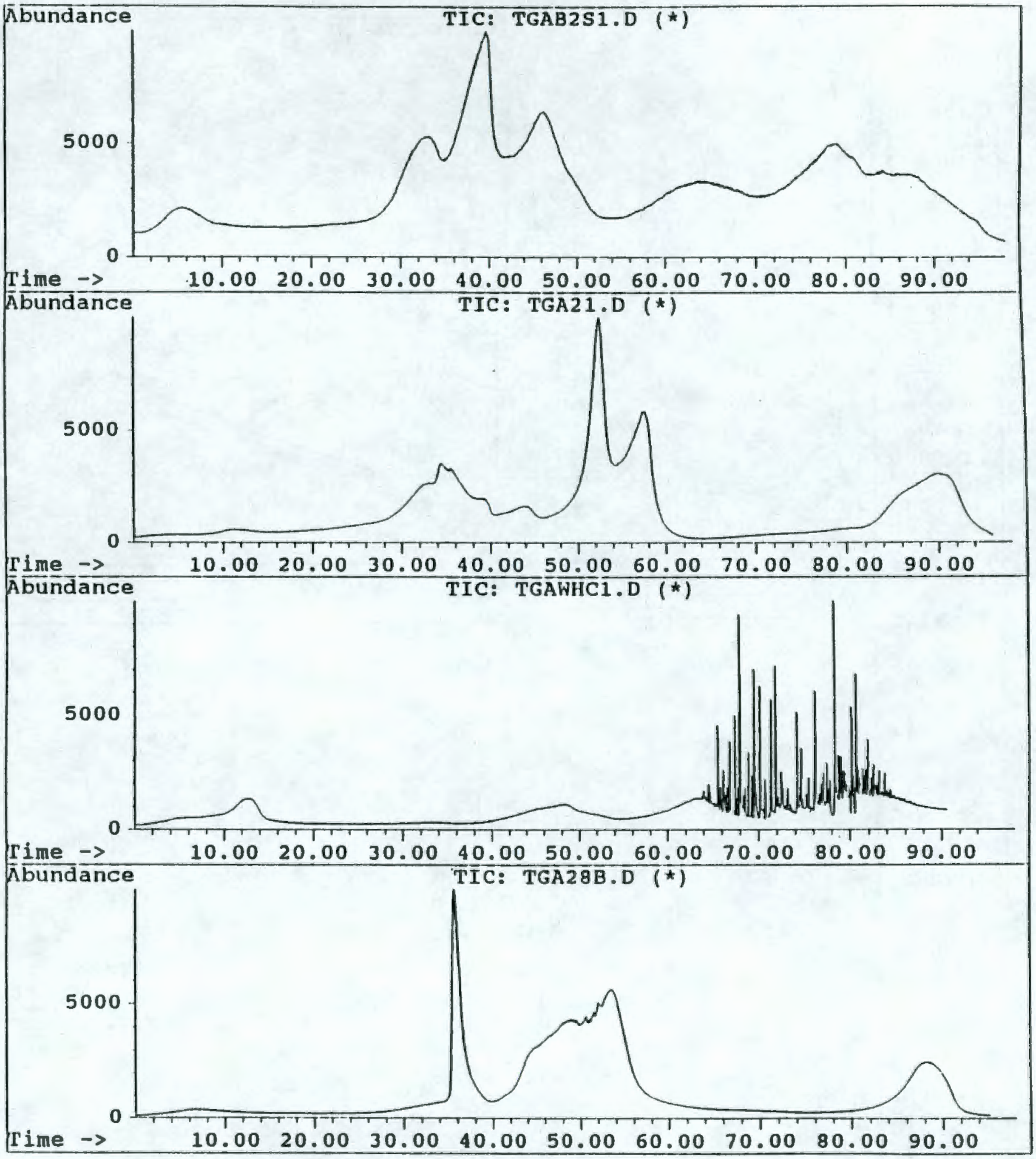


Figure 2-9. Extracted Ion Traces for Mass Ion 28 for the Thermal Decomposition of Flowsheet Materials: Batch 2 Solid 1, FECN-21, WHC-1 and FECN-28b.



Solid 1, FECN-21, WHC-1, and FECN-28b plus nitrate/nitrite. The cause of the sharp spikes (noise) on WHC 1 are unknown and may have resulted from an air leak in the system. The differences in thermal behavior/gas production of the various flowsheet materials are apparent. The weight loss after heat-up to 500 °C for the various samples was Batch 2 Solid 1 = 41.56 percent, FECN-21 = 39.72 percent, WHC-1 sample holder broke, and FECN-28b = 33.22 percent.

The nitrate/nitrite mix was also run by itself for comparison and lost only 2.41 percent weight over the same temperature range.

Results showing the individual mass ions 18, 28, 30, an 44 for FECN-21 and FECN-28b are presented in Figures 2-10 and 2-11, respectively. One of the significant differences between these results and the results from Batch 2 Solid 1 is the production of nitrogen (mass 28) at a lower temperature, 300 °C. The maximum exothermic reaction results in the production of nitrogen gas, not the nitrogen oxides as seen with Batch 2 Solid 1 at these temperatures.

In preparation for the ferrocyanide aging experiments, a Mossbauer spectrometer was repaired. The Mossbauer is capable of determining the oxidation state and form of iron present in solid samples nondestructively. Standard samples have been run to determine operational parameters such as concentration and temperature. A dewar for low-temperature data collection is being evaluated. A sample alignment problem is currently being corrected. A representative spectrum of a ferri/ferrocyanide mix taken at room temperature is shown in Figure 2-12. The dark line is the best calculated peak fit.

Catalyst/Initiator and Diluent Studies--The catalyst, initiator, and diluent studies was delayed during the early part of the quarter due to experimental difficulties. During these initial investigations the samples were forced out of the TTX reaction vessel during heat up. These problems were overcome by using different wash solutions to remove traces of the parent solution and different drying methods to prepare the materials for TTX evaluation. This material no longer pushes the test mixture out of the reaction vessel. The TTX tests of the dried material give a minimum explosion temperature near 290 °C.

The studies have begun with the investigation of the effects of sodium EDTA, nickel hydroxide, chromium (III) hydroxide, and ferric hydroxide on the TTX for the reaction mixture of washed sodium nickel ferrocyanide (FECN-28b) and equimolar sodium nitrate/nitrite. Based on preliminary analysis of the data, there appear to be minimal effects of any of the catalysts or initiators at the level tested. The minimum explosion temperature observed in these tests is about 290 °C. Although there appears to be little effect on the TTX, the brisance of the explosion varied between test mixtures. Only a qualitative measure of the sound level and tone was used. The data is currently being analyzed using statistical methods to determine if the additives have a significant effect on TTX.

Figure 2-10. Selected Ion Traces from the Thermal Decomposition of FECN-21 Flowsheet Material.

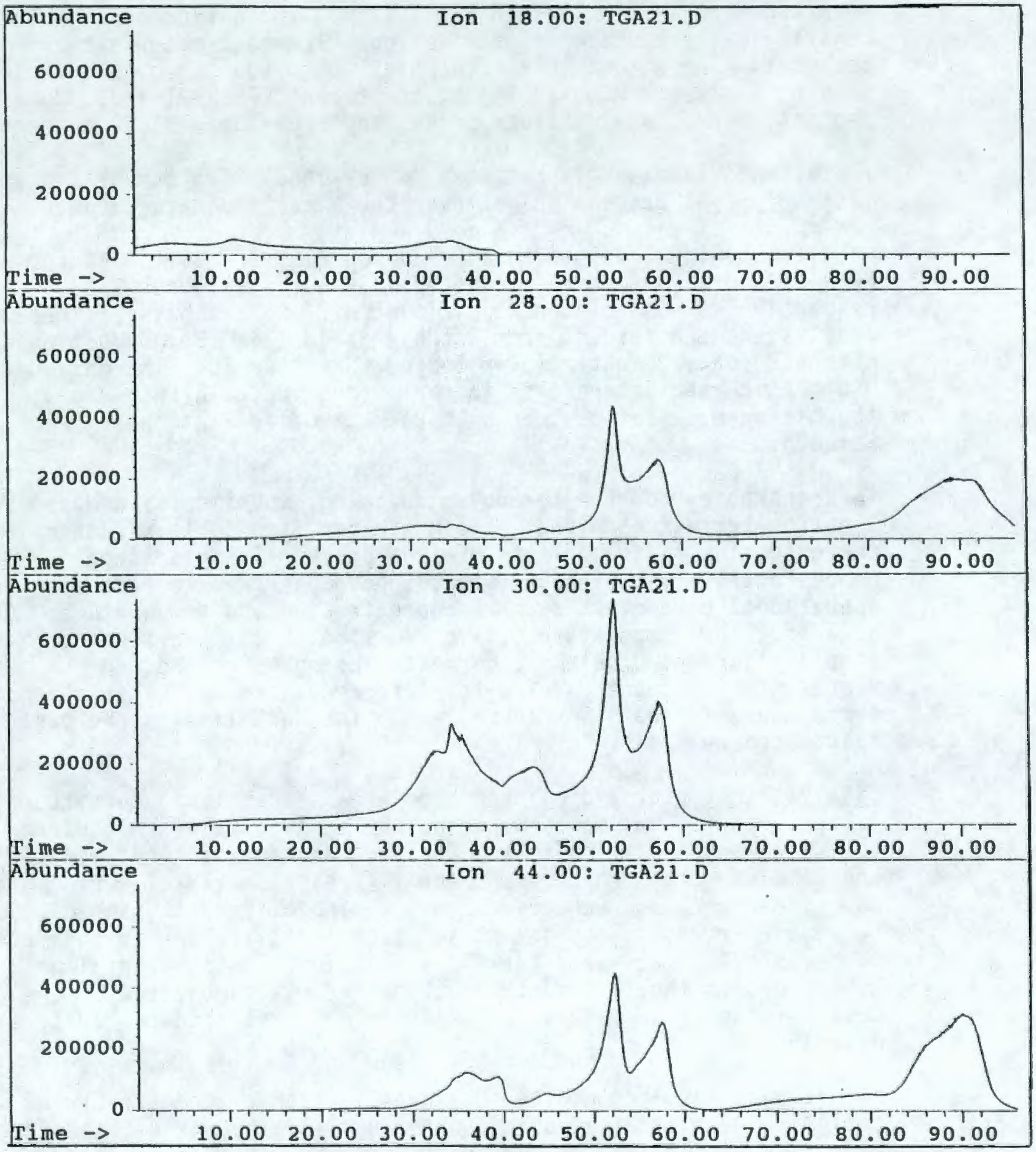


Figure 2-11. Selected Ion Traces from the Thermal Decomposition of FECN-28b Flowsheet Material.

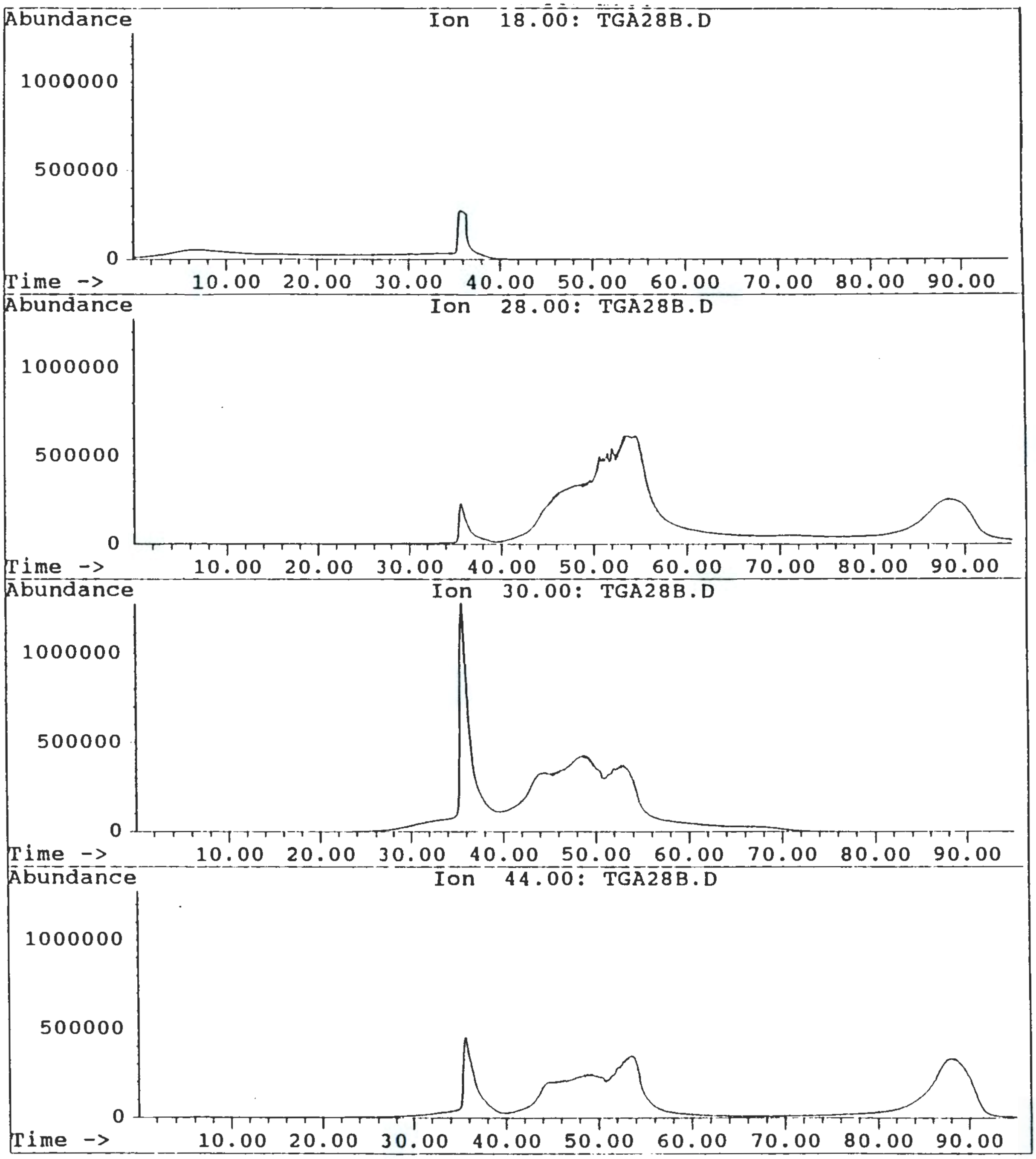
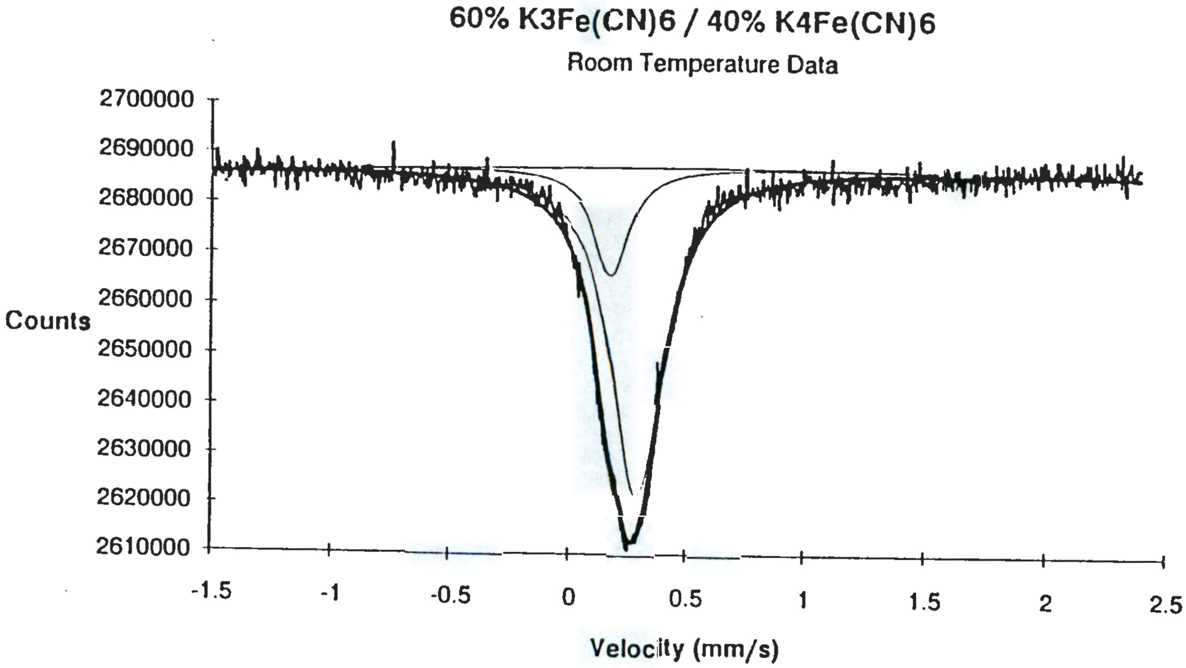


Figure 2-12. Representative Mossbauer Spectrum for a Ferri/Ferrocyanide Mix Obtained at Room Temperature.



Sensitivity and Large-Scale Thermal Tests--WHC and DOE tank advisory panel (TAP) have expressed concern about the sensitivity and large-scale tests. The concern is whether the material to be tested represents what is present in the tanks and whether a large-scale test would be of value. The objectives of this task were reviewed and modified. It was decided to defer the large-scale test at this time. The emphasis of the LANL testing will be on the non-thermal sensitivity tests. The mixtures selected for the sensitivity testing are a stoichiometric mix of sodium nickel ferrocyanide and equimolar sodium nitrate/nitrite and a mix representative of the in-farm scavenging flowsheet.

The first mix will be prepared at LANL by mixing washed vendor-supplied sodium nickel ferrocyanide. The materials have been shipped to LANL for the first mix. The presence of sulfate in the vendor-supplied material delayed the shipment. Prior to sending the ferrocyanide to LANL the decision was made to wash the material to remove the sulfate.

Reactivity Studies--Thermodynamic calculations were made to estimate the quantities of water, sodium nitrate, and a typical diluent, sodium aluminate, which will adsorb/remove the heat evolved in the ferrocyanide oxidation at 200, 250, and 307 °C. For the reaction of 1 g of sodium nickel ferrocyanide with 1.6 g of sodium nitrate at 200 °C, 3 g of water, 23.7 g of sodium nitrate, or 61 g of sodium aluminate would be required to absorb the heat released and prevent further temperature rise. Lesser amounts of each diluent would be required to adsorb the heat generated at the two higher temperatures.

The energetic properties of the various ferrocyanide materials have been evaluated. The vendor-supplied material (WHC-2) behaved similarly in TTX testing and in DSC tests to sodium nickel ferrocyanides prepared in the PNL laboratory, although differences do exist. In the TTX testing the minimum explosion temperature observed was about 290 °C for the vendor supplied material. The minimum observed exothermic reaction temperature with equimolar sodium nitrate/nitrite was 240 °C using the DSC. The general shape of the thermogram was similar to other ferrocyanides reacting with this oxidant, however the relative magnitudes of the various exothermic peaks differed from the washed sodium nickel ferrocyanide prepared in the PNL laboratory. The in-plant ferrocyanide (WHC-1) did not explode in TTX testing and only a small exothermic peak was observed in the DSC. This is consistent with the fact that this material contains very little ferrocyanide.

- **Planned Work For Subsequent Months.** Characterization of ferrocyanide materials will continue to determine their compositions as a function of how they were formed. Emphasis will continue on the species precipitated from the scavenging processes used for the high-level waste.

  Continue aging studies by performing gamma radiolysis, chemical oxidation, and hydrolysis/metathesis in highly alkaline solutions.

Chemically characterization of the aging products will be determined including the ratios of iron (II) to iron (III) and the effect of pH on the solubility of the ferrocyanide precipitates.

Continue reactivity studies of ferrocyanides representative of those precipitated during the scavenging campaigns.

Determine additional studies to address the effects of catalysts. Evaluate the effects of elements present in the ferrocyanide waste that have known catalytic effects, especially silver and copper.

In conjunction with LANL and PNL define the exact test mixtures to be evaluated and conduct a suite of sensitivity tests on representative synthetic material believed to be in the various ferrocyanide tanks. Conduct bounding tests with commercially prepared sodium nickel ferrocyanide mixed with stoichiometric amounts of nitrate/nitrite.

The annual report for FY 1991 PNL chemical reaction studies will be prepared and issued.

- **Problem Areas and Action Taken.** Aging studies have not been adequately designed. Aging processes will be studied by performing gamma radiolysis, chemical oxidation, and hydrolysis/metathesis in highly alkaline solutions. The details of these experiments need to be determined and planned. Methods to follow aging reactions have been determined, but the ferrocyanide material to use, that most closely simulates the in-tank material, has not been identified. Once this material is identified and becomes available, experimentation can be initiated. This work is very important because it must be determined if the ferrocyanide bearing material is still in the tanks as laid down, whether significant changes have occurred, and whether some of the ferrocyanide may have become soluble and been carried to other tanks during transfers or when the tanks were pumped as part of the interim stabilization program underway for all single-shell tanks at Hanford.

- **Milestone Status.**

  - July 31, 1991: Completed first meeting of tank waste science panel on ferrocyanide issues.

  - September 30, 1991: Completed status report of ferrocyanide characterization studies.

  - September 30, 1991: Completed status report of ferrocyanide characterization studies.

  - September 30, 1991: Completed status report of chemical nature of iron and cyanide in wastes.

- September 30, 1991: Completed screening and scoping studies initial set for catalysts, initiators, and diluents.
- November 30, 1991: Annual report of FY 1991 - On Schedule

2.5.2 Ferrocyanide Propagation Studies

Propagation studies are underway at Fauske and Associates, Inc. (FAI) under contract to WHC. These propagation tests are designed to test whether certain ferrocyanide materials will ignite and burn along a given length of material. The propagation velocity is a key parameter assuming ignition of the ferrocyanide and oxidant mixture. Certain mixtures will propagate at a rapid rate and others will burn slowly or not at all. The amount of water present in the material often makes the difference between reactions that will propagate and those that won't.

The overall test apparatus configuration at FAI is shown in Figure 2-13. The components of the apparatus include the following:

a. Sample holder and support stand.

b. Sample holder and support stand heater.

c. Insulation

d. Containment vessel (ultra high pressure up to 5000 psig).

e. Instrumentation and data acquisition system.

f. Igniter and power supply.

g. Nitrogen or argon pressure and purge system.

The 10 mm diameter sample holder is shown in more detail in Figure 2-14. It consists of a vertical thin wall tube with thermocouples located at the fixed axial positions identified in the figure. A burn can be initiated by the small nichrome igniter wire driven by a 12 volt dc power supply. The sample holder is a disposable item and can be easily fabricated and replaced if damaged in any test. A 25 mm sample holder has also been fabricated. Heaters and insulation are also shown in Figure 2-14. The heater is used to pre-heat the sample to achieve the desired initial sample temperature and is turned off during the ignition and reaction propagation event.

Scoping tests were conducted during the April-June quarter with commercially available potassium ferrocyanide in order to test out the apparatus and data acquisition system. Several modifications were required to achieve the desired response time and to assure collection of all data for extremely fast propagation velocities. In addition the effects of different parameters (such as density of the sample, moisture content, confinement pressure, stoichiometry, particle size, and geometry) were also investigated.

Figure 2-13. Overall Test Equipment Configuration.

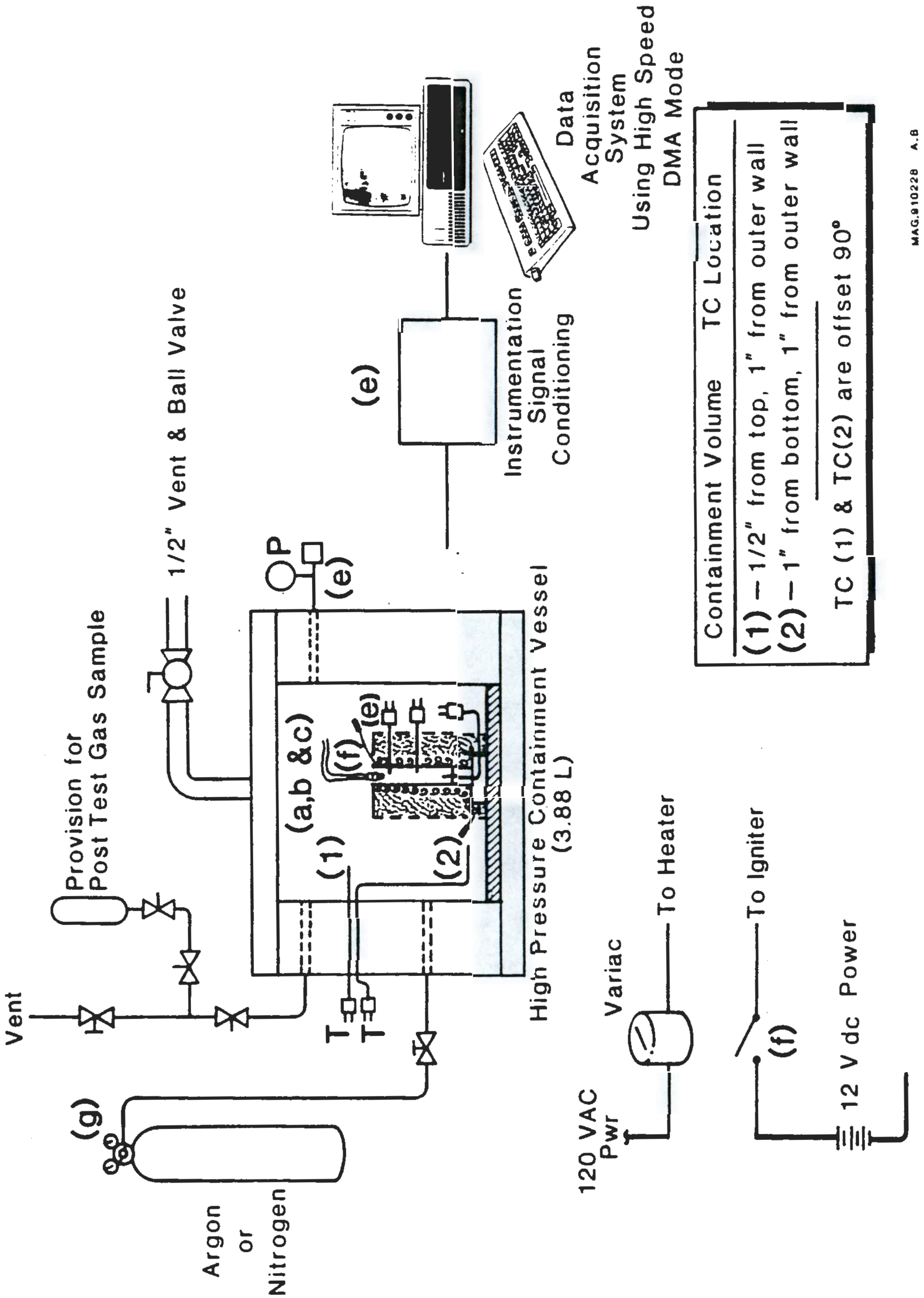
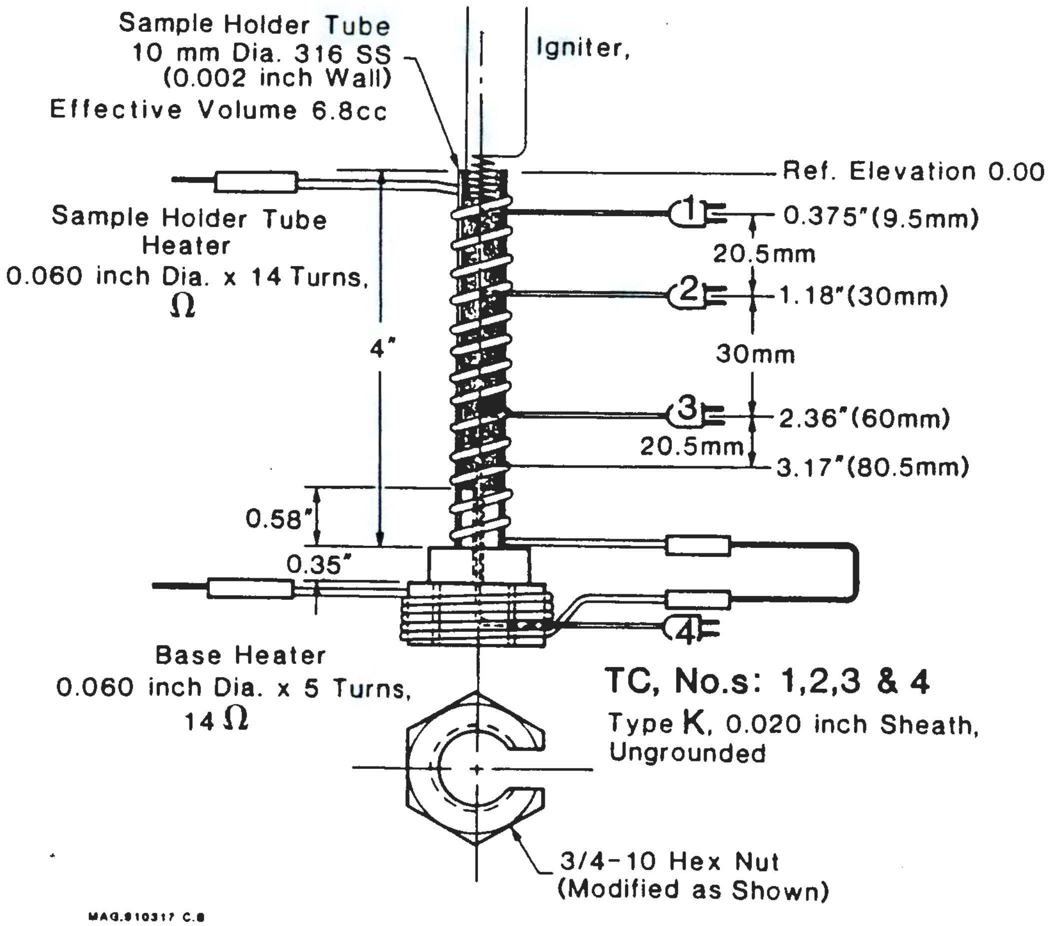


Figure 2-14. 10 mm Diameter Sample Holder Used in Burn Velocity Tests.



Conclusions drawn from these scoping tests are summarized below:

- Hydrated material is much less reactive than anhydrous material.
- Increased confinement pressure enhances propagation.
- Propagation rates were greater for 25 mm diameter than for 10 mm diameter tests.
- Gas results show the most favored reaction(s) produces carbonates with nitrogen and some CO_2 gases released.
- **Progress During the Reporting Period.** Propagation tests were completed this quarter on stoichiometric fuel/oxidant mixtures of $Cs_2NiFe(CN)_6$ and NO_3/NO_2 (3 to 1 mole ratio). Preliminary operations included moisture determination, particle size analysis, and packing density. Tests were run using the 10 mm diameter tube, at two pressures: 1 atm and 50 atm. Test results are shown in Table 2-2.

  Fauske and Associates also completed adiabatic scanning calorimetry (ASC) evaluations of the in-plant flowsheet synthetic material (WHC-1) provided by WHC. The flowsheet settled sludge was centrifuged at FAI to simulate a 30 equivalent gravity-year settling period and was then tested in the calorimeter. Because of the low ferrocyanide concentration, there was only weak exothermic activity and no sign of propagation even after the temperature was raised above 350 °C. These results suggest that no propagating reaction can occur in the tank bulk ferrocyanide material scavenged by the in-plant flowsheet. This flowsheet was used for all ferrocyanide-bearing material transferred to the BX Farm (4 tanks) and the BY Farm (10 tanks). Evaluation for other fuel sources (such as sodium citrate) in addition to the ferrocyanide in the waste needs to be completed since there is excess oxidant (nitrate/nitrite) in the waste.

- **Planned Work for Subsequent Months.** Other flowsheets (such as the in-farm and T-Plant flowsheets), which were used to produce smaller quantities of ferrocyanide waste at Hanford, need to be evaluated to determine their reactive properties. The in-farm flowsheet material with the highest ferrocyanide concentration is being produced on a pilot-plant scale (see Section 2.4.3) so that propagation tests can be performed with that material. This material will be ready for testing next quarter.
- **Milestone Status.**
  - November 30, 1991: Complete report on in-plant flowsheet tests - On Schedule
  - July 31, 1992: Complete report on in-farm and T-Plant flowsheet propagation tests

Table 2-2. Propagation Test Results with Cesium Nickel Ferrocyanide

CsNiFeCN PROPAGATION RATE

Stoichiometric CsNiFeCN Mix	Bulk Density	Propagation rate (cm/s) 1 ATM	50 ATM
Hydrous	1.25	0.44	1.6
Anhydrous	1.30	0.58	-

2.6 DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 90-7.6

"The Board had recommended 'that an action plan be developed for the measures to be taken to neutralize the conditions that may be signaled by alarms.' Two types of measures are implied: actions to respond to unexpected degradation of a tank or its contents, and actions to be taken if an explosion were to occur. Your implementation plan stated that 'the current contingency plans . . . will be reviewed and revised if needed.' We do not consider that this proposed implementation of the Board's recommendation is adequately responsive. It is recommended that a written action plan founded on demonstrated principles be prepared as soon as possible, that would respond to indications of onset of abnormal temperatures or other unusual conditions in a ferrocyanide-bearing tank, to counter any perceived growth in hazard. A separate emergency plan should be formulated and instituted, covering measures that would be taken in event of an explosion or other event leading to an airborne release of radioactive material from the tanks, and that would protect personnel both on and off the Hanford Site. The Board believes that even though it is considered that the probability is small that such an event will occur, prudence dictates that steps be taken at this time to prepare the means to mitigate the unacceptable results that could ensue."

2.6.1 Action Plan for Response to Abnormal Conditions

The *Action Plan for Response to Abnormal Conditions in Hanford Radioactive Waste Tanks Containing Ferrocyanide* (Cash and Thurman 1991) was prepared in response to DNFSB recommendations. The action plan describes the steps to be taken if a temperature increase trend above the tank temperature baseline is measured in any of the ferrocyanide tanks.

- **Progress During Reporting Period.** The action plan (Cash and Thurman 1991) issued earlier did not include criteria, monitoring frequencies, reporting requirements, and action response levels for flammable gases nor did it include actions to be taken if a ferrocyanide tank starts to leak. Responses to DOE were made on these issues this quarter.

  Requirements for flammable gases cannot be provided until gas sampling of the first ferrocyanide tank is completed and laboratory analyses of the samples are reported. The first tank (241-BY-104) is now scheduled for sampling during the first quarter of FY 1992, but analytical results will not be available until the second quarter of FY 1992. The frequency at which periodic monitoring should be completed is still being evaluated. Composition of the gas within the ferrocyanide tanks is expected to change very slowly with time. A computer model will be developed which will predict changes with time as a function of atmospheric conditions, tank temperatures, and hydrogen gas generation rates due to radiolysis of water. The presence of organic compounds in the waste will also be considered.

  Work is currently in progress for the ferrocyanide tanks to identify the quantities of liquid required in a tank for safety reasons and

the relationship of this liquid to drainable quantities. If a leak in a ferrocyanide tank were to occur before this work is completed, a decision to pump or not to pump a leaking tank would have to made based on the available information. If it is determined that pumping the leaking tank can be safety accomplished, the affected tank would be pumped in accordance with established emergency pumping procedures. If it should be determined, for safety reasons, that drainable quantities of liquid have to remain in a leaking tank, it can be assumed that water would be added to the tank to maintain the required level of drainable liquid. Of the 24 single-shell tanks classified as ferrocyanide tanks, 17 were interim stabilized before the identification of the ferrocyanide safety issue. The remaining seven tanks are scheduled for stabilization in FY 1993.

- **Planned Work For Subsequent Months.** The action plan (Cash and Thurman 1991) will be modified to include criteria and responses for abnormal levels of flammable and toxic gases and implemented during the second quarter of FY 1992.

  An engineering study will be started to evaluate the potential risk of pumping (interim stabilization) a ferrocyanide tank, especially if that tank should be discovered to be leaking. Criteria and recommendations will be developed as a result of this study by June 30, 1992.

- **Problem Areas and Action Taken.** None.

- **Milestone Status.** Updating the action plan (Cash and Thurman 1991) to incorporate flammable gas monitoring criteria is now scheduled for December 30, 1991.

2.6.2 Response to an Airborne Release From a Ferrocyanide Tank

If a radioactive release from a ferrocyanide tank were to occur, it would be detected by one or more radiation monitoring systems. Significant airborne or ground surface releases that spread beyond the immediate tank or tank farm would be detected by the tank farm area radiation detectors. These monitoring systems are on all tank farms. An emergency event involving an underground radioactive waste tank is a unique event with potentially serious consequences both onsite and offsite. DOE and WHC have analyzed the potential impacts of an event involving one of these tanks and have taken additional steps so that emergency personnel will be able to take mitigating actions in a timely fashion. These analyses resulted in development of the *Tank Farm Emergency Response Stabilization Plan* (WHC 1991) in March 1991. The plan includes predetermined mitigative actions for terminating the emergency phase and providing a transition to the recovery phase. Acknowledging that an event could range from minor to major releases, the plan addresses responses in four distinct and defined steps that will cover the range of consequences. The stabilization plan (WHC 1991) provides quick, preplanned actions that can be used to stabilize an emergency event at an underground radioactive waste tank.

- **Progress During Reporting Period.** WHC is in the process of upgrading the Hanford Site emergency response actions to cover tank farm emergency events. Additionally, planned emergency exercises have been conducted as scheduled, with additional exercises planned this calendar year. Emergency event recognition and classification procedures to respond to tank farm emergencies have been developed. The procedures were drafted and are currently undergoing final internal WHC review. Concurrence and approval of these procedures by DOE is required before training can be conducted and the procedures and training can be validated by an exercise. The criteria, which forms the basis for these procedures, was presented to DOE-HQ on September 19, 1991, with tentative verbal approval of the concept and criteria at that time.

  A radiological field team exercise was completed as well as a WHC Emergency Management (Emergency Control Center, and Emergency Management Center) exercise. County/State Emergency Management teams were also exercised as scheduled.

- **Planned Work For Subsequent Months.** Procedures for emergency event recognition and classification to respond to Tank Farm emergencies will be finalized and provided to DOE for review and approval. Following issuance of the procedures, a validation exercise will be conducted for emergency response organizations and a critique issued. An exercise is also planned to demonstrate Protective Actions necessary for Columbia River Closure.

- **Problem Areas and Action Taken.** Approval of the emergency event classification criteria is necessary prior to RL and HQ approval of Event Recognition and Classification Procedures for any Hanford Facilities. Verbal concurrence was obtained from DOE-HQ Defense Programs and Environmental Management emergency management personnel and a letter was sent to DOE-HQ Environmental Management and Restoration, on September 19, 1991 by RL, requesting formal DOE-HQ approval of the criteria. The subject procedures require DOE review and approval and have taken considerably longer than anticipated. Following DOE approval the procedures will be issued.

  The River Closure drill was scheduled for Friday September 19, 1991, but due to mechanical problems with the Hanford Patrol jet boat, the drill has been postponed with a scheduled completion date of October 25, 1991. The concerns expressed by WHC Emergency Planning and Safeguards and Security concerning availability of the Hanford Patrol boat, have been raised to RL.

- **Milestone Status.** Development and issuance of Tank Farm Emergency Event Recognition and Classification procedures has slipped to December 2, 1991.

  Conduct of validation exercises of emergency response organizations and the issuance of a critique for Tank Farm Emergency responders is behind schedule and has slipped to December 2, 1991. The radiological field teams drill was completed on schedule. The WHC Emergency Management exercise was completed on schedule. The

Protective Actions associated with Columbia River Closure has slipped to October 25, 1991. The County/State Emergency Management exercises were completed on schedule.

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3.0 SCHEDULES

The schedules shown in Figure 3-1 on the next three pages have been statused for the quarter ending September 30, 1991. Some milestones have been completed on or ahead of schedule while others have slipped from one to several months. Explanations for each slippage, where applicable, are described above in Section 2.0 for each affected task activity.

Delays were encountered this quarter in completing and approving pre-start items identified in the readiness review process for startup of vapor space sampling of 241-BY-104, the first ferrocyanide tank intrusive activity. These items were completed on October 8 and vapor space sampling activities started October 14, 1991. The actions required to resolve vapor space startup details have caused a slippage of other intrusive tank activities such as auger sampling of 241-BY-104 and vapor space sampling and push mode core sampling of 241-C-112. Preparation and approval of the required safety assessment, environmental assessment, and "finding of no significant impact" documentation for each one of these activities is taking much longer than previously estimated.

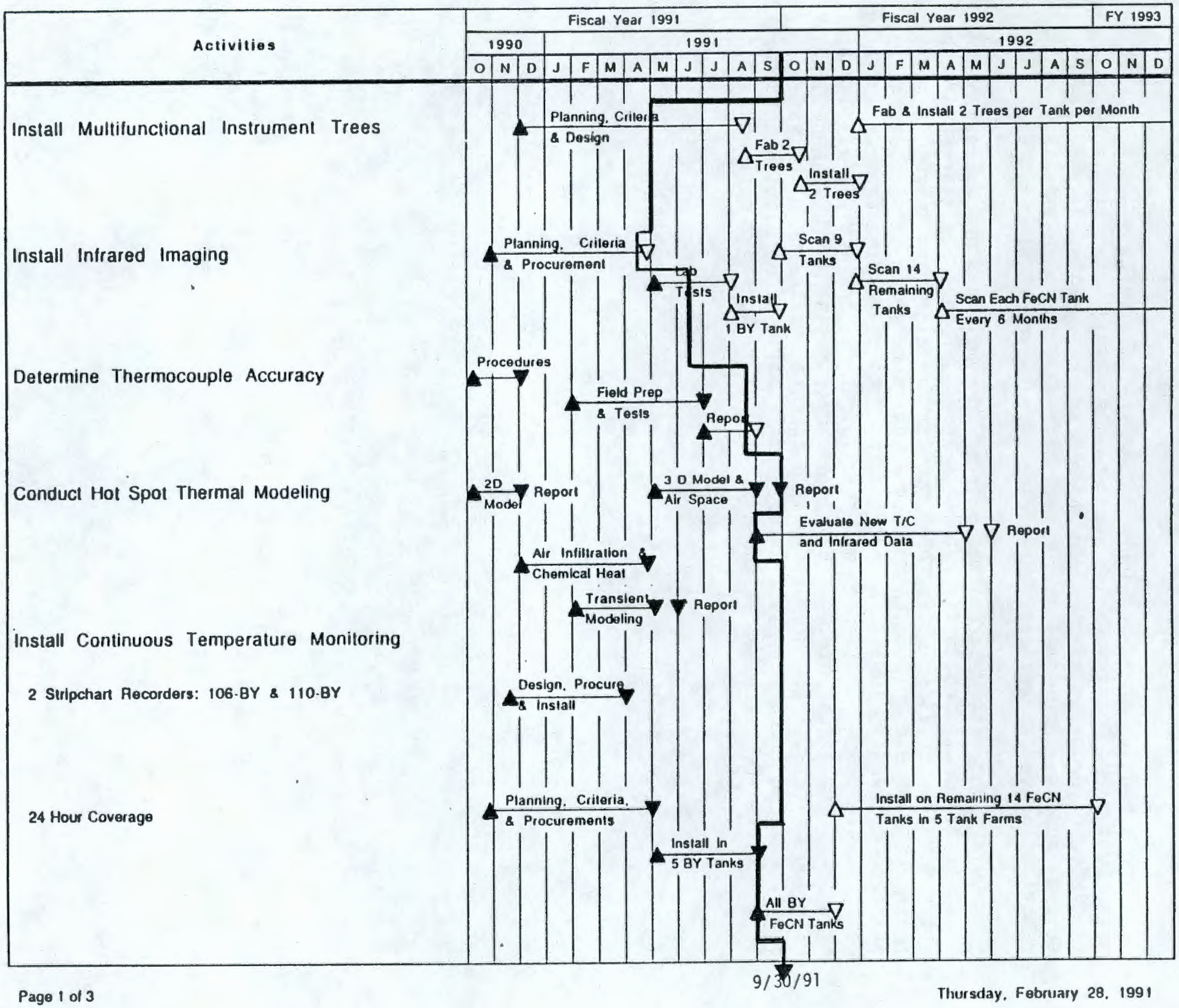

Figure 3-1. Schedule. (sheet 1 of 3)

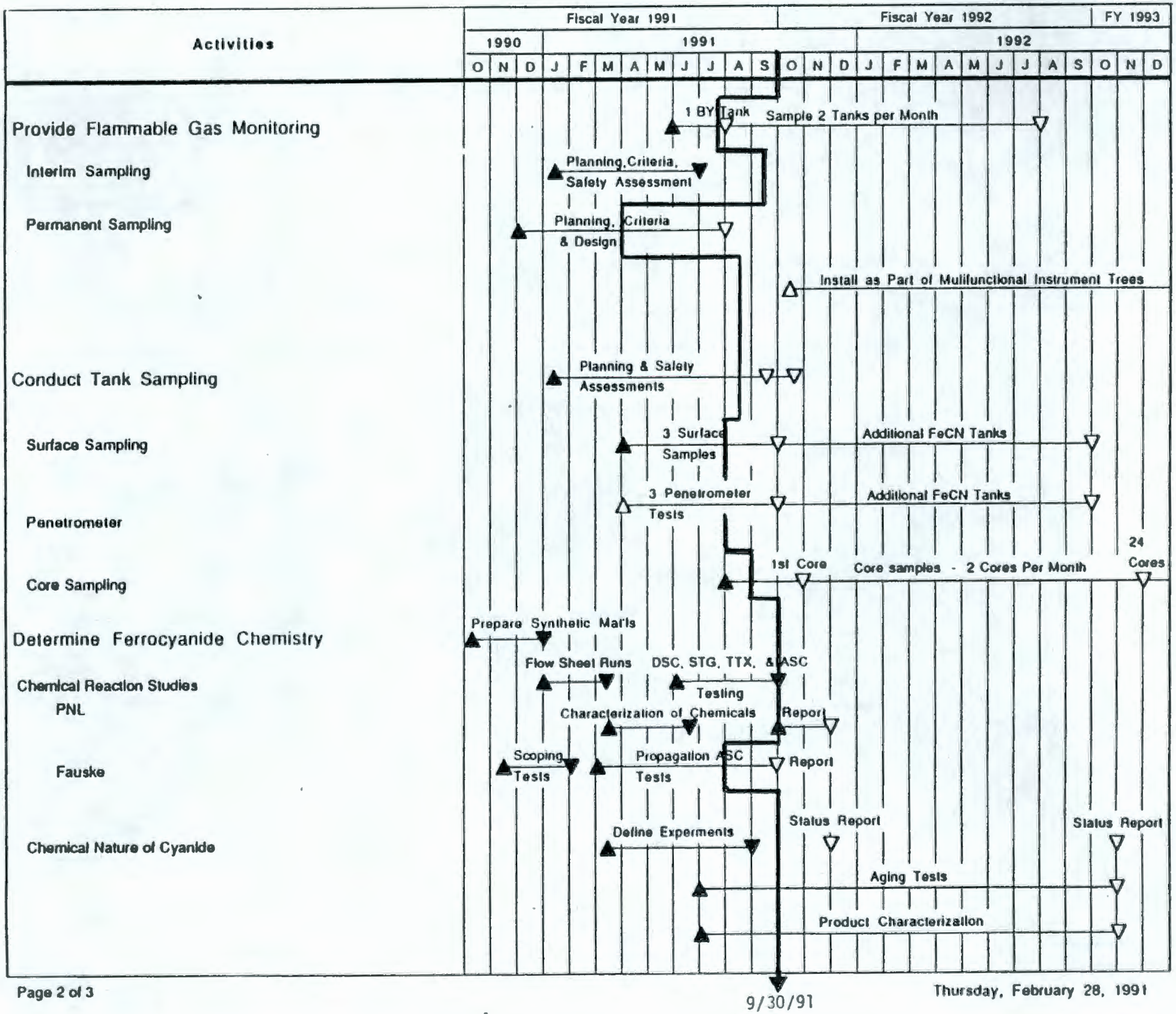

Figure 3-1. Schedule. (sheet 2 of 3)

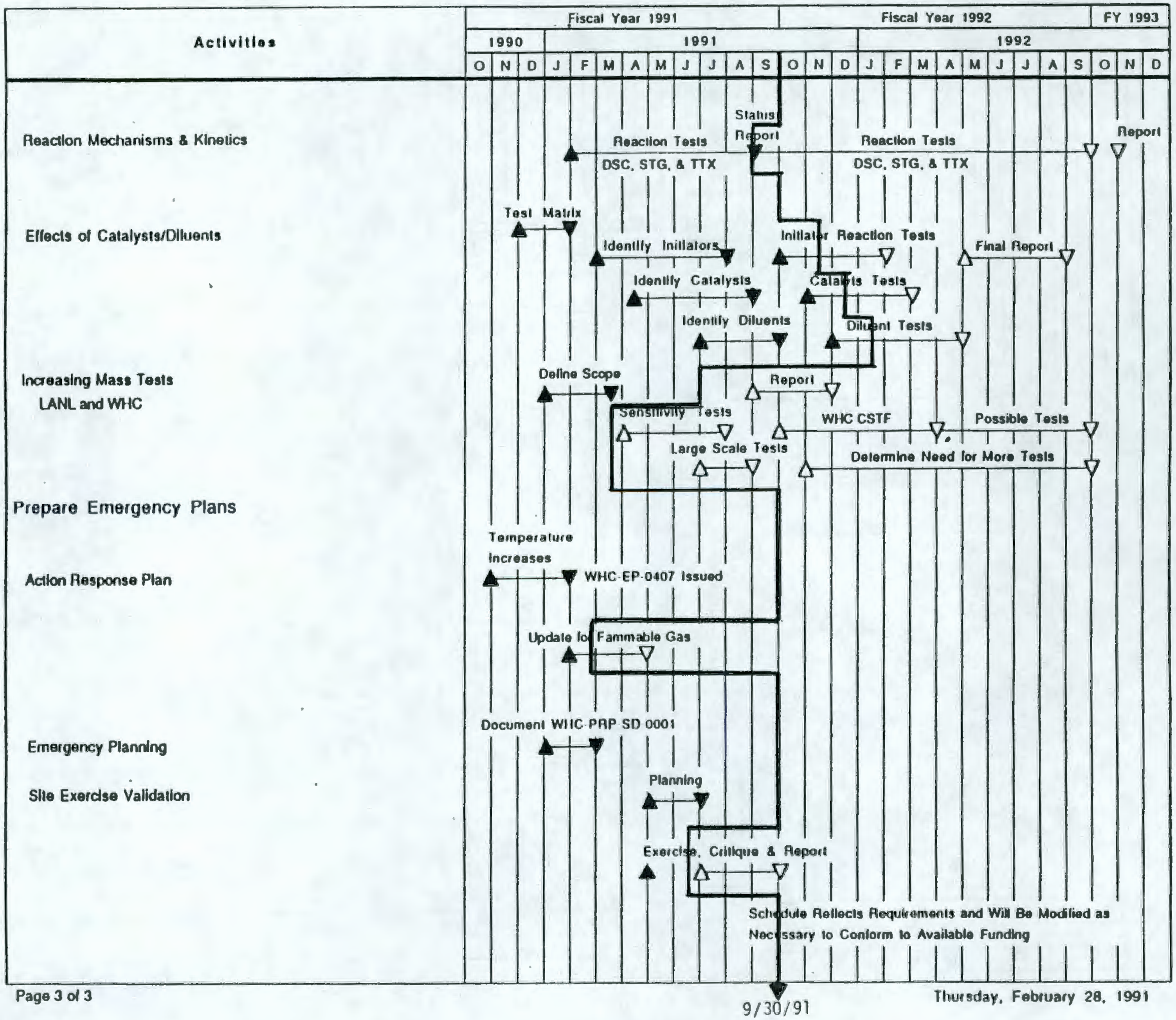

Figure 3-1. Schedule. (sheet 3 of 3)

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APPENDIX A

LIST OF FERROCYANIDE TANKS

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Table A-1. Revised Summary of Contents and Status of Ferrocyanide Tanks[a].

Tank	Total Waste Volume (1,000 gal)	FeCN (1,000 g mol)	Heat Load (Btu/h)	Maximum Temperature[b] (°C)	(°F)	Status
102-BX	96	< 1	10,000	22	71	Stabilized
106-BX	45	< 1	10,000	21	69	Not Stabilized
110-BX	199	< 1	10,000	20	68	Stabilized
111-BX	230	< 1	10,000	23	73	Not Stabilized
101-BY	387	< 1	8,200	23	74	Stabilized
103-BY	400	66	8,600	27	81	Not Stabilized
104-BY	406	83	17,000	54	130	Stabilized
105-BY	503	36	37,700	46	114	Not Stabilized
106-BY	642	70	12,200	55	131	Not Stabilized
107-BY	266	42	14,500	28	83	Stabilized
108-BY	228	58	23,000	38	101	Stabilized
110-BY	398	71	25,200	51	124	Stabilized
111-BY	459	6	34,200	33	92	Stabilized
112-BY	291	2	<10,000	30	86	Stabilized
108-C	66	25	10,000	26	78	Stabilized
109-C	66	30	10,000	27	81	Stabilized
111-C	57	33	<10,000	26	78	Stabilized
112-C	109	31	<10,000	29	85	Stabilized
101-T	133	< 1	<10,000	23	73	Not Stabilized
107-T	180	5	<10,000	23	74	Not Stabilized
118-TX	347	< 3	4,900	23	74	Stabilized
101-TY	118	23	<10,000	20	68	Stabilized
103-TY	162	28	<10,000	21	70	Stabilized
104-TY	46	12	<10,000	22	71	Stabilized

Total Gal: 5,834,000

[a]Based partially on information in Hanlon, 1991.
[b]September 1991.

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APPENDIX B

METRIC CONVERSION CHART

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Metric Conversion Chart

Into Metric			Out of Metric		
If You Know	Multiply By	To Get	If You Know	Multiply By	To Get
Length			Length		
in.	2.54	cm	mm	0.04	in.
ft	30.48	cm	cm	0.4	in.
Mass (weight)			Mass (weight)		
lb	0.453515	kg	kg	2.2	lb
Volume			Volume		
gal	3.78541	L	L	0.264172	gal
Temperature			Temperature		
Fahrenheit (°F)	Subtract 32 then multiply by 0.55555...	Celsius (°C)	Celsius (°C)	Multiply by 1.8, then add 32	Fahrenheit (°F)

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