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ANALYSIS OF THE
OCTOBER 5, 1979 LITHIUM SPILL AND FIRE
IN THE LITHIUM PROCESSING TEST LOOP

by

V. A. Maroni, R. A. Beatty, H. L. Brown,
L. F. Coleman, R. M. Foose, C. C. McPheeters,
M. Slaweki, D. L. Smith, E. H. Van Deventer,
and J. R. Weston

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

Prepared for the U. S. DEPARTMENT OF ENERGY
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Printed in the United States of America
Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

NTIS price codes
Printed copy: A05
Microfiche copy: A01

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Printed in the United States of America
Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

NTIS price codes
Printed copy: A05
Microfiche copy: A01

ANL-81-25

DE82 009489

Distribution Category:
Magnetic Fusion Energy
(UC-20)

ANL-81-25

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9700 South Cass Avenue
Argonne, Illinois 60439

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Chemical Engineering Division

December 1981

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ABSTRACT

On October 5, 1979, the Lithium Processing Test Loop (LPTL) developed a lithium leak in the electromagnetic (EM) pump channel, which damaged the pump, its surrounding support structure, and the underlying floor pan. A thorough analysis of the causes and consequences of the pump failure was conducted by personnel from CEN and several other ANL divisions. Metallurgical analyses of the elliptical pump channel and adjacent piping revealed that there was a significant buildup of iron-rich crystallites and other solid material in the region of the current-carrying bus bars (region of high magnetic field), which may have resulted in a flow restriction that contributed to the deterioration of the channel walls. The location of the failure was in a region of high residual stress (due to cold work produced during channel fabrication); this failure is typical of other cold work/stress-related failures encountered in components operated in forced-circulation lithium loops. Another important result was the isolation of crystals of a compound characterized as Li_xCrN_y . Compounds of this type are believed to be responsible for much of the Fe, Cr, and Ni mass transfer encountered in lithium loops constructed of stainless steel. The importance of nitrogen in the mass-transfer mechanism has long been suspected, but the existence of stable ternary Li-M-N compounds (M = Fe, Cr, Ni) had not previously been verified.

Conclusions reached from the analysis of the failure, evaluations of the LPTL operating conditions, and experience with other lithium loop systems include the following: (1) in liquid metal loop systems such as the LPTL, a cover-gas pressure venting system should be used to guard against overpressurization of the dump tank during an emergency situation; (2) further studies are needed on stress, strain, and fatigue as well as mass transfer phenomena in large flowing-lithium systems constructed from stainless steel, with particular emphasis on the effects of impurities and magnetic fields; and (3) fire suppressants and fire-fighting procedures recommended for use on lithium fires could be improved further.

I. INTRODUCTION AND BACKGROUND

A. Summary of the Incident

The Lithium Processing Test Loop (LPTL)--a 50-gal, electromagnetically pumped liquid-lithium loop--was constructed in and by the Argonne National Laboratory/Chemical Engineering Division (ANL/CEN) to carry out lithium-blanket development work for the U.S. Department of Energy/Office of Fusion Energy (DOE/OFE). Construction of the LPTL was completed in December of 1977, and operations were started in January of 1978. The LPTL, thereafter, achieved ~9500 h of operation (excluding several maintenance shutdowns) up to the end of August 1979. Following some required repairs of heaters and instrumentation, the LPTL was restarted on October 4, 1979.

At approximately 12:40 p.m. on October 5, 1979, a leak developed in the channel of the EM-pump, which spilled ~20 gal of lithium onto the floor of the LPTL enclosure (a metal-lined, concrete cell). The spilled lithium caught fire immediately, setting off a smoke alarm which summoned the ANL Fire Department (ANL/FD). The ANL/FD arrived in less than three minutes from the time of the alarm and had the fire under control in about forty minutes. The principal consequences of the fire were destruction of the EM-pump, damage to the LPTL facility floor pan, and the deposition of a finely powdered residue of lithium compounds and fire extinguishant powder (mostly graphite) on walls, floors, and equipment surfaces within the LPTL cell and in adjacent corridors and laboratories. The failure-detection and safety-shutdown systems performed as designed; and, with the exception of the residue deposited outside the LPTL cell, the leak, the heat from the fire, and related effects were well contained. However, several facility- and procedure-related difficulties encountered during the incident gave evidence of need for corrective action in order to reduce the impact of any similar incidents in the future. These difficulties involved the procedures and hardware used for cover-gas pressure control, the methods used to seal off the LPTL cell in the upper levels, the reliability of the scrubber pump, and the optimum choice of fire suppressant material. They are discussed in greater detail in Sections II through V, and recommendations for improvements are given in Section VI.

Immediately after the incident on October 5, 1979, the supporting agency, DOE/OFE, was notified of the events and immediate consequences, and written authorization was received from DOE/OFE to conduct a thorough analysis of the incident and to include the analysis activity itself as an objective/milestone for the FY 1980 program on lithium processing technology. Accordingly, L. Burris, Jr., Director of CEN, approved the formation of an incident analysis team to investigate the causes, effects, and lessons learned from the spill, the fire, the cleanup/repair, and the examination of failed components.

B. Incident Analysis Team: Membership and Approach

The persons assigned to the Incident Analysis Team (IAT) together with a summary of their qualifications are listed below.

V. A. Maroni: (Chairman)

A member of ANL/CEN, leader of the Fusion Reactor Research Group, and principal investigator of the program that directed LPTL operations.

J. R. Weston:

A member of ANL/CEN, the operator of the LPTL facility, and one of the key individuals involved in extinguishing the fire and conducting the cleanup/repairs.

M. A. Slawewski:

A member of ANL/CEN and of the ANL/CEN Emergency Brigade; also the lead designer of the LPTL instrumentation and control systems.

C. C. McPheeters:

A member of ANL/CEN, leader of the CEN Sodium Technology Group, with over fifteen years of experience in liquid-metal systems design and operation.

D. L. Smith:

A member of the ANL Materials Science Division (MSD) with over fifteen years of experience in liquid metals technology, primarily in the area of materials compatibility, corrosion processes, and mechanical properties effects.

H. L. Brown:

ANL/CEN Assistant Division Director; maintenance overseer for plant, laboratories, and engineering facilities.

L. F. Coleman:

The ANL/CEN Safety Coordinator and emergency supervisor.

R. M. Foote:

A captain in the ANL/FD and the person in charge of extinguishing the fire in the LPTL facility.

R. A. Beatty:

A member of the ANL/Quality Assurance Division (QAD) assigned to ANL/CEN.

F. A. Cafasso:

The Associate Division Director within ANL/CEN responsible for all fusion-related work within CEN, including LPTL operations.

D. S. Webster:

Deputy Director of ANL/CEN.

The analysis followed the procedures described in Ref. 1. Six separate subgroups, each having a subgroup leader and two additional members, were set up to explore the principal factors/concerns that contributed to, or resulted from, the incident. These factors/concerns related to: (1) the chronology of

events and scenario for the incident, (2) the manner in which the LPTL enclosure (cell) contained the fire and fumes therefrom, (3) the manner in which the ventilation-exhaust/scrubber system performed during the incident, (4) the performance of the fire extinguishants used during the incident, (5) the analysis of the failed pump channel and adjacent components, and (6) incident documentation and quality assurance analysis. The findings in each of these six areas and the resulting recommendations are described in the remaining sections and appendices of this report. A summary is given in Section VI.

C. Description of the LPTL Facility

The principal features of the LPTL system are shown in Figure I-1. The EM-pump circulates lithium contained in the reservoir tank (64-gal capacity) through one of four side streams. The four side streams are (1) a bypass; (2) a molten-salt (alkali halide) extraction tank which operates like a pseudo-mixer settler; (3) a series combination of a hot getter trap containing zirconium and operating at 500-650°C and a cold trap operating at 200-300°C to remove oxygen, nitrogen, carbon, and other impurities formed from the constituents of the structural materials; and (4) an auxiliary branch which is intended to house add-on experiments. The loop also contains two permeation-type hydrogen monitors, a 64-gal capacity dump tank, four magnetic flow meters, a motorized dump valve, and 10 bellows-sealed process control valves which are designed for high-temperature liquid metal service.

The LPTL is designed for operation up to 30 psig and 550°C except for the getter trap which is designed for operation up to 650°C. Normal cover-gas operating pressure was ≤ 4 psig of ultra-high-purity argon in the reservoir, the dump tank, and the salt tank. The total lithium in the entire system was in the 40- to 45-gal range from the time of initial startup. The tanks and other vessels and the piping are Type 304L stainless steel (SS). Some components in contact with the liquid lithium were not readily available in Type 304L stainless steel (SS) and substitute materials were used. For example, the valve bodies were Type 316-SS and the valve seats were stellite. The flowmeter channels were Type 304-SS and the pump channel was Type 316-SS. The 316-SS, 304-SS and stellite were expected to perform as well in service with liquid lithium as 304L-SS under the operating conditions proposed for the LPTL (see Ref. 2). Vermiculite, Kaylow, calcium silicate, and KaoWool (all halogen-free insulations) were the only insulating materials used on LPTL. All joints that were contacted by liquid lithium were welded, and, to verify their integrity, all accessible boundary welds were dye-penetrant checked, radiographed, and helium leak tested.

The system piping is 1/2-in. Schedule 40 pipe joined with full-penetration butt welds. An exception to this piping size is the 1-in. Schedule 40 dump line connecting the reservoir to the dump tank. The piping layout is designed to avoid exceeding allowable stresses due to thermal expansion up to the maximum operating temperature. All lithium piping is installed with a downward slope so that the lithium in the system can drain into the dump tank by gravity and gas bubbles can migrate to the cover gas in the reservoir tank.

The EM-pump in the main piping provides the driving force to circulate the liquid lithium through the piping system. This pump (a product of Mine Safety Appliances Co.) is a two-stage electromagnetic conduction pump with

Fig. I-1. Lithium Processing Test Loop

a pumping section of Type 316-SS tubing. The flow rate of this EM-pump is controlled from zero to a maximum of approximately 4 gpm at 10 psi by an adjustable auto-transformer. Information on the type of pump used in the LPTL is given in Ref. 2. The selection of this pump for use with lithium was not expected to lead to any operational problems apart from those which might be encountered in sodium systems of a similar design.

The LPTL vessels (the dump tank, the reservoir, the salt tank, the cold trap, and the getter trap) are heated by tubular-type rod heaters attached to the outer surface of the vessels. In addition, separate heaters are wound around all pipe sections and the remaining components. These heaters are Nicrome wire, insulated with asbestos and glass and covered with a braided Monel wire sheath (GE wire). Normal rating of this type of heater is 10 W/ft. All of the 1/2-in. lithium valves are heated with clamshell-type heaters having Nicrome elements embedded in a ceramic casting which is molded to conform to the body of the valve. This type of heater is capable of supplying up to 30 W per square inch of inside area of the casting.

Leak detection is provided by a separate wrap of GE wire around all vessels and piping. The heating and leak-detection wraps are enclosed in a thin shell of stainless steel shim stock (retained by steel bands). This shell is expected to contain lithium (flowing through a leak) for a sufficient length of time to permit the leak detector to be activated and the shutdown systems to respond. The only section of the entire LPTL system that was not protected by a leak detection wrap was the center portion of the EM-pump (between the bus-bar brazes), which is where the leak occurred in the October 5, 1979 incident.

The LPTL complex is controlled from a console located on the second level of the surrounding high bay area outside the LPTL cell. The console contains nine individual relay racks. Access to each rack is possible from the rear. All power and control leads are routed from the console to the LPTL cell through four separate 2-in. wireways. Both instrument and power leads run to outlet panels which have been distributed outside the cell for ease of connection to all LPTL heaters, thermocouples, leak sensors, etc.

A carbon steel catch pan (with elevated grating) covered the floor under the LPTL system and was large enough to contain all the lithium in the system in the event of a major lithium leak from the piping or components. There are inlet ducts to a filtered exhaust system near the bottom and top of the cell. The exhaust system removes the normal heat loss from LPTL components and, in the event of a fire, should also remove the smoke. A smoke detection signal from either of the two smoke detectors inside the LPTL cell automatically activates a water scrubber, through which the exhausted air passes before being discharged through a bank of "absolute" air filters. The exhaust blower has a rated capacity of 6,000 cfm.

More detailed descriptions of the LPTL and its operating characteristics are presented in the LPTL Operations Manual² and in Refs. 3-7.

D. LPTL Safety Systems (Summary)

The LPTL complex has two types of safety alarms: those that require loop shutdown and complete lithium dumping sound a bell at the console, and those that require other action sound a buzzer. In addition, there are other interlock and bypass circuits to provide for complete system safety. Table I-1 lists the safety features for this system.

Circuitry (see Fig. I-2) is provided for the main safety alarms, i.e., lithium leak, smoke, and low flow, so that an immediate indication of which alarm caused system shutdown can be seen. Whenever any of the main safety

Table I-1. LPTL Safety Systems

-
- Electrical shorting-type leak detectors on all tanks, lines, valves, and valve bonnets.
 - Photoelectric smoke detectors on LPTL cell exhaust.
 - Induction-type level monitor for low liquid level in the reservoir.
 - Low-flow shutdown of lithium EM-pump.
 - Interlock on dump valve to prevent operation in the event of low temperature.
 - Manual back-up operator for dump valve.
 - Main tank heaters provided with individual high-temperature shutdown.
 - Power level on line and valve heaters manually set to limit temperature.
 - Principal power outage sensor to prevent shutdown due to short-duration power outage.
-

alarms are tripped and the system dumps, all remaining alarms are locked out, and only one main safety alarm light is illuminated on the control panel. This feature is inoperative only when a main power outage is of sufficient duration to dump the system. All high-temperature alarms shut off the heaters in the affected zone. The heaters stay off until they are manually reset.

Each leak detector is incorporated into a fail-safe circuit. Under no-leak conditions, the leak relay is energized from a low-voltage DC source through an 800 Ω current-limiting resistor. When a leak develops in any of the leak-detector circuits, the relay is shorted out, an indicator light on the alarm panel goes on (locating the zone wherein the leak occurred), and the dump valve opens so that the lithium can drain into the dump tank. The only exception to this course of action is if a leak develops in the dump tank. In this one instance, an alarm light flashes on the alarm panel, a buzzer sounds, and the laboratory central surveillance system (CSS) receives a coded signal reporting the conditions. (No lithium dumping would occur at this time unless initiated manually.) Should a leak of any type occur, push-button switches are provided on the console to aid the operator in identifying which of three possible zones transmitted the leak signal.

Two photoelectric-type smoke detectors are located in the LPTL cell near the openings to the cell air exhaust system. An indication of smoke from any of the two detectors in the LPTL cell will trigger a light on the control panel, sound a bell, shut down the heaters, open the dump valve, activate the exhaust scrubber system, and summon the fire department.

Fig. I-2. LPTL Control System Block Diagram

A low lithium level in the reservoir tank is sensed by a Mine Safety Appliances (MSA) inductive level probe inserted in a well that penetrates the top of the tank. The level is shown by an indicator on the control panel. An electronic alarm card with adjustable set point, whose input parallels the level-indicator meter input, is used to set the level at which a low-level alarm occurs. When a low lithium level occurs, a light on the main shutdown alarm panel will light, a bell will sound, the system will shut down, and the dump valve will open and dump all lithium. A bypass switch and flashing light are provided for operations where manual lithium level adjustment in the reservoir tank is required.

A lithium flow indicator-controller is provided for indication of lithium flow in the main bypass line. The low-flow point is set at some predetermined value; and when low flow is indicated, a light is triggered on the process alarm panel, a buzzer sounds, and the EM-pump is shut down. A 10-sec delay is provided for flow perturbations of short duration. A bypass switch and flashing light are provided for the flow indicator-controller for operations where known lithium-flow changes in the system are made.

The shutdown alarms (e.g., lithium leaks, smoke detection, and low-level indication) provide the necessary surveillance of malfunctions in LPTL. These alarms, however, rely on the capability of the dump system to deposit all the lithium into a large dump tank in the event of a malfunction. The LPTL dump system consists of a motor-operated valve and the necessary control circuitry for proper operation of the dump valve. The valve can be operated manually with the use of momentary contact push buttons mounted on panels. Also located on the panel are a valve position indicator and pilot lights which show whether the valve is fully open or fully closed. When the LPTL is operating normally, the dump valve is fully closed. Under automatic emergency dumping conditions, the shutdown alarm system energizes the circuit that causes the dump valve motor to open the valve. The dump valve must be at the proper temperature, and emergency power must be available for the motor to operate. The motor operator will open the valve until an adjustable torque switch in the operator deenergizes the starter coil. Lithium will start to dump as soon as the valve seat is lifted. Approximately 10 sec is required to take the valve from full-close to full-open and vice versa.

Another mode of dump-valve operation occurs when principal power is lost for more than 2 min and only emergency power is available. In this case, a time-delay switch closes, engaging the motor starter coil which energizes and opens the dump valve.

E. Operational Characteristics of the LPTL

The operational characteristics of the LPTL and its precursor test stand in ANL/CEN, the Lithium Mini-Test Loop (LMTL), are listed in Table I-2. The principal objective of the LPTL and related studies in ANL/CEN is to develop and test methods for tritium recovery, impurity control, and process monitoring for liquid-lithium fusion reactor blankets. The types of impurity control methods that have been investigated on the LPTL (and the LMTL) include cold trapping, getter trapping, and molten-salt extraction. Figure I-3 shows the range of achievable lower limits in impurity control levels determined for each of these methods from work done on the LPTL and the LMTL; these findings are discussed in more detail in Ref. 3. Two impurity monitoring techniques

Table I-2. Characteristics of the Lithium Processing Test Loop and the Lithium Mini-Test Loop

Characteristic	Lithium Processing Test Loop	Lithium Mini-Test Loop
Loop Capacity	~50 gal	~1 L
Construction Material	Mostly 304L-SS (some 316-SS)	304 and 316-SS
Operating Temperature		
Main Loop	400-500 °C	450-550 °C
Getter Leg	400-500 °C	550-600 °C
Cold-Trap Leg	180-300 °C	---
Salt-Extraction Leg	500-530 °C	---
Length of Operation	>9500 h	~10,000 h
Experimental Capabilities		
Direct Sampling	Filter pipette	Filter pipette
Cold Trapping	Mesh-packed trap	---
Getter Trapping	Zirconium-packed trap	Zirconium-packed trap
Hydrogen Monitoring	Permeation-type	---
Salt Extraction	LiF-LiCl-LiBr	---

Fig. I-3. Achievable Lower-Limit Impurity Control Levels for Selected Lithium Processing Methods

(one based on electrical resistance measurements and the other based on the use of metal membranes that are permeable to hydrogen) have also been investigated using the LPTL. Results of this work are summarized in Table I-3 and are discussed more thoroughly in Ref. 7.

The materials-related performance of the LPTL and the LMTL has also been studied in some depth. In particular, there have been a number of failures involving special components that contain cold-worked or highly stressed sections of thin stainless steel. The characteristics of these failures (including the pump failure documented in Section IV of this report) are summarized in Table I-4. It is worthy of note that these failures have been limited to pump channels and valve bellows and that no other structural failures have occurred in pipe or tubing sections, welds, flow-meter channels, and the like. However, there has been evidence in both the LPTL and the LMTL of long-term mass transfer effects involving the constituents of the structural materials (iron, chromium, and nickel in the case of both systems), as discussed later.

In summary, the processes involved in corrosion of austenitic stainless steels by lithium appear to involve (1) a rapid (<500 hr) depletion of nickel in the near-surface region of the stainless steel due to dissolutive attack by lithium, which reaches depths of up to 40 μm depending on temperature and impurity level but slows down parabolically in rate with increasing depth; (2) a longer-term (>1000 hr) general dissolutive corrosion, which involves all the main structural elements, Fe, Cr, and Ni, and may be assisted and, in

Table I-3. Development of On-Line Monitors Using the LPTL^a

-
- An electrical-resistance-type probe has been used to monitor cold-trap performance.
 - Real-time monitoring of hydrogen-isotope cold trapping has been achieved.
 - Instantaneous sensitivity to concentration changes in hydrogen and/or nitrogen impurity of 100 appm has been observed.
 - A permeation-type hydrogen meter employing a Nb-1 Zr membrane has been tested.
 - Marginal sensitivity was achieved at 375°C.
 - Extrapolated sensitivity at 500°C is <10 appm of hydrogen.
 - A combination of resistivity- and permeation-type meters can be used to selectively follow hydrogen isotopes and nitrogen.
-

^aSee Ref. 7 for details.

fact, dominated by effects of impurities such as carbon and nitrogen; and (3) the growth of dendritic deposits of both iron and chromium, which often occur in higher temperature regions or, in the case of iron, in regions of high magnetic field such as an EM-pump channel.

Examples of the types of effects described in the preceding paragraphs are illustrated in Figs. I-4 through I-7. Figure I-4 contains a composite of photographs and photomicrographs of the Nupro-Type-T valve failure that occurred on the LMTL. A number of cracks were found in the outer convolutions of the bellows, as shown in the upper right-hand insert. The microscopic results reveal that much deeper intergranular cracking occurred in the acutely curved section of the bellows than along the flattened section. The etched specimen (lower right) shows clear evidence of the residual stress in the bellows material. Photographs obtained from the SEM analysis in the region of the failed pump channel in the LMTL are shown in Fig. I-5. Here again, the point of failure was at a position of high residual stress, i.e., one of the sharply curved corners of the rectangularly deformed tube from which the channel was fabricated.

Examples of the consequences of the general corrosion processes discussed above are illustrated in Figs. I-6 and I-7. In the upper two horizontal rows of inserts in Fig. I-6, the iron (ferrite) buildup, chromium segregation and dendrite formation, and nickel depletion can be seen in the comparative electron microprobe scans. Nickel deposition in a lower-temperature region of the

Table I-4. Component Failures and Their Analysis

Failed Component	System	Operating, h	Temperature History	Nature of Failure	Cause of Failure
EM-Pump Channel	LMTL	~7200	450-500 °C	Crack in seam of rectangular section.	Stress-related rupture in a heavily cold-worked region.
Nupro-Type-T Valve	LMTL	~7000	450-500 °C	Valve seizure in open position (no evidence of leakage).	Crack in bellows due to repeated operation (over 300 cycles).
Nupro-Type U Valve	LPTL	~3400	380-480 °C	Leakage of lithium into valve bonnet.	Crack in bellows, possibly due to being struck by a tool during a repair operation.
EM-Pump Channel	LPTL	~9600	380-480 °C	Massive leak of lithium through two large cracks in curved edge of channel.	See Section IV of this report.

**MICROSCOPIC EXAMINATION OF THE FAILED
BELLOWS FROM THE 10,000 HOUR LMTL TEST***

- MATERIAL: 321- or 347-SS (Determination in Progress)
- TEMPERATURE: 450-500°C
- HISTORY: >300 Operating Cycles

*[Courtesy of P. F. Tortorelli and J. H. DeVan (ORNL)]

200 400 μm 1200 1400
0.01 50X in. 0.05

20 40 60 μm 120 140
0.001 500X in. 0.005

20 40 60 μm 120 140
0.001 500X in. 0.005

20 40 60 μm 120 140
0.001 500X in. 0.005

Fig. I-4. Microscopic Examination of the Failed Bellows from 10,000-h LMTL Test

Polished
Sample

| 100 μm

Etched
Sample

| 100 μm

Fig. I-5. SEM Analysis of LMTL Pump Channel Failure

Fig. I-6. Specimens of Pipe Sections from 10,000-h LMTL Test

Fig. I-7. SEM Analysis of LPTL Corrosion Test Rod (Material: 304-SS; exposure temperature: $410 \pm 20^\circ\text{C}$)

same loop is indicated in the lower right-hand insert (EM pump channel set). Figure I-7 contains a collection of corrosion results obtained by exposing a 304-SS test rod to the lithium in the LPTL reservoir tank during early operating phases. This rod showed a decreasing rate of ferrite layer formation (nickel depletion) with time (up to ~600 h) and the absence of evidence of intergranular attack (etched sample).

The metallurgical information summarized in this section has been included in the report to provide a basis for the interpretation of results of the LPTL pump-channel failure analysis discussed in the following sections (particularly Section IV).

F. Quality Assurance Aspects of LPTL Construction and Operation

This section contains a summarized evaluation of the aspects and levels of quality assurance that were applied during planning, design, construction, operation, and maintenance of the LPTL facility. The failure analysis verified that quality assurance elements appropriate to a one-of-a-kind experimental test facility like the LPTL had been planned and implemented. Detailed specifications were written for the fabrication and installation of all vessels and piping. Preliminary and final design/operation safety reviews were held within ANL/CEN. Cognizant individuals outside ANL were solicited for informal review and comment on the design details and drawings for the LPTL prior to construction.

Provisions implemented by the Project Manager to ensure personnel safety, validity and reproducibility of experimental results, reliability and availability of the facility, and maximum project productivity included, but were not limited to, the following:

- design in conformance with accepted codes and standards
- documented finite-element stress analyses of the LPTL piping network
- design reviews
- safety reviews
- written analyses of abnormal conditions
- detailed specifications for procurement of one-of-a-kind components
- certified materials, weld procedures, and welders for critical welds
- procurement source surveillance and quality control
- procedures for weld inspection and weld repair
- nondestructive evaluation of welds (dye-penetrant test, radiography, and helium leak test)
- procedures for system operation

- operator orientation and training
- preoperational evaluation using checklists
- emergency team training
- fire department personnel training
- use of operation logbooks
- retention of construction, inspection, and system operation records.

During the incident, all of the facility control and safety shutdown systems in the facility, which had been operating to that point for two years, functioned normally. The first safety sensor to detect a problem and initiate automatic-shutdown procedures was the smoke detector (as expected for the location and type of failure involved). Shutdown and initiation of lithium drainage occurred according to expectation. A gas leak that developed in the cover-gas region of the reservoir tank during the incident created a cover-gas pressure imbalance, which caused lithium to be aspirated from the dump tank and through the open leak holes in the pump channel. (This condition was mitigated during the incident by venting the cover-gas system to 0 psig.) Other system response features which created undesirable conditions were (1) the smoke containment characteristics of the cell and (2) the intermittent inoperability of the scrubber pump motor. These malfunctions are discussed in more detail in Section III. Plans for assuring that these malfunctions will not have a high probability of occurring in a subsequent incident of the same type are discussed in Section VI and in Appendix E.

II. KEY FEATURES OF THE LPTL FACILITY THAT HAD AN IMPACT ON THE CONSEQUENCES OF THE OCTOBER 5, 1979 INCIDENT

A. Introduction

There are a number of features of the LPTL facility that had a direct bearing on several of the major consequences of the October 5, 1979 incident. These relate to the characteristics of the building area that houses the LPTL facility, the ventilation and scrubber systems for the LPTL cell, the fire control provisions in place at the time of the incident, and the status in understanding of failure modes and effects for the LPTL at the outset of operation. The information on these features summarized in this section is intended to serve as a preamble to the discussions of the causes and effects of the incident in Sections III and IV.

B. LPTL Deployment in Building 205

The LPTL facility is housed within a laboratory area designated H-126 (see Fig. II-1) in ANL's Building 205 (CEN Division). H-126 is a high bay area with a ceiling height of 60 ft and base dimensions of 30 by 60 ft. The area is fully enclosed on three sides, with a partial wall along the fourth side (west end) separating H-126 from a corridor. The separation wall extends

Fig. II-1. Schematic Drawing of Air Handling System and H-126 Laboratory in which the LPTL Facility Is Installed.

to a height of 15 ft. Within H-126 is a solid concrete structure surrounded by five steel platform levels and an external staircase. The concrete structure is divided into three elongated cell areas with common inner walls. All cell walls including partition walls are 18-in. thick and consist of Rebar high-density concrete. The lithium loop occupies the lower three levels of the center cell (cell 2), with the major vessels and tanks located on the first two levels.

C. Ventilation System for H-126

Originally, H-126 was planned and constructed as a single purpose (experimental), high-bay, engineering area with an air supply and exhaust system designed to serve this purpose. All fresh air enters H-126 at ceiling height and free flows down to all levels and areas. Initially all the air supplied plus a variable amount from adjacent corridors was discharged through the cell areas and into the H-126 exhaust system. The single purpose dedication of H-126 became obsolete with the termination of CEN's fuel reprocessing development efforts. In the interim years, the H-126 structure has been modestly reconstructed and transformed into multiple-use areas; and as a consequence, the exhaust ventilation system was altered over the years. Although the exhaust ventilation rate, 6000 cfm, has remained unchanged, two high static blowers were installed to sustain the required flow for longer periods of

time. Air from these two areas plus the cells passes through a wet scrubber box at the minimum rate of 6000 cfm. The scrubber was originally installed as part of a now defunct fluoride volatility program to protect against the possible dispersal of halogens to the atmosphere and was operated on a 24 hour a day basis. For the LPTL construction, cell equipment and containers associated with the fluoride volatility program were removed, and the scrubber was shut-down but remained intact.

At the first floor level, the center cell (cell 2), where the LPTL is housed, has double aluminum doors equipped with filters. The third level of cell 2 has a solid double door and the fifth level has a sliding door. All air entering cell 2 is pulled through these entrances. On the south part of the cells, there are large entrances, approximately three feet wide by six feet high, on each cell level with the exception of the second level. The main floor level has three entrances, one for each partitioned section; the third level has similar openings; the fourth has two and the fifth three openings. There is no direct air supplied to any of the cells. The direct air supplied to the periphery of H-126 is proportioned from a central system supplying air to three engineering areas, designated G, H, and J. The total air supplied from this system is 50,000 cfm, of which approximately 5500 cfm is direct air to H-126. However, the H-126 exhaust system is independent and consists of two 3000 cfm continuous operating blowers and one non-operating 3000 cfm blower as a standby. All air exhausted from the area is filtered through 14 high-efficiency particle (HEPA) filters before discharge from the building.

Preparatory to the installation of the lithium loop, cell 2 was isolated by blocking all intercell openings at each of the five levels. The interior cell structure was changed very little. At each level of cell 2, an open grating extends five feet into the cell, creating an open slot along the length of the north cell wall and thus allowing air to freely circulate through all five levels.

D. Scrubber System for Cell 2 of H-126

A scrubber system is used under emergency conditions for air exhausted from the LPTL cell and the remainder of H-126. In the event of a fire in the LPTL cell, the scrubber is automatically energized with actuation of either one of two photoelectric-type smoke detectors located near the cell 2 exhaust ducts. Manual operation of the system is also possible from a starter located at the scrubber pump. Once the pump is started by any means, only manual operation can shut the system down.

The scrubber is comprised of a spray chamber containing three plates with four orifices in each plate. A pressure nozzle is aligned to direct a water spray at each orifice. Baffles plus a demister pad located further down stream reduce water and particulate carryover through the ducts leading to HEPA filter in the fan loft.

The scrubber system is shown in Fig. II-2. The scrubber pump is a vertical drive unit capable of providing 100 gpm of water at 63 psi. The system piping is stainless steel except for the drain line from the scrubber which is black iron pipe. A system of valves and filters is provided for filtering the scrubber feedwater or for filter backwash. The scrubber is located in the

Fig. II-2. Scrubber System (PI = pressure indicator; F = filter)

area adjacent to the fifth level cells; and, when operated, water is recirculated between it and a surge tank located in a corridor on the second floor of H-wing. Normally, the system is operated with from 200 to 225 gal of water in the surge tank.

During the LPTL incident on October 5, 1979, the water recirculation was automatically started at 1240, when the smoke detector was activated, but shut off at 1249 due to a pump motor overload. Subsequent operation of the recirculation system until 1400 was in a manual mode, with a cycle that was estimated to be about 10 min operating and 8 min off to cool the motor overloads. After 1400, the system was shut down since the incident had been brought under control, and noxious fumes were no longer being generated.

Tests of the water circulation system were performed on October 15, 1979 (10 days after the incident). These tests revealed that, for efficient spraying of water from the jets in the scrubber, the throttling valve between the surge tank and pump had to be kept wide open. Furthermore, under these conditions, the current drawn by the pump (which had a 10 A rating) was 16.5 A; hence, the pump could only be operated about 55% of the total time, with the remainder of the time required for cooling of the pump overload to permit system restart.

The scrubber system had been installed during the late 1950's. At that time, it was used for continuous operation in the removal of halogen gases exhausted from nuclear fuel reprocessing experiments being conducted in the cells. This work continued until the mid-1960's when it was discontinued.

The scrubber had only been used sparingly since that time, primarily for effluents from other experiments (notably fluidized-bed combustor off-gases). In the five years prior to the construction/startup of the LPTL, the system had not been used, and preventive maintenance on the system was not performed. Upon installation of LPTL, the scrubber was cleaned, flushed, and placed in operating condition. The system was checked on a regular basis and was found to be in good working order for emergency operation in the event of a fire in the LPTL cell. Unfortunately, the years had taken their toll on the water pump through shaft and bearing corrosion and seal problems. When called upon to operate for an extended period of time during the incident, the pump overloaded due to the added load caused by the corroded bearings and shaft. The pump was capable of sustained operation since it had once been used in this manner. However, tests of the pump operation never lasted more than 5 minutes, and this short running time did not reveal the pump overload problem.

E. Fire Control Provisions for the LPTL Facility

Fire control requirements for the LPTL facility were established prior to initial start-up (Ref. 2 of this report). Extinguishants and equipment to control a 50 gal lithium spill were provided, including two 30 lb Lith-X ANSUL extinguishers and ten 45 lb containers of bulk Lith-X sealed in plastic liners to prevent contamination until needed in an emergency. The smoke detection system, described in Section I.D and in Ref. 2, provided a direct signal to the ANL Fire Department (ANL/FD). Prior to the initiation of LPTL operations, exercises in lithium fire extinguishing were held with ANL/FD personnel, during which procedures and extinguishant materials were tested.

F. Failure Modes and Effects Analysis for the LPTL

An analysis of plausible failure modes and effects for the LPTL facility was conducted by the LPTL design team during system design phases, preceding the final safety review prior to startup. The scope of these analyses covered perceived abnormal conditions that could occur in the as-designed facility. Documentation of the analyses is included in the LPTL Operations Manual.²

III. DESCRIPTION OF THE OCTOBER 5, 1979 INCIDENT

A. Introduction

This section describes details of events before, during, and after the October 5, 1979 incident, and contains various plausible scenarios for the incident that are believed to be consistent with the accumulated observations and reports. This information was collectively reviewed by the Incident Analysis Team.

B. Pre-Incident Conditions

On August 2, 1979, the LPTL was shut down for repairs. Up to that time, there had been 9544 accumulated hours of lithium circulation in the LPTL. The items to be repaired were (1) the inlet valve to the getter trap and (2) heaters on the reservoir tank. The bellows of the getter-trap inlet valve (a hand-operated Nupro valve) had failed on November 10, 1978, after being in

service for 3513 h. This failed valve was cut out, and a straight section of pipe was welded in as a replacement. The twelve stainless steel sheathed heaters on the reservoir tank had been failing one at a time during operation until only five were providing heat. Sheathed heaters from another manufacturer were used as replacements.

On October 4, 1979 at 1420, the LPTL was started and steady flow was established at a 2.1-mV (~ 0.4 gpm) reading for the main flow. The Variac setting for the EM-pump powerstat was 18% with a 5-A supply current. Temperature of the reservoir tank was stable at 396°C; temperature of the EM-pump channel was 300°C; and temperature of the piping was around 300°C, with the individual line heaters providing continuous heat to their respective piping segments. The power setting of the EM-pump was observed to have less of an effect on the flow-meter reading than had been observed during prior operation of the LPTL. Settings on the Variac from 18 to 35% produced the same reading of 2.1 mV on the main flow meter. When the pump powerstat setting was decreased below 18%, the flow rate signal also decreased. Table III-1 shows the lithium flow rates observed at various pump settings during early stages of LPTL operation and near the time of the October 5, 1979 incident. Within the first 100 h after initial startup, pump Variac settings of 18% and 22% produced flow-meter readings of ~ 6 and ~ 7 mV, respectively. After 8000 h of operation, Variac settings of 18% and 34% resulted in total flow-meter readings (main flow plus cold-trap flow) of ~ 5 and ~ 8 mV, respectively. Just before the failure, an 18% Variac setting produced a flow-meter signal of only 1.8 mV. This low flow-meter signal was evidence that a flow blockage was building up somewhere in the main flow circuit. (A flow-meter calibration is shown in Table III-2. Flow rates are given in terms of L/min, gal/min, and ft^3/min , and velocities in ft/s are given for 1/2- and 1-in. schedule 40 pipe.) By closing the main flow valve the flow rate could be stopped, but the valve only affected the flow rate near the closed position. Rotation of the handle for the main flow valve was checked to determine how the valve handle was functioning; it was found to rotate properly. A buzzing vibration could be felt near the EM-pump by touching the insulation on the piping near the pump. The pump Variac setting was left at 18% to keep the lowest power to the pump for a 2.1-mV flow-rate signal. It was thought that partial blockage at the main flow valve or at a cold spot in the piping may have occurred. Although the lithium flow rate was low, it was sufficient to equalize the temperatures in the piping system. If there was an insoluble partial plug at the valve or at a cold spot in the piping system, the total flow could be increased by establishing a bypass flow in the cold-trap piping. This bypass flow could be started by heating the cold trap in order to melt the lithium in that section of the system. On October 4, 1979 at 1600, the cold-trap bottom was wrapped with insulation to heat up the lower portion to the melting point of lithium and to establish lithium flow in that branch. This was the last operation performed on the LPTL on October 4, 1979.

C. Conditions During the Incident

During the morning of October 5, 1979, attempts were made to improve the lithium flow in the system. Two activities were undertaken: (1) addition of a second layer of insulation to the cold trap to hasten thawing of the lithium in the trap, and (2) surging of the main pump power as an attempt to break up the flow blockage. At 0900 the pump power was surged by alternately lowering

Table III-1. Comparison of EM-Pump Supply Current with Flow Rates at Start-Up and After 8000 h Operation

Date	LPTL Operating Time, h	EM-Pump Supply Current, A	EM-Pump Variac Setting, % of Powerstat Full Scale	Main Line Flow Rate, mV	Cold-Trap Line Flow Rate, mV	Temperature of Bulk Lithium, °C
12/28/77	0-start	4.5	17	3.8	---	318
01/11/78	3	5.0	18	6.3 ^a	---	360
	19	5.0	18	5.8	---	360
01/16/78	50	5.0	18	5.8	---	360
	56	6.0	22	7.0	---	390
06/08/79	8,224	5.0	18	5.5	2.3	350
	8,230	5.0	18 ^b	3.2	1.8	350
		5.5	34	5.5	2.3	350
10/04/79	9,544	5.0	18	2.1	---	396
10/05/79	9,566 ^c	5.0	18	1.8	---	396

^aInitial flow rate was indicated by a 6.3 mV signal, which dropped to 5.8 mV in 16 h.

^bFlow-meter leads were changed to a different recorder. When the leads were connected to the new recorder, the measured flow-rate signals had changed. By a slight increase in current, the flow-rate readings were increased to the previous flow-rate settings.

^cThis is the time of the incident.

Table III-2. Calibration of Main LPTL Flowmeter

Flow-Meter Signal, mV	Lithium Flow Rate			Lithium Flow Velocity, ft/s	
	L/min	gal/min	ft ³ /min	For 1/2 in. Pipe	For 1 in. Pipe
1	0.621	0.164	0.0219	0.173	0.0609
2	1.242	0.328	0.0439	0.346	0.122
3	1.863	0.492	0.0658	0.520	0.183
4	2.484	0.656	0.0877	0.693	0.244
5	3.105	0.820	0.111	0.866	0.305
6	3.726	0.984	0.132	1.04	0.365
7	4.347	1.148	0.154	1.21	0.426
8	4.968	1.312	0.176	1.39	0.487
9	5.589	1.477	1.208	1.56	0.548
10	6.210	1.641	0.219	1.73	0.609
11	6.831	1.80	0.241	1.90	0.670
12	7.452	1.968	0.263	2.08	0.731

and increasing the Variac setting several times. The lower and upper limits of these power surges were in the range from 10 to 30% of Variac full scale. The 0900 power surging met with little success. The flow increased from 1.8 to 1.9 mV. However, between 0930 and 1130, the flow declined smoothly from 1.9 to 1.3 mV. At 1130 the pump power was surged again by the same procedure used at 0900. Only moderate success was achieved again, with the flow increasing to ~ 1.7 mV by 1140.

The following paragraphs describe the sequence of events following the pump surge at 1130:

- 1130: Immediately following the pump surge, the temperature reading on thermocouple TR2-2, which was on the pump channel upper midsection (bottom side, down-stream end), began to rise rapidly (see Fig. III-1). Thermocouple TR2-2 was not connected to any high-temperature alarm interlocks; hence, no notification was possible and the operator was unaware of the condition.
- 1145: Thermocouple TR2-2 reached 680°C —a temperature rise of 350°C in 15 min. (The operator was still unaware of the condition.) Since the highest bulk lithium temperature in the system (in the reservoir tank) was 390°C , the higher temperature adjacent to the pump channel indicates that an exothermic reaction was occurring.
- 1145: The system operator entered the LPTL cell, lower level, to install more insulation on the cold trap. He remained in the cell until

Fig. III-1. Temperature in Area of Electromagnetic Pump Immediately before and during Incident.

approximately 1205, unaware of the rising temperature at the pump tube. He did not notice any irritant in the air or other evidence of an exothermic reaction. (Since the nose is a sensitive detector of alkali-metal smoke, it was concluded that little, if any, smoke was evolved during this phase of the incident.) The operator left the LPTL location at ~1205.

- 1155: TR2-2 showed a decrease in temperature from a high of 688°C.
- 1215: Thermocouple TR2-6 on the pump channel's lower midsection (bottom side, up-stream end, and lower end of slope of pump tube) began to increase rapidly in temperature. Thermocouple TR2-2 went off scale and presumably burned out shortly after this time. The smoke detector alarm was activated. This event resulted in: (1) notification of the fire department and central surveillance, (2) activation of a local alarm, (3) startup of the ventilation air scrubber, and (4) shutdown of all power to the LPTL except power to the temperature and flow recorders and indicators, the dump-valve operator, and the microprocessor. All these system shut-down features appear to have operated as designed.
- 1240 to
- 1315: The main lithium flow meter indicated a large spike at the instant of system shutdown (probably due to electrical noise generated by the instantaneous shutdown). Following this spike, the main flow-meter voltage fluctuated erratically, increasing to peaks of ~5 mV and ~6 mV at 1250 and 1300, respectively, as shown in Fig. III-2. (See Table III-2 for correspondence of meter voltage and flow rate.) The flow was very irregular but always large and positive during the entire incident (the positive direction is from the pump to the reservoir tank).
- 1255: Pump bus-bar temperatures (pump current core, midsection) increased rapidly and went off-scale, thereby indicating that either the lithium was reacting exothermically with the copper or the fire had spread to the region of the thermocouples.
- 1315: The operator reduced cover-gas pressure in the reservoir and dump tank to 0 psig.
- 1318: Leakage of lithium from the pump stopped, and the fire was considered under control. Also, the flow-meter signal dropped to zero at about this point in time, as shown in Fig. III-2.
- ~1430: The fire was essentially out, and the smoke had, for the most part, cleared out of the cell.
- 1440: The temperatures in the vicinity of the bottom of the reservoir tank and the reservoir outlet pipe began to rise (see Fig. III-3).
- 1505: The reservoir-tank bottom reached a maximum temperature of 410°C, then began declining.

Fig. III-2. Main Lithium Flow between Pump and Reservoir Tank.

Fig. III-3. Temperatures in the Lower Section of the Reservoir Tank.

1520: The temperature of the reservoir-tank outlet pipe reached a peak of 305°C, then began declining.

1520 to
~1800: The temperature of the reservoir-tank bottom remained constant at ~355°C, then began declining.

Further details of the fire fighting operation, including the incident summary report of the ANL Fire Department, appear in Appendix B.

D. Post-Incident Consequences and Recovery

1. Damage Assessment

The principal consequences of the incident in the LPTL facility were as follows: (1) The EM-pump and associated heaters, thermocouples support, and insulation were destroyed (see Fig. III-4). (2) The metal floor pan under the EM-pump as well as the wall linings around it were spattered and burned (see Fig. III-4). (3) There was a layer of dusty residue on all horizontal and vertical surfaces within the cell housing the LPTL (cell 2). The most notable effect of the incident (both during the fire and after its extinguishment) was an obvious lack of containment of the particulate and smoke generated from the fire. This was evident by the appearance of smoke in corridors and laboratories adjacent to the LPTL complex. It appeared that the copious quantities of particulate smoke and hot gases (generated by the fire and contributed to by the powdery fire suppressants applied to the fire) overwhelmed the exhaust system and escaped through all openings, including doorways, door sills, open seams in the ceiling area at the fifth level of cell 2, and other paths. In particular, an appreciable quantity of smoke was released through the first level doorway during the fire; this door was left open a large part of the time for fire-fighting access.

Examinations and tests of the ventilation system servicing the LPTL cell and adjacent areas revealed that (1) there was no direct air supply to cell 2; (2) the H-126 ventilation exhaust fans moved 6000 cfm through the scrubber with scrubber water flow on or off; and (3) a negative pressure of an estimated 0.05 in. of H₂O existed in the LPTL cell with the cell openings in their intended operational condition (there was no evidence that the cell openings were not in their intended operational condition during the incident, with the exception of the first level door which was open during most of the fire-fighting operation).

Fire-fighting efforts and thermal effects of the fire could have accounted for all particulate release from the cell. Movement of firemen in and out of the first level doorway plus the extreme turbulence associated with a fire centered at floor level about five feet from the doorway (three feet wide by five feet high) caused copious release of particulate. The fire heated the air rising in the cell to the high level exhausts. If the cell pressure was 0.05 in. H₂O negative at the floor level, a temperature increase of 60°C in the rising air would have caused a positive pressure in the cell relative to the outside at the same level at all levels above ten feet, thereby causing release of particulate through all openings at heights greater than ten feet above the cell floor.

a. **PUMP SECTION**

b. **FLOOR PAN**

Fig. III-4. LPTL Pump Channel Failure

2. Composition and Distribution of the Dispersed Particulate

Much of the dispersed particulate and residue from the fire that was deposited outside the LPTL cell had to be cleaned up immediately after the incident (within 72 h) to permit resumption of work in these areas. However, there were still sufficient quantities of the residue in localized areas to permit a survey analysis of the composition and approximate distribution. This survey was conducted during the week following the incident (the incident occurred on a Friday) and is summarized in Table III-3.

Table III-3. Results of the Analysis of Residue Samples Taken after the Lithium Fire in the LPTL Facility^a

Location	Area Sampled, ft ²	Density, g/ft ²	Composition ^b
Slag (floor of cell)	---	---	Graphite, LiOH-H ₂ O, Li ₂ CO ₃
LPTL Cell (1st level)	0.25	34	Graphite, NaCl
LPTL Cell (above EM-pump)	2	7	Graphite, Li ₂ CO ₃ , NaCl(vm) ^c
LPTL Cell (2nd level)	4	5	Graphite, NaCl(m) ^d , Li ₂ CO ₃ (vm), α-SiO ₂ (m)
LPTL Cell (3rd level)	4	4	Graphite, NaCl(m)
LPTL Cell (4th level)	11.4	5	Graphite, α-SiO ₂ (m), Li ₂ CO ₃ (vm)
LPTL Cell (5th level)	4.5	7	Graphite, Li ₂ CO ₃ (vm), NaCl(vm)
Glove-box roof (1st level)	8	0.7	α-SiO ₂
Instrument room (2nd level)	11	0.01	α-SiO ₂ (vm)
Ledge (4th level)	5	4	α-SiO ₂ (m)
Enclosure roof (4th level)	10	1.5	α-SiO ₂ , Graphite
Instrument box (6th level)	3	0.06	Graphite, Li ₂ CO ₃
Computer-room roof (7th level)	71	0.01	Graphite, Li ₂ CO ₃ (vm), NaCl(vm)

^a Samples collected by P. A. Finn and N. R. Chellew of ANL/CEN.

^b Outside cell samples were sometimes contaminated with large amounts of CaCO₃, CaMg(CO₃)₂, and CaSO₄·2H₂O produced by other experiment programs.

^c vm = very minor.

^d m = minor.

The information presented in Table III-3 indicates that a very high level of particulate residue was present in the cell area after the fire. The particulate consisted primarily of extinguishant materials, *i.e.*, graphite, NaCl and α -SiO₂. (The types and quantities of extinguishants used are listed in Table III-4.) In the slag sample from the cell floor, both LiOH·H₂O and Li₂CO₃ were found on the level farthest removed from the fire (the fifth level). The analyses of samples obtained outside the cell show that both fire extinguishant and lithium compounds were carried to all affected areas. The observation of a pH of ~11 (litmus) in samples from these areas indicates that additional basic material was present, probably LiOH·H₂O.

Table III-4. Fire Extinguishant Used on the LPTL Fire

Material	pH		Major Constituents
	(litmus)	(meter)	
Sand	5	8	α -SiO ₂
Lith-X	9	8	Graphite, LiCl
Metal-X	7	9	NaCl
Lith-3	6	7	NH ₄ Cl, α -SiO ₂ , vermiculite

The conclusions drawn from the survey are the following: (1) lithium reacted with both air and the constituents of the extinguishants to form numerous volatilized compounds (Li₃N, Li₂O, Li₂C₂, etc.), (2) much of the aerosol formed in the lithium fire consisted of very fine particulate which was carried a considerable distance from the fire; and (3) the aerosol contained not only lithium compounds but also the constituents of the extinguishant materials.

3. Analysis of LPTL Lithium

After the incident, several analyses were conducted to determine the condition of the LPTL lithium: (1) the quantities of lithium remaining in the dump tank and the reservoir tank were measured, (2) core samples were taken from the two tanks to measure impurity levels as a function of metal depth, and (3) visual observations were made of the reservoir-tank interior to determine the extent of the reaction product layer on the lithium surface. The results of these analyses are given below.

The lithium level in the dump tank was found to be just below the bottom of the drain pipe, *i.e.*, no more lithium could have been expelled from the dump tank. The lithium level in the reservoir tank was at the level of the bottom exit pipe. (The exit pipe protrudes ~4 in. above the bottom of the tank leaving a pool of ~5 gal when the tank is drained.) The impurity levels found in the two tanks are shown in Table III-5.

Table III-5. Impurity Levels in Lithium Core Samples from the Reservoir Tank and Dump Tank

Depth	Nitrogen Level, wppm	
	Reservoir Tank	Dump Tank
Surface	Pure Li_3N	6,000
3/4-in. Below Surface	5,000	1,000
1-1/2-in. Below Surface	5,000	1,000

Two small ports were opened at the top of the reservoir tank. A fiber-optics light source was inserted through one of the ports, while visual observations were made through the other. The lithium surface was found to be covered with a multi-colored layer of reaction products (predominantly lithium nitride of varying stoichiometry). Formation of Li_3N on the lithium surface is probably related to the temperature rise observed in the reservoir tank between 1430 and 1530 h (see Fig. III-3). A photograph was taken through the port and enlarged for closer examination (see Fig. III-5). The surface layer was disturbed by scraping with a metal tube; it was found to be fairly thin and easily broken and was moved aside. Beneath this layer (of perhaps 1-2 mm thickness) was bright, metallic lithium.

E. Evaluation of the Pre-Incident, Incident, and Post-Incident Observations

In this section, a plausible scenario has been proposed for the conditions and progression of events that led to and occurred during the incident. This scenario, although speculative and somewhat inconclusive, is supported in large part by the observations drawn from the previously given results. For convenience of discussion, the scenario is divided into four phases: (1) pre-incident, (2) leak initiation, (3) lithium leak and fire, and (4) post-incident.

1. Pre-Incident Phase

For this phase, two scenarios (designated A and B) have been proposed; scenario A is considered to be the most likely.

Scenario A. The nitrogen concentration was quite high in the lithium of the LPTL during its start-up prior to the incident (~1,300 wppm). This high nitrogen level could have two effects on the pump tube: (1) lithium nitride could precipitate in the cool region adjacent to the pump bus bars, causing an increasing flow restriction, and (2) the high nitrogen activity could accelerate corrosion of the pump tube along the edges of the flattened pump tube. Residual stresses from the tube-forming operation, coupled with possible thermal stresses, would make the stainless steel pump tube more susceptible to attack by the high-nitrogen lithium. This attack may have been progressing for many hours during previous loop operation as well as during the October 4, 1979 start-up operation. Stress corrosion cracks may have formed partway through the tube wall in the highly deformed region.

Fig. III-5. Lithium Surface in Reservoir Six Months or More after Incident.

Scenario B. The high nitrogen concentration in the LPTL lithium could have resulted in precipitation of lithium nitride in the cool region adjacent to the pump bus bars. The precipitated lithium nitride could create a flow restriction which, in turn, would have caused the pump to cavitate

(supported by the vibration noted by the operator prior to the incident). The cavitation and associated turbulence would then have reduced the diffusion boundary layer at the pump tube wall. The reduction in the diffusion layer would have accelerated the corrosion rate along grain boundaries in this region.

2. Leak Initiation

The pump surge performed at 1130 may have provided the final stress that caused the weakened pump tube to fail. The tube could already have been cracked most of the way through the wall and the extra pressure caused by surging the pump power might have been sufficient to open up a small lithium leak. Once outside the tube wall, the lithium had ample materials with which to react (including air and thermal insulation). These reactions would have occurred at a relatively low temperature at first since the leak was small, and thus might not emit detectable smoke (smoke would be expected if heat were vaporizing the reactive compounds, thereby causing a vapor-phase reaction instead of a liquid-gas or solid-gas reaction). The reaction products (aided by increasing heat input from the reaction) would have very rapidly corroded the pump tube, thus enlarging the leak path. This progression of events is supported by the successive rise in temperatures on the pump tube adjacent to the leak (Fig. III-1). As the temperatures continued to rise, sufficient smoke was emitted to activate the smoke alarm.

3. Lithium Leak and Fire

When the smoke alarm was activated, all power to the loop, including the pump and heater, was shut down, with the exception of (1) the dump valve operator and line heater, (2) temperature and flow recorders and indicators, and (3) the microprocessor. The dump valve was opened and the lithium began to flow into the dump tank. At some time during the fire, enough lithium entered the dump tank to cause an indication on the level probe. (Post-incident measurements indicate that a level rise of only ~1-2-in. would be required to activate the level probe. We cannot say, therefore, whether more than 5 gal of lithium entered the dump tank at any time during the incident.)

By the time the smoke detector was activated, the fire was well under way. Apparently, within five minutes of the smoke detector activation, the plastic face of a differential pressure gauge near the reservoir tank melted and deformed. Since the plastic face was part of the system pressure boundary, the pressure in the reservoir tank was released to near atmospheric pressure. Argon was still flowing through the reservoir tank, but the reservoir tank pressure was probably not much more than that associated with the pressure drop through the few feet of 1/4-in. tubing connecting the differential pressure gauge to the reservoir. (It is apparent from this experience that a plastic-face pressure gauge should not be part of the pressure boundary in an alkali-metal system, even though it may be located far from the high-temperature region. At least, careful consideration should be given to the possibility of such materials becoming overheated in the event of a liquid-metal fire.) This imbalance in pressure could have caused lithium in the dump tank (at 4 psig) to be forced upward into the system and past the leak point shown in Fig. III-6, with some flow actually being forced upwards toward the reservoir tank. When the dump tank was empty, gas may have begun to flow up

Fig. III-6. Postulated Hydraulic Diagram of LPTL during the Incident.

the dump line, through the horizontal line to the pump and out the leak. As the gas passed the tee, it would tend to pick up intermittent slugs of lithium coming down from the reservoir tank. These slugs of lithium would be accelerated to a high velocity by the 4 psig pressure (supplied to the dump tank) operating against atmospheric pressure at the leak location. Some of the lithium came out of the leak; however, a significant fraction could have been carried past the leak location where it impacted the column of lithium leading up to the reservoir tank. The continual impacting of these slugs of lithium at the base of the column would have caused an irregular but positive flow of lithium from the pump region up into the reservoir tank. (At 1318, after the argon pressure was manually vented, it was noted that the lithium leak had stopped. The venting also coincided with cessation of flow through the main flow meter. However, this cessation may have been a coincidence, because post-test examination showed that all the lithium that could be expelled from the system already had been, *i.e.*, the lithium leakage may have stopped because none of the remaining lithium in the system was in a location from which it could be aspirated to the leak.)

Several other explanations for the large positive indication on the flow meter during the fire have been considered. Some of these explanations are: (1) electrical noise, (2) thermal emfs, (3) gas bubbles travelling through a more or less stagnant lithium region, and (4) the pump still effectively being on. These explanations were rejected for the following reasons.

(1) Little or no electrical noise would be expected because essentially all the system power was turned off during this period. An electrical noise spike can be observed at the instant of system shutdown at 1240; however, in the following period, the flow meter should show no influence from electrical fields, particularly of the magnitude observed. (2) Thermal emfs could explain the magnitude of the signals observed; however, thermocouples located near the flow meter registered a temperature rise of $<10^{\circ}\text{C}$. A temperature rise of several hundred degrees would be required to account for the signals observed. (3) Oscillation of the lithium column or passage of gas bubbles through a rather stagnant lithium region would result in alternating positive and negative flow-meter signals. The signals that were observed oscillated about a strongly positive voltage, rather than about zero. (4) The pump could not have remained on with line power off. Other events such as opening of the dump valve and turning off the heaters could not have occurred without the pump also having been turned off. In any event, the pump would have stopped operating at about 1300 because at that point it was severely damaged by the fire.

The conclusion of all this is that (1) a positive, irregular lithium flow existed from the pump region toward the reservoir tank during the entire time period of the fire, and that (2) the lithium was not moved by the EM-pump but was probably moved by an aspiration-type process involving differential gas pressure.

4. Post-Incident Phase

At ~1440, about an hour and twenty minutes after the lithium leak had stopped, temperature rises were observed in the reservoir tank and in the outlet pipe at the bottom of the reservoir tank (Fig. III-3). These temperatures reached levels greater than pre-incident operating levels (410°C maximum). Also, instead of cooling down slowly as would be expected, the reservoir-tank temperature remained constant at 355°C for about three more hours before it began to cool.

These observations indicate that a slow, exothermic reaction was occurring in the reservoir tank well after the external fire had been extinguished. It is probable that air was leaking in through the failed pressure gauge. Since oxygen, nitrogen and moisture all react readily with lithium, there is no built-in mechanism for limiting the air-leak reaction. A protective layer may have accumulated on the lithium pool surface at the base of the reservoir (*i.e.*, at the level of the exit pipe penetration) to stop the reaction before all the lithium was consumed.

IV. METALLURGICAL AND CHEMICAL ANALYSES OF THE FAILED EM-PUMP CHANNEL

A. Preface

The results of the metallurgical and chemical (M&C) analyses performed on the failed pump channel after its excision from the LPTL are discussed in this section. The bulk of the metallurgical and analytical work reported here was done within, and by members of, ANL/CEN. However, selected samples were sent to five outside organizations that expressed a desire to participate in the

pump channel analysis. Although most of the analysis results presented in this report are those obtained by ANL/CEN, several key findings by the outside organizations are also cited to add clarity and reinforcement. It is planned that a more detailed report of the combined findings by all participating organizations will be published after all the analytical efforts have been completed and the results reported to ANL.

B. Approach to the ANL/CEN Analyses

The pump channel was excised from the LPTL and subjected to some abrasive descaling to remove most of the cakey residue adhering to the outside surfaces. A picture of the channel following the descaling operation is shown in Fig. IV-1. An enlarged view of the region where the leak occurred is presented in Fig. IV-2. Prior to beginning the M&C analysis, an analysis plan was written and approved by the LPTL Incident Analysis Team (see Appendix C). Preparatory to sectioning the channel, its ends were sanded smooth. Momentary sparking and flaming was observed in the process, indicating the presence of unreacted lithium adhering to the inner walls of the channel. In order to prevent any further loss of information beyond that which may have occurred in the interim period between the LPTL fire and the start of the M&C analysis, specimen-sectioning cuts were made on a Buehler cut-off wheel using Freon TF as a lubricant. (Water was not used during any of the cutting to avoid hydrolysis of lithium-containing residue on the surfaces and in the grains of the channel.) For the most part, this procedure appeared to preserve the integrity of the normally hydrolyzable phases present in some sections of the pump channel as discussed below.

Fig. IV-1. Photograph of Failed EM-Pump Channel
Excised from LPTL.

Fig. IV-2. Expanded View of Region of EM-Pump Channel Where Leaks Developed.

As per the analysis plan for the pump channel (see Appendix C), Sections 10, 11, 12, and 16 of the channel (see Fig. IV-1) were the first ones prepared for examination. Appendix D summarizes the manner in which some samples were dispensed to other laboratories for additional examination. The principal analytical tools used in these analyses were the scanning electron microscope (SEM) with energy dispersive X-ray analysis (EDAX), the ion microprobe mass analyzer (IMMA), X-ray diffractometry (X-RAY), and wet-chemical analysis (CA).

C. Results of ANL/CEN Analyses

The most notable observation of the ANL/CEN analyses was the finding of rather extensive solid plugs of nonconductive material in Sections 5 and 11 (refer to Fig. 3 of Appendix D). As such, these plugs were in the two regions of highest magnetic and electrical field during EM-pump operation, since they are located at the two positions along the channel where the current-carrying bus bars and the magnet pole pieces of the first and second pump stages butt to the channel. Also, these plugs occur at the point of greatest heat loss in the pump, since the brazed-on bus bars and, to a certain extent, the magnet pole pieces conduct heat away from the channel at an appreciable rate. Resolution of whether these plugs occurred as a result of electromagnetic effects, pseudo-cold trap effects, or the fire itself appeared to represent a key to determining the causes and effects of the incident. While other plugs were found in regions of the pump channel and adjacent piping, these plugs were mostly solid lithium and were significantly different in character from the two in Sections 5 and 11.

An extensive analysis was conducted on the plug in Section 11 and on specimens of the pump channel itself taken from Section 11 and adjacent sections. Although, as was mentioned above, this plug was nonconductive electrically, it

did contain appreciable quantities of ferromagnetic material. Visual observation of the plug in cross section (see Fig. IV-3) reveals two dominant phase regions and several other minor (in area) regions of distinguishably different texture. It is believed that many features of the chemistry of these regions were preserved by the procedure of polishing and mounting the specimen in a helium dry box, using nonaqueous lubricants.

Fig. IV-3. Cross-Section View of Plug Region in Section 11 of the Failed LPTL Pump Channel.

Among the more interesting areas of apparent homogeneity in Fig. IV-3 is a dark loop-shaped band $\sim 0.3\text{-mm}$ thick, which encircles a region of crystals with very large grain size. The dark band appeared to be composed primarily of chromium (SEM/EDAX), with carbon being a major non-metallic constituent (IMMA); and the encircled region was found to be nearly pure Li_3N (X-RAY, CA). It seems from the view in Fig. IV-3 that the dark loop-shaped band (Cr-C band) had at one time been contiguously adherent to the entire inner circumference of the channel and that it may have been partially detached prior to or during the fire. The region outside the band is a mixture of phases (mostly ceramic in nature), with iron-rich and nickel-rich inclusions (SEM/EDAX). The section of channel wall not contacted by the Cr-C band is severely eroded on both the inner and outer surfaces, but it is suspected that most of this erosion is due to the fire and the accompanying high temperatures. In all, $\sim 40\%$ of the original pipe wall has been eroded away at the thinnest point. The principal elemental and crystalline materials found in the plug in Fig. IV-3 and in adjacent regions are summarized below.

Nitrogen, Carbon, and Oxygen: Lithium nitride, Li_3N , is a major phase present throughout the plug region in Fig. IV-3; this was confirmed by both chemical analysis and X-ray diffraction. The large-grain crystalline region bounded by the Cr-C band is essentially all Li_3N . This bounded region occupies roughly 25% of the cross section of the plug. The primary constituents of the "heterogenous region" outside the Cr-C band are lithium, oxygen, and hydrogen which have reacted together to form a ceramic matrix of unresolvable composition (by X-RAY). It is not clear whether the actual composition of this region evolved during the fire or as a result of attack by moist air after the fire had been extinguished.

Another informative discovery involved a reddish crystalline material scrapped from the interior surface of the pipe in Section 15. This material was found to be mostly Li_2NCN (X-RAY), a compound which is known to crystallize from lithium containing near-saturation levels of nitrogen and carbon. At the time of the maintenance shutdown in August of 1979, the LPTL was saturated with carbon at a fairly low cold-trap temperature ($\sim 200^\circ\text{C}$), and there was evidence from a filtered sample taken out of the LPTL reservoir tank that the nitrogen level was higher than the normal value of 900 wppm. This condition at the August 1979 shutdown may have contributed to the plug formations in Sections 5 and 11 and to the startup difficulties encountered in October of 1979 (see Section III-B).

As a follow-up to the concern about high nitrogen in the LPTL during the October 1979 start-up, samples of lithium from the piping immediately downstream of the EM-pump were analyzed for nitrogen. The bulk lithium in this pipe section, after being cored from the pipe, was found to contain ~ 900 wppm of nitrogen by the micro-Kjeldahl method (the normal value measured in the LPTL during its later stages of operation); but in cases where uncored samples (pipe plus lithium) were analyzed, the equivalent nitrogen level in the lithium was ~ 5000 wppm. There appear to be two plausible explanations for this finding: (1) Li_3N and/or other hydrolyzable nitrides were in solution during LPTL operation but precipitated and settled against the pipe wall during cooling of the lithium, or (2) an insoluble surface layer of ternary nitrides (e.g., one containing structural material constituents) built up during LPTL operation. (Some evidence will be presented in the following text to support at least the existence of such ternary nitrides.) Interpreting the nitrogen-related data is somewhat complicated by our inability to determine how much nitrogen was introduced to the system during the leak and fire and how much was actually present at the time of the October 4, 1979 startup.

Iron, Chromium, and Nickel: Finely divided deposits of iron, chromium, and nickel were found in relatively high abundance throughout the region of the plug. The heterogeneous region outside the Cr-C band contained iron (~ 25 wt%, CA), chromium, and nickel in approximately the same weight ratio as is present in 304 stainless steel (72:18:9). Considering the region of the plug as a whole, the iron appeared to be present in at least four forms: (1) ferrite (most probably from the ferrite layer that normally forms at stainless steel/lithium interfaces), (2) iron-rich needle-like hexagonal crystals of nearly pure iron 0.1 to 0.2 mm in length, (3) multifaceted plate-like crystals up to 0.4 mm across (see Figs. IV-4 and IV-5) composed of 86-93 wt % Fe, 7-14 wt % Ni, and 0 to 1 wt % MN (EDAX), and (4) discreet, uniformly sized spheres of iron (~ 15 μm in diameter) present primarily in the heterogenous phase.

SEM PHOTO

X-RAY SCAN (Fe)

Fig. IV-4. Iron Crystals from LPTL Pump Channel.

In addition to the Cr-C band described previously, ruby red rod-like crystals (~100 to 200 μm in length, see Fig. IV-6), which were composed mainly of lithium (X-RAY), chromium (SEM/EDAX), and nitrogen (X-RAY), were plucked from a specimen taken from Section 12 of the pump channel. They were shown not to be Cr_2N or CrN ; and, based on comparative X-ray diffraction analyses, they appeared to be isostructural with compounds of the type Li_xMn_y , where M could be chromium. These crystals were readily hydrolyzable, but there was not enough material collected to examine the hydrolysis products.

In a series of independent experiments conducted at ANL by W. F. Calaway, it was found that iron, chromium and nickel concentrations in lithium under LPTL operating conditions (200 to 500°C and 500 to 1000 wppm nitrogen) can range from 10 to 60 wppm. In these experiments, the source of iron, chromium and nickel was stainless steel; hence, the equilibrium activities of each metal are likely to be much the same as existed in the LPTL at the time of the failure. Calaway's work further showed that nitrogen was an important factor affecting chromium dissolution, particularly at the higher end of the temperature range (*i.e.*, toward 500°C), where Cr^{6+} was the predominate chromium species (implying species of the type Li_xCrN_y), whereas Cr^0 predominated at lower temperatures. The iron and nickel solubilities also seemed to be affected by nitrogen activity, but no definitive evidence concerning oxidation states was derived. The results of this work are summarized in Fig. IV-7.

D. Metallurgical Condition of the EM-Pump Channel

Despite careful examination of the EM-pump by visual inspection as well as radiography and micrometallurgical methods, it has not been possible to

Fig. IV-5. Modifications of Iron Crystallites Found in the LPTL Pump Channel.

Fig. IV-6. SEM/EDAX Study of Ruby-Red Crystals Found in LPTL Pipe Section

draw any definite conclusions regarding the specific cause(s) of the failure that resulted in the leak and fire of October 5, 1979. Most of the scorched, scaly, and crusted residue on the inner and outer surfaces of the channel has been attributed to the fire. The Cr-C band in Fig. IV-3, the plug region bounded by it, and the channel surfaces to which it is still adherent are presumed to have been much less affected by the fire than the heterogenous region of the plug and the channel surfaces in contact with it. The only sections of the channel that showed signs of severe thinning were those in direct contact with the heterogenous plug material. Other sections of the channel, including those near the leak holes, exhibited much less evidence of thinning; and it was generally concluded that all observable thinning was due to corrosion which took place during the fire, not before.

Along the inner channel surfaces in Fig. IV-3, in the region where the Cr-C band had become detached from the wall, there appears to be high residual stress in the surface of the structural metal as evidenced by a series of uniformly spaced, optically distinguishable layers of stainless steel. This layering is not present along the surfaces of the channel that are still bonded to the Cr-C band and is believed to be a result of the fire incident.

Fig. IV-7. Concentrations of Dissolved Fe, Ni, Cr, and N in Lithium Samples Equilibrated with Stainless Steel

E. Analyses by Outside Organizations

Because of community-wide interest among lithium experimentalists in the events, causes, and consequences surrounding the LPTL pump channel failure, a selected set of specimens from the pump channel and from nearby sections of piping were forwarded to members of other organizations (per their request and/or concurrence) for supplementary analysis. Their efforts were also expected to bolster the information input to the pump channel analysis. The organizations and specific individuals receiving samples were:

- Oak Ridge National Laboratory (J. H. DeVan)
- ETEC, Canoga Park, CA (N. J. Hoffman)
- Colorado School of Mines (D. L. Olsen)
- Hanford Engineering Development Laboratory (W. F. Brehm)
- Westinghouse Research Center (M. G. Down)

The specific instructions transmitted to these organizations are given in Appendix D. At the time of writing of this document, most of these organizations had started, but not completed, their intended analyses of the LPTL specimens; hence, a complete report of their findings is not yet available. However, some early input from these other organizations has helped to confirm and augment several aspects of the CEN-conducted analyses. The preliminary reports from the other participating organizations are discussed below.

Oak Ridge National Laboratory (ORNL): Workers at ORNL have conducted preliminary optical metallographic examinations of metallic specimens from Sections 11 and 16. They found the normal type of corrosive attack on the lithium-exposed surface of each specimen, as described in Section I.E. of this report. The specimen from Section 16 which ORNL received was one that was not contacted by the fire in the pump channel area. This specimen included a pipe-section fabrication weld made by an outside vendor (Atomic Welding, Inc.). The ORNL investigators found several cracks (indicating a lack of complete fusion) along the inner surface of the root pass of this weld; these cracks would give cause for weld rejection if appropriate ASME code qualifications were applied. ORNL also found several voids in the root pass of this weld, which showed evidence of having been infiltrated and attacked by lithium. These voids are believed to have been present in the original weld and contiguous with the surface cracks.

As a follow-up to this finding by ORNL, seven pipe-section welds in the vicinity of the lower level on-line hydrogen meter (three welds produced by ANL in the field, and 4 welds produced by Atomic Welding, Inc. in section fabrication) were removed from the loop and prepared for radiographic and microscopic examination. No other evidence of serious weld faults was found.

ETEC: Workers at ETEC examined the grain structure and microhardness characteristics of specimens from Sections 10 and 12 of the pump channel. They confirmed the findings of the CEN-conducted analyses which showed sharply delineated regions of large and small grain size. [It has not been clear from any of the analytical work done on the pump channel whether the observed grain structure (1) is the same as it was in the as-received channel, (2) was reordered during operation of the LPTL (doubtful), or (3) is in its present state as a result of the fire.] The ETEC microhardness data, although only of semi-quantitative value at this time, do indicate that some sensitization of the channel material may have occurred during the fire. This is evidenced by the presence of recrystallized regions with a high frequency of KNOOP hardness in excess of 200 in the ETEC specimen from Section 10.

Colorado School of Mines (CSM): Investigators at CSM are using optical metallography on etched and unetched mounted specimens from Sections 12 and 16 of the pump channel. The key findings of their work to date are: (1) the thick inner and outer ceramic corrosion layer surfaces of Section 12 (similar to those shown in Fig. IV-2 for Section 11) were formed rapidly, most probably during the fire, (2) there was residual cold work in the bend region of the pump channel (confirming the findings of other groups), and (3) there was no evidence of flaws in the weld in the CSM half of Section 16 (see comments on ORNL findings above).

Hanford Engineering Development Laboratory (HEDL): Investigators at HEDL received samples from Sections 10 and 11 of the pump channel. At the time of writing of this document, they were performing optical microscopy and were planning ion and electron microprobe studies. They reported that they were using water-free cutting methods similar to the ones employed in the ANL/CEN analyses so that hydrolysis of lithium-bearing species would be avoided. They had found (1) Fe-Si and Fe-Ni and pure Cr deposits in the interior surface regions of their pump channel specimens (confirmed in ANL/CEN findings) and (2) some optical activity in a number of the surface layers examined, perhaps indicating the presence of oriented ceramic crystallites.

Westinghouse Research Center (WRC): Workers at WRC received samples from Sections 11 and 12 of the pump channel. Their plans for analysis of these specimens were to conduct X-ray diffraction and secondary ion mass spectrometry which focused on the low-Z element inventory and chemical composition of the plug material that developed in the vicinity of the EM-pump bus bars. Results from these studies should provide an important supplementary set of information on the plug material.

V. POST-INCIDENT TESTS OF LITHIUM FIRE EXTINGUISHING AGENTS

A. Preface

The airborne residue produced by the lithium fire and the extinguishing process (see Section III.D.2) created what proved to be the most costly and troublesome aspect of post-incident recovery. Analysis of samples of the residue collected from corridors and laboratories adjacent to the LPTL cell showed the presence not only of lithium compounds (mostly hydroxides and carbonates), but also constituents of the various extinguishing agents used to suppress the fire. (Typical findings were listed in Table III-3.) From an assessment of the consequences of this residue, it seemed advantageous to future LPTL operations that methods be examined for minimizing (1) the amount of smoke produced during fire suppression and (2) the length of time required to effect complete extinguishment. To this end, a series of tests was planned by the subgroup specifically assigned to the analysis of fire extinguishant performance (see Section I.B); these tests were carried out with the assistance of the ANL Fire Department. The nature of these tests is described below.

B. Description of Tests

The test consisted of dispersing selected liquid-metal fire extinguishing agents over the surface of pre-ignited one-pound (1 lb) blocks of lithium metal. The criteria for selection of the extinguishing agents were that (1) the agent not contain halogen salts, (2) the agent not contain oxides or salts of other metals (e.g., sodium, magnesium) that might react with molten lithium to free those other metals and create an environment for their combustion in air, and (3) the agent be one which has been used successfully in liquid-metal fire extinguishing applications. On this basis, the following six materials were selected and procured for testing:

- (1) Lith-X (Graphite-LiCl mixture)
- (2) Graphite Microspheres
- (3) G-1 Powder (Pyrolytic graphite)
- (4) Lith-3 Powder (NH_4Cl - αSiO_2 -vermiculite mixture)
- (5) Dry Silica Sand
- (6) Lithium Carbonate (Li_2CO_3)

The tests were conducted in an all-metal ventilated scrubber hood in ANL Building D-206. Each lithium ingot (1-lb size) was placed in an iron pan, and the pan was set on a bed of sand contained in a stainless steel tray. The ingot was ignited with an acetylene torch and allowed to burn until the lithium was completely melted and the surface of the lithium pool was at least half covered with the familiar coral-like glow indicative of burning lithium. Each agent was applied by sprinkling it uniformly over the burning surface until the fire was sufficiently suppressed that no fumes or flames were visible. In cases where small burn-throughs occurred following complete coverage of the surface of the pool, small amounts of agent were reapplied to those spots.

The key results of the tests are summarized in Table V-1. The graphite microspheres appeared to have the best fire suppressant characteristics in that (1) little or no smoke evolved during or after their application and (2) less material was required to achieve levels of extinguishment comparable to those of the other suppressants tested. The performance of Lith-X was only slightly inferior to that of the graphite microspheres. Pyrene G-1, Lith-3, and Li_2CO_3 performed poorly in terms of smoke generation, fire suppression rate, and frequency of burn-through after complete coverage. In particular, Lith-3 and Li_2CO_3 appeared to be reactive with the burning lithium. The dry silica sand (test results not shown in Table V-1) was also reactive, with much popping, cracking, and sparking after application directly to the areas of burning lithium. However, the sand was effective as a diking material around the edges of the burning lithium pool.

C. Discussion

The mechanism of combustion in a lithium fire has been determined and characterized in previous studies.^{8,12} In particular, the most recent work done at HEDL^{8,9} describes detailed studies of the burning of lithium in controlled environments and in the presence of other combustible materials. Specific conclusions from these studies, in the context of their comparison with liquid-sodium fire experience, are given below:

- Both sodium and lithium react in air. The lithium-air reaction occurs more rapidly and produces higher reaction temperatures. Both are inert in argon; lithium reacts with nitrogen, while sodium does not. Techniques exist which conservatively predict the temperature and pressure from both a sodium pool and spray-fire source in an air atmosphere.
- Both sodium and lithium react exothermically with concrete; however, lithium-concrete reactions are more energetic than sodium-concrete reactions. It is prudent to avoid lithium or sodium contact with concrete by designed features such as steel liners.
- Reactions from shallow sodium or lithium pools in an air atmosphere can be extinguished by powder applications. However, inert gas flooding is most effective in controlling medium-to-large spills. Catch pans are helpful in containing a liquid metal spill and may provide a closed system for inert gas flooding.

Table V-1. Results of Lithium Fire Suppressant Tests Conducted in Conjunction with LPTL Failure Analysis

Test Number ^a	Agent Tested	Applications Time, min	Weight of Agent Applied, lb	Notes
1	Lith-X	4	~3.3	Slow solidification.
2	Lith-X	13	~3	Rapid solidification.
3	Graphite Microspheres	2	~1	Very little smoke during and after application.
4	Graphite Microspheres	1	~1.5	Same as Test No. 3.
5	Pyrene G-1	6	~3	Much black smoke during and after application.
6	Pyrene G-1	2	~5	Same as Test No. 5.
7	Lith-3	3	~1	Numerous burn-throughs. Evidence of reaction with agent.
8	Lith-3	—	~2.7	Same as Test No. 7.
9	- - - - - VOID - - - - -			
10	Li ₂ CO ₃	—	~2	Yellow flames indicated reaction. Fire was not suppressed.

^aAir flow in scrubber = 6000 cfm. All test used 1-lb lithium ingot.

- Aerosols produced by liquid-metal spills can be controlled in air cleaning systems with collection efficiencies greater than 99.9%. Very large mass loadings are possible with large collection efficiencies in an aqueous scrubber system.

These conclusions, which were made independently by the HEDL workers, are in agreement with the consequences of the LPTL fire. In addition, HEDL recommended (1) the implementation of a catch pan and an aqueous scrubber system and (2) the use of powder extinguishants; these are already used in the LPTL. Also, the finding that the molten lithium did penetrate and react with the concrete under the floor after breaking through the bottom of the stainless steel catch-pan is understandable in light of the work of the HEDL investigators. [The breaching of the catch-pan is not viewed as a normal occurrence, but is not totally surprising either, considering the temperatures reached in the burning pool (>1000°C) and the fact that the floor pan was made from relatively thin (1/16 in.) steel sheet.]

The findings with respect to the dispersed aerosol from the LPTL fire are also understandable on the basis of existing knowledge of lithium fire characteristics. In the tests described in Refs. 8 to 11 and in Section B above, liquid lithium is found to react exothermically both with constituents of air (oxygen and nitrogen) and with moisture, forming an aerosol that is white and finely dispersed. This aerosol is predominantly Li_2O for ignition in air, but converts to the carbonate and hydroxide either while airborne or after settling. The reaction products that remain at the reaction site are usually found to consist mainly of equal parts lithium nitride and lithium oxide. The lithium pool temperatures can reach 1040°C , and flame temperatures up to 1260°C have been measured. (At these temperatures, the density of lithium is approximately one-third that of water.) The flame or fire zone is usually confined to a porous, coral-like growth of lithium nitride which floats on the liquid lithium metal and acts as a catalyst and a wick. The spread of flame across the liquid metal is normally not rapid.

To expeditiously extinguish a lithium fire, the fire zone present at the lithium nitride wick should be coated with an inert blanket of extinguishant. The bulk molten metal should be cooled as quickly as possible to prevent further outbreaks of fire; hence, the desirable characteristics of a fire extinguishant would be one that allows the extinguishant to float on the lithium surface and permit rapid cooling of the lithium pool. It is for this reason that dry inert powders have been employed in liquid-lithium fire suppressant applications. Also, because of the low density of lithium at high temperatures, the technique of applying the extinguishing agents is as important as the correct selection of extinguishant for the particular conditions of the fire. In particular, heavy rapid additions prove to be less efficient in suppressing a fire than lightly applied uniform coatings.

VI. CONCLUSIONS AND RECOMMENDATIONS

The principal conclusions of this analysis of the component failure and accompanying lithium leak and fire that occurred in the LPTL facility on October 5, 1979 can be summarized as follows:

- There is strong evidence for a direct connection between high residual stress and structural failure in liquid lithium systems constructed from 300-series stainless steel. The rate of intergranular penetration is much greater in regions where the steel exhibits residual stress from forming operations, such as are performed on EM-pump channels of the type that failed on the LPTL.
- The LPTL pump-channel failure could have been brought about by a number of factors, as discussed in Section III.D. The most likely cause could have been related to a combination of (1) excessive local corrosion in the heavily cold-worked region of the pump channel, (2) other erosive effects along the pump channel due to corrosion product slugs in the vicinity of the bus bars that restricted lithium flow, and (3) manual surging of the pump current (during the startup phase just before the failure), which further exacerbated the stresses in the channel.

- The severity of the LPTL incident would have been greatly reduced had there not been a simultaneous failure of the differential pressure device (DPD) on the reservoir tank. Alternatively, if the cover-gas pressure control system had been designed so that the cover-gas regions in the reservoir and dump tanks were both vented to atmospheric pressure at the time the dump valve was activated, very little lithium would have been lost through the leak in the pump channel, despite the failure of the DPD.
- The amount of airborne soot from the fire suppressant would have been reduced considerably if (1) the LPTL cell was more tightly sealed, particularly at the top of the third level and around the third-level doors and (2) graphite microspheres were used as the fire suppressant instead of the other extinguishants that were actually employed.
- The response of the LPTL safety systems and the ANL Fire Department to the alarms was nearly perfect. Even considering the problem of the excessive leakage caused by the DPD failure, the entire incident was handled skillfully and effectively by all parties involved at the time of the incident. There were no injuries to personnel and damage to adjacent experimental areas and to the LPTL itself was minimal. The entire cost of recovery from the incident, including repairs to the LPTL was under \$25,000.

The key recommendations derived from the results of this analysis, from prior experience with lithium loops, and from a number of other independent studies related to the handling and use of liquid lithium may be stated as follows:

- There is a need for improved understanding of the impact of stress, strain, and fatigue on the response of components in a lithium environment, including the effects of impurities (O, N, and C in particular).
- It would be beneficial to the operator of lithium loop systems to have functional, real-time on-line monitors for nitrogen, oxygen, hydrogen, and carbon. The need for a nitrogen meter appears to be preeminent in this regard.
- Some work has been done in recent years on the improvement of lithium fire control methodology, e.g., that done by HEDL, but more is needed. A fire suppressing material that rapidly cuts down the aerosol generation rate during a lithium fire would be very beneficial.
- Additional examination is needed of the causes, characteristics, and potential consequences of the trapping of magnetic corrosion products in the high magnetic field regions of electromagnetic components (i.e., pumps, flow meters) used on lithium loops. It may be necessary to routinely install magnetic impurity traps on lithium systems in order to prevent magnetic trapping from occurring in undesirable locations.

- There appears to be considerable incentive for the continued development and subsequent utilization of advanced structural alloys (e.g., refractory metals) as construction materials for large lithium systems. Also, although stainless steels appear marginal for long-term use in flowing lithium circuits, appreciable benefit could be realized by the development of corrosion inhibition techniques.

With regard to restart of the LPTL, the failure analysis team prepared a list of recommended action items. The most important of these were: (1) installation of a leak detection probe around the pump channel, (2) fabrication of a stress-free circular pump channel, (3) complete reconditioning of the scrubber system, and (4) a shakedown test (three days of attended operation) prior to continuous running in an unattended mode. A complete listing of these action items is given in Appendix E. It is worthy of final note that a circular pump channel/bus bar assembly suitable for implementation in a conduction-type EM-pump was actually fabricated and installed in an MSA Style V pump. A picture of the pump channel/bus bar assembly is shown in Fig. VI-1.

Fig. VI-1. Reconstructed MSA Style V Pump Section with Circular Pump Channel

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APPENDIX A

ANL CHEMICAL ENGINEERING DIVISION FAILURE ANALYSIS PROCEDURE (DRAFT)

Prepared by

R. A. Beatty

Argonne National Laboratory
Quality Assurance Division

November 1979

SCOPE

This procedure is provided as a guide for Managers. Provisions of the procedure considered appropriate to the particular Failure Analysis (FA) may be selected for implementation.

1.0 Program Manager/Group Leader (PM) appoint Failure Analysis (FA) team.

- 1.1 Consider as team leader a Project Manager or Responsible Scientist with needed technical expertise.
- 1.2 Include project key personnel: those with greatest incentive to prevent reoccurrence; those with most knowledge of problems and solutions; those best able to plan so that irreplaceable evidence is not lost.
- 1.3 Include personnel from related projects who may be implementing team findings.
- 1.4 Include Project Quality Engineer (PQE) as team member or to monitor FA progress through close coordination with FA team leader.
- 1.5 Include others as appropriate; safety, engineering, etc.

2.0 Team should address the following items, if applicable:

2.1 Agree on purposes of the FA

- 2.1.1 the purpose of FA is prevention: prevent further loss or damage; minimize negative impacts on program; prevent reoccurrence; prevent destruction of irreplaceable evidence during disassembly, clean-up or other activities, etc.

2.2 Establish the facts of the failure.

2.3 Define problems that caused the failure or that arose as a result. All problems identified should be reported in the draft FA report.

2.4 Define goals of the FA and which problems from 2.3 that the team should analyze.

2.4.1 Team goals should include:

- 2.4.1.1 find the most likely cause of the failure
- 2.4.1.2 define failure mechanisms
- 2.4.1.3 recommend corrective actions (CA),
CA responsibility and schedule

- a) CA to remedy current failure
- b) CA to remedy other problems that resulted from this failure

- c) CA to prevent other existing potential failure
- d) CA to prevent failure in future projects.

- 2.4.2 Team should assure that problems from 2.3 not analyzed by the team are analyzed by others.
- 2.5 Define ground rules, record keeping, information handling, information release, specific means (procedures) for carrying out elements of the plan, etc.
- 2.6 Select criteria for evaluation of problem solutions.
- 2.7 Determine FA cost restraints.
- 2.8 Determine need for GIDEP Report or Request (Government-Industry Data Exchange Program).
- 2.9 Determine need for Unusual Occurrence Report (reactor related).
- 3.0 FA team should submit initial FA plan and tentative schedule for Program Manager concurrence.
 - 3.1 The FA should begin promptly. A recent major FA required more than 60 days.
 - 3.2 Concentrate planning effort on defining steps of the specific analyses required. Determine what tests will be performed and in what sequence. Determine what disassembly will be done, by whom, how and when. Determine what visual observations will be made and how they will be recorded. Determine how best to ensure that critical or irreplaceable evidence required in a complete failure analysis will not be lost during disassembly, clean-up, sampling, etc. Other determinations suggested by FA team.
- 4.0 FA team members perform tasks assigned by team leader. Listed in Enclosure (1) are examples of tasks and concerns that may be appropriate to the FA.
- 5.0 FA team members should ensure recording of each action on assigned tasks, observations made, and significant results of evaluations.
 - 5.1 The FA plan should be reviewed and updated based on findings after completion of each step until the FA is closed out.
- 6.0 The FA team should meet, as required by the team leader, to ensure that:
 - 6.1 findings are reviewed and FA redirected, if required
 - 6.2 significant data are fully recorded
 - 6.3 team members interact

- 6.4 criteria recorded in the FA plan are adequately addressed and that planned goals are achieved.
- 6.5 problems selected in 2.4 are reported, analyzed and preferred resolution determined.
- 7.0 FA team should issue final report of the failure analysis and, when appropriate, interim reports and CA recommendations requiring immediate action. The FA team should include proposed distribution in their recommendations.
 - 7.1 FA team should provide draft of reports for comments by division safety coordinator (if there are safety concerns).
 - 7.2 PM should review and approve report and assign responsibility for CA implementation.
 - 7.2.1 Team leader should coordinate issue, distribution and implementation of PM approved report.
 - 7.2.2 Team leader should ensure CA follow up, CA completion and documentation of CA completion.
 - 7.2.3 Team leader should ensure that a system is set up to review CA effectiveness and continued implementation.
 - 7.3 The failure may be particularly significant or have great impact on other activities/schedules/funding/programs. In this event, PM may choose to have a formal review of the FA draft report. Selected specialists/consultants could review and comment before PM approval and distribution of the final report.
 - 7.4 The PM should ensure that FA results are reported to organizations having a need to know.
- 8.0 FA Closeout
 - 8.1 A copy of significant notes by team members should be forwarded to the team leader.
 - 8.2 The team leader should ensure that permanent records of the FA are archived in an effective Record Management Center.
 - 8.3 The team leader should notify the PM in writing when no further action by FA team members is considered necessary and the FA has thus been closed out.

TASK AND CONCERNS THAT MAY BE APPROPRIATE FOR FAILURE ANALYSIS

1. Review operating log.
2. Review project reports.
3. Review safety meeting minutes as well as reports.
4. Review design review minutes and reports.
5. Inquire about documentation of close out of corrective actions, open issues, etc.
6. Inquire about the criteria for past decisions (e.g., likelihood of failure). Consider redefine criteria in light of failure.
7. Identify other systems having equal likelihood of failure.
8. Identify other apparatus (division, ANL, government/industry) that lessons learned may apply to.
9. Identify records needed for failure analysis that are not being kept.
10. Were likely failure modes recorded and analyzed during design stage?
Were resulting management actions recorded?
11. Should CA already taken be continued?
12. Are additional emergency plans needed? By adjacent labs?
13. Have recent actions been taken, which if allowed to mature are likely to prevent similar failures?
14. Are critical operating procedures controlled, formally revised, reviewed and approved?
15. Are additional procedures needed?
16. Is additional training needed?
17. Are new personnel immediately oriented and adequately trained?
18. Should operators of critical systems be qualified by examination?
19. Should the process or apparatus be qualified by performance test?
20. Have suitable preparations been made to minimize losses in the event of such failures:
 - 20.1 Have information sources been identified to expedite equipment restoration?
 - 20.2 Did the emergency system and communications operate satisfactorily?

21. Are policies being implemented: project, division, ANL, DOE?
22. Are provisions adequate for funding repair, clean up, other contingencies?
23. Are provisions adequate to contain the effects of such failures and minimize impact on adjacent facilities?
24. Record the damage that actually resulted from the failure itself.
25. If the experience from the failure had been applied at the beginning, how could the failure have been prevented.
26. Estimate the cost of recommended CA.
27. Analyze CA cost/benefit, when appropriate.

APPENDIX B
REPORT OF ANL FIRE DEPARTMENT

ARGONNE NATIONAL LABORATORY
OHS - FIRE DEPARTMENT
REPORT OF FIRE and/or EMERGENCY
VEHICLE RESPONSE

Time of Notification: 1240 Date: 10/5/79

Alarm:

Rec'd by: Fireman Gusich
 (name)

vla: 2nd by GAS at 1243 hrs 1st by CFAC
 (tel. no.) (radio) (other)

Reported by: Mr. Lang
 (name) (Div. or Dept.)

Type of Incident: Fire alarm activation & report of lithium fire

Bldg. No.: 205 Room No.: H-126 Other: _____

Distance: .4 mi. Arrival Time: 1243 Under Control: _____

AES: Mr. L. Coleman Time Emergency Secured: 1656

FD Men and Vehicles Responding:

Time of Response: 1241 1241

Vehicle: Engine #1 Engine #2 Command Car

Officer: Capt. R. Foose Lt. W. Blaess Chief Veerman

Driver: C. Fenolio J. Nystrom

Hydrantman: T. Gusich

Breakman: J. Marasco

Nozzleman #1: C. Jones

Nozzleman #2: _____

Pumping Time: _____

Time of Return to Quarters:
1343 1343

Hose Lines Used: 1" Booster ft., 1-1/2" Hose ft., 2-1/2" Hose ft.

Type Nozzle/s Used: _____ Ladders Used: _____ No. of Sprinklers Open: _____

Extinguishers Used:

By Whom: Capt. Foose, Lt. Blaess, FF Jones Div. or Dept.: OHS-FD Type: 30# Lith-X: 30# Dry Powder of Size: numerous quantities

Large quantities of bulk Lith-X, Met-L-X, and dry silica sand

FD Respirator/s Used:

By Whom: Capt. Foose, Lt. Blaess, FF Jones Div. or Dept.: OHS-FD Time Used: 60 minutes each

Chief Veerman, FF Marasco OHS-FD 30 minutes each

List Other Equipment Used: Long handled scoop shovels, smoke ejectors, spare Scott bottles, etc.

Equipment Checked by Radiation Safety? ☒ Yes ☐ No Contaminated? ☐ Yes ☒ No

Radiation Safety Representative: In attendance

List Contaminated Equipment: None

OHS-FD-14 (7/74) (over)

FOR USE BY DEPT. OFFICE ONLY	
EMERGENCY VEHICLE RESPONSE:	
FIRE	☒
FALSE ALARM	☐
OTHER	☐
INVESTIGATION ONLY:	
REPORTABLE FIRE	☐
NON-ANL FIRE	☐
OTHER	☐

Date of Report: 10/5/79 Reported by: Robert Foose Title: Captain
Officer in Charge: Robert M. Foose

Building 205 Fire
10/5/79
1240 hours

SUMMARY OF OPERATIONS AND STORY OF FIRE (CONT'D.)

burning through. After that hour, no more extinguishers were deemed necessary. At 1850 hours I requested Mr. Girard to call in Mr. Propper of Communications to attempt to return the fire alarm system in the cell to a normal condition. At 2014 hours Mr. Propper and I went to the building and replaced the two smoke heads, reset the system OK. Relieved the fire watch who, upon my determination, was no longer necessary. Mr. Weston indicated that he would be there until approximately 2200 hours and then I would tour the fire scene at approximately 2330 hours (see Inspection Tour reports).

NOTE: During the course of the emergency many things transpired that are not indicated on this report (the calling in of additional men, the accumulations of additional fire extinguishers, bulk extinguishants, additional self-contained breathing apparatus, the fine work of Plant Maintenance personnel, technicians, and especially Mr. Weston, and of course the professionalism shown by the fire fighting crew at the scene.) Chief Veerman's able assistance, direction, and cooperation was greatly appreciated. The assistance of the Fire Inspectors, Mr. Lindberg, OHS Safety group and many others is appreciated.

APPENDIX C
PUMP CHANNEL ANALYSIS PLAN

U.S. DEPARTMENT OF ENERGY

ARGONNE NATIONAL LABORATORY

9700 SOUTH CASS AVENUE, ARGONNE, ILLINOIS 60439

ANL-CENI-77

PLEASE DO NOT USE THIS NUMBER FOR REFERENCE EXCEPT IN OTHER INTERNAL DOCUMENTS OF THE CHEMICAL ENGINEERING DIVISION-ANL

TELEPHONE 312/972-4547

December 18, 1979

TO: Distribution

FROM: V. A. Maroni *v/am* Chemical Engineering Division

SUBJECT: Examination Plan for the First Series (Phase I) of Analyses
of the Failed LPTL Pump Channel

This memorandum describes the steps that will be taken in analyzing the first sample sections to be cut from the failed LPTL pump channel. (The pump channel is hereafter identified as that section shown in the photo insert in Attachment I.) The motivation for the present plan stems from (1) a desire to get on with the examination so that we can begin to draw conclusions about the cause of the failure and to formulate plans for eliminating the cause, and (2) an equally compelling desire not to lose valuable information by entering the examination phase too hastily. To avoid, as much as possible, the latter problem, a plan has been conceived (by D. L. Smith, E. H. Van Deventer, and myself) wherein we section only a part of the channel at this juncture and carryout a series of micro-metal-lurgical analyses to determine, among other things, what additional steps should be taken in subsequent analyses. In particular, the section of the pump channel containing the leak-holes will not be cut into at this time. Instead we will cut out sections for which a "reasonably" symmetrical equivalent exists in the remaining uncut sections. In this way, should we decide at a later date that a different sectioning, mounting, examining, etc., approach is necessary, we will still have original material to start with. To further preserve sample replaceability, each vertically cut numbered section (see Attachment I) will be further cut longitudinally and one half of that section will be archived. Also, the initial cuts (at 10, 13, and 16) will be made with a "dry" saw so that the unsectioned remainder is not further contacted with water or other lubricant beyond what has already occurred. This dry cutting should be done so that the amount of heating to the section is minimal. Subsequent cuts of the sections to be examined will be made with a cut-off wheel in an inert environment, using facilities in J-101 or D-205. The sequence of steps for these "Phase I" examinations is then as follows:

1. Measure the distance of each cut location from location 1 in Attachment I to $\pm 1/32$ of an inch.
2. Dry cut at locations 10, 13, and 16 (See Attachment I). The section to the right of a cut-line number will be given that number.

3. Using the cut-off wheel in J-101 separate sections 10, 11, and 12, and then make a symmetrical longitudinal cut through each section. Tag and archive one of the half-sections of each full section.
4. Vertically slice and tag five (5) 0.5 cm-thick subsections from one of the half-sections of each section. The code numbering will be done in the following way.

Notes:

- a. A (exposed face in the inserted photo) or
B (covered face in the inserted photo).
 - b. U (upper) or L (lower) longitudinal section
 - c. 01 to XX depending on number of verticle cuts made.
5. After each subsection is cut, a marking will be made on that subsection to preserve the identity of the A face.
 6. The samples will then be distributed to the various organizations conducting the examinations. A list of desired procedures and analyses will be generated, but for the most part we will let individual examiners use their own established procedures during Phase I.

All persons involved in the analysis of Phase I specimens are expected to keep records of the types of analyses performed, the instrumental conditions and parameters used, and the nature of the results. All data, photos, charts, and other output should be marked so that they are directly traceable to the respective sample. In addition, each organization conducting an analysis will be asked to look for and record evidence of specific corrosion-related phenomena, e.g., intergranular attack, non-stoichiometric surface attack, dissolutive corrosion, and grain restructuring.

The actual procedures used throughout Phase I are expected to be the reasonably well established ones normally used in this type of analysis and will be documented at a later time to permit some flexibility in this first series of examinations. Let's plan to meet on Thursday, December 20, 1979 at 3:30 p.m. in the L-186 Conference Room in Building 205, to discuss this plan further.

VAM:mlw
Enclosure

Distribution:

J.R.Weston, CEN
M.A.Slawecki, CEN
C.C.McPheeters, CEN
D.L.Smith, MSD
H.L.Brown, CEN
L.F.Coleman, CEN
R.M.Foose, OHS
R.A.Beatty, QAD
F.A.Cafasso, CEN
D.S.Webster, CEN
FA File (Original)

ATTACHMENT I

Diagram of the Reference Position, Cutting Pattern and Numbering Scheme for the Failed LPTL Pump Channel

APPENDIX D

LETTER TO OTHER LABS CONDUCTING METALLURGICAL ANALYSES

US DEPARTMENT OF ENERGY

ARGONNE NATIONAL LABORATORY

9700 SOUTH CASS AVENUE, ARGONNE, ILLINOIS 60439

TELEPHONE 312/972-

4547

March 4, 1980

TO: Distribution

FROM: V. A. Maroni

SUBJECT: Analysis of Specimens from the "Failed"
Channel of the LPTL EM-Pump.

As all of you are aware, on October 5, 1979 we experienced a major leak and, associated with it, a small fire in the Lithium Processing Test Loop (LPTL) Facility at ANL. The leak was the result of a failure in the EM-pump channel. Following the incident, an Incident Analysis Team (IAT) was set up at ANL to conduct an investigation of the causes, impacts, and experience gained. This group has had several meetings and assignments of investigatory responsibilities have been made. One of the initial tasks of the IAT was to develop a plan for sectioning and analyzing the pump channel. In connection with this analysis, which is progressing at ANL, those of you on the distribution list have expressed an interest in receiving some specimens of the failed pump channel and neighboring pipe sections for your own analyses. I appreciate your willingness to conduct these investigations and, accordingly, am forwarding herewith two specimens to each of you for study as you see fit. The remainder of this memorandum contains information which may be of use to you in your analyses, together with some guidelines for preserving important details that relate to sample identification and handling.

The actual spillage of lithium was the result of two rather large holes which developed in the center of the EM-pump channel. A picture of the extracted pump channel and a blowup of the region of the holes is shown in Figure 1A/1B. Because rather high temperatures were reached in the vicinity of the pump channel at the time of the fire, it is believed that wastage, which occurred during the fire, caused the holes to open to their present size. It is not likely that we will ever be able to determine how large the hole(s) were when the leak first started. We do know from thermocouple readings in the vicinity of the pump channel before, during and after the fire (see Figure 2) that the leak may have started as much as 40 minutes before the fire was actually detected (via the smoke alarm). The innermost section of the pump channel was the only section of the entire LPTL system that was not wired for leak detection.

Analyses of specimens from the pump channel conducted to date at ANL have revealed some noteworthy features. Solidified material inside the pump channel (mostly reacted lithium residue) was found to contain several discrete phases with sharply defined X-ray diffraction patterns. These include Li_3N , a compound of the type $\text{Li}_x\text{M}_y\text{N}_z$ (where M appears to be chromium) and several other phases that we have not as yet been able to identify. This

residue also contains appreciable quantities of finely divided needle-like crystallites of α -iron. The pump channel itself is heavily pitted and in some places appreciably thinned out by what appears to be some type of an erosive process that occurred throughout the channel. Samples of unreacted lithium, cored from a section of pipe downstream from the pump channel, were found to contain essentially the same nitrogen level (900 wppm) as had been present in lithium samples taken when the LPTL was last operating. However, analyses of the pipe section from which this lithium was cored showed a much higher level of hydrolyzable nitrogen (evolved as NH_3) than was expected. We suspect that there may be some ternary nitride phases which have infiltrated the first 20 or 30 μm of the inner surface of the LPTL piping (304-SS) during its 9500 hours of operation (mostly at around 400°C).

Each of the samples you have received has been sliced from the channel or adjacent piping with a Buehler cut-off wheel, using Freon TF as a lubricant to avoid hydrolyzing surface and subsurface impurities. Each sample has been code numbered so that it can be traced to its original location in the pump channel. The exact locations of your samples are shown in Figure 3. Since the pieces are essentially symmetrical, each piece has a strip of red tape on one side. It will be important to keep track of which side(s) you have examined with reference to the red tape. Please do not lose track of your sample identities. In addition, we further ask that you keep a record of the washing, mounting, polishing and etching procedures you apply to these samples as well as the important instrument parameters used during your analyses. Since most of us will be attending the Liquid Metals Conference at HEDL in April, I will try to arrange a meeting of all specimen recipients so that we can exchange ideas on how to best conduct the analyses and what to look for. In the meantime, do not hesitate to contact me (Telephone: 312-972-4547; FTS: 972-4547) if you have any questions between now and the conference. After our meeting at HEDL, I will prepare a list of answers to all questions concerning this analysis activity and distribute it to your offices.

Thank you in advance for the time and effort you will be spending on your investigations of the LPTL specimens. I look forward to getting together with all of you at HEDL in April.

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Chemical Engineering Division

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Fig. 1.A Photograph of the failed EM-pump channel excised from the LPTL.

Fig. 1.B Expanded view of the region of the EM-pump channel where the leaks developed.

Fig. 2. Plot of EM-pump channel interior thermocouple readings before, during, and after the smoke alarm signal.

Fig. 3. Locations of specimens and sample notation for samples distributed with this memorandum.

APPENDIX E

LIST OF ACTION ITEMS (Actions approved for implementation on the LPTL facility are indicated by a check mark or circle around number; the items with circles will be completed before the three-day attended operation test while the checked ones will be done at the start of unattended operation.)

- ✓ (1.) Analyze the lithium in the reservoir and dump tanks. Reduce the nitrogen level in the lithium by mechanically removing the Li_3N layer on the lithium surface in the reservoir tank.
- ✓ (2.) Conduct a flush-out of the lithium piping (with argon gas) from (a) by-pass line to dump tank, and (b) reservoir to dump tank before pump installation.
- ✓ (3.) Recheck results of weld qualification tests. Radiograph and destructively test welds in Sections 1 and 14. Consider other weld examinations. Check on other nondestructive test methods (e.g., ultrasonic) for examining welds.
- ✓ (4.) Leak-check seals associated with salt tank and reduce leaks for the entire gas system.
- ✓ 5. Analyze thermal stress in the pump duct. If stress in pump duct exceeds two-thirds yield stress of stainless steel at operating temperature, pump should be modified.
- ✓ (6.) Recondition the scrubber:
 - (a) Remove old scrubber pump.
 - (b) Check pipes connected to pump.
 - (c) Replace pipe if necessary.
 - (d) Clean scrubber spray heads.
 - (e) Install new scrubber pump.
 - (f) Test scrubber.
- ✓ (7.) Repair damaged power leads.
- ✓ (8.) Replace damaged thermocouple extension wires.
- ✓ (9.) Install new EM pump. Connect pump to supply leads.
- ✓ (10.) Connect line heaters that have been replaced.
- ✓ (11.) Install one extra leak detector lead for pump channel.
- ✓ (12.) Add lithium to system.
- 13. Change the design of the new EM pump, i.e.,
 - ✓ (a) Replace copper bus bars with nickel ones to reduce the heat sink problems.
 - ✓ (b) Replace the flattened pump channel with a circular one.
 - (c) Replace the bus bars with ones that "float" with respect to their brazes (i.e., a flexible joint in the bus bar) so as to reduce strain.
 - (d) Rotate the EM pump 90° to reduce the strain on the pump channel caused by flexible joints in bus bars.

- ✓(14.) Install a high temperature alarm and shut-down for at least one location on the EM pump channel.
- 15. Install an automatic check valve to vent the cover-gas system to atmospheric pressure during an emergency shut-down.
- 16. Install a magnetic trap in front of the EM pump to extend the longevity of the system.
- ✓17. Define current expectations for future LPTL use.
- ✓18. Specify design changes. With revised design criteria, a design review for safety should be considered.
- ✓(19.) Prepare a plan for preoperation check-out.
- (20.) Conduct a three-day test of the LPTL after completion of repairs. Prepare a test plan for LPTL operation during the 3-day test.
- 21. A formal review of procedures of all work activities should be performed by the Project Manager.
 - (a) Specify requirements for each step.
 - (b) Specify criteria that verify requirements are met.
 - (c) Identify critical subsystems for which written specification is needed to assure that requirements have been met.
- 22. Define evaluations of facility components to be done at end-of-life analysis. Assure that LPTL operation does not destroy end-of-life evaluation data.
- ✓23. Inspect all critical components of facility; recalibrate instrumentation.
- 24. Prepare or revise written procedures for critical activities.
- ✓25. Verify that all critical systems actually perform per design requirements.
- ✓(26.) Procedures for normal operation of all subsystems should be understood and rehearsed by operators and backup operators.
- ✓(27.) Procedures for abnormal operation (identified in a safety review) should be understood and rehearsed by operators and backup operators.
- ✓(28.) Objective evidence exists that in the event of another fire, the October 1979 incident would be prevented by using the following criteria:
 - (a) The lithium dump operation should be designed to proceed smoothly and rapidly.
 - (b) The LPTL cell containment and the exhaust system should be modified to adequately contain and remove the aerosol from the fire.

- ✓(29.) Verification should be made that each critical subsystem performs adequately throughout its operation range.
- ✓(30.) Visual and instrument indications should verify acceptable performance throughout all shakedown tests.
- 31. Subsystem conditions after shakedown tests should be checked to verify that there is no evidence of deterioration or abnormal operation due to the tests.
- ✓(32.) The log of system performance during the three-day test and subsequent operation should be maintained as a complete record.
- 33. Prepare plan of responsibility matrix and acceptance criteria for preoperational checkout.
- 34. Prepare plan of responsibility matrix and acceptance criteria for three-day fully attended system test.
- 35. Identify LPTL subsystem design requirements and safety factors.
- ✓(36.) Identify revised procedures for normal and abnormal operation.
- ✓(37.) Review procedures in light of accident scenario.
- ✓(38.) Review operating/safety procedures with system operators.
- 39. Identify operating range of each subsystem.
- (40.) Review test plan to assure each critical subsystem is evaluated throughout normal operation range.

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