# EMERGING TECHNOLOGY REPORT: RECLAMATION OF LEAD FROM SUPERFUND WASTE MATERIAL USING SECONDARY LEAD SMELTERS

RISK REDUCTION ENGINEERING LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY CINCINNATI, OHIO 45268

# NOTICE

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# FOREWORD

The Superfund Innovative Technology Evaluation (SITE) Program was authorized in the 1986 Superfund Amendments. The program is a joint effort between EPA's Office of Research and Development and Office of Solid Waste and Emergency Response. The purpose of the program is to assist the development of hazardous waste treatment technologies necessary to implement new cleanup standards which require greater reliance on permanent remedies. This is accomplished through technology demonstration designed to provide engineering and cost data on selected technologies.

The Risk Reduction Engineering Laboratory (RREL) is responsible for planning, implementing and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis for support of the policies, programs, and regulations of the EPA. This publication is one of the products of that research and provides a vital communication link between the research and the user community.

This project consisted of demonstration of the use of secondary lead smelters to reclaim lead from a variety of materials, including materials found at Superfund sites as well as other lead-containing wastes. The demonstration consisted of feeding materials from five sources – three Superfund sites, and two other sources, to a secondary lead smelter to determine if the smelter could economically reclaim the lead.

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# ABSTRACT

There are over 3,000 sites across the United States contaminated wtth lead. Techniques to remediate these sites include standard stabilization/disposal technologies, reclamation of lead using secondary lead smelters, soil washing, and biological removal technologies.

Through a Cooperative Agreement with the U.S. Environmental Protection Agency's Risk Reduction Engineering Laboratory, the Center for Hazardous Materials Research CHMR), in conjunction with a major secondary lead smelter, has demonstrated that secondary lead smelters may be used economically to reclaim lead from a wide range of lead-containing materials frequently found at Superfund sites. Such materials include battery case materials, lead dross, and other debris containing between 3 and 70 percent

During the study, CHMR and the smelter reclaimed lead from five sets of materials, including two Superfund sites containing primarily battery cases, and one battery breaker/smelter site with a variety of lead-containing materials. Between 4 and 1600 tons of materials from each of these sites were excavated and processed at the smelter, while the research team assessed the effects on furnace operation and performance. Two additional sets of materials, one from the demolition of a house containing lead-based paint, and the other consisting of blasting abrasive material from work on a bridge coated with lead paint, were also processed in the smelter. The results showed that it was technically feasible to use the secondary ead smelter to reclaim lead from all of the materials. CHMR considered the use of materials from a sixth site, which contained approximately 80% soil and 20% waste battery cases. The site was dropped from consideration for the study, because the smelter could not accept materials with such a high soils concentration

CHMR also assessed the economics of using secondary lead smelters to reclaim lead from Superfund sites, and developed a method for estimating the cost of reclaiming lead. This method develops cost as a function of material excavation, transportation and processing costs combined with cost benefits received by the smelter (in the form of recovered lead, reduced fuel usage and/or reduced iron usage). The total remediation costs using secondary lead smelters for the sites and materials studied varied between \$35 and \$374 per ton, based on January 1994 market prices for lead. The costs were primarily a function of lead concentration, the market price for lead, distance from the smelter, and the amount of materials which become incorporated into slag from the process, although other factors affected the economics as well. Materials with high concentrations of lead were significantly less expensive to remediate than those with bw concentrations. The cost to remediate materials which left few slag residues in the furnace was lower than the cost to remediate materials which contained a significant amount of material that remained in the slag.

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# 1.0 INTRODUCTION

Lead is used in the production of various consumer and commercial items, from automobile and equipment batteries to paints to crystal. This widespread use has made it one of the most common contaminants at sites on the National Priorities List (NPL). The most common current treatment of lead contaminated wastes at Superfund sites is immobilization, either on-site or in a landfill. Remedial approaches which involve recovery of lead are preferred over immobilization, which wastes the lead. One such remedial approach is the use of secondary lead smelters for recovery.

The initial sections of this report provide a brief overview of the sources and types of lead contamination and available remediation technologies. The remaining sections present results from a study of secondary lead smelting as a reclamation technology for lead-containing waste material at Superfund sites. The economics of reclamation are examined to determine if the technology is economically competitive with other technologies. Finally, a prognosis is given for the use of secondary lead smelters for lead recovery.

#### 1.1 SOURCES OF LEAD CONTAMINATION

The prevalent use of lead in paints, gasoline additives, and other products resulted in wide dispersion throughout the environment. A review of the literature [1-11] reveals the following sources of lead in the environment:

- Lead acid battery breaking activities;
- Primary and secondary lead smelting and refining;
- · Production of lead acid batteries;
- · Production, storage and distribution of gasoline with leaded additives;
- Solder use and manufacture;
- Plumbing;
- Ceramics and crystal manufacture:
- · Paints (houses, bridges, ships), and paint abrasive blasting material;
- Wire manufacture and coating;
- Automobile demolition (auto fluff);
- · Construction demolition (typically in plumbing and paints);
- Production and use of fishing sinkers;
- Pesticide production and use;
- Cathode-ray tube production and use;
- Rifle ranges and munitions dumps, including state game land and military ranges;
- Ammunition and explosive manufacturing;
- Sewage sludge;
- By-products from metal production (e.g., electric arc furnace dust from steel production);

- · Radioactive shielding (from x-ray machines to reactors); and,
- Other metals mining, smelting, and alloying (copper, zinc, cadmium, and chromium).

These uses and sources of lead have contributed to many types of lead contamination, including lead contaminated soils, battery breaker sites, and integrated breaker/smelter sites. These are discussed below.

#### 1.1.1 Lead Contaminated Soils

Lead in soils can be from a variety of sources. The range of lead contamination from 436 Superfund sites surveyed is between 0.16 and 366,000 mg/kg, compared to up to 200 ppm in typical uncontaminated soils [12, 13] and 51,000 mg/kg in lead ores used at a major primary lead smelter [1]. Lead has been detected in soils in urban environments at concentrations of up to 15,000 mg/kg due to a combination of automobile exhaust, lead paints and batteries [15]. Lead is commonly found in soils at battery breaker sites in concentrations up to 100,000 mg/kg [8] and at gun clubs, with concentrations up to 10,000 mg/kg [9].

Small amounts of lead are naturally present in the form of galena (PbS) although upon exposure to air it is converted to carbonates, sulfates, oxides and sulfur salts. Anthropomorpic lead sources in soils typically include acetates, organometallic compounds, oxides, elemental lead, sulfates, halides, sulfides, and silicates [16].

The United States Environmental Protection Agency (EPA) has set acceptable lead levels in soils at a range of between 500 and 1,000 mg/kg. Otherlimits may be used based primarily on the likely uptake of lead in children or the proximity of the site to residential areas [17].

#### 1.1.2 Battery Breaker Site Materials

The components of lead-acid batteries include the battery case, lead electrodes (typically screens), spacers that separate the electrodes and prevent shorting, sulfuric acid, and lead battery paste ( $PbSO_4$ ). In the past, the lead in lead-acid batteries was commonly removed by cracking or breaking the battery shell, draining the sulfuric acid into surface impoundments or lagoons, and pulling out as much of the metallic and paste lead as possible.

The battery cases, spacers, and some of the paste were sometimes improperly disposed or piled. Battery cases were often disposed in drainage ditches and pits, and then buried with soil. Sometimes the cases were burned in pits on site to recover additional lead. These materials, when segregated from the soil, have lead concentrations ranging from 5,000 to 200,000 mg/kg [8]. Battery cases historically were made from ebonite rubber, which is a hard, black rubber containing coke and coal dusts. In the late 1970's, battery manufacturers switched to polypropylene cases, which are readily recycled.

Among the materials typically found at battery breaker sites are [1,4,19]: broken or whole battery cases, lead scrap, battery paste, sulfuric acid, lead debris and, (if the battery parts were burned) partially incinerated battery parts. There were approximately 20 battery breaker sites on the NPL in 1991 [4].

## 1.1.3 Integrated Breaker/Smelter Site Materials

Some battery breaking plants also reclaimed lead on-site at smelting facilities. These facilities would sell the recycled lead to battery manufacturers for use in the production of new lead-acid batteries. Materials found at these sites include the same materials found at battery breaker sites, as well as alloys, slag, emissions control dusts, dross, lead oxides, and calcium sulfate sludge from emissions scrubbers.

## 1.1.4 Other Lead-Containing Materials

Other lead containing materials frequently found at Superfund sites include lead munitions and shot, automobile fluff, and lead additive residues. Lead is very common, and its forms vary greatly: one site, for example, contains over 10 million kg of lead-containing plastic wire insulation, and a second site contains 20 million kg of broken lead-containing crystal, accumulated over several decades.

## 1.2 AVAILABLE REMEDIATION TECHNOLOGIES

There are several options for treating lead contaminated sites, including soil washing or extraction, immobilization, and reclamation. There are many variations of each type of remediation technology. A

survey of records of decision (RODs) [18] indicated that over 70 percent of lead-contaminated sites were treated with some type of immobilization technology, usually stabilization or disposal in a landfill permitted to receive hazardous waste. Less than 10 percent were treated using washing (size separation or extraction) technologies, and less than 10 percent were treated using a reclamation process to recover usable lead. The remainder were treated using thermal technologies such as incineration because the material was contaminated with organic as well as heavy metal contamination. Incineration destroys the organic material, but there is typically lead-contaminated ash remaining after treatment. This ash usually retains the metals and therefore may also be hazardous and require separate treatment and disposal. Several current treatment technologies are described below.

## 1.2.1 Solidification/Stabilization

Solidification/stabilization technologies reduce the leachability of contaminants in the soil. Stabilization refers to mixing of waste with reagents such as flyash to produce an end product that has about the same consistency as the original waste, but with significantly reduced contaminant leachability. Solidification involves mixing the waste with ingredients, such as binders or concrete, in cement mixers, pug mills, or other types of equipment to produce solid blocks of material with considerable strength. The main goal is to lock the contaminants into the material so they do not leach out [1,4,21]. The two technologies are often used together [20,21].

## 1.2.2 Capping

Capping a waste site reduces the mobilization of the lead by containing it on the site. This prevents direct contact of the contaminated material with the public and the surroundings. Capping is attractive for lead-contaminated soils, since the lead is often relatively immobile in some soil systems [3]. Capping may be performed with compacted clay, synthetic liners or both.

### 1.2.3 Washing

Washing usually involves slurrying the waste with an aqueous solution, and then physically or chemically removing the contaminants using acid, chelating agents, size separation, or solvents. Some soil washers separate the heavy metals based on the principle that most contaminants are concentrated in the finer soil fractions. These technologies remove the finer soils (which must be treated or disposed) and return the coarse material to the site as clean soil [27].

Although washing is generally used for soils, some companies are developing technologies to remove and wash the battery case material. These systems use gravity separation or other techniques to segregate the battery cases from soil, rocks, or other debris, and scrubbers are used to thoroughly wash the cases. The clean battery cases can then be landfilled as non-hazardous waste [23,28]. It is difficult to remove all the lead from ebonite battery cases, because the lead permeates the slightly porous ebonite.

Most soils washing technologies produce either a soils fraction with high lead concentration or an hydroxide sludge, which must be disposed. However, several recently developed technologies purport to remove lead from contaminated soils and produce metallic lead as a product [35], eliminating the need to dispose of residuals.

Solidification/stabilization capping, and washing have been proven to be effective in remediating heavy metals in soils [4,19]. The use of these technologies, however, has not been as successful when there is large debris, such as battery cases or metallic lead, in the waste stream. Reclamation using secondary lead smelters provides a more feasible alternative for such materials.

# 2.0 LEAD RECLAMATION USING SECONDARY LEAD SMELTERS

Secondary lead smelters typically reclaim lead from spent lead-acid batteries. The use of secondary lead smelters to reclaim lead from Superfund or other waste feeds involves slight rnodifiitions to the normal smetting process. This section provides a description of typical secondary lead smelting processes, as well as the modifications required to process the waste material.

#### 2.1 TYPICAL SMELTING OPERATIONS

The evaluation was conducted at Exide/General Battery Corporation's secondary lead smelter location in Reading, Pennsylvania. A schematic of the process is shown in Figure 1. The operations at this smelter are typical of those found in the secondary lead smelting industry. As part of normal secondary lead smelting operations using reverberatory and blast furnace combinations, spent batteries received at Exide's Reading smelter are crushed to release the sulfuric acid. Next they are processed through a sink/float system to separate the battery cases from the heavier lead particles. The plastic battery cases are recycled on-site for use in the production of new battery cases.

Reverberatory furnaces are charged with lead from the sink/float system as well as other lead containing material, and are fueled with natural gas and oxygen. These furnaces are tapped for slag, which typically contains 60 to 70 percent lead, and a pure (soft) lead product.

Blast furnaces are charged with the slag generated from the reverberatory furnaces as well as other lead-containing materials, and are fueled by coke and air enriched with oxygen. Iron and limestone are added as fluxing agents to enhance the lead production in the furnace by preventing much of the lead from remaining in the blast furnace slag. Typical feed concentrations for coke, iron, and limestone are 5.7, 8, and 0.9 weight percent, respectively [29]. The blast furnaces are tapped continuously to remove lead and intermittently to remove the slag. The blast slag, which contains primarily silica, iron oxides, and some lead, is usually not hazardous and is transported to an off-site residual waste landfill for disposal.

Lead produced in the blast and reverberatory furnaces is transferred to the refining process where additional metals are added to make specific lead alloys. The lead is then sent to the casting operations where it is molded into ingots ("pigs") for use in the manufacture of new lead-acid batteries. Waste materials from Superfund and other sites are fed either to the blast or reverberatory furnaces, depending on the material type, particle size, density, and other parameters. The modifications made to enable processing of this waste material are discussed in the following section.

# 2.2 USE OF SECONDARY SMELTERS IN LEAD RECLAMATION FROM WASTE MATERIAL

Prior work involving the use of secondary lead smelters to reclaim lead focussed on battery cases. In general, waste material was simply put through a smelter to dispose of it. There were no quantitative attempts made to analyze the affects on the furnace, or if the reclamation itself was successful [29]. One goal of this research was to determine how the smelter furnaces react to the addition of various waste materials.

The first step in the reclamation process, shown schematically in Figure 2, acquiring and transporting the material to a secondary lead smelter. Generally, this involves excavation or collection, pre-processing, and transport to the smelter. The lead-containing waste material is typically excavated from lead-acid battery Superfund sites or collected from other sources, such as bridge blasting or demolition operations.

Next, the material may require processing prior to entering the furnace. Preprocessing includes screening to remove soil, large stones, or noncontaminated debris. Materials larger than 12 inches cannot be fed into the reverberatory furnaces. These larger materials may cause jams in the belt system that feeds the reverberatory furnace, or they may remain unburnt in the furnace for too long a period of time. The soil and debris removed during pre-processing may be treated using one of the technologies described in section 1.2, since soil cannot be processed through a secondary smelter.

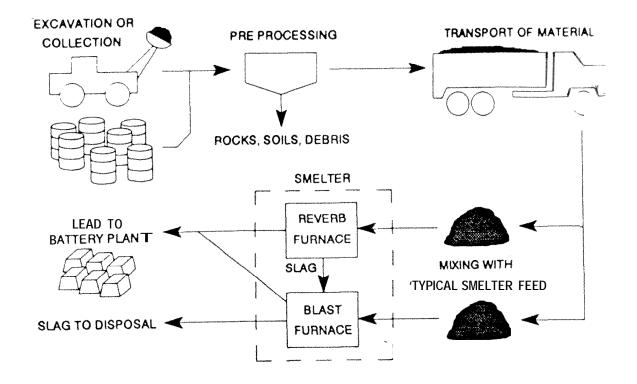


FIGURE 1 SCHEMATIC OF SMELTING PROCESS

The pre-processed material is mixed with regular furnace feed from the crushing and sink/float system. The feed rate is determined by lead content, size of the material, fuel values, and other parameters.

# 2.3 EVALUATION METHODOLOGY, SITES, AND MATERIALS

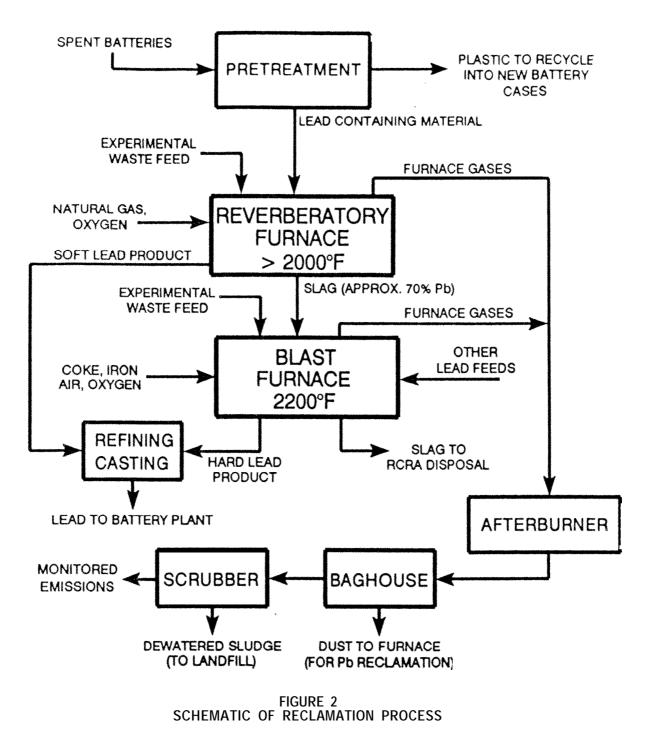
The basic methodology during the project was to acquire quantities of the materials to be tested, characterize these materials, process them through the secondary smelter, and observe their effects on the furnaces through sampling and data collection. The primary objective of the tests was to determine how much and what types of materials could be fed to the furnaces without causing shutdowns or otherwise adversely affecting smelter furnace performance.

# 2.3.1 Acquisition of Materials

Materials from three Superfund sites as well as two additional sets of lead-containing materials were processed during this project. The following sections provide a short description of each of the file evaluations. The feed rates are presented as weight ratios of test material to total furnace feed. Table 1 presents a summary of the materials tested and the evaluations. More information on three of the sites (Tonolli, NL Industries, and PennDOT) can be found in appendices A, B, and C, respectively.

# Tonolli Superfund Site

The Tonolli site is a 30 acre battery breaking and smelting facility located in Nesquehoning, PA. Piles of ebonite rubber and polypropylene battery case pieces were tested, without any pre-processing. The material had an average lead concentration of 3.5 percent. Approximately 84 tons of material were fed at a ratio of 10 percent through a reverberatory and blast furnace. The material was too large to be readily processed In the reverberatory furnace, but was successfully processed in the blast.



Hebelka Superfund Site

The Hebelka site was a former automobile junk and salvage yard located in Weisenburg Township, PA. The site contained battey case debris mixed with soil that had an average lead concentration of 14.7 percent. Approximately 20 cubic yards of material were transported to the smelter. This material was first

percent. Approximately 20 cubic yards of material were transported to the smelter. This material was first reduced in size to less than <sup>1</sup>/<sub>4</sub>-inch with a hammermill. The material was successfully fed to the reverberatory furnace at a feed ratio of 17 percent.

#### Demolition Waste

Demolition waste was obtained from a Housing and Urban Development (HUD) project. The waste consisted of demolition debris mainly wood) coated with lead based paint and had a lead composition of between 0.5 and 1 percent. The test material was shredded in a pallet shredder before It was smelted The demolition debris was processed through both reverberatory furnaces at feed ratio of 10 percent test material, by weight. At this weight ratio the test material comprised 50 percent of the volume fed to the furnace. This high volumetric ratio caused malfunctions in the furnaces, so the feed ratio was reduced to 5 percent, at which point the material was successfully fed.

#### NL Industries Superfund Site

The NL Industries site in Pedricktown, NJ, was an integrated battery breaking, smelting, and refining facility with its own on-site landfill. There were a wide variety of materials at the site including lead slag, dross, debris, ingots, hard heads (large chunks of metallic lead), battery case debris, baghouse bags, and contaminated pallets and iron cans. The evaluation was conducted in two parts: a preliminary investigation and a full-scale investigation.

		Т	able 1 Summ	nary of the Evaluation	ns		
Site	Site Description	Site Size	Test Duration	Type of Mat'l	Amt. Processed	Avg. Lead Conc.	Feed wt. Ratio
Tonolli	Integrated battery breaker	30 acres	5 days	Rubber & plastic battery cases	84 tons	3.5%	20%
Hebelka	Auto junk & salvage yard	20 acres	1 day	Mainly plastic battery cases (some rubber)	12 tons	14.7%	17%
Laurel House	Residence	N/A	1 day	Wood demolition debris	4 tons	1%	5%
NL Industries	Integrated battery breaker & smelter	11 acres	3 months	lead slag, debris, dross, ingots, cases, baghouse dusts	1570 tons	30 to 60%	20 to 50%
PennDOT	Bridge repainting site	N/A	1 day	Iron shot bridge abrasive	6 tons	3.2%	13%

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During the preliminary investigation, approximately 370 tons of all types of the above materials were processed. Analyses revealed an average lead concentration of 57 percent. The larger pieces of debris were removed and processed through a blast furnace, while the bulk of the material was fed into a reverberatory furnace at feed ratios of up to 100 percent. The feed was sufficiently dense to cause breakdowns in the reverberatory furnace conveyor feed system, so the feed ratio was reduced to 50 percent test material, by weight.

During the full scale operation, approximately 1200 tons of material were transported to the smelter over a three month period. For the first two months, the test material, which contained approximately 50 percent by weight lead, was processed in the reverberatory furnace, with a feed ratio of 20 to 30 percent. The ratio was limited due to high amounts of calcium in the NL material. The high calcium concentration slowed the operation of the furnaces.

The test material for the last month of the investigation consisted mainly of larger pieces of slag and debris with an average lead concentration of 30 percent. This material was charged directly to one of the blast furnaces, at a feed ratio of approximately 30 percent.

#### Pennsylvania Department of Transportation

The Pennsylvania Department of Transportation PennDOT) used an iron-shot abrasive blasting material to remove old lead-based paint from a bridge in Belle Vernon, PA. Sixteen 55 gallon steel drums of this material, containing an average of 3.2 percent lead, were processed at the smelter.

The bridge blasting material was primarily iron (60 percent) with 5 percent calcium and 5 to 10 percent moisture content. The material contained too much moisture to be incorporated into the reverberatory feed. The test material, including the drum, was fed to one of the blast furnaces at a feed ratio of approximatety 13 percent, by weight.

#### **Bypass 601 Site Material**

Material was obtained from the Bypass 601 Superfund site in Concord, NC. The material consisted of battery cases, mixed with soil. Overall, it contained approximately 1 percent lead. The material contained approximately 80 percent soil and 20 percent battery cases. Based on this, the use of the reclamation technology was deemed not to be feasible, unless a suitable separation method for the cases and soil was developed. Although relatively simple size separation methods for separating the soil from the battery cases exist, they did not appear likely to be economically viable, because the soil contained significant rocks and other debris which would remain with the battery cases, and because a majority of the lead would stay in the soil. Therefore, the site was dropped from consideration by this study.

#### 2.3.2 Smelter Furnaces

Exide's secondary smelter has two reverberatory furnaces and two blast furnaces. Each reverberatory/blast combination Is similar in design and construction, as well as **production** potential. During the evaluation, one furnace (either the reverberatory or blast, de**pending on the** waste material) was charged with regular smelter feed and the other was charged with regular feed mixed with certain amounts of the waste material. This provided "control" and "test" furnaces, and comparisons could be made between the two. Because the furnaces normally undergo wide fluctuations in production, the comparisons were only valid when the furnaces were fed materials over four to six shifts.

Reverberatory furnaces are often used to remove metallic lead, and to produce a fused slag feed for the blast furnaces. The reverberatory furnace has a two to three hour residence time. The most serious problem encountered by introducing new feeds into the reverberatory furnaces was the stacking of unburnt material inside the furnace. This can cause impaired performance or even damage to the furnace. This condition takes from one to several hours to occur, so the furnaces were still monitored after the test feed was stopped.

Blast furnaces are used to reduce lead oxides to metallic lead, and remove them as product. The blast furnace has a residence time of several hours. Therefore the monitoring of the blast furnace charged with test material was continued for several hours after the waste feed is discontinued. This allowed time to more accurately determine if the test feed had any positive or negative affects on the blast furnace performance.

## 2.4 DATA AND SAMPLE COLLECTION

Project personnel collected samples and data to assess the furnace performance, characterize the input material, and characterize the furnace outputs. Table 2 shows the parameters that were measured and how they were obtained. The input material parameters were characterized to provide information related to the feed, so that comparisons of the effects of different feeds could be made. The furnace performance parameters, such as air flow, oxygen usage, fuel usage, and furnace feed rates provided measurements of the furnace performance while the experiments were conducted, principally indicating when production levels were falling or materials were clogging in the furnace. The output parameters were the most important ultimate measurements of furnace performance -- including both production rates and quality, as well as residuals generation.

The data generated from measurements and sample analyses were used to compare the performances of the test and control furnaces. The amount of lead in each product is useful in making a mass balance for the lead. The other parameters, such as oxygen, air, and fuel, are useful in determining the cost for processing the test material.

The procedures for obtaining the samples (marked "S") in Table 2 varied depending on the materials to be sampled. For samples of incoming waste materials, representative samples were obtained by compositing two to four individual grab samples obtained from various locations and depths within the pile. Generally, a total of one to two kilograms of such materials were obtained. Duplicate samples were obtained for approximately 25% of the samples. Samples of lead and slag produced in the furnaces were obtained through the use of a small crucible placed into the lead or slag stream.

Input Material Characterization	Furnace Performance Parameters	Furnace Output Parameters
total lead (S)	test material in the feed (M)	lead production rates (M)
sulfur (S)	air flow (M)	slag production rates (M)
silica (S)	% oxygen enrichment (M)	slag viscosity (0)
calcium (S)	fuel usage (M)	% lead in the slag (S)
moisture content (S)	lead inputs (S)	% sulfur in the slag(S)
density (M)	iron inputs (S)	back pressure (M)
particle size distribution (M)	% test material in feed (M)	sulfur aioxide emissions (M)
BTU value (S)		calcium sulfate sludge (S)

TABLE 2INPUT, OUTPUT, AND OPERATING PARAMETERS

(S)-Sample (M)=Measurement (O)=Operator Observation

Furnace performance and output parameters were monitored through a combination of review of operators notes and logs, and direct observation by CHMR personnel. In general, production data were obtained exclusively through operator logs, since that was the only form available for the data. These logs are accurately kept and monitored by plant management (who use the data for accounting purposes, and therefore require accuracy). CHMR obtained copies of the logs when such data were to be used.

## 2.5 QUALITY ASSURANCE/QUALITY CONTROL

The project was conducted in conformance with the approved Quality Assurance Projed Plan (QAPP), dated October 28, 1991.

The QAPP procedures related to several sets of parameters, including those listed in Table 3. The analytical or measurement methodology is also listed.

Some of the total parameters, including total lead, sulfur, iron, silica, calcium, were also determined using semiquantitative emissions spectrographic methods, which determined an approximate concentration of a wide range of compounds. These methods were employed when approximate measurements ( $\pm 30\%$ ) were sufficiently accurate, or as screening methods to determine if a wide range of elements were present in a sample.

TYPE OF PARAMETER	PARAMETER	ANALYTICAL OR MEASUREMENT TECHNIQUE
Test Material Parameters		
	Total lead	SW 846 6010 (digestion 8 ICP
	Total sulfur	ASTM D129-64 (digestion & ICP
	Total Silica	Spectrography
	Total Calcium	SW 846 7140
	Moisture	Drying/weighing
	Density	Weigh known volume
Furnace Perf. Parameters		
	Percentage Test Material in Furnace Feed (weight)	Ratio of charges, density of materials
	Air Flow to Furnaces	Plant rotameters
	Oxygen Enrichment	Plant setting
	Total furnace feed	Plant shift records/ observations
	Fuel Usage	Plant flowmeters
Furnace Output Parameters		
	Lead quality	Spectrography (measure impurity concentrations)
	Lead production rates	Plant shift records
	Slag production rates	Plant shift records
	Percent lead in slag	SW 846-6010
	Percent sulfur in slag	ASTM D129-64 or equivalent
	SO <sub>2</sub> Concentration in Stacks	Plant CEM (calibrated weekly)

TABLE 3 Methods Used to Measure Test Parameters

Quality control procedures for the laboratory analyses included quality control both within and without the analytical laboratory. For quality control within the analytical laboratory, CHMR reviewed the laboratory

standard procedures for conformance with the standard analytical methods. These procedures called for the use of periodic duplicates, spiked samples, and blanks.

Quality control procedures outside the analytical laboratory related mainly to in-plant measurements. Many of these measurements were obtained from plant shift data, which are presumably accurate. The production data was corroborated where possible by CHMR personnel observations -- how many charges of material were fed to the furnaces during a glven shift? When was the furnace operating? How long did they pour product or slag? These observations showed consistency between the numbers obtained and the practices observed. For example, over two shifts when CHMR personnel noted a 30 percent reduction in the number of charges fed to the furnaces, there was a corresponding 28 percent decrease in the weight of material fed to the furnace noted on the shift records, and a decrease in lead production of over 20 percent. Based on these observations, CHMR believes that the production data were accurate to within  $\pm 10$  percent.

Other observations, sutfur dioxide emissions data, for example, were obtained from instruments which are required by operating permits to be maintained and calibrated. Although it was not the focus of the study, CHMR personnel noted no obvious deviations from those requirements while on site.

Material density was obtained by weighing a known volume -- typically a carboy -- of material, and/or weighing an entire truckload of material as it was dumped at the plant site. This latter method was believed to be more accurate, because the volume used could be determined based on truck bed size before the material left the waste site and settled in the truck. From analyses of the feed samples from the Tonolli study see Table A-1), CHMR found a range of bulk densities of 23.2 to 28.4 lb/ft<sup>3</sup> (average 25.3 lb/ft<sup>3</sup>) using the carboy method, and 23.7 to 31.4 lb/ft<sup>3</sup> (average 27.6 lb/ft<sup>3</sup>) using the truck weight measurement. The overall average of the densities was 26.7 lb/ft<sup>3</sup>. The relative percent difference (RPD) (calculated as the difference between the duplicate analyses divided by the average of the two values), between the methods of calculating was 8.7 %.

The most pressing quality control issues related to the sampling. Battery case piles are notoriously nonhomogeneous, because the particles within them segregate both according to density and particle size. In one early experiment, for example, CHMR obtained total lead concentrations ranging between 3 and 16 percent.

To reduce errors introduced by the non-homogeneity, CHMR took composite samples from several areas within a pile (3 to 4). In addition, the samples were often taken of the material as it was loaded onto the truck for transportation to the smelter. This provided a more representative sample of material actually sent to the smelter.

During the NL Industries study (Appendix B), CHMR obtained duplicate analyses of eight input material an slag samples between two different analytical laboratories see especially Tables B-1 and B-5). The RPD for ead concentration ranged between 0.9% and 42%, with an average of 17.9%.

Overall, CHMR's estimates of the total lead concentrations and other parameters, where they are based on averages from several samples, are estimated to be within 20 percent of the actual value.

# 3.0 RESULTS AND DISCUSSION

In general, the study demonstrated that various materials may be processed in secondary lead smelters with relatively few effects on overall furnace performance. The most significant effects were caused by processing materials in a furnace without property pre-processing it, or by processing too much material at one time. For example, the battery case pieces in the Tonolli feed were so large that they burned too slowly in the reverberatory furnace, causing a reduction in the furnace production rate. Later, battery case material from the Hebelka site was successfully processed in the reverberatory furnace after it had been shredded in a hammermill to a particle size of less than <sup>1</sup>/<sub>4</sub>-inch. The NL Industries material was initially unsuccessfully processed at 100 percent feed ratio because it was too dense for the feed system. When the ratio of test material to total feed was lowered to 50 percent, the material was processed with few problems.

More extensive summaries of the evaluations from these sites (NL Industries, Tonolli, PennDOT, Hebelka and HUD) may be found in Appendices A through E, respectively.

#### 3.1 **PRODUCTION PARAMETERS**

The lead and slag production from the furnaces varied with each evaluation. Table 4 shows three lead and slag production parameters derived from the daily production of lead and slag at the smelter. The demolition matenal evaluation is not included in Table 4 because this test was performed primarily to qualitatively determine if the furnaces could process the light, wooden material.

	Γ	Evaluation				
Furnace	Parameter	Tonolli	Hebelka	NL Industries	PennDOT	
Reverb	WL % Test Feed	N/F	17%	20 to 50%	N/F	
	% Pb in Test Feed	N/F	14.7%	57%	N/F	
	Pb Prod. Ratio	N/F	1.20	0.90	N/F	
Blast	WL % Test Feed	10%	N/F	30%	13%	
	% Pb in Test Feed	3.5%	N/F	30%	3.2%	
	Pb Prod. Ratio	1.3	0.9	1.25	1.1	
	Pb Loss Ratio, L <sub>R</sub>	0.6	•	1	2.6	
	Slag Ratio	0.97	1.00	0.97	0.97	

TABLE 4LEAD AND SLAG PRODUCTION COMPARISONS

Composition of blast slag was not analyzed

N/F means that the test material was not fed to that furnace.

## 3.1.1 Lead Production Ratio

The Lead Production Ratio is the ratio of the amount of lead produced by the furnace processing test material to that produced by the furnace processing regular feed. This parameter can be determined for the reverberatory furnaces only when the furnaces are fed waste material, but can be determined for the blast furnaces in all cases, since the blast furnaces indirectly receive the test feed through the reverberatory slag. A value greater than 1 indicates that the furnace processing the mix of test and regular feeds produced more lead than the control furnace.

The Ratios for all the other evaluations were different than 1, but not significantly so given the relatively short durations of each of the tests as compared to the three month NL Industries evaluation.

For the NL Industries material, the lead production ratio for the reverberatory furnace is consistently below 1, averaging 0.9 over the three month period in which the materials were added to the furnace. However, during the same time period, the blast furnace associated with the "test' reverberatory furnace **p**roduced significantly more lead than its control counterpart. It appeared that the use of the test material **sh**ifted the production of lead from the reverberatory to the blast furnace. This shift is attributed to the relatively high concentration of antimony and lead oxides in the waste feed material. Antimonial lead and lead oxides melt at higher temperatures than elemental lead, and are therefore more likely to be removed in the blast furnaces, which operates at a higher temperature than the reverberatory furnaces.

#### 3.1.2 Lead Loss Ratio

The secondary lead smelter has two major outlets for the lead input: production and blast furnace slag (waste). The Lead Loss Ratio  $(L_R)$  Is the ratio of the loss of lead in the slag from the test furnace (total slag produced in the test furnace multiplied by lead concentration in that slag) to the loss of lead in the control fumace. This ratio can only be calculated for the blast furnaces because the blast slag is a waste product from the process. An  $L_R$  value greater than 1 means that more lead is lost in the blast slag produced by the furnace(s) processing test material than in the control furnace(s).

The Tonolli evaluation produced a much lower  $L_R$  than 1, while the value for the PennDOT evaluation was 2.6. The composition of the blast slag produced in the Hebelka evaluation was not determined. The apparent low value during the Tonolli experiment is most likely due to normal variations in the furnace operations and sampling procedures, and it is not attributable to a significant increase in efficiency. Over the course of the experiment with the PennDOT materials, the lead concentrations in the slag were high and appeared to be climbing in both furnaces, even though the furnaces were not fed the same feed. Therefore it is difficult to determine how significant the apparent increase in lead loss was. However, there is reason to believe that some of the increased  $L_R$  was due to the feed, since approximately twice as much iron than is usually fed was added during the course of the experiment, and additional iron binds lead to the slag. Lead concentration in the slag and quantity produced when the NL Industries site material was processed were not different than when typical feed was processed. Since this was the longest evaluation in terms of time, this may show that most material will have no effect on furnace performance in the long run.

### 3.1.3 Slag Disposal Ratio

The Slag Disposal Ratio ( $S_R$ ) is the amount of blast slag produced by the test furnace divided by the amount of slag produced by the control furnace. This is another term that is exclusive to the blast furnaces because reverberatory slag is fed to the blast furnace, while the blast slag is disposed. In all of the evaluations, the  $S_R$  was effectively 1, which means the test and control furnaces produced nearly equal amounts of blast slag.

#### 3.2 FEED RATIOS OF SUPERFUND MATERIALS TO TOTAL FEED

Secondary lead smelters normally accept feed materials derived principally from the breaking and demolition of lead-acid batteries. These normal feedstocks consist primarily of lead grates, terminals, and sludge from the batteries. This material typically contains 60 to 70 percent lead, by weight with the remaining materials consisting of plastic, rubber, wood scrap, sulfur, dirt and residues from the batteries, and moisture and oxides combined with the lead. Superfund wastes and other materials, which contain lower concentrations of lead and higher concentrations of other constituents that may be harmful to the furnaces, must be blended with "normal" feed prior to being led to the furnaces.

The mix ratio of normal feed to waste materials was one of the parameters tested during this study. Based on furnace performance and operations results, it was possible to determine whether the furnace performed successfully or unsuccessfully at any given feed ratio. A test is considered to be unsuccessful if the feed ratio of test material has to be lowered, or discontinued altogether.

The tests show that the successful mix ratios may be correlated with the percentage of lead in the waste material. Figure 3 shows the feed ratios which were successful, plotted against the percentage of lead in the waste material (i.e., prior to blending with normal feed). The results are nearly linear from streams containing 3 percent lead to those containing 60 percent lead. This delineates the region marked

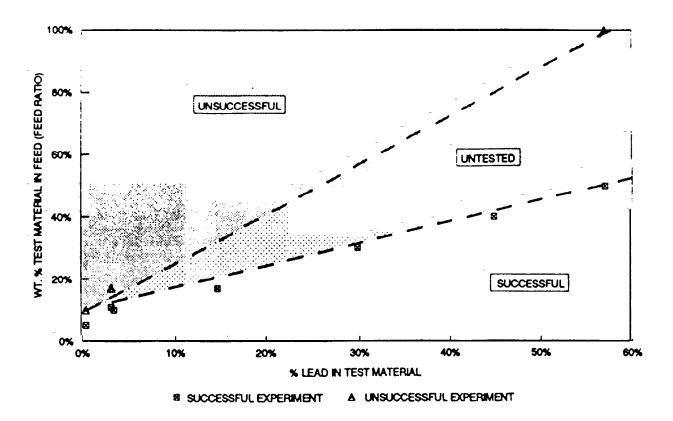


FIGURE 3 PERCENT LEAD IN TEST MATERIAL VERSUS FEED RATIO

the waste material (i.e., prior to blending with normal feed). The results are nearly linear from streams containing 3 percent lead to those containing 60 percent lead. This delineates the region marked "successful" on the figure. When the feed ratios versus lead concentration for unsuccessful runs are plotted, a second region (the "unsuccessful" area) emerges. Finally, a third region, in which no tests were performed is also marked on the figure. No test feed with more than 60 percent lead was fed to the furnaces, so the regions in the range above 60 percent lead cannot be determined from the experimental results.

It is important to note that the test results represent an empirical observation based on the tests performed. Some of the correlation can be easily accounted for-- lead concentration affects density for example, and material which is too dense is not easily conveyed into the furnaces, while material which is too tight will fill up the furnaces and cramp out other feeds. Much of the correlation, however, is not easily accounted for, and is simply based on furnace operator observations. CHMR believes that further study may help delineate the "untested" region, but that it may be more practical to simply perform a treatability study on a given feed to more accurately determine the acceptable feed ratio.

## 3.3 BENEFICIAL EFFECTS OF CERTAIN MATERIALS ON FURNACE PERFORMANCE

Some materials, particularly ebonite battery cases (which contain coal or coke dusts), represent a potentially significant source of energy to the furnaces. The battery case materials had average BTU-values of over 11,060 BTU per pound. Coke is a typical feed to a blast furnace. Ebonite rubber cases were successfully substituted for a portion of the coke in the blast furnace. Battery cases will not substitute for all of the coke since the large chunks of coke provide structure inside the furnace, which battery cases cannot provide.

Other materials represent substitutes for other furnace feeds. Iron is typically fed to a blast furnace to help separate lead from the slag. The PennDOT bridge blasting material represented a source of iron and calcium in the blast furnace. The material contained so much iron (over 60 percent) and so little lead in comparison (approximately 3 percent), that it may be considered an iron source which happens to be contaminated with lead, rather than a lead source to the furnace. In this capacity, it represents a potentially beneficial reuse for a material which most state and municipal authorities have found difficult to dispose. This material would have to be added at a slower rate than during the test run to be used as an iron source

#### 3.4 APPLICABILITY OF LEAD RECOVERY FROM WASTE MATERIALS

Broadly speaking, lead reclamation in secondary smelters is an applicable technology whenever sufficient lead exists in a form which can be economical retrieved from the Superfund material. However, there are certain materials, such as materials containing large amounts of soil, In which the lead may not be easily reclaimed using the technology. This section describes the procedures to be used to determine applicability and the range of applicability determined by the experiments.

Generally, to determine if a material can be processed through a secondary lead smelter, a two step procedure is required: material characterization followed by a pilot or treatability study at the smelter. Table 5 lists the typical parameters which must be included in a material characterization. Not all these parameters were obtained or measured during the study: many are based on requirements obtained from the smelter. Note that the smelter operators are not interested in the TCLP leachability of the metals in the waste, but the total metal content. The leachability only determines the regulatory status (hazardous, non-hazardous) of the material, and does not affect its properties in a smelter.

The table is intended to provide an approximate gauge of the materials that can be processed through the smelter. Because there is such a wide range of lead-bearing materials found in the environment, it is probably necessary to discuss a particular waste feed with smelter personnel before deciding whether the lead is suitable for reclamation. Some wastes may be blended with other materials to make suitable feedstock. A high calcium waste, for example, may be blended with other materials with little or no calcium and fed to the furnace. Of course, this blending may significantly increase the cost of storage and processing. Other materials, such as metallic aluminum, are prohibited in furnaces.

After characterizing the waste material to determine its suitability in a smelter, a pilot-scale study may be necessary to better determine the effects on the furnaces and the appropriate operating parameters. These will likely have significant bearing on the economics of treating the material. The demolition material tested during this study, for example, had to be shredded and blended relatively slowly with normal furnace feed. This increased the handling and storage requirements by more than 50 percent. A more detailed cost analysis is presented later in this paper.

Constituent	Lower limit (lf applicable)	Upper limit (If applicable)	What does parameter affect?
Total lead	1%	100%	Economics (based on lead recovery), feed rates
Total antimony	N A	N A	Needed to determine refining requirements
Total arsenic	N A	2 %	Lead quality, refining
Total copper	NA	2%	Lead quality, refining
Total halogens	NA	<1%	Damage flue gas system, permits
Total tin	NA	2%	Lead quality
Total iron	NA	none	Feed rates (iron is a furnace feed)
TCLP - metals	NA	NA	Overall need to process
Physical state	No liquids or gases	No liquids or gases	Conveyance, furnace performance, permits
Total sulfur	NA	10%	CaSO <sub>4</sub> sludge production, sulfur emissbns
Total silicate	none	20%	Furnace performance
Total soil content	none	25%	Furnace performance, economics, slag production
Fuel value (BTU/lb)	none	NA	Economics
Total calcium	none	10%	Furnace performance, feed ratios
Total aluminum	none	1%	May cause explosions in the furnaces
Particle size	5 microns	lm	Loading, conveyance
Oil and Grease	none	<2%	Permits, feed system, material handling
Moisture	none	15%	Heat requirements, may cause steam explosions
Density	none	none	Loading, conveyance, economics
Ash content (excluding lead)	none	70% (est.)	Slag production, furnace performance, economics

 TABLE 5

 REQUIRED CHARACTERIZATION PARAMETERS

# 3.5 LEAD RECLAMATION EFFICIENCY

In determining whether the process is suitable for Superfund activities, it is important to determine if the secondary lead smelting process actually reclaims lead from the various materials. Unfortunately, there was no way to precisely measure the extent to which the lead from the Superfund materials is reclaimed after these materials are mixed with regular furnace feeds. Therefore, a methodology was developed to estimate the minimum reclamation efficiency based on conservative assumptions regarding the partitioning of lead inside the furnaces.

Generally, lead inputs to the smelter include feeds to the reverberatory and blast furnaces. During normal smelting operations, outputs from the smelter include [29]:

- Lead production from both furnaces (usually between 99.5 and 99.7 percent of the lead feed);
- Blast furnace slag (which contains 0.3 to 0.5 percent of the lead feed);
- Calcium sulfate sludge from the emissions control system (which contains approximately 0.004 percent of the lead feed); and,
- Fugitive and stack air emissions (which contain less than 10<sup>-5</sup> percent of the lead feed).

The furnaces reclaim over 99 percent of the lead fed to them. The only significant outlet for lead besides lead product is the slag, which must be landfilled. For the purposes of estimating reclamation efficiency, the slag may be estimated to contain 1.5 to 2 percent lead, which the literature suggests can be maintained over a variety of operating conditions [30].

The mininum reclamation efficiency can be estimated noting that the lead partitions between the lead product, which is essentially pure lead, and slag, which contains 1.5 percent (by weight) lead. The percentage of any feed material partitioning to the slag is approximately equal to the concentration of non-lead, noncombustible materials in the feed (which is approximately equal to the ash content minus the lead concentration in the feed). Thus, for test material with a measured ash content of 50 percent, and 30 percent lead, it is reasonable to assume that the remaining 20 percent of the feedstock will become part of the slag. If we further assume that the slag produced by Superfund materials is indistinguishable from that produced by normal feeds operating under the same furnace conditions, and therefore will contain the same weight fraction of lead as the slag. This can be compared with the original amount of lead in the Superfund material to determine a reclamation efficiency:

#### $\epsilon = 1 - [Pb_{e}] ([A] - [Pb])/[Pb]$

where  $\varepsilon$  is the minimum reclamation efficiency; [Pb<sub>2</sub>] is the lead concentration in the slag; [A] is the ash content of the test material (as a weight fraction); and [Pb] is the weight fraction of lead in the feed. Based on this estimate, the minimum reclamation efficiencies for the various feedstocks used during the experimental program ranged from a high of 99.5 percent for the Pedricktown material with 45 percent lead and 60 percent ash content, to a low of 10 percent for the PennDOT material, with an ash content of 70 percent, a lead concentration in the slag of approximately 3 percent, and a lead concentration in the slag of approximately 4%. (Note that the lead concentration in the slag was especially high dunng the time that the PennDOT material was run for reasons which appeared to be independent of the PennDOT feedstream. The lead reclamation efficiency would normally be considerably higher.) The results show that lead reclamation always occurs, even when minimum reclamation conditions are assumed.

# 4.0 LEAD RECLAMATION ECONOMICS

The cost of using secondary lead smelters for the recovery of lead from Superfund sites is determined by the cost of the three basic operations:

- On-site excavation and preprocessing of the material (C<sub>F</sub>)
- Transportation (C<sub>T</sub>)
- Processing at the smelter (C<sub>P</sub>)

These costs are discussed in further detail below.

#### 4.1 ON-SITE EXCAVATION

Excavation costs for Superfund materials vary depending on the health and safety requirements for given sites, contaminants and contaminant concentration. For battery cases, or lead debris, dross, etc., which are relatively accessible on the site, and require little more than dust control during excavation, the costs range between \$5 and \$15 per cubic yard. On-site costs, denoted as  $C_{E}$ , will Increase if additional on-site processing is required.

#### 4.2 TRANSPORTATION

Transportation costs for hazardous wastes are dependent on the amount of material transported, and the distance transported. Typical costs range between \$0.20 and \$0.35 per ton-mile (transport 1 ton 1 mile). For most material, the cost of transportation,  $C_{T}$ , is estimated as:

#### $C_{T} = $0.3 \text{ D/mile}$

where D is the distance from the site to the smelter in miles. Note that for longer distances (over 300 miles), alternate means of transportation (i.e. tandem trucks, rail shipment, etc.) may be more economical.

The transportation economics shift slightly for some materials, particularly those with specific gravities significantly less than 1. These materials may include whole (i.e., uncrushed) battery cases which, because of their structure, have relatively large void volumes and therefore a low density. Unless these materials are crushed on-site, the transportation costs will be based not on their weight but on their volume. This will necessitate a correction factor  $\Phi$  on the transportation cost equation which is equal to the inverse of the material bulk density in ton per yd<sup>3</sup>, and a maximum of 1. Thus, the overall transportation cost can be estimated as:

## $C_{T} = 0.3 \ \Phi D$

where the dimensionless correction factor  $\Phi$  is equal to max (1/p,1), in which p is expressed in dimensions of ton per yd.<sup>3</sup>

#### 4.3 PROCESSING AT THE SMELTER

Based on the results of the study, processing costs for the materials vary significantly depending on the concentration of lead, the market price for lead, and the percentage of the feed which becomes slag. As the market price for lead or the concentration of lead in the feed material decreases, the cost of processing Superfund materials will increase, because the lead represents a salable commodity generated during the reclamation. If the material contains a greater fraction of constituents which exit the furnace in the slag fraction, then the cost increases commensurate with the disposal costs of slag. Most of the other parameters (for example, a slight increase in oxygen usage in the furnace) have little overall affect on the cost of processing.

CHMR has developed a model for estimating the costs associated with processing Superfund materials at a smelter. The model breaks the cost into four major categories: a base cost (per ton) to cover a portion

of normal smelting costs, additional production costs specific to the material, additional disposal costs, and offsets of the value of the recovered lead and reductions in other feeds (if any) to the furnace. These are described further below.

## 4.3.1 Base Cost C<sub>B</sub>

**According** to the literature [12, 17, 27, 29] the cost of processing material in a smelter is approximately **\$0.10±0.02** per pound or **\$200±** 40 perton of material processed. Note that this is not the cost of producing a pound of lead which is typically estimated at between **\$0.12** and **0.17** per pound, but is the cost of processing a pound of feed. The two are related, since furnace feeds [including coke, iron and limestone] contain 55 to 65 percent lead. This cost includes the cost of handling and loading the material, maintaining the smelter, normal furnace inputs (coke, natural gas, oxygen, etc.), labor costs, environmental compliance costs, etc.

# 4.3.2 Additional Production Costs Cac

These costs include additional labor and equipment costs associated with handling an atypical feed at the smelter. Included in this category are costs associated with maintaining a staff member on-site during loading to ensure that the proper materials are loaded onto the trucks (estimated at \$6 per ton); labor costs associated with inspecting, unloading, and mixing the material at the smelter (\$10 per ton); costs for additional management time and attention for the material (\$3 per ton); additional capital costs at the smelter, including loading equipment, storage areas, and piloting costs (estimated at \$18 per ton); legal and contractual costs associated with handling Superfund materials (\$10 per ton); and miscellaneous costs, such as analytical costs (estimated at \$10 per ton). These additional production costs remain relatively fixed independent of the amount or types of matenials processed, at **\$60±10** per ton.

For light materials, the additional processing costs will be based on their volume, rather than weight. More than likely, however, the smelter will choose to crush or grind them before feeding them to the furnaces, at a cost of between \$10 and \$15 per cubic yard. To denote this, a correction factor  $\Psi = $15$  per ton is applied when the bulk density of the material is below 0.8 tons per yd<sup>3</sup>. If the density is above 0.8 tons per yd<sup>3</sup>,  $\Psi = 0$ .

## 4.3.3 Additional Disposal Costs C<sub>disp</sub>

Additional disposal costs associated with processing Superfund materials include the disposal of slag and calcium sulfate sludge. Based on measurements taken at the smelter, CHMR determined that typical smelter feeds produce in the range of 200 lbs of slag per ton of feed 10 percent slag in the feed), and 60 lbs of sludge per ton of feed (based on 3 percent sulfur in the feed). This material is typically disposed of at a cost of \$150 per ton. The cost to dispose of 200 lbs of slag and 60 lbs of sludge is included above as part of the overall cost to process the material. However, some Superfund and waste materials may contain significantly higher percentage of compounds which will end up in the slag, or sulfur which will produce sludge. Therefore, the overall processing cost must be adjusted to include the cost to dispose of the waste materials produced by the feed. This adjustment is calculated as:

$$C_{disp}$$
 \$150 ([N] - 0.1) + \$100 ([S] - 0.03)

where [N represents the percentage of non-combustible, non-lead, and non-volatile material in the superfund material, and [S] is the sulfur content of the feed. [N] can then be calculated by:

where [A] is the ash content of the waste material, measured at or above 1500°F, and [Pb] is the percentage lead in the material. The correction factor to the lead concentration corrects for the appearance of lead oxides in the lead. The overall disposal cost differential is therefore:

4.3.4 Offsets for the Value of Lead and Reduction of Other Furnace Feeds

The value of the recovered lead in a waste material is given by:

$$C_{Pb}^{=} - \epsilon_r P [Pb]$$

where  $E_r$  is the reclamation efficiency for the feed in question (assumed to be approximately 1), P is the market price for lead in dollars per ton and [Pb] is the weight fraction of lead in the material. The negative sign on the cost indicates that this is not a "cost" per se, but in reality a credit for the lead content to the smelter. The January 1994 market price for lead was approximately \$700 per ton. This is down from over \$750 per ton in 1991, but up from \$450 per ton in 1992. The volatility in the price of lead is due to large sales of lead from the former Soviet Union and the price is anticipated to stabilize during 1994 [32].

A second benefit from Superfund materials is decreased coke usage. For materials such as battery cases, which can be essentially a supply of coke to the furnace, the change in required coke is related to the heat value of the Superfund materials (which in this discussion will be denoted by [BTU] and given in units of BTU per pound). If coke were used only as a fuel source inside a furnace, and if the battery case materials burn similarly to coke, then the reduction in coke usage based on addition of a Superfund materials would be equal to the ratio of the BTU value of the Superfund material to that of coke (i.e., if you feed 1 BTU of energy to the furnace from the Superfund material, you would save 1 BTU's worth of coke in the furnace). However, coke is not used only as a fuel source, but also to provide structure to the materials inside the furnace. In addition, battery cases, with different sizes and shapes, do not burn at the same relativity homogenous rate as coke input to a furnace. Therefore, a one-to-one reduction in coke requirements for the Tonolli feed, in which a 10 percent feed of a material with a heating value of 12,000 BTU per pound allowed for a reduction of 30 percent in the coke usage, CHMR estimates that approximately 25 percent of the heat-value of a Superfund waste can be supplied to offset the blast furnace requirements for coke. Based on this, the BTU-value of coke (13,000 BTU per pound) and the market price or the coke used in the furnace (approximately \$150 per ton), the cost savings from the BTU-value of the Superfund material can be estimated as:

wher e  $C_{coke}$  is in terms of dollars per ton of feed material. The cost savings include savings from not having to add the coke as part of the base feedstock to the furnace. The 2,000 BTU per pound differential in the equation is included because below a minimum level, the material probably provides no realizable benefit to the furnace, and may even increase fuel requirements because of its thermal capacity.

Likewise, the content of a Superfund material may provide significant benefit to the furnace. The extent of reduction allowed by the addition of Superfund materials containing iron could not be estimated from the experiments conducted, because the smelter fed the material at a rate significantly higher than they usually feed iron. However, assuming a 75 percent tradeoff from metallic iron input to the furnace, and based on the market price of scrap iron (\$130 per ton), the potential cost avoidance savings are:

where [Fe is the weight fraction iron in the feed. Note that the equation predicts that there is no benefit if the iron fraction is below 0.1. The formula includes a correction to avoid double counting the iron which needs to be disposed.

Thus, overall cost benefits and avoidance  $C_A$  based on the lead in the Superfund material and reductions of coke and iron usage is:

$$C_A = -P [Pb] - 0.007 ([BTU] - 2,000) - 360 max([Fe] - 0.1, 0)$$

with [BTU] in BTU per pound and [Fe] as weight percentage metallic iron in the feed, and the lead reclamation efficiency assumed to be 1.

#### 4.3.5 Net Smelter Processing Cost

The net cost to process the waste materials in a secondary lead smelter, is therefore:

$$\label{eq:C_p} \begin{split} C_{\rm P} = C_{\rm B} + C_{\rm apc} + C_{\rm disp} + C_{\rm A} \\ C_{\rm P} = 256 + 150 \ \mbox{[A]} \cdot (165 + \mbox{P}) \ \mbox{[Pb]} + 100 \ \mbox{[S]} \cdot 0.007 \ \mbox{[BTU]} - 360 \ \mbox{max}(\mbox{[Fe]} - 0.1,0) \end{split}$$

where  $C_P$  is in units of dollars per ton of material.

#### 4.4 OVERALL PROCESS ECONOMICS

The overall process economics can be determined by combining the various components (excavation transportation, and processing the materials):

$$C_{Tot} = C_E + C_T + C_P$$

where  $C_{Tct}$  is the overall cost of processing in dollars per ton. Fixing the cost of excavation and required pre-treatment at \$15 per ton, inserting a function of distance (D) from the smelter (in miles) for transportation costs, and combining the processing cost for the material,  $C_{Tct}$  becomes:

$$C_{Tot} = 271 + 0.3\Phi D + \Psi + 150 [A] + 100 [S] - (165 + P) [Pb] - 0.007 [BTU] - 360 max([Fe] - 0.1, 0)$$

with  $C_{Tot}$  in dollars per ton of material. [A] is the percent ash content of the waste material; [S], [Pb], and [Fe are the percent sulfur, lead, and iron concentrations, respectively. [BTU] is the heating vaue in BTU: an P is the price for lead in dollars per ton.

Correction factors  $\Phi$  and  $\Psi$  are functions of the bulk density of the material (in ton per yd<sup>3</sup>) where  $\Phi$  = max [1/  $\rho$ , 1) and  $\Psi$  = 0 if p> 0.8 ton per yd<sup>3</sup> and \$15 per ton if  $p \le 0.8$  ton per yd<sup>3</sup>. Neither correction factor will likely apply if the material is crushed on-site to increase its density and decrease handling costs before being shipped to the smelter, but, of course, the cost of this crushing must then be added to pre-processing and excavation costs.

Based on this cost model, the overall cost of processing the materials from the sites studied during this research, have been calculated, and are presented in Table 6. The table includes two costs, the first based on a conservative market price for lead (650 per ton) and the second based on a more plausible long-term cost for lead (\$750 per ton). Note that the overall cost of using secondary lead smelters as a treatment technology for Superfund sites is dependent on the lead content and market price for lead and total ash content (i.e., slag generation potential) of the material.

Site	P=\$650/t Cost/ton	P=\$750/t Cost/ton	Distance D (miles)	% Ash [A]	% Lead [Pb]
Tonolli	\$228	\$224	40	20	3.5
Hebelka	\$174	\$160	75	30	14.7
Demolition Material	\$374	\$373	100	4	1
NL Industries	\$80	\$35	200	65	45
PennDOT	\$231	\$228	250	70	3.2

TABLE 6 COST OF REMEDIATING SITES

# 5.0 CONCLUSIONS

Lead is a common contaminant at Superfund sites, present at between 30 and 50 percent of the sites, and reclamation is one viable option for remediating the sites. The following conclusions may be drawn from the study of reclamation of lead from Superfund sites using secondary lead smelters.

- Lead was successfully reclaimed in a secondary lead smelter from a variety of materials, including battery case pieces, dross, lead debris, spent abrasive materials, and demolition material contaminated with lead paint. The lead concentration in these materials ranged form 1 to 45 percent.
- The economics of reclaiming lead from Superfund sites are dependent on lead concentration, the market price for lead, distance from the smelter, the amount of materials which become incorporated into slag from the process, iron content, BTU-value of the wastes, and to a lesser extent, sulfur concentration.
- The cost for recovering lead from the five sites selected for this project, based on a conservative price for lead (\$650/ton), ranged between \$80 and \$374 per ton of materials.
- The Superfund material must generally be mixed with regular furnace feed prior to being processed. The acceptable weight ratios of the Superfund material to overall furnace feed (Superfund plus regular materials) were found empirically to be a function of percentage lead in the Superfund materials, with the approximate linear function: Mix ratio = 0.1 + 0.5[Pbfeed], where [Pb<sub>feed</sub>] is the concentration of lead in the Superfund material.

Overall, CHMR concludes that secondary lead smelters provide a viable alternative to stabilization and disposal for the treatment of wastes found at battery breaker and secondary lead smelter Superfund sites. as well as for other commonly found lead-containing waste streams.

Factors in selecting reclamation using secondary lead smelters for the materials found at Superfund sites include:

- Lead concentration:
- Material type (dross, debris, slag, soil, etc.);
- Density of the material;
- Sulfur, calcium, iron, antimony, soil, aluminum, and silica concentrations;
- Moisture content (smelters generally cannot accept wet materials); Presence of organic wastes, haiogenated materials, and gaseous materials (which typically are prohibited in smelters); and, BTU-value.

In all cases, a thorough analysis of the site materials is anticipated to be necessary before the material is accepted. In some cases, a treatability study to determine the allowable mix ratios and effects of the material on the furnaces may be necessary before a stream is accepted by the smelter.

## 6.0 RECOMMENDATIONS FOR LEAD RECLAMATION AS A REMEDIAL APPROACH

Table 7 provides a comparison of the use of secondary lead smelting to other remediation technologies described in the paper for a variety of feeds. The table includes all costs associated with processing each feed in the technology indicated. The table shows that secondary smelting is the most economical alternative for lead debris, and competitive for battery cases.

Secondary lead smelting has other advantages over the alternative technologies, including:

- It is a permanent solution The lead is reclaimed and put back to its original use. The long term effects of other technologies, such as landfilling or stabilization, are still uncertain.
- <u>It reduces liability</u> Once the material is processed in the smelter, it is no longer a waste, but a product permanently ending the long term CERCLA liability of disposing the waste.
- <u>It uses existing technology</u> This eliminates the need to develop mix ratios for solidiiition or stabilization
- <u>Smelter receptivity</u> In a limited informal survey, CHMR found that virtually all secondary lead smelters are permitted to accept lead-containing hazardous wastes, and that approximately half were amenable to doing so.

CHMR estimates that the total amount of battery case material and other debris which could be processed annually in the U. S. secondary smelters is approximately 300,000 tons. This is based on 25 tons per day per furnace, and 280 production days per year. It appears at this point that several U. S. smelters are currently planning to accept Superfund materials.

Technology	Media	Price Range/ton	Comments
Stabilize/Capping	Soils Battery Cases	\$30-100 \$50-150	Limited application
Stabilize/Disposal	Soils	\$50 - 200	
Physical Separation	Soils Battery Cases	\$60-200 \$60 250	Process + dispose
Extractive Washing	Soils Battery Cases	\$60 - 250 \$75 - 300	Metal Recovery
Secondary Lead Smelting	Battery Cases Debris Other Materials	\$100- 250 \$60 - 200 \$100- 300	Metal Recovery

#### TABLE 7 COMPARISON OF SMELTING AND OTHER TECHNOLOGIES

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# APPENDIX A

# RECLAMATION OF MATERIALS FROM BATTERY CASE PILES AT THE TONOLLI SUPERFUND SITE IN NESQUEHONING, PA

# Description of Evaluation and Results and Discussion

July 23, 1992

# 1.0 DESCRIPTION OF EVALUATION

The purpose of the evaluation was to determine the feasibility and economics of the recovery of lead from battery case materials. The Tonolli Corporation Superfund site in Nesquehoning, Pennsylvania was used as the source of these materials. The materials were processed at Exide's Reading, Pennsylvania, secondary lead smelting facility.

Exide's Reading smelter is primarily engaged in recycling lead from lead-acid batteries, such as those utilized in automobiles and the recycling of polypropylene rom battery cases for subsequent reuse. Exide uses both reverberatory and blast furnaces to recycle lead. These furnaces and other plant operations are typical of the secondary lead smelting industry.

This appendix includes a description of the evaluation, and a discussion of the results. The conclusions from this evaluation have been ncorporated into those found in the main body of this report.

#### 1.1 EXPERIMENTAL DESIGN

During the evaluation, the material from the Tonolli site was fed to both the reverberatory and blast furnaces. Initially, the test material was fed directly to each furnace using a front-end loader, which loaded one toad of test material, then several loads of regular feed (depending on the mix ratio), then another bad of test material, and so on. However, it became apparent that the furnaces would better handle the test material if it were initially mixed with the regular feed, and the resultant mixture charged to the furnaces.

The plant is equipped with two blast and two reverberatory furnaces. Generally one reverberatory furnace and one blast furnace were utilized for processing the Tonolli material, which the second reverberatory furnace and blast furnace remained in normal, routine operation. Conducting the test in this manner allowed for comparison of the operation of the lest" furnaces with the "reference" or "control" furnaces.

During the evaluation, the operating parameters (fuel usage, oxygen usage, slag production, lead production, etc.) for each set of furnaces were obtained. In addition, samples of slag and lead were obtained from each furnace to be used to determine effects on production quality.

#### 1.2 CHRONOLOGY OF EVALUATION

The following represents a chronology of the activities conducted in support of the evaluation, between September 5 and 13, 1991.

#### 1.2.1 Material Acquisition and Sampling

On September 5, 1991, five dump trailers of material were loaded with battery case materials from the Tonolli Superfund site in Nesquehoning, Pennsylvania. Because the battery case materials were stored in piles at the Tonolli site and no separation of materials was necessary, no problems were encountered during loading activities. No dusting problems were encountered during loading activities. A total of 167,750 pounds of material was transported in five dump trailers, ranging from 42 to 48 cubic yards of material per trailer.

Composite samples of each battery case pile were collected during the loading process at the Tonolli site. The samples were subsequently analyzed to determine:

- bulk density;
- percent of the hard rubber material in the total sample;
- percent of polypropylene plastic in the total sample;
- percent of other miscellaneous material (i.e. soils, concrete, etc.);

- percent of metallic lead in the total sample;
- lead concentration using the Toxicity Characteristic Leaching Procedure (TCLP) on rubber cases;
- total sulfur; and
- percent of lead contained in the rubber, soil, concrete, etc.

The analytical results of this material are presented in Table1 A-1. A discussion of these results appears later in this report.

#### 1.2.2 Major Activities

This section describes the major activities that occurred during this evaluation. These activities are presented chronologically starting with the first day of processing through the smelter.

#### Day One

Activities completed during the first day of the test included density determination of test material, which was determined by weighing trucks as they entered and exited the plant, and dividing by the truck bed volume. One blast furnace was charged at a 7 percent test material feed ratio and one reverberatory furnace was charged at an 11 percent feed ratio, in accordance with the QAPP. The test material feed ratio is calculated by dividing the weight of "test" material charged to the furnace by the total weight of feed (including "test' material and typical feed).

#### Day Two

Early in the second day, both blast furnaces were running sluggishly and there was a crust accumulating within the reverberatory furnace which was being charged with test material. Based on the amount of coke added during the previous night, the plant management concluded that the blast furnaces were overcoked. This was unrelated to the evaluation, but Exide management still decided that it would be best to refrain from charging additional test feed to the blast furnaces until the build up within the furnaces was reduced. The accumulation of the crust in the reverberatory furnace was determined to be related to the experiment. Therefore, test feed input to that furnace was suspended.

Sample	Type of Sample	Bulk Density (lb/tt <sub>3</sub> )	Hard Rubber (wt %)	Plastic (wt %)	Concrete Soil, etc. (wt %)	Sulfur (wt %)	Metallic Lead (wt %)	Total Lead (wt %)	TCLP Lead (mg/L)
1	Composite from trucks taken at site	24.4	76.3	22.5	1.0	3.10	0.25	0.34	130.9
2	Composite from trucks taken at site	23.2	70.7	27.8	2.6	†	0.05	0.82	111.8
3	Composite from trucks taken at site	16.9*	67.9	27.5	2.6		0.05	0.47	131.3
4	Composite from trucks taken at site	28.4	79.9	18.0	2.0		0.10	1.57	159.4
5	Composite taken at smelter	-		•	-	1.93		15.7	
6	Composite taken at smelter					2.71		7.3	
7	From piles on site	•						1.15	
8	From piles on site (duplicate of 7)	hate			3.0	-	0.10	0.28	•

# TABLE A-1 ANALYTICAL RESULTS FROM TEST MATERIALS

\* Value was found to be anomalous by q-test at 90% confidence limit

Notes: Samples 5 and 6 contained noticeable quantities of reddish sludge, which apparently was a lead oxide compound. Therefore the total lead concentration In these samples is significantly higher than those for other samples.

Additional bulk densities were calculated by obtaining weights and volumes from the trucks. These were calculated to be: 29., **27.3**, **23.7**, 31.4 and 26.6 lb/ft<sup>3</sup> respectively on trucks 1 through 5. The overall average of all the values obtained was  $26.7 \pm 1.7$  lb/ft<sup>3</sup>.

The test material was not fed into the blast or the reverberatory furnace again until later in the afternoon. At that time, the test feed was introduced at an 11 percent weight ratio into blast furnaces #1 and #2 and reverberatory furnace #1. The test material was fed to both blast furnaces to ensure that both furnaces would contain the same material at the start of the third day of the experiment. During the third day, Exide planned to use blast furnace #1 for processing of test materials.

#### Day Three

The test material feed ratio in Blast Furnace #1 was increased to 13 percent and Blast Furnace #2 was fed regular feed to serve as the control. The test material was pre-mixed with the regular feed prior to charging the furnaces to eliminate layering of test materials within the furnaces. Exide decided to reduce coke input to the blast furnaces because the test material was serving as a supplemental source of carbon. The reverberatory furnace remained at a feed ratio of 11 percent of the test material. Again, material accumulated in the reverberatory furnace throughout the day.

#### Day Four

The reverberatory furnace showed continued signs of material buildup, crusting and sluggish performance. Exide decided not to feed test material to the reverberatory furnaces until the next day. The blast furnace test feed ratio was held constant at 13 percent test material.

#### Day Five

Beginning at 8:00 AM on the final day of the test, the reverberatory furnace was fed at a ratio of 7 percent test material. No crust accumulation in the reverberatory furnace was noted. However, the reverberatory furnace still performed sluggishly. Exide plant management hypothesized that the sluggish performance was attributed to the slow rate of burning associated with the relatively large pieces of material in the test feed. They concluded that it was not viable to process Tonolli site materials in the reverberatory furnaces without size reduction of the battery case materials.

Both blast furnaces were fed test material at 20 percent by weight until the operation of one of the furnaces was discontinued in order to complete scheduled repairs and maintenance. The testing was continued using the second blast furnace and the overall evaluation was concluded by 10:00 PM on September 13, 1991 when all the Tonolli material had been processed.

# 2.0 RESULTS AND DISCUSSION

In order to assess the viability and economics of the process, CHMR/Exide recorded the settings of various plant parameters, the production figures for both sets of furnaces, and stack parameters. In addition CHMR obtained samples of the input material slag and lead. The sampling was conducted in accordance with the QAPP. The results from the sampling and analysis are presented and discussed below.

All statistical analyses were conducted according to the procedures outlined in Section 5 of the QAPP. All confidence ranges, q-tests, analyses of variance (ANOVA analyses), etc. were performed based on 90 percent confidence limits.

In-depth analyses were performed only on the blast furnace data. No statistical analyses were conducted on reverberatory furnace parameters, because the experienced furnace operators had already determined that the reverberatory furnaces could not accept the battery case material feed as received from the Tonolli site.

#### 2.1 TEST MATERIALS

The test materials consisted of pieces of rubber and polypropylene, typically shredded to approximately 1 to 3 inches in size, combined with reddish-brown sludge, dust, bits of concrete and iron, small wood scraps and pieces of cloth. Because of the varying particle sizes and shapes, the material was not densely packed.

The test materials were sampled for various parameters, including density, lead content, sulfur content, TCLP values, percentage rubber, and other parameters. The results from these analyses are presented in Table A-1, which shows lead concentrations ranging between 0.28 percent and 15.7 percent, with an average of 3.5 percent.

The wide range in analytical results from analyses of the Tonolli material was attributed to non-homogeneity in the material, rather than errors in the analyses. CHMR reviewed the analytical procedure and QA/QC notes for the analyses, and determined that no obvious errors in analyses existed. Analyses 1 through 4 were of samples composites of the material on the trucks loaded from the site (composited from four different grabs of material taken from the trucks). Analyses 5 and 6 were each composites of material obtained from five different locations in the pile of material present at the smelter. Analyses 7 and 8 were duplicate analyses of material from one location in the pile. The composites for analyses 5 and 6 included one grab which contained large amounts of reddish brown sludge. This sludge apparently contained a very high fraction lead. Thus, by averaging all the analytical results, CHMR obtained an average which was representative of approximately 30 grab samples taken of the material. Based on this, CHMR concluded that much of the Tonolli material contained relatively low concentrations of lead, with scattered higher concentrations, and an average concentration of 3.5%.

# 2.2 FURNACE INPUT PARAMETERS

The input parameters recorded included air flows, oxygen consumption, back pressure, and a record of materials introduced into the furnaces. These parameters were recorded by Exide and CHMR personnel during each shift. Table A-2 presents a summary of effects of the test material on the various parameters.

## 2.2.1 Blast Air

Air enriched with oxygen is used in the blast furnace to oxidize coke and other combustible material to maintain a sufficiently high temperature to melt and fuse the material in the furnace. The air is enriched with oxygen to produce higher flame temperatures, more efficient burning, and as a side benefit, smaller blowers are required to introduce extra oxygen than are required to move the required larger volumes of air. The required rates of air and oxygen inputs to the furnaces are determined by the operators and set manually. The air and oxygen settings are recorded at the beginning of each shift, and whenever they are changed.

# TABLE A-2 SUMMARY OF EFFECTS ON BLAST FURNACE PERFORMANCE

Parameter	Effect of AddIng Test Feed to Furnace	Probable Cause	Response
Air	Increase on furnace #2	Probabty due to build-up; otherwise insignifcant	Small problem no response is necessary
Oxygen	No observed difference		
Back Pressure	Increased	Partially due to over-coking	Decrease particle size
SO <sub>2</sub> Emissions	No observed difference		
Lead Production	No observed difference		
Slag Production	No observed difference		
Lead Concentration in Slag	Increased from 2.2 to 3.7%	Over-coking	Reduce coke input
Lead Concentration in Slag	Reducing environment	Too much carbon	Reduce coke input; increase iron input
Sulfur in slag	No observed difference		
Antimony in Slag	No observed difference		
Silica in Slag	No observed difference		
FeO in Slag	No observed difference		

The relative air consumption in Blast Furnace #2 while regular feed only was processed was **6.75±0.25**, and while the Tonolli material was processed was **7.42±0.12**. The difference in air consumption in Furnace #2 between times when test feed was processed and when it was not appears to be largely due to a significantly decreased air consumption rate on Wednesday, September 11, which occurred when no test feed was processed in the furnace. It also correlates to a time when the overall production rate of the furnace decreased, possibly due to attempts by the operators to reduce coke buildup in the furnace. This buildup was only partly attributable to the test feed.

#### 2.2.2 Back Pressure

The furnace back pressure is a measure of the pressure against which the air blowers must push in order to force air into the furnace. The back pressure increases as the air required by the furnace increases. At constant air, the back pressure is a measure of the resistance of the material inside the furnace to the flow of air. The back pressure increases when small particles are fed to the furnace or if material begins to back up inside the furnace.

The use of test feed caused an increase in back pressure in the blast furnace. The average back pressure while regular feed was processed was  $4.95\pm0.6$  inches of water, and  $7.42\pm0.4$  inches of water when test feed was charged to the furnace.

Although some of the apparent correlation between back pressure and test feed input may be attributable to buildups within the fumace caused by over-coking, the evidence is strong that the Tonolli material significantly contributed to the back pressure increase. The most likely reason for the increase in back pressure while processing Tonolli material is that the Tonolli material was large enough that it burned relatively slowly, but sufficiently small to fill some of the pore spaces inside the furnace feed shaft.

The increase in back pressure seen during the evaluation probably had little affect on overall furnace performance or overall process economics. However, as the back pressure increased, the blowers had to work harder to blow air into the furnace, and at higher back pressures (above 30 or 40 inches of water), the blowers will stall. Therefore, based on the data, which suggest a 5 inch back pressure increase per 10 percent addition of battery case material, the maximum sustainable battery case material feed ratio is between 30 percent and 50 percent by weight.

# 2.3 FURNACE OUTPUT PARAMETERS

CHMR monitored the output of parameters such as sulfur (in the form of  $SO_2$  and calcium sulfate sludge) lead, and slag.

# 2.3.1 Sulfur

The Tonolli material was found to contain **2.6±1.0** percent sulfur. Overall, this concentration did not represent a significant difference with regular feed material, which may contain 2 percent to 5 percent sulfur. Therefore, no significant differences in overall plant sulfur emissions were anticipated. However, CHMR/Exide monitored the fate of sulfur in the process to ensure that processing the test material did not result in an increase in production of calcium sulfate sludge, SO<sub>2</sub> emissions, or sulfur concentration in the slag.

Approximately 50 percent of the sulfur entering a blast furnace leaves as oxides in the flue gas stream. Nearly 100 percent of this sulfur at Exide's Reading smelter is removed as calcium sulfate sludge from the  $SO_2$  scrubber system. However, a small portion of the sulfur in the flue gas is released as stack emissions. Therefore, CHMR/Exide monitored both the calcium sulfate sludge generation and  $SO_2$  concentrations in the stack emissions.

#### SO<sub>2</sub> Emissions

CHMR monitored both stacks at the smelter. Each stack system is connected to the corresponding blast and reverberatory furnace. The average sulfur stack emission in system #1 was 64±6 ppm, while the average stack emission in system #2 was 69±9 ppm. Both of these values were below Exide's permitted average of approximately 110 ppm. No statistically significant differences exist between the emissions from the two systems. The overall average plant SO, concentration was 66±6 ppm.

ANOVA analyses indicate no differences were demonstrated between  $SO_2$  emissions when the test material was being processed, and when it was not being processed, for either set of furnaces. In addition, there is no significant correlation between  $SO_2$  concentrations as a function of test material input ratio. Therefore, CHMR/Exide conclude that the TonIII feed material did not have a significant affect on the  $SO_2$  concentration in the stacks.

#### Calcium Sulfate Sludge Production

During each shift, specific gravity measurements of slurry generated from the air emission control equipment (scrubbers) were taken by the operator on duty. The specific gravity is related to the calcium sulfate concentration in the slurry, and therefore gives a measure of total sulfur uptake in the sludge. Typical slurry specific gravity is between 1 .1 and 1.3. Slurry specific gravities for both air emission control systems averaged **1.18±0.02**, and showed no correlation with test feed input.

At the end of each shift, the amount of slurry was calculated by counting the number of 10 cubic yard tankers transported from the scrubbers to the on-site sludge dewatering facility. The number of tankers varied only between 2 and 4 per shift, with a total of 9 trucks per day except Thursday, September 12, when 8 trucks were transported. Normally, between 8 and 10 tankers per day are transported. Therefore, CHMR/Exide conclude that there was no significant increase in calcium sulfate sludge production due to processing the Tonolli material in the blast furnaces.

# 2.3.2 Slag and Lead Production and Quality

The amount of slag and lead produced by the blast furnaces were critical experimental parameters. The amount and quality of lead produced is important for obvious reasons, as lead is the salable product from smelter operations. The slag generation rate was also important, as slag disposal represents a significant operating expense. The lead concentration in the slag is also important because it represents a potential loss of salable lead product.

Lead and slag production for each furnace during each shift were measured. ANOVA statistical analyses were performed to determine if significant differences existed between the amounts of slag or lead produced while the test materials were run versus the amounts produced while only regular feed was processed in the furnaces. The tests indicated no statistically significant change in lead or slag production

rates while the test feeds were run in either furnace. The only significant drop in production during the week occurred in Furnace #2 on September 10, when lead production dropped due to over-coking.

Lead samples from the blast furnaces were obtained during each shift. The samples were analyzed by Exide personnel using Spectrometer Lab Test 201 equipment to determine concentrations of trace metals in the lead. The results from the lead analyses indicated no significant increase in contaminants in the lead product.

Composite samples of slag were taken as the slag was being tapped from the furnaces. The molten slag samples were collected by the Exide operators by inserting a shovel into the furnaces in accordance with the QAPP. Table A-3 shows the results of the analyses of slag samples taken from the furnaces when they were fed regular feed, and regular feed and test feed mixed (with the test feed ratios noted). The table shows no consistent differences in sulfur, silica, antimony, and iron oxide concentrations when the test feed was processed in the furnaces. The results do show a significant difference in lead concentrations in the slag. When only regular feed was processed, the lead concentration was  $2.21\pm0.6$  percent. When Tonoiii material was added, the lead concentration increased to  $3.7\pm0.7$  percent.

Much of this difference may be attributable to the problems encountered when both furnaces were overcoked earlier in the week. The over-coking problem was intensified by the addition of test feed. High coke increases the concentration of lead in the slag. On Wednesday, September 11, the plant responded by reducing the amount of coke and increasing the iron fed to the furnace to maintain a reducing environment with less coke. The data show a decrease in the lead content in the slag beginning during the third shift on September 11 (samples 13 and 14) and continuing throughout the remainder of the week. By September 12 and 13 (samples 15 and 16), the lead concentration in the slag was reduced, and a relatively stable slag composition was maintained at 2.5 percent, which is approximately equal to the lead concentration in slag when no Tonolli input was fed to the furnace.

# TABLE A-3 BLAST SLAG ANALYTICAL RESULTS

Sample	Lead (%)	Sulfur (%)	SiO <sub>2</sub> (%)	FeO (%)	Antimony (%)
1	2.8	3.0	24.8	30	1.5
2	3.1	3.9	29.5	32	0.1
3	1.1	5.2	15.1	63	
4	2.7	3.0	24.4	36	0.5
5	1.9	4.9	21.7	46	0.5
6	2.5	5.0	4.7	30	1.0
7	1.0	2.9	23.9	43	-
AVERAGE	2.2±0.6	4.0 <u>+</u> 0.8	23 <u>+</u> 3.4	40 <u>+</u> 8.6	0.72±0.5

# **REGULAR FEED ONLY**

# REGULAR FEED AND TEST FEED MIXED (% test feed in parentheses next to sample number)

Sample	Lead (%)	Sulfur(%)	SiO <sub>2</sub> (%)	FeO (%)	Antimony (%)
8 (7%)	3.3	1.4	33.9	29	0.3
9 (7%)	5.3	3.6	30.0	24	0.2
10 (11%)	4.8	5.9	21.4	47	2.2
11 (11%)	2.6	2.1	32.6	33	0.6
12 (13%)	3.3	1.2	32.6	30	0.2
13 (13%)	4.7	4.5	21.2	44	0.4
14 (13%)	4.5	5.2	2.36	42	0.4
15 (13%)	2.5	1.8	28.0	48	0.4
16 (20%)	2.5	3.2	18.8	55	0.2
Average	3.7 <u>+</u> 0.7	3.2 <u>+</u> 1.0	27 <u>+</u> 3.7	39 <u>+</u> 6	0.5 <u>+</u> 0.4

All confidence intervals based on 90% confidence level.

# APPENDIX B

# RECLAMATION OF LEAD FROM THE NL INDUSTRIES SUPERFUND SITE IN PEDRICKTOWN, PA

Description of Evaluation and Results and Discussion

November, 1993

# 1.0 INTRODUCTION

The Center for Hazardous Materials Research (CHMR) and Exide Corporation (Exide) performed a joint research study to determine the feasibility and economics of using secondary lead smelters for the recovery of lead from leadcontaining materials. Under the project CHMR/Exide conducted an evaluation to determine the feasibility and economics of reclaiming lead from the NL Industries, Inc. Superfund site in Pedricktown, NJ. This evaluation was conducted in two stages: a preliminary investigation and a larger-scale evaluation.

As part of the preliminary investigation, a total of 19 trucks with 20 to 40 cubic yard capacities were loaded with lead-containing material on January 29 and 30, 1992. The trucks represented different types of material found on the Pedricktown site with lead contents ranging between 36 and 65 percent. A total of 372 tons of material were processed during this initial investgatton. Information gathered from the preliminary test assisted CHMR/Exide in determining the feed rates and types of material to be processed through the secondary lead smelter.

The purpose of the preliminary study was to process the different types of material through the furnaces and determine the initial response of the furnaces to the material. This provided CHMR/Exide with the information necessary to complete the next stage of the evaluation, which consisted of processing the material over a longer time through the secondary lead smelter.

After receiving permission from the EPA Project Officer and the EPA Remedial Project Manager (RPM), the second stage of the investigation was initiated. Materials were excavated, loaded, and transported to Exide's secondary smelter in Reading, PA. The material was weighed and stored in an area isolated from other feed material prior to beginning the evaluation. During the second evaluation, which occurred over three months, approximately 1200 tons (2.4 million pounds) of Pedricktown material were processed.

# 2.0 DESCRIPTION OF EVALUATION

The purpose of the evaluation was to determine the feasibility and economics of processing the material from the NL Industries, Inc. Superfund site using secondary lead smelting technology. The initial step was a preliminary test during which 19 truckloads were processed. This was followed by a larger-scale investigation that spanned three months.

# 2.1 RECLAMATION PROCESS

The reclamation of lead from Superfund and other lead-containing materials is based on existing lead smelting procedures and basic pyrometallurgy. The materials are first excavated from Superfund sites or collected from other sources. Then they are preprocessed to reduce particle size, and to remove rocks, soil, and other debris. The materials are then transported to the smelter.

At the smelter, the materials are fed either to the reverberatory or blast furnaces, depending on particle size or lead content. The two reverberatory furnaces normally treat lead from waste lead-acid batteries as well as other lead-containing material. The furnaces are periodically tapped to remove slag, which contains 60 to 70 percent lead, and a soft, pure lead product. The two blast furnaces treat the slag generated from the reverberatory furnaces, as well as larger sized leadcontaining waste. These fumaces are tapped continuously for lead, and intermittently to remove the slag, which is transported off-site for disposal. The reverberatory and blast furnace combination at Exide can reclaim lead from batteries and waste with greater than 99 percent efficiency.

### 2.2 SMELTING PROCESS DESCRIPTION

Exide's Reading, PA, secondary smelter is primarily engaged in the reclamation of lead from spent lead-acid batteries and other lead-containing materials. Exide reclaims lead using both reverberatory and blast furnaces. The plant operations are typical of the secondary lead smelting industry.

### 2.3 EXPERIMENTAL DESIGN

The material obtained from the Pedricktown site varied in size, lead content, and overall texture and structure. These factors determined the method of processing through the secondary lead smelter. The majority was processed through the reverberatory furnaces, while the blast furnaces were used for larger pieces of material, such as lead hard heads (large chunks of metallic lead) and ingots found at the site.

Approximately 372 tons of material were processed during the preliminary investigation. Since the objective of the preliminary investigation was to study the effects of the Pedricktown material on furnace performance, this stage of the evaluation featured the most diverse variety of material.

During the large-scale evaluation, CHMR/Exide processed approximately 1200 tons of material. The majority of material was processed through the reverberatory furnaces over a two month period. During the third month, the material had to be processed through the blast furnaces due to its size and composition.

# 2.4 EXPERIMENTAL FEED

Tables B-1 through B-4 provide the results of detailed analyses of the NL Industries site material during the preliminary and large-scale investigations. Table B-1 shows the results of analyses of samples taken during a preliminary field reconnaissance to the site during December 1991. The parameters analyzed included lead, antimony, calcium, silicates, sulfur, arsenic, tin and iron were analyzed mainly to determine the metallurgical aspects of the feed and its suitability in the smelting process. Further analyses were conducted of the material which was fed to the furnaces during January 1992. The analytical results are presented in Table B-2. Composite samples of the material were taken as it arrived in trucks at the smelter. Analyses of lead and silica were conducted to provide baseline metallurgical data for the materials. Fluorine and chlorine were analyzed to quantitatively demonstrate

that the material contained negligible quantities of these elements, and no halogenated compounds. Aluminum was analyzed because there was a concern among plant management that the material may contain some aluminum, which is prohibited in the furnaces, and sodium was analyzed for because it represented a potential impurity in the lead products. In general, the results showed no major areas of concern for the material to be fed to the smelter, and a feedstock which contained over 50% lead.

Table B-3 shows the results of analyses conducted on composite samples taken of the material sent to the smetter during the longer term evaluations, August through October 1992. The analyses included moisture analyses for the August and September material, because moisture levels appeared to be a potential concern to furnace operators. The results showed that the levels were not of concern. The material shipped during October which was fed to the blast furnace, appeared to be similar to reverberatory slag and was composed primarily of lead and iron One load of wooden pallet material was analyzed for Btu content, since it was anticipated to burn in the furnaces.

#### 2.5 CHRONOLOGY OF THE EVALUATION

The following provides the schedule and different methods used for processing the material during the preliminary and large-scale investigation.

#### 2.5.1 Preliminary Test - January 1992

Nineteen trucks brought a wide variety of the debris from the site to the smelter at Exide Initially, test material was fed directly into the reverberatory furnaces as 100 percent of the total feed. This caused various malfunctions in the furnace, including build-ups in the furnace and breakdowns of the belt feed system. The material was successfully fed at a 50 percent weight ratio into the reverberatory furnaces. No material was fed to the blast furnaces.

#### 2.5.2 Large-Scale Test - August through October 1992

The larg e-scale investigation took place through the three months that were used to process the material. During the first two months, the material consisted primarily of dross, slag, and debris, which was processed mainly through the reverberatory furnaces. The material during the third month consisted of large pieces of slag, which was processed through the blast furnaces at the smelter. The slag also featured a lower concentration of lead than the previous material. The following paragraphs provide a short summary of the materials, and the methods used for processing the material.

#### 2.5.2.1 August

The initial 19 trucks for the large-scale investigation transported various types of dross material, which were mixed with regular feed at a 40 percent (by weight) ratio and fed into the reverberatory furnace. The larger sized material was sorted out and processed through the blast furnaces. These operations continued until the initial truckloads of material had been depleted. The average lead concentration of the dross material was 48 percent.

	Metal Concentration	entration						
Common Transferration	Lead (percent)	Antimony (percent)	Calcium (CaO)	Silicates (percent)	Sulfur (percent)	As (percent)	Sn (tin) (percent)	Fe as % FeO
sample lype/Notes			(percent)					
Dross from Pedricktown	60.3%	0.1 to 1%	0.1 to 1%	1 to 10%	1.0%			The second state of the se
Duplicate of Sample A	60.9%		0.2%		0.8%			1.0%
88% dross, 6% metal, 6% plastic	50.5%	0.1 to 1%	0.1 to 1%	2.1%				
Dross	61.8%	1 to 10%	0.1 to 1%	1.3%				
76% dross, 21% metal, 3% plastic	63.1%	1.3%				0.14%	0.58%	
Dross (EXCLUDED FROM TEST)	5.3%			68.2%				
Duplicate of Sample F	5.4%		0.3%	29.8%				10.0%
73% dross, 23% metal, 3% plastic	69.0%	1.2%				0.11%	0.23%	
86% dross, 13% plastic	39.7%	0.4%				0.04%	0.20%	
Slag & debris	56.1%							
Battery casing & debris	52.8%							
Lead hard head mat'l	35.8%							
Red Dross	57.3%							
Lead debris	57.3%							
Soft lead dross	54.1%							
Orange/yellow Dross	60.9%							
Black Dross	61.1%							
AVERAGES:	50.1%	0.97%	(excl.	1.7% (exci. Sample F)	0.10%	0.34%		

TABLE B-1 FIELD SAMPLE DATA NL INDUSTRIES (PEDRICKTOWN) SITE DECEMBER 1991

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### TABLE B-2 FEED STREAM ANALYSES NL INDUSTRIES EVALUATION PRELIMINARY EXPERIMENT JANUARY 1992

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Sample Type/Notes	Quantity O Processed (Ibs)		on Flouride (percent)	Chloride (percent)	Silicates (percent)	Aluminum (percent)	Sodium	Other analyses (percentages)
LEAD DEBRIS	40,530	57%	<0.0001	0.51%	6.9%	0.55%	1.35%	
LEAD DROSS	87,310	61%	<0.0001	0.06%	3.1%	0.16%	0.38%	
LEAD SLAG	18,280	54%	<0.0001	0.12%	3.5%	0.18%	0.28%	
SLAG AND DEBRIS	42,620	61%'	<0.0001	0.11%	3.4%	0.41%	0.59%	
SLAG AND DEBRIS	81,740	61%	<0.0001	0.04%	1.5%	0.22%	0.62%	
SOFT LEAD DROSS	44,210	54%	<0.0001	0.08%	1.6%	0.26%	0.52%	
LEAD SULFATE	30,050	59%	<0.0001	0.06%	7.9%	0.12%	0.12%	
SLAG AND DEBRIS	34,950	65%	<0.0001	0.14%	2.4%	0.22%	0.16%	
LEAD HARD HEAD MATERIAL	40,460	36%	<0.0001	0.04%	5.5%	0.40%	0.65%	
BATTERY CASING AND DEBRIS	32,680	53%	<0.0001	0.08%	4.7%	0.85%	0.13%	
MATERIAL FROM INSIDE BLDG	77,040	55%	NA	NA	4.2%	NA	NA	S: 3.3 CA:0.9 Sb: 0.6%
SLAG AND DEBRIS	43,070	65%	<0.0001	0.14%	2.4%	0.22%	0.16%	
COMPOSITE OF TWO PILES AT SITE	45,100	63%	NA	NA	3.3%	NA	NA	S: 2.7 Ca: 0.3 Sb: 0.8%
SLAG AND DEBRIS (incl. B2 P3&4)	84,360	59%	<0.0001	0.24%	3.63%	0.34%	0.68%	
SLAG AND DEBRIS	42,070	61%	<0.0001	0.11%	3.4%	0.41%	0.19%	
<b>TOTAL</b> Weighted Average:	744,470 lb	<b>)s</b> 58%	0.00%	0.13%	3.59%	0.32%	0.49%	

# TABLE B-3 FEED STREAM ANALYSES NL INDUSTRIES EVALUATION AUGUST - OCTOBER 1992

	,		oncentration		Ciliantes	0		0.1
Sample Type/Notes	Processed (Ibs)	Lead (percent)	Moisture (percent)	Antimony (percent)	Silicates	Sulfur	Iron (FeO)	Other
AUGUST MATERIA		(percent)	(percent)	(percent)	(percent)	(percent)	(percent)	analyses (%)
Lead Dross & debris		43%	9.8%	0.8%	5.7%	2.7%	6 9.6%	
	133,779							
Lead Dross & debris	112,656	55%	10.4%	NA				
Lead Dross & debris	105,615	54%			7.9%			
Slag & debris	182,470	44%	12.6%	0.6%	4.2%	3.0%	% 1.4%	1
Slag & debris	262,700	47%	8.7%	3.4%	3.7%	6 0.9	% 4.6%	6 Ca 0.53%
SEPTEMBER MATE	RIAL							
Lead debris	486,630	57%	13.4%	NA	NA	2.1%	6 4.0%	)
Black dross	138,140	51%	11.7%	2.3%	NA	0.8%	6 NA	1
Iron cans	13,230	<1%	NA	NA	NA	N	A > 80%	Iron feed
Battery Cases & debr	,	44%	9.6%	NA	NA	1.09	6 5.1%	
Pallets	39,810	7%	NA	NA	NA	N/		
Baghouse bags	49,000	69%	NA	NA	NA	N/	A NA	N N
OCTOBER MATERIA	ΔI							
Wooden pallets from		6.5%	NA	NA	NA	NA		Btu/lb: 4945
Slag	600,900	32%	NA	NA	NA	NA		
Slag & debris	261,820	28%	NA	NA	NA	NA	=	Ca: 0.14%
Total Drassasad	2 457 200 1	ha						
Total Processed: Weighted avg.com	2,457,300   position:	43.1%	11.3%	2.0%	4.7%	1.8%	6 8.5%	)

# 2.5.2.2 September

In September, a total of 19 truckloads containing various types of lead debris were processed through the smelter's reverberatory furnace. The material varied in overall weight and density, with an average lead concentration of 53 percent. In addition, the material had relatively high amounts of calcium and therefore needed to be processed through the reverberatory furnaces at tower feed rates (20 to 30 percent of typical feed, by weight) than the material processed during the previous month. An excess of calcium in the reverberatory furnaces causes build-ups inside the furnaces.

# 2.5.2.3 October

CHMR/Exide performed the final stage of the large-scale investigation in October. The material consisted primarily of slag and debris, which was larger in size than previous material, and therefore needed to be processed through the blast furnaces. This slag and debris material contained lead concentrations of approximately 30 percent. The final 20 truckloads of the Pedricktown material were processed by early November 1992.

# 3.0 **RESULTS AND DISCUSSION**

In order to assess the viability and economics of the process, CHMR/Exide obtained production sheets, samples, sample results and other general information of furnace performance to evaluate the processing of the material. All statistical analyses were conducted according to the procedures outlined in Section 5 of the Quality Assurance Project Plan for the project. Analysis of variance (ANOVA) was performed on the data sets. The ANOVA compares two experimental means to determine if a significant difference exists between them. The null hypothesis is that no difference exists between the data set. The governing equations are as follows:

$$Z=ts_{\sqrt{\frac{n_1+n_2}{n_1n_2}}}$$

and

$$D = |x_1 - x_2|$$

where *s* is the standard deviation across all data, *n* is the number of samples analyzed, *t* is from statistical tables (based on 90 percent probability level), and x is the calculated mean. When Z > D, the null hypothesis is not rejected, and no significant difference between the means has been demonstrated. On the other hand, when D > Z, then a significant difference does exist. The q-test, which rejects outlying data, was also performed on each data set as required prior to the ANOVA.

## 3.1 TEST MATERIAL

The Pedricktown material was analyzed for percent moisture, silica, antimony, iron oxide, lead, sulfur, and calcium. Table B-4 presents a summary of the analytical results of the material processed over the entire investigation. The January material was not analyzed for all of these parameters, and the October slag and debris material was only analyzed for total weight percent of lead.

# 3.2 SUMMARY OF PRELIMINARY INVESTIGATION RESULTS

CHMR/Exide conducted the preliminary investigation in which material was processed through the smelter between January 29 and February 2, 1992. During this preliminary investigation, CHMR/Exide processed approximately 372 tons of material containing between 36 and 65 percent lead. The results from the preliminary investigation showed that the material could be processed through the reverberatory furnaces at feed rates of up to a maximum of 50 percent by weight test material. Higher feed rates caused build-ups and break-downs in the furnace feed system due to the weight of the test material, which was heavier than the typical Exide feed.

# 3.2.1 Lead and Slag Production and Quality

The amount and nature of the lead and slag produced by the reverberatory furnaces when processing the test material were critical experimental parameters, since the lead is the salable product from the smelter, and the reverberatory slag is a feed to the blast furnaces.

Blast furnace lead and slag production were not monitored during the preliminary experiment, because the test feed material was fed exclusively to the reverberatory furnaces.

# TABLE B-4 NL INDUSTRIES FEED MATERIAL – SUMMARY ANALYSIS

		FULL-SCALE	TEST	
	JANUARY	AUGUST	SEPT	OCT
PARAMETER	MATERIAL	MATERIAL	MATERIAL	MATERIAL
% MOISTURE	N/A	10.1%	8.7%	N/A
% SiO2	3.6%	5.0%	N/A	N/A
% ANTIMONY	N/A	1.0%	2.3%	N/A
% FeO	N/A	3.1%	4.6%	N/A
% LEAD (AVG)	56%	46%	52%	31%
% LEAD (RANGE)	36 to 65	42 to 52	7 to 69	6.5 to 31
# OF FEED PILES	15	3	6	1
% SULFUR	3%	1.3%	1.3%	N/A

# 3.2.2 Reverberatory Furnace Lead Production and Quality

The quality of the lead produced by the reverberatory furnaces during the January test was determined through the use of Spectrometer Lab Test 100 analysis on-site, which measures the concentration of trace metals in the lead product. CHMR/Exide representatives collected 20 lead samples from the test reverberatory furnace, and 10 lead samples from the control reverberatory furnace. The average results of these samples can be seen in Table B-5, which shows a comparison of the lead produced during processing of the test material and the lead from the control reverberatory furnace processing typical feed. The values given for the metals are relative units. The table shows that the quality of the lead produced by the test reverberatory furnace is comparable with typical reverberatory lead. All of the experimental means are statistically equivalent at a 90 percent probability level except for that of antimony: **0.249±0.0028** units of antimony when test material was fed versus **0.041±0.008** when only regular feed was fed. This was anticipated, because the test material was known to contain relatively high concentrations of antimony.

Total lead production in the test and control reverberatory furnaces remained roughly equivalent during the evaluation, with perhaps a slightly significant increase in production for the furnace fed test feed. The effect of the material on total furnace production was more precisely quantified during the longer term evaluation.

# 3.2.3 Reverberatory Slag Production and Quality

CHMR obtained samples of the reverberatory furnace slag for both the test and control furnaces. The slag was analyzed for total lead, antimony, silicates, sulfur, and iron. The results from the analyses are presented in Table B-6. The results showed a significant decrease in lead concentration in the slag -- from an average of 73% in the control furnace to 48% in the test furnace. At the same time, the concentration of silicates and iron increased in the slag from the furnace fed test feed.

Total slag production for the furnaces was only cursorily reviewed, since it was better quantified during the longer term evaluation. It seemed to decrease slightly during the evaluation, although the decrease was only barely statistically significant.

The combination of a decrease in lead concentration with a potential decrease in slag production indicates a significant decrease in the amount of lead flowing from the reverberatory furnace to the blast furnace. At the same time, lead production in the reverberatory furnace increased, Indicating that at least some of the lead input was effectively shifted from the slag to lead product in the reverberatory furnace.

# TABLE B-5 REVERBERATORY LEAD FROM TEST MATERIAL VERSUS TYPICAL FEED LEADFOR JANUARY, 1992 PRELIMINARY INVESTIGATION

REVERBERATORY LEAD FROM TEST MATERIAL (TEST FURNACE)

	Pb	Sb	Sn	As	Cu	NI	Bi	Ag	Fe	Te	Zn
AVG LEAD FROM TEST MATERIAL (x):	13,812	0.249	0.000	0.000	0.037	0.012	0.012	0.003	0.000	0.004	0.000
STANDARD DEVIATION (S):	483	0.073	0.000	0.000	0.004	0.008	0.003	0.001	0.000	0.001	0.000
NUMBER OF SAMPLES (n):	20	20	20	20	20	20	20	20	20	20	20
	13812	0.249			0.037	0.012	0.012	0.003		0.004	
TRUE MEAN (μ):		±	0	0	±	±	±	±	0	±	0
	186	0.028			0.002	0.004	0.001	0.000		0.000	

TRUE MEAN  $\mu = x \pm \frac{St}{\sqrt{n}}$ . VALUE FOR t BASED ON 90% PROBABILITY LEVEL.

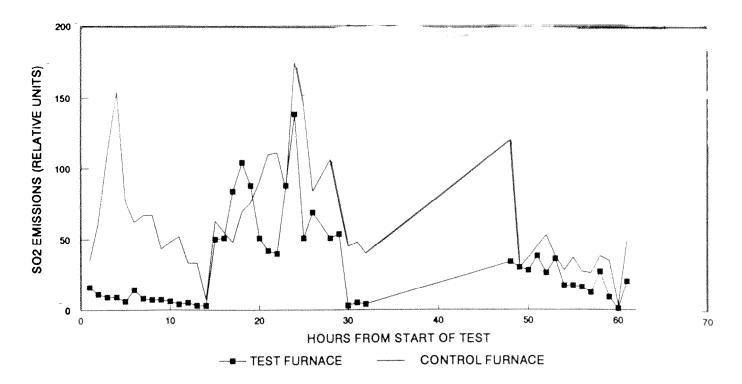
# REVERBERATORY LEAD FROM TYPICAL FEED MATERIAL (CONTROL FURNACE)

	Pb	Sb	Ŝn	As	Ĉu	NI	Bi	Ag	Fe	Te	Zn
AVG LEAD FROM TYPICAL FEED (x):	13317	0.041	0.000	0.000	0.040	0.020	0.012	0.004	0.000	0.004	0.000
STANDARD DEVIATION (S):	779	0.014	0.000	0.000	0.006	0.006	0.003	0.001	0.000	0.000	0.000
NUMBER OF SAMPLES (n):	10	10	10	10	10	10	10	10	10	10	10
	13317	0.041			0.040	0.020	0.012	0.004		0.004	
TRUE MEAN $(\mu)$ :	±	±	0	0	±	±		±	0	±	0
	446	0.008			0.004	0.004	0.002	0.001		0.000	l l

TRUE MEAN  $\mu = x \pm \frac{S_t}{\sqrt{n}}$ . VALUE FOR t BASED ON 90% PROBABILITY LEVEL.

# TABLE B-6REVERBERATORY FURNACE SLAG COMPOSITIONDURING TEST WITH NL INDUSTRIES MATERIAL - - January 1992

Lead Date         Antimony (percent)         Sulfur (percent)         Fe (percent)           TEST 29-Jan-92         78.7% 58.3%         3.4% 4.2%         5.8% 5.8%         4.4% 3.6%           29-Jan-92         43.9% 5.6%         20.3% 4.2%         10.6% 5.8%         10.6% 4.4%           29-Jan-92         43.9% 42.2%         6.6% 14.9%         2.2% 3.6%         3.6% 10.4%           29-Jan-92         49.7% 42.2%         6.6% 14.9%         2.2% 3.6%         3.6% 10.4%           29-Jan-92         49.7% 5.4%         11.3%         10.4% 19.2%           30-Jan-92         40.1% 9.9%         9.3% 11.4%         9.5%         12.9% 12.9%           30-Jan-92         41.6% 9.9%         13.1%         16.7% 12.9%           31-Jan-92         71.5%         2.9% 1.5%         3.8% 11.5%           31-Jan-92         82.8% 2.2%         1.5% 3.8%         3.8% 1.5%           01-Feb-92         20.2% 3.2%         2.3% 2.3%         23.0%           01-Feb-92         29.9% 4.7%         18.8% 2.13%         21.3% 2.4%           01-Feb-92         29.9% 4.3%         3.8% 17.6%         22.1% 2.4%           02-Feb-92         20.2% 3.2%         22.4% 3.3%         2.3% 2.4%         2.4% 2.4%           02-Feb-92         20.2% 3.4%		Concentrat	tion			
TEST FURNACE           29-Jan-92         78.7%         3.4%         3.3%         0.7%           29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         49.7%         6.8%         15.3%         8.7%           42.2%         6.6%         14.9%         2.2%         3.6%           29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         43.5%         6.6%         15.1%         19.2%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.1%         7.3%         12.1%         12.0%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         28.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         29.9%         4.7%<		Lead	Antimony	Silicates	Sulfur	Fe
TEST FURNACE           29-Jan-92         78.7%         3.4%         3.3%         0.7%           29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         49.7%         6.8%         15.3%         8.7%           42.2%         6.6%         14.9%         2.2%         3.6%           29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         43.5%         6.6%         15.1%         19.2%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.1%         7.3%         12.1%         12.0%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         28.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         29.9%         4.7%<	Date Sampled	(percent)	•		(percent)	as % FeO
29-Jan-92         78.7%         3.4%         3.3%         0.7%           58.3%         4.2%         5.8%         4.4%         3.6%           29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         43.9%         5.6%         20.3%         8.7%           42.2%         6.6%         14.9%         2.2%         3.6%           29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         33.5%         6.6%         15.1%         19.2%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.5%         2.9%         5.1%         5.3%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         82.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           02-Feb-92         29.1%         3.6%         19.2%         25.4%      0					-u /	
29-Jan-92         78.7%         3.4%         3.3%         0.7%           58.3%         4.2%         5.8%         4.4%         3.6%           29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         43.9%         5.6%         20.3%         8.7%           42.2%         6.6%         14.9%         2.2%         3.6%           29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         33.5%         6.6%         15.1%         19.2%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.5%         2.9%         5.1%         5.3%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         82.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           02-Feb-92         29.1%         3.6%         19.2%         25.4%      0						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	TEST FURNACE					
29-Jan-92         43.9%         5.6%         20.3%         10.6%           29-Jan-92         49.7%         6.8%         15.3%         8.7%           42.2%         6.6%         14.9%         2.2%         3.6%           29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         33.5%         6.6%         15.1%         19.2%           30-Jan-92         54.0%         4.4%         9.5%         12.9%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.1%         7.3%         12.1%         5.3%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         82.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         9.8%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           01-Feb-92         29.9%         3.2%         21.1%         20.4%	29-Jan-92	78.7%	3.4%	6 3.3%		0.7%
29-Jan-92         49.7%         6.8%         15.3%         8.7%           42.2%         6.6%         14.9%         2.2%         3.6%           29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         33.5%         6.6%         15.1%         19.2%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         40.1%         9.9%         13.4%         16.7%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.1%         7.3%         12.1%         12.0%           31-Jan-92         47.8%         6.0%         14.5%         3.8%           31-Jan-92         47.8%         6.0%         14.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           02-Feb-92         20.2%         3.2%         22.8%         26.7% <th></th> <th>58.3%</th> <th>4.2%</th> <th>6 5.8%</th> <th>4.4%</th> <th>3.6%</th>		58.3%	4.2%	6 5.8%	4.4%	3.6%
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	29-Jan-92	43.9%	5.6%	6 20.3%		10.6%
29-Jan-92         49.7%         5.4%         11.3%         10.4%           29-Jan-92         33.5%         6.6%         15.1%         19.2%           30-Jan-92         54.0%         4.4%         9.5%         12.9%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         43.7%         11.4%         9.3%         17.3%           30-Jan-92         43.7%         11.4%         9.3%         17.3%           30-Jan-92         47.1%         7.3%         12.1%         12.0%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         82.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         30.2%         3.2%         21.1%         20.4%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           01-Feb-92         29.9%         4.7%         18.8%         21.3%           02-Feb-92         20.2%         3.2%         22.6%         26.5%	29-Jan-92	49.7%	6.8%	6 15.3%		8.7%
29-Jan-92         33.5%         6.6%         15.1%         19.2%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.1%         7.3%         12.1%         12.0%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         82.8%         2.2%         1.5%         3.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         30.2%         3.2%         21.1%         20.4%           01-Feb-92         34.3%         3.8%         17.6%         22.1%           02-Feb-92         29.1%         3.6%         19.2%         25.4%           02-Feb-92         29.1%         3.6%         19.2%         26.5%           02-Feb-92         20.2%         3.2%         2.3%         23.%		42.2%	6.6%	<b>6 14.9%</b>	2.2%	3.6%
30-Jan-92         54.0%         4.4%         9.5%         12.9%           30-Jan-92         40.1%         9.9%         13.4%         16.1%           30-Jan-92         43.7%         11.4%         9.3%         17.3%           30-Jan-92         41.6%         9.9%         13.1%         16.7%           31-Jan-92         47.1%         7.3%         12.1%         12.0%           31-Jan-92         47.8%         6.0%         14.5%         11.5%           31-Jan-92         82.8%         2.2%         1.5%         38%           31-Jan-92         82.8%         2.3%         23.0%         9.8%           01-Feb-92         28.3%         2.3%         23.0%         19.4%           01-Feb-92         30.2%         3.2%         21.1%         20.4%           01-Feb-92         30.2%         3.2%         21.1%         20.4%           01-Feb-92         30.2%         3.2%         22.8%         25.4%           02-Feb-92         29.1%         3.6%         17.6%         22.1%           02-Feb-92         20.2%         3.2%         22.8%         26.7%           02-Feb-92         20.2%         3.2%         21.5%         24.6%	29-Jan-92	49.7%	5.4%	6 11.3%		10.4%
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	29-Jan-92	33.5%	6.6%	6 15.1%		19.2%
30-Jan-92       43.7%       11.4%       9.3%       17.3%         30-Jan-92       41.6%       9.9%       13.1%       16.7%         31-Jan-92       47.1%       7.3%       12.1%       12.0%         31-Jan-92       71.5%       2.9%       5.1%       5.3%         31-Jan-92       47.8%       6.0%       14.5%       11.5%         31-Jan-92       82.8%       2.2%       1.5%       3.8%         57.4%       2.0%       3.8%       0.3%       9.8%         01-Feb-92       28.3%       2.3%       23.0%       19.4%         01-Feb-92       30.2%       3.2%       21.1%       20.4%         01-Feb-92       34.3%       3.8%       17.6%       22.1%         02-Feb-92       29.9%       4.7%       18.8%       21.3%         02-Feb-92       20.2%       3.2%       25.4%       26.5%         02-Feb-92       20.2%       3.2%       26.5%       26.5%         02-Feb-92       20.2%       3.2%       21.5%       24.6%         AVERAGE:       48.2%       5.4%       12.4%       2.3%       12.3%         STD. DEV.       17%       3.9%       6.4%       2.60%       <	30-Jan-92	54.0%	4.4%	6 9.5%		12.9%
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	30-Jan-92	40.1%	9.9%	6 13.4%		16.1%
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	30-Jan-92	43.7%	11.4%	6 9.3%		17.3%
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	30-Jan-92	41.6%	9.9%	6 13.1%		16.7%
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	31-Jan-92	47.1%	7.3%	6 12.1%		12.0%
31-Jan-92       82.8%       2.2%       1.5%       3.8%         01-Feb-92       28.3%       2.3%       23.0%       19.4%         01-Feb-92       30.2%       3.2%       21.1%       20.4%         01-Feb-92       30.2%       3.2%       21.1%       20.4%         01-Feb-92       30.2%       3.2%       21.1%       20.4%         01-Feb-92       29.9%       4.7%       18.8%       21.3%         01-Feb-92       29.9%       4.7%       18.8%       21.3%         01-Feb-92       29.9%       4.7%       18.8%       21.3%         01-Feb-92       29.1%       3.6%       19.2%       25.4%         02-Feb-92       20.2%       3.2%       22.8%       26.5%         02-Feb-92       12.4%       1.3%       30.3%       26.7%         03-Feb-92       25.9%       4.0%       21.5%       24.6%         AVERAGE:       48.2%       5.4%       12.4%       2.3%       12.3%         STD. DEV.       17%       3.9%       6.4%       2.60%       2.70%         29-Jan-92       71.9%       3.9%       6.4%       2.60%       2.70%         29-Jan-92       79.4%       3.8%	31-Jan-92	71.5%	2.9%	6 5.1%		5.3%
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	31-Jan-92	47.8%	6.0%	6 14.5%		11.5%
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	31-Jan-92	82.8%	2.2%			3.8%
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$						9.8%
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	01-Feb-92					
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	01-Feb-92					20.4%
$\begin{array}{cccccccccccccccccccccccccccccccccccc$						
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	01-Feb-92			6 17.6%		22.1%
02-Feb-92       20.2%       3.2%       22.8%       26.5%         02-Feb-92       12.4%       1.3%       30.3%       26.7%         03-Feb-92       25.9%       4.0%       21.5%       24.6%         AVERAGE:       48.2%       5.4%       12.4%       2.3%       12.3%         STD. DEV.       17%       3%       7%       2%       8%         CONTROL FURNACE       7%       3.9%       6.4%       2.60%       2.20%         29-Jan-92       71.9%       3.9%       6.4%       2.60%       2.20%         29-Jan-92       76.9%       3.7%       4.7%       2.20%       2.20%         29-Jan-92       76.9%       3.6%       2.6%       2.70%       2.90%       2.70%         29-Jan-92       79.4%       3.8%       3.7%       2.70%       2.90%       3.6%       2.90%       3.4%       2.90%       3.4%       2.90%       3.4%       2.30%       3.4%       3.3%       5.2%       0.5%       3.1-Jan-92       78.9%       3.4%       4.1%       2.30%       3.6%       3.2%       2.80%       3.6%       3.6%       4.2%       2.80%       3.8%       3.6%       3.6%       3.8%       3.6%       3.8% <td< th=""><th>02-Feb-92</th><th></th><th></th><th></th><th></th><th>25.4%</th></td<>	02-Feb-92					25.4%
02-Feb-92       12.4%       1.3%       30.3%       26.7%         03-Feb-92       25.9%       4.0%       21.5%       24.6%         AVERAGE:       48.2%       5.4%       12.4%       2.3%       12.3%         STD. DEV.       17%       3%       7%       2%       8%         CONTROL FURNACE       17%       3.9%       6.4%       2.60%       29-Jan-92       76.9%       3.7%       4.7%       2.20%       29-Jan-92       26.6%       2.70%       29-Jan-92       76.9%       3.7%       4.7%       2.20%       29-Jan-92       76.9%       3.6%       2.6%       2.70%       2.90%       2.70%       2.9-Jan-92       79.4%       3.8%       3.7%       2.70%       3.6%       2.70%       3.4%       2.90%       2.70%       3.6%       3.4%       2.90%       3.4%       2.90%       3.4%       2.90%       3.4%       4.1%       2.30%       3.6%	02-Feb-92					
03-Feb-92       25.9%       4.0%       21.5%       24.6%         AVERAGE:       48.2%       5.4%       12.4%       2.3%       12.3%         STD. DEV.       17%       3%       7%       2%       8%         CONTROL FURNACE       71.9%       3.9%       6.4%       2.60%         29-Jan-92       76.9%       3.7%       4.7%       2.20%         29-Jan-92       76.9%       3.7%       4.7%       2.20%         29-Jan-92       79.4%       3.8%       3.7%       2.70%         29-Jan-92       79.4%       3.8%       3.7%       2.70%         30-Jan-92       79.4%       3.8%       3.7%       2.30%         31-Jan-92       78.9%       3.4%       4.1%       2.30%         31-Jan-92       79.7%       3.6%       4.2%       2.80%						
$\begin{array}{cccccccccccccccccccccccccccccccccccc$						
STD. DEV.         17%         3%         7%         2%         8%           CONTROL FURNACE         29-Jan-92         71.9%         3.9%         6.4%         2.60%           29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         79.4%         3.6%         2.6%         2.70%           29-Jan-92         79.4%         3.8%         3.7%         2.70%           30-Jan-92         86.5%         3.9%         3.4%         2.90%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%						
CONTROL FURNACE         71.9%         3.9%         6.4%         2.60%           29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         44.3%         3.6%         2.6%         2.70%           29-Jan-92         79.4%         3.8%         3.7%         2.70%           30-Jan-92         79.4%         3.8%         3.7%         2.70%           31-Jan-92         86.5%         3.9%         3.4%         2.90%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%	AVERAGE:	48.2%	5.4%	<b>12.4%</b>	2.3%	12.3%
29-Jan-92         71.9%         3.9%         6.4%         2.60%           29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         44.3%         3.6%         2.6%         2.70%           29-Jan-92         79.4%         3.8%         3.7%         2.70%           30-Jan-92         79.4%         3.8%         3.7%         2.90%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%	STD. DEV.	17%	3%	ώ 7%	2%	8%
29-Jan-92         71.9%         3.9%         6.4%         2.60%           29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         44.3%         3.6%         2.6%         2.70%           29-Jan-92         79.4%         3.8%         3.7%         2.70%           30-Jan-92         79.4%         3.8%         3.7%         2.90%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%						
29-Jan-92         76.9%         3.7%         4.7%         2.20%           29-Jan-92         44.3%         3.6%         2.6%         2.70%           29-Jan-92         79.4%         3.8%         3.7%         2.70%           30-Jan-92         86.5%         3.9%         3.4%         2.90%           71.6%         2.7%         1.8%         5.2%         0.5%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%	CONTROL FURNACE					
29-Jan-92         44.3%         3.6%         2.6%         2.70%           29-Jan-92         79.4%         3.8%         3.7%         2.70%           30-Jan-92         86.5%         3.9%         3.4%         2.90%           71.6%         2.7%         1.8%         5.2%         0.5%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%	29-Jan-92	71.9%	3.9%	6.4%		2.60%
29-Jan-9279.4%3.8%3.7%2.70%30-Jan-9286.5%3.9%3.4%2.90%71.6%2.7%1.8%5.2%0.5%31-Jan-9278.9%3.4%4.1%2.30%31-Jan-9279.7%3.6%4.2%2.80%	29-Jan-92	76.9%	3.7%	<b>4.7%</b>		2.20%
30-Jan-92         86.5%         3.9%         3.4%         2.90%           71.6%         2.7%         1.8%         5.2%         0.5%           31-Jan-92         78.9%         3.4%         4.1%         2.30%           31-Jan-92         79.7%         3.6%         4.2%         2.80%	29-Jan-92	44.3%	3.6%	<b>2.6%</b>		2.70%
71.6%2.7%1.8%5.2%0.5%31-Jan-9278.9%3.4%4.1%2.30%31-Jan-9279.7%3.6%4.2%2.80%	29-Jan-92	79.4%	3.8%	3.7%		2.70%
31-Jan-9278.9%3.4%4.1%2.30%31-Jan-9279.7%3.6%4.2%2.80%	30-Jan-92	86.5%	3.9%	3.4%		2.90%
31-Jan-9278.9%3.4%4.1%2.30%31-Jan-9279.7%3.6%4.2%2.80%		71.6%				
31-Jan-92 79.7% 3.6% 4.2% 2.80%	31-Jan-92	78.9%	3.4%	4.1%		
	31-Jan-92	79.7%	3.6%	4.2%		
AVERAGE: 73.0% 3.5% 3.9% 4.0% 2.1%	AVERAGE:	73.0%			4.0%	<b>2.1%</b>
STD. DEV. 11% 0% 1% 1% 1%	STD. DEV.	11%	0%	6 1%	1%	5 1%



ANOVA ANALYSIS OF EMISSION DATA

]	TEST	CONTROL
VARIABLE (EXPANATION)	FURNACE	FURNACE
x1,x2 (AVERAGE DATA VALUES)	0.49	1.00
s1,s2 (STANDARD DEVIATIONS)	0.49	0.59
n1, n2 (NUMBER OF DATA)	45	45
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.64	1.64
s (STANDARD DEV. ACROSS ALL DATA)	0.60	
<sub>1</sub> Z = ts ((n1 + n2)/(n1 n2)) ^ .5	0.21	
D = abs(x1 - x2)	0.51	
Signif. difference iff $D > Z$ :	Significant dif	ference
Conclusion:	$x^2 - x^1 = 0.5$	1 ± 0.19

# FIGURE B-1 Sulfur Dioxide Emissions from the Test and Control Furnaces – January 1992

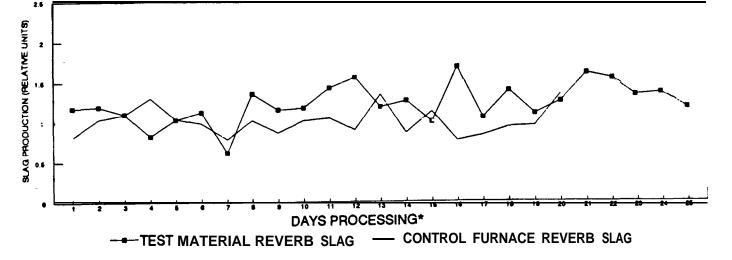
these analyses were not made available to CHMR, although reportedly the vast majority of slag samples were non-hazardous. The amount of blast slag produced is a far more important parameter, and if is discussed below.

# 3.3.2 Lead and Slag Production

Figures B-2 through B-7 present the lead and slag production for the three months of the large-scale investigation. The data used to generate the figures was taken from Exide's daily productions sheets. All of the values In the figures are normalized so that the average production of the control furnace is 1. This was done to protect confidential Exide production information.

[	TEST	CONTROL
VARIABLE (EXPLANATION)	FURNACE	FURNACE
x1, x2 (AVERAGE DATA VALUES)	1.22	1.00
s1, s2 (STANDARDDEVIATIONS)	0.24	0.18
n1, n2 (NUMBER OF DATA)	25	20
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.66	1.71
(STANDARD DEV. ACROSS ALL DATA)	0.24	-
$Z = ts ((n1+n2)/(n1 n2)) ^ .5$	0.12	. 1
D = abs(x1 - x2)	0.22	
Signif. dfference iff D > Z:	Significant difference	
Conclusion:	$x^2 - x^1 = -0.22 \pm 0.10$	0

# AUGUST REVERBERATORY SLAG PRODUCTION



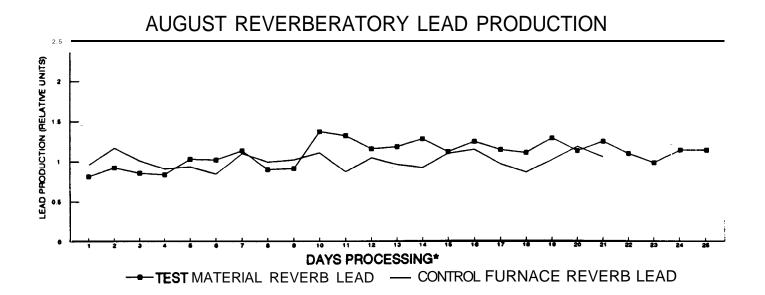
# FIGURE B-2 Comparison of Reverberatory Slag Production- Test and Control Furnaces – August 1992

Different figures are given for lead and slag production for each month of the investigation. Each figure contains a statistical analysis of each month's production based on which furnace the test material was fed. For example, if the reverberatory furnace was being fed with test material, then the analysis of variance was performed on the reverberatory slag and lead produced.

# 3.3.2.1 August

Figures B-2 and B-3 show that test material processed through the reverberatory furnaces in August generated a significant statistically increase in lead and slag productions. This was an unanticipated result, since the test material purportedly contained a lower concentration of lead than the regular feed. No adequate explanation was found, except perhaps that the test feed material could be processed faster than the regular feed, thereby increasing overall furnace throughput.

	TEST	CONTROL
VARIABLE (EXPANATION)	FURNACE	FURNACE
x1, x2 (AVERAGE DATA VALUES)	1.09	1.00
s1, s2 (STANDARD DEVIATIONS)	0.15	0.10
n1, n2 (NUMBER OF DATA)	25	21
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.66	1.70
s (STANDARD DEV. ACROSS ALL DATA)	) 0.14	
$Z = ts ((n1+n2)/(n1 n2))^{.5}$	0.07	
D = abs(x1 - x2)	0.09	
Signif. difference iff D > Z:		
Conclusion:	$x^2 - x = -0.09 \pm 0.06$	6



# FIGURE B-3 Comparison of Reverberatory Lead Production- Test and Control Furnaces --August 1992

# 3.3.2.2 September

The statistical analyses for the reverberatory lead and slag production in September (figures B-4 and B-5) ielded the same quantitative results as in Agust, with statistically significant increases in the reverberatory h

throughput rates.

	TEST		CONTROL
VARIABLE (EXPANATION)	FURNACE		FURNACE
xI, x2 (AVERAGE DATA VALUES)	1.24		1 .00
s1, s2 (STANDARD DEVIATIONS)	0.34		0.23
n1, n2 (NUMBER OF DATA)	32		26
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.64		1.65
s (STANDARD DEV. ACROSS ALL DATA)	0.31		
$Z = ts ((nl + n2)/(n1 n2)) \land .5$	0.14		
D = abs(x1 - x2)	0.24		
Signif. difference iff D > Z:	Significant	difference	]
Conclusion:	$x^2 - x^1 = -$	0.24 ± 0.12	<u>i</u> i

SEPTEMBER REVERBERATORY SLAG PRODUCTION

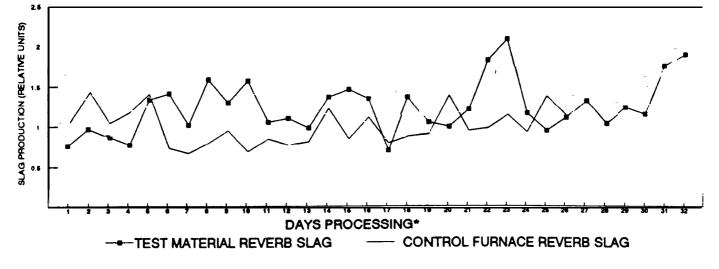
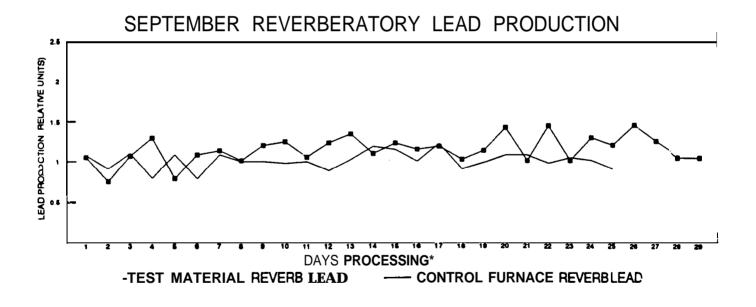


FIGURE B-4 Comparison of Reverberatory Slag Procfuctton- Test and Control Furnaces -September 1992

# 3.3.2.3 October

Figures B-6 and B-7, which show October blast furnace lead and slag production, indicate that the average amount of lead produced in the furnace fed test feed was statistically higher than that produced than that in the control. This difference was most probably due to a higher throughput caused by the use of the test material. There was no significant difference demonstrated in the amount of slag generated in October between the furnace fed test materials and the control furnace.

	TEST	(	CONTROL
1 VARIABLE (EXPANATION)	FURNACE	I	FURNACE
xI, x2 (AVERAGE DATA VALUES)	1.14		1.001
s1, s2 (STANDARD DEVIATIONS)	0.16		0.11
n1, n2 (NUMBER OF DATA)	29		25
t1,t2 (FOR 90% CONFIDENCE LIMIT)	1.64		1.66
s (STANDARD DEV. ACROSS ALL DATA)	0.15		]
$Z = ts ((n1 + n2)/(n1 n2)) ^.5$	0.07		i
D = abs(x1 - x2)	0.14		
<b>Signif</b> . difference iff D > Z:	Significant	difference	and a second second
Conclusion:	x2 – xl =	-0.14 ± 0.06	and a subsection of the subsection of the





# 3.3.3 Overall Blast Slag Production

Since blast slag is costly to dispose of, an increase in blast slag production would cause a commensurate increase in costs. The amount of blast slag produced during each shift was determined based on Exide's daily production reports and compared for the furnace systems fed test and regular feed. Table B-7 presents an ANOVA analysis for the blast slag production during the three month investigation. The analysis showed that there was no significant **difference** demonstrated between the average test furnace slag production. Therefore, it was concluded that the use of **the** test feed would not cause a significant increase in disposal costs.

	TEST	CONTROL			
VARIABLE (EXPANATION)	FURNACE	FURNACE			
x1, x2 (AVERAGE DATA VALUES)	0.95	1.001			
s1 , s2 (STANDARD DEVIATIONS)	0.15	0.10			
n1, n2 (NUMBER OF DATA)	16	16			
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.75	1.75			
s (STANDARD DEV. ACROSS ALL DAT	ΓA) 0.13				
Z = ts ((n1 +n2)/(n1 n2)) ^ .5	0.08				
D = abs(x1 - x2)	0.05				
Signif. difference iff D > Z:	No difference	demonstrated			
Conclusion: $x = 0.97 \pm 0.04$					

# OCTOBER BLAST FURNACE SLAG PRODUCTION

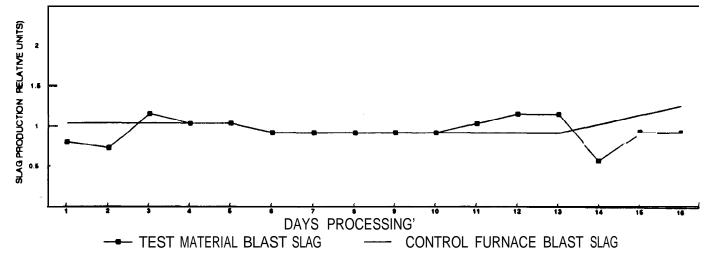


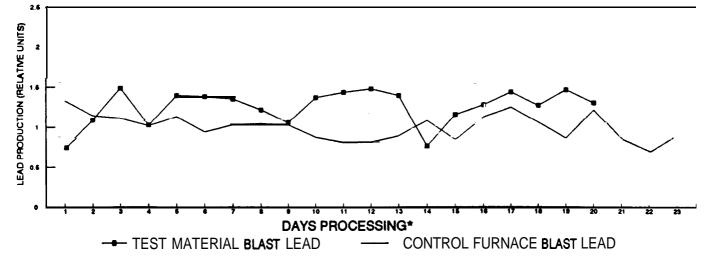
FIGURE B-6 Comparison of Reverberatory Slag Production- Test and Control Furnaces -October 1992

# 3.3.4 Sulfur Dioxide Emissions

CHMR did not study the sulfur dioxide emissions during the large-scale investigation because the preliminary investigation revealed that the emissions did not change significantly when test material was processed through the furnaces and the emissions are well controlled by Exide's scrubber system Therefore, CHMR assumed that the sulfur emissions would remain relatively constant, and did not obtain or analyze data on them. Plant personnel indicated that no extraordinary exceedances of permit conditions occurred during the three months that the tests were performed.

	TEST	CONTROL
VARIABLE (EXPANATION)	FURNACE	FURNACE
x1, x2 (AVERAGE DATA VALUES)	1.25	1.00
s1, s2 (STANDARD DEVIATIONS)	0.22	0.16
n1, n2 (NUMBER OF DATA)	20	23
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.71	1.66
s (STANDARD DEV. ACROSS ALL DATA)	0.23	
$Z = ts ((n1 + n2)/(n1n2))^{.5}$	0.11	
D = abs(x1 - x2)	0.25	
Signif. difference iff D > Z:		
Conclusion:	$x^2 - x^1 = -0.25 \pm 0.10$	)

OCTOBER BLAST FURNACE LEAD PRODUCTION



# FIGURE B-7 Comparison of Reverberatory Lead Production- Test and Control Furnaces-October 1992

# 3.3.5 Calcium

Several loads of material at the site contained up to 50 percent calcium. Small quantities of such materials could be blended with Exide's regular feedstock material. However, it is not possible for Exide to process large quantities of such material through its blast or reverberatory furnaces, as such materials tend to impair furnace production.

# TABLE B-7 ANOVA Analysis of Blast Slag Production – August - October 1992

	TEST	CONTROL
VARIABLE (EXPANATION)	FURNACE	FURNACE
x1, x2 (AVERAGE DATA VALUES)	0.87	1.00
s1, s2 (STANDARD DEVIATIONS)	0.44	0.43
n1, n2 (NUMBER OF DATA)	60	65
t1, t2 (FOR 90% CONFIDENCE LIMIT)	1.64	1.64
s (STANDARD DEV. ACROSS ALL DATA)	0.44	
$Z = ts ((n1 + n2)/(n1 n2))^{0.5}$	0.129	
D = abs(x1 - x2)	0.128	
Signif. difference iff D > Z:	No difference dem	onstrated
Conclusion:	$x = 0.94 \pm 0.06$	

ALL PRODUCTION VALUES HAVE BEEN NORMALIZED TO 1 BY DIVIDING THEM BY THE "NORMAL FEED" TO PROTECT EXIDE CONFIDENTIAL INFORMATION

# **APPENDIX C**

# THE USE OF SECONDARY SMELTING TECHNOLOGY TO RECLAIM LEAD FROM IRON-SHOT BRIDGE BLASTING MATERIAL

Description of Evaluation and Results and Discussion

November, 1993

## 1.0 INTRODUCTION

The Pennsylvania Department of Transportation (PennDOT) uses iron shot abrasive material to blast old paint from some state bridges. PennDOT collects the spent material and stores it in 55 gallon drums for disposal. CHMR obtained 16 drums (approximately 6.5 tons) of the blasting material from PennDOT to perform a qualitative evaluation to determine if the material could be processed through a secondary lead smelter for lead recovery. The main goal of the investigation was to determine if the material could be processed without causing any mechanical or production problems in the furnaces at Exide's secondary lead smelter in Reading, PA. The material used was from a bridge in Belle Vernon, PA. CHMR observed the modifications, if any, that were necessary to process the material in the most efficient manner.

This appendix details the evaluation conducted on iron shot abrasive material used by the Pennsylvania Department of Transportation (PennDOT) to blast the paint from a bridge in Belle Vernon, PA.

#### 2.0 METHODOLOGY

The material arrived at the smelter in sixteen 55 gallon drums. The material was inspected by Exide personnel upon its arrival at the site, and they determined that the material contained too much moisture, which was anticipated to cause problems in the reverberatory furnaces. Plant personnel advised that the material, including the barrel, be placed directly into Blast Furnace #2. The other blast furnace (#1) was processing regular feed, and this was to be used as a control furnace.

Approximately 4 hours into the experiment, CHMR personnel at the site were informed that the control furnace had been fed with a different type of "typical" feed material than the test furnace. This precluded CHMR from making any direct comparisons between the two blast furnaces. This was later verified upon analysis of the lead produced by each blast furnace prior to processing any test material. Even before the experiment, blast furnace #2 was producing lead that was significantly higher in antimony, arsenic, and tin. CHMR decided to compare the production during the experiment with the production in the same furnace before processing the test material.

A front-end loader was used to feed the barrels into the blast furnace at the rate of one barrel per charge, with the furnaces being charged approximately twice every hour. All 16 of the barrels were fed into the test furnace approximately 7 hours after the start of the experiment. Residence time in the furnaces ranges up to 3 hours. Therefore, all test material was processed roughly 10 hours after feeding the first barrel. Furnace operating parameters, such as back pressure and air and oxygen rates, were recorded and blast furnace slag samples were collected by CHMR personnel approximately every hour.

The material obtained from PennDOT varied in size and overall composition. A composite sample of the blasting material was obtained by removing some of the material from each barrel prior to processing through the blast furnace. The material was dirty brown in color with visible blue-green specks in it. The specks were most likely paint from the bridge. The sample also seemed to contain some dirt and small wood pieces. Table C-1 provides the analysis for the composite sample of the material as received.

Parameter	Composition
Iron	39.7 to 63.4%
Calcium	1.8%
Lead	3.2%
TCLP Lead	2.5 mg/L
Total Sulfides	7.0 mg/kg
Other	31.6 to 55.3%

TABLE C-1 PennDOT BLASTING MATERIAL

These results show that the main constituent of the blasting material is iron. The lead concentration is relatively low compared with reverberatory slag, which is typical blast furnace feed and which contains between 60 and 70 percent lead. The blasting material is not TCLP hazardous for lead, as the test yielded a value less than 5 mg/L.

# 3.0 RESULTS AND DISCUSSION

CHMR obtained daily production sheets, sample results, and other general information about furnace performance from Exide personnel and through direct observation in order to evaluate the processing of the bridge blasting material through the blast furnaces at the smelter.

#### 3.1 TEST MATERIAL

A composite sample of the 16 drums of the abrasive blasting material was collected prior to testing. test material was low in lead (3 to 4 percent) compared to typical blast feed, which averages 65 to 70 percent lead. The main component in the material is iron, since it is an iron shot abrasive blasting material. The iron is assumed to be beneficial to the blast furnace performance since iron is usually added to the furnace as a fluxing agent. The 55 gallon barrels that contained the blasting material also provided iron in the furnace.

#### 3.2 FURNACE PERFORMANCE

Discussions with plant management and furnace operators indicated that the test material did not cause any back-ups or other mechanical problems in the blast furnace. The observed results were expected since the amount of material being fed into the furnace was very small compared to the amount of typical feed. Plant personnel also mentioned that the smelter should be able to accept other iron-shot based abrasive material. However, they can not accept other types of blasting material that are silica based (such as "Black Beauty") because a high silica content has the potential to cause problems in the furnaces.

## 3.3 LEAD AND SLAG QUALITY AND PRODUCTION

The amount and nature of the slag and lead produced by the furnace are critical experimental parameters. The quality of the lead is important since the lead is the marketable product from the smelter. The blast furnace slag is one of the waste streams from the smelter, and disposal is costly.

#### 3.3.1 Lead Quality

The results of the lead analyses are presented in Table C-2. The analyses were performed on-site by Exide personnel using Spectrometer Lab Test 201 equipment. This equipment determines the amounts of trace metals in the lead sample. The "Lead Before Experiment" are samples taken from each blast furnace prior to processing the test material. These initial samples show that the lead produced in blast furnace #1 contains significantly different amounts of antimony, tin, arsenic, copper, and cadmium than the lead produced in blast furnace #2. This was due to the different feed piles that were being used for each furnace, according to Exide personnel. The difference in the feeds is what led CHMR to abandon furnace #1 as a valid control furnace.

	Pb	Sb	Sn	As	Cu	NI	Cd	Ċa	Se	S	A
LEAD BEFORE EXPERIMENT:	_			-		1					·······
BLAST #1 03:05 PM	9.636	1.95	0.43	0.08	0.45	0.04	0.15	0.11	0.01	0.03	0.01
BLAST #2, 03:05 PM	8,568	5.08	1.14	0.47	0.33	0.04	0.57	0.11	0.00	0.05	0.01
TEST MATERIAL BLAST LEAD:					t						
BUST #2,04:00 PM	8,566	5.08	1.17	0.44	0.31	0.04	0.53	0.01	0.01	0.04	0.00
BLAST #2,05:35 PM	10,236	5.15	1.08	0.42	0.28	0.03	0.46	0.10	0.01	0.03	0.02
BLAST X2. 07:00 PM	11,243	4.62	1.00	0.25	0.28	0.02	0.35	0.10	0.01	0.03	0.02
	از,	4.95	1.08	0.37	0.29	0.03	0.44	0.07	0.01	0.03	0.01
MEAN TEST BLAST LEAD:	1.500	± 0.321	± 0.09	± 0.12	± 0.02	± 0.01⊺	0.10	± 0.06	± 0.00	± 0.01	± 0.01

TABLE C-2 TEST MATERIAL BLAST LEAD ANALYSIS

$$MEAN \mu = x \pm \frac{1 s}{\sqrt{n}} .$$

The section in Table C-2 labeled "Test Material Blast Lead" refers to the samples taken from the test furnace during the processing of the abrasive blasting material. The "Lead Before Experiment" are.initial samples taken prior to testing. The table shows no significant difference, based on a 90 percent confidence limit, between the metals concentrations taken before and during the experiment.

# 3.3.2 Slag Quality

Table C-3 provides the analyses of the slag from the test blast furnaces. The results from blast furnace #1 were not used for the evaluation. CHMR decided to concentrate on the slag taken from blast #2 the test furnace, to see how the composition of the slag changed over the course of the evaluation. An initial slag sample was taken from the test furnace prior to testing, followed by five additional samples at various times during the processing of the test material. These samples were composites of two to three furnace taps.

Sample	% Pb	% Fe	Reactive Sulfides	_
Initial	3.75	38.7	1.12 ppm	ļ
1	5.10	44.7	ND*	1
2	3.90	40.8	ND*	i
3	2.45	42.5	0.23 ppm	ı
4	3.55	46.9	0.24 ppm	Ì
5	6.25	47.4	0.25 ppm	_

TABLE C-3 ANALYSIS OF SLAG SAMPLES

The data indicate that the test material had some small effects on the composition of the slag. The percent iron in the slag showed a slight increase, which may be due to the high iron content of the test material. The reactive sulfides present in the slag were slightly lower during the processing of the test material.

# 3.3.3 Slag and Lead Production

It is difficult to make a determination about the production of lead and slag since the material was only being fed for a 7 hour period. CHMR obtained Exide's daily production sheets from 9 days before the evaluation through the day after. The production sheets indicated that there was basically no affect on the amount of lead and slag produced during the test when compared to production data from before the test. CHMR concludes this is because of the relatively low feed ratio (10 percent by weight) of the test material to the furnace.

# 3.4 SULFUR DIOXIDE EMISSIONS

CHMR did not obtain sulfur dioxide emissions data for this evaluation. The iron-shot abrasive blasting material was initially low in sulfides (7 ppm) and was not expected to raise the sulfur dioxide emissions. Previous evaluations conducted with materials containing 2 to 3 percent sulfur showed no effect on emissions, which are well controlled.

# 3.5 FURNACE PARAMETERS

Certain furnace parameters, such as oxygen and air usage, and furnace back pressure, were monitored during the evaluation, and the results are given in Table C-4. The amount of oxygen needed in the test blast furnace was increased slightly (approximately 100 ft<sup>3</sup> per hour) after roughly 3 hours because of the

test material. Air usage did not fluctuate at all, and back pressure seemed to behave normally. There were no reports of any build-ups or other complications in the furnace that could be attributed to the test material.

 TABLE C-4

 TEST BLAST FURNACE OPERATING PARAMETERS

	<b>BLAST AIR</b>	BLAST O2	BACK	
TIME	(ft³/mìn)	(ft³/hr)	. PRESS (psi).	COMMENTS
12:30 PM	1000	4500	1.88	START FEEDING AT 12:45 PM
01:00 P M	1000	4500	1.88	FEEDING 1 BARREL PER CHARGE
01:45 PM,	1000	4500	1.88	ONE CHARGE PER HALF HOUR
02:40 PM	1000	4500	1.75	
03:00 PM	1000	4600	1.81	
03:40 PM	1000	4600	1.94	
04:30 PM	1000	4700	1.63	
05:00 PM	1000	4700	1.81	
06:00 PM	1000	4600	1.69	
07:00 PM	1000	4600	2.06	LAST BARREL AT 07:00 PM

# 4.0 CONCLUSIONS

Based on the study, CHMR concluded the following:

- The test material was fed to the furnace at a feed ratio of approximately 10 percent of the typical feed that is processed through the smelter. No effect was found on lead and slag production and quality, production costs, sulfur dioxide emissions, and furnace performance.
- The material can be easily fed into the blast furnace. The blasting material arrived in 55 gallon drums, and were charged directly into the furnace when processing this material.
- Exide plant management believe that they can accept iron shot abrasive blasting material. They cannot accept silica-based blasting material.
- Costs of processing the material in a secondary lead smelter are estimated to be below \$150 per drum.

These conclusions were based on one relatively short term test, with a relatively small amount of material processed. CHMR recommends additional testing and further monitoring of daily production data to determine if the blasting material has adverse effects on the slag and lead production and to better determine the process economics.

APPENDIX D

# RECLAMATION OF LEAD FROM BATTERY CASE MATERIALS FROM THE HEBELKA SITE

Description of Evaluation and Results and Discussion

February 5, 1992

## **1.0 INTRODUCTION**

The Center for Hazardous Materials Research (CHMR) and Exide/General Battery Corporation (Exide) engaged in a joint research study to determine the feasibility and economics of using Exide's secondary lead smelters for the recovery of lead from lead-containing materials from Super-fund sites and other facilities. As part of that project, CHMR/Exide processed approximately 8 tons of battery case materials from the Hebelka Corporation (Hebelka) site in Upper Macungie Township, Pennsylvania.

The primary purpose of the evaluation was a qualitative determination of whether or not battery case materials have negative affects on the reverberatory furnaces when the size is reduced significantly. A previous investigation showed that large pieces of battery cases slow the reverberatory furnace production. The Hebelka Superfund site was originally an auto salvage yard containing approximately 1,000 cubic yards of rubber battery case material from an unknown battery breaker source. The materials consisted of large broken pieces of rubber and polypropylene battery cases.

CHMR/Exide received permission in January 1992 from Patrick Augustin, the EPA Project Officer for the research, and Fred McMillan, the Hebelka Remedial Project Manager from EPA Region III, to proceed with the evaluation. The material was processed on February 5, 1992.

The main conclusion from the evaluation was that the battery case materials, after being milled to less than <sup>1</sup>/<sub>4</sub> inch, could be successfully fed to the reverberatory furnaces at a weight ratio of at least 17%.

The purpose of the evaluation was to determine if reducing the size of battery case materials affects reverberatory furnace performance. The Hebelka Super-fund site in Upper Macungie Township, Pennsylvania, was used as the source of these materials. The materials were processed over a five hour period at Exide's Reading, Pennsylvania, secondary lead smelting facility.

#### 2.1 RECLAMATION PROCESS

The reclamation of lead from Superfund and other lead-containing materials is based on existing lead smelting procedures. The material are first collected or excavated. Next, they may be preprocessed to reduce particle size, and to remove rocks, soil, and other debris. The materials are then transported to the smelter. An overview of the reclamation and smelting process can be found in the main report.

# 2.2 EXPERIMENTAL DESIGN

The main objective of the evaluation was to feed Hebelka material to the furnaces at a known feed ratio and determine if the material significantly changed furnace operation or parameters. This was a short term test lasting only five hours, but the intention was to see if reducing the size of the battery case material would allow it to be processed in the reverberatory furnaces. During the Tonolli experiment, one of the conclusions was that large battery case pieces (up to 14") cause build-ups in the reverberatory furnaces. This is because the larger pieces burn more slowly. Smelter management determined that a relatively short term test would be sufficient to detect buildups within the reverberatory furnace.

The material from the Hebelka site was reduced in size in a hammermill until it passed through a ¼ inch screen. After size reduction, the material was fed to one reverberatory furnace. No test material was fed to the blast furnaces, as the ability of blast furnaces to reclaim lead from battery case material was evaluated in a previous study (Tonolli). The second reverberatory furnace was fed typical material. Conducting the test in this manner allows for comparison of the operation of the "test" furnace with the "control" furnace.

During the evaluation, the operating parameters (fuel usage, oxygen usage, slag production, lead production, etc.) for each set of furnaces were obtained. In addition, samples of slag and lead were obtained from each furnace to be used to determine effects on production quality.

# 2.3 EXPERIMENTAL MATERIAL

The Hebelka material processed during this evaluation consisted primarily of chunks of rubber battery cases, ranging in size from a fraction of an inch to full-sized (approximately 12-inch by 8-inch x 6-inch) battery cases. The cases were black in color, and appeared to be a mixture of ebonite rubber with some polypropylene cases. Much of the material was covered with a thick reddish-purple sludge, which contained approximately 80% of the lead. Tests of the material showed average concentrations of 14.7% lead. The composition of the material is further characterized in Table D-1.

# 2.4 CHRONOLOGY OF THE EVALUATION

The following represents a chronology of the activities conducted in support of the evaluation between January 24 and February 5, 1992.

#### 2.4.1 Material Acquisition (1/24/92)

Eight tons of the battery case material were excavated and loaded in two trucks by an EPA contractor. After the excavation and loading of the material, the equipment and trucks were decontaminated under EPA supervision. The material was transported to Exide's smelting facility weighed, and then stored in an area isolated from other feed material.

# 2.4.2 Size Reduction (2/5/92)

The waste battery case material varied in size between a fraction of an inch and large pieces up to 14 inches. Based on a previous experiment with rubber battery case material from the Tonolli Super-fund site, it was determined that rubber battery case material would need to be reduced in size prior to processing the material.

The material leaving the hammermill ranged in size from a fine dust to a particle size around ¼ inch. The material was collected in cubic yard boxes and mixed on-site with regular feed material before processing. The material in each box weighed approximately 800 pounds.

#### 2.4.3 Processing in the Furnace (2/5/92)

The regular reverberatory furnace feed consisted of spent battery parts, lead oxides, sludge and other lead containing material, mainly from Exide's sink/float separation system. The rubber battery cases were mixed with regular feed material at a 17% weight ratio (e.g., 17 pounds of material to 83 pounds of regular feed). The mixed material was fed into reverberatory furnace #1. Reverberatory furnace #2 served as the experimental control. The material was processed between 12:20 PM and 5:20 PM on February 5, 1992.

During the experiment, reverberatory furnace #1 was tapped twice for slag. The slag from the furnace flowed well and there was no noticeable build-up within the furnace. CHMR personnel continued to monitor the furnace performance until 7:30 PM, with no problems noted in the furnace.

# 3.0 RESULTS AND DISCUSSION

In order to assess the viability of the process, CHMR/Exide recorded the settings of various plant parameters, the production figures for both reverberatory furnaces, and stack parameters. In addition, CHMR obtained samples of the input material, and output slag and lead. The sampling was conducted in accordance with the EPA approved Quality Assurance Project Plan (QAPP). The results from the sampling and analysis are presented and discussed below.

#### 3.1 TEST MATERIALS

The test material was analyzed for lead, silica, sulfur, carbon, and iron concentrations, as well as bulk density, and heat value. Table 1 shows the results of analytical tests on the test materials. The results for silica and sulfur are below the thresholds at which the material would impair the furnaces. The carbon and iron percentages are favorable for smelting. The amount of lead in the material (14.7%) is considerably below that of normal reverberatory furnace feeds, which average about 60% lead.

Sample #	Pb (%)	S (%)	SiO <sub>2</sub> (%)	C (%)	Fe (%)	Density (lb/ft <sup>3</sup> )	Heat (BTU/lb)
1	13.9	3.3	5.2	52	0.45	58	9670
2	18.4	1.3	4.2	60	0.36	51	7820
3	11.7						
Average	14.7	2.3	4.7	56	0.41	55	8750

Table D-1. Hebelka Battery Case Material Analysis

#### 3.2 SLAG AND LEAD PRODUCTION AND QUALITY

The amount and nature of slag and lead produced by the furnaces were critical experimental parameters. The amount and quality of lead produced is important because lead is the salable product from the smelter. The reverberatory slag characteristics also important, because this slag is used as feed material for the blast furnace.

Composite slag samples were taken as the slag was being tapped from the furnaces throughout each shift from reverberatory furnace #2. Grab samples of slag were taken from furnace #1. The molten slag samples were collected by the Exide operators by inserting a shovel into the furnaces and extracting the sample, in accordance with the QAPP. The results of the slag analysis are presented in Table D-2.

The amount of lead production was determined by collecting Exide's daily production reports, which detail the entire smelter activity for each day. The quality of the lead was determined through analysis with a Spectrometer Lab Test 100. This analyses, which is performed on-site by Exide personnel, provides the amount of several trace metals in lead samples taken from the furnace. The results from these tests can be seen in Table D-3.

Performing a paired two-sample T-test for means for each metal in the table revealed that only the antimony showed a difference between the two means. The test was performed with a 95% confidence level.

Sample	Furnace	Pb (%)	S (%)	Sb (%)	C (%)
1	Test	72.0	8.2	3.0	0.76
2	Test	42.2	1.3	5.8	0.20
3	Test	62.4	7.1	2.8	0.74
4	Test	73.1	10.5	1.5	2.56
5	Test	59.1	7.3	2.7	1.42
6	Test	65.7	6.9	3.0	0.06
	Average	62.4	6.9	31	0.96
Sample	Furnace	Pb (%)	S (%)	Sb (%)	C (%)
7	Control	52.5	6.5	3.1	0.85
8	Control	60.8	6.1	4.6	0.99
• • •	Average	56.7	6.3	3.9	0.92

Table D-2. Composition of Slag Samples from Reverberatory Furnaces

# **3.3 INPUT FURNACE PARAMETERS**

The input parameters recorded included furnace fuel and oxygen usage. These parameters were recorded, and are summarized in Table D-4. The fuel usage remained constant for both furnaces. The oxygen had to be increased in both furnaces, however, the increase was greater for the test furnace than for the control furnace. This is consistent with a furnace being fed a material that is high in carbon content.

# 3.4 SULFUR

Rubber battery cases typically contain between 2% and 10% sulfur. The Hebelka rubber battery casings contained an average of 2.3% sulfur. Once inside a furnace, this sulfur could exit the process by three means: through stack gas emissions, through the calcium sulfate sludge generated by the emissions control system, or in the slag.

Time	Furnace	Sb (%)	Sn (%)	As (%)	Cu (%)	Ni (%)	Bi (%)	Ag (%)	Fe (%)	Zn (%)	Te (%)
6:30	Test	0.1910	0.0001	0.0002	0.0374	0.0162	0.0125	0.0023	0.0006	0.0005	0.0033
6:30	Control	0.1009	0.0006	0.0004	0.0438	0.0320	0.0118	0.0021	0.0006	0.0007	0.0045
14:00	Test	0.1805	0.0001	0.0002	0.0462	0.0462	0.0105	0.0018	0.0006	0.0004	0.0025
14:00	Control	0.0669	0.0002	0.0001	0.0435	0.0313	0.0107	0.0017	0.0005	0.0003	0.0030
16:15	Test	0.1801	0.0001	0.0002	0.0412	0.0478	0.0108	0.0017	0.0006	0.0004	0.0022
16:15	Control	0.0507	0.0000	0.0001	0.0332	0.0245	0.0103	0.0017	0.0005	0.0003	0.0023
19:00	Test	0.1750	0.0001	0.0002	0.0347	0.0244	0.0110	0.0016	0.0005	0.0003	0.0014
19:00	Control	0.0741	0.0001	0.0001	0.0359	0.0249	0.0107	0.0015	0.0005	0.0004	0.0026
Average	Test	0.1817	0.0001	0.0002	0.0399	0.0337	0.0112	0.0019	0.0006	0.0004	0.0024

Table D-3. Trace Metals in Lead Samples.

Average				0.0002							
Average	Control	0.0732	0.0002	0.0002	0.0391	0.0282	0.0109	0.0018	0.0005	0.0004	0.0031

	Oxygen us	sage (cfm)	Gas usage	(rel. units)
Time	Test	Control	Test	Control
13:30	1500	1500	6.8	8.0
14:00	1500	1900	6.8	6.0
14:30	1500	1700	6.8	8.0
15:00	2100	1800	6.8	6.0
15:30	2200	1800	6.8	6.0
16:00	2200	1800	6.8	6.0
16:30	2200	1900	6.8	6.0
17:00	2000	1800	6.8	6.0
17:30	2100	1800	6.8	6.0

Table D-4. Furnace Parameters During Test.

#### 3.4.1 Sulfur Dioxide Emissions

In order to feasibly process the Hebelka material, it is important that the test materials do not cause an increase in sulfur dioxide (SO<sub>2</sub>) emissions above Exide's permitted emissions limits of approximately 110 parts per million (ppm). No such increase was anticipated because the concentration of sulfur in the normal furnace feed is about 1 to 2%, and also because Exide has a sulfur dioxide control system. However, CHMR/Exide still monitored sulfur dioxide emissions to determine if an increase occurred.

CHMR/Exide obtained data concerning sulfur dioxide emissions from the facility's SO<sub>2</sub> Continuous Emission Monitor (CEM). Average and initial data values were collected by the operator and CHMR/Exide project personnel on duty during each shift. Each stack system is connected to the corresponding blast and reverberatory furnaces. The average stack emission in system #1, to which the test material was fed, was 56 ppm, while the average stack emission in system #2 was 78 ppm. Thus, the emissions remained considerably below the permit levels of 110 ppm.

#### 3.4.2 Calcium Sulfate Sludge

During each shift, specific gravity measurements of slurry generated from the air emission control equipment (scrubbers) were taken by the operator on duty. The specific gravity is related to the calcium sulfate concentration in the slurry, and therefore gives a measure of total sulfur uptake in the sludge. Slurry specific gravity measurements for both stacks averaged 1.25, which indicates no significant difference between the slurry produced in the furnace system fed the Hebelka material and that produced in the system fed only normal feeds.

At the end of each shift, the amount of slurry was calculated by counting the number of truckloads transported from the scrubbers to the on-site sludge dewatering facility. No increase was found in the number of truckloads produced each shift.

APPENDIX E

# RECLAMATION OF LEAD FROM DEMOLITION MATERIAL FROM A HUD RENOVATION

Description of Evaluation and Results and Discussion

December 1991

# 1.0 INTRODUCTION

The Center for Hazardous Materials Research (CHMR) and Exide/General Battery Corporation (Exide) engaged in a joint research study to determine the feasibility and economics of using Exide's secondary lead smelters for the recovery of lead from lead-containing materials found at Superfund sites and other sources. As part of this study, CHMR/Exide processed demolition material (primarily wood) from the renovation of a women's shelter in Montgomery County, PA.

One common, but often overlooked, source of lead is demolition material from residential housing coated with lead based paints. Many houses, especially older ones, have interior and exterior wood which can contain lead in excess of 1%. The current treatment for lead-containing demolition waste is to place it into sanitary landfills with other municipal wastes. As an alternative to landfilling, CHMR and Exide evaluated the potential for processing lead-containing demolition material in a secondary lead smelter.

The demolition material is from the front and back porches of the Laurel House, a women's shelter in Montgomery County, PA. The deteriorating porches were coated with lead based paint. The Montgomery County Housing and Community Development (MCHCD) Office contracted an environmental firm to perform the necessary abatement. Nearly five tons of lead-containing waste was generated.

The main goal of this short term experiment was to determine the effects that a wooden feed would have on the reverberatory furnaces. After receiving consent from the EPA, the demolition material was transported to Exide's secondary lead smelter in Reading, PA, and accumulated until December 16, 1991, when the material was processed through the smelter.

# 2.0 DESCRIPTION OF EVALUATION

The purpose of this evaluation was to determine the affects on furnace production that a wood-based feed would have. Material was acquired from the Laurel House, a woman's shelter in Montgomery County, PA. The demolition materials were processed over **a** ten hour period at Exide's Reading, PA smelter.

# 2.1 RECLAMATION PROCESS

The reclamation of lead from Superfund and other lead-containing materials is based on existing lead smelting procedures. The material are first collected or excavated. Next, they may be preprocessed to reduce particle size, or to remove rocks, soil, and other debris, The materials are then transported to the smelter. An overview of the reclamation and smelting process can be found in the main report.

# 2.2 EXPERIMENTAL DESIGN

The main objective of this evaluation was to process the demolition material in the reverberatory furnaces at a known feed ratio and determine if the material significantly changed furnace operation or performance. Unlike other evaluations, material was charged to both reverberatory furnaces. Therefore, this test did not have a "test" and "control" furnace and so comparisons can only be made to determine how both furnaces performed with respect to each other while processing the same feed.

The preprocessing phase for this evaluation involved reducing the size of the demolition material with a pallet shredder, and then storing in an area separate from other feed material at the smelter. During this evaluation, certain operating parameters for each reverberatory furnace were obtained, including air and natural gas usage. Samples of slag and lead were obtained from the reverberatory furnaces for analysis.

# 2.3 EXPERIMENTAL MATERIAL

The demolition material consisted primarily of pieces of wood coated with lead-based paint. The material arrived at the smelter on pallets in three to six foot lengths. The material had an average lead concentration of 1.2%, and an average TCLP lead level of 13.7 mg/l. The ash content of the material was determined to be 4% by weight.

# 2.4 CHRONOLOGY OF THE EVALUATION

The following is a chronology of the activities conducted for this evaluation between November 21 and December 16, 1991.

# 2.4.1 Material Acquisition

MCHCD contacted CHMR in early November and expressed interest in working with the lead reclamation project. CHMR consulted with Patrick Augustin of the EPA, who agreed that the material could be processed as part of the research project. Starting on November 21, the environmental contractor responsible for the lead abatement transported several pallets of the demolition material to the smelter in Reading, PA. Once at the smelter, the material was weighed, processed through a

pallet shredder, and stored separate form other smelter feed material.

# 2.4.2 Day One (12/15/91)

CHMR personnel arrived at the site and were introduced to smelter management and supervisors. CHMR personnel also received health and safety training regarding the operations and equipment at the smelter.

The density of the demolition material was determined by weighing a known volume of the material This procedure was duplicated seven times, and the average density was found to be 15.7 lbs/ft<sup>3</sup>.

Three composite sample of the initial demolition material were collected in accordance with section 3.6 of the Quality Assurance Project Plan (QAPP). Grab samples of the lead and slag produced by the reverberatory furnaces prior to processing any test material were also collected.

# 2.4.3 Day Two (12/16/91)

More samples of the reverberatory lead and slag were taken before feeding the demolition material The test material and regular feed were mixed prior to starting the experiment.

At 10:30 AM, a mixture containing 10% by weight (50% by volume) demolition material was charged to both furnaces. At approximately 1:30 PM, plant management noticed a buildup of material in both of the reverberatory furnaces. Test feed to the furnaces was suspended and oxygen flow was increased to try and correct this problem.

Exide plant management decided that the feed ratio should be lowered to 5% by weight to try and prevent any further buildups from occurring in the furnaces. The mixed feed was started again at 3:00 PM and continued until 8:45 PM, with no other problems noticed in the furnace. All slag and lead samples taken during the course of the investigation were taken in accordance with the QAPP.

#### 3.0 RESULTS AND DISCUSSION

CHMR obtained enough data to determine whether or not the material could be successfully run at the tested feed ratios. The test was not long enough to determine the total effects that processing the demolition material had on the furnaces. The following sections give more detailed information on the operating parameters for the two reverberatory furnaces.

#### 3.1 INPUT PARAMETERS

CHMR personnel recorded air flows, oxygen volumes, natural gas usage, and types of material charged into the furnace. No major changes were noted in air or natural gas usage throughout the length of the experiment. The percent oxygen enrichment had to be increased for a short time to reduce the buildups in the reverberatory furnaces that occurred from processing the test material at a 10% by weight mixture. Overall the results were inconclusive: insufficient material was processed to adequately determine if changes in process settings, i.e., natural gas, oxygen, were necessary to process the demolition material.

Three samples of the test material were collected prior to the start of the evaluation, and two were sent to separate laboratories. Table E-1 shows the composition of the feed material. The material has a lead concentration of 0.96% in one analysis and 1.5% in another. The average leachable lead is 13.7 mg/l, based on TCLP analysis.

Feed	TCLP Pb	Pb	Pb	Ash
Sample	(mg/l)	(ppm)	(%)	(%)
1	11.3	9,594	0.96	
2	16.1		1.5	4

Table E-1. Ana	vsis of	Demolition	Material
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#### 3.2 SLAG PRODUCTION AND QUALITY

The reverberatory slag production and quality are important concerns because the slag is used as feed to the blast furnaces. CHMR acquired Exide's daily production sheets for the week of December 13 through December 18, 1991. The test appeared not to affect the slag production at all, however, since the test was short term with only 9,000 pounds processed, no conclusions can be drawn about how the demolition material affected the slag production.

Composite slag samples were taken as the slag was tapped throughout each shift. These samples were taken by inserting a shovel into the furnace and removing the slag, in accordance with section 3.6 of the QAPP. At the end of each shift, CHMR personnel collected the samples and split them with Exide. The reverberatory slag samples were analyzed for percent lead, tin, antimony, and arsenic. Tables E-2 and E-3 below show the analysis of the slag samples taken before, during, and after the evaluation. Although furnace #1 showed a slight decrease in slag lead content, Exide personnel said that it is in the acceptable range for the reverberatory slag. Furnace #2 showed a slight increase, but leveled out during the evaluation.

Table E-2. Reverberatory Furnace #1 Slag Samples.

When	Pb (%)	Sb (%)	Sn (%)	As (%)
Before	60.4	3.40	0.80	0.22
During	55.3	5.07	1.09	0.29
During	50.0	1.25	0.25	4.25
After	47.2	6.56	1.50	0.36

Table E-3. Reverberatory Furnace #2 Slag Samples.

When	Pb (%)	Sb (%)	Sn (%)	As (%)
Before	51.4	5.34	1.52	0.43
During	66.5	3.60	0.66	0.24
During	69.3	0.56	0.06	2.80
After	66.2	3.28	0.62	0.22

# 3.3 LEAD PRODUCTION AND QUALITY

The production and quality of lead is an important parameter because lead is the salable product from the smelting process. CHMR anticipated that the demolition material would cause a decrease in the production of lead. This would not be caused by any change in the slag, but because the demolition material comprised much of the volume of the feed. Therefore, any decrease would be due to the decreased throughput. However, the material was not run long enough to notice any affects on the lead production.

Lead samples were collected throughout the evaluation and analyzed with a Spectrometer Lab Test 100. This analyses, which is performed on-site by Exide personnel, provides the amount of several trace metals in lead samples taken from the furnace. CHMR could not get this data, however Exide plant supervisory personnel noted that there was no change in lead quality throughout the evaluation

### 3.4 SULFUR

Once material is inside a furnace, the sulfur present in the demolition material can exit the process by three means: through stack gas emissions, through the calcium sulfate sludge generated by the emissions control system, or in the blast and reverberatory slag.

# 3.4.1 Sulfur Dioxide Emissions

Sulfur dioxide  $(SO_2)$  emissions cannot exceed Exide's permitted emissions limits of 110 ppm.  $SO_2$  emissions at Exide are constantly monitored and recorded on a strip chart recorder. However, at the time of the evaluation, Exide was having mechanical problems with their  $SO_2$  Constant Emission Monitor (CEM). Plant personnel estimated that the sulfur dioxide levels never exceeded 100 ppm based on the limited data that they could acquire.

# 3.4.2 Calcium Sulfate Sludge

Calcium sulfate sludge is generated by the emissions control equipment (scrubbers) at the smelter. Specific gravity measurements of the slurry were taken approximately every hour by Exide personnel. The specific gravity is related to the amount of calcium sulfate in the slurry and therefore allows for an estimate of sulfur uptake in the sludge. The average specific gravities over the course of the experiment were 1.22 and 1.20 for furnaces 1 and 2, respectively. There was also no increase in the daily amount of calcium sulfate sludge generated.

# 4.0 CONCLUSIONS

Based on the limited evaluation conducted, CHMR/Exide concluded the following:

- It is technically feasible to reclaim lead from wood demolition materials, provided they are appropriately sized to fit in the furnaces.
- The material burns well in the furnaces, and did not present any major difficulties based on its combustive properties (i.e., did not cause a build-up within the reverberatory furnaces).
- The economics of recovering lead from such materials are difficult to assess. However, the value of the material as a feed to the smelter is minimal because it contains such a low concentration of lead. The use of secondary lead smelters is only likely to be economical when the demolition material is a hazardous waste, and therefore cannot be disposed economically in a municipal or demolition landfill.