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Technology Development

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INTERIM REPORT

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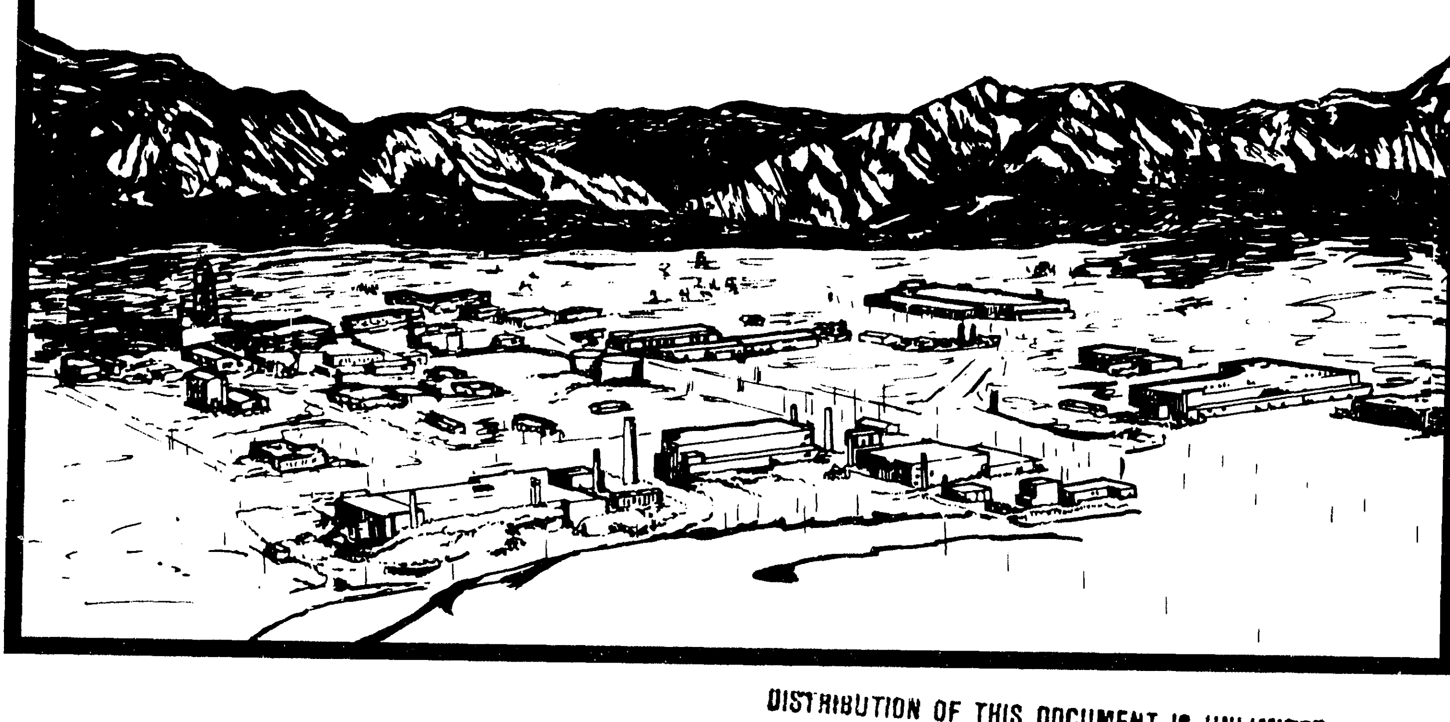
EG&G ROCKY FLATS



EQUIPMENT EVALUATION FOR LOW DENSITY POLYETHYLENE ENCAPSULATED NITRATE SALT WASTE AT THE ROCKY FLATS PLANT

INTERIM REPORT
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EXECUTIVE SUMMARY

Mixed wastes at the Rocky Flats Plant (RFP) are subject to regulation by the Resource Conservation And Recovery Act (RCRA). Polymer solidification is being developed as a final treatment technology for several of these mixed wastes, including nitrate salts. Encapsulation of nitrate salts with low density polyethylene (LDPE) has been the preliminary focus of the RFP polymer solidification effort.

Literature reviews, industry surveys, and lab-scale and pilot-scale tests have been conducted to evaluate several options for encapsulating nitrate salts with LDPE. Most of the effort has focused on identifying compatible drying and extrusion technologies. Other processing options, specifically meltration and non-heated compounding machines, were also investigated.

The best approach appears to be pretreatment of the nitrate salt waste brine in either a vertical or horizontal thin film evaporator followed by compounding of the dried waste with LDPE in an intermeshing, co-rotating, twin-screw extruder. Additional pilot-scale tests planned for the fall of 1993 should further support this recommendation.

Preliminary evaluation work indicates that meltration is not possible at atmospheric pressure with the LDPE (Chevron PE-1409) provided by RFP. However, meltration should be possible at atmospheric pressure using another LDPE formulation with altered physical and rheological properties: Lower molecular weight and lower viscosity (Epoline C-15). Contract modifications are now in process to allow a follow-on pilot scale demonstration. Questions regarding changed safety and physical properties of the resultant LDPE waste form due to use of the Epoline C-15 will be addressed.

No additional work with non-heated mixer compounder machines is planned at this time.

INTRODUCTION

Mixed wastes generated and stored at the Rocky Flats Plant (RFP) are subject to regulation under the Land Disposal Restrictions (LDR) of the Resource Conservation And Recovery Act (RCRA). The Federal Facilities Compliance Agreement [1] (FFCA II) allows RFP to continue operations while pursuing the development and implementation of treatment technologies for both newly generated and stored mixed wastes.

The Comprehensive Treatment and Management Plan [2] (CTMP) describes the DOE strategy for achieving compliance with the RCRA LDR. The CTMP also identifies technical performance criteria, schedules, and treatment methods for the various mixed wastes at RFP. The CTMP identifies polymer solidification as a potential final treatment technology for several mixed wastes, including nitrate salts, lead metal, ash, sludge, and secondary waste generated from organic destruction technologies.

Encapsulation of nitrate salts with low density polyethylene (LDPE) has been the preliminary focus of the RFP polymer solidification effort. As a low level mixed waste destined for disposal at the Nevada Test Site (NTS), the final LDPE waste form must meet stringent regulatory and disposal site criteria, including free liquid, dispersible solid, and Toxicity Characteristic Leaching Procedure (TCLP) requirements. Initial results show promise for LDPE encapsulation as a final treatment technology for nitrate salts.

This interim report describes information compiled from industry surveys, courses, and consultations. Results from on-going process equipment evaluations required for LDPE encapsulation of nitrate salts are also summarized.

BACKGROUND

Current Process Scheme

Aqueous waste streams from throughout the Rocky Flats Plant (RFP) are treated by Liquid Waste Treatment Operations in Building 374. Wastes from production operations are treated by hydroxide precipitation to remove actinides. The wastes are predominantly composed of the nitrate salts of various metals. Chlorides and sulfates are also significant components. The effluent from the precipitation process is combined with low specific activity wastes such as laundry wastes and incidental waters from solar evaporation ponds and interceptor trenches. This solution is concentrated to an approximate 35 weight % brine in a quadruple-effect evaporator and is then dried in a spray dryer. The spray dried salts are hollow microspheres with diameters ranging from less than 0.3 microns to greater than 10 microns (approximately 0.15 number fraction less than 0.3 microns). The nitrate salts have low specific activity (approximately 400 pCi/g) and have a bulk density ranging from 0.35 to 0.60 g/cc. The spray dried nitrate salts are combined with brine and cement to produce the "saltcrete" waste form.

Alternate Process Scheme

The advantages / disadvantages of low density polyethylene (LDPE) encapsulation of nitrate salts, and the characteristics of the final LDPE waste form have been discussed in previous reports [3,4,5], and therefore will not be repeated here. The processing alternatives to the current "saltcrete" waste form include compatible combinations of drying and extrusion technologies and other processing options.

The three (3) drying equipment options considered for evaluation include a vertical thin film evaporator, a horizontal thin film evaporator, and a horizontal rotary blender/dryer. The three (3) extrusion equipment options considered for evaluation include a single screw extruder, an intermeshing counter-rotating twin screw extruder, and an intermeshing co-rotating twin screw extruder. The two (2) other options considered for evaluation were a mixer/compounder and a meltration evaporator.

OBJECTIVES

The objectives of this work are:

To survey the polymer compounding industry for best demonstrated technologies and to access industry expertise.

To establish a technology baseline for future development effort.

To identify and evaluate alternate process equipment for application to low density polyethylene (LDPE) encapsulation of nitrate salts.

To investigate alternate processes for preliminary compatibility and to demonstrate most likely alternate equipment on a pilot scale.

INFORMATION ASSESSMENT

INDUSTRY SURVEY

Both a literature search and an industry survey have been conducted to provide an understanding of the current state of technologies within the polymer compounding industry. Several companies have been contacted for information and assistance. A wide spectrum of related vendors of ancillary equipment have also been contacted. Attendance at seminars, conferences, and trade exhibitions has provided valuable avenues for contact. Selected Trip Reports are included as Appendix 1. Contractors within the DOE / DOD complex have also been accessed for information. In addition, a consultant has been retained to facilitate communication with other facilities involved in polymer solidification of waste, especially foreign nuclear power facilities. Information complied from these varied sources has been instrumental in assigning priorities among the alternate processes, in establishing selection criteria for the major equipment options, and in identifying the most likely alternate process equipment.

The companies contacted include:

Adtechs Corp. (JGC)
American Leistritz Extruder Corp.
Artesan Ind.
Berstorff Corp.
Draiswerke Inc.
Fitzpatrick Co.
GEA Canzler Co.
Hardy Instruments
Killion Extruders Inc.
LCI Corp.
Pacific Nuclear Services
Paul O. Abbe Inc.
Polymer Processing Institute (at Stevens Institute of Technology)
Rheometrics Inc.
Tecnetics Inc.
Teledyne Readco
Thermal Engineering of Arizona
Welding Engineers Inc.
Werner Pfleiderer Corp.
Yamato Scientific America Inc.

The DOE / DOD contacts include:

Martin Marrietta / ORNL
Westinghouse / Hanford
Indian Head Naval Surface Warfare Center Continuous Processing Facility
Associated Universities Inc. / Brookhaven National Laboratory

COURSE WORK

Information has also been gathered from classroom instruction and course work. A variety of courses were completed. Subjects range across many related areas.

Surveys of available process equipment and performance characteristics, funaamental principles of operation and operating envelopes, theory and industry consensus practices, and impact of feed material properties on process design, are all subjects of study.

Courses attended include:

Class Title	Sponsoring Organization
Extrusion Workshop	Killion Extruders Inc. Newark, New Jersey, June, 1992
Polymer Processing Operations	AICHE Fall National Meeting Minneapolis, Minnesota, August, 1992
Mixing and Compounding in Polymer Processing	University of Wisconsin at Milwaukee April, 1993
Polymer Properties	AICHE Spring National Meeting Houston, Texas, March, 1993
Particle Enlargement by Granulation and Compression	Powder and Bulk Solids Conference Chicago, Illinois, May, 1993
Feeder Performance and Design Criteria for Efficient Discharge of Bulk Solids	Powder and Bulk Solids Conference
Advances in Communition: Fine Grinding	Powder and Bulk Solids Conference
Designing Mechanical Conveyors / Feeders for Handling Bulk Solids	Powder and Bulk Solids Conference

EXTRUDER EVALUATION

The fine particle size and low bulk density of the RFP nitrate salt waste stream and stringent waste acceptance criteria make the selection of proper processing equipment of extreme importance. Of primary importance is the ability of the extruder to thoroughly compound LDPE with the salt and consistently extrude the product. While many extruders are capable of mixing powders with the LDPE melt, selection of the optimum unit will maximize product quality and minimize operational difficulties. The purpose of this section is to: 1) establish criteria to be used in extruder selection, and 2) to evaluate commonly available compounding technologies against these criteria. Ultimately, this will result in the selection of the compounder that should best meet the requirements for the immobilization of the nitrate salt waste stream.

SELECTION CRITERIA

The selection criteria determine the most likely equipment option within the compounding operations and also provide a benchmark of comparison for the meltration evaporator alternative. In addition, the selection criteria provide the framework for decisions affecting both upstream (drying operations and ancillary equipment) and downstream (ancillary equipment) processing alternatives. The selection criteria described in the following text were developed from literature reviews, course work, and RFP laboratory scale experience [6-20].

1. Dispersive Mixing

As the salt is distributively mixed with the LDPE it tends to agglomerate into clumps within the melt. The dispersive mixing process breaks up these clumps and wets the surface of the salt with liquid polymer, thereby encapsulating the salt. With dissimilar materials such as nitrate salt (polar) and LDPE (nonpolar), the wetting of each particle surface with the liquid is unfavorable and considerable energy must be expended to bring it about. This type of mixing is called dispersive mixing because it tends to disperse the powder into the melt. Two mechanisms are responsible for dispersive mixing; 1) shear stress, and 2) elongation.

First, shear stress can overcome the attractive forces holding the particles together, thus breaking up the salt clumps and dispersing the particles throughout the LDPE. The shear stress can be increased by reducing the distance between the two surfaces and by increasing the viscosity of the liquid. This shear tends to cause a mechanical failure of the weakest elements in the mixture. In this case that would be the salt clumps which first stretch and then break. Care must be taken to conduct this mixing at the proper polymer temperature so that melt viscosity and shear stress will be optimized.

Second, elongation of the melt can cause mixing. The salt clumps can be viewed as behaving like a bubble filled with particles. The stretching of the bubble produces an instability in the bubble, essentially a 3 dimensional wave. When the amplitude of this wave reaches the diameter of the stretched bubble, the bubble spontaneously reconfigures itself into several smaller bubbles. The process can then be repeated on the smaller salt clumps formed until individual particles are encapsulated. Simply forcing the fluid through a narrow gap produces an elongation that disperses the particles.

Twin screw extruders provide both shear mixing through movement of the screws and elongational mixing by forcing the melt through gaps between the screws. The addition of mixing elements can optimize this and provide nearly complete wetting of the particles with the LDPE melt. Single screw extruders provide little shear mixing and virtually no elongational mixing. The circulation of the melt in the screw channels is such that the shear mixing near the barrel is canceled by shear in the opposite direction when the melt circulates near the screw.

Elongational mixing by the melt moving across screw flights is minimal because only a small fraction of the melt crosses the flights. Mixing elements can be effective by forcing the melt through a narrow gap. However even with mixing elements the single screw extruders are considered only fair at dispersive mixing.

2. High Filler Content

With a high waste loading being crucial to the economics of this process, the ability of the unit to process mixes with a high filler content is very desirable. A small increase in waste loading could result in a large improvement in the economic viability of the technology.

Twin screw extruders are designed for compounding high filler volumes into polymers. This is primarily a result of superior distributive and dispersive mixing capabilities. Although single screw extruders are occasionally used for compounding powders into polymers, the efficacy is much less than that of twin screw units for high filler contents and fine powders.

3. Powder Feeding Capability

The product of both the current nitrate salt spray dryer and all anticipated replacement dryers is expected to be a fine powder. Therefore, the ability to easily feed fine powders is essential.

Single screw extruders depend on a vertical gravity feed of the powder (filler) down the throat of the extruder. This can be very inefficient for powders that have a small particle size and low density. Such powders will tend to "entrap air and become fluidized"[5] in vertical feeders. In the case of the nitrate salt waste stream, a significant number fraction of the particles (0.15) is less than 0.3 microns. This fraction will be difficult to feed with a single screw extruder but can be fed with a twin screw unit. Twin screw extruders can be equipped with a side feeder that provides a positive displacement pumping action to force the powder into the extruder barrel. This will ensure adequate feeding of even the fine powder expected in the nitrate salts.

4. Versatility

With final waste treatment decisions pending for both the waste stream feedstock and the physical and institutional constraints of installation and operation, versatility of the extruder is a necessary attribute. A versatile system will allow rapid process changes in response to change in the waste stream or physical environment of the unit. A rigid process would prevent such response, causing undue operational difficulties and requiring additional resources to accomplish the same waste treatment.

The modular screw design of twin screw extruders greatly increases versatility, allowing exchange of various sections as dictated by process requirements. For example, downstream feed ports can be added at the optimum location for compounding, devolatilization ports can be placed for maximum water removal, and mixing sections can be changed as required to maintain product quality. Single screw units cannot be altered without considerable time delays and extensive rework at the factory.

5. Screw Speed.

High screw speed is desirable because it increases shear stress and with it, dispersive mixing. Under the proper operating conditions, additional pressure generation and product output can also be obtained by increasing the screw speed. This can be used as an operating parameter to adjust for variations in the waste.

Counter-rotating twin screw extruders are somewhat limited in their screw speed. Co-rotating twin screw extruders have a higher screw speed. Single screw extruders typically operate at relatively low screw speeds when compared to twin screw extruders.

6. Multiple Feed Ports

The most obvious feed port for both the salt and the LDPE is the throat of the extruder. Several problems are associated with feeding the nitrate salts through this feed port. First, as mentioned above, the physical form of the salt may not feed well due to the inefficiency of the gravity feed requirements of this location. Second, once fed into the extruder, the salt can interfere with the melting of the LDPE. The LDPE is melted through frictional forces applied by the screw. The salt, being spherical in shape, can act as ball bearings, reducing friction and inhibiting the melting of the LDPE. Third, the salt can be very abrasive and increase barrel wear. The most desirable feed port for the RFP application would be a downstream side feed port. This type of feed port uses a screw mechanism to pump the salt into the barrel, thus avoiding problems of gravity feeding. Because the feed port is downstream, the LDPE can be melted by frictional forces before the salt is introduced. Melting the LDPE before the salt is introduced allows the LDPE to coat the barrel and reduce barrel wear. Finally, the salt can be introduced at the position down the barrel where the viscosity is optimum for mixing the salt into the LDPE.

Twin screw extruders (co- or counter-rotating) have a modular barrel design that allows feeding the salt through a downstream side port. Single screw extruders do not have this modular design and generally require feeding at the barrel throat.

7. Operating Stability and Product Consistency

Operating stability is of crucial importance where mixed waste is being treated. An instability during operation could lead to a shutdown of the unit and a hazardous release, or an uncertifiable product. This could result in unnecessarily increased exposure of operators and maintenance personal to both radioactive and hazardous material. An example of such instability is the surging in extrusion rate seen when certain minor constituents are included in materials starve-fed to single screw extruders. Closely related to operational stability is product consistency. Instability in operation can severely affect product quality. Quality requirements for waste acceptance are stringent. A product that consistently meets or surpasses all criteria is the minimum acceptable. This requires a process that is robust with regard to changes in the feedstock and operating parameters so that a consistent final waste form can be produced.

Consistency and stability of operation are well established characteristics of twin screw extruders. Twin screw extruders are designed to mix and convey the polymer melt with consistency. In this regard, the counter-rotating twin screw is considered superior because its positive conveyance of the melt also eliminates variations caused by changes in the viscosity of the melt. When drag flow is the predominant mechanism for conveying the polymer melt, as in single screw extruders, the extent of screw fill can vary. This results in variations in the extruder output rate, or surging. Only counter-rotating intermeshing twin screw design provides positive displacement of the melt and prevents surging. For overall long term product consistency the counter-rotating twin screw is considered the optimum choice, followed closely by co-rotating twin screw extruders and distantly by single screw extruders.

8. Pressure-Generating Capability

High pressure generation capability allows the extruder to stand alone without an auxiliary melt-pump. This increases the overall simplicity of the process, thereby reducing the system footprint requirements, installation time and cost, maintenance, and down-time after installation. Both twin screw and single screw extruders are expected to generate sufficient pressure for operation.

9. Fully Self-Cleaning Action

Fully self cleaning action (or fully self-wiping action) occurs when the leading edge of one screw's flights fully wipe the trailing edge of the other screw's flights. Each screw then fully cleans the other screw. The self-cleaning action prevents buildup of the polymer on the screws and prevents retention of the product in the extruder. This is desirable because retention of the product in the barrel can result in contamination of later runs. In addition, self wiping is desirable because it improves mixing of the material contacting the screw with the material in the screw channel.

As can be deduced from the above discussion, two screws are required for self wiping to be possible. Therefore, this feature is available only on twin screw fully intermeshing extruders.

10. Devolatilization and Degassing

The nitrate salt waste stream contains about 70%-85% air. In addition, the salt may contain small amounts of water that was not removed in the drying process or was reabsorbed during storage. Devolatilization and degassing are similar. The major difference is that in devolatilization, the heat of vaporization must be supplied by the equipment to bring the liquid into the gaseous state. This requires good heat transfer and mixing characteristics. In degassing, the substance is already in a gaseous state. During the compounding process, good mixing will separate the gas/vapors from the salts. The salt will be encapsulated and the air and volatilized water must be vented from the extruder barrel to prevent bubble formation in the product.

The excellent mixing and heat transfer capabilities of twin screw extruders provide good devolatilization characteristics. Additionally, the modular design provides the ability to place vent ports down the barrel as needed. Devolatilization from single screw extruders is less efficient than from twin screws due to poorer mixing and heat transfer and less flexibility in placement of vent ports. However, single screw extruders may have adequate devolatilization for the expected water content in the nitrate salts.

11. Distributive Mixing

Distributive mixing occurs when one phase (or liquid) is distributed throughout another phase, the first phase being immiscible in the second. This kind of mixing typically occurs in the following sequence: 1) separation of a stream into two or more parts, 2) reorientation of the parts with respect to each other, and 3) recombination of the parts. Several repetitions of this sequence are required to provide sufficient distributive mixing. In the case of compounding a powder into a polymer, the first phase is the powder (which displays the characteristics of a fluid), and the second phase is the melted LDPE. Distributive mixing ensures intimate contact between these two phases so that the subsequent dispersive mixing (discussed above) can more readily occur. It should be pointed out that even though distributive and dispersive mixing are discussed separately they always occur together. Distributive mixing does not occur without dispersive mixing and dispersive mixing does not occur without distributive mixing.

Twin screw extruders are considered excellent distributive mixing tools if mixing elements are present and fair without the elements. With two screws, more opportunity is available for the sequence of separating, reorienting, and recombining streams. Even without mixing elements the twin screw mechanism of shifting the melt from channels in one screw to those in the other provides fair mixing. Without mixing elements single screw extruders have no mechanism for mixing and are inadequate for the task. Adding mixing elements to the single screw somewhat improves distributive mixing but this capability is limited.

12. Industry Usage

Industry has been compounding fine powders into polymers for many years. The selection of an extruder for a similar application should reflect the cumulative experience of industry. Industry uses co-rotating twin screw extruder for similar processes about 80% of the time with most of the remainder using counter-rotating twin screw extruders. Single screw extruders are seldom used when fine powders are to be compounded into polymers.

13. Cost

While the cost of an extruder for a given throughput is of consideration, the actual cost of the unit is a minor portion of the overall implementation cost, and of secondary consideration to the suitability of the unit for the task. The cost of selecting a unit that produces an unacceptable product would dwarf the cost of even the most expensive unit.

Single screw extruders are, on average, about one third the cost of twin screw extruders with the same throughput capacity.

EVALUATION

Twin screw co-rotating and counter-rotating intermeshing and single screw extruders were rated against the evaluation criteria. This evaluation is summarized in Table 1.

TABLE 1: SELECTION CRITERIA FOR COMPOUNDING OPERATIONS
(Extruder)

KEY: +, Good; 0, Adequate; —, Poor

EXTRUDER FEATURES	TWIN SCREW - COUNTER - INTER MESHING	TWIN SCREW -CO -INTER MESHING	SINGLE SCREW
Dispersive Mixing This is the most important feature for nitrate salt encapsulation. Dispersive mixing breaks up the agglomeration of particles and ensures coating of each particle by the polymer.	0 without special mixing element + with	0 without special mixing element + with	— without special mixing element 0 with
High Filler Content High waste loading is crucial to the economics of this process. Therefore, the ability of the unit to process mixes with a high filler content is very desirable.	0	+	—
Powder Feeding Capability With the product of both the current dryer and all anticipated replacement dryers being a fine powder, the ability to feed fine powders is essential.	+	+	0
Versatility A more versatile system would allow rapid change to the process in response to changes in the waste stream and for processing of other waste streams under consideration. The modular format greatly increases versatility. In combination with controlled pumping and wiping characteristics, it facilitates matching and retrofitting screws/barrels to specific process tasks. In addition, modular design reduces the space required for maintenance of the extruder, and would be the format of choice for glovebox applications.	+	+	—
Screw Speed High screw speed is desirable because it increases shear stress and thereby dispersive mixing.	—	+	+
Multiple Feed Ports The most desirable feed port for our application would be a downstream side feed port. This allows melting of the polymer before introduction of the salt. Better polymer melting, improved compounding of the waste salt, and reduced barrel wear are advantages of this feature.	+	+	—

Operating Stability Consistency and stability of operation is crucial to producing a quality product. When not using pressure as the mechanism for conveying the polymer melt, the extent of screw fill can vary, resulting in variations in the extruder output rate, or surging. Only a counter-rotating intermeshing twin screw design provides positive displacement of the melt and prevents surging.	+	0	0
Pressure Generating Capability (positive displacement) High pressure generation allows the extruder to operate without an auxiliary pump, and often enhances feeding and processing capabilities.	+	0	0
Self Cleaning Action Self cleaning is desirable because it minimizes hold-up of material in the barrel. Also, mixing of the material contacting the screw with the material in the screw channel is improved.	+	+	—
Devolatilization and Degassing Devolatilization and degassing ports are essential for the removal of remaining water and trapped gases.	+	+	0
Distributive Mixing Distributive mixing provides an even distribution of all components throughout the polymer mix, and ensures uniformity of the final product. Uniformity is desirable in order to maximize the probability of meeting waste acceptance criteria.	0 without special mixing element + with	0 without special mixing element + with	— without special mixing element 0 with
Industry Usage Industry has been compounding fine powders into polymers for many years. The selection of an extruder for processing waste of a similar physical form (fine powders) should reflect the cumulative experience of industry. Industry experts recommend and almost exclusively use twin-screw technology.	0	+	—
Cost While the cost of an extruder for a given throughput is of consideration, the actual cost of the unit is a minor portion of the overall implementation cost, and of secondary consideration to the suitability of the unit for the task.	—	0	+
TOTALS	7+; 2—	10+; 0—	2+; 5—

SUMMARY

Counter-rotating intermeshing twin screw extruders have good dispersive mixing characteristics, generally feed polymers and fillers well, have a self-cleaning action, and a good pressure generating (positive displacement) capability. They are capable of successfully extruding a variety of materials with minimum down-time for setup. They are capable of producing a very stable melt flow. Screw speeds are lower than with other extruder types, resulting in somewhat lower throughput rates, but this may be offset by the fact that better pressure generating capability can enhance feeding and processing efficiency.

Co-rotating intermeshing twin screw extruders have most of the advantages listed above for counter-rotating extruders. Additionally, they are capable of accepting a higher filler content, can generate a higher screw speed, and are more extensively used in the polymer processing industry than counter-rotating intermeshing twin screws.

Single screw extruders in industry are used primarily for "profile" extruding. That is, they are used for extruding shapes such as film, sheet, rod, etc. They are only marginal for the compounding applications expected at RFP.

Based on the above comparison of extruder characteristics, the co-rotating twin screw unit will most closely match RFP needs. Nearly as good and also quite acceptable is the counter-rotating twin screw extruder. The single screw extruder is expected to be significantly inferior to either the co- or counter-rotating twin screw and its ability to adequately perform the anticipated task is highly questionable.

DRYING EVALUATION

The three (3) drying equipment options considered for evaluation include a vertical thin film evaporator (VTFE), a horizontal thin film evaporator (HTFE), and a horizontal rotary blender / dryer. Pilot-scale evaluation tests using surrogate nitrate salt waste were conducted at vendor facilities.

VERTICAL THIN FILM EVAPORATOR (VTFE)

The VTFE consists of a tubular, heated jacket surrounding a finned rotor which is custom designed for the material to be dried. Typical applications for the VTFE include suspensions or slurries of nitrates, chlorides, sulfates, etc., as well as organic compounds.

The material to be treated is introduced to the VTFE through the top of the unit, above the heated zones. The spinning rotor spreads the material over the heated interior wall in a thin film. The material is dried to a solid, free flowing powder as it moves down the heated wall. Vapors leave the dryer at the top, where they can be collected and removed from the system.

Advantages of VTFE's include:

- Single-step operation - Dilute feed materials dry to free-flowing solids in a single pass.
- Reduced energy requirements - Less than 1.15 pounds of steam are required per pound of water removed.
- Contact drying - Thermal degradation of heat sensitive or hazardous products is minimized by low residence time.
- Fouling of the thermal surface is minimized or even eliminated by the scraping action of the rotor blades.

Pilot-scale evaluation tests were performed during August 1993 at the LCI Corporation Process Division Test Center in Charlotte, North Carolina. LCI's model CP-0050 with a heat transfer surface of 5.4 ft^2 was used in the test.

The vertical unit was operated for 2 hours at a 35 wt % solids feed rate of approximately 40 lbs/hr. Minimal fouling was observed on the rotor. The product produced was a free-flowing powder with a maximum of 0.5% moisture. The apparent bulk density was 0.93 - 0.94 gm/cc as determined using ASTM method 218.

The feed rate was increased to 50 lbs/hr and signs of fouling in the unit appeared shortly afterward. The build-up of material on the mid-portion of the rotor may have been caused by the lack of transport elements in this section of the rotor or by incorrect spacing tolerance between the wiper blades and the outer shell.

Photographs 1 through 4 depict the test VTFE and its components. A sample of the dried product is shown in Photograph 5. Test observations are included in Appendix 2.

HORIZONTAL THIN FILM EVAPORATOR (HTFE)

The HTFE, like the VTFE, consists of a tubular, heated jacket surrounding a spinning rotor. Material is forced into the heated zone by an augur feed blade at the feed end of the rotor. Conveying and distributing blades are also incorporated in the rotor design to facilitate drying and movement of the material along the length of the dryer. The unit produces a solid, free flowing powder from the slurry as it moves along the heated interior wall.

Typical applications for the HTFE include slurries, filter cakes, and wet powders of nitrates, chlorides, sulfates, etc., as well as organic compounds.

Rotor design may be customized to optimize dryer performance depending on the application and feed materials.

Advantages of HTFE's include:

- May be operated under vacuum or as an atmospheric contact dryer.
- Minimal airborne dust as no sweep gas is required.
- Rotor design prevents the product from sticking to the heated wall.
- Reduced energy requirements - Less than 1.25 pounds of steam is required per pound of water removed.
- Single-pass, "plug-flow" drying.

Pilot-scale evaluation tests of the HTFE were performed during August 1993 at the LCI Corporation Process Division Test Center in charlotte, North Carolina. LCI's model D-0100 with a heat transfer area of approximately 10 ft^2 was used in the test.

The horizontal unit was operated for 1.5 hours at a 35 wt % solids feed rate of 104 lb/hr. Minimal fouling was observed on the rotor, indicating that higher feed rates are feasible. This test run produced a free-flowing powder with a maximum 0.5% moisture content. The apparent bulk density was similar to the product from the vertical dryer, but the average particle size and distribution were significantly larger.

Photographs 6 through 9 depict the test HTFE and its components. A sample of the dried surrogate nitrate salt produced in the test HTFE is shown in Photograph 10. Test observations are included in Appendix 2.

HORIZONTAL ROTARY BLENDER/DRYER

The Pacific Nuclear Services (PNS) RVR-800 is a horizontal rotary blender/dryer designed specifically to evaporate liquids from nuclear power plant waste solutions. The unit is typically used to produce a free-flowing powder of approximately 10% moisture with no visible free liquids. Frequently, a paraffin wax is added to the dryer once a powder is formed. The heat of the dryer melts the paraffin wax and paddles in the dryer mix the molten paraffin with the waste powder. The mix is discharged into a 55 gallon drum, where it solidifies upon cooling.

Brookhaven National Laboratory (BNL) coordinated a demonstration at the PNS test facilities in Columbia, South Carolina in May 1993. Dry Rocky Flats surrogate nitrate salt waste (500 pounds) was mixed with 930 pounds of water in the blender/dryer to produce a 35% solids solution. After five hours of operation, a thick paste formed. After seven hours of operation, particles ranging from 1mm to 1/2 inch in size with a moisture content of 2-to-3% formed. From this point, agglomeration proceeded rapidly, and abrasion of hard balls of product against the walls of the unit created a significant amount of noise. Approximately eight hours after the test began, the PNS operator stopped the test because of concerns that abrasion of the product against the walls of the dryer could be damaging the unit.

The final product consisted of a fine powder and hard pellets ranging from approximately 2 mm to marble-sized. A few very large chunks of material were also observed. Moisture analysis on the pellets indicated a moisture content of 0.96%. When the pellets were crushed, the moisture released increased to 1.82%. The dried surrogate nitrate salt produced in the RVR-800 horizontal rotary blender / dryer is shown in Photographs 11 through 13. Test observations are included in Appendix 3.

Use of the PNS RVR-800 rotary blender/dryer unit is not recommended for pretreatment of the RFP nitrate salt waste prior to polyethylene extrusion. First, the low throughput rates of the unit make it impractical for the application. Multiple units would have to be installed in an existing facility with limited available floor space in order to meet the throughput requirements of the RFP liquid waste treatment facilities. Second, the large particle size distribution of the final product is not compatible with the polyethylene extrusion process. An additional unit process, such as a grinder, would be required to produce a material that could be effectively processed in an extruder. Finally, the long-term reliability of the RVR-800 dryer for this particular application is questionable. The RVR-800 dryer was designed to remove visible liquids and produce a free flowing powder, not to produce a powder with less than 0.5% moisture. Based on the noise produced by the unit at the lower moisture ranges, it is apparent that frequent operation of the unit under these conditions could result in early wear and possible failure of the unit.

The RVR-800 blender/dryer should be considered, however, for concentrating other smaller volume mixed waste streams at RFP, especially prior to cementation or microwave solidification. The unit is probably not a viable pretreatment to polymer encapsulation.

OTHER PROCESSES

Processing schemes other than drying and extrusion were also evaluated for polymer encapsulation of the nitrate waste stream. These included "meltration" and compounders other than heated extruders.

MELTRATION

The "meltration" process involves the simultaneous devolatilization and encapsulation of waste brine and thermoplastic material in a vertical thin film evaporator. If successful, the need for microencapsulation of waste using an extruder would be eliminated.

A preliminary screening evaluation was conducted during May and June 1993 at the LCI Corporation Process Division Test Center in Charlotte, North Carolina. The results from these tests indicate that meltration is not possible at atmospheric pressure using the LDPE provided by RFP (PE-1409) due to its relatively high melting point. However, meltration should be possible at atmospheric pressure using a LDPE with a lower molecular weight and viscosity. LCI recommended and performed additional tests with Epoline C-15. Photographs 17 through 19 depict a preliminary evaluation sample of surrogate nitrate salt encapsulated in Epoline C-15. The screening evaluation report form LCI is included in Appendix 4.

The basic composition and structure of the two resin polymers are similar (i.e. low density polyethylene) except for differences in molecular weight. Decreasing the molecular weight of polymers with similar structure and composition results in a decrease in melt viscosity. The large difference in melt viscosity and molecular weights of the C-15 and PE-1409 resins is reflected by the large difference in the Melt Indices (MI). The MI of the PE-1409 is 50 gms/10 minutes while the MI of the C-15 is 4,200 gms/10 minutes (a factor of 84). Although the lower molecular weight/melt viscosity may allow processibility through the TFE, there will be decreases in the quality and properties of the final solidified waste form. The overall changes to the properties of the polyethylene are not expected to result in a solidified waste form which is unacceptable; however, the degree of these changes in quality and properties should be determined and compared. The following properties should be evaluated for the final solidified waste form using the C-15 resin:

1. Mechanical Properties

As the molecular weight of the polymer is lowered, the mechanical properties of the resin decrease. The decreased properties include stiffness, tear strength, hardness, tensile strength, compression strength, low temperature toughness, and resistance to environmental stress cracking. Also, the lower molecular weight could result in increased crystallinity (although this is highly dependent upon the branching on the molecule), which increases the brittleness and decreases the impact/toughness (especially at lower temperatures) of the polyethylene resin.

- The compression strength and the low temperature impact resistance between the waste forms solidified with PE-1409 and C-15 should be determined and compared.

2. Physical Properties

Decreases in the molecular weight decreases the glass transition temperature, softening point, and the melt viscosity of the resin. The lower molecular weight could affect the thermal stability of the resin (especially if very low molecular weight contaminants are contained in the resin), although this is not expected. However, the addition of certain salts and excess thermal exposure during processing can affect the thermal stability of the resin.

- Thermal transition, softening points, the melt viscosity, and the relative thermal stability of resin/salt mixtures should be determined.

- The compression strength of the two mixtures should be tested at an elevated temperature (60 C).

3. Combustibility

The nitrate salts in the brine are considered to be oxidizers which upon mixing with an organic polymer could result in a combustible mixture. Previous studies have determined that nitrate salts and PE-1409 do not form a combustible mixture [5]. Normally, the combustibility of such a mixture should not be affected by molecular weight of the polymer until very low molecular species are formed during thermal decomposition. If the thermal stability of the polymer is similar to the decomposition temperature of the nitrate salts (380 - 400 C), an easily ignitable gas mixture of the low molecular weight polymer decomposition products and the oxides of nitrogen could be formed.

- Tests should be conducted to determine if the addition of the nitrate salt (especially if dispersed in fine particle) could affect the autoignition temperature, combustibility, and the burning rate of the resin.

- Screening tests should be conducted with the differential scanning calorimetry (DSC) on the salt/polymer mixture for exothermic reactions (indicating oxidation) prior to testing with the TFE equipment.

4. Stability of Solidified Waste Form

The change in molecular weight of the resin is not expected to affect the stability. The rotating blades of the TFE create high shear rates/forces which could create hot spots and cause decomposition (oxidation, etc.) of the resin. If the resin undergoes thermal decomposition, this could affect the overall stability and life of the resin during storage of the solidified waste.

- Screening analysis by Fourier Transform Infrared (FTIR) spectrometry should be conducted to determine if the resin has been oxidized or undergone decomposition during the TFE processing. If decomposition is detected, then consideration should be given to accelerated storage/life studies.

5. Radiation Stability

The very low levels of radiation (approximately 200 pCi/gm) in the salt should not significantly affect the life of the solidified waste form [21]. However, one of the mechanisms of radiation decomposition is scission which results in a reduction in the molecular weight of the polyethylene molecule. Because of the lower molecular weight of the C-15 resin, the radiation resistance of the C-15 resin is expected to be lower.

- No testing is required for radiation stability.

6. Leachability

The different processing method and the lower molecular weight of the polymer may affect the structure of the matrix.

- To ensure that the salt particles/pockets are not connected, a leaching/immersion test should be conducted.

7. Excess Water Retention/Dispersion of Salt

The final solidified form should have no water/brine pockets or foaming which can be formed during the evaporation of the water.

- The amount of water retained in the solidified waste should be determined.

- The dispersion of the salt should be evaluated.

8. Microbial Decomposition

Microbial decomposition of the lower molecular weight polyethylene is not expected since the molecular weight is above 500 [5].

- No microbial resistance testing of the solidified waste form is needed.

9. Cost and Availability

The cost and availability of the Eastman resin from other sources is not known at this time. Resin with the same composition and properties should be available from at least two different sources.

- A brief cost analysis between the two resins should be conducted.

- Alternate sources of the C-15 resin should be identified.

GELIMAT MIXER/COMPOUNDER

The Gelimat mixer/compounder is used in the plastics compounding industry to compound low bulk density powders and low cost fillers in more expensive thermoplastic polymers. The Gelimat unit imparts a very high degree of shear mixing force to the material by high speed mechanical agitation.

A courtesy demonstration with the Gelimat mixer / compounder was provided by Draiswerke Inc.. The demonstration was performed during June, 1993, at the Draiswerke Inc. facility in Allendale, New Jersey.

A mixture of 50 weight % technical grade sodium chloride and 50 weight % LDPE was produced. Photographs 14 through 16 depict samples from the courtesy demonstration.

FUTURE WORK

Equipment evaluations are continuing. As evaluations progress and upstream process parameters become defined, follow-on demonstrations and selection of specific equipment will become appropriate. Information for ancillary equipment will also be refined. Development of process design envelope information will begin.

Drying Operations

The Statement of Work (SOW) in place with LCI Corp. is being modified for bulk processing (200 kg range) of surrogate nitrate salts. The thin film evaporator-dried surrogate nitrate salts would then be made available for follow-on pilot scale extrusion operations demonstrations.

Extrusion Operations

Two (2) SOW's are in place with American Leistritz Extruder Corp. to provide for pilot scale demonstrations of several surrogate nitrate salt forms. Initial process design envelope information will be generated.

Other Operations

The Epoline C-15 meltration option will be investigated. While the lower molecular weight (and resultant lower viscosity) may allow greater processibility, the change will also decrease the quality and properties of the final low density polyethylene waste form. The overall decrease is not expected to result in an unacceptable waste form; however, the extent of the decrease will be determined. Safety questions, including potential exothermic reactions between the Epoline C-15 and the nitrates salts will be evaluated prior to pilot scale demonstrations.

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Photographs 1 - 4: VTFE

Photograph 5: VTFE dried salt

Photograph s 6 - 9: HTFE

Photograph 10: HTFE dried salt

Photograph 11 - 13: PNS dried salt

Photograph 14 - 16: Gelimat dried salt

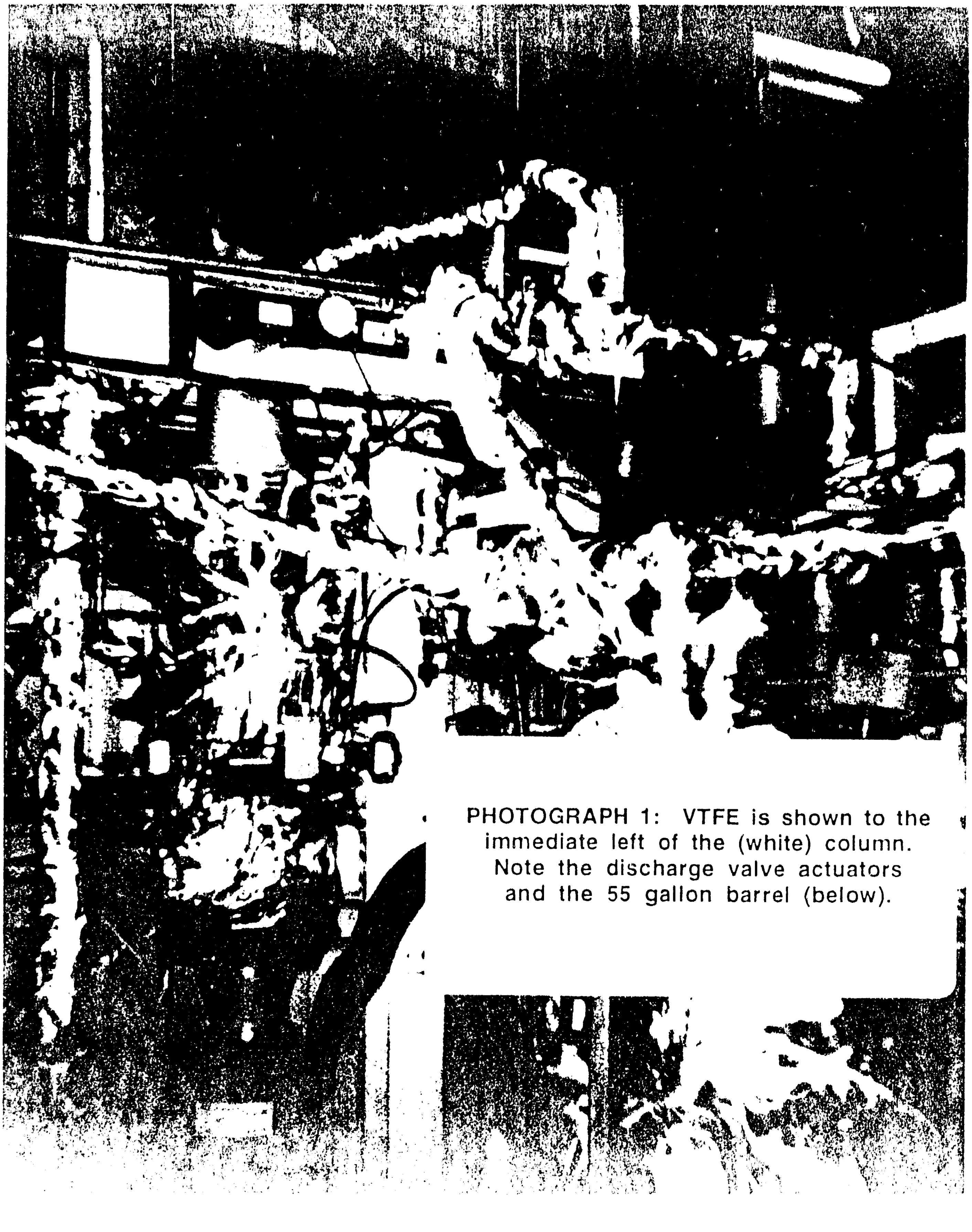
Photograph 17 - 19: Epoline C-15

Appendix 1: Trip Reports

Appendix 2: V & H TFE test observations

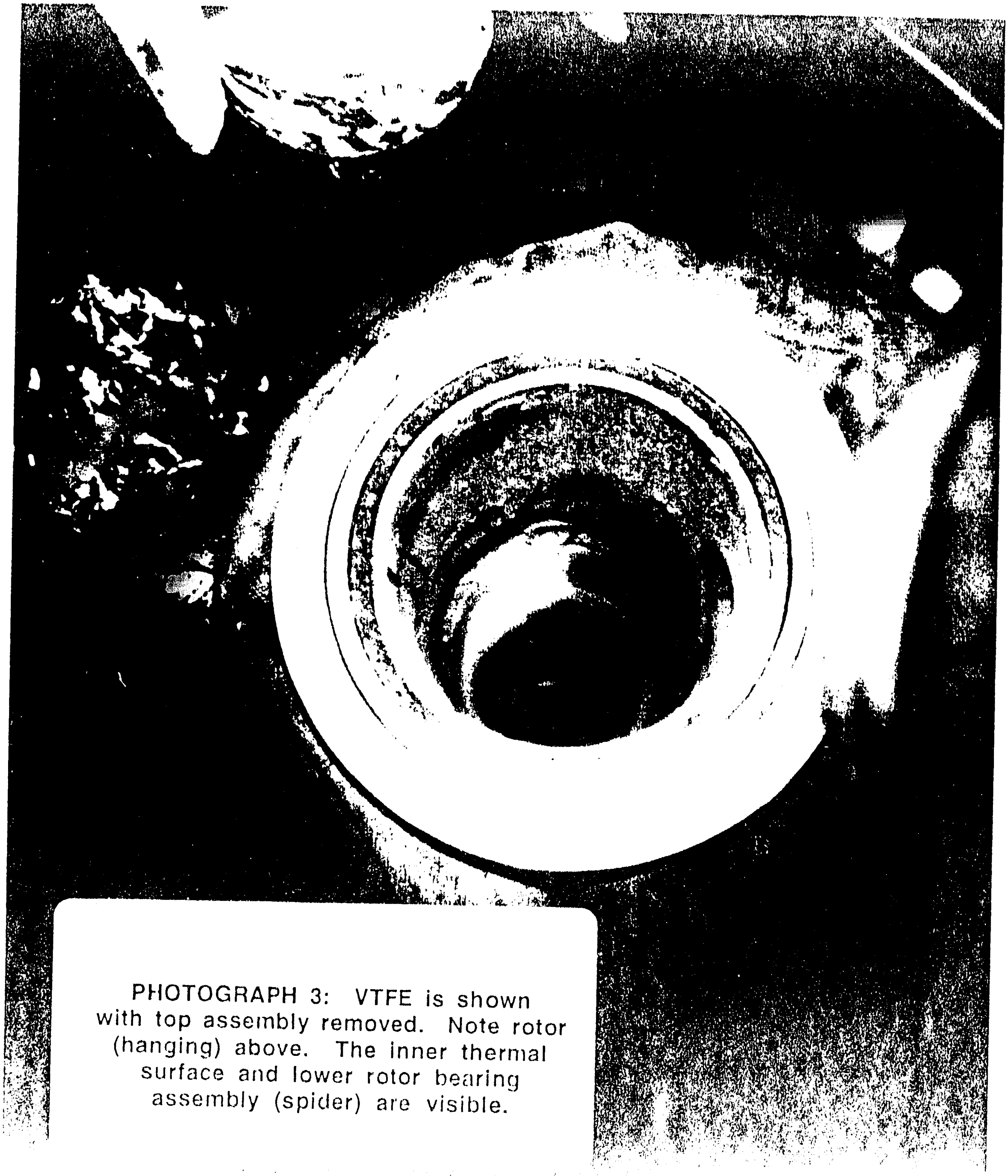
Appendix 3: PNS test observations

Appendix 4: LCI screening evaluation report

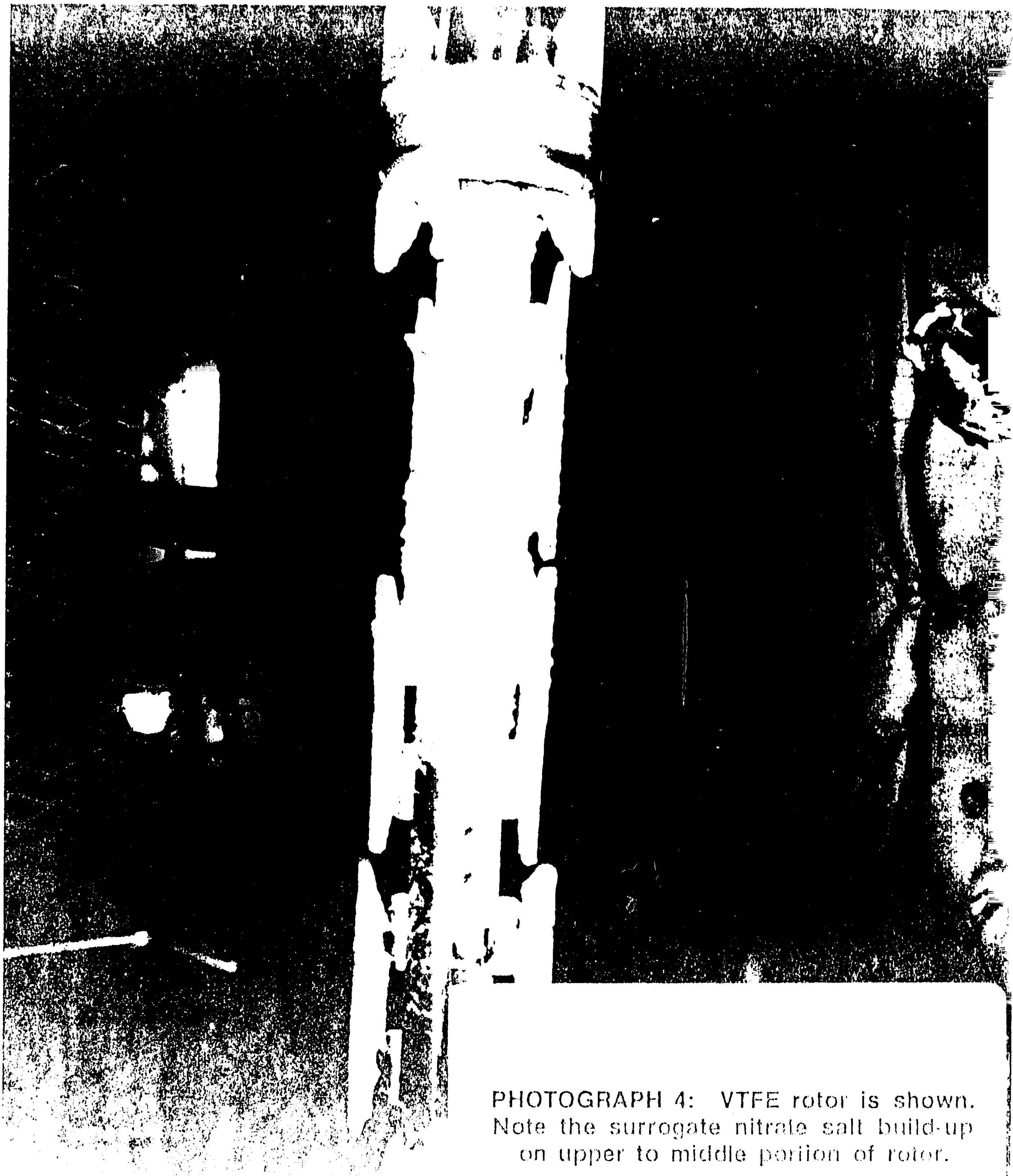


PHOTOGRAPH 1: VTFE is shown to the immediate left of the (white) column. Note the discharge valve actuators and the 55 gallon barrel (below).

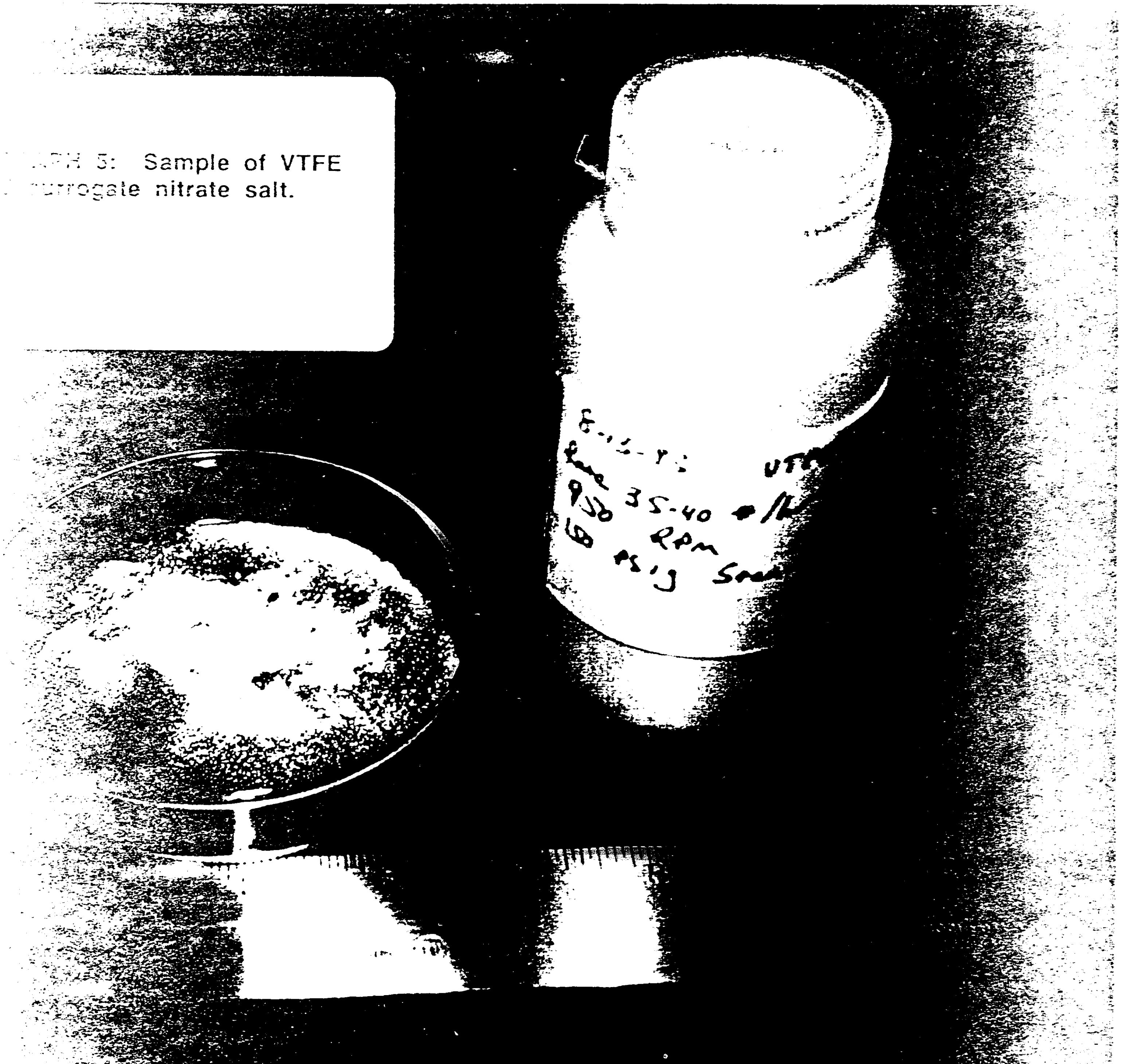
PHOTOGRAPH 2: VTFE equipment layout is shown. Material feed tank is in foreground right; VTFE is behind LCI Corp. technician at left.



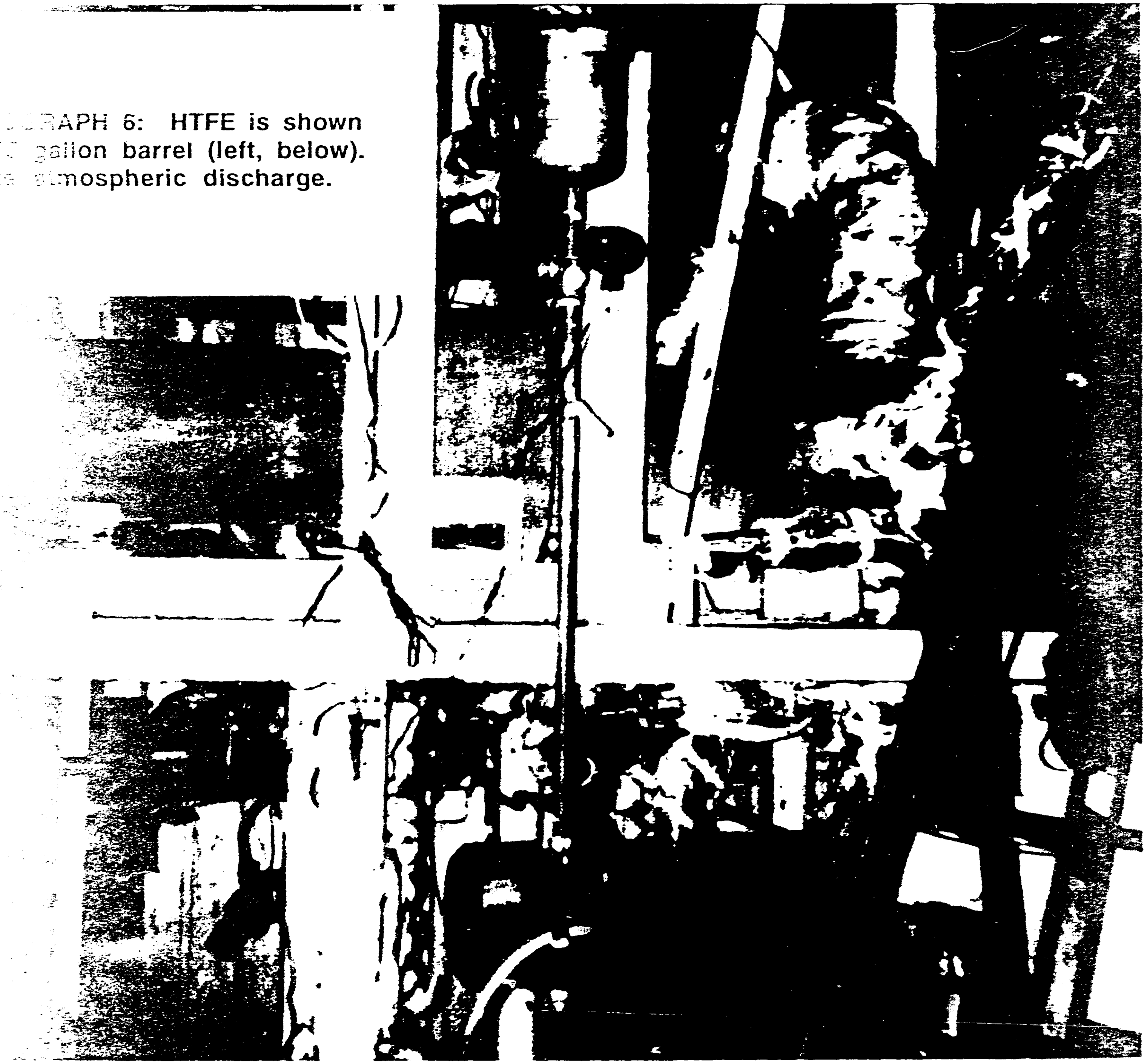
PHOTOGRAPH 3: VTFE is shown with top assembly removed. Note rotor (hanging) above. The inner thermal surface and lower rotor bearing assembly (spider) are visible.



PHOTOGRAPH 4: VTFE rotor is shown. Note the surrogate nitrate salt build-up on upper to middle portion of rotor.

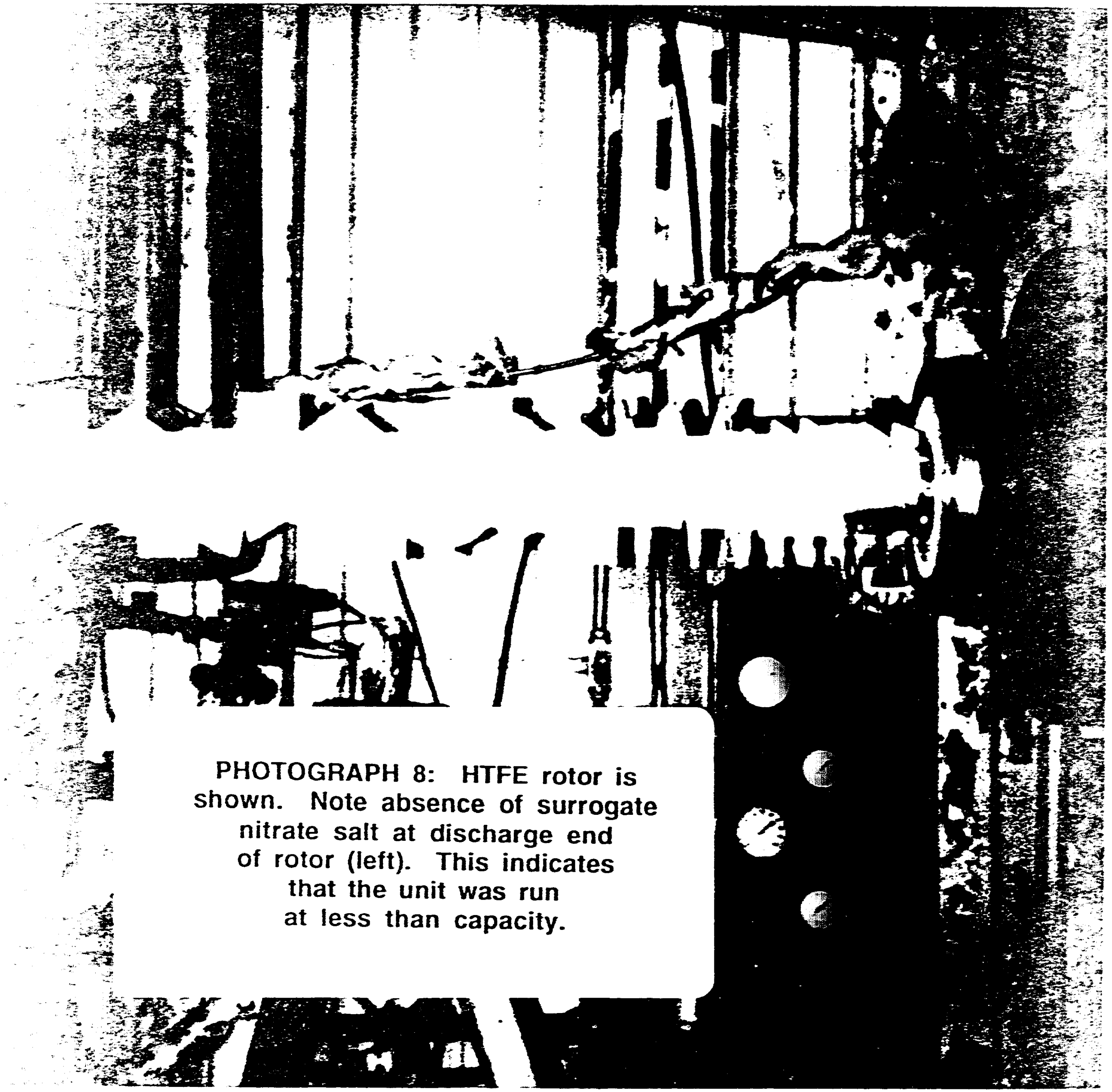


PH 5: Sample of VTFE surrogate nitrate salt.

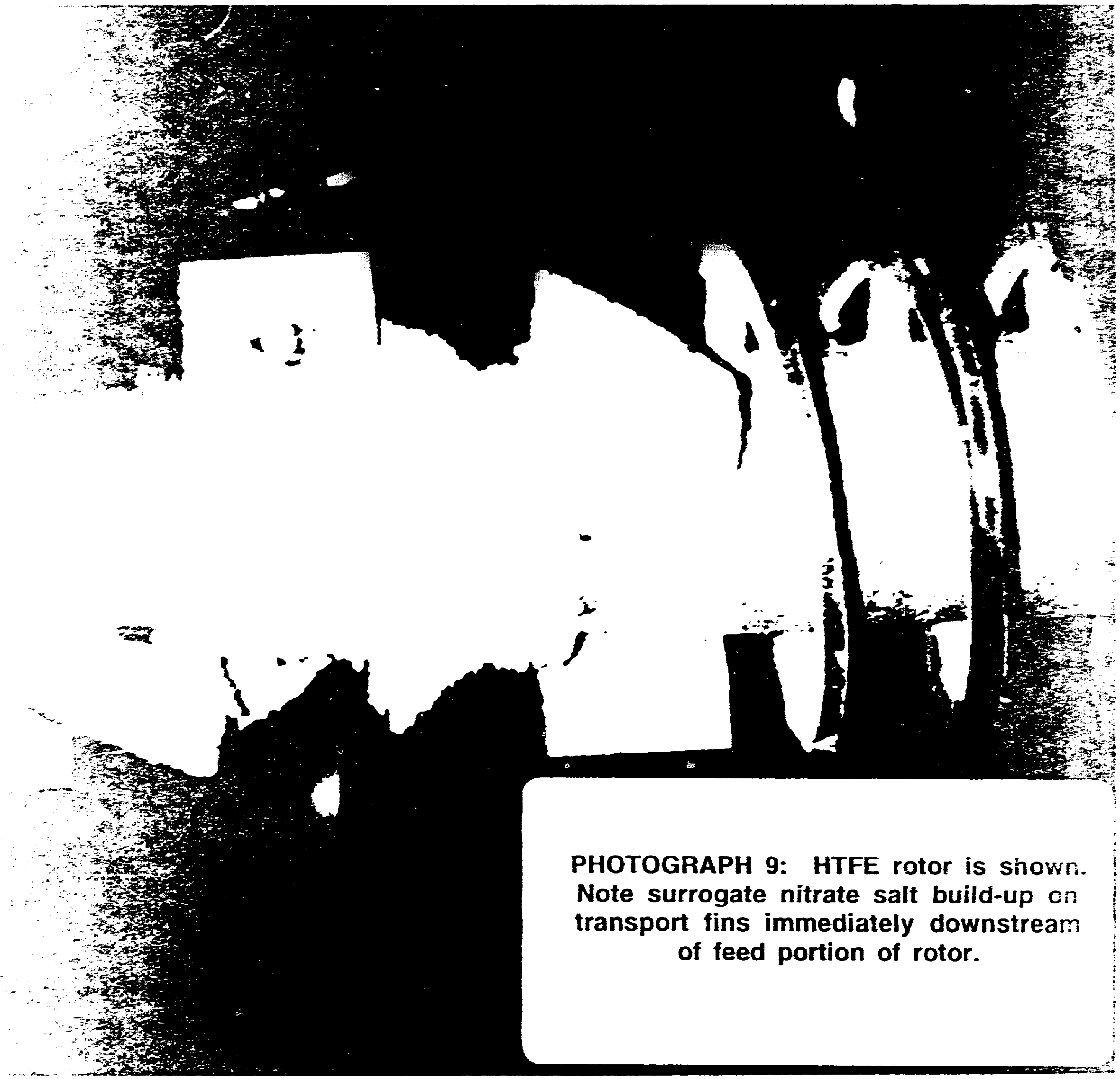


...RAPH 6: HTFE is shown
... gallon barrel (left, below).
... atmospheric discharge.

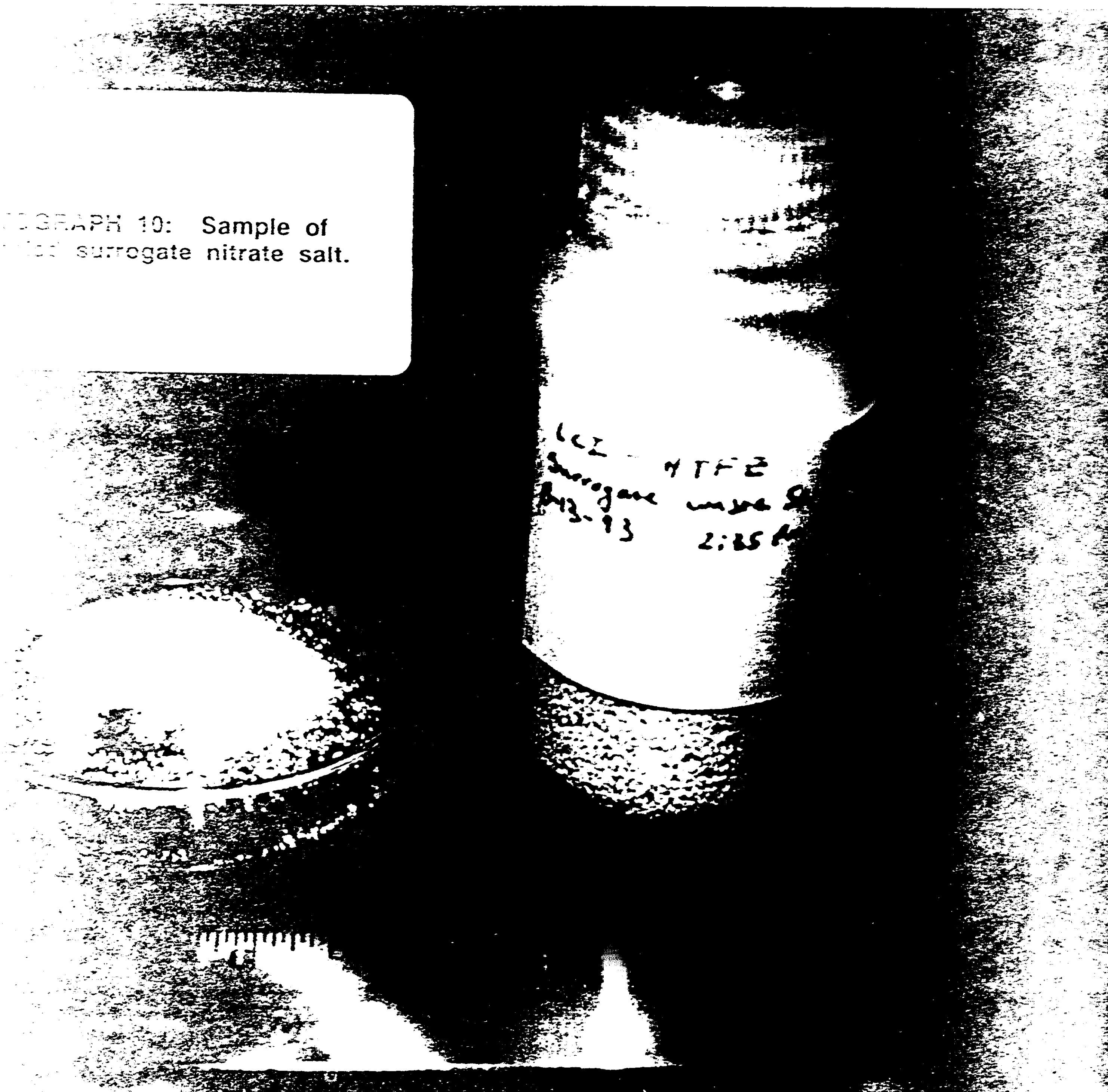
PH 7: HTFE equipment
shown. Drive motor
own below HTFE.
is shown to right.



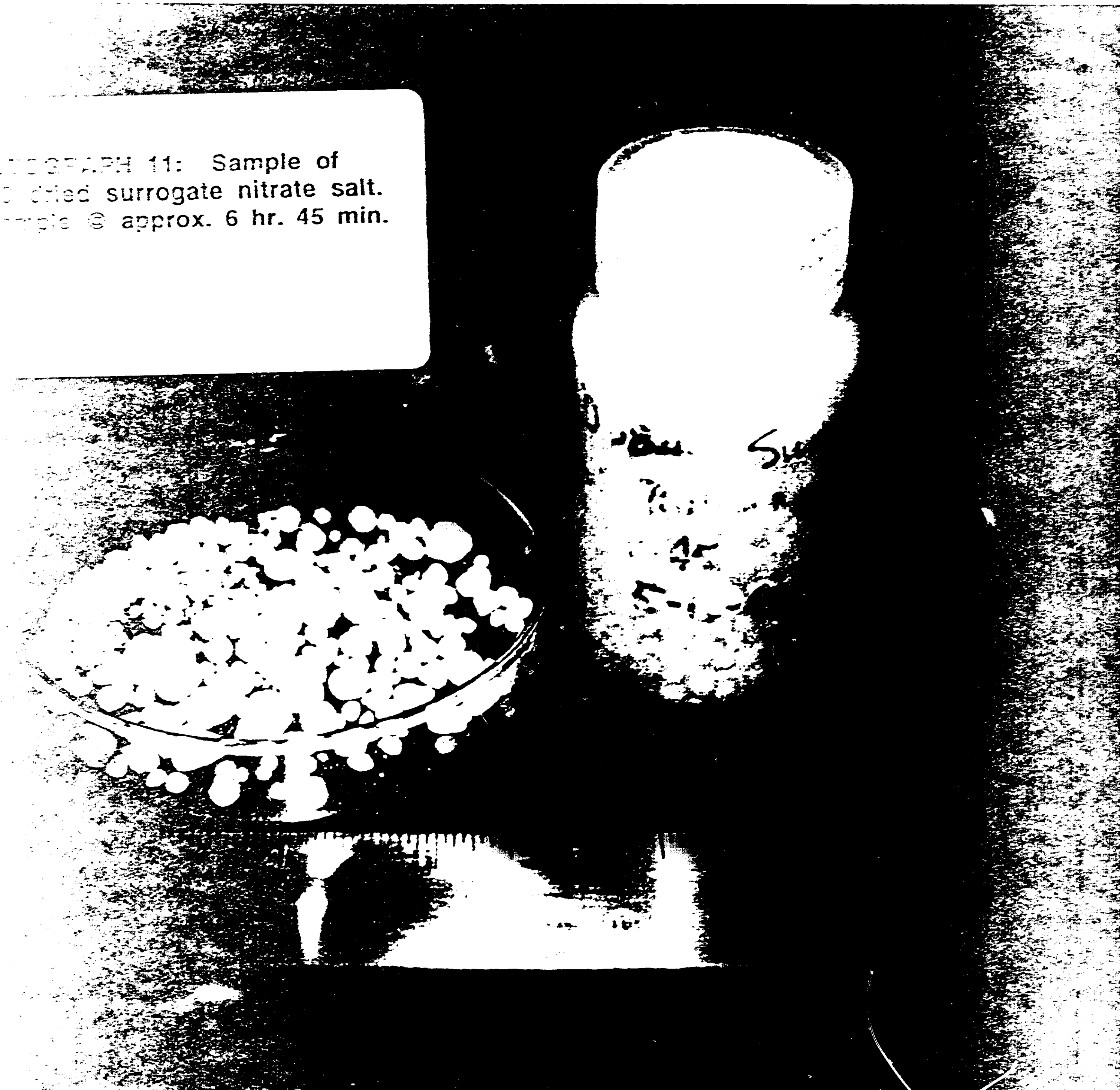
PHOTOGRAPH 8: HTFE rotor is shown. Note absence of surrogate nitrate salt at discharge end of rotor (left). This indicates that the unit was run at less than capacity.



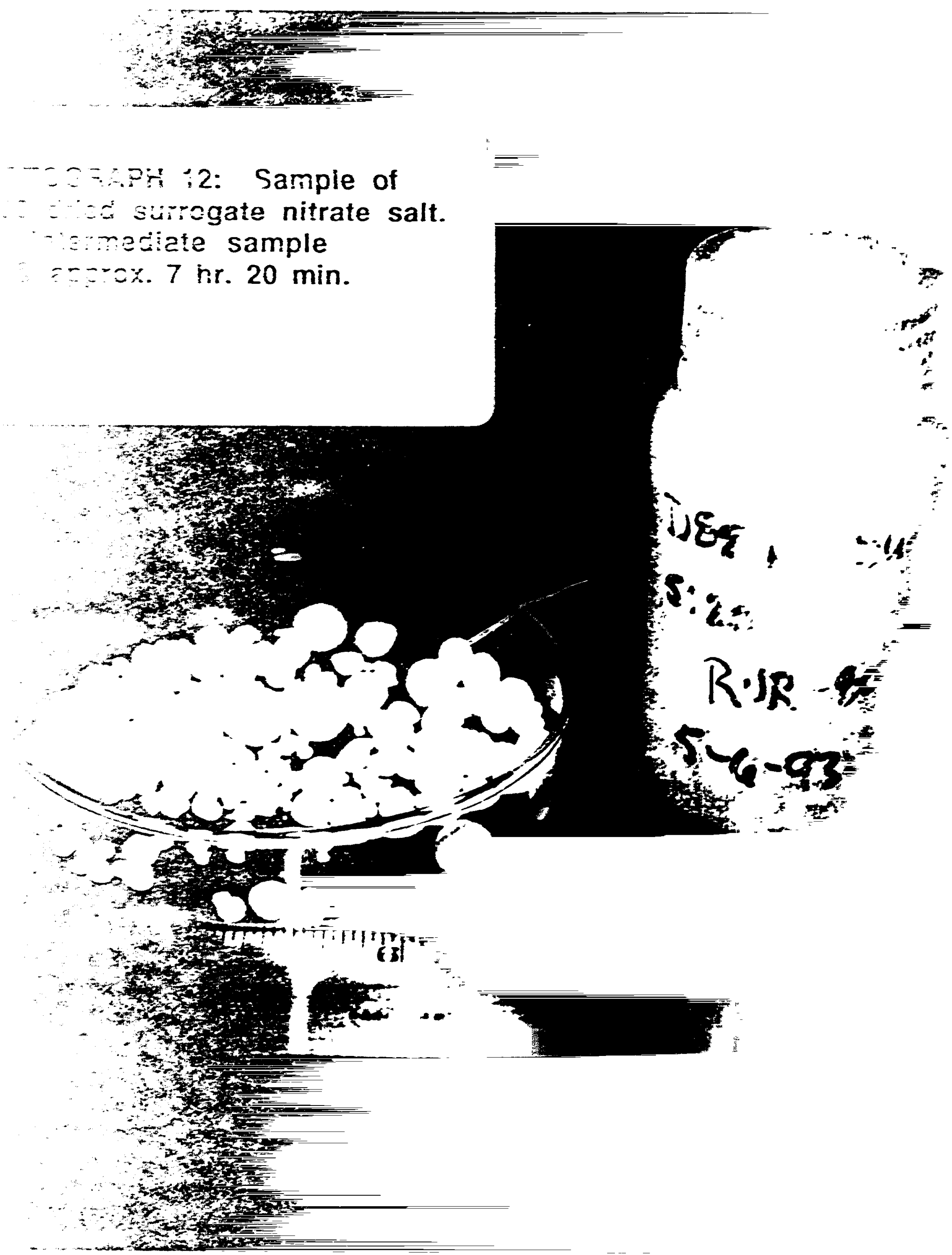
PHOTOGRAPH 9: HTFE rotor is shown. Note surrogate nitrate salt build-up on transport fins immediately downstream of feed portion of rotor.



GRAPH 10: Sample of surrogate nitrate salt.

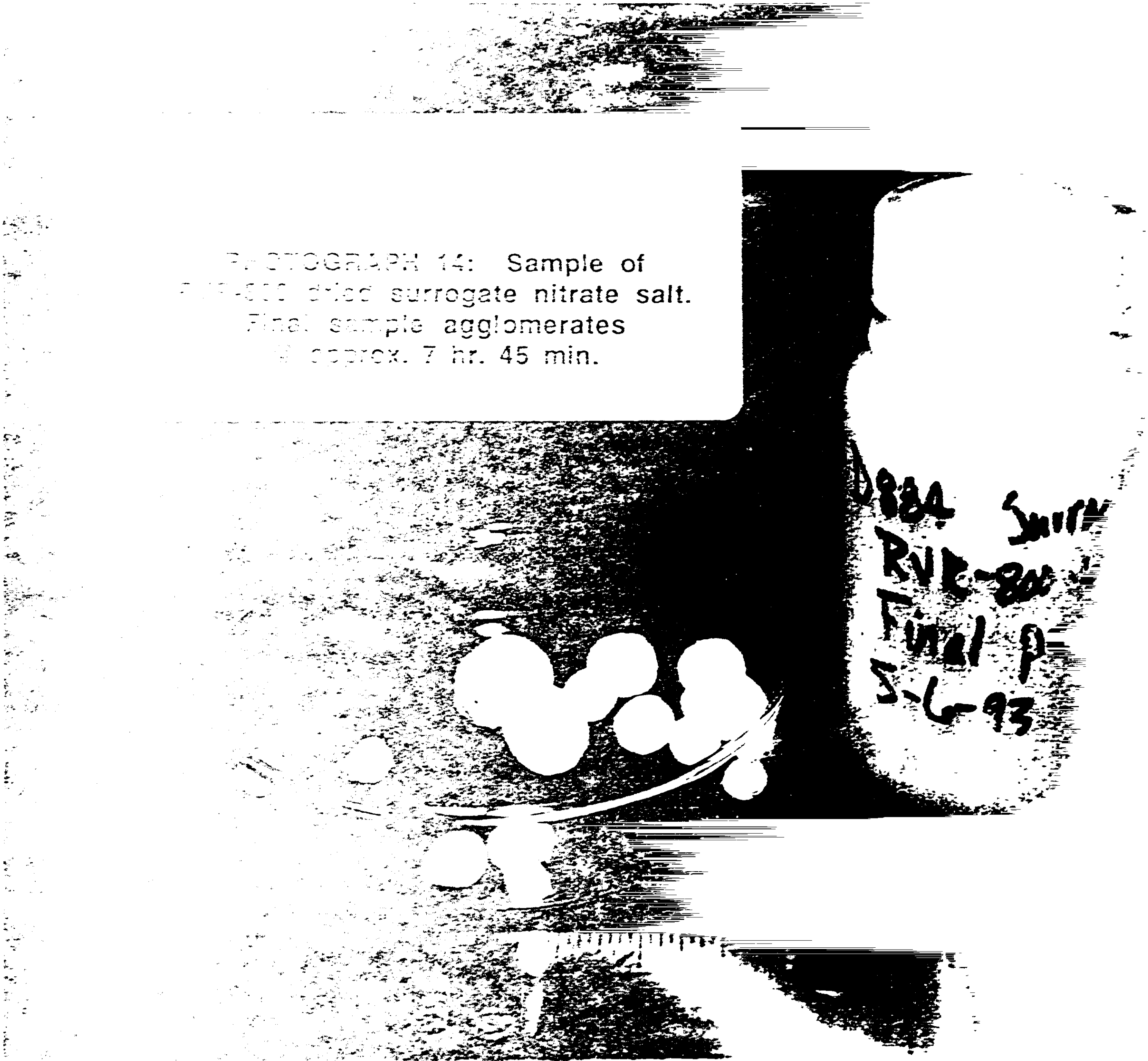


PHOTOGRAPH 11: Sample of
[illegible] dried surrogate nitrate salt.
Sample @ approx. 6 hr. 45 min.

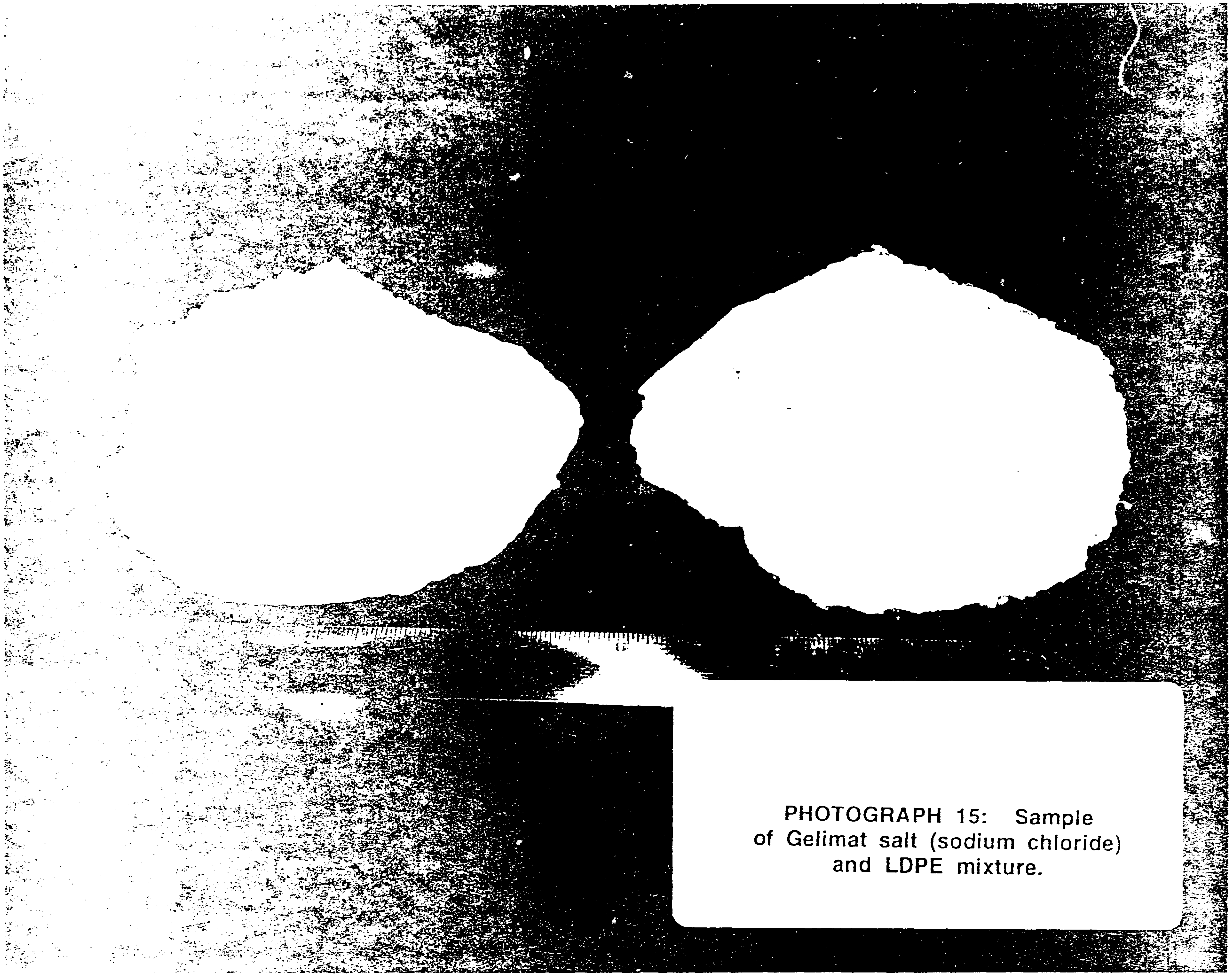


...OGRAPH 12: Sample of
... dried surrogate nitrate salt.
...ntermediate sample
... approx. 7 hr. 20 min.

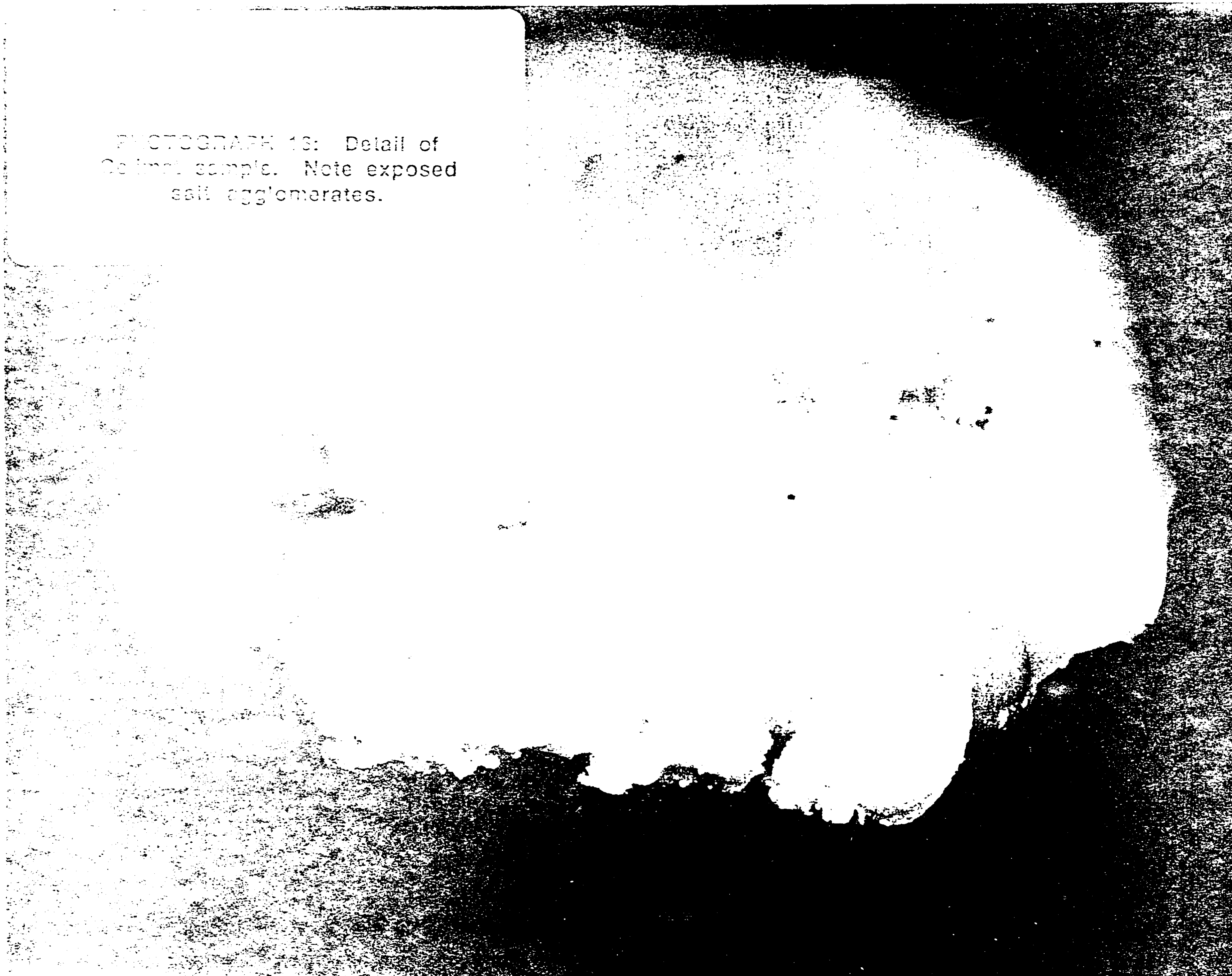
PHOTOGRAPH 13: Sample of [illegible] dried surrogate nitrate salt. Final sample powder (@ approximately 7 hr. 45 min.).



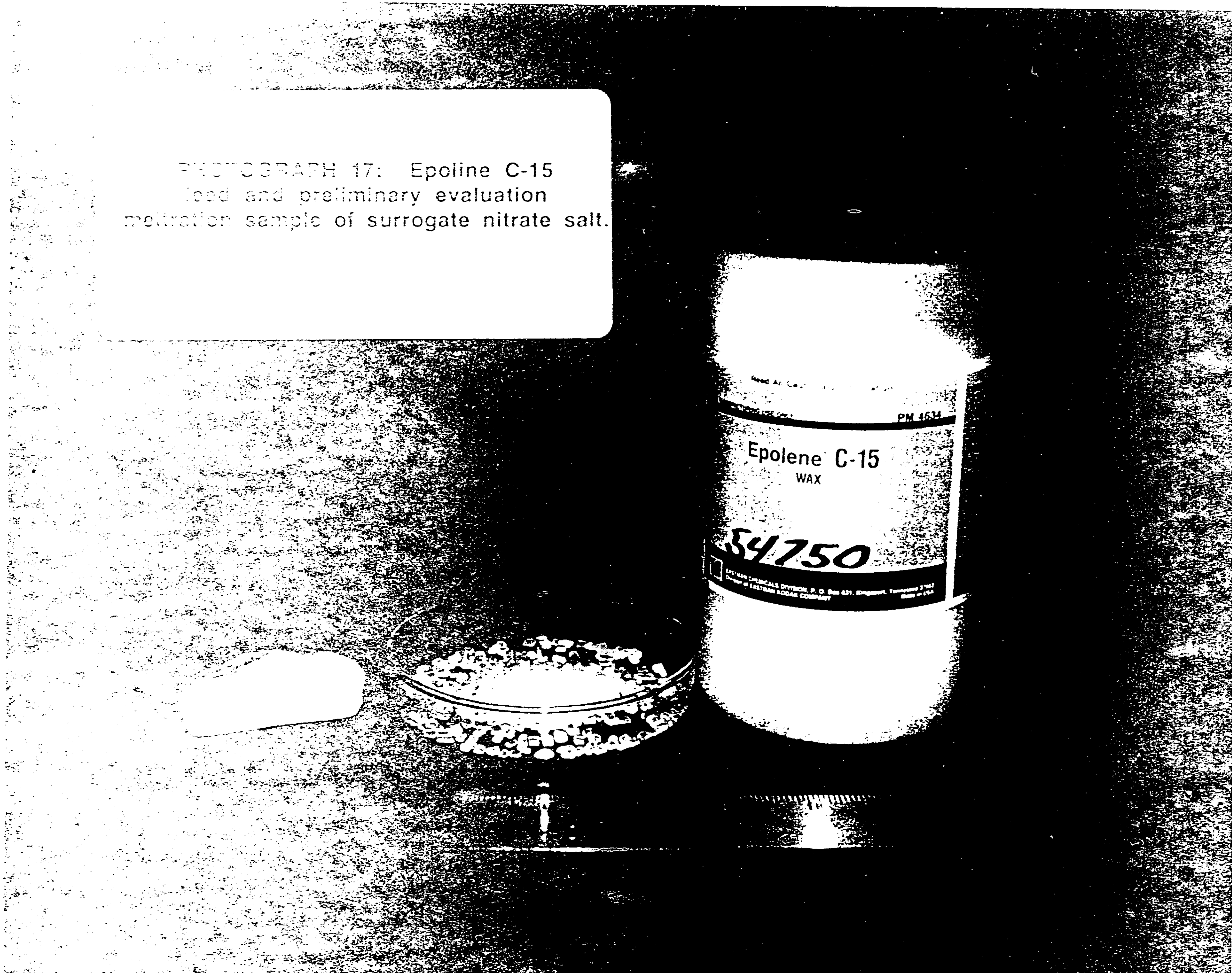
PHOTOGRAPH 14: Sample of RVF-800 dried surrogate nitrate salt. Final sample agglomerates @ approx. 7 hr. 45 min.



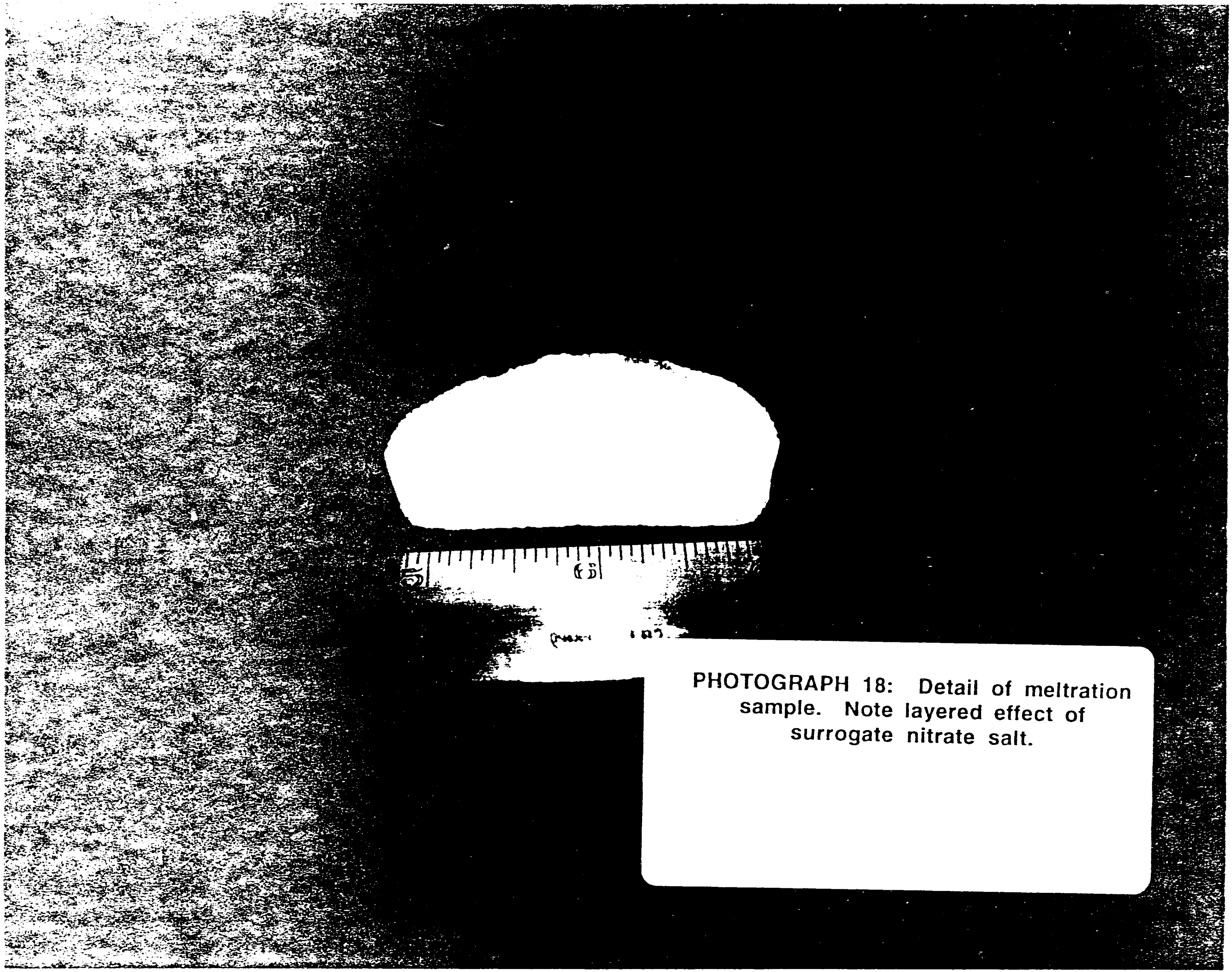
PHOTOGRAPH 15: Sample of Gelimat salt (sodium chloride) and LDPE mixture.



PHOTOGRAPH 13: Detail of [illegible] sample. Note exposed salt agglomerates.



PHOTOGRAPH 17: Epoline C-15 feed and preliminary evaluation meltration sample of surrogate nitrate salt.



PHOTOGRAPH 18: Detail of meltration sample. Note layered effect of surrogate nitrate salt.

APPENDIX 1

SELECTED TRIP REPORTS

TRIP REPORT

KILLION EXTRUDERS INC. AND WERNER PFLEIDERER CORP.

JUNE 1992

Ray Jantzen, Bob Yudnich, and I traveled to Killion Extruders for a two-day workshop on extrusion technology. We also attended a meeting with Killion technical staff to discuss RFP extrusion problems and possible solutions. Additionally, we visited Werner & Pfleiderer Corporation, a twin-screw extruder manufacturer.

The extrusion workshop was designed primarily for Killion clients in the plastics industry. However, presentations on extrusion theory, extruder design, screw design and construction materials, and data acquisition and control systems were valuable. Also of value was the overview of extrusion applications we received. The workshop consisted of both classroom lectures and "hands-on" extruder operation in the following areas:

1. Extruder theory
2. Dies
3. Pelletizing
4. Tubing extrusion
5. Blown film, cast film, and sheet extrusion
6. Profile (odd shape) extrusion
7. Coextrusion
8. Data acquisition and control systems (classroom only)

The meeting with Killion technical staff was attended by the above RFP personnel and Paul Nardone and Margaret Henke of Killion. The meeting was extremely valuable in that we were able to discuss, with experts in the field, solutions to specific RFP extrusion concerns, such as the following:

Air Entrapment—Contributes to low density. It was Killion's opinion that the RFP method of sample collection contributes to air entrapment. However, it is difficult to distinguish between air entrapment and cooling voids.

Cooling Voids—Caused when the outside of the extruded product cools faster than the inside. Void pressure differences can be used to distinguish between air entrapment and cooling voids. Also, the quicker the extruded product cools, the more voids are produced. Slowly cooling the product can decrease cooling voids and add strength.

Screw Configuration—Killion recommended that RFP identify a method to accurately determine product homogeneity before commencing discussions to define an optimum screw configuration. This would include a vented screw configuration for the Building 374 vented extruder. A hardened screw will cost about $800.00 more than a conventional screw, but will afford a much longer service life.

Die exit port transition from horizontal to vertical—Killion feels that this is not necessary. The present method of horizontal extrusion is sufficient. If vertical extrusion is desired, a heated hose capable of a 90° bend can be used. Killion has a heated hose vendor's name. They also presented the idea of placing the storage container receiving the melt on a platform that depresses as the container fills. This will keep the extruder die nozzle at the same distance from the melt pool throughout the extrusion process, minimizing "noodling" and air entrapment.

Homogeneity—Because of difficulties encountered in determining homogeneity in the extruded product, RFP personnel asked Killion for recommendations. They suggested extruding the product using a sheet die. This would result in a thin product that could be more readily examined. They also suggested extruding the material in a rod, as is presently done, and then cutting random samples or taking a series of samples over a specified time. These samples could be analyzed for homogeneity. Additionally, Gary Schmidt, the lab manager at Werner & Pfleiderer, was asked about homogeneity. He suggested that we view the product microscopically using cross-polarized light. The polarized light will fluoresce the polymer, in effect creating a contrast between the polymer and the salt crystals. He also took a portion of a 50-weight percent salt loaded sample we brought with us and prepared it using an instrument called a Carver Press. The press subjected the sample to heat and pressure, which flattened the sample to a thin disk, in which could be seen the salt crystals.

During classroom discussion Killion mentioned that the PVC molding placed around walls at floor level in many office buildings is filled with up to 40-weight percent clay. As this means the molding manufacturers are experienced in extruding filled polymer, we asked for company names. Killion suggested contacting Joe Petrozelli or Charlie Martin of American Lestritz in Somerville, N.J., or Bill Davis of Alpha Dexter in Newark.

We asked about our method of "starve-feeding" the extruder, meaning that we do not fill a hopper with material to be extruded, but instead "trickle" the material into the feed throat from the calibrated hoppers. The screw is always visible. They indicated this method will aid in producing a homogenous product. They recommended introducing the salt at the back of the feed throat to allow the screw to better start the mixing process.

Extruder Heat Zone Temperatures—The primary consideration for determining individual heat zone temperature is screw drive motor amperage. The amperage should be in the approximate center of its range (0-30 amps for the 881 extruder). Low amperage may indicate too high a temperature. Conversely, high amperage may indicate too low a temperature. Zone one (the feed section) temperature should be low relative to the other zones to insure there is no pre-melting of the polymer. A low zone one temperature will also allow better mixing of the polymer and salt and afford more consistent mechanical transport of the polymer/salt mix.

Killion Laboratory Fees—We discussed with Killion the possibility of issuing a contract to them to research extrusion parameters for nitrate salts dried by various drying technologies, such as thin film evaporation or ring drying. Lab fees are as follows:

Set up fee:	$500.00	
Hourly rate:	180.00	(4-hour minimum)
Daily rate:	1,940.00	

Killion emphasized that discounts for "long-term contracts" are available.

Glovebox Applications—Single-screw extruders may have very limited application in glovebox operations. The main reason is that there must be sufficient room to remove the screw if this becomes necessary. This would mean an area in front of or in back of the extruder barrel equal to the length of the screw plus enough room to work. It is for this reason that a twin-screw extruder be seriously considered for any glovebox applications. Twin-screw extruders are configured with the barrel and screws in sections.

Yamato Spray-Dried Salt—Killion has 1.5 pounds of spray-dried salt from Yamato to be used to optimize screw configuration. This may not be the most desirable dried material for the research effort. A more applicable salt may be one that has undergone thin film evaporation or ring drying.

Data Acquisition and Control System—Both Killion and Werner & Pfleiderer supply data acquisition and control systems for their extruders. The Killion system is a "touch screen" system with security levels, complete parameter control and statistical process control capability. It is unknown whether the Werner & Pfleiderer system has a touch screen.

Both systems can be as sophisticated as desired. The Killion system can be retrofitted to the Building 881 extruder at a cost of approximately $26,000.00. The Building 374 extruder is already configured to accept a control system. The cost of this system would be approximately $23,000.00. For research and development purposes, purchase of this system should be seriously considered.

TRIP REPORT

POWER AND BULK SOLIDS CONFERENCE

MAY 1993

Ray Jantzen and I traveled to the Rosemont Convention Center, Chicago, Illinois to attend the Powder Bulk Solids Exhibition and conference. This exhibition along with the technical classes that we attended are directly applicable to the Polymer Solidification Project.

The Polymer Solidification Process will use an extruder to encapsulate waste in a matrix of low density polyethylene (LDPE). One of the primary candidate waste streams for this process is a low level radioactive, mixed hazardous, nitrate salt waste which has been spray dried. The spray drying process at Rocky Flats Plant produces a spherically shaped salt particle with a diameter which falls in the range of .3 to 10 micron. The bulk density of this material is very low.

The small particle size and low bulk density of the salt presents several problems for the Polymer Solidification Process. Recent experiments have revealed that the single screw lab scale extruder had difficulty extruding the Rocky Flats Plant (RFP) spray dried salt and ow Density Polyethylene (LDPE) mixture. The extruder had problems maintaining a constant output throughout the extrusion process. The Polymer Solidification team speculated that this inconsistent extrusion output is due to the fine particle size and low bulk density of the slats. Waste Projects sent Bob Yudnich and Ray Jantzen to the Powder Bulk Solids Exhibition and Conference to view and evaluate equipment that may help to solve this problem.

This exhibition is the worlds largest trade show for the dry processing industry with more than 550 exhibitors in attendance and hundreds of new products on display. The following is a brief description of classes attended and exhibits visited at the solidification project were discussed or displayed.

Exhibits Visited

Draiswerke, INC., 3 Pearl Court, Allendale, NJ 07401.

Conversations with process engineers at the convention indicated that the Gelimat Mixer/compounder manufactured by the Draiswerke Company, could possible be used to encapsulate the spray dried salts. Draiswerke representatives told us that their compounding machine had many advantages over extruders including:

1) The Gelimat does not have any problems encapsulating very small size light weight materials such as the spray dried salts.
2) The Gelimat is less expensive than an extruder.
3) The machine is capable of imparting a very high degree of shear mixing force to the material in its chamber by means of high speed (up to 48 meters per second tip speed mechanical agitation. The amount and duration of this shear force can easily be increased or decreased by varying the mixing time.

The Gelimat mixer/compounder is used primarily in the plastics compounding industry. This industry combined the more expensive plastic resins (such as polyethylene with over cost filler material (such as bentonite, clay or fly ash) to form a finished product.

The machine incorporates a series of high speed blades which mix the filler material and plastic inside a chamber. The action of the blades produces frictional heating in the material by imparting an extreme degree of mechanical shear force to the material contained int he chamber. It is a batch type process that can easily be automated. Draiswerke manufacturers two models in this equipment, the batch type Model G and the continuous process Model S. Operation in the Model S is as follows; material can be automatically metered into the chamber, mixed for a certain period of time (20 seconds is common), and then a door is opened in the bottom of the chamber and the mixing action of the blades causes the contents of the chamber to be completely expelled so that the whole process can be repeated. The Polymer Solidification team plans on talking to Draiswerke in the near future about testing for RFP.

Tecnetics Industries INC., 2180 Old Highway 8, St. Paul, MN 55112.
This company sells volumetric feeders that are similar to that of the AccuRate Corporation. AccurRate feeders are currently being used by the Polymer Solidification team, but Tecnetics offers some additional features over AccuRate at a lower cost. These features include a bayonet style quick disconnect on both the feed tube and the helix. Also, the Tecnetics hopper is much easier to detach from the feeder which is very beneficial when removing unused material during cleaning. In addition, Technetics representatives claim that their polyurethane hopper is stronger and more abrasion resistant than the AccuRate vinyl hopper.

Fitzpatric Company, 832 Industrial Drive, Elmhurst, IL 60126.
This firm makes compacting equipment for condensing fine, aerated powders such as the spray dried salts. The compacting equipment forces fine powders between two counter rotating rolls. As the volume decreases throughout the region of maximum pressure, the material is formed into a solid compact referred to as a briquette. It is also a common practice to form compact granules with this type of machinery. The Polymer Solidification team is considering methods of increasing the bulk density and particle size of the spray dried salts to improve extrusion. It may be possible to run the spray dried salts through such a machine to form a granule so that it could be successfully extruded.

However, there are inherent problems in compacting spray dried materials and testing would be both necessary and prudent prior to a purchase of equipment. Work is in progress at this time to identify an outside contractor capable of spray drying surrogate nitrate salts for use in these type of tests. Also, an investigation is underway to identify, for purchase, lab scale or bench scale equipment which could be used under a ventilation hood. The purpose of this equipment will be compaction or densification of RFP spray dried salts in Building 374.

APPENDIX 2

TEST OBSERVATIONS - VTFE

AUGUST 1993/LCI CORP.

August 12 Vertical TFE

Materials for the testing was received at approximately 9:00 AM and LCI technicians began mixing of the surrogate salt brine shortly after this time. The test run was started at approximately 11:30 AM.

12:24 PM A note is made of problems arising due to erratic feed into the vertical TFE. The test operator speculates that the cause is undissolved solids in the feed which are causing erratic behavior in the feed valve. This valve is a needle type valve known as a Masoneilan Valve. The operator decides to shut down the test and replace the feed valve with a Moyno Feed Pump. This is a pump that depending on the speed that it is turned, delivers progressively greater quantities of material at an accurate rate.

2:24 PM The Moyno Pump is installed and running and some powder is coming out of the bottom valve. The test setup included two rotary lock type valves in series beneath the TFE discharge. The purpose of having two valves was so that it would be possible to maintain pressure lock on the unit during operation and still be able to discharge material on a timed interval basis. For some reason these valves were not working during the test and the unit was run open to atmospheric pressure.

3:35 PM After performance of a Periodic Rate Check the operator notes that the collected material shows evidence of retained moisture. This is indicated by caking and no free flowing powder. Prior to this time the feed rate was calibrated at approximately 80 LB/HR based on drive revolutions.

3:50 PM Pulled the rotor out and found fouling (dough-like build-up of material on the surface) in the center portion of the rotor. The operator speculates that this is probably due to too high of a feed rate. They will clean the rotor and the TFE up and then try again with a lower feed rate.

5:10 PM Unit is back together and running and we are getting wet stuff out of the discharge again. The material cycles between liquid aNd dry powder. The operator thinks that the feed rate is still too high. The drive unit on the Moyno is turning at 52 revolutions per minute which equates to about 73 pounds per hour. This is as low as this particular drive unit will turn. The operator suggests that we change the drive unit to one which will turn slower and try the test at a lower feed rate in the morning. I agree.

5:45 PM We pulled the rotor for inspection and cleaning. It is not as badly fouled as last time (no doughy build-up on the shaft and the pendulum blades are free moving) indicating that we are on the right track by lowering the feed rate and that we need to go lower. The operator also mentions that the spyder at the bottom of the unit is plugged and that this is also commonly caused by too high of a feed rate.

6:00 PM We agree to call it a day. We will change the drive unit on the Moyno Pump in the AM and try again with the vertical unit.

August 13 Vertical TFE

8:30 AM The drive unit has been changed and the Moyno Pump is turning at 32-34 RPM. The rate yesterday was 52 RPM. The feed rate is calculated to be around 42 LB/HR. The operator reports that the final moisture analysis on the product sample taken yesterday was 0.5%. b This figure comes after having cooked the material all night at 200 degrees C with 30 LB vacuum. The sample moisture content was reported at 0.3% after approximately one hour of cooking in the oven. What this means is that the last bit of moisture left the material only after being in the oven for a long time. It should also be noted that in performing the moisture analysis it was necessary to remove the sample for the oven and transfer it to an analytical balance for weighing. While the sample was on the balance one could observe the weight increasing due to the hygroscopic properties of the material.

9:10 AM A rate check performed by the operator verifies that the feed rate is 40 LB/HR. The operator comments that this is the first rate check that he has done during this test wherein the figures added up and made sense (ie. bottoms and vapor in proper proportion to feed material).

9:25 AM The operator shows me a sample from the feed tank which contains undissolved solids. He feels that the material is probably Calcium Sulfate which is formed from a reaction of Calcium and Sulfate ions in the feed material. He has had recent experience with this compound and states that he believes that it has an inverted solubility curve which means that the more this material is heated the less soluble it becomes. He tells me that this can add to fouling problems in TFE's. (Upon returning to RFP, I confirmed the above by talking to a chemist in our group. This compound and its unusual property may be consideration in the design of any future drying systems at RFP as these ions are present in considerable proportions in the Nitrate Salt waste stream).

9:36 AM We are getting what appears to be a good steady supply of dry powder out of the unit. I took a sample and performed bulk density and moisture analysis on it as well as taking note of the flow properties.

Bulk Density	.93-.94 gm/cc
Moisture Analysis	0.3% after one hour in the oven at 200 degrees C and 30 LB vacuum
Flow Properties	The material flowed through the funnel but it was necessary to facilitate the flow by tapping on the funnel with a hard object.

Other Observations The material appears to be of a finer granule size than material produced yesterday.

Bottoms pH-=5 Feed pH=12

10:36 AM Increase the revolutions of the Moyno Pumps to 42-44 RPM's. This equates to a feed rate of approximately 50 LB/HR.

11:06 AM During the rate check the operator notes the presence of wet material in the sample which indicates fouling of the unit. We will shut down and pull the rotor for inspection.

11:34 AM Minimal fouling around the middle set of blades of the rotor. The operator makes a comment that this could be indicative of the need to check the clearance on these blades.

At this point in time the test of the vertical unit was terminated.

August 13 Horizontal TFE

Preparations were started for the test on the horizontal TFE unit.

1:40 PM We are now running and getting product out of the horizontal unit. It is more granular and much larger particle size that the product from the vertical unit. The Moyno is turning at 80 RPM's which gives a feed rate of approximately 100 LB/HR.

I took a sample and performed bulk density and moisture analysis on it as well as taking note of the flow properties.

Bulk Density	.87 gm/cc
Moisture Analysis	0.2% after one hour in the oven at 200 degrees C and 30 LB vacuum
Flow Properties	The material flowed well through the funnel with no help required
Other Observations	The material is of a much coarser granule size than that produced in the vertical TFE.

3:56 PM The operator will perform one more rate check after approximately 1.5 hours running at 100 LB/HR. We will then pull the rotor and check for signs of fouling. A bulk density check is also performed at this time.

Bulk Density	92.7 ± 0.1 gm/100cc

4:30 PM After some difficulty the rotor has been removed and is ready for inspection. It should be noted that the rotor on the horizontal unit was much more difficult to pull than that of the vertical unit. This would be a consideration in choice of designs (vertical over horizontal) and possibly in the design of the glove box enclosure if it is required. The primary problem was that the rotor had to be winched out of the shell. The only support for the rotor during this operation was the rotor elements riding on the heat-exchange shell. This may be of consideration in a larger unit with a heavier rotor.

Some fouling is evident on the feed end of the rotor. The cause of this is probably due to the doughy stage that the material goes through as it dries and there is a strong likelihood that this problem could be either reduced or eliminated by adding more of the "pusher" type blades (transport fins) in this region. The operator also notes that the entire last 1/3 of the rotor is free of any material build-up. This indicates that all drying has occurred previous to this point and therefore we were operating the unit at well below capacity.

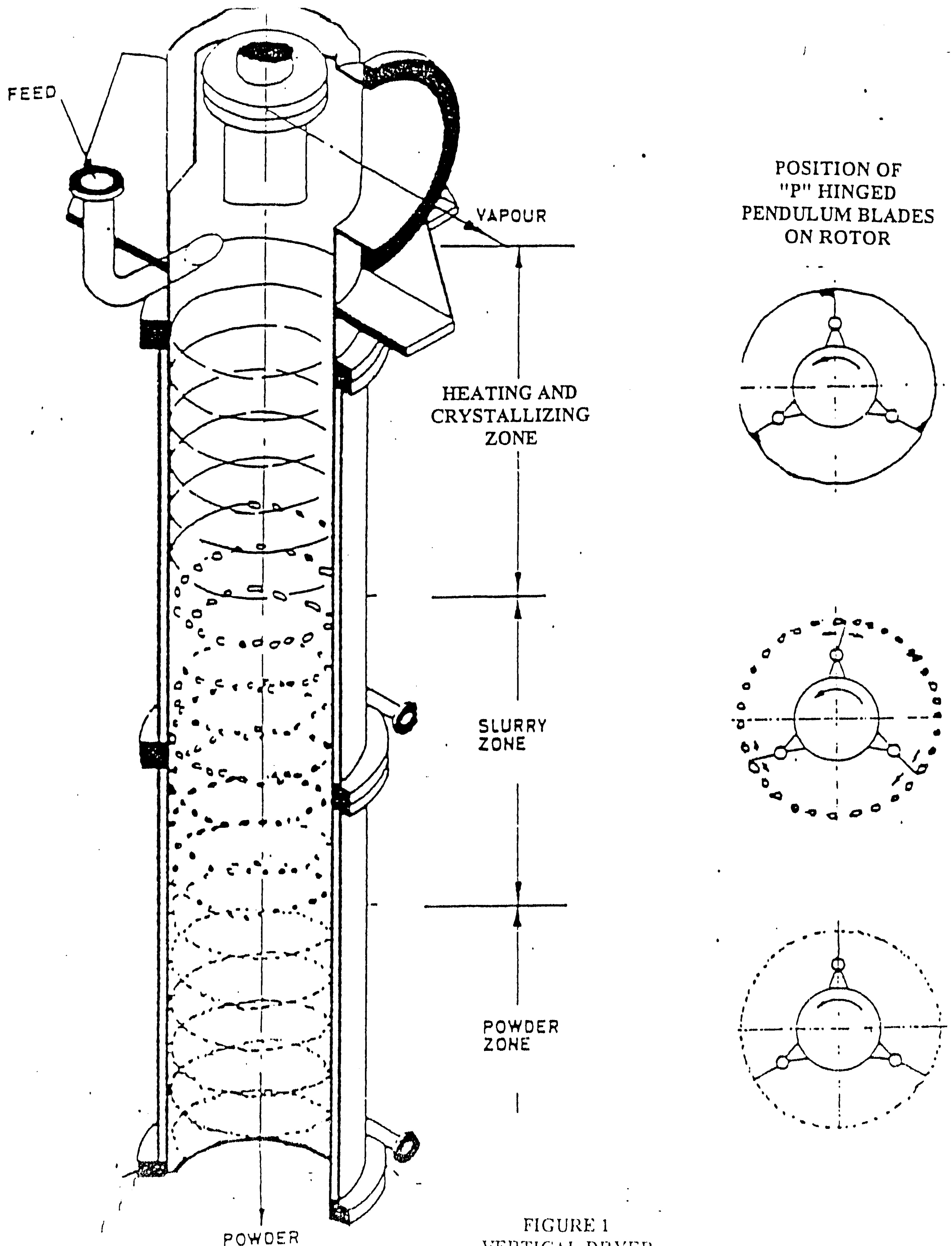


FIGURE 1
VERTICAL DRYER

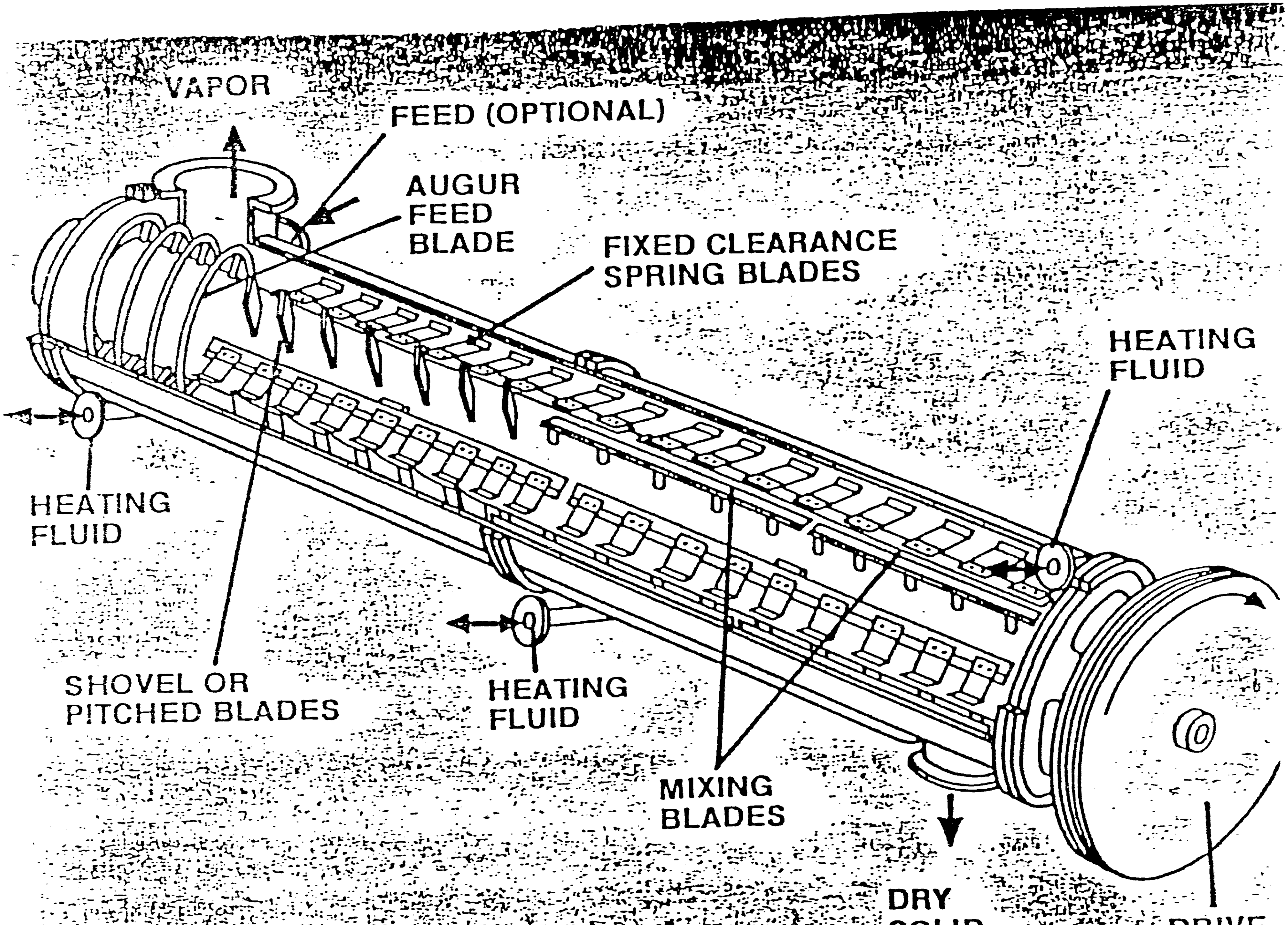
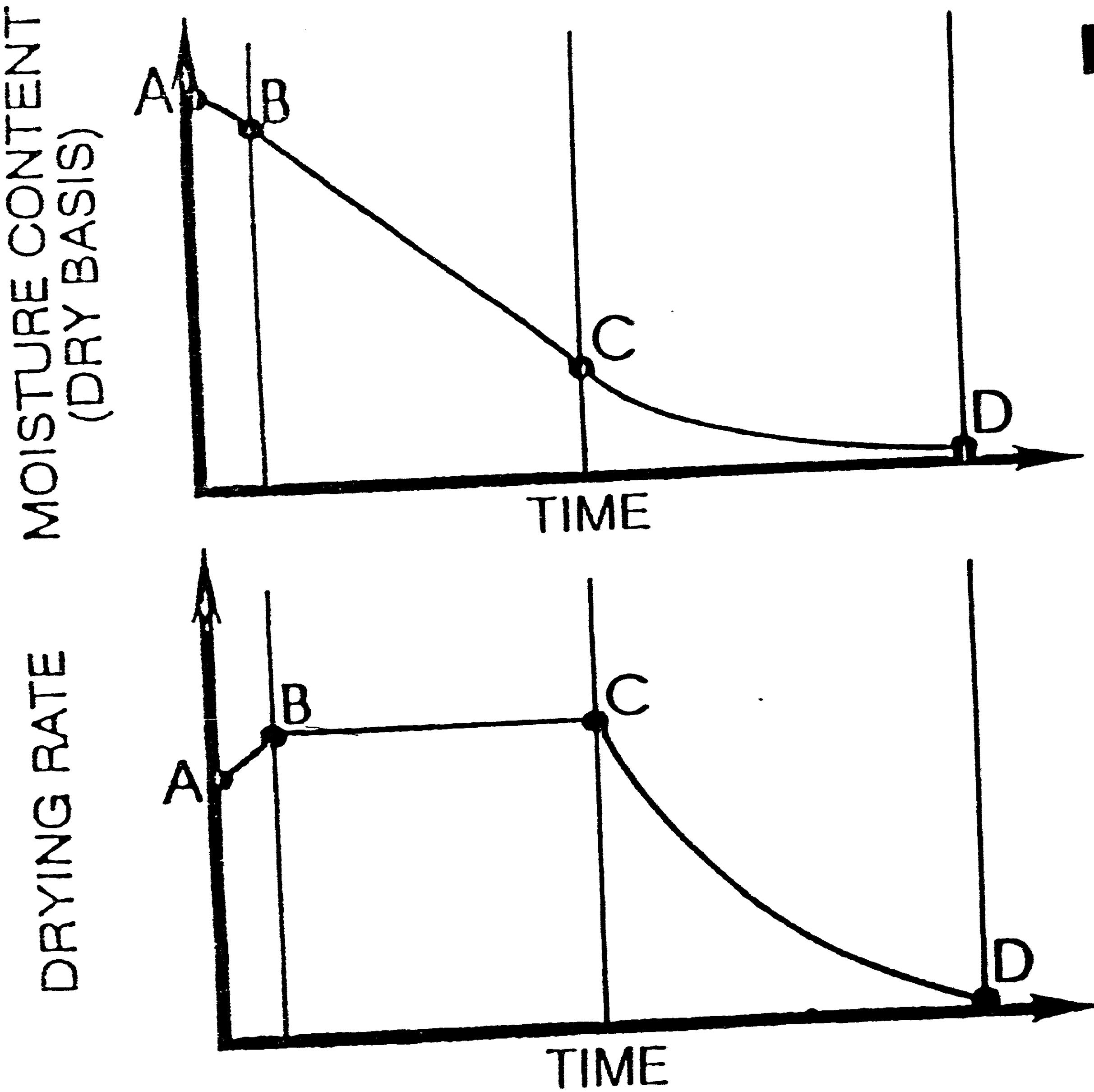


FIGURE 2
HORIZONTAL DRYER



DRYING PERIOD

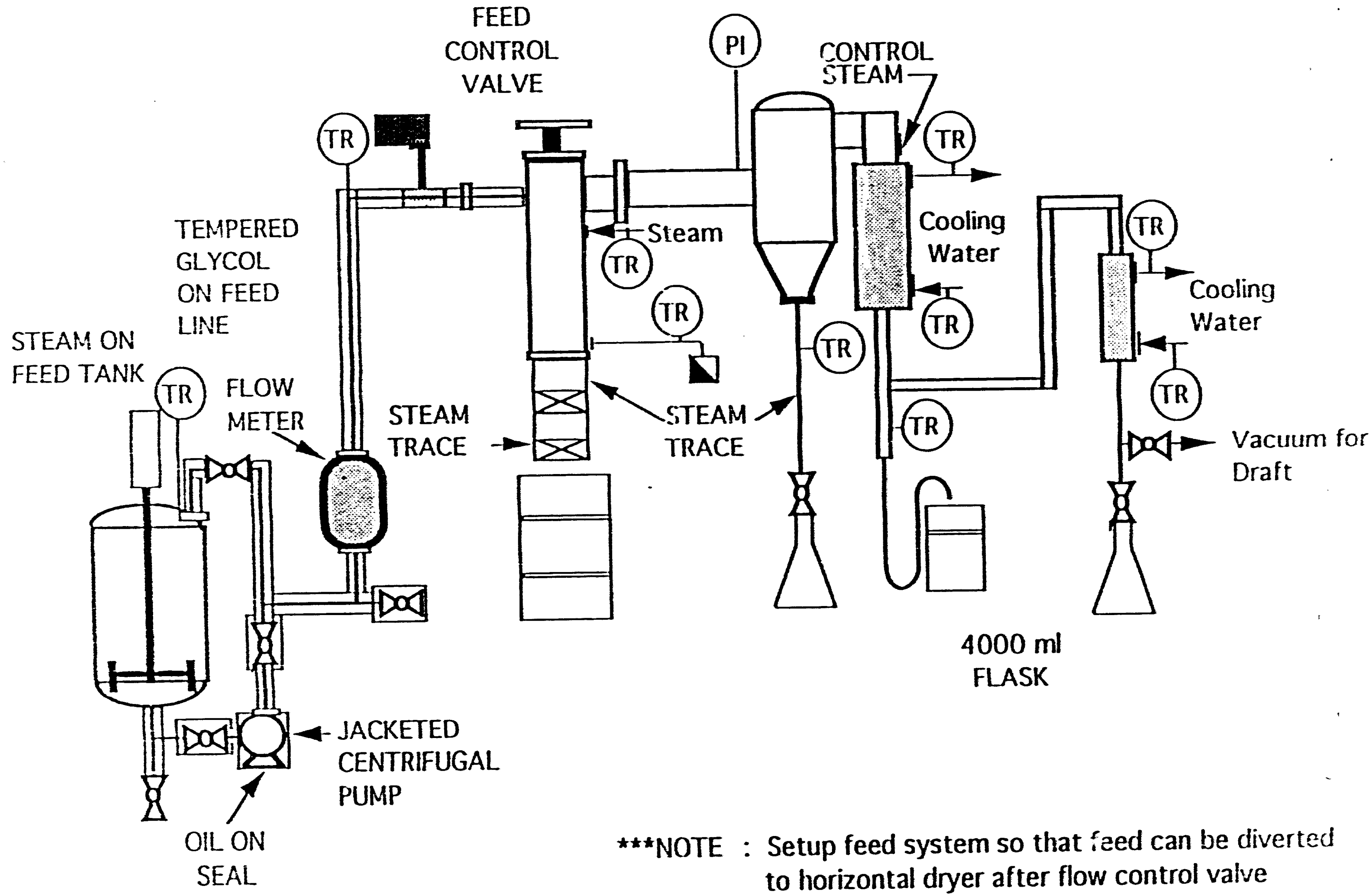
A TO B EVAPORATIC OF BULK LIQUID.

B TO C CONSTANT RATE DRYII PERIOD

C TO D FALLING RATE DRYII PERIOD

FIGURE 3
DRYING PERIODS

EG&G TEST 94D019



***NOTE : Setup feed system so that feed can be diverted to horizontal dryer after flow control valve

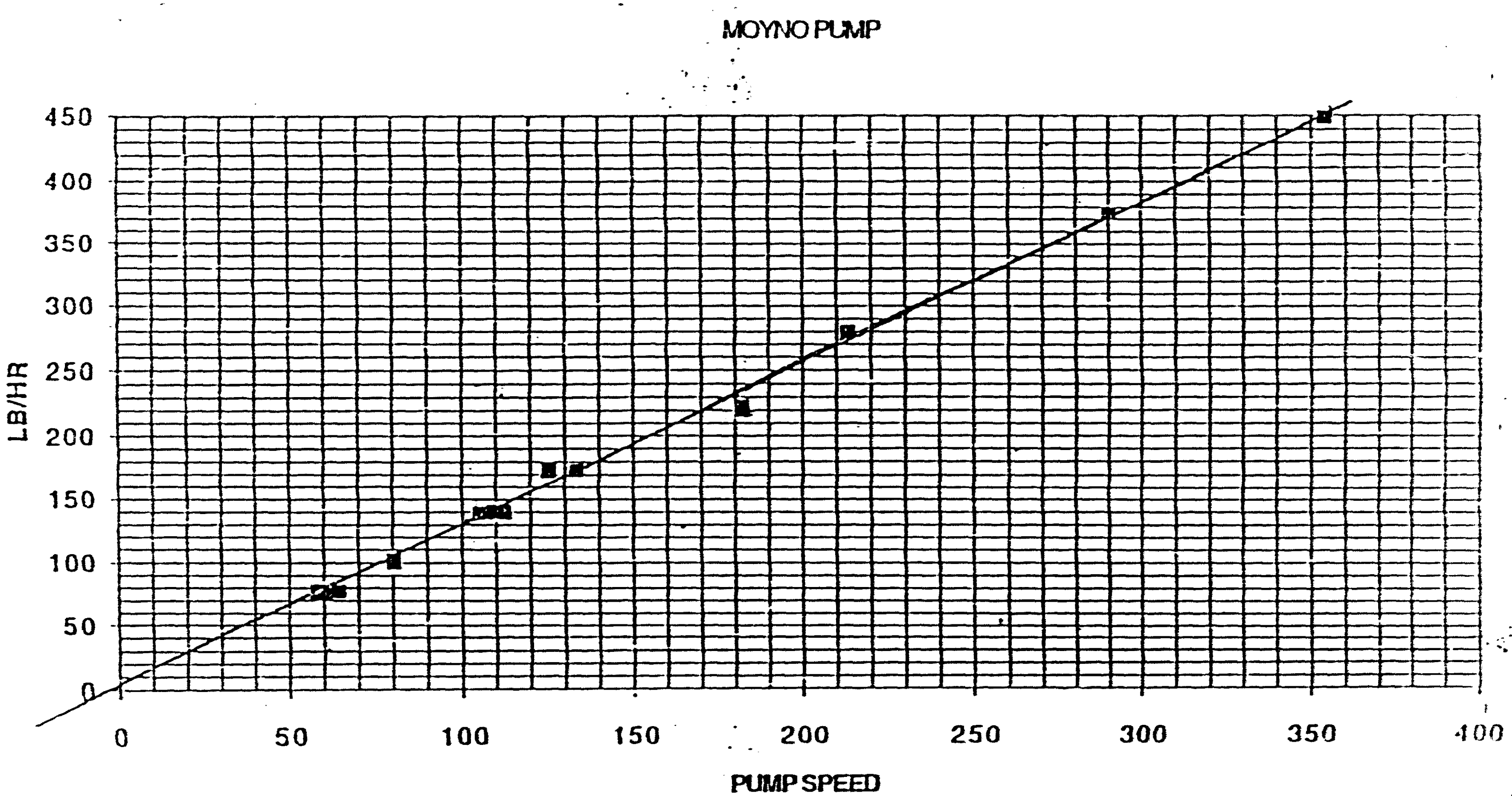


CHART 1
FEED CALIBRATION CHART
FOR MOYNO PUMP

EG&G TEST T93D019

Run #	1	2	3	4	5	6
Length of Run (min.)	6	6	6	6	6	6
Product	Ultralo... State/C					
Date	8/11/93	8/12/93	8/13/93	8/13/93	8/13/93	8/13/93
Time	1500	1525	0910	10:15	11:10	11:30
Operating Press (mmHgA)	ATM	ATM	ATM	ATM	ATM	ATM
Steam Press (psig)	150	150	150	150	150	150
Rates (lb/hr)						
Feed Rate by Flowmeter	79-88	90	36	36-37	50	50
Feed Rate by Addition	97	67	40	46	50	52
Bottoms	8.3/2.4/55	5.5/2.4/31	1.5/0.3/12	3.0/0.3/17	1.6/0.4/12	1.8/0.4/14
Vapor	6.2/2.4/38	6.0/2.4/36	5.2/2.4/28	5.3/2.4/29	6.2/2.4/38	6.2/2.4/38
% Overhead	39%	40%	70%	63%	76%	73%
Temperatures (°C)						
Feed Tank	83	83	75	78	80	80
Feed Line	83	83	76	79	80	81
Bottoms	43	46	55	79	93	97
Vapor at Evaporator Outlet	102	101	99/103	100/110	100/112	102/112
Dryer Heat In	187	188	187	188	188	188
Dryer Heat Out	187	188	187	188	188	188
First Condenser Coolant In	33.1	33.2	29.6	30.8	32.6	32.6
First Condenser Coolant Out	34.2	34.2	30.2	31.8	33.2	34.1
First Distillate	42.3	43.4	31.3	30.6	31.1	31.6
Second Condenser Coolant In						
Second Condenser Coolant Out						
Rotor Speed (RPM)	952	952	950	950	950	950
Feed Pump Spd (RPM)	50	50	32	32	42	42
Rotor Amps	5.5	5.6	5.5	5.5	5.4	5.5
% Moisture	0.5%		0.2%			

160

CHART 2

EG&G TEST T93D019

Horizontal Dryer

Run #	7	8				
Length of Run (min.)	6	6				
Product						
Date	8/13/93	8/13/93				
Time	1500	1600				
Operating Press (mmHgA)	ATM	ATM				
Steam Press (psig)	200/170	200/170				
Rates (lb/hr)						
Feed Rate by Flowmeter	100	100				
Feed Rate by Addition	107	113				
Bottoms	4.2/0.3/39	4.8/0.3/45				
Vapor	6.2/2.4/68	4.2/2.4/68				
% Overhead	63.6%	60.2%				
Temperatures (°C)						
Feed Tank	83	83				
Feed Line	82	83				
Bottoms						
Vapor at Evaporator Outlet	122/103	123/112				
Dryer Heat In	190/196	191/197				
Dryer Heat Out	192/196	192/197				
First Condenser Coolant In						
First Condenser Coolant Out	39	40				
First Distillate						
Second Condenser Coolant In						
Second Condenser Coolant Out						
Rotor Speed (RPM)	600	600				
Rotor Amps	7	7				

CHART 3
EG&G TEST T93D019

	MOLE	MOLE	MOLE	GM
	Ca	SO4	CaSO4	CaSO4
0	0.012	0.012	0.025	3.355
5	0.012	0.012	0.024	3.232
10	0.011	0.011	0.023	3.123
15	0.011	0.011	0.022	3.024
20	0.011	0.011	0.022	2.934
25	0.01	0.01	0.021	2.853
30	0.01	0.01	0.02	2.779
35	0.01	0.01	0.02	2.717
40	0.01	0.01	0.019	2.65
45	0.01	0.01	0.019	2.592
50	0.009	0.009	0.019	2.54
55	0.009	0.009	0.018	2.491
60	0.009	0.009	0.018	2.445
65	0.009	0.009	0.018	2.403
70	0.009	0.009	0.017	2.363
75	0.009	0.009	0.017	2.326
80	0.008	0.008	0.017	2.291
85	0.008	0.008	0.017	2.259
90	0.008	0.008	0.016	2.228
95	0.008	0.008	0.016	2.199
100	0.008	0.008	0.016	2.172
MW				
CASO4				
136				

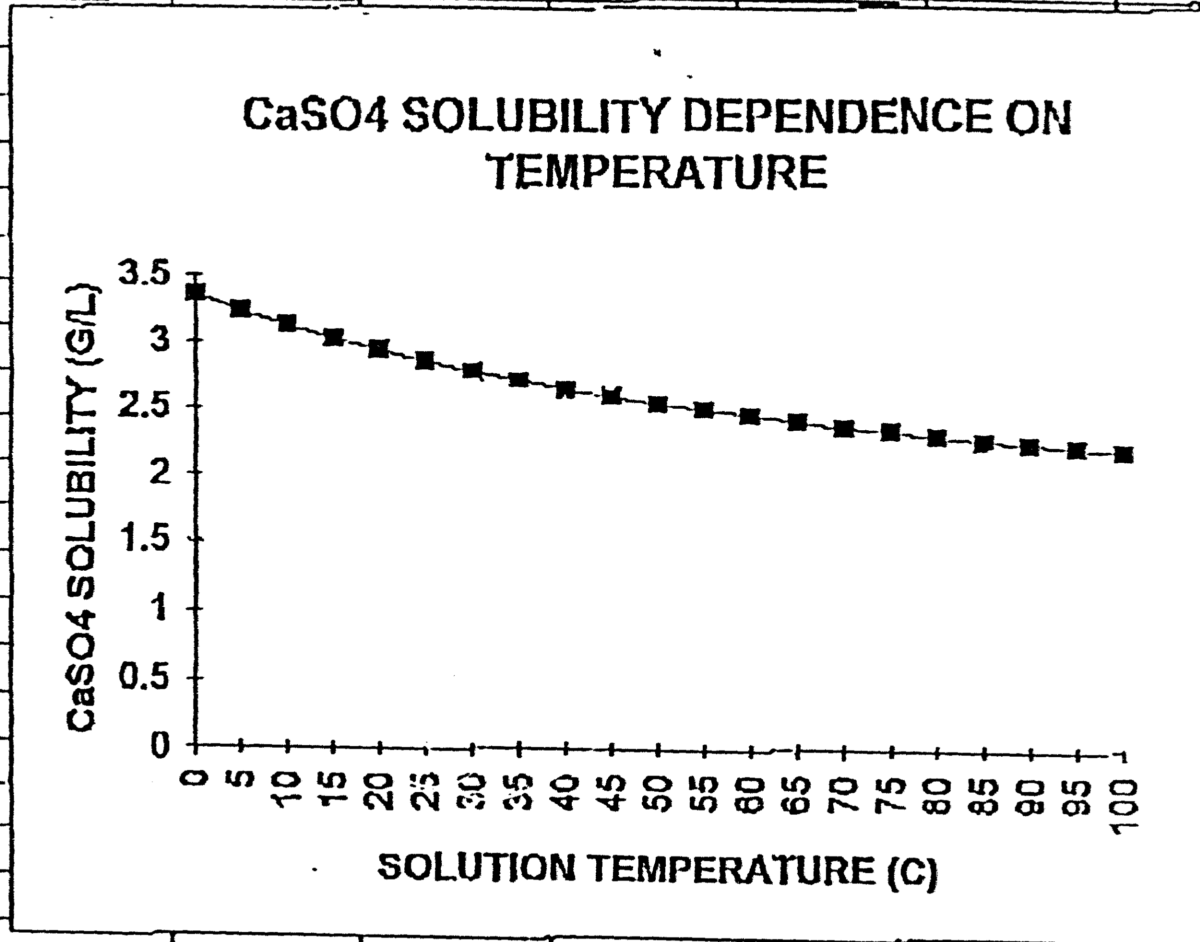


CHART 4
SOLUBILITY DEPENDENCE

APPENDIX 3

TEST OBSERVATIONS

HORIZONTAL ROTARY BLENDER/DRYER

MAY 1993 PNS

PNS RVR - 800 Demonstration

Description

Horizontal unit with design basis of 800 ga/day. Unit designed and built to evaporate liquids from nuclear power plant waste solutions. Unit designed to produce a free-flowing powder of typically 10% moisture with no visible free liquids. Frequently add a paraffin wax to the dryer once a powder is formed. The heat of the dryer melts the paraffin wax and paddles in the mixer mix the molten paraffin with the waste powder. The mix is discharged into a 55-gal drum, where it solidifies upon cooling.

PNS is currently working on a vertical design which will have a better likelihood of producing a free flowing powder with a moisture content of less than 1%.

Test Demonstration

500 pounds of dry D884 surrogate was mixed with 930 pounds of water in the blender/dryer to produce a 35% solids solution. The solution was not pH adjusted to represent the actual salt waste stream. The surrogate salts were prepared by BD Chemical and shipped in two batches (sodium nitrate in one batch and all other constituents in the other batch) to prevent segregation due to the relatively large particle size of the N_aNo_z salts.

Drying of the surrogate solution began at approximately 10:00 AM. At 3:00 PM, a thick paste formed. At 3:25 PM, a doughy paste formed, and by 3:40 PM, the material resembled a flaky dough. At 4:45 PM, a sample was collected by dumping some product from the bottom of the blender/dryer. No free standing liquids were observed, and the particle size ranged from 1 mm to 1/2 inch. The moisture analysis on the particles indicated 2% residual moisture. When the particles were crushed, the moisture content increased to 2.7%.

After the sample was collected at 4:45 PM, the PNS operator began "shocking" the dryer by driving the temperature up and down to accelerate the drying process. At 5:09 PM, dusting was observed in the dryer. Also, the product was beginning to agglomerate. The wipers in the unit began rubbing hard balls of product against the walls of the unit, creating a significant amount of noise. At 5:20 PM, some product was dumped to attempt to accelerate drying and reduce abrasion of the product against the walls of the dryer. The product consisted of very hard marble-sized pellets and a fine powder. Moisture analysis on the pellets indicated a moisture content of 0.96%. When the pellets were crushed, the moisture released increased to 1.82%.

At 5:45 PM, the PNS operator stopped the test because of concerns that over possible damage to the dryer unit. That abrasion of the product against the walls of the dryer could be damaging the unit. By this time, abrasion of the product against the walls of the blender/dryer was creating a considerable amount of noise. The temperature in the dryer at the completion of the test was 180°.

The final product consisted of a fine powder and hard pellets ranging from approximately 2 mm to marble-sized. A few large chunks of material ranging from 1 1/2 inch x 3 inches were observed. The PNS operator indicated that some larger sized material may have remained inside the blender/dryer cavity.

The product material was streaked with grey due to cross-contamination from a SRS test the day before. The SRS surrogate waste consisted of a class F fly ash spiked with heavy metals to simulate the output from their proposed mixed waste incinerator. The specific heavy metals added and their corresponding concentrations were not provided to RFP, but the PNS operator stated that one of the metals added was mercury and that concentrations of some of the metals were as high as 1500 ppm.

Use of the PNS RVR-800 blender/dryer unit is not recommended for pretreatment of the RFP nitrate salt waste prior to polyethylene extrusion. First, the low throughput rates of the unit make it impractical for the application. Multiple units would have to be installed in an existing facility with limited available floor space in order to meet the throughput requirements of LWTO, Second, the large particle distribution of the final product is not compatible with the polyethylene extrusion process. An additional unit process, such as a grinder, would be required to produce a material that could be effectively processed in an extruder. Finally, the long-term reliability of the RVR-800 for this particular application is questionable. The RVR-800 was designed to remove visible liquids and produce a free flowing powder, not to produce a powder with less that 0.5% moisture. Based on the noise produced by the unit at the lower moisture ranges, it is apparent that frequent operation of the unit under these conditions could result in early wear and possible failure of the unit.

The RVR-800 should be considered, however, for concentrating other smaller volume mixed waste streams at RFP, especially prior to cementation or micro wave solidification. When a vertical version of the RVR-800 is available, it should be evaluated for its ability to effectively dry some of the smaller volume mixed waste streams at RF prior to polymer encapsulation.

APPENDIX 4

PRELIMINARY EVALUATION AND RECOMMENDATIONS
(PES #E378-P94005)

PREPARED FOR EG&G, ROCKY FLATS

BY

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Charlotte, NC 28297-8804

1.0 OBJECTIVES

The objective of this evaluation is to determine if LCI Thin Film Technology can be used to concentrate and solidify nitrates salts in a matrix of low density polyethylene. The mixture on a dry basis is composed of:

37.3% sodium nitrate
31.6% potassium chloride
17.7% sodium sulfate
6.2% calcium carbonate
5.2% sodium fluoride
2.0% magnesium chloride

The feed concentration is to be 35% total solids, 65% water.

2. EVALUATION MEANS

The solids, which were pre-blended, and polyethylene were provided by EG&G. The sample of solids was mixed in a beaker and placed on a heated stir plate. The properties of the evaporating sample were observed for solubility, boiling point, and handling properties.

These observations coupled with LCI's previous experience, both in the pilot plant and in the field, allows the proper selection of processing equipment.

3. EVALUATION PROCEDURE AND OBSERVATIONS

A 200 gram sample was mixed from 70 grams of solids and 130 grams of water. The sample was mixed in a 300 ml beaker. The sample volume was 160-165 ml as indicated by the graduations on the beaker. At 30 ^{o}C the sample was a slurry. A teflon mixing bar was placed in the beaker with the sample and the beaker was placed on a heated stir plate. The temperature was monitored with a mercury thermometer which has a range of 0-400 ^{o}C. The sample temperature increased slowly to 102 ^{o}C before it began to boil. The sample did not completely dissolve. When the temperature of the solution reached 105 ^{o}C, 125 ml remained. at 109 ^{o}C, 75 ml remained. At this point the sample began to get thick. Some polyethylene beads were dropped into the sample. The beads floated but did not melt.

A few of the polyethylene beads were placed in a Brookfield Thermocell. This is a temperature controlled device normally used for measuring viscosity. The temperature was slowly ramped from room temperature to 100 ^{o}C. The sample did not melt. The first signs of melting occurred at 115 ^{o}C but the sample was not flowable. Even as the temperature was ramped to 140 ^{o}C the sample did not flow. A curve of viscosity vs. melt point for the polyethylene provided which was Chevron PE 1409 is shown in Figure 1.

We decided that a polyethylene with a lower viscosity and lower melting point would be needed. A sample of Epolene C-10 and C-15 was acquired from

Eastman Chemical. Viscosity measurements were made on these samples with a Brookfield DV-II using a Thermocell and an LV-31 spindle. The curves of viscosity vs. temperature are shown in Figure 2.

Another sample of surrogate solution was mixed using 70 grams of solids and 130 ml of water. This time the solids were blended using a solids kneader. The sample was not totally in solution at 80 °C. The beaker was placed on the heated stir plate and was gradually hated. The sample began to boil at 104 oC but was still not completely in solution. The sample volume was approximately 15 ml. When the volume was concentrated to about 100 ml the temperature was 1067 °C. Seventy (70) grams of C-15 polymer was added and the stir bar was removed and the sample was stirred manually with a spatula while the solution was being concentrated. When the remaining mass reached 150 °C the mixture was poured into aluminum weigh dishes. The sample solidified to a strong block of polymer with embedded solids.

4. <u>CONCLUSION</u>

A process for simultaneous concentration and imbedding in polyethylene in a thin film evaporator using Chevron PE 1409 would not be possible at atmospheric pressure. The boiling point of the mixture is too low and the melting point of the polyethylene is too high. It might be possible to accomplish the process under pressure (10-40 psig) to elevate the boiling point. Still the viscosity of the polyethylene will require a positive transport rotor as the polymer will not flow by gravity. A process using Epolene C-15 for simultaneous concentration and embedding should work in a standard thin film evaporator. The viscosity and melt point are low enough for atmospheric operation.

5. <u>RECOMMENDED COURSE OF ACTION</u>

A pilot test in LCI's test center will be necessary for generating samples for EG&G's evaluation of final waste form properties. The test can generate samples with various solids loading and generate data for scale-up to commercial rates.

Test Unit:	Standard Thin Film Evaporator (5.4 sq. ft.)
Required Time:	3-5 days
Product Requirement:	Enough solids for mixing 3000 lb. of surrogate solution and 1000 lb of Epolene C-15

Figure 1

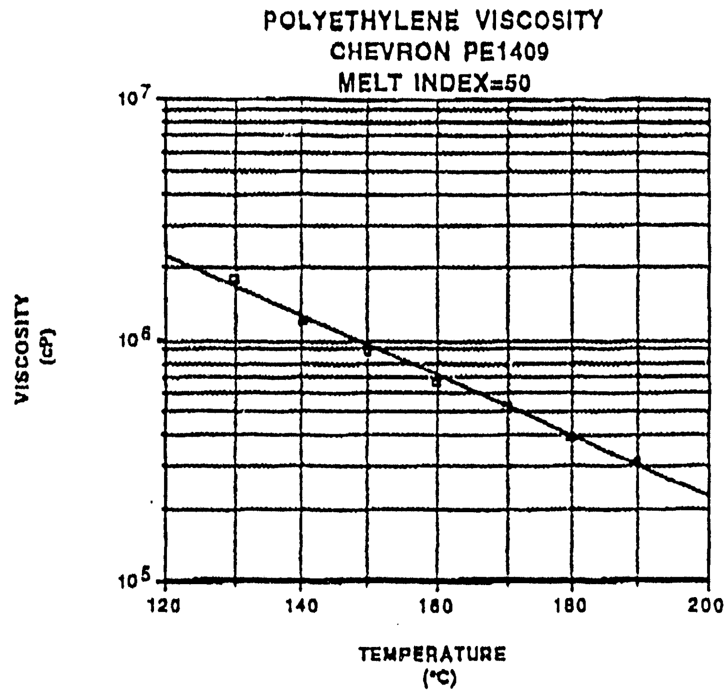
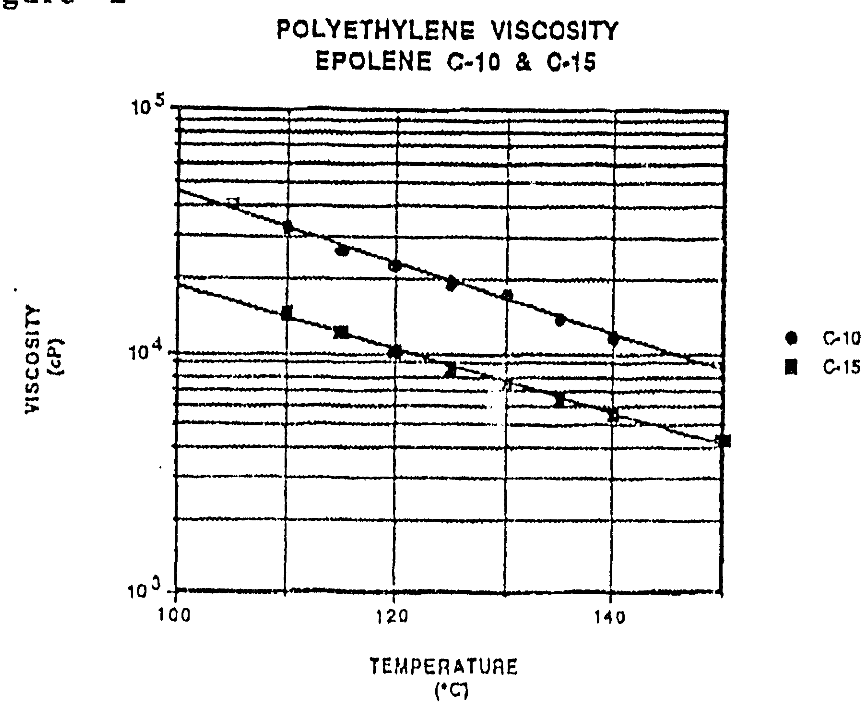


Figure 2



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