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METHODS FOR MONITORING INITIATING SOURCES GENERATED IN PYROTECHNIC PROCESSING EQUIPMENT

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Conditions leading to an accident in batch processing of pyrotechnic materials were investigated. Levels of temperature, impact, friction, and electrostatic energy were defined for five typical pyrotechnic materials. The most sensitive of these materials yielded the following thresholds: temperature, 510°C; impact, 41 in-lb; friction, 10^8 Newtons/square meter, and electrostatic energy, 29 millijoules.

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20. Abstract (continued)

Means of detecting these levels of driving force were sought for processing operations currently used: mixing, blending, granulating, pressing and drying.

A sound (IMS) system is outlined for further testing and control. This system should permit the earliest possible detection of potential problems and allow more time to act on a potentially hazardous situation.

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Mr. Louis Avrami, Dr. Frank Taylor, Mr. Ted Boxer and Dr. Burton Werbel all offered helpful inputs to this program. Louis Avrami arranged for experiments to be conducted at the Picatinny Arsenal pyrotechnic processing facility and added materially to the IMS development. Some of the photographs in this report were supplied by Dr. Frank Taylor.

Many Franklin Institute personnel contributed to the work reported here. C. W. Hargens supplied the guidance on the sound measurements as well as analysis of the recordings. W. J. Dunning made the measurements and supplied the instrumentation. F. W. Sweeney provided the IMS measurements and selected chemical tag materials.

FOREWORD

The basic objective of this program was to investigate the possibility of detecting at the earliest possible time the presence of any energetic stimuli which could cause initiation, during processing, of the various types of compositions employed for making pyrotechnic end-items.

Considered in this study was early detection of (1) solvent vapors and/or "tag" materials generated by the composition due to the creation of "hot spots", (2) unusual sounds indicating the unwanted and dangerous presence of frictional and impact forces, (3) the accumulation of electrostatic charge, or (4) any other discernible form of energy. Early detection would forewarn operators of the growth of these possible initiation sources so that they would have sufficient time to escape the processing area. Concurrently devices could be automatically activated to quench the initiation reactions, thereby preventing widespread propagation leading to possible destruction of life and property. This program presents some preliminary thoughts for accomplishing these goals.

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1. INTRODUCTION

Fires and explosions have occasionally occurred in pyrotechnic production facilities. Normally, facilities have been built around the premise that accidents would happen. Accordingly, buildings were constructed to relieve excessive pressure and attempts were made to keep bulk explosives, pyrotechnics and other reaction materials to some minimum quantity. In early production efforts, loss of life was high in any accident that occurred. Though vastly improved facilities exist, we are still not completely confident of safety in the production of materials that explode or deflagrate.

Response speed is paramount in bringing about control of a runaway reaction or conditions leading to such reactions in a pyrotechnic batch production facility. Early detection of a reaction or the factors responsible for bringing about an initiation and rapid, reliable countermeasures will reduce loss of life and property.

To achieve fast response it is necessary to rapidly detect conditions leading to possible initiation of the pyrotechnic materials being processed. Such a hazard detected early enough can lead to:

1. Shut down of processing

2. Evacuation of personnel from the plant

3. Application of automatic cooling or fire fighting apparatus, or

4. Warning to look for the source of the problem.

Any or all of these measures would lead to preservation of life and property, the primary objective of this study.

The main batch processing procedures considered for this study were mixing, blending, granulation, drying and consolidation. The machines indicated below were taken as exemplary for purposes of this study.

Mixers are generally of the Muller-Mixer type which use a fold and roll process to blend constituents. Mixing is done in a metal (usually stainless steel) bowl. Figure 1-1 shows the machine used at Picatinny Arsenal.

Granulating is accomplished by drawing an oscillating, bladed roller across a stainless steel screen. The roller has fixed linen-filled phenolic blades which contact the screen. Figure 1-2 shows a typical granulator.

Dry blending is carried out in a device similar to a ball-mill. The agate balls normally used in a ball mill are replaced by conductive rubber stoppers.

Consolidation of illuminants, delays and igniter mixes is carried out in a pressed die that is activated in a hydraulically operated press.

Typical pyrotechnic mixes used in batch processing are listed in Table 1-1 which shows characteristic sensitivities to stimuli from a number of sources. These were extracted from various PADS (Picatinny Arsenal Determination of Safety). The materials listed in this table are those chosen as typical materials.

Excitation modes may include impact, friction, electrostatic or other forms of thermal energy. In each of these types of stimulus, the sensitivity of the pyrotechnic mix must reasonably be defined to that particular type of stimulus and the conditions leading to activation of the mix by that stimulus known. Only in this manner can the "driving force" be reasonably defined.

Several methods for the detection of incipient hazards were investigated. Each of these relies on occurences that may lead to initiation of the pyrotechnic mixes. Impact and friction would both result in heating of the mix and sound changes in the operating equipment. Electrostatic charge production would produce changes in the electric

Figure 1-1. Lancaster Mixer - 4000 Grams Capacity

Figure 1-2. Stokes Granulator

Table 1-1. Formulations and Sensitivities of Pyrotechnic Compositions Used in Program

Composition No.	Formulation
SI-193, Igniter	Boron - 25% KNO_3 - 75% VAAR - 1% (added)
PFP-555, Photoflash Powder	Al - 40% $BaNO_3$ - 30% $KClO_4$ - 30%
FY-1451, Illumination Composition	Mg - 46% $NaNO_3$ - 45% Laminac - 9%
FY-306, Decoy Composition	Mg - 54% Teflon - 46% Nitrocellulose - 2.6% (added)
DP-973 Delay Composition	Boron - 10% $BaCrO_4$ - 90% VAAR - 1% (added)

Sensitivity Test	Flare Yellow 1451 / Flare White 306	Igniter 193	Delay Powder 973	Photoflash Powder 555
Friction (Fiber Shoe)	0	0	0	0
(Steel Shoe)	0	X	Sparks	Snaps
Impact* (Inches)	17-19	10	24	40
Electrostatic Discharge** (Joules)	11.02	0.124	.029	2.14
Auto Ignition Temp. °C	510	602	560	856
Pads	104	298	349	560

NOTES:

X - Reacts

O - No reaction

* - Picatinny Arsenal Drop Test

** - BuMines Apparatus

fields surrounding the mix. The discharge itself would result in electromagnetic disturbance, heat, possibly sound and motion of the mix. Heating of the mix in any manner would increase the production of infrared radiation from the batch being processed. Initiation of any of the mixes would result in the production of light, flame, IR, and ions.

Interests were focused on the *early detection* of any stimulus that could bring about initiation of the mixes in pyrotechnic processing. For this reason, and because equipment **was available,** the ion mobility measurements were given some priority. The rationale was that molecules would be emitted early in the stages of temperature rise before initiation of the mix actually took place. Questions to be answered were many and we undertook answering these in order of their importance. The questions are:

1. Do the typical pyrotechnic mixes issue ions when heated below the initiation temperature?
2. What constituents of the mix contribute ions?
3. How much of the mix must be heated to detect ions?
4. In what volume of air (room size) may airborne ions be detected?
5. What are false alarm possibilities and consequences?
6. What approximate early warning time frames might be expected?

Similar questions apply to sound and IR and to any other method considered for detection.

Ions and IR measure the results of thermal activation of the mix irrespective of the activation source: friction, impact, or external heating. Sound, if applicable to this problem, could actually measure the driving force that may cause heating: impact or friction in the mix or in machinery operating on the mix.

Electrostatic sources were also given some consideration mainly in measurements of the resistivity of the mixes and in their ability to generate charge.

Initially relative sensitivity measurements were made on the pyrotechnic mixes to compare them with each other and with some primary explosives previously tested.

Brief examination was made of possible extinguishing systems including deluge systems, their response, activation times and general results. No experimental work was carried out on extinguishing systems, but information was gathered and weighed for potential use of extinguishment.

2. HAZARDS ANALYSIS

2.1 GENERAL

Inspection of the pyrotechnic processing plant at Picatinny Arsenal leaves little doubt that safety has been considered carefully in layout, design and equipment location. One wonders what possible condition could cause problems in a plant so constructed and maintained.

Processing itself causes work to be done on pyrotechnic materials and all sources of energy that could potentially cause problems in processing must be considered in order to arrive at detection levels that are meaningful. For this reason we observed the equipment in operation without pyrotechnics present and with simulated pyrotechnic materials present in the operations. In addition, we asked for and received information from equipment manufacturers for most of the equipment described earlier. The question that must be addressed in order to arrive at suitable detection methods is "What are the causes of pyrotechnic accidents?"

Initiation of pyrotechnic materials basically results from heat generated within the composition; this heat may result from fire, impact, electrostatic discharge, friction or electric current.

Some hypothetical accidents would be difficult to foresee either by detection means or by planning. Sudden mechanical equipment failure, lightning strikes and heavy falling objects are possible examples.

Most accidents, we believe, do not happen suddenly; but rather conditions leading to the accident build up to the point where an accident does happen. Normally the materials being processed are subject to friction, impact, compression and indirectly, the generation of electrostatic charges.

One objective of this study is to determine what levels of these stimuli could be troublesome. Once these levels have been determined, there is a basis for selecting a stimulus that will not be to the point where ignition is imminent. The detection level for any warning device must be well below this threshold and well above the normal operating range of the equipment. Many of the hazardous levels have been determined for the typical pyrotechnic materials being considered in this project. These are recorded in Table 1-1.

2.2 FRICTION

Friction tests are made using a 20-kilogram weight on the end of a pendulum. The surface of this weight is faced with a shoe of either fiber or steel. Initially the weight is raised one meter above an anvil containing grooves into which the pyrotechnic sample is placed. The pendulum is allowed to swing past the anvil so that the shoe is in frictional contact with the anvil. The results of this test on the five typical pyrotechnic materials (shown in Table 1-1) indicate that no material tested is sensitive to friction from the fiber shoe; however, igniter, delay and photoflash mixes do react to the steel shoe as indicated in the table.

In addition to the results of the friction pendulum tests, several quantitative tests were made on frictional sensitivity [1] . Threshold initiation levels were determined for SI 193 as 7×10^8 newtons per square meter over a range of velocities from about 0.1 to 2.4 meters per second. Sensitivity was increasing with increasing velocity.

In general, the results of the referenced friction study showed that frictional pressures around 10^8 Newtons per square meter should be detected in order to ascertain a hazard below the threshold level for most pyrotechnic materials being processed. Frictional pressures around 10^9 newtons per square meter will undoubtedly initiate many of the pyrotechnic materials under consideration in this study.

Apparently a good level for detection in practice is 10^7 to 10^8 newtons per square meter at surface velocities from 0.5 to 2 meters per second.

2.3 IMPACT

The impact sensitivities of the pyrotechnic compositions considered in this study range from 10 to 40 inches. These sensitivities were determined using the Picatinny Arsenal impact machine which uses a drop weight of two kilograms. Thus the energy on impact for the more sensitive of the pyrotechnic materials is 20 inch kilograms or 44.1 inch-lbs (Table 1-1).

The sensitivity reported here represents one reaction in ten drops from the indicated height. Impact is applied to a fluted striker assembly which works against an anvil holding the explosive sample. The area of the striker and supporting anvil is small so that the impact energy acts upon a relatively small area.

2.4 ELECTROSTATICS

2.4.1 General

The electrostatic sensitivity of the typical pyrotechnic materials, according to the Bureau of Mines Test Apparatus, ranges from 29 millijoules for DP 973 to 11.02 joules for the Flare Yellow 1451 and Flare White 306 mixes. Nearly three orders of magnitude are represented in this span (Table 1-1).

The problem of static electricity with pyrotechnic materials rests mainly in the generation of charge, ability to retain that charge, and in enough capacitance to store and deliver charge quickly through the mix. Sensitivity of the pyrotechnic mix to activation by discharge is also important.

How and where to sense a charge is the main question to be answered if charge generation is a problem.

Currently the atmosphere in processing areas is maintained at a humidity of at least 60%.

2.4.2 Charge Generation by Pyrotechnic Materials

Charge generation by electrostatic means requires a contact between dissimilar materials followed by separation of these materials. In all but drying oven operation, the pyrotechnic materials are so treated. In order to test the charge producing capabilities of the materials, they were allowed to flow down an aluminum channel which is part of the apparatus shown in Figure 2-1. A hopper located at the upper right is used to store the powder. Upon command the powder is released and allowed to flow down the aluminum channel and into the foil-lined container held in a laboratory beaker which is electrically insulated from the ground plane. An electrostatic voltmeter is connected from the receiver foil to the ground plane.

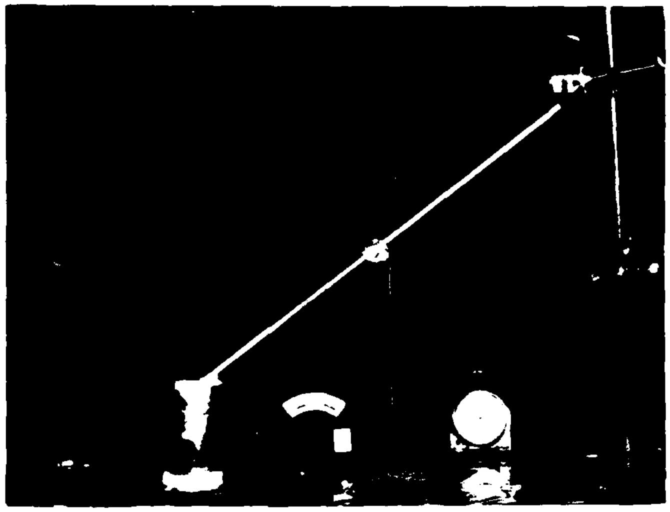
Several tests were made using this apparatus.

All five materials were examined for their ability to generate electrostatic charge by sliding 15-gram samples down a 36-inch-long aluminum channel (one-inch web) inclined at 35° to the horizontal. A conical receiver was made of aluminum foil and then mounted in a glass beaker. Results are shown in Table 2-1.

With 60% relative humidity none of the materials generated a measurable electrostatic charge. Sand was used as a reference material because it was found to be a good electrostatic generator when separated from the aluminum channel.

After 3 hours of conditioning at 28% relative humidity, selected as a relatively low value of humidity, the 15-gram sand sample generated 2700 volts on the receiver assembly; FW-306,450 volts, and FY-1451, 525 volts. None of the other three pyrotechnic materials showed evidence of charge generation under the conditions described.

After conditioning for 65 hours, additional tests were made with results about the same as those for three hours. FY-1451 showed a slight increase in charge production.



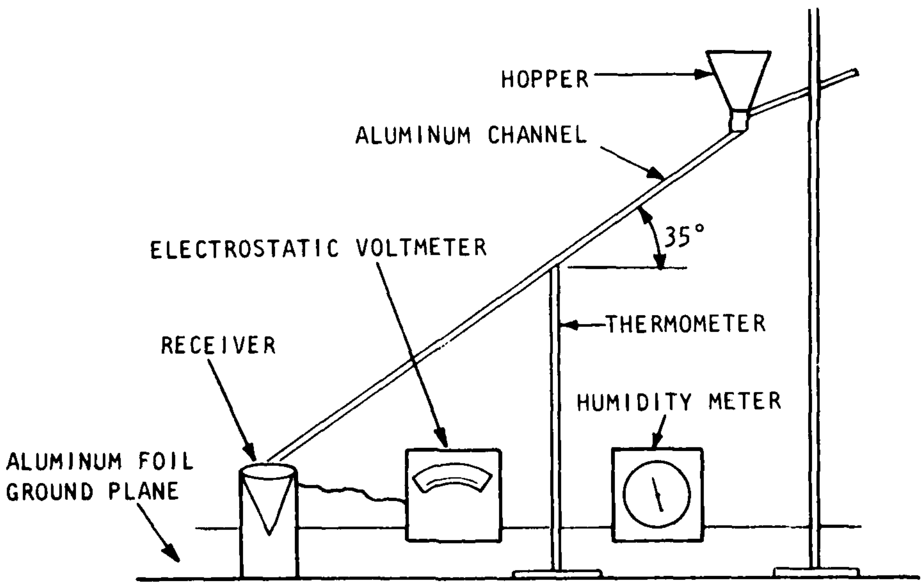


Figure 2-1. Apparatus for Determining Electrostatic Charge Production in Pyrotechnic Powders

Table 2-1. Dynamic Electrostatic Charge Build-up on Pyrotechnic Materials

		VOLTAGE ON RECEIVER (VOLTS)	
		28% RH	28% RH
Material	60% RH	3 Hrs. Conditioning	65 Hrs. Conditioning
SI 193	0	0	0
PFP 555	0	0	0
FW-306	0	450	425
FY-1451	0	525	700
DP 973	0	0	0
SAND	0	2700	2700

Conditions:

Load Capacitance = Approx. 15 Picofarads
Receiver Capacitance= Approx. 10 Picofarads
Meter Capacitance = Low Range - 15 Picofarads
High Range - 10 Picofarads

Note: High range used on voltaqes over 1000 volts.

The generated energies in the worst cases were approximately 31.6 ergs (3.16 microjoules) for FW 306 and 85.8 ergs (8.58 microjoules) for FY 1451. Each sample was at 28% relative humidity and had been conditioned for 65 hours at that humidity. Sample weight was 15 grams.

2.4.3 Resistivity Measurements

A contributing factor to the safety aspects of manipulating powdered materials rests in the materials resistivity. Limits have been established on resistivity ranges that are indicative of the relative safety of using such powders [2]. These limits are given in Table 2-2.

Table 2-2. Rule-of-Thumb Electrostatic Safety Rating for Powders in Terms of Resistivity

Resistivity (ohm-cm)	Anti-Static Rating
greater than 10^{13}	Zero
10^{12} to 10^{13}	Poor
10^{11} to 10^{12}	Moderate
less than 10^{10}	Good

Resistivity measurements were started on the pyrotechnic materials in order to make assessment of the potential for static electricity build-up with these materials. A teflon cell with copper electrodes of known area was used to house the pyrotechnics. Spacing was measured using the difference between the closed electrodes (without the pyrotechnic materials present) and sample-filled holders. Resistance measurements were made with a Kiethley Milliohmeter.

Table 2-3 shows the results of these tests. The magnitude of resistivity indicates that a marginal condition for charge build-up exists in as far as safety is concerned.

Table 2-3. Resistivity Measurements of Sample Pyrotechnic Materials

Material	Resistivity (ohm-cm)
DP 973	1×10^{13}
SI 193	1.8×10^{10}
PFP 555	9.65×10^{9}
FW 306	1.5×10^{13}
FY 1415	2×10^{13}

More studies were made of the electrical properties of the five pyrotechnic materials to be used as examples in this program. The results are shown in Table 2-4 for the materials in a loose state and in a compressed state. If the results of Table 2-3 are compared with the results in Table 2-4 it becomes clear that the measured value of resistivity has increased up to three orders of magnitude. The difference can be attributed to the humidity conditions prevalent during the measurements. The lower values of resistivity were obtained with the humidity at approximately 40%. The higher values of resistivity were obtained with the humidity at about 20%. This humidity change is one that crosses some important safety barriers. In light of the rule-of-thumb guidelines advanced in Table 2-2 the resistivity has changed from good to fairly good static resistance to poor-to-zero static resistance in every case.

Table 2-4. Electrical Properties of Typical Pyrotechnic Materials

Material	Resistivity (ohm-cm) Loose	Resistivity (ohm-cm) Compressed	*Electrostatic Sensitivity (joules)
DP 973	1.01×10^{14}	8.5×10^{13}	0.029
SI 193	3.42×10^{13}	9.56×10^{12}	0.124
PFP 555	4.80×10^{13}	2.09×10^{12}	2.14
FW 306	1.46×10^{14}	1.21×10^{13}	>11
FY 1451	2.65×10^{14}	1.29×10^{14}	>11

Humidity was about 20%

*From Picatinny Arsenal PADS

These measurements confirm the well-known belief that relative humidity is an extremely important parameter to be monitored and controlled. Resistivity as a function of humidity should be determined for the materials being processed. Humidity levels should then be monitored in the areas that pyrotechnics are being processed with alarms being set at levels where the resistivity changes from, say 10^9 or 10^{10} to 10^{12} ohm cm occur, if indeed this transition ever occurs. This procedure would reduce the buildup of electrostatic energy in the materials being processed.

Humans have a capacitance of about 500 picofarads (maximum) with some safety factor applied. The voltage to which a human may normally be charged without any special precautions as to clothing and insulation is approximately 20,000 volts. The energy stored on a human under these nominally maximum conditions is 100 millijoules. The third column in Table 2-4 shows the sensitivity of the sample pyrotechnic materials to electrostatic discharge. From the charged human under these conditions only the delay powder, DP 973, and the igniter mix, SI 193, would appear to be subject to initiation. Where these materials are being processed operators and personnel in the vicinity of the materials should be monitored for the presence of electrostatic charge. Entry to the working area could be a convenient place for a sensor to check the electrostatic condition of personnel entering certain areas. One machine checks the footwear for conductivity from foot to foot. Other commercial equipment monitors the electric field surrounding the person without physical contact to the individual.

2.5 THERMAL

2.5.1 General

The five typical pyrotechnic materials have been tested for autoignition temperature. The term autoignition is normally used interchangeably with cook-off. Tests to indicate and determine this temperature are run in an oven with the pyrotechnic sample contained in the oven and with a thermocouple imbedded in the sample [3]. Oven temperature is increased very slowly until the material under test begins to supply heat to the oven. At this point some automatic control working on sample and oven temperatures regulates the oven temperature to prevent heat exchange from oven to sample. Eventually the sample reacts rapidly at the autoignition temperature for that material.

The lowest autoignition temperature for the five pyrotechnic materials tested was 510°C, this being for ignition mix SI 193. Implications from these data are that a temperature much lower than this must be used for detection of potential thermal problems.

2.5.2 Tests on Small Quantities of Pyrotechnic Materials at Low Level Excitation

Experiments were conducted to determine the response of small quantities of pyrotechnic materials to low level electrical excitation. Test fixtures were constructed as follows: An Atlas match-comb bridge, mounted on a metal-laminated holder, was placed in a smaller-diameter piece of Tygon tubing. The pyrotechnic materials were loaded into the open cup formed by the tubing. A thin plastic cap was glued to the open end of the assembly.

To assess the performance of these devices and to note the reaction of each of the pyrotechnic materials, each of the first five devices made was exposed to a relatively high stimulus constant current pulse of 5 amperes magnitude, three seconds duration. This amplitude and time were chosen only to be large enough to assure firing. Results are given in Table 2-5.

Table 2-5. Observed Responses of Typical Pyrotechnic Materials

Material Identity	Observations
SI 193	Pop - Very bright flash - Little noise or residue
PFP 555	Loud pop - Some light - Little residue
FW 306	Loud crack - Started fire in tubing
DP 973	Sparks - Deflagration - Little noise - Started fire in tubing
FY 1451	Did not ignite - Bridge wire burned out - Did not ignite on second trial

Enough of the bridge wire-pyrotechnic devices were manufactured to establish a firing distribution with respect to applied current. The exception is FY 1451, which could not be functioned by this method.

After loading and testing of hot bridge wire devices was completed, Bruceton tests were made on four of the five typical pyrotechnic materials, with the results indicated in Table 2-6.

Devices made with the second flare material (FY 1451) were not able to be fired at first. Subsequently we packed this material more tightly into the bridgewire assembly. Some of these devices did fire under this condition at about 5 amperes. This current level is much higher than for the other four materials. Furthermore, the behavior of these items was such that the bridgewire opened without setting off the pyrotechnic mix in a number of trials. We reason that the size of the particles in this mix, the largest of the five mixes tested, prohibited good contact between the mix and the bridgewire.

A supply of the hot wire devices loaded with pyrotechnic mixes was retained for experimental use in detector evaluation.

Table 2-6. Functioning Currents for Hot-Wire Excited Pyrotechnics

Pyrotechnical Material	Current (Amp) for Functioning Probability (%) of							Confidence (%)
	0.1	1	10	50	90	99	99.9	
DP 973	1.346 1.338	1.371 1.365	1.407 1.403	1.454	1.503 1.507	1.542 1.549	1.571 1.580	90 95
SI 193	1.045 1.009	1.133 1.103	1.265 1.246	1.454	1.671 1.696	1.866 1.916	2.022 2.094	90 95
PFP 555	2.048 2.023	2.120 2.100	2.222 2.209	2.360	2.506 2.521	2.627 2.652	2.719 2.753	90 95
FW 306*	2.484 2.438	2.606 2.569	2.785 2.762	3.030	3.296 3.324	3.522 3.572	3.696 3.766	90 95

*The bridgewire used for FW 306 was not the same as used for the other materials. Comparisons cannot be made on the basis of similarity.

Pulse Application time was 3 seconds.

The pyrotechnic materials are listed in the order of decreasing sensitivity. The mean firing currents increase going from top to bottom on the table. Flare yellow mix, FY 1451, could not be tested Bruceton fashion, but it appears to be the least sensitive of the pyrotechnic mixes to the stimulus. Stimulus was from a fine bridgewire in each case.

The main purpose of these tests was to determine the amplitude of the current pulse of 3 seconds duration that would yield a certain functioning probability. Three seconds is a relatively long pulse duration compared to thermal time constants of most wire bridge initiation. Knowing these values, items could be excited at various levels below the point at which functioning occurred. In this manner, some data could presumably be taken on detector responses under excitation of this type, i.e., from a very small thermal source.

In general, and with the exception of FY 1451, distribution of firing current for the pyrotechnic mixes was fairly tight. This narrow distribution indicates that the entire system, including the pyrotechnic mix, bridgewire and exposure system, are well controlled.

3. DETECTION METHODS

3.1 GENERAL

The stimulus levels from various forms of excitation have been defined within reasonable limits. The problem at this point is to establish that the temperatures of the pyrotechnic mixes are approaching the 510°C autoignition temperature, that frictional levels are in the vicinity of 10^7 to 10^8 newtons per square meter, that impacts are nearing 41 inch lbs, or that static electric levels of less than 29 millijoules are present. These are essentially the challenges that are to be faced in arriving at detectors that are feasible for use in pyrotechnic batch processing.

The approach to finding detectors that could effectively monitor the aforementioned stimuli was to examine existing literature on fire, ion or smoke detection apparatus. The applicable devices and methods in the literature were practically nonexistent. Reasoning was that the mixes themselves would probably produce some form of gas or vapor as they were heated to a point well below the ignition temperature. The frictional and impact stimuli will undoubtedly produce sounds that could exceed the masking sounds from the equipment. Only experiment could answer these questions reasonably.

Efforts were concentrated on the detection process. The actions to be taken in the event of signal or alarm where examined only briefly. The detection methods and approaches are discussed further in this section of the report.

3.2 ION MOBILITY SPECTROMETER (IMS)

3.2.1 Operation of IMS

Ion mobility spectrometry involves the simple process of ionizing molecules by a beta source, moving the ions through an electric field,

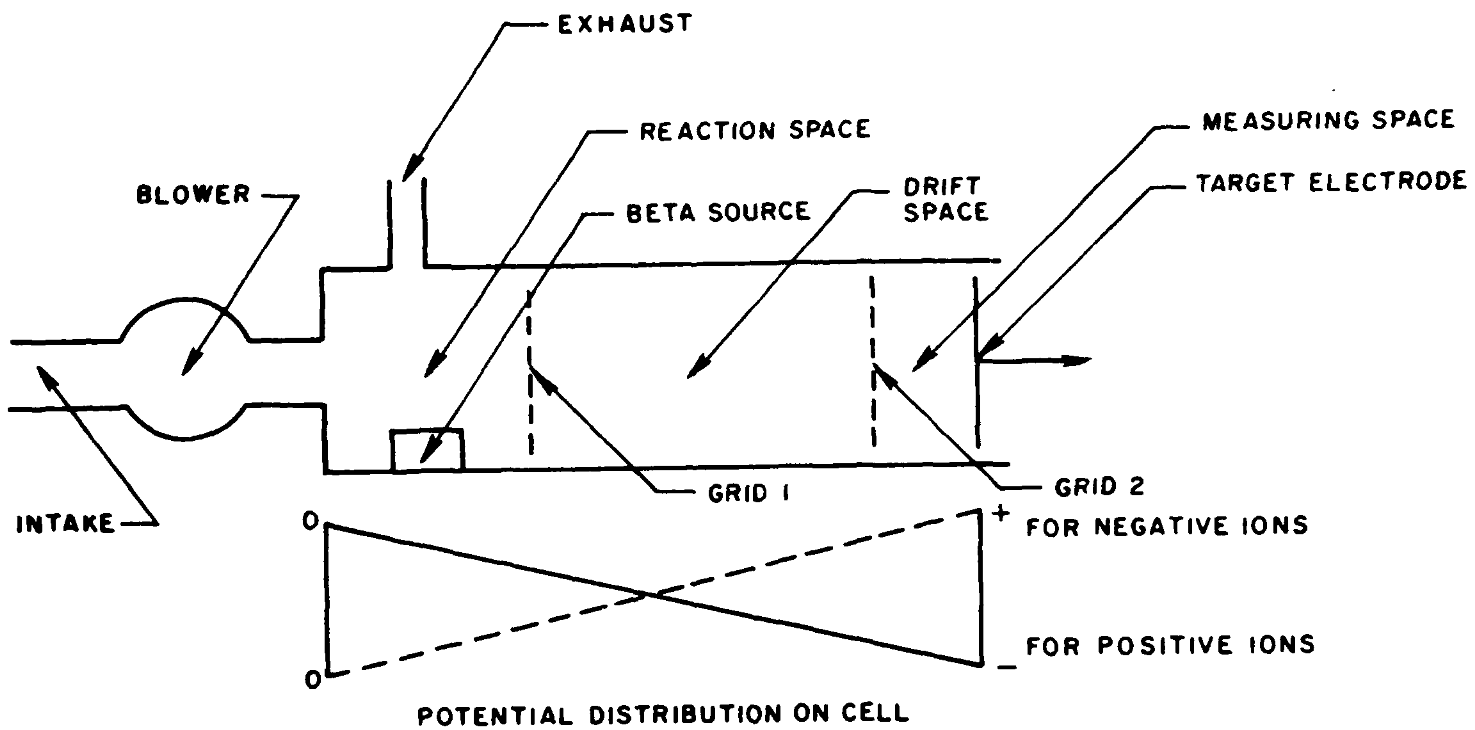


Figure 3-1. Basic Structure of Cell for Ion Separation and Presentation

and determining their velocities as they hit a collecting target. The signal generated by the impinging ions is depicted on an oscilloscope. The pattern generated gives a detailed "fingerprint" of the masses (in reality, e/m) of all the species in the IMS detection chamber.

Operation of the IMS head is as follows: The air sample is taken through the intake by pressure created by the blower. Excess air is exhausted and a fresh sample in the reaction space is maintained by the flow through this space. The air sample may contain molecules in vapors from the sample space. A beta source is contained in the reaction space so that the molecules of the sample are ionized. These ions are moved toward the target electrode by virtue of the electric field maintained along the cell axis. Grids 1 and 2 are designed to gate ions in such a way that they begin traverse of the measuring space together. Arrival time at the target electrode is different for each ion type.

When start-time is taken to be the "open" pulse on grid 2, and an oscilloscope is triggered on this pulse, the current time picture on the oscilloscope reveals a signature containing all of the ions in the drift space. There is time separation of the resultant current stream. The current contribution from an ion type is therefore present for discrete time intervals. This phenomenon allows detection by a timed-gate pulse technique.

3.2.2 Tests on Pyrotechnic Materials and Binders

An IMS unit was available to make measurements on samples under conditions of our choice. Our first interests were to determine if such materials chosen as typical pyrotechnic materials (listed in Table 1-1) would give a signature on the IMS and at what temperature.

Experiments were conducted at first with a Mettler furnace using very small samples of the pyrotechnic. Detection by this technique was marginal because, we supposed, of the very small quantity of material. There were signatures of a type, but with very short time of exposure, too short a time to view on an oscilloscope carefully or to photograph the resultant trace.

For this reason a larger "oven" was made using a small test tube wrapped with nichrome wire as shown in the apparatus of Figure 3-2. This oven was calibrated for temperature in terms of the voltage applied to the heater wire.

Test tubes containing the pyrotechnic samples were placed inside the heater coil and the voltage on the coil set to give the temperature desired. The IMS intake was placed 2 inches above the mouth of the test tube. The temperature was increased until such time that a signature was observed on the oscilloscope connected to the IMS system.

Immediate results were obtained using this system. Results were recorded from the face of the oscilloscope using Polaroid film. The temperature at which first positive detection was achieved is indicated in Table 3-1 for the five typical pyrotechnic materials. Typical signatures for the materials are shown in Appendix A.

A clear indication is present where detection temperatures are shown either by a change in the air spectrum or by new peaks in the display.

PFP 555 did not give an indication, probably because there is no binder and because the components are not reactive at the temperature limits of these tests.

Generally the positive ion was detected at a lower temperature than the negative ion, the exception being VAAR.

3.2.3 Tag Materials

Early in the reasoning process tag materials were considered a possible solution to the problem of providing early warning that overheating was occurring in pyrotechnic processing. The experiments conducted with the five pyrotechnic materials, while successful in all but one instance, demonstrated that a universal tag would afford some advantage. Finding a suitable tag material would probably allow a choice of detection temperature; a uniform signature more amenable to a single, sensitive detector design; a greater efflux of ions and a simpler signaling and warning system.

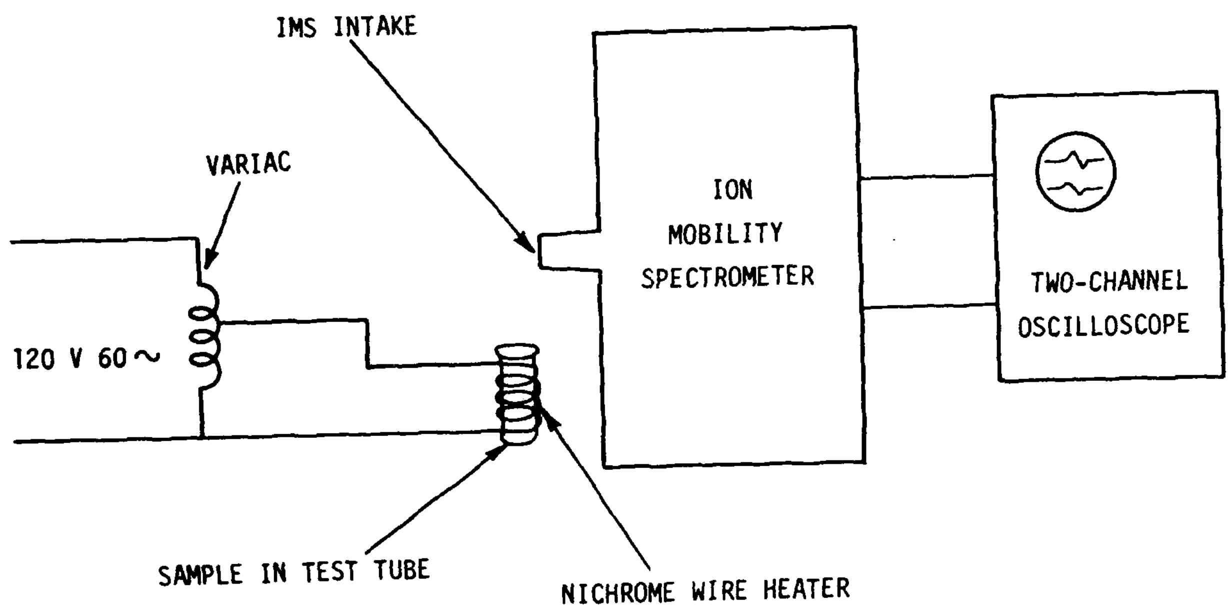


Figure 3-2. Apparatus for Obtaining IMS Signatures of Pyrotechnic Samples and Tag Materials

Table 3-1. Ion Mobility Detection Temperatures of Untreated Pyrotechnic Mixes, Laminac and VAAR

Material	Auto Ignition Temp °C	Detection Temperature (°C)	
		Positive Ion	Negative Ion
DP 973	560	58	268
SI 193	602	87	125
PFP 555	856	None	None
FW 306	510	80	155
FY 1451	510	120	240
Laminac	—	100	210
VAAR	—	195	178

For these reasons, we set about the task of examining potential tag materials. To aid in this effort, some information on IMS signatures was available along with experience in the chemical and physical nature of tag materials [4].

A review of chemical literature related to IMS, including the library referenced earlier, resulted in the choice of a number of tag materials for test and further examination. These are listed in Table 3-2.

The quantities of tag materials anticipated to be needed would be relatively small. The tag materials would be thoroughly mixed with the pyrotechnic raw materials. One point to be made here is that little is known of the compatibility of the potential tag materials with the pyrotechnics. This problem must be resolved prior to their use with pyrotechnic materials.

These materials were evaluated in terms of IMS signature.

3.2.4 Volume-Time Tests with Succinic Anhydride Tag

The IMS apparatus was programmed to detect the characteristic lines of succinic anhydride, a tentative tag material, by the normal test-tube technique. The tube containing the sample is heated via an electrically heated wire wrapped around the test tube. Initially, the compound signature is obtained on an oscilloscope and the coding set for response to this compound. Volumetric tests were made under the conditions listed below.

Temperature	Volume (cu ft)	Time to Detector
147°C	4.5	45 seconds
	400	2 minutes

There was no intentional air circulation in these tests other than the intake blower on the IMS unit.

Other findings are that the positive ion response generally results in contamination of the IMS head. The negative ion is not as contaminating. Contamination appears to be an important adverse consideration in the use

Table 3-2. Prospective Tag Materials

1-octadecanol
1-nitrobenzoic acid
cetyl alcohol
3,5-dinitro benzoyl chloride
tetradecanol
*phthalic anhydride
N-(p-hydroxyphenyl) glycine
aniline hydrochloride
diallyl amine
hexadecyl amine
*malonic acid
decanoic acid
*succinic anhydride

Those compounds marked with an asterisk are most sensitive in response.

of the IMS; therefore, the negative ion response appears to be more desirable from this point of view.

3.2.5 Background Test for Contaminants

Air samples were collected in the pyrotechnic storage area at Picatinny Arsenal as well as at various locations in the processing buildings to ascertain the type of background that could be expected using the IMS. The collected samples were brought to the IMS at The Franklin Institute and these were checked for spectral response. The response was minimal. Indications are that the IMS will operate under the conditions encountered. Processing was not being carried out at the time of the tests.

3.3 SOUND

3.3.1 General

Impact and friction both result in localized heating sources that could achieve high temperatures in relatively small areas of the pyrotechnic mix. Frictional heating could conceivably be dispersed by virtue of the motion of the pyrotechnic mix in the blender and eventual detection by a "sniffer" technique similar to the IMS. Impact may occur a number of times with various intensity levels less than the critical value. This kind of an event would probably result in intense local heating without much opportunity for the relatively small total quantity of heat to be spread through the mix. It appears that impact would not cause enough molecular output to cause tripping of IMS detection. For this reason impact in particular is a good prospect for detection by means of sound; frictional sounds may also be detected readily.

3.3.2 Methods and Approach

Sound was originally considered as a detection means because operators have expressed their experiences with processing machinery. Strange sounds have been observed prior to a breakdown either with or without action on the part of the operator. Prompt reaction has resulted in preserving equipment, avoiding costly repairs and preventing injury.

Processing pyrotechnics is different from most processing procedures in that the material being processed is potentially dangerous. A more vigilant observation is necessary, beyond the normal ability of humans to hear and observe. Equipment for sound reception, amplification and treatment can possibly be designed to be more discerning. Details for the equipment, methods and plans are contained in Appendix B. The objective at this point in the program was to experiment with operating machinery and demonstrate the feasibility of using sound equipment with proper sensitivity and fidelity to detect sounds due to friction and impact.

3.3.3 Experiments and Results

Arrangements were made to observe operating processing equipment at the Picatinny Arsenal batch processing plant. Appendix B gives test conditions and shows the results of spectral analyses of tape recordings made on the processing equipment.

This procedure was used so that the sounds generated in the processing equipment during normal operation could be compared with faults intentionally placed in the equipment. What was found is typified by the rendition of the spectral output of the 4000-gram mixer (see Figure 3-3).

In normal operation the sound spectrum is confined to peak amplitude around 1 KHz with a range from 500 Hz to 1.5 KHz. An impact on the mixer bowl with an automatic center punch (much below the critical 41 inch lbs) resulted in a peak rising at 3 KHz. While this reading was obtained via a tape recording, a real-time detection of this type of phenomenon is feasible and apparently practicable.

In addition to impact, definite observations were made on scraping machine parts, and on a jammed roller. These signals were discernible with the equipment operating.

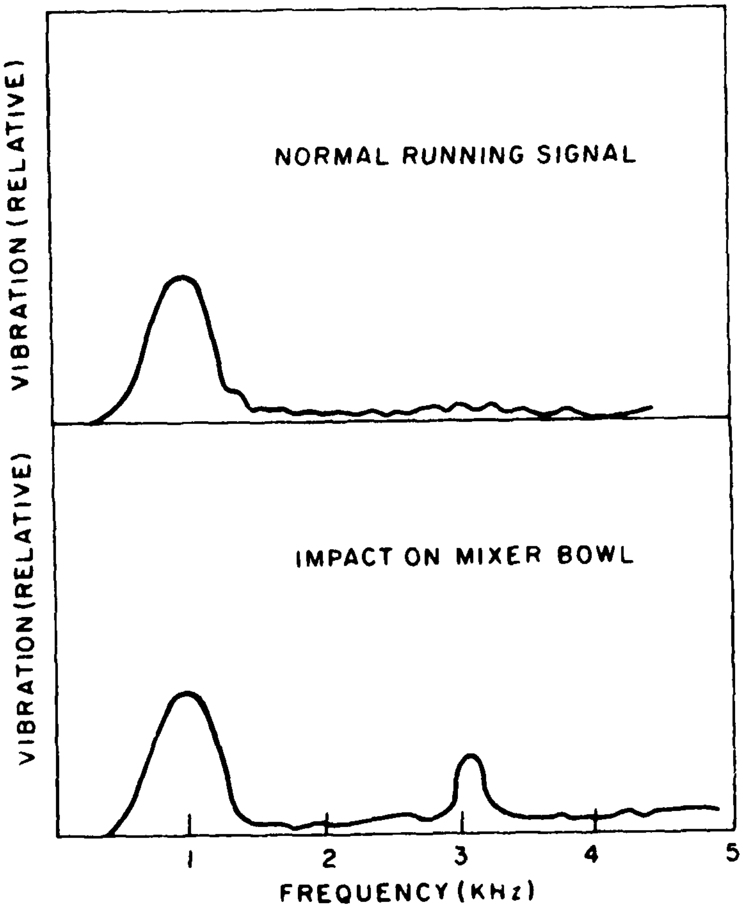


Figure 3-3. Typical Sound Spectrum Outputs from Pyrotechnic Machinery-(4000-gram mixer)

3.4 INFRARED (IR) AND HEAT

3.4.1 General

Of obvious interest in the detection of potential problems, IR and other heat detectors afford some advantages. Two basic approaches were considered as a potential solution to this problem. One was the sensing of IR radiation from the surface of the material being processed and the other temperature measurement in the materials being processed.

3.4.2 Infrared

We examined literature on the IR equipment available. Some would be suitable for the purposes of monitoring surface temperature; however, the cost, we believe, was too high to be considered for that purpose.

Methods considered were a video-type of scanner that would cover an entire area, "remembering" the thermal picture of the scanned area through the use of stored information. Subsequent scans would be compared on a variable time basis with the stored information. Difference and rate of change would be examined for use as alarm information. This method would require installation in each room and would be effective but extremely expensive.

Cost estimates on such a system would be based on one IR scanner of the video type for each of the processing locations. A dual conversion may be potentially the most economical approach to the entire problem. An IR viewer, similar to the night vision devices used by the military, would be an intermediate converter. The screen on the night vision device would be coupled optically to a TV converter tube. The result would be an IR video signal scan of the room or equipment. Component costs per room would be on the order of $2,000 provided present equipment is amenable to the longer wavelengths needed to detect temperatures in the 200°C range. The storage and comparison equipment is estimated to cost approximately $40,000 including the recording equipment.

A Williamson Corporation HS detector system was selected because the price was reasonable, it covered a relatively large field of view, and detected changes in temperature that seemed appropriate for early warning.

Efforts to evaluate the unit failed. A unit was requested for evaluation, and delivery was made several months later. The unit did operate but the optical axis was distorted and we feared other problems would become evident. For this reason the unit was never evaluated to our satisfaction. The performance that was witnessed allowed differentiation of small, warm to hot bodies on a room-temperature background. A good feature of the equipment was a background integration, which apparently accepted very slow changes in temperature without an alarm. Sudden changes did cause an alarm.

3.4.3 Thermocouples

Fast-response thermocouples (in the millisecond range) were considered for installation in contacting, non-rotating elements of processing equipment. The Namac thermocouple appears applicable for this purpose.

The thermocouple is essentially a bolt with the thermocouple element on the face of the bolt. The surface is renewable and highly responsive. The one major and defeating problem is that the thermocouple must be in contact with the critical pyrotechnic, i.e., the pyrotechnic material that is being elevated in temperature. A large number of contacting thermocouples would be needed to sense temperatures at enough locations to assure reading elevated temperatures.

3.5 OTHER METHODS INVESTIGATED

3.5.1 Literature Sources

A number of potential sources for information on detection were reviewed including specialized literature such as the Proceedings of the Annual Seminars of the Armed Forces Explosive Safety Board, National Fire Protection Association publications and equipment descriptions on available protection systems. There was much of value in all of these sources, but none seemed directly applicable to the problem of detecting the kinds of stimulus of direct interest to this program with the possible exception of ion and flame detectors and an occasional reference to pyrotechnic fires and equipment. Commercial literature was reviewed searching for specific equipment that was potentially useful for detection. The most applicable of the related literature is referenced by manufacturer in Appendix C.

3.5.2 Quenching

Quenching pyrotechnic fires is a subject of great controversy. Water deluge systems are commonly used in explosive operations where water is effective against the type of fire, e.g., in propellant plants where operations are in line and where fires can be localized by the rapid application of large quantities of water. In batch operations with pyrotechnics the feeling is that water may create additional problems. The main concern is that many of the mixes would generate hydrogen in the presence of water and heat. Hydrogen in the area of a fire would be subject to explosion and further damage to the site and possibly injury to personnel.

Burning magnesium flares have been extinguished by the use of water [6]. These experiments were conducted outside in open air. No significant amount of hydrogen was detected in the vicinity of the fire while water was applied. Both salt and fresh water were the *only* extinguishing materials found to be successful in controlling a burning flare.

The exemplary-pyrotechnic materials are *not* physically the same as the consolidated flares. Reaction rates on these materials were found to be relatively high as evidenced by experiments done here using electric matches loaded with small quantities of the typical pyrotechnic materials (See Section 2.5.2).

Present recommendations deal mainly with the use of deluge systems for areas using explosive chemicals including TNT, gun propellants and similar materials. For these materials nozzles passing 50 gallons per minute or more at a pressure of 40 psi or greater are recommended. Using this same method of application to pyrotechnics cannot be clearly recommended at this time. There is insufficient evidence to produce a clearly beneficial effect from the installation of such a system.

Specific and useful references from this grouping of literature are few. Most detectors are of the type that function upon detection of evidence that a fire has begun. Conventional fires pose a threat to pyrotechnic operations. Early detection of conventional fires is important because of the possibility that pyrotechnics could become involved with the occurrence of a spreading conventional fire.

3.5.3 TSG Gas-Sensing Semiconductor

A solid state detector was fully evaluated (5). This detector is manufactured by Figuro Engineering, Osaka Japan. It is small, of low cost and great sensitivity. The principal drawback preventing its application in pyrotechnics is its broad responsiveness to air-borne vapors. For example, it responds to many of the solvents used in pyrotechnic processing such as acetone and alcohol.

The device was evaluated for pyrotechnic applications by exposing it to the output from pyrotechnic "squibs" described earlier. Only when the pyrotechnic actually fired was there an appreciable output from the detector.

At this point it appears that the TSG detector would serve only to indicate that a reaction had occurred or that acetone or alcohol vapors are present. The alternative would be to design and develop a selective process to assure that only certain vapors or gasses get to the TSG detector. This selective process is already available in the IMS detector.

3.5.4 Off-Center Load Indicator for Press Operation

The practice of hydraulic press consolidation apparently does not lend itself well to conventional means of detecting problems. The consolidation procedure was examined on several occasions in order to ascertain possible means of detecting problems. The nature of the operation reduces the effectiveness of the IMS procedure because of the relatively good seal in the volume containing the pyrotechnic. While sound may be a possible means of monitoring operations, the process is transitory and monitoring may require rather special procedures. (These are worth further consideration.)

Picatinny Arsenal personnel suggested that off-center loads could conceivably cause a failure of the die and subsequent broaching of die components in the consolidation area.

A potential solution to this problem rests in an instrumented die base. A four-quadrant base would probably suffice. Each quadrant would provide a signal indicative of the load. As long as the load was uniform on all quadrants, nearly identical signals would be provided. These would be combined for essentially no output unless an unbalance in one or more of the quadrant loads was experienced, at which time an alarm would be provided or automatic shut-down of the press would be initiated.

4. CONCLUSIONS

4.1 DETECTION

With the techniques studied,by far the best system uncovered is believed to be a combined ion mobility and sound system that will provide early warning of potential problems. The ion mobility spectrometer as stated is selective to certain specific molecules. These molecules exist in greater numbers as temperature is reached. The pyrotechnic mixes themselves evolve ions or molecules that can be detected long before ignition temperature is reached; however, each mix may have different molecules present, or as in the case of PFP 555, none that are readily detectable. Tag chemicals consistent in the yielding of readily detectable molecules make possible single-purpose detectors that are keyed to temperature evaluation in the mixes during processing. Thus, direct detection of the pyrotechnic mixes overheating could generally be achieved using the IMS procedure. Each pyrotechnic material would need to be examined for emissions, and multipurpose detectors made for each of the materials. In the event that one material lacked an output that could be detected, it would be necessary to tag that material.

This process appears applicable whenever an open process is being carried out. Mixers, pelletizers, ovens and possible blenders and presses are operations that can be monitored by sensing molecules from the mixes themselves.

Sound transducers mounted on operating equipment offer early warning of mechanical problems. Process difficulties like impact and friction produce a different spectrum than normally operating equipment. The sounds generated by improper mixer and pelletizer functioning were readily detected in laboratory examination of sound spectra. Real time processing of sound from operating equipment is technically feasible in mixing and pelletizing operation. Blending is probably another operation that can be monitored by sound with reasonably good results. Consolidation operations are most probably marginal for this kind of monitoring.

A quadrant load base appears a favorable asset in monitoring consolidation by hydraulic press. Off-center loads are detectable by the process.

Electrostatic activation appears remote with current practice because most responsive materials do not generate appreciable charge with ambient humidity above 50 to 60% at room temperature. Those materials which do generate charge are relatively insensitive to static stimulus.

4.2 QUENCHING

Quenching of pyrotechnic reaction in general is still a questionable practice. If a quenching agent is used, water appears to have the most desirable effect; however, this conclusion is based only on tests with flares in the open.

In closed areas hydrogen generation could be a problem. A water deluge may minimize this problem to some degree by limiting the time that hydrogen could be generated.

Another beneficial effect of water quenching would be to limit involvement of adjacent area or personnel in the flame-heat follow-up to the fire event.

Currently available quenching systems apply water at the rate of 50 gallons per minute at pressures of 40psi from each outlet.

5. RECOMMENDATIONS

5.1 GENERAL

To take advantage of the findings of this study, the following courses of action are recommended:

1. Development of simplified IMS detection system specifically for tag materials in pyrotechnic processing.

2. Development of sound monitoring systems for pyrotechnic processing equipment.

3. Definitions of detection limits for these two equipments in terms of local conditions.

4. Installation of prototype units in a processing facility.

5. Study system performance and determining capability of the equipment with pyrotechnic operation.

6. Adjust the monitoring equipment to operate most effectively.

5.2 POTENTIAL EARLY DETECTION METHODS

5.2.1 Ion Mobility

Several promising tags were uncovered that allow a choice of the temperature at which detection could be achieved. It is suggested that these tags be examined for compatibility with pyrotechnic mixes in general and with the five typical mixes in particular.

Minimum quantities of these materials for effective detection should be determined carefully as a function of room size and detector sensitivity. The effects of interfering species, if any, need to be determined so that a reliable and effective system can be designed around specific tag materials. Any contamination effects of the ion mobility detector should also be evaluated.

The next logical step is to develop a detector system which will be effective in applications such as those in pyrotechnic facilities.

Such a system should be designed, constructed, tested and ultimately installed in a processing plant as an interim system. It is suggested that lots of pyrotechnic mixes be injected with the tag material or materials and that these be processed in the normal fashion, monitoring equipment on. No warning event should occur in this operation unless a malfunction occurs. The signal level should be monitored during this operation to observe the below-threshold signal and perhaps to establish the threshold level. Next, after the area has been cleared of pyrotechnics, a small quantity - several grams at most - should be placed in the room. The mix should be placed in an appropriate container and the temperature increased slowly until a clear signal, well above the noise level, is detected. Parameters of interests in the determination are size and weight of pyrotechnic, area in contact with heat producer, amount of tag material in the mix, interface temperature and time to activation.

A two-detector, two-tag approach at this point would offer the advantage of determining heating rate. The rate is important in assessing the degree of the problem. Higher rates usually mean more serious problems.

Many of these parameters will have been determined in laboratory tests prior to the experiment in the pyrotechnic facility.

The data then derived will permit setting up and locating the warning level for the equipment. Plans should include a six-month or more trial installation with the alarm system output being monitored by recording equipment. Periodic activation tests should be made.

5.2.2 Sound or Vibration

The sound approach to early detection of potential problems has been investigated only briefly. Findings so far indicate that impacts, friction and probably other troublesome conditions can be resolved by this approach.

Primary indications are that warning sounds are from resonances in the machine structures affected by friction or impact. These signals stand out upon playback of sound recordings through a spectrum analyzer. Figure 3-3 is a rendition of the appearance of the oscillogram. Normal running of the 4000-gram mixer is indicated by the top trace. Impacts on portion of the mixer bowl resulted in ringing of the mixer bowl at a frequency around 3 KHz, resulting in a pronounced rise in response at that frequency.

The most optimistic view at this point would be that problems are going to show up at specific frequencies in the sound or vibration spectrum from the equipment. Accelerometers may be mounted at one or more locations on the equipment and connected as is shown in Figure 5-1. Their output would be fed to an amplifier (one amplifier per accelerometer). The amplifier output would be fed to several filters designed to pass signals that could represent problems in the equipment. Output signals from the filters would activate an alarm system that could include light or sound warnings or the signal could be used for automatic shut-down of the operation.

5.2.3 Other Means of Detection

Infrared

While no great emphasis was placed on other methods of detecting potential problems, several were considered. IR methods are without doubt useful in monitoring surface temperature on equipment. Detection is possible with several degrees of change (perhaps 10) over an area of a few square inches of pyrotechnic. "Viewing" the material is a potential problem although it appears that mixing and granulating operation could readily be converted to use IR. Consolidation and blending operations present what appears to be an insurmountable problem for IR except on a long time basis. IR could most readily be used to sense the

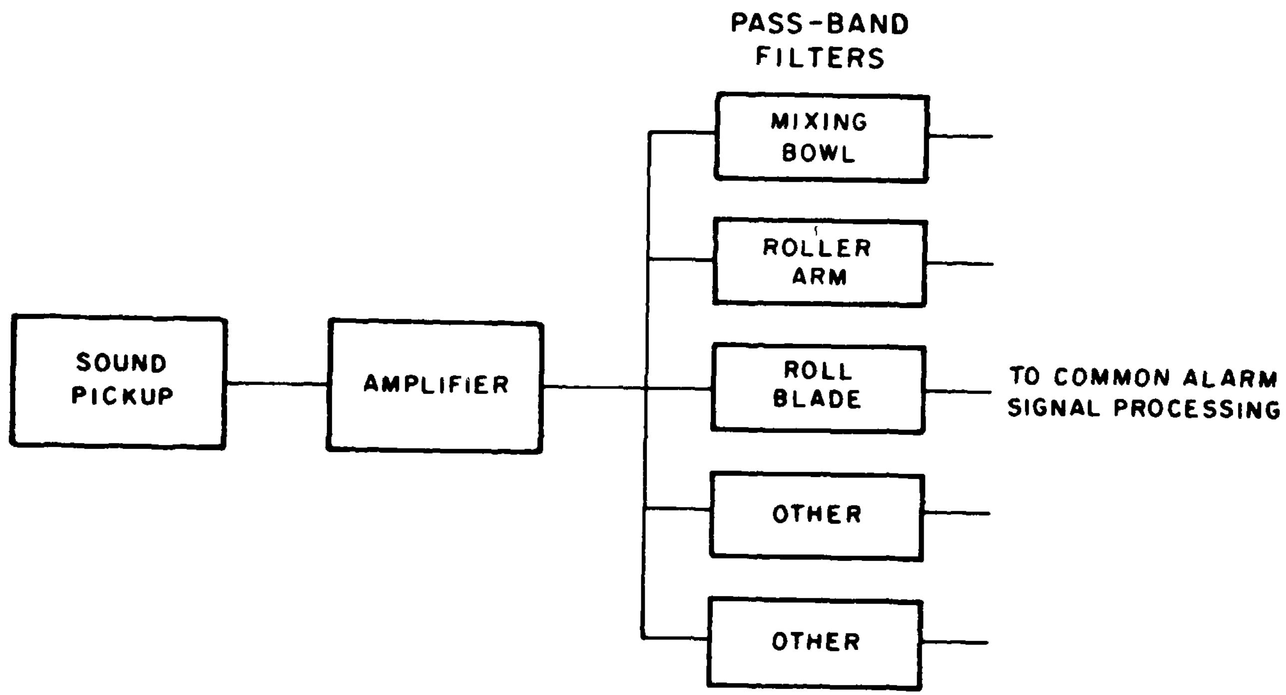


Figure 5-1. Proposed Sound Monitoring System for Pyrotechnic Processing

beginning of burning or overheating of equipment and material in the vicinity of operations. Fast response is one of the potentially good qualities of IR systems and for this reason should be considered for the purpose of activating quenching mechanisms.

Electrostatic Energy

Normal protection procedures, water vapor injection and personnel-read gauges are not reliable enough to preclude static accumulation and sparking. A humidity sensor with warning light and/or sound should be investigated to ensure high humidity levels in processing areas. Such sensors are available and regularly used in home appliances (clothes dryers). A practical, safe, low-humidity warning device should be devised.

Off-Center Indicator

Consolidating operations using hydraulic presses to drive compression die represent a difficult problem for detection. One of the main concerns, however, appears to be created by an uneven load on the die-cylinder system which would cause broaching inside the fixture, accompanied by part failure and heat, possibly followed by ignition of the pyrotechnic.

This problem could be averted by the use of an instrumented die base. A four-quadrant base would probably serve as a function in this effort, each quadrant instrumented with strain gauge or piezoelectric transducers selected on the basis of present signal amplitude indicating the degree of balance on the die. Unbalance above a certain predetermined level would halt load application to the die.

5.2.4 Concept of Installation

The essential elements of a detector installation have already been discussed. At this point the installation concept is considered.

One concept is shown in Figure 5-2, which depicts a three-room installation. Here each of the rooms has one IMS head and one sound transducer. The IMS head would require power and would therefore be shielded and in a dust-resistant enclosure to minimize electrical-explosive

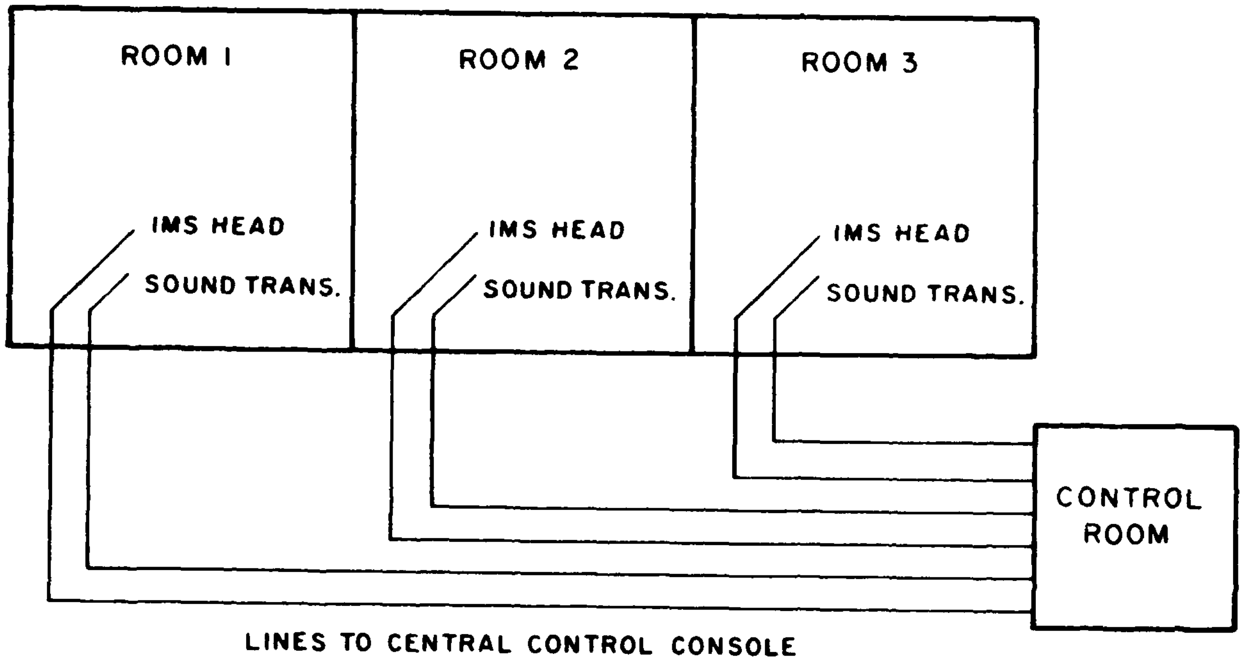


Figure 5-2. Proposed System for Monitoring Pyrotechnic Processing Equipment

interaction. Alternately, a hose-type connection could be used to draw air samples out of the processing room for use in a detector mounted in other than the processing room itself. The detector could be mounted on the outside wall of the room and the sample conveyed to the detector. The longer the air path, the greater the delay in detecting the ions from the tag material; however, the detection process is designed to give very early warning of impending problems and this delay may be of little importance. Alternately, the entire sampling process could be carried out at one central location with a single IMS head serving all rooms. The centrally located detector would be fed samples from all operating locations via an air hose. Any indication of problems could be treated from the central location.

The vibration gauges planned for use are passive, i.e., they require little or no electrical inputs, and their electrical output is minimal. They do not constitute an electrical hazard under normal circumstances.

Figure 5-2 shows one sound transducer per room. There is no reason that several could not be used in each room. The signal from the vibration or sound transducer would be conveyed to the control room via wires and there treated by one or more of several techniques to give warning of abnormal operation.

Anticipation is that passing the sound through a series of filters, as previously shown in Figure 5-1, will be adequate once problem areas are better defined. However, comparison techniques may also be employed if necessary. This would involve storing sounds from recent operation and comparison of these with those sounds currently being received.

A concept of a control and alarm console is shown in Figure 5-3. This panel would serve as a centrally located control center for the equipment. A three-room control is conceived here, each room control being identical. Features shown include provision for Alarm, Test, Calibrate and Locate.

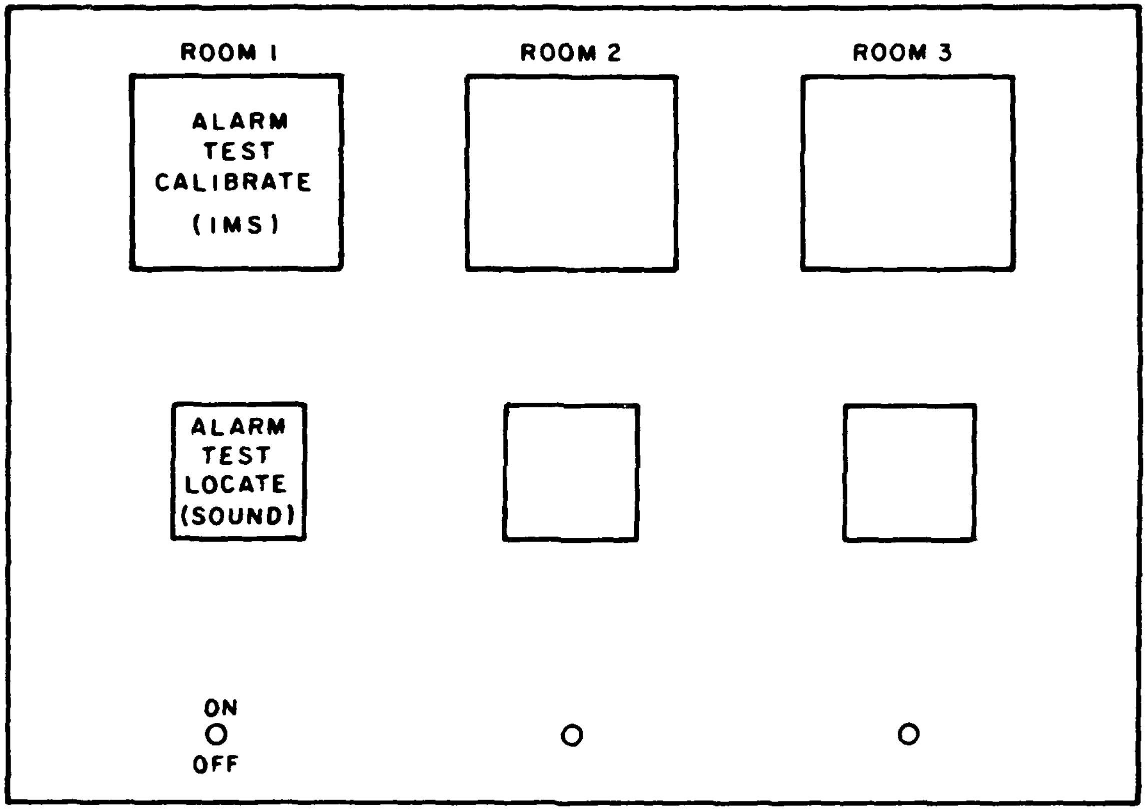


Figure 5-3. Concept of Control and Alarm Console

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Also see Appendix C for Sources of Commercial Literature.

APPENDIX A

ION MOBILITY SPECTROMETER ANALYSIS

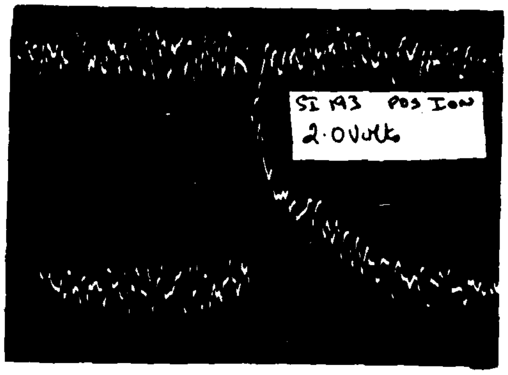
SI 193

POSITIVE ION

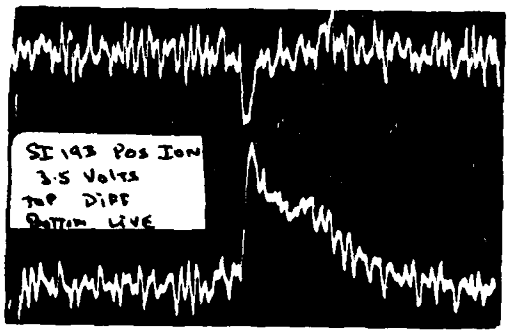
Temp °C	Remarks
30	Slight change
50	Slight change
65	Air peak diminishes in favor of a very large broad peak centered at 14 millisec
87	Air peak diminishes in favor of a very large broad peak centered at 14 millisec
110	Air peak diminishes in favor of a very large broad peak centered at 14 millisec
145	No change
195	No change
225	Air peak returns to near normal height
252	No change
295	No change
300	Air peak normal
350	No change
>350	Flashed

SI-193
Positive Ion

Room Temp



30°C

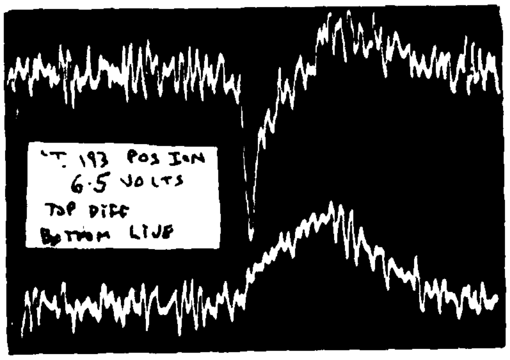


50°C

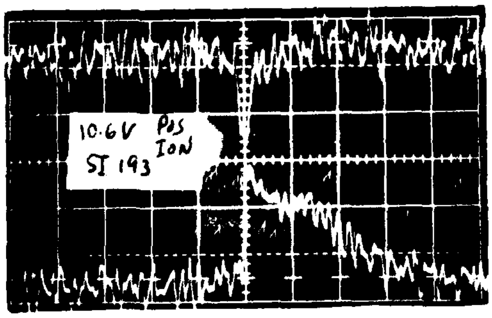
Detection
Definite

87°C

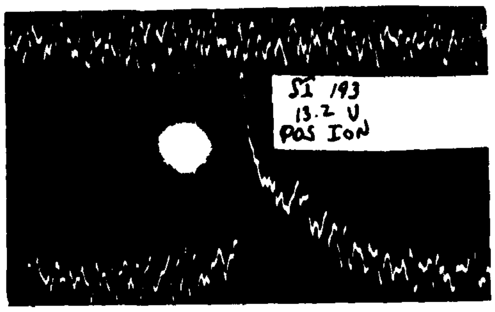
SI-193
Positive Ion



110°C



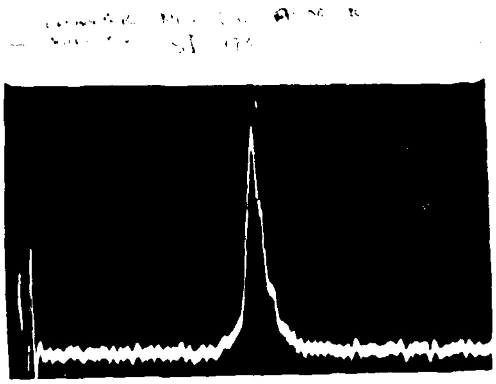
225°C

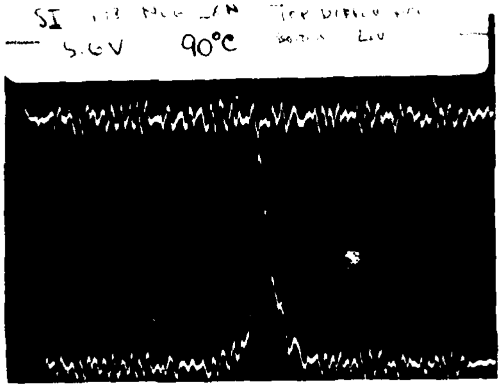


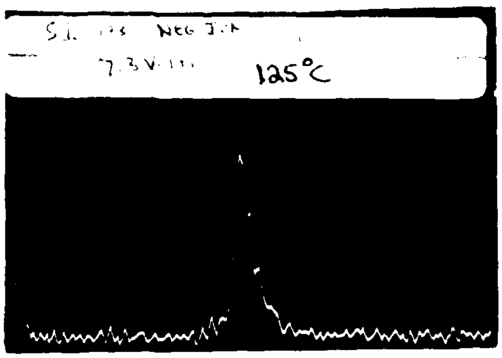
SI 193
NEGATIVE ION

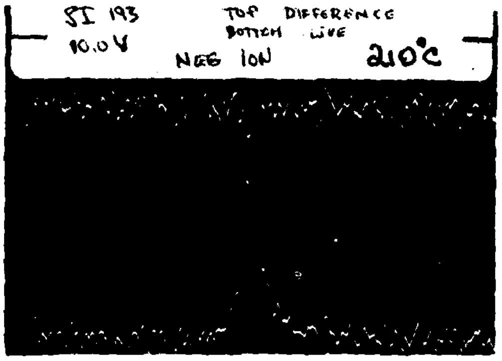
Sample SI-193 Igniter Boron/potassium nitrate/vinyl alcohol acetate resin 431°C, 25/75/1 to 25/75/1 (added)

Temp °C	Remarks
30	Photo of clean air spectrum no change
45	No change
60	No change
90	No change
125	Slight decrease in air peak
210	Slight decrease in air peak
285	Slight decrease in air peak
325	No change
370	No change
>370	No change

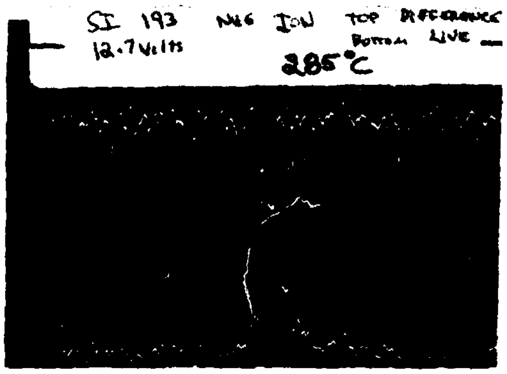



90°C


125°C



Decreased Air
Peak



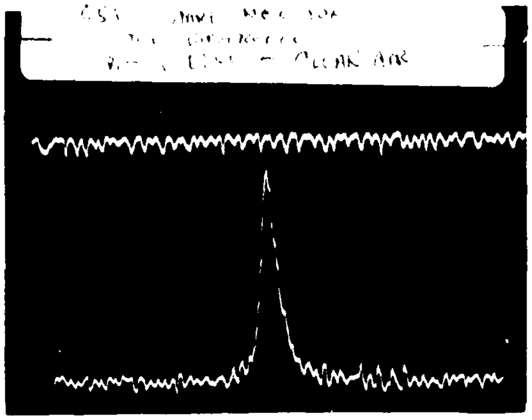
Definite
Detection

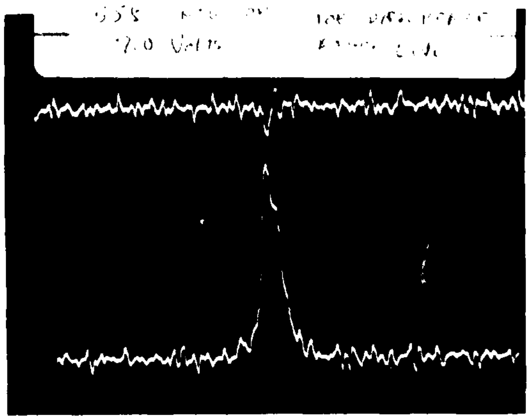
PFP-555

NEGATIVE ION

Formulation: Aluminum/Barium Nitrate/Potassium Perchlorate - 40/30/30

Temp °C	Remarks
38	No change from air spectrum
43	No change from air spectrum
78	No change from air spectrum
120	Shoulder developing on main (air) peak
180	Shoulder developing on main (air) peak
210	Shoulder developing on main (air) peak
255	Shoulder developing on main (air) peak
330	Shoulder developing on main (air) peak
355	Shoulder developing on main (air) peak
>360	No change





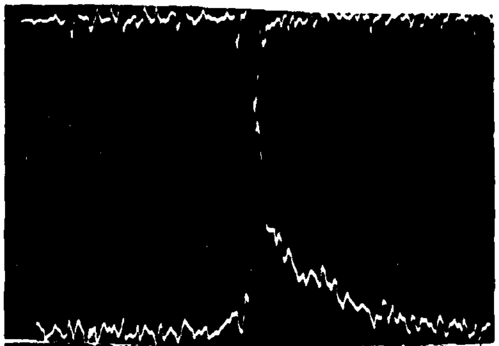
PFP-555

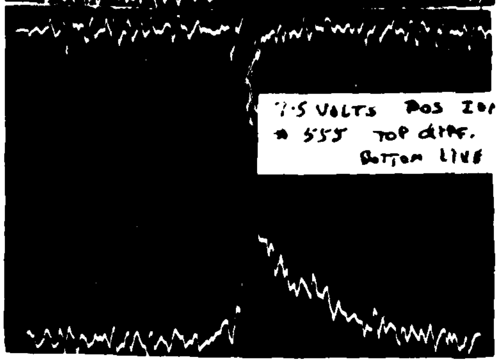
POSITIVE ION

Formulation: Aluminum/Barium Nitrate/Potassium Perchlorate - 40/30/30

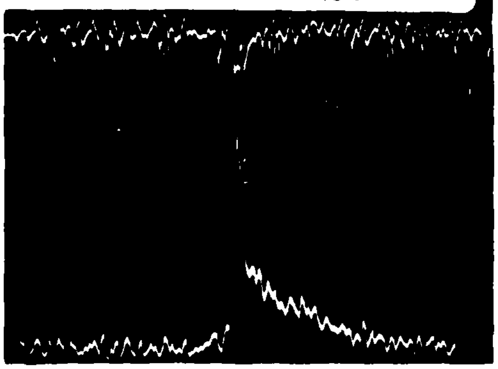
Temp °C	Remarks
38	No change
50	No change
67	No change
88	No change
110	Slight decrease in air peak no new peaks
135	No change
180	No change
210	No change
240	No change
265	Air peak increases to original height
300	Spectrum like that of clean air
330	Spectrum like that of clean air

FORMULATION 555 TOP DIFFERENCE
BOTTOM LIVE
6.5 Volts





POSION. TOP: DIFFERENCE
— 555 BOTTOM LIVE
13.0V

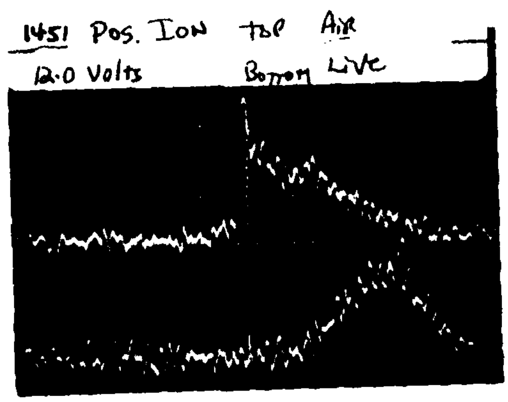


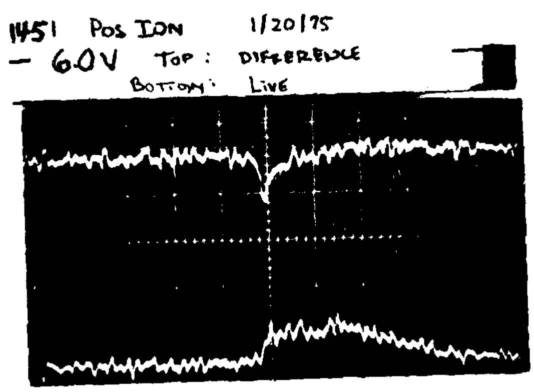
FY-1451

POSITIVE ION

Formulation: Magnesium/Sodium Nitrate/Laminac - 46/45/9

Temp °C	Remarks
40	Air spectrum dirty from start no change
60	Main peak shorter and broader but no new peaks
100	Main peak shorter and broader but no new peaks
120	Main peak shorter and broader but no new peaks
150	Main peak shorter and broader but no new peaks
210	Air peak flattens even more no new peaks
265	Air peak completely gone, new peak, very broad peak
325	No change
>350	No change


1451 Pos. Ion Top Air
12.0 Volts Bottom Live


1451 Pos Ion 1/20/75
− 6.0V Top: Difference
Bottom: Live

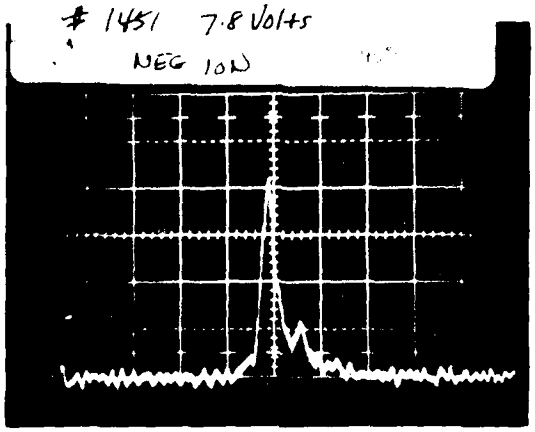
FY-1451

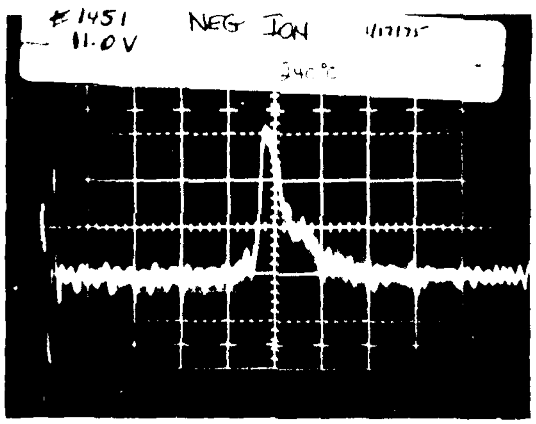
NEGATIVE ION

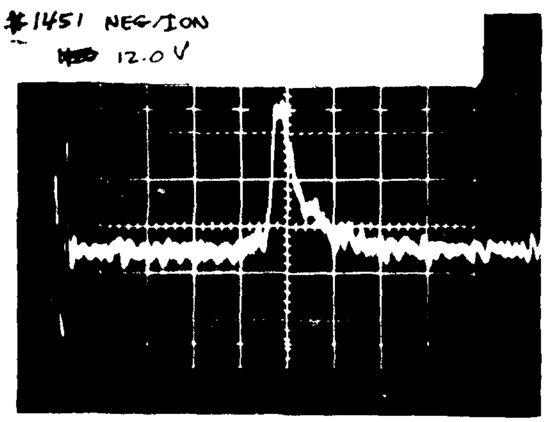
Formulation: Magnesium/Sodium Nitrate/Laminac - 46/45/9

Temp °C	Remarks
40	No change from air spectrum
145	Slight peak at 11.0 millisec
180	Second peak too small to resolve from noise
240	No new peaks but major air peak becomes shorter-broader
270	No new peaks but major air peak becomes shorter-broader
295	No change
325	Broader-shorter
350	Broader shorter still

#1451 Flare Yellow
Negative Ion







APENDIX B

SOUND ANALYSIS

1. Proposed Approach
2. Results of Study at Picatinny Arsenal Batch Processing Facility

ACOUSTICAL METHODS APPLIED TO THE
DETECTION OF CHANGES IN PROPELLANT MIXING ACTION

1. GENERAL CONCEPTS

Changes in the internal sounds emanating from pyrotechnic formulations and propellants during the mixing process may have value as emissaries of approaching danger. The sound signature of the mixing equipment containing the explosive components can be determined in a variety of ways and may be analyzed for distinctive characteristics. This can be done at first on a strictly exploratory basis and, if useful as suggested above, later as a continuous real-time analysis with possibilities for actuating a warning signal.

The best method for deriving sound from the process is by close mechanical coupling to the structure. This would be done by applying one or more accelerometers solidly to pick up structure-borne sound. The sound coupling would be in this way far more effective than through the air path. Accelerometers which are quite small and produce a voltage directly proportional to acceleration provide a convenient pickup for sound. Furthermore one can integrate their signals once or twice to obtain respectively velocity and displacement if that is desired. However, the acceleration signal has the largest amplitude and contains all the frequency components that are to be analyzed.

The analysis usually takes the form of a conversion of the wave from the time domain to the frequency domain. Thus its spectral content is derived, and this is the more convenient characterization for use in identifying diagnostic changes.

2. EXPERIMENTAL PROCEDURE

To begin an investigation of the potentialities of the concepts discussed above one should try to instrument the mixing apparatus with accelerometers for the securing of structure-borne sound recordings. Some thought should be given to the principal mechanisms of sound generation in the mixer itself by virtue of its moving parts (machine sounds) and in the chemical mix. It would seem desirable to so far as possible suppress the former and emphasize the latter. It is possible that the machine sounds may have frequency components differing from those of the mixing sounds and thus could be distinguished by electric filters.

The probable propagation paths of sound generated by the two sources should be considered in the locating of the sound pickups. Careful choices in placement and orientation may help to bring out the desired information.

At first one should make recordings of the mixing machine empty. Then it should be studied under normal operating conditions. If variations in the chemical mix components are part of the pattern of operation, these too should be recorded and identified with the changes as accurately as possible.

Prior to making any recordings of the test machine in operation, background levels and spectral characteristics should be recorded with the apparatus at standstill. This is a measure of the vibrations transmitted to the test machine from those around it and from other sources in the plant. These could be substantial and might confuse the analysis of the data if their presence went unrecognized or uncompensated.

3. APPARATUS REQUIRED FOR DATA ACQUISITION

The simplest instrumentation will consist of a General Radio (GR) Type 1560-P52 accelerometer, attached by magnetic clamp if the machine components are iron, connected to a GR Type 1551-C Sound-Level Meter through a Type 1560-P11B Vibration Pickup System control box. This furnishes a calibrated measure of the acceleration signal so that the response of various attachment points can be compared quantitatively. The output of the sound-level meter will also drive a 600-ohm line over a considerable distance so that the magnetic tape recorder can be operated remotely from the danger area.

The magnetic tape recorder can be any type that has reasonably good (high-fidelity entertainment quality) frequency response. Instrumentation type recorders such as the Nagra or General Radio may not be essential in this case because the great dynamic range (low noise level) which makes them superior is probably not necessary here. The level of the mixing sounds will determine these requirements, and so they cannot be estimated until some preliminary measurements have been made.

Initially we propose to use for recording purposes a Model 1260 Ampex tape deck. If this is not satisfactory, a Nagra IV-D will be used. These together with the sound and vibration pickup equipment listed above or its equivalent will allow us to bring sample acoustic data to the Laboratories for analysis.

Some indications of the levels and frequency ranges involved will be obtained during the field tests and may dictate alternate procedures. For this purpose a GR Type 1933 Precision Sound-Level Meter and Analyzer may be used initially in the field trials to locate the octave bands in which the sounds predominate. This instrument is capable of receiving the signal from an accelerometer or microphone and measuring each of 10 octave band's energy content. Over the band center frequency ranges from 31.5 Hz to 16,000 Hz it is limited only by the capabilities of the vibration pickup (accelerometer) or the microphone, whichever is used.

4. REDUCTION AND ANALYSIS OF DATA

Although an elementary form of data reduction and analysis can be performed as indicated above through the use of an octave band analyzer, these results are not expected to be sufficiently discriminating for our intended use. To accurately characterize the properties of the sounds of concern here we will probably wish to know the spectral content in considerable detail. Such analyses are performed in the Laboratories using one of several audio-frequency spectrometers. For spectrum averaging or steady state signal analysis the Saicor, Model SAI-51B is useful. For the spectral analysis of short-duration sounds, 1 to 40 seconds, and transients the Kay spectrometer is well suited. Each has its own analysis bandwidth limitations and other operational capabilities which need not be discussed further until we learn more about the signals to be analyzed.

Our Sound Laboratory also provides narrow band analyzers for studying steady, unvarying signals. Typical of this class is the General Radio Sound and Vibration Analyzer, Model 1564-A which has 1/3 and 1/10 octave continuously tunable capabilities. This type of equipment can be useful in checking the spectral detail in the vicinity of peaks located by means of the spectrometers mentioned above.

5. APPLICATION OF RESULTS OF THE ACOUSTICAL STUDY

The overall objective of the study is an attempt to find a distinguishing sound component indicative of unsafe mixing action. This may or may not be possible for the reasons that no such sound phenomenon exists or, if it does, it may be difficult, impractical or impossible in the present state of the art to separate it from the interfering machinery noises. However, the investigation will be designed to answer these questions.

If we are fortunate in discovering some unique acoustic signal indicative of dangerous action, it can be used in various warning schemes.

C. W. Hargens
Acoustical Engineering
14 April 1975

03G-C3942-01
9/3/75

PICATINNY ACOUSTIC ALARM STUDY

Recordings of Structure-Borne Emissions, The Alarm Concept

It was hypothesized that incipient malfunction of the blending and granulating machines would generate abnormal structure-borne sounds and that these might be used as a continual indicator of equipment status. To test the correctness of this assumption, experiments were conducted principally to determine the nature of normal sounds within the equipment and by simulation alterations which might be detectable acoustically.

An accelerometer was mounted on various machine components such as the support arm for the roller of the Lancaster blender, and its output amplified and recorded for later analysis in the laboratory. The spectral analysis was subsequently performed at the Franklin Institute using a real-time spectral analyzer capable of capturing the frequency distribution of such sounds as impacts and scuffs of the roller.

Appendix 1 lists 21 test conditions, and of these a number of sound recording excerpts were selected. Appendix 2 lists 21 spectral samples which are documented by photographs in the following section.

Spectral Changes During Malfunction or Malfunction Simulation

Acceleration calibration was performed using a General Radio accelerometer calibrator operating at 1g and 100 Hz. The recording of the spectral analysis of the calibration is given in photograph no. 1.

The spectrum of a normally operating Lancaster blender is shown in photograph no. 2. Here the abscissa corresponds to a frequency range of 1 KHz. Inspection of the recording shows that most of the sound energy during normal operation is below 150 Hz.

When the roller binds and produces a scraping or scuffing sound, quite audible in the recording, the spectrometer shows a prominence at about 150 Hz. This is illustrated clearly in photograph no. 3 and again in no. 7. Repeat analyses without the scrape sound present are given in photographs 4 and 5 to show the consistency of the low-frequency spectrum. Some scraping sounds produce not only a peak in the 150 to 200 Hz region but a general broadband noise as shown in Fig. 6.

A rather different spectrum is produced by impacts. These are shown clearly in Figures 8, 13, 14, 15, 16 and 17. Appendix 2 should be consulted to determine the frequency range of the abscissa which was changed in some of the recordings in order to be sure that higher frequency responses were not missed or to spread the recording for optimum analysis.

Impacts at different places produced somewhat varied response, but, in general, the resonant frequency of the container was evident.

Several tests were conducted with varying amounts of sodium nitrate to simulate the normal mixing compounds and their dampening effects, if any, upon the acoustic response. The listing in Appendix 2 will also serve to clarify differences between the blender and the granulator.

Conclusions

From this preliminary set of measurements, it appears that one could distinguish sounds produced by malfunctioning of the equipment. Much more extensive tests should be conducted before the design of alarm filters and frequency selective indicators is attempted. However, the spectral changes appear to be so marked that a rather positive indication can be expected. Most of the effects are far from marginal.

A number of alarm and verification procedures can be visualized. First an automatic system would show spectral changes or general sound level alterations. This could initiate primary shutdown, or an operator could heed the warning and listen with headphones to compare mentally the characteristic sound of normal function with that producing the alarm.

Still more powerful indications could be achieved by combining in an "and" circuit the acoustics and some other sensing signal, such as temperature or chemical effluents.

Recommendations

We propose to make additional experiments with better controls on the variables involved. For example, we would like to try placement of the accelerometer on various locations to optimize its ability to distinguish between machine noises and malfunctions of various specific types. We feel that this could result in a discriminating system so accurate that it could list the type of malfunction occurring.

We would like to further analyze the old and new tape recordings to obtain a much larger body of spectral data than we now have. This is necessary because we did note fluctuation between various scrapes, scuffs and impacts for the same location. For design purposes, it would be well to have quantitative data on the marginal differences and deviations for basically similar conditions.

Further experiments to investigate the damping effects of the ingredients would be valuable.

Beyond this we visualize the design and construction of a demonstration model with illuminated displays which would indicate the type of malfunction. Such a prototype or model might be useful to Picatinny personnel in explaining the nature of the detection system to others concerned with the subject.

C.W. Hargens
Acoustical Engineering

LANCASTER BLENDER
STOKES GRANULATOR

PICATINNY ARSENAL

1 "Accelerometer mounted on the support arm for the roller of the 4000 gm. Lancaster Blender - at 70 dB" - Cut for approx. 5" - Machine running for approx. 1'20" - then 5 impacts on roller - Approx. 20"

2 "5 impacts on side blade" - Machine running approx. 30".

3 "5 impacts on edge of pan" - machine running approx. 30".

4 "1000 grams of sodium nitrate added to mixer" - machine running approx. 30".

5 "Adding lump of sodium nitrate" - hits and cuts for approx. 30" - Machine runs for approx 1'15"
Prior impacts due to lump of sodium nitrate passing under roller.

6 Machine running - no impacts - approx. 1'5"

7 "Accelerometer NO. 6473 mounted on the base casting that carries the bearing ridge (?) for the drum - recorded at 90 dB." Machine running for approx. 1'55" - impacts at approx. 1 min for approx. 20 sec. 5 center punches on roller itself.

8 "5 center punches on edge plate" - machine running approx. 20 sec.

9 "5 center punches on rim of drum" - machine running approx. 15 sec.

10 Machine running for approx. 10 sec - then silence for 15" - no announcement.

11 "Calibration signal 1G at 100 Hz" - some sort of machine noise - then silence for 6 sec.

12 Same machine noise resumes - then "following will be an empty signal on the front bearing housing of a Stokes Granulator - Accelerometer S/N 6473" - short cut - silence - hum - low signal level for approx. 20 sec. - then - "at 100 dB followed by 10 sec. silence.

13 Machine running 20 sec - then - "empty granulator at 100 dB" - followed by 30 sec. of dead tape.

14 "Granulator with sodium nitrate" - machine runs for 30 sec.

15 "Granulator empty with a possibly 24 mesh screen" - machine runs for approx. 30 sec. then - "Stokes granulator with approx. 24 mesh screen with small amount of sodium nitrate ground until material exhausted" - followed by 10 sec. silence.

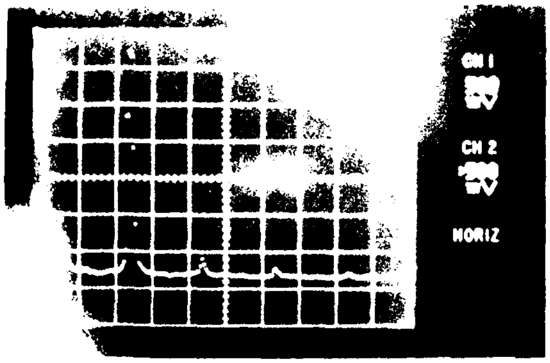
16 "1g 100 Hz calibration signal - accelerometer 6473-AT90DB" - 10 sec of 100 Hz tone.

17 "Small mixer - 1st position on bearing plate holding the roller" - machine runs approx. 5 sec. - then cut for 5 sec. - then "at 80 dB" - some mike hits - machine runs for approx. 10 sec. - dead for 10 sec. - machine runs approx. 10 sec.

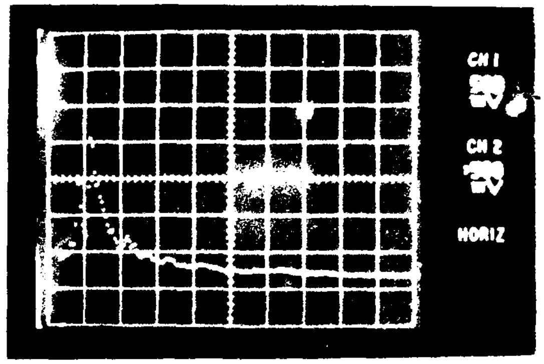
18 "At 70 dB - on side scraper blade" - then approx. 10 sec. ambient noise - machine runs approx. 10 sec. - Cut for 2 sec. - Machine for 7 sec. - then - "Correction - last at 80 dB.

SOUND SPECTROGRAMS

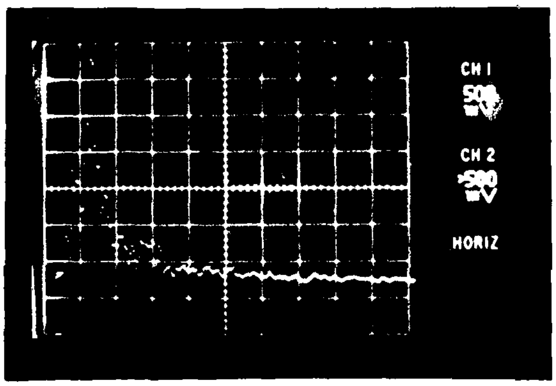
Photo No.	Conditions
1	70dB - Range 500 Hz
2	Lancaster Blender (#1) Range 1 KHz Interg = 16 Machine only - No scrape
3	Same as No. 2 Scrape sound - real-time Range 1 KHz
4	Same as #3 - No scrape sound
5	Repeat of #4
6	Same as #3. Different scrape
7	Repeat of above - 3rd scrape
8	Impact on edge of pan - Range 5K
9	Same area as #8. No impact. Range 5K
10	Blender with 100 grams sodium nitrate added. Range 1K
11	Lump sodium nitrate added - Range 1K
12	Accel. mounted as described in #7 Page 2. No impacts - 5K
13	Same as #12 - impact on roller - 5K
14	Same as #13 - different impact - 5K
15	Same - impact on rim of drum - 5K
	STOKES GRANULATOR
16	Empty granulator at 1000dB - 10K
17	With sodium nitrate - 10K
18	Set up as in #15 page 3 - small amount of sodium nitrate 24 mesh screen - 10K
19	Cal tone as in 16 page 4 - 500 Hz
	SMALL MIXER
20	As in #17 page 4 - 1 KHz
21	As in #18 page 4 - at 80 dB 5 KHz


CH 1
CH 2
HORIZ

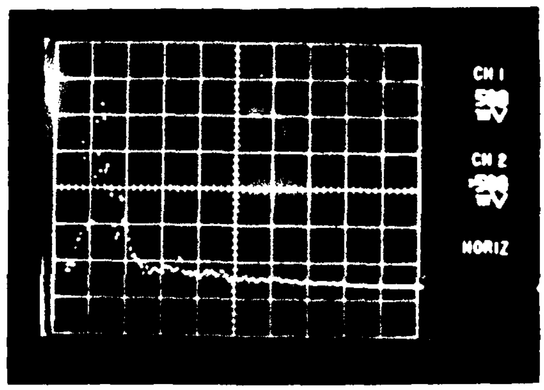
1


CH 1
CH 2
HORIZ

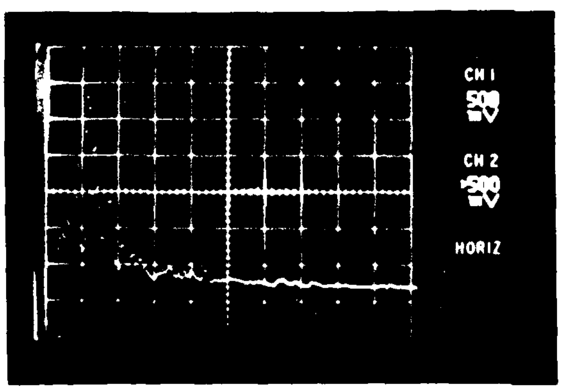
2


CH 1
CH 2
HORIZ

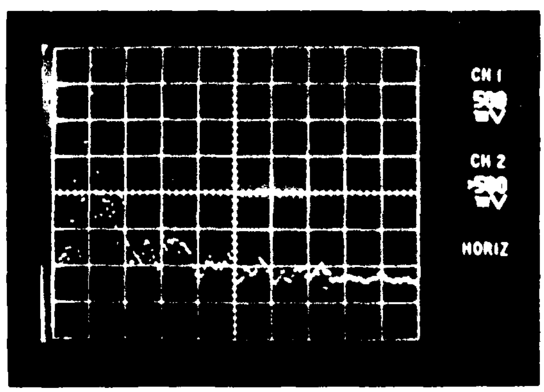
3


CH 1
CH 2
HORIZ

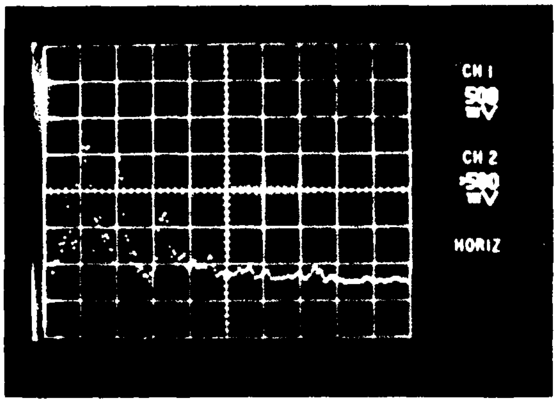
4


CH 1
CH 2
HORIZ

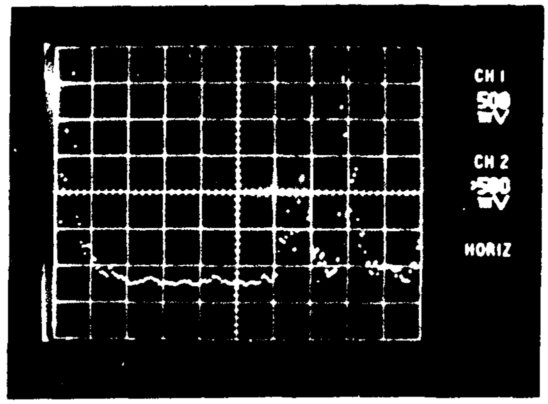
5


CH 1
CH 2
HORIZ

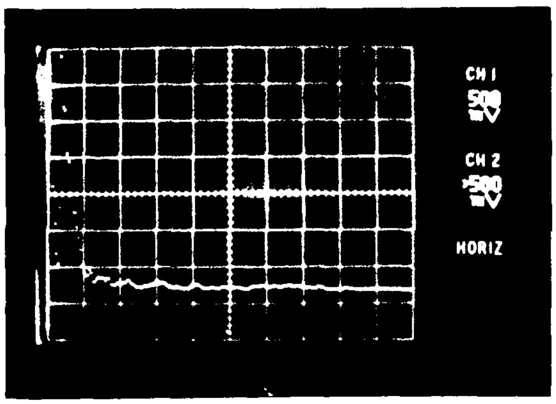
6


CH 1
500 mV
CH 2
500 mV
HORIZ

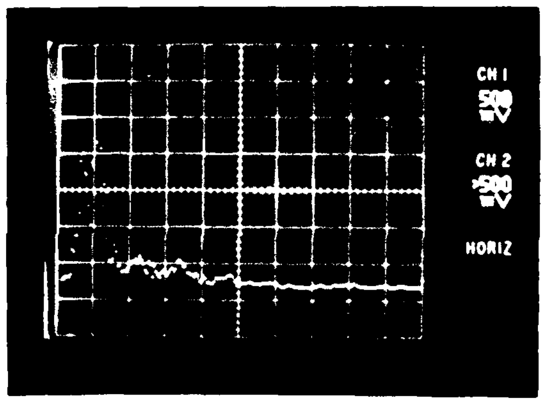
7


CH 1
500 mV
CH 2
500 mV
HORIZ

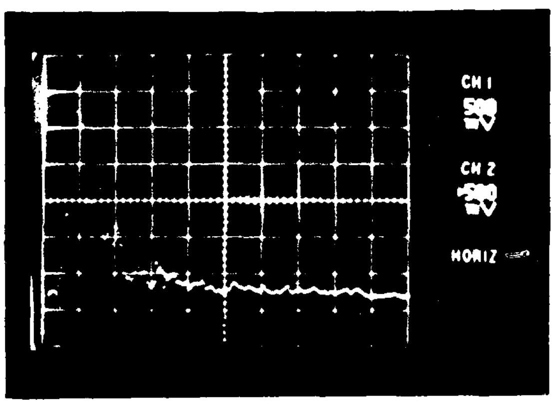
8


CH 1
500 mV
CH 2
500 mV
HORIZ

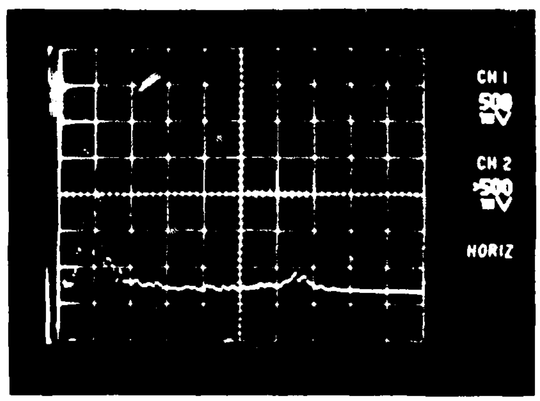
9


CH 1
500 mV
CH 2
500 mV
HORIZ

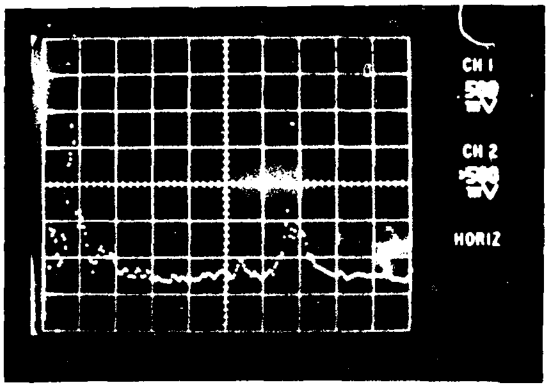
10


CH 1
500 mV
CH 2
500 mV
HORIZ

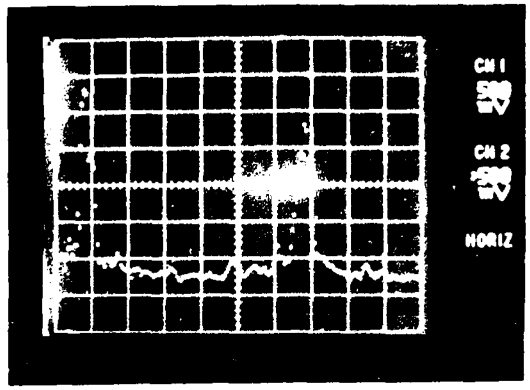
11


CH 1
500 mV
CH 2
500 mV
HORIZ

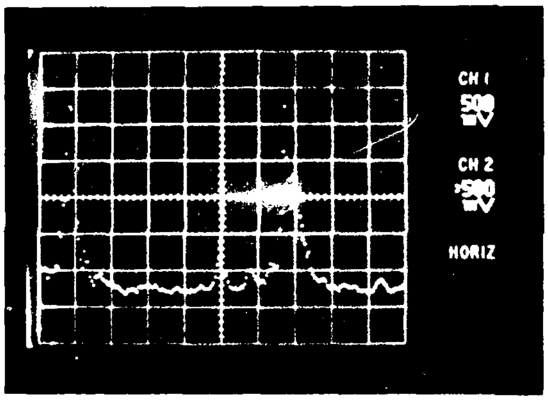
12



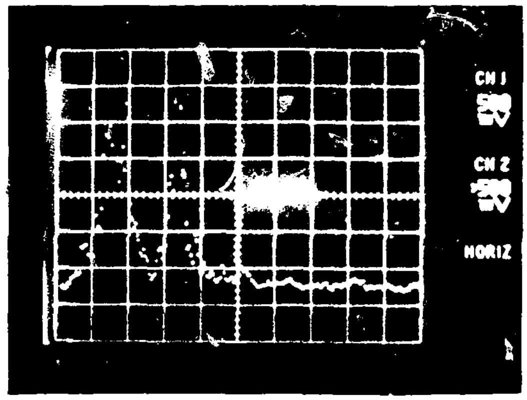
13



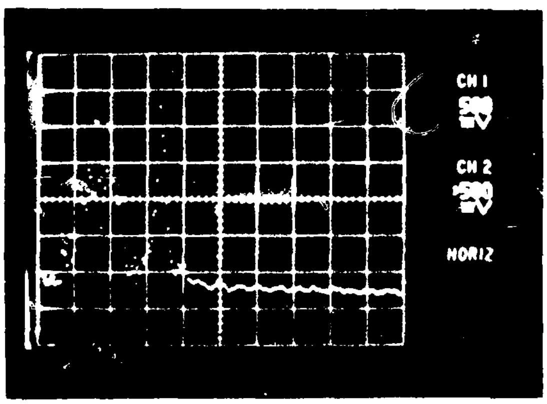
14



15



16



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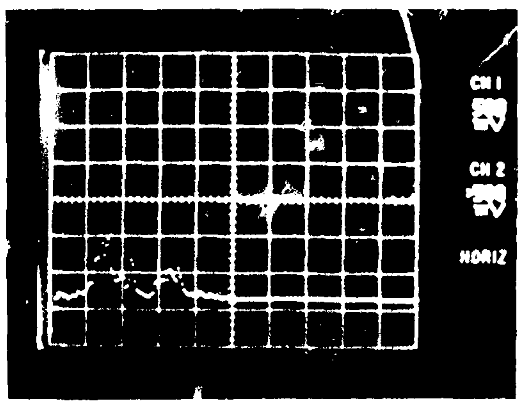

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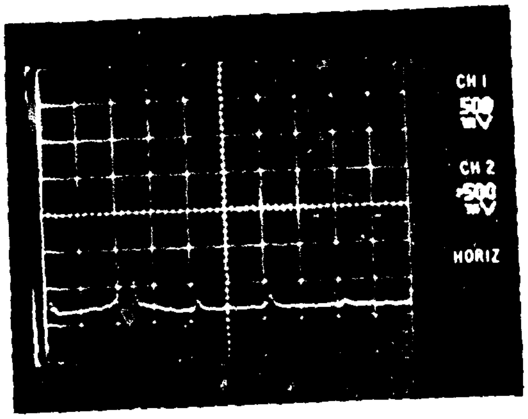
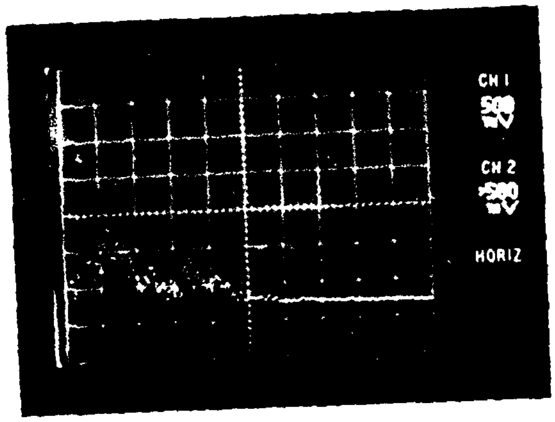
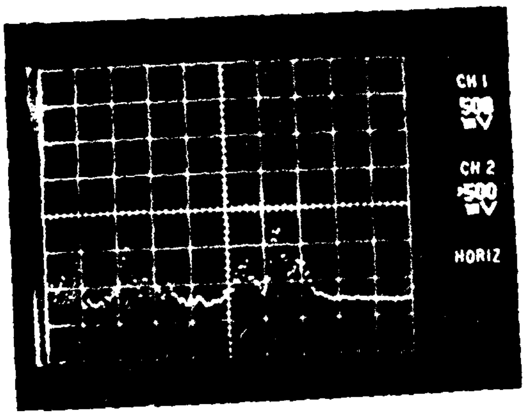

CH 1
500 mV
CH 2
500 mV
HORIZ

19


CH 1
500 mV
CH 2
500 mV
HORIZ

20


CH 1
500 mV
CH 2
500 mV
HORIZ

21

APPENDIX C

RELATED COMMERCIAL LITERATURE

RELATED COMMERCIAL LITERATURE

1. William Wahl Corporation, 12908 Panama St., Los Angeles CA 90066

    Heat Prober Thermometer (Thermistors)
    Temp-Plate-Color Change, Self-Adhesive Sensors
    Surface Temperature Thermometers
    Heat-Spy Infrared Thermometer

2. Milletron, 632 Sanford Blvd., Mt. Vernon NY 10550

    Infrared, non-contacting thermometer

3. Oriel Corp., 15 Market St., P.O. Box 1395, Stamford CT 06904

    Infrared, Golay Cell

4. Williamson Corporation, 116652 Main St., Concord MA 01742

    Industrial Process Temperature Control Systems

5. Figaro Engineering Inc., 2-15-6 Higashitoyonaka, Toyanaka City, Osaka, Japan

    T6S - Gas Sensing Sem. Conductor

6. Monroe Electronics Inc., 100 Housel Ave., Lyndonville NY 14098

    Stat-Arc - Portable Static Monitor

7. Consolidated Devices Inc., 895 Waverly Ave., Holtsville NY 11742

    Multi-Channel Thermocouple Instrumentation
    Transmitters, Alarms, Accessories

8. Spectron Instruments, P.O. Box 891, La Habra CA 90631

    Acur-Temp - Non-Contacting Temperature Sensors

9. Fenwal Incorporated, Ashland MA

    Explosion Protection Systems - Bulletin MC-215A

10. Namac Corp., P.O. Box 8, Indian Head MD

    Fast response thermocouple

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