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CONCEPT DEVELOPMENT FOR DETECTION OF LEAKS IN WP MUNITIONS (DRY PROCESS)

by

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August 1975

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DEPARTMENT OF THE ARMY
Headquarters, Edgewood Arsenal
Aberdeen Proving Ground, Maryland 21010

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Existing methods of leak detection were reviewed to determine their applicability to detect leaks in selected white phosphorus munitions immediately following production line filling and the relatability to the existing oven test method. Heated anode (halogen detector), electron capture (sulfur hexafluoride detector), infrared absorption (carbon dioxide), and mass spectrometer (helium detector) were found to have the needed sensitivity and ability for fully automated monitoring. Compatability tests of

20. continued

dichlorodifluoro methane and sulfur hexafluoride with liquid white phosphorus indicated no reaction between these tracer gases and the WP.

PREFACE

The work described in this report was authorized under PA, A 4932, Project No. 5751250, MIPR B5061 and TWR. No. 6107. It was performed at the NASA National Space Technology Laboratories (NSTL) for the Edgewood Arsenal Resident Laboratory (EARL) by the General Electric Company under Contract No. NAS8-27750. The work was completed in June 1975.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

REPORT ON

CONCEPT DEVELOPMENT FOR DETECTION OF LEAKS IN WP MUNITIONS

(DRY PROCESS)

1.0 INTRODUCTION

1.1 Objective. The objective of this study was to determine methods of leak detection which would be appropriate for application to dry process production line filling of white phosphorus munitions. The scope of work was restricted to consideration of only those methods for which existing off-the-shelf detectors were available. The range of application requirements was limited to detection of leaks in munitions M302, M156, M375, M60, M416 and M110.

1.2 Authority. The work described in this report was authorized under PA, A 4932, Project 5751250, MIPR B5061, and TWR 6107. It was performed at the NASA National Space Technology Laboratories (NSTL) for the US Army Edgewood Arsenal Resident Laboratory (EARL) and NASA/NSTL by the General Electric Company under Contract No. NAS8-27750 during the period April 1975 through June 1975.

1.3 Background. White phosphorus is commonly used in bursting type munitions for production of smoke screens and signals. Current production of white phosphorus (WP) munitions utilizes a wet filling process, whereby the empty munition is completely submerged in liquid WP. After the munition is filled, a certain amount of WP is aspirated, leaving a predetermined void space at the top of the munition. The munition is then sealed by pressing a burster well into the munition opening.

A "dry filling process" is being considered to replace the above described manufacturing process, wherein liquid WP is vacuum-transferred from a storage area to the munition. In this process the munition is filled to a predetermined level leaving a void space at the top of the round. CO_2 under pressure is then used to force the remaining WP back into the storage area leaving a heavier-than-air gas blanket above the WP. The munition is then sealed by pressing the burster well into the round. The advantages of the dry filling process over the wet process, one of which is cleanliness, is not the concern of this report and will not be discussed here.

Following the munition sealing,common to both processes, is an oven test whose primary function is associated with the detection of existing leaks and those leaks which may be produced during storage due to increases in temperature and pressure. The current failure rate is based upon visual observation of extruding WP during the six to eight hour test. The object of this report is to consider leak detection methods whereby leaks may be detected in a shorter amount of time, at room temperature, and with less probability of exposure of WP to the atmosphere.

2.0 TECHNICAL DESCRIPTION

2.1 White Phosphorus Filling Line

2.1.1 White Phosphorus Munitions. Six types of munitions are under initial consideration for production line filling using the "dry fill" process for which an alternate leak detection method is being considered. These munitions are:

- 60mm, M302
- 2.75 in, M156
- 81mm, M375
- 105mm, M60
- 155mm, M416
- 155mm, M110

These munitions are loaded with WP from the top and are sealed with a burster well ranging in diameter for the first four from 20.3mm to 59.5mm.[1] The seal is a metal-to-metal pressed fit between the steel casing and either an aluminum (M156 and M375) or a steel burster well. A silicone, white lead or cosmoline lubricant is used to assist in easier and more effective sealing. The shell casing of all the munitions for which drawings were available are one piece cast or brazed so that the only source of leak is in the press seal area, as shown in figure 1. The seals are located at the top of the munition, so that leak detection techniques need consider only this area of investigation.

2.1.2 Munition Filling Line. A basic operation outline of the WP munition dry process production line is shown in figure 2. In the proposed dry filling operation, liquid WP at approximately 140°F is vacuum transferred to the open munition which has been purged with CO_2. After filling to the required height, the WP is cleared from the filling line by back pressuring with CO_2. This leaves the void space in the munition filled with CO_2 prior to insertion of burster well. After insertion, the burster well is pressed into the munition. In the wet fill process, three munitions are sealed simultaneously. This pressing operation has the effect of pressurizing the void space in the top of the munition. Table 1 gives the expected void volumes andpressures after this operation[2]. It is assumed that one of the objectives of the new process implementation will be a full automation capability with a minimum of personal material or munition handling.

Following the pressing operation the munition is painted and marked. The munitions are then transferred to an oven where the round is heated to maintain the WP in molten state in order to facilitate detection of a leaking round and simulate worst case storage conditions. One of three types of oven test is then conducted:

1. 180°F for 8 hr. 2. 160°F for 8 hr. 3. 207°F for 15 min.

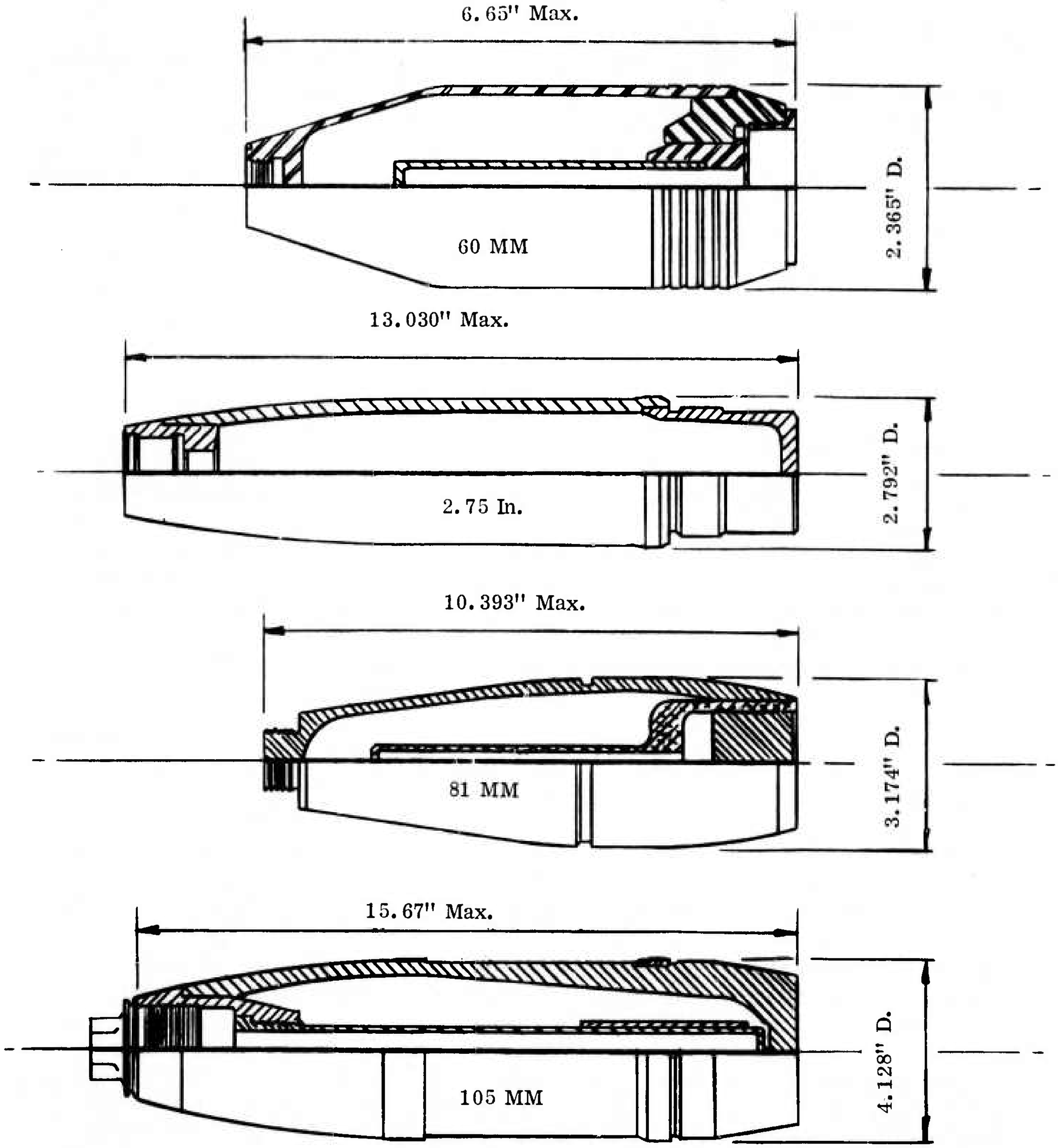

Figure 1. WP Munitions

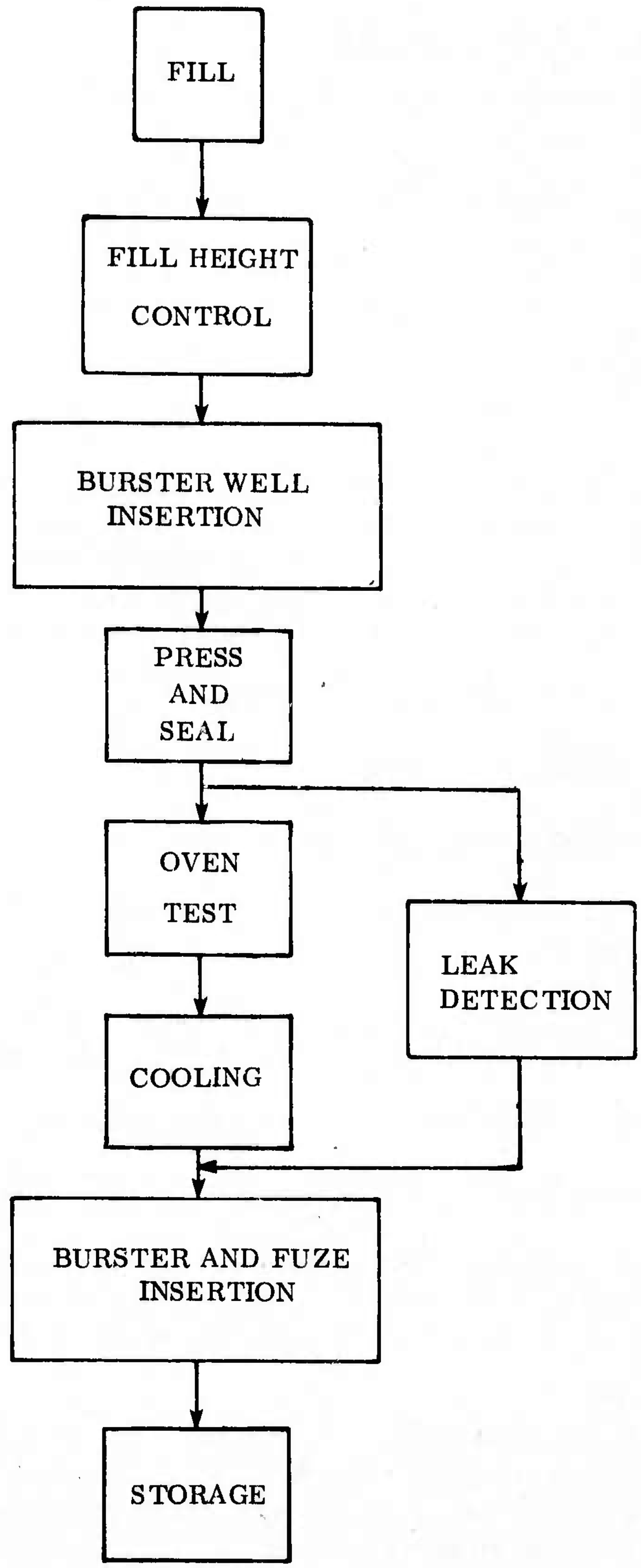

Figure 2. Preliminary WP Munition Production Line

Table 1. Expected Munition Void Volume and Pressure After Filling

Munition	Total Volume (cc)	Void (%)	Void Volume (cc)	Void Pressure (psi)
M302	205	5	10.25	30.7
M156	398	3	12.0	20.8
M375	475	2	9.5	146.0
M60	1152	7	80.6	19.0
M416	1595	7	111.6	37.5
M110	3998	3	120.0	17.6

Automatic smoke detectors and visual inspection following the oven test are used to identify the leaking rounds which are then placed in containers of water. Edgewood Arsenal representatives indicate munition failure rate due to leakage is as large as 1% for some rounds. Although the primary causes of leakage have not been investigated and are not within the workscope objectives, the areas of greatest concern seem to be:

- Seal closure reliability, including bad fit or marred surface
- Defect in burster well
- Overfill of munition and lack of void

2.1.3 Need for Leak Detection. An alternate method of detecting leaking munitions is being considered which will:

- Reduce the probability of exposure of WP and P_2O_5 to the environment and thereby reduce the personnel hazard of the oven test.
- Reduce the cost and energy consumption present in the oven test.
- Reduce the intraround propagation of leakers inside the oven.

2.2 Leak Detection. There are two areas of concern in leak detection; one is leak measurement and the other is leak location. In this study we are interested only in determining whether or not a leak exists in a particular munition and are not concerned with its actual location.

Since the physical dimensions of a leak, i.e., its length and width are generally unknown, the size of a leak is described in terms of its leak rate. The leak rate or mass flow rate (Q) for a gas is best described by the number of molecules escaping per unit time. When the temperature of a system is known, the ideal gas law relates the mass flow number to the more readily determined dimensions of

$$\frac{\text{Pressure x Volume}}{\text{Time}}$$

Although there is a wide variety of units used for leakage description, this report shall use engineering designation for Q in atm cc/sec.

2.2.1 Evaluation Criteria. The following discussion summarizes the considerations used in the selection of techniques appropriate for detecting leaks in WP munitions.

Applicability - An appropriate leak detection technique must be suitable for operation either at atmospheric pressure, or should experience little or no detrimental effects due to frequent opening to the atmosphere as might be encountered for detectors requiring vacuum operation. The munitions are sealed prior to leak checking so that all testing must be done external to the munition. Due to the already hazardous nature of WP the technique should not add significantly to the possible hazards environment. The detector should be applicable to production line operation and able to withstand the plant environment of contaminant background of the atmosphere, noise and or vibration.

Sensitivity - While it is recognized that no object is totally leak tight, there is a point below which a leak is acceptable or tolerable as determined by such considerations as reaction rates, expected storage life, extra cost for added sensitivity, or background noise.

One objective of this report is to comment on the relatability of the leak detection technique with the oven test. During the oven test the temperature of the WP filled munition is raised to some specific temperature for a definite length of time. During this time the interior of the munition experiences an increase in pressure due to temperature increase of the gas occupying the head space in the round and the volume expansion of the White Phosphorus.

The leak rate, Q, is related to this difference between the pressure inside the round and the atmosphere outside. The exact functional dependence on the pressure is determined by the physical dimensions of the leak and the magnitude of the pressure differential[3]. The three primary categories are turbulent flow ($Q > 10^{-2}$ atm cc/sec), laminar (10^{-1} atm cc/sec $> Q > 10^{-6}$ atm cc/sec) and molecular ($Q < 10^{-6}$ atm cc/sec) where:

Turbulent flow

$$Q \propto \left[P_1^{\,2} - P_2^{\,2} \right]^{1/2},$$

Laminar

$$Q \propto \left[P_1^{\,2} - P_2^{\,2} \right],$$

Molecular

$$Q \propto \left[P_1 - P_2 \right],$$

An increase in the pressure difference as occurs in the oven test, therefore, correponds to an increase in the leak rate. Pressure increases due to the pressing operation using estimates of the volume expansion, β, of WP between 140°F and 180°F of $5x10^{-4} \cdot C^{-1}$ can be estimated using the relation[4,5].

$$P = \frac{P_i V_i T_f}{T_i (V_t - V_i) (1 + \beta \Delta T)}$$

where (V_t-V_i) represents the volume of WP in the rounds. The predicted pressure increases are given in the following table:

Munition	Initial Pressure	Final Pressure
M302	30 psi	53 psi
M156	20 psi	44 psi
M375	146 psi	437 psi
M60	19 psi	30 psi
M416	37 psi	60 psi
M110	17 psi	37 psi

Current leak detection by means of observing WP extruding from the munition is indicative of gross leak which correspond to gaseous leak rates on the order of 10^{-2} to 10^{-3} atm cc/sec.

For purposes of correlation an increase in sensitivity of leak detection of three orders of magnitude should compensate for the difference in pressure change encountered in the oven test. Therefore, it is expected that a leak detection sensitivity of 10^{-5} to 10^{-6} atm cc/sec at room temperature should be comparable to the existing oven test leak detection. In this region the leak will have laminar flow characteristics. Although the leak rate is also inversely proportional to the viscosity of the escaping gas, there is not sufficient difference in the viscosity of most gases, except hydrogen and helium, to make any one significantly better for dilution than another.

Basic to a discussion on the sensitivity necessary for a particular detector is the selectivity of the detector or its ability to differentiate between the substance being sensed for and other gases possibly present in the background environment.

Response/Time - Production estimates of 8000 rounds per 6.5 hour shift allows approximately three seconds per station per round. This represents the time available for a sensor to detect a leaking munition. However, multiple or carousel type loading and handling facilities may be employed to extend the time for detection and recovery on the order of 10 to 20 seconds. It should be noted that with an expected failure rate on the order of 1 percent or less, leak detection of several rounds simultaneously may be faster and more efficient than individual testing. This consideration will depend ultimately upon the size of the munition and the leak detection method used. Additional considerations affecting the response time but independent of the type of detector are discussed in a later section.

Operation - Critical to the successful use of a sensor on an automated production line is the complexity of its installation, operation and maintenance in addition to the skills required of the personnel who will be operating the instrument. Another point for consideration under operational criteria is the expected lifetime or downtime required for maintenance of the detection system. Although the degree of automation, whether semi or full, is a consideration, it is not of prime concern here since electronics systems may be easily built into the specific material handling equipment to give any degree of automation desired.

Cost - Initial cost of a detection system is a large factor in its selection, however, it is usually proportional to the sensitivity and, therefore, cannot be used as a sole determining factor. Other considerations affecting the cost will be maintenance and operating expense including such things as parts and tracer gases used for detection.

2.2.2 Leak Detection Methods. Many methods of leak detection were considered for application to this problem. There exists a number of physical and chemical principles upon which a leak detector system may be based. These may be divided into three categories:

- Detection of the leaking munition material, P_4, P_2O_5 or CO_2 .
- Detection of an added or tracer material used specifically for leak detection.
- Detection of physical or chemical changes in or around the munition, i.e., pressure, temperature or color.

In the case of White Phosphorus filled munitions the primary candidate material for detection would be either White Phosphorus, phosphorus pentoxide or the filler gas contained in the munition head space. Since one of the objectives of this study was to find a method which would supplement or replace the current oven test and thereby reduce the probability of exposure of WP, methods utilizing its properties were not considered. For similar reasons a detector for P_2O_5 was not pursued. This study, therefore, restricted itself to techniques appropriate to the detection of escaping gases from the head space in the sealed munition and detection of physical and chemical changes in or around the munition.

This study considered only those systems of detection which were commercially available as off-the shelf items which would require, at most, material handling or tooling modifications for application to the production line.

Although a wide variety of techniques were considered, time available did not permit a complete analysis of all of them but required early elimination of the majority of the candidates based upon assumptions of the criteria for this particular application.

This section summarizes the characteristics of commercially available techniques used for leak detection. Approximately one hundred companies were contacted of which approximately half supply some type of leak detector. The remainder no longer supply leak detection equipment or only furnish engineering support for equipment installation. Detector descriptions and specifications were obtained from manufacturers literature, conversation with applications engineers and from the Leakage Testing Handbook[3].

Ultrasonics - This technique depends upon the detection of ultrasonic sound waves in the frequency range 36kHz to 44 kHz generated by pressurized gas flowing through a leak. Since laminar flow does not generate sound, this method is applicable to leaks in the turbulent flow range above 10^{-2} atm cc/sec. Available detectors such as those from Hewlett Packard and Dawe Instruments are highly directive in nature and lend themselves more to leak location techniques. In addition high noise levels present in a manufacturing environment make this detector inappropriate for production line monitoring of small leaks.

Chemical Indicator - The chemical indicator technique of leak detection is primarily used for location of leaks of the order of 10^{-3} atm cc/sec. The two basic types of chemical indicators are based upon either a color change due to a chemical reaction with a soluble dye placed in the area of the leak (available from Metronics Associates, Inc.) or observation of a penetrant liquid usually of a fluorescent nature which has penetrated the leak due to the internal pressurization (supplied by Magnaflux Corp.).

Both techniques use essentially a static testing technique requiring longer inspection times for greater sensitivity. Since this method is dependent upon visual inspection for possible minute areas of color change or dye presence it does not lend itself to fast or automated leak detection.

Bubble Testing - One of the easiest and least expensive methods of leak detection is bubble testing. Two basic techniques used in bubble testing are liquid immersion, and liquid application with liquid immersion being the more sensitive. The maximum sensitivity of bubble testing is in the range of 10^{-4} to 10^{-5} atm cc/sec. Although manufacturers of bubble solutions such as American Gas and Chemical Corp., Cargille Scientific Inc. and the Heckerman Corp., claim detection capability below .25 atm cc/hr or 7×10^{-5} atm cc/sec, the detection in this region is very dependent upon the observation and alertness of the operator and is therefore not well suited for production line automated leak detection. The solubility of the tracer gas in test liquid also affects this lower detection limit where a leak may dissolve in the liquid before being observed.

Pressure Rise - The pressure rise technique consists of detecting a pressure change in a system enclosing the item being tested. The leak rate is related to a change in pressure Δp inside a fixed volume, V, by the relation

$$Q = \frac{\Delta PV}{t}$$

The smaller the volume the faster the leak may be detected. One advantage of this method is that it is independent of the tracer gas used and therefore does not require any special gas handling other than that already used. Available sources of pressure differential manometers and McCleod gauges such as MKS, Datametrics, Federal Products Corp., enable detection of pressure changes on the order of .01 psi. This technique requires complete enclosure of the munition with the ultimate sensitivity dependent upon the volume of open space in the test chamber. Evacuation of the test chamber prior to testing would improve sensitivity but would also lengthen cycle time. The major difficulty with this method is the unknown pressure changes due to outgassing and changes in the temperature of the munition during the test. Maximum sensitivity for this application is therefore estimated to be of the order of 10^{-4} atm cc/sec.

Flow measurement - Another common method of leak detection related to the pressure rise method is the measurement of volume change in the test chamber due to the leak. This technique would use a wet type gas meter or movement of a small liquid plug inside a glass capillary tube connected to the test chamber for detection. This method has the same disadvantages as the pressure technique and is therefore not useful for detecting leaks smaller than 10^{-4} atm cc/sec.

Thermal Conductivity - The thermal conductivity technique, widely used for measuring vacuum pressures, is also applicable to detecting leaks at atmospheric pressure. This method will detect the presence of any gas with thermal conductivity different from air. Typical gases used for leak detection in conjunction with thermal conductivity sensors are helium, hydrogen, carbon dioxide, methane, freons, and other hydrocarbons. Sensitivity may be as high as 10^{-5} atm cc/sec for helium but rapidly decreases for other gases due to their different thermal conductivity. Some manufacturers are GOW-MAC Instrument, Ion Tract Instruments and Uson Corporation with costs ranging from \$500 to \$3000 depending upon sensitivity and portability. This method is not considered applicable for production line monitoring due to its poor selectivity and low sensitivity.

Another type of detector similar to the thermal conductivity detector and used for detection hydrocarbons such as propane, butane, methane, benzene derivatives and carbon monoxide is the catalytic detector. In this sensor the presence of a combustible gas raises the temperature of the catalyst which is monitored by a heat sensing element. Precision Gas Products and Environmental Metrology Corp. are some of the manufacturers. This technique is not considered useful in this application due to the need of a combustible hydrocarbon as a tracer gas which would increase the hazards already present.

Flame Ionization - Flame ionization is a measuring technique adapted to leak detection which finds its greatest use in the detection of combustible hydrocarbons. With a concentration sensitivity of 10 ppm and a sampling rate of about 100 cc/sec it has a leak rate sensitivity of about 10^{-3} atm cc/sec. For slower sampling rates of the order of 1 cc/sec the sensitivity may be lowered to the order of 10^{-5} atm cc/sec. However, since it detects primarily combustible gases its use is not considered applicable here.

Light Absorption - Leak detection by light absorption gives very good selectivity for determining the presence of tracer gases.

Infrared sensors will detect concentrations in the range of ppm, so that with an internal pumping speed in the range of 1 cc/sec, leak rate sensitivity of the order of 10^{-6} atm cc/sec is obtainable. Modern Controls is the only supplier of an IR detection system for CO_2 leaks. Their system employes a carousel arrangement for multiple testing. The system performs the actual testing after a partial vacuum has been pulled around the sample. A unique feature of their system, which has been incorporated into the unit for detection of gross leakers or poor sealing test fixtures, is a pressure sensor set into the vacuum pumping system which looks for a maximum allowable pressure remaining in the test chamber after a fixed period of pumping.

This leak detection system is relatively new in its commercial availability and the actual IR detection unit has not been refined as a general type detector and is only available as part of the complete material handling and sensor package. As such, the detector portion is sold at approximately $15,000.

Halogen Detector - The halogen detector utilizes a heated anode, usually platinum, at about 900°C. The characteristic ion emission from this hot surface varies in proportion to the concentration of halogenated compounds present. Halogen compounds are those containing any of the elements fluorine, chlorine, bromine or iodine. Thus the halogenated hydrocarbons such as dichlorodifluoromethane (freon 12) and the other freons are easily detected using this type sensor. Principal sources for this detector are the General Electric Company (Model H-25), Ion Track Instruments (leak finder) and Inficon Inc. (hld-2). The sensing unit samples the atmosphere at a rate of about 10 cc/sec and claims a maximum sensitivity of the order of 10^{-7} to 10^{-8} atm cc/sec. Although its primary use is found in hand held sniffer applications its sensitivity and sampling rate make it appropriate for application to automated production line monitoring with little modification.

Electron Capture - The most recent addition to leak detection techniques uses the principle of electron capture to detect electronegative gases. A constant electron current is maintained in the detector by ionization of a carrier gas flow of argon/methane across a weak radioactive tritium source[6]. Detection is accomplished when the presence of an electronegative gas causes a decrease in this current by capturing available electrons. The relative sensitivity or electron capture coefficients of various types of compounds are given below:

Compound	Relative Sensitivity
trifluoro-, monochloro-	0.1 - 10
monobromo-, dichloro-,	10 - 100
trichloro-, dibromo-, hexafluoro-,	100 - 1000
monoitro-, benzene-.	100 - 1000

Due to the arc suppressant characteristics and blanketing nature of SF_6, sulfur hexafluoride is commonly used as a tracer gas for leak detection using the electron capture device. With a sampling rate of about 2 cc/sec the principle suppliers, Varian (2310 Bantam Leak Detector) and Ion Track Instruments (ITI Leakmeter), claim sensitivity of the order of 10^{-8} atm cc/sec for SF_6. It is also used primarily in manual operation but has the capability for use in automated production line applications.

Helium Mass Spectrometer - Helium mass spectrometry is the most commonly used method for leak detection. Its sensitivity in optimum operating conditions can be as high as 10^{-12} atm cc/sec and its selectivity in detecting the presence of helium without interference from other gases is unmatched by any other technique. Helium being a light inert gas, will penetrate any leak without any possibility of closing or sealing it and is therefore aptly suited for leak detection. Although the mass spectrometer may be tuned to detect other gases by their atomic weight, interferences become a problem as the detected mass is increased. The mass spectrometer is basically composed of four components:

1) Mass spectrometer tube

2) Stable electrometer amplifier

3) High vacuum section for spectrometer tube environment which must be maintained below 10^{-4} Torr.

4) Mechanical pumping section for initial pump down or interface between atmosphere and high vacuum

Several manufacturers supply helium mass spectrometers for use in production line monitoring of leaks, among them are:

Aero Vac Corp. (Model 18-702)

Dupont Instruments (Type 24-120B/038) formerly Consolidated Electrodynamics Corp.

Edward High Vacuum Inc. (Model LT104)

Varian/Vacuum Division (925-40 Porta test and Integra test and VFT)

Veeco Instruments Inc. (Model Ms-17 UFT)

These units range in price from $7,500 up to $15,000 for the basic unit requiring manual loading. Helium, if it is used as the tracer gas, will have to be used in conjunction with a heavier gas such as CO_2 in order to maintain an insulating blanket over the WP during the filling operation. For a mixture 1 percent He in CO_2 the maximum sensitivity would be reduced to the range of 10^{-8} atm cc/sec, still more than sufficient for the sensitivity needed.

Radioisotope Detection - The radioisotope procedure employs diluted krypton 85 as a tracer gas and a detector which measures the beta emission from the leaked gas. Maximum measurement sensitivity is in the range of 1 microcurie per cubic meter of activity. The sensitivity of this technique may be as high as 10^{-13} atm cc/sec. The Krypton source may be mixed with N_2 or CO_2 in a proportion to give the desired sensitivity, in particular about 10 microcurie per 10^{-5} cc, while reducing the cost of the tracer gas. This mixture would cost approximately $100/liter or approximately $1.00 per unit tested in this sensitivity range. Equipment for this leak detection method is manufactured by Tracor-Northern, with the radiation detector costing approximately $4,000. An AEC license is necessary for possession and use of the Krypton 85 leak test equipment[7]. Response time for detection of radiation from leaks of the order of 10^{-6} atm cc/sec would be approximately 15 to 45 sec with a 30 sec 'dead' or downtime in the case of saturation. For these reasons this method does not seem appropriate for leak detection in WP munitions.

2.2.3 Testing Considerations. The function of the proposed detection method will be leak measurement or detection rather than determining the specific location. The test configuration must therefore ensure a low background of the tracer gas being detected in order to have the necessary reliability. This may be accomplished in several ways:

- purging the immediate vicinity of the detector and tested round with some non-detectable gas

- completely enclosing the test munition during the test

- performing the test at a 'safe' distance from the filling operation to ensure low background

There are two modes of leak detection, static and dynamic. The static method sometimes called accumulation testing involves placing the pressurized test object in a closed chamber and monitoring the change in partial pressure or concentration of the tracer gas as a function of time. The partial pressure change of the tracer gas is given by

$$\frac{dp}{dt} = Q/V$$

where Q is the leak rate, and V is the volume of the chamber accumulating the gas. For high density tracer gases, such as Freon or SF_6 that tend to stratify, circulation may be necessary to ensure detection. In dynamic testing the system around the test object is continually pumped. This mode of testing includes both the manual sniffer probe measuring technique operating at atmospheric pressure and those methods which operate under a vacuum such as the light absorption on mass spectrometer detectors. For a pumping speed, S, the partial pressure is given by

$$V\frac{dp}{dt} = Q - PS ,$$

which reduces to

$$P = \frac{Q}{S}\left(1 - \exp\frac{-St}{V}\right)$$

For fast pumping speeds and small volumes S/V is small and P quickly approaches Q/S so that the partial pressure of the tracer gas depends only on the size of the leak and the pumping rate. Thus any fixture which is placed around the test object should be as small a volume as possible. When the round is removed, the tracer partial pressure is given by

$$V\frac{dp}{dt} = -PS$$

which reduces to

$$P = P_o\, e^{\frac{-St}{V}}$$

The quantity V/S is a measure of the minimum time necessary for detection and also time for clean up to remove any residual gas before retesting.

Another consideration for selection of a particular method over another is the cost of the tracer gas used. Relative costs of the primary gases being considered for detection are

• Mass spectrometer	He -	\$0.15/cu ft
• Infrared detector	CO_2	\$0.04/cu ft

- Halogen detector CCl_2F_2 \$0.41/cu ft
- Electron Capture SF_6 \$1.30/cu ft

  Ar/CH_4 \$0.27/cu ft
- Other gases N_2 \$0.09/cu ft

Although these costs may be reduced by mixing with less expensive gases, this will increase the sensitivity needed by the same order of magnitude as the reduction in concentration.

One consideration in the loading of the tracer is the possibility of introducing and sealing the tracer gas at a pressure higher than that generated by the prossing operation indicated in table 1. Although Q is proportional to P^2 and an increase in P would increase Q and therefore lower the sensitivity requirements, this increase would introduce additional hazards in a storage environment where rises in the temperature would generate even greater pressures.

2.3 Compatability Tests. Of prime concern to the successful use of a tracer gas for leak detection is the compatability between the tracer gas and the white phosphorus. Current use of CO_2 in the loading of WP munitions indicates its obvious compatability. In order to give other applicable techniques of leak detection any further consideration, preliminary chemical compatability tests were made. Those gases considered were dichlorodifluoromethane (Freon 12) and sulfur hexafluoride (SF_6). A schematic of the test apparatus is shown in figure 3. In the preliminary tests liquid White Phosphorus was dropped under vacuum onto a hot plate to maintain its molten state and expose it to any environment inside the test chamber. The tracer gas was then introduced at atmospheric pressure. The compatability of the WP and the gas was monitored by visual observation of the WP and the atmosphere inside the test chamber as additional heat was supplied to the WP. These tests showed no detectable reaction between the WP and either dichlorodifluoromethane or sulfur hexafluoride.

3.0 RESULTS

The survey of commercially available leak detectors has found approximately fifty manufacturers of leak detectors which may be classified according to eleven different categories depending upon the basic physical and chemical principles of their operation. They have been analyzed and compared primarily on the basis of their maximum sensitivity and capability for automated operation on a rapid production line. Little attention has been given to the material handling or actual testing setup of the detector or munition since the actual configuration of the production line, space available, or mechanical restrictions are undefined and outside the workscope of this report. Details of detectors characteristic of individual manufacturers not specifically germain to this application, such as, material handling features have not been explicitly reported since in all cases actual application will necessitate modification of existing apparatus. Chemical compatability tests have been performed on specific gases to determine their eligibility as tracer gases for use in conjunction with specific leak detectors.

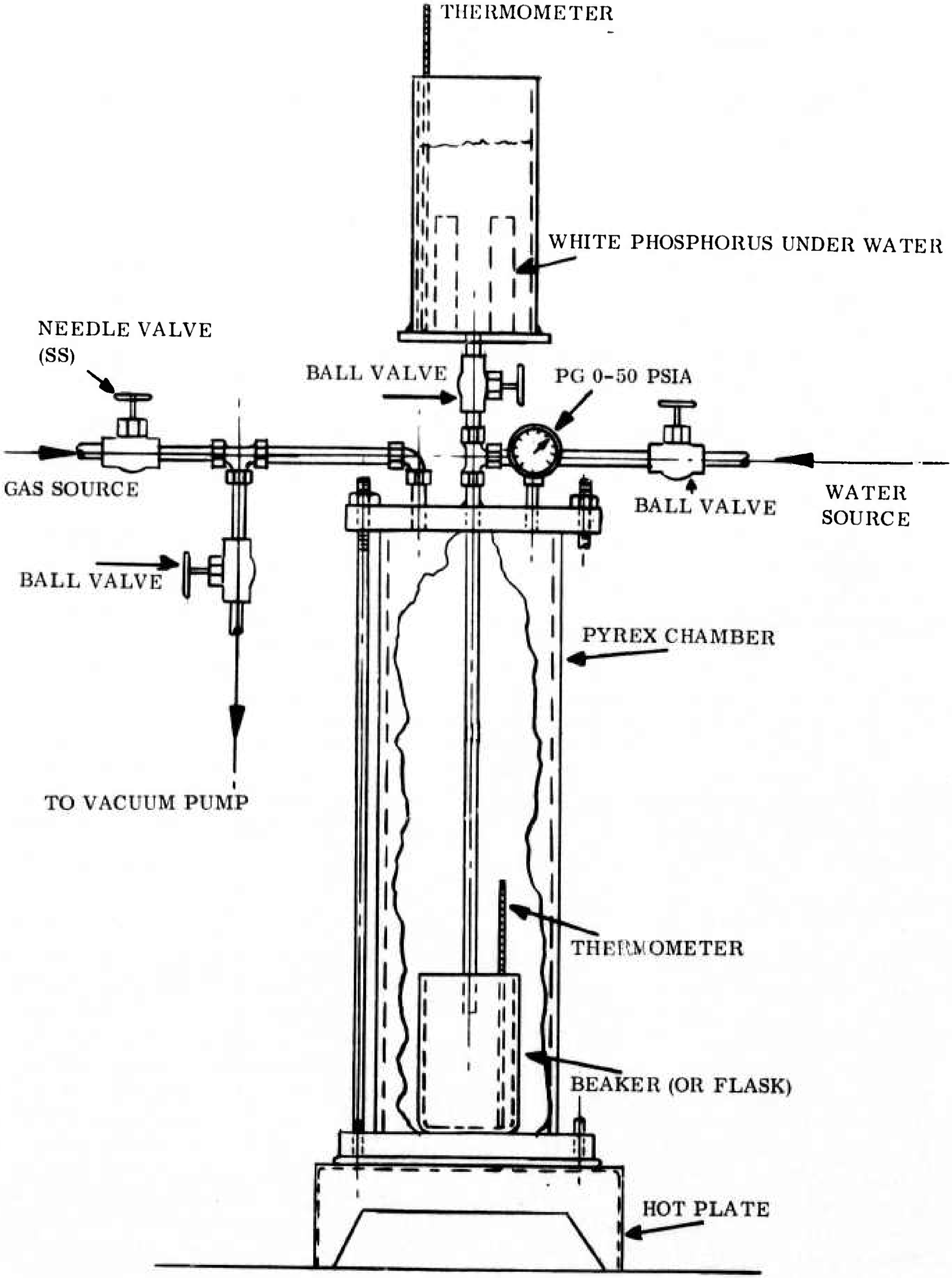

Figure 3. Test Setup

4.0 CONCLUSION

Comparison of evaluation criteria and leak detector characteristics on the basis of their sensitivity and capability for automated monitoring indicate four types of detectors appropriate for application to the WP production line. They are:

- Heated anode detector (Halogen tracer)
- Electron capture detector (Sulfur hexafluoride tracer)
- Infrared Absorption detector (carbon dioxide detector)
- Mass spectrometer detector (Helium tracer)

The remaining methods were rejected on the basis of insufficient sensitivity, use of hazardous tracer gases, and inability to apply to automated production line operation.

Each of the above methods are applicable to automated production line operation. The halogen and SF_6 are primarily manual-operated units but may be easily modified for automated operation at atmospheric pressure using a hooded test vessel for trapping or collecting escaping tracer gases. Although the IR detector and mass spectrometer may be operated in the sniffer mode, they are most efficient when the test item is at least under a partial vacuum. Therefore, these detectors require a vacuum system for their operation.

An additional operational consideration is the complexity of their operation. This may be interpreted as complexity of interpretation of when a leaker is present or complexity of maintenance. As far as complexity of interpretation is concerned each detector is equipped with an audible alarm when tracer gas is present above a prescribed level. This level is dependent upon the particular background characteristics of the filling line. The complexity of maintenance will in general be directly proportional to cost.

In the order of their sensitivity to their particular tracer gases, the mass spectrometer is the most sensitive. The SF_6 and Halogen detector have comparable sensitivity and are about an order of magnitude more sensitive than the IR detector.

Each of the four types of detectors have fast response times on the order of a few seconds with recovery from 100 percent overload or saturation in less than 10 sec. This recycling aspect of the recovery time depends upon the testing consideration such as the time required for the pumping system to clear any tracer gas before the next item is tested.

Each type of detector uses a different tracer gas. The costs of these gases are one consideration which has been summarized in table 2.

A factor affecting this consideration is the possibility of diluting the gas and thereby increasing the sensitivity needed or reducing the cost such as in the case of the mass spectrometer or SF_6 detector, respectively. An additional factor affecting the use of helium as a tracer gas is its tendency to rapidly diffuse. Although, this increases its ability to find extremely small leaks, it also affects its ability to form a blanket over the WP or remain in

Table 2. Characteristics of Applicable Leak Detector Techniques

	HEATED ANODE	ELECTRON CAPTURE	IR ABSORPTION	MASS SPECTROMETER
Tracer Gas	Halogen (Freon*)	SF_6	CO_2	He
Sensitivity (Maximum) atm cc/sec	$10^{-7} - 10^{-8}$	$10^{-7} - 10^{-8}$	10^{-6}	$10^{-9} - 10^{-11}$
Operation	Operating life of sensor 1000 hr Does not require vacuum	Requires Argon/ Methane currier gas Does not require vacuum system	Uses partial vacuum Includes pressure sensor for detecting gross leaks	Requires vacuum system Unknown amount of He remaining during sealing
Cost	Under \$2,000 per unit Sensor Element ≈ \$100 Freon ≈ \$0.41/cu ft	Under \$3,000 per unit SF_6 ≈ \$1.30/cu ft Ar/CH_4 ≈ \$0.27/cu ft	Detector unit about \$15,000 CO_2 ≈ \$0.04/cu ft	Basic mass spectrometer and vacuum system cost about \$8000. Helium ≈ \$0.15/cu ft
Supplier	General Electric Co., Ion Tract Instruments, Inficon	Ion Track Instruments, Varian Assoc.	Modern Controls	Aero Vac Corp., Dupont Instruments, Edwards High Vacuum, Inc., Varian Assoc., Veeco Instruments Inc.

*Use of tradmark name not imply endorsement only refers to most common name of class of halocarbons.

the head space before sealing. This factor cannot be effectively evaluated without actual tests however.

The remaining major point of comparison between these detectors is their cost. In some instances it has been difficult to divorce the cost of the actual detector from material handling aspects which have been factored into the most appropriate type detectors. Therefore, the detectors have been grouped into three categories: Below \$5,000 between \$5,000 and \$10,000 and above \$10,000. Variations in cost of up to \$2,000 exist depending upon manufacturer and special features. Table 2 lists the approximate costs of these detectors. As previously mentioned, the material handling and toolings costs have been factored out as well as possible to present these detectors on an equal footing. It is estimated that these costs will be between \$10,000 to \$100,000 depending upon the multiple item handling features.

Existing infrared scanners were also considered for application to leak detection of WP munitions. Their unique capability of high resolution temperature measurement enable them to locate leaks by the presence of thermal gradients around a leaking hot gas. However, no suppliers were found which have detectors applicable to automated testing. Mention is made here as a point of information as to their value in monitoring liquid level inside the munition after sealing to ensure the presence of a head space.

No leak detection devices were found from commercially available sources which could be easily modified for selectively monitoring the presence of WP or P_2O_5.

The methods of leak detection mentioned above are effective in detecting leaks in the WP munition which are above the level of WP in the munition. Leakage due to defects in the burster well, in particular the part thereof which is in contact with the WP, cannot be detected by any of these methods. If this area is considered a possible or probable source of leaks, additional leak detection tests on the burster well prior to sealing is recommended.

5.0 RECOMMENDATIONS

It is the recommendation of this study that evaluation tests be performed on actual WP munitions using the recommended methods of leak detection to verify their snesitivity and to determine the method of testing most appropriate for the WP munitions.

It is further recommended that such tests be performed in conjunction with the existing oven test and on the same munitions to determine the correlation between the different test methods.

NOTE

Use of manufacturers names and model numbers are not meant to imply endorsement but only to indicate possible and by no means only source of supply or additional information.

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