The POOR MAN'S PRIMER MANUAL

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by

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Warning

The manufacture and possession of explosives such as those used in primers is illegal without proper authority and licenses from the Bureau of Alcohol, Tobacco and Firearms. Do not use this book to create the described explosives and primers without licenses and authorization from the afore mentioned agency. Further, authorization for the proper agencies on the local and state level may be required.

The procedures in this book are dangerous and result in products that are extremely dangerous. Whenever dealing with explosives of any type, all precautions as set forth in industry standards for experimentation and production of high explosives should be strictly adhered to. Failure to strictly follow the industry standards may result in either physical injury or death. Therefore, the information in this book is presented solely for academic purposes only. Neither the author, publisher nor distributors of this book assume any responsibility for the use or misuse of information contained herein.

Introduction

The present handbook is intended for use by the students of priming mixes used in small arms primers. It is a compilation of information available from a variety of unclassified and publicly available publications, such as books, periodicals, government specifications, patents and manufacturer's catalogues.

The majority of printed data relating to the priming mixes is intended for the research and development community of chemists, physicists and engineers involved. The presentations assume that the reader has a thorough base of college courses in chemistry, physics and/or engineering. These texts focus on the details of chemical reactions of the materials involved, in their crystalline structure, reactions under a variety of physical conditions and stimuli. Such information is most important and necessary in the design of new and/or better mixes as well as to the engineer involved in the selection and operation of production equipment. The explosive characteristics of the materials involved are discussed at length.

Regretfully, the attention given to the actual preparation of such materials and / or mixes is considered almost secondary. Most texts describe the laboratory preparation of priming mix components with a single concise paragraph at best. The preparation processes are brief, sometimes incomplete and generally leave many questions unanswered. One of the objectives of this volume was to collect and study the process descriptions from different sources. This published information has been digested and presented in a format familiar to the students of high school chemistry courses. Each primary explosive preparation is presented as a separate and independent chapter. Where suitable and possible, graphs and flow charts have been used as a substitute for actual text.

Each of these priming mix chapters contains pertinent data on equipment, materials, safety precautions, preparation procedures and quality control information. Only the tests have been consolidated under a separate heading. This approach allows for a quick and easy preparation of a given priming mix.

The present material is devoid of chemical reaction equations, thermodynamic calculations of decomposition reactions,etc. It contains only information on the safe and practical preparation of the various priming mixes using the simplest and readily available equipment.

The book is further restricted to primers of percussion type,as used in small arms. Electric primers used generally in crew-served and or on-board automatic weapons are a specific area of interest.

While there are only six basic percussion primers in use throughout the world, there are dozens of electric primers for different cartridges and weapons. To discuss them in detail is beyond the scope of this book.

The present text describes not only the laboratory preparations, but also the "one-pail" size operations which are suitable for small and medium (approximately 2 million primers per week) size production lines. It must also be pointed out very emphatically that extrapolating the processes from "one-pail" size to larger vessels should not be considered. Such escalations of reaction vessels require knowledge of heat transfer, reaction time, and other special considerations.It is due to the relation between the surface area and volume of each individual piece of equipment.For example:

The volume of a glass beaker is $\frac{3.14 \times 4'' \times 4'' \times 4''}{4} = 50.24$ cubic inches (823.3 ml)

the surface area of the beaker in contact with the work contents is

$$\frac{\pi D^2}{4} + \frac{\pi DH}{4} + \frac{3.14 \times 4 \times 4}{4} + 3.14 \times 4 \times 4 = 62.80 \text{ square inches } (405 \text{ cm}^2)$$

thus every one square inch of surface has to heat or cool 0.8 cubic inches of work material (1 cm^2 to 2.03 ml).

If the laboratory system is increased to a level of a stainless steel pail (generally used for many operations),the relationship between work volume and the available heating-cooling surface of the pail becomes:

$$\text{surface } \frac{\pi \times 10'' \times 10''}{4} + \pi \times 10' \times 7'' = 78.5 + 220 = 300 \text{ in}^2 \ (1935 \text{ cm}^2)$$

and the respective volume of work will be

$$\frac{\pi \times 10'' \times 10''}{4} \times 7'' = 550 \text{ cubic inches } (8935 \text{ ml})$$

Now each square inch of the pail surface heats/cools 1.8 cubic inches of the work material (1 cm^2 for 4.62 ml). This requires correspondingly greater capacity of the heating/cooling liquid (water) to maintain the proper operation cycle. This "one-pail" may be considered the practical safe limit of the off-the-shelf equipment. Further increase in the reactor volume requires specially designed units capable to maintain the required temperature control of the whole volume, not only the layer adjacent to the vessel surfaces. Such large capacity vessels-reactors are usually jacketed, with additional internal coils, baffles and stirrers designed to expose all contents uniformly to rapid heating/cooling cycle.

Another alternative is to use multiples of "one-pail" units. This approach was in use since the very beginning of the Fulminate of Mercury production. The "one-pail" method and its multiples,is also suitable for small and medium size marufacturing plants,as inexpensive and flexible.

However,for large, particularly military, plants with large volume of only limited types of primers production, the continuous process methods may be preferable. Although their initial cost may be high, they pay for themselves in relatively short time by substantial personnel and work area reduction. Furthermore, they contain only a small volume of in-process materials (explosives) and may be remotely controlled, which reduces personnel hazards considerably. These features not only increase the safety but also allow for wider choice of plant location.

Interestingly, the technicians and workers in the priming mix production sometimes do not have any chemistry schooling at all. Yet they have been trained to perform the work required. The essential prerequisites for preparation of the priming mixes are: absolute adherence to the safety instructions, full and undivided attention to the work performed, healthy respect for the materials to be handled.

COMPLACENCY HAS NO PLACE IN THIS BUSINESS

It is hoped that the information provided will assist the students and workers in their efforts.

It is customary to devote a separate chapter at the beginning of most technical texts to the history and development of the subject under discussion. The author feels that the story of priming mixes and the small arms primers has been presented in so many books and so eloquently, that is would be presumptuous on his part to try it. Instead the following is a short list of books describing the development of the priming mixes and their application, providing an ample background:

United States Firearms,The First Century;Butler,David F.,Winchester Press ,NYC, 1971

Early Percussion Firearms;Winant,Lewis, Wm. Morrow & Co., NYC, 1959

Chapter 1

General Primer Data

The primer is considered the most critical component of a small arms cartridge. It is located in the base (head) of the cartridge case; held by friction and/or interference fit, and in the military cartridges, also by crimping in place.

With a few exceptions, the small arms cartridge primers are of the percussion type. This means that the primer is actuated by impact of the firing pin (striker) on the primer cup.

During this impact the cup is deformed and the priming mix retained between the cup and the anvil is ignited. To be considered a good primer, it must satisfy the following requirements:

1- Reliability - A primer should produce reliable ignition of the cartridge propellant under the adverse conditions of extreme ambient temperatures from - 40°C to +55 °C.
2 - Uniformity - The propellant ignition should be uniform to assure uniform muzzle ballistics.
3 - Sensitivity - A primer should conform to the standards of sensitivity to firing pin impact established by the weapon and ammunition designers and users (military).
4 - Clean burning - The priming mix should burn without harmful corrosive residue affecting the weapon and to a lesser degree the cartridge case (important for reloaders).
5 - Stability - The primer must retain above performance characteristics under storage for extended periods of time at varying temperatures. This is important to the manufacturer and the end user, so they do not have to worry unduly about inventory and stored product turnover.
6 - Safety last, but not least, the primer must be safe to reasonable handling during manufacture, cartridge loading and packing, and in storage.

The small arms percussion primers are of three basic types:

1- Berdan (anvil integral with the cartridge case)
2 - Boxer (anvil seated in the primer cup)
3 - Shotshell (primer cup and anvil inside a battery cup)

These primers are described on the following pages:

Berdan type primers are without integral anvil. The anvil in this case is an integral part of the cartridge case and is located at the bottom of the primer pocket.

The Berdan primer pocket usually has 2 small-diameter flash holes. The theoretical advantage of this system was:

a - Simple primer construction and manufacture with inherently less chance for defectives.
b - Positive location of the anvil.
c - Smaller flash holes preventing fractured powder grains from entering into the primer pocket.
d - Difficulty in reloading of fired case.

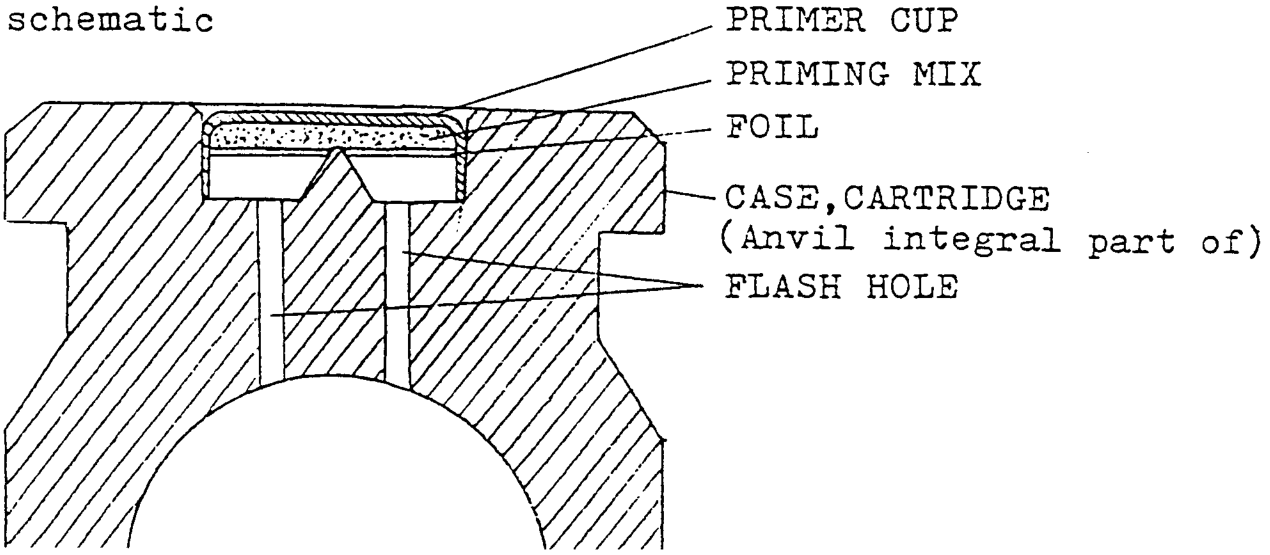
The last item is of importance in areas where ammunition is under strict government control.

This type of primer is prevalent in Europe.

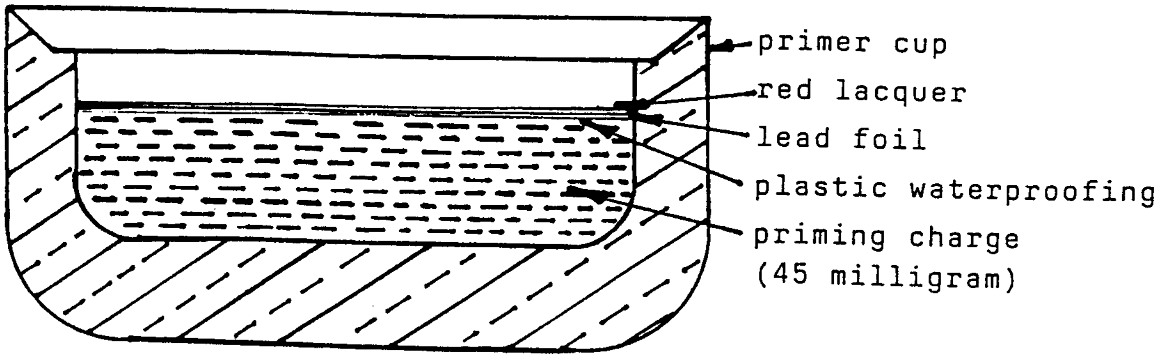
Boxer type primers have the anvil as an integral component of the primer itself. The cartridge case, has only a simple pocket with a single, relatively large, centrally located flash hole.

PRIMER,PERCUSSION,
BERDAN TYPE

schematic



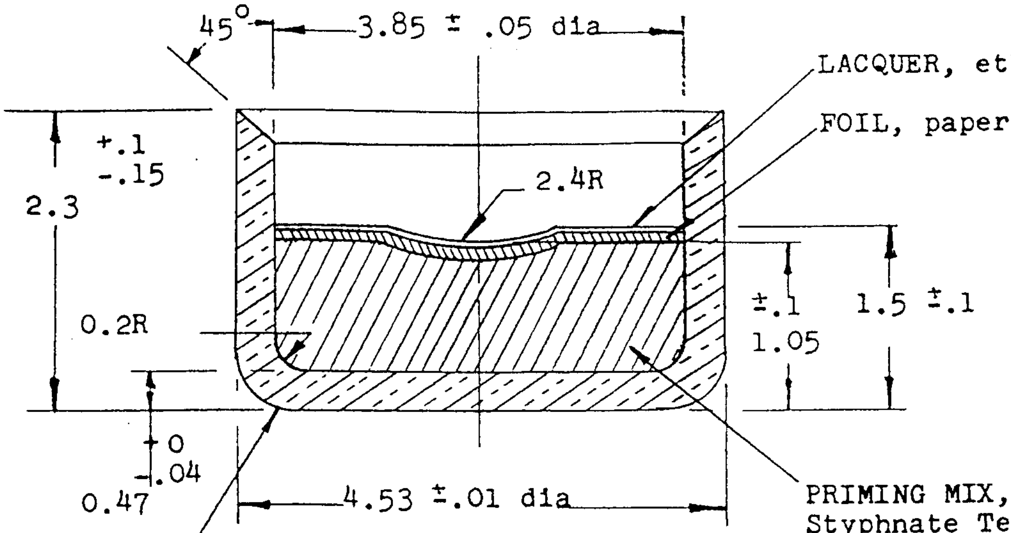
BERDAN TYPE PRIMER
LAPUA MANUFACTURE
FOR 7.62 x 39 CARTRIDGE



dry charging priming mix composition:

Lead styphnate
PbO_2
Sb_2S_3
$Ba(NO_3)_2$
Ca-silicide

BERDAN TYPE,
SMALL PISTOL SIZE

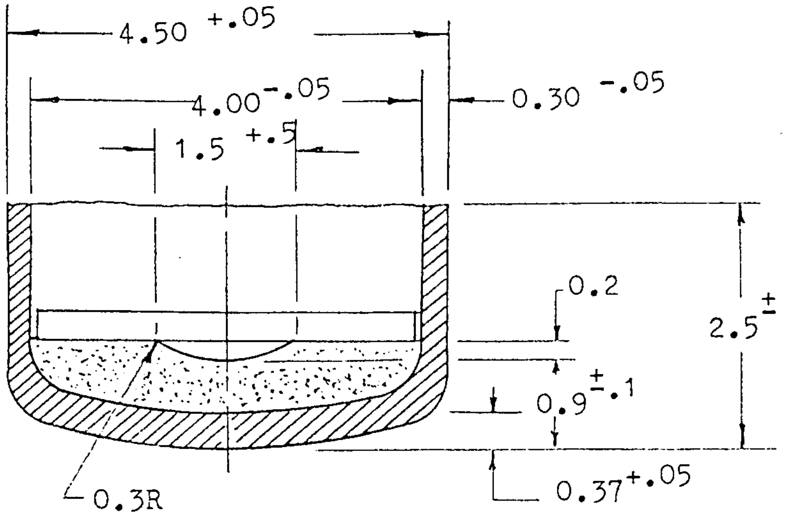


LACQUER, ethyl cellulos

FOIL, paper, .15-.20 thick

PRIMING MIX,Lead Styphnate Tetrazene type charge, nominal:
21 ± 2 mg
compressed with 70Kg dead load

PRIMER CUP, cartridge brass alloy,
bottom hardness (Vickers diamond pyramidal,
130 with 2.5 Kg load)
side hardness,150 max.



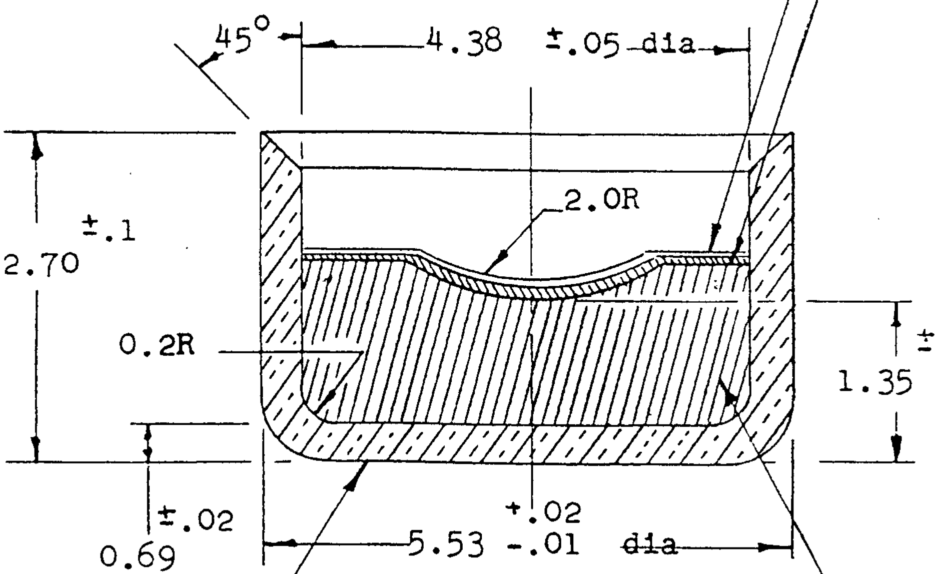
PRIMER CUP,
cartridge brass

FOIL,PAPER
.15 thick

PRIMING MIX
Lead Styphnate-
Tetrazene type

Note: dimensions are in millimeters

PRIMER,PERCUSSION,
BERDAN TYPE
LARGE RIFLE SIZE



LACQUER, ethyl cellulose

FOIL, paper, .15-.20 thick

PRIMING MIX, Lead Styphnate
Tetrazene type,
charge, nominal: 39 - 3 mg
compressed with 70 Kg dead
load

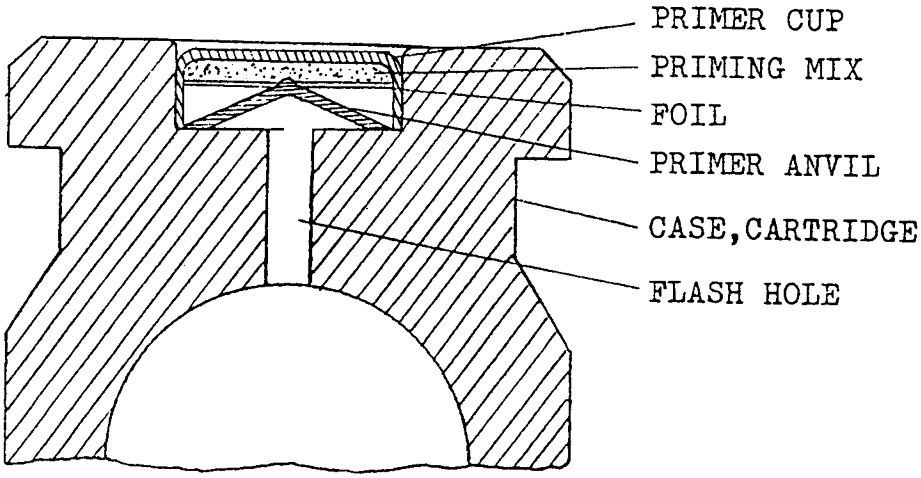
PRIMER CUP, cartridge brass alloy
hardness (Vickers diamond pyramidal,
with 2.5 Kg load)
bottom : 130
side : 150 max.

Some of the advantages of this system are:

a – Anvil and primer cup are consolidated while priming mix is wet and relatively insensitive.
b – Cartridge case construction and manufacture is simplified.
c – Cartridge case is easily reloadable, by punching out fired primer.

The boxer primer has demonstrated its reliability in billions of military and commercial cartridges, thus nullifying the principal theoretical advantage of the berdan primer. The boxer primer is used and preferred in the USA.

PRIMER, PERCUSSION,
BOXER TYPE
schematic



Battery cup primer is used almost exclusively in shotshell cartridges. The usual shotshell case is made from paper and/or plastic material. The case head is reinforced with a thin metallic cup, holding the case body and the base wad together. It is needed also for positive extraction of fired case from some shotguns.

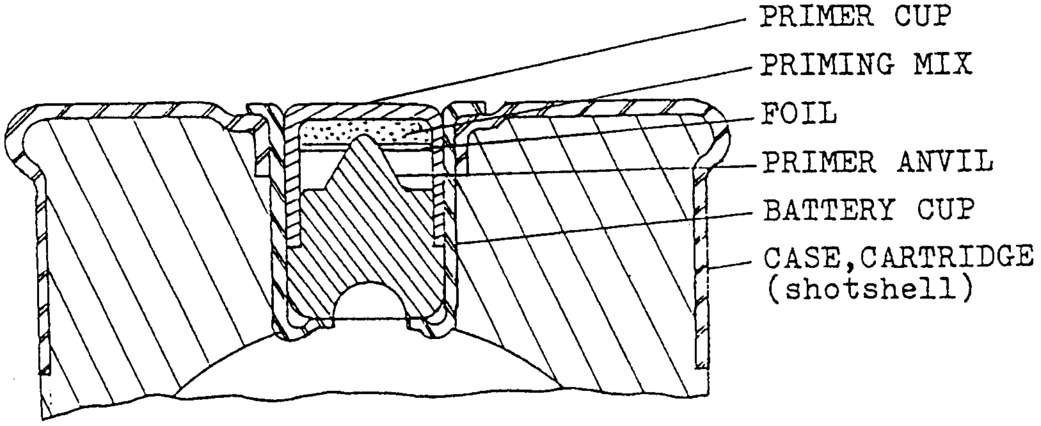
Since the primer needs a solid, non-elastic surface to rest on, for positive ignition, a small metallic piece is added to the primer assembly. This piece is called "battery cup".

In the past, there were several types of battery cup primers, but at present the 209 Winchester type is used almost exclusively in U.S., Asia and Europe.

The shotshell primers may have the anvil as a separate piece, or as an integral part of the battery cup. As with the boxer and berdan primers, both types of shotshell primer have some advantages and disadvantages.

PRIMER, PERCUSSION,
BATTERY CUP TYPE

schematic (in shotshell)



For reliable and safe performance, regardless of the type, the primer must be seated in the cartridge case quite tightly and rigidly. Loose primer, when impacted by the firing pin, may bounce back and/or "give" without ignition. Even if ignition occurs, the priming mix flame and gases will leak around the cup back into the action, and the propellant in the case may not be ignited. Thus it is extremely important that the primer fit the case correctly and is seated against the priming pocket bottom.

All these conditions require close tolerances, both of the primer and the cartridge case primer pocket. The following tables give data as established in the industry.

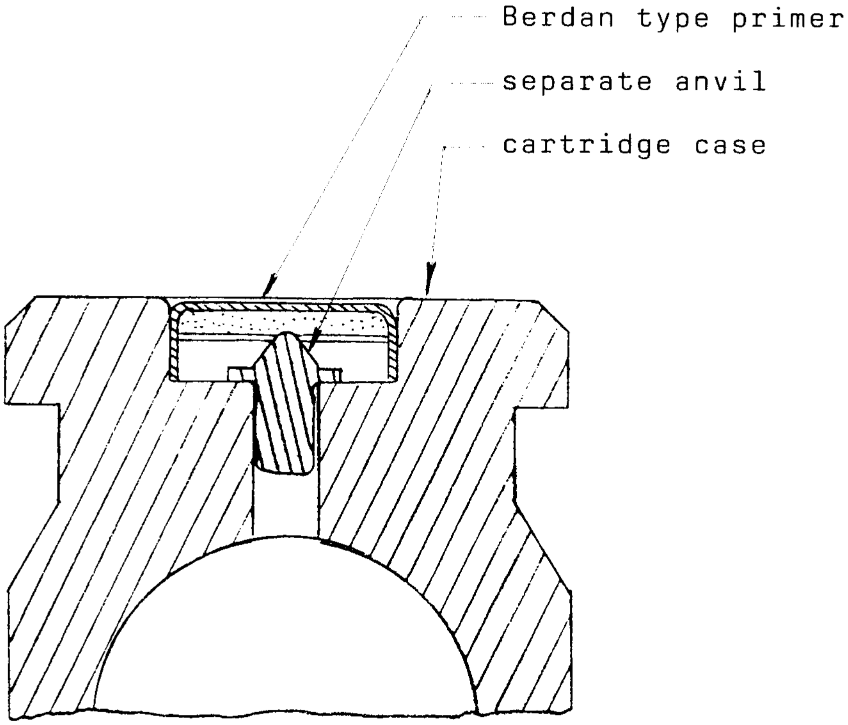
There is a third primer type which was patented years ago. This system used a Boxer type (no anvil) cartridge case with a separate anvil and a Berdan type (no anvil) primer. This priming system had the following advantages:

a – Using a cartridge case with a central flash hole and no anvil integral with the case allows for a simpler case manufacture.

b – Use of Berdan type primer allows a larger priming mix charge which is particularly important in large capacity cartridges (Magnum type); and in cartridges with small diameter cases needing small size primers.

However, the classic Berdan and Boxer primers have been made for generations and there is not an urgent need for other types. Furthermore, the seating of this new type anvil would require modifications to existing cartridge loading machines with extra stations for orienting, seating and inspection of the anvil.

This priming system is illustrated below:



Through the years the percussion primers in the U. S. A. have been standardized into the following sizes:

Small pistol
Large pistol
Small rifle
Large rifle
Battery cup shotshell
.50 cal. (for military use)

The following table lists some of the cartridges and their respective primers:

Primer type

SMALL		**LARGE**	
PISTOL	**RIFLE**	**PISTOL**	**RIFLE**
.25ACP	.22 Hornet	.30-40	.243 Win.
.32ACP	.222 Rem	.44-40	.250 Sav.
.32 Colt	.25-20	.44 Spl.	.270 Win.
.357 Mag	.32-20	.44 Mag.	7x57 Mau.
.38 Spl.	.30 Carb.	.45 Colt	.30-30
.380 Auto	5.56x47	.45 ACP	.30-06
.30 Mau.			.300 Mag.
9mm Para.			7.62x51
.41 Colt			.375 H&H
			.458 Win.

Note: all shotshell gauges, from .410 through 10-ga. use one size battery cup primer.

PRIMER, PERCUSSION, SMALL ARMS CARTRIDGE

TYPICAL COMMERCIAL SPECIFICATIONS

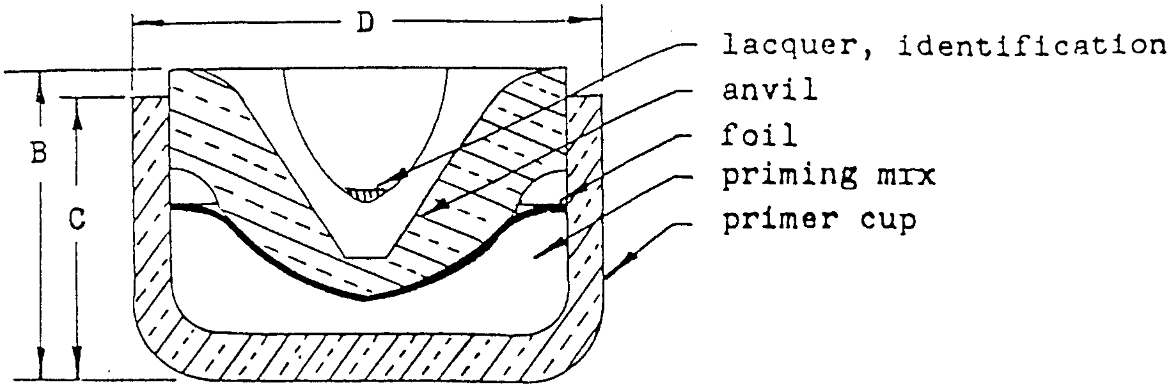
PRIMER SIZE		LARGE		SMALL	
		rifle	pistol	rifle	pistol
DIMENSIONAL - mm					
Diameter - D		5.38+.01	5.38+.01	4.47+.01	4.47+.01
Cup height - C		3.12+.07	2.92+.07	2.92+.07	2.92+.07
Unseated height - B		3.30+.05	3.07+.05	3.07+.05	3.07+.05
PRIMING MIX PELLET WT - mg					
Individual		38.90±4.85	29.80±2.80	22.65±2.80	22.35±1 .90
Avg. of 10		37.25±1.65	28.85±0.95	21.70±0.95	21.70±0.65
Median of 7		38.90±2.60	29.80±1.45	22.65±1.45	22.35±1.00
Median range of 7		23.00			
Median of 25		38.87±1.92			
Median range of 25		7.20			
SENSITIVITY					
(When seated in cartridge case)					
Drop ball weight - gram		103.4	56.7	56.7	56.7
Ball drop height - mm					
100%	rimmed case	275	250	325	255
	rimless case	305	290	335	205
H + 5 5	rimmed case	300	290	375	286
	rimless case	350	325	380	235
H - 2 S	rimmed or rimless case	75	75	75	50

STABILITY IN STORAGE: 1 year at 70°C in dry air or 46° C at 85% relative humidity

Notes: 1) The 5.56 x 45 military cartridge has a 100% drop sensitivity, using a 103.4 gram ball with 305 mm drop 2) the higher sensitivity values for rimless cases are due to account for the case shoulder cushioning effect.

PRIMER,PERCUSSION,
SMALL ARMS CARTRIDGE

SCHEMATIC ASSEMBLY



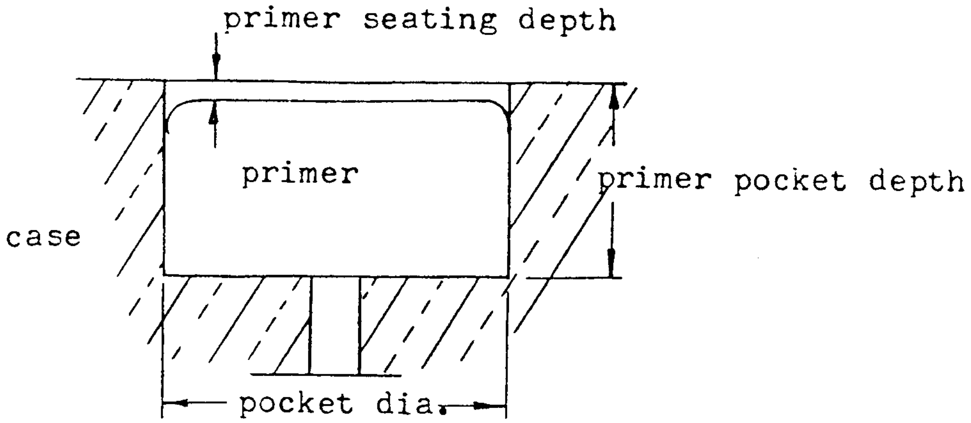
PRIMER ,PERCUSSION 50 cal. Browning cartridge M36A1

DIMENSIONAL - mm	
Diameter - D	8.06 - .03
Cup height - C	5.47 + .14
Unseated height - B	7.11 + .20
PRIMING MIX PELLET WT.-mg	
Individual	110.0 + 26.0
Median of 7	122.0 + 12.0
SENSITIVITY	
(When seated in cartridge case)	
Drop ball weight - gram	207.0
Ball drop height - mm	
100%	380
H + 5 S	455
H - 2 S	75

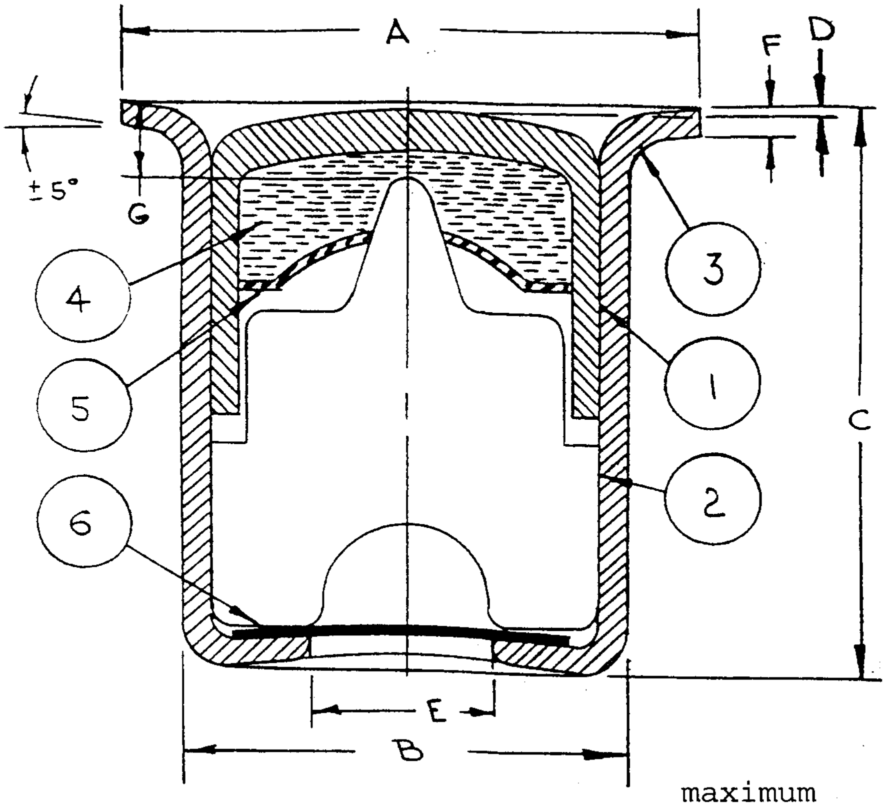
STABILITY IN STORAGE: 1 year at 70°C in dry air or 46°C at 85% relative humidity.

CARTRIDGE CASE PRIMER POCKET
for Boxer type primers

PRIMER SIZE	PRIMER POCKET DIMENSIONS (in millimeters) DIAMETER	DEPTH	PRIMER SEATING DEPTH
Small pistol	4.41 +0.02	2.96 +0.08	0 +0.15
Small rifle	4.41 +0.02	2.96 +0.08	0 + 0.15
Large pistol	5.31 +0.02	2.96 +0.08	0 +0.15
Large rifle	5.31 +0.02	3.22 +0.008	0 + 0.15
.50 cal. Browning	8.01 +0.02		0 +0.15



General Primer Data



1 - primer cup
2 - anvil
3 - battery cup
4 - priming mix
5 - priming mix foil
6 - battery cup foil

Note: for best results seat primer in cylindrical pocket 6.04 - 6.07 mm diameter solid aginst flange.

	maximum specification	acceptance specification
Pellet weight - mg avg. of 10	54.40 ± 4.00	54.00 ± 1.50
Pellet height - mm		1.90 ± 0.10

Sensitivity (56.7 gram steel ball)		drop height - mm
100 %	steel die	203
	shotshell case	255
H + 4S	steel die	191
	shotshell case	
H-2S	steel die	51
	shotshell case	51
0%	steel die or shotshell case	51

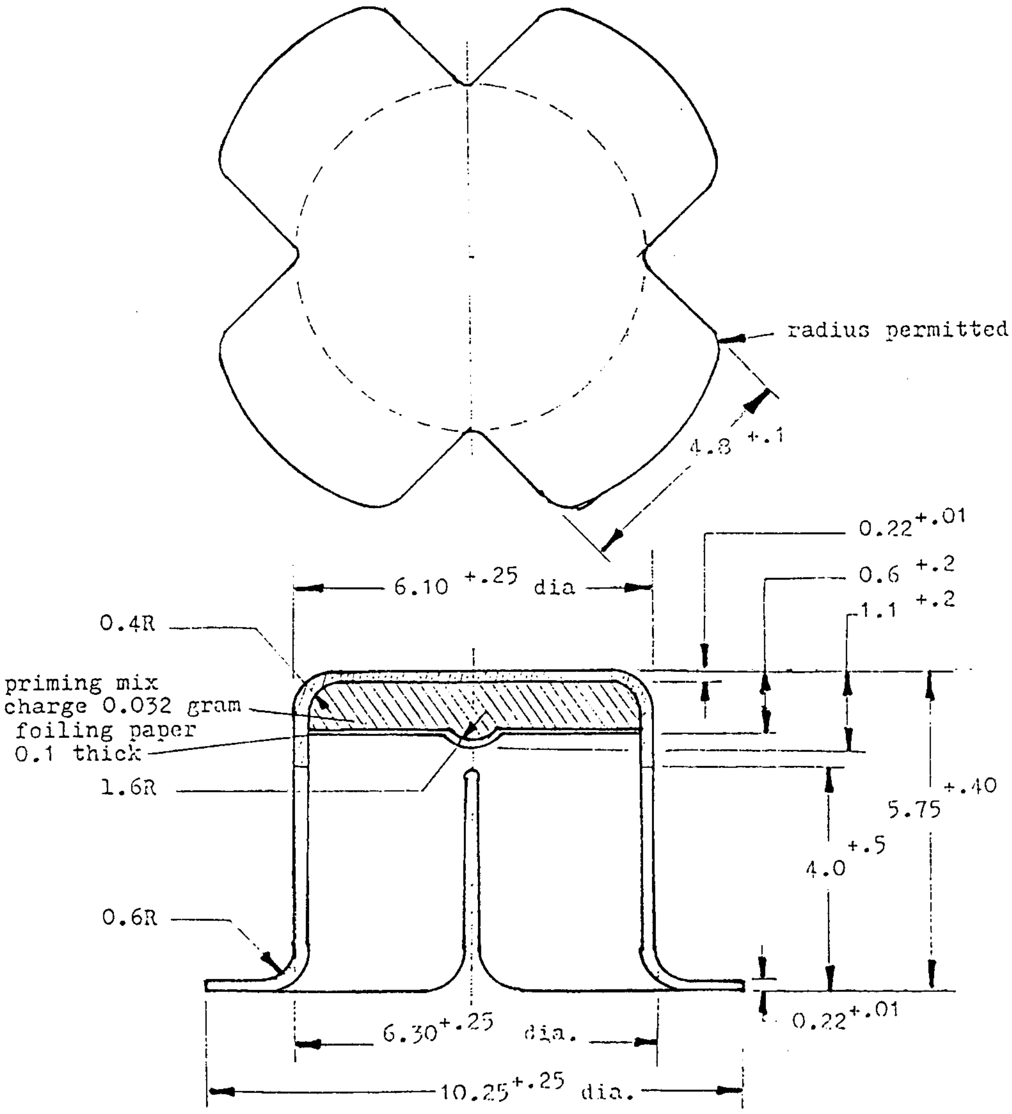
Dimensions - mm	maximum specification	acceptance specification
A	7.97 ±0.25	7.97 ± 0.15
B	6.10 ±0.03	6.09 ± 0.017
C	7.72 ±0.10	7.69 ± 0.05
D	0.12 ± 0.12	0.15 ± 0.05
E	2.95	
F	0.30 ± 0.12	0.18 ± 0.02
G	0.95± 0.06	

Stability - 1 year at 70°C dry air or 46°C and 85% relative humidity

No primer discussion would be complete without mentioning the original percussion cap, theoretically obsolete, yet revived by the increased interest in the muzzle loading black powder arms.

The percussion cap, from which the modern primer evolved, is a thin copper, or brass cup filled with mercury fulminate or chlorate base priming mix. It is not seated in a cartridge case (there is none), but on the nipple of the muzzle loader arm. It is fired by the impact of a hammer, which compresses the cap and mix against the nipple rim. The flame and combustion materials of the ignited mix are carried through the nipple flash hole to the black powder charge in the barrel.

Following is a drawing of a typical musket (rifle) percussion cap:



Percussion primer manufacture may be divided arbitrarily into three semi-independent phases:

1 - Metallic components manufacture, i.e. primer cups, anvils, and battery cups.

2 - Priming mix manufacture.

3 - Primer loading, assembly and racking.

Each of the above phases, although relating to the same product, is actually a completely independent discipline.

The manufacture of primer cups, anvils and battery cups, is exclusively a metal forming process, requiring knowledge of presses, dies and punches, annealing and washing techniques, lubricants, etc. It is a field of metallurgists and mechanical engineers, with emphasis on mass production of small, highly accurate metallic components. It is comparable to processes used in the watch and electronic industries.

The principal metallic components of a small arms percussion primer are the cup and the anvil. In the berdan type primer the anvil is separated from the cup and is an integral part of the cartridge case. In the 209 type shotshell primer the battery cup is an additional component holding the primer cup and the anvil. The battery cup not only acts as a container, but also assures a positive surface for the components to rest on. The battery cup creates a more rigid support for the primer and an improved uniformity for the primer ignition.

The primer cups are invariably made from brass, mainly to prevent reaction between the wet priming mix and the cup material. Furthermore, brass is easily formed and soft enough not to cause excessive wear of production tooling.

In a large volume production the primer metallic components are made from a strip of the appropriate metal/alloy fed automatically into a high speed press (over 100 strokes/minute). The actual forming is done in a block containing 3-6 or even more individual dies and punches. Thus a single press running at 120 strokes/mm (for example) with 5 sets of tools will make 600 components every minute of running. Operating this press one shift/year will produce 600 pcs/min x 50 mm/hr x 2000 hrs/year, almost 60 million pieces. This is an average production capacity for many ammunition plants. For laboratory purposes, requiring only a few hundred (or even thousand) pieces, the metallic components may be obtained preferably by purchase from the large manufacturer, or by reconditioning and cleaning fired primers. The tools and procedure for such operations have been described in detail in several publications devoted to survival, reloading ammunition and even government publications. The reconditioning requires only a couple of nails shaped with file/grinder into punches and a pair of needle nose pliers for handling the tiny anvils. The skills for this are acquired during the work.

More details on the metallic components are given in Appendix C.

The priming mix manufacture is, in contrast, a chemical operation with the added problem of handling sensitive and dangerous materials. All priming mix production methods are of the type commonly used in cosmetic, pharmaceutical and food processing industries.

Handling, washing, blending, weighing, and other operations are routinely performed by production and laboratory personnel throughout these industries.

Even the equipment used in primer manufacture is of the type and models used in these above fields.

In many respects some pyrotechnic operations in preparation of tracer and/or incendiary mixes, flares, etc. are potentially more hazardous than priming mix manufacture because they are handled in dry state and larger quantities, while the primer mix is essentially wet throughout the whole process.

The primer mix is made in two parts - the high explosive premix and the non-explosive premix.

The high explosive premix/mix is represented by Mercury Fulminate, Potassium Chlorate and TNT, or mechanical mixture of Lead Styphnate, Tetrazene and PETN.

The nonexplosive premix is a mechanical mixture of Barium Nitrate, antimony Sulfide and Aluminum; or other fuels and oxidizers.

The details of preparation of these mixes are in the respective chapters of the text.

The LAP (loading - assembly - packing) of small arms primers may be compared to other explosive and pyrotechnic operations in that a sensitive chemical composition is inserted into a small container and packed for additional handling. Although the priming mix is classified as a primary explosive, it is handled in smaller quantities at any one tine thus requiring different operating procedures than loading of tracer-incendiary projectiles, detonators, etc.

The detail equipment layouts for the primer charging and assembly depend on the type of priming mix used. Dry mixes are charged by remote control equipment behind barricades. The wet mixes are charged traditionally by hand. Either method uses a series of plates holding approximately 500 components each. Thus each sequential operation is performed on 500 pieces at the same time. Assuming that each operation takes about 30 seconds (actual production rate); the real production rate becomes:

1000 pcs/min x 50 mm/hr x 2000 hrs/shift-year 100 million primers per year; again this volume represents industrial capacity production. The sequence of operations for some primers is illustrated in Appendix B.

For small experimental laboratories a set of plates of approximately 75 x 75 mm (3 x 3 inches) holding 25 pieces each would be a convenient size. Such plates should be made from non-sparking metal (stainless steel, brass, aluminum), or even plastic and hardwood. The plates should be stacked into a "sandwich" and all holes should be drilled through. In this manner there would be a good alignment of the components during assembly.

The industrial operations use multiple plates of each type for steady production flow. However, for the laboratory a single plate for each operation is adequate. The necessary information for the design and preparation of the laboratory plates is given in appendix D.

Chapter 2

Priming Mix Compositions

An ideal priming composition would be :

a - Single chemical.
b - Safe and easy to load into cups.
c - Sensitive to friction and impact then assembled into primer.
d - Stable in storage, at high temperatures.
e - Non-hygroscopic.
f - Generate hot gases and/or particles to assure positive and uniform propellant ignition at low temperatures.
g - Powerful, thus requiring only a small individual charge.
h - Allow 100% function testing of non-destructive type.
i - Raw materials to be non-critical, readily available and inexpensive.

Unfortunately there is no single material satisfying all above requirements. The present mixes are a compromise favoring one or several critical parameters, while downgrading the others.

All priming mixes, regardless of the composition and materials used may be divided into two basic groups:

a - Dry mixes in which all mix components are charged into the primer cup in the dry (powder) form. The primer is then assembled with the other components in the appropriate manner.

b - Wet mixes in which the wet and dry components are blended into a wet/moist paste which is then charged into the primer cup.

The anvil and battery cup are then assembled with the wet charged cup into a complete primer.

The classic example of the dry mixes are those based on the Mercury Fulminate. Mercury Fulminate was the first successful primary explosive discovered and used. It is easy and relatively safe to prepare from readily available materials. It does not require any special equipment. It can be made using usual kitchen containers and sink. Yet it gave quite satisfactory performance and was used in small arms primers even in World

War II. However, the inherent danger of dry mix charging as well as the unwanted effect of Mercury Fulminate on brass cases encouraged research for other priming mixes. Furthermore, the key material used, namely metallic mercury, could not be produced locally in countries lacking in minerals of mercury compound. Thus the Mercury Fulminate was the first and probably the last example of dry mix .Yet because of its key characteristics of simple preparation and quite satisfactory performance it will be used for years to come.

The search for a perfect priming mix led to the first acceptable wet mix, the Chlorate priming compositions known in the United States as the Frankfort Arsenal H-48 and the #70. The H-48 was used until spring of 1917 when the Winchester priming mix NF47 was adopted by the U.S. Army under the name of FA#70. Additional historical information on these mixes is given in *Hatcher's Notebook.*

While these mixes allowed wet charging and safer handling, they did not eliminate the corrosion effect of the Potassium Chlorate. The search for a better wet mix led in the 1930's to the Lead Styphenate-Tetrazene mix currently used.

The wet priming mixes, as used now are not perfect but have the following desirable features:

1 - Simple operation - not requiring technically skilled personnel. Even the priming mix manufacture may be carried out by workers without special chemical education.
2 - Simple equipment - the equipment used is selected for ease of maintenance, disassembly, cleaning and reassembly. A maximum use of non—corrosive, non—sparking materials and explosion-proof electrical components has been applied. Aside from foiling and assembly presses, all units can be adjusted by the operators themselves.

3 - Minimum personnel - the fewer people exposed to the explosives during manufacture, the less chance of an accidental mistake and the less possibility of casualty. Wet mix allows the use of minimum personnel during each operation.

Under normal operating conditions a work team of four female operators and one charger-supervisor will assemble 20,000 primers per standard 50 minute hour. During a 40 hour week and 50 week work year, the production figure of 40 million primers per one shift-year is arrived. Such realistic figure has been achieved by the industry on a year-by-year basis.

To provide the priming mix for this volume of production requires 2.5 man-time on the same time basis as described above. Again, in practice a 3 operator team does the priming mix work and the duties of material handlers etc. Thus a work force of 8 people using wet mix accomplishes the work of 17-25 persons using the mercury fulminate, or other dry mix method.

4 - Process sheets, material flow charts and all equipment have been tested by experience in a variety of locations. Development is continuously carried out to

improve the wet mix system to reduce the personnel, while retaining and/or increasing the production rate, always maintaining the safety as prime requisite.

5 - General health safety practices used in the chemical and other industries are also rigidly followed.

6 - All phases of the operation are synchronized to keep the quantity of explosives at any one place to a minimum. The wet mix system is balanced so that each complements the next one and the production line does not need to receive shipments of, nor to store large quantities of incoming explosives. In an integrated line, only non-explosive materials are shipped from the outside vendors and stored in quantities necessary for continuous production. When PETN is used, it is received in wet state in quantities of 25-30 Kg and does not present a serious storage problem.

7 - All explosives are manufactured, tested, weighed, handled and stored in wet condition.

8 - The primer cups are charged and assembled into finished primers with wet mix. The possibility of an accident during the primer assembly is considerably reduced. In the event of a mix explosion during the cup foiling and/or anvil seating in the respective presses, the firing is limited to one primer only. A firing of one, or even several primers (usually intentionally during tests) does not damage the machine, nor the tooling. The operator notices such accident only by the noise of the firing.

9 - The primer becomes active only after storage in a drying room. Even here it is stored in such manner that the propagation of explosion from one primer to another is virtually eliminated.

Various priming mixes have been used and are still in use, based on Potassium chlorate, Mercury fulminate, and other primary explosives. However, experience has shown that for small arms primers, the Lead styphnate-Tetrazene wet mix is the optimum choice. While some mixes have more suitable characteristics for specific ignition devices, not subject to this discussion, the safety of the LS-T wet mix in manufacture and assembly of the primer itself are unsurpassed. It is mainly for this reason that the LS-T wet mix is used in all modern small arms primers.

The currently used wet priming mixes may be separated by the type of cartridge to be used in:

Rimfire
Small pistol (9 x 19 Para, .38 Spl., etc.)
Large pistol (.45 ACP, .44 Mag., etc.)
Small rifle (5.56 x 450
Large rifle(7.62 x 51)
Shotshell (battery type)
Large caliber ammunition (.50, 20MM, 30MM, etc.)

The percentage of the components of a specific mix may vary from manufacturer to manufacturer. The usual determining factor is the type and quantity of cartridge propellant to be ignited.

The following tables give formulations of different priming mixes. The percentages are in weight except for gum solutions which are in milliliters per 1,000 grams of mix.

Table 1

Mercury Fulminate base priming mixes

Material	**Percent by Weight (dry)**					
	a	b	c	d	e	f
Mercury Fulminate	50	50	28	27	37.5	14
Potassium Chlorate	25	-	14	37	37.5	41
Potassium Nitrate	-	25	-	-	-	-
Antimony Sulfide	25	25	21	29	25	33
Ground glass powder	-	-	35	6	-	11
Shellac (dissolved in ethanol)		-	2	1	-	1

Note: a – Italian military mix of WWII
b – French (military) mix of WWI
c – U.S. commercial shotshell mix of 1946
d – German
e – British
f – Austrian

The choice of formulation depends on the availability of raw materials (metallic Mercury), propellants to be ignited, size and characteristics of primer cup, etc.

The addition of glass to the formulations with reduced percentage of mercury fulminate is apparently needed to increase sensitivity of the mix.

The basic priming mix consists of:

mercury fulminate

potassium chlorate

antimony sulfide

The chlorate oxidizes the carbon in the compound to CO_2, increasing the completeness of the combustion and the explosion efficiency.

An 80/20 % pot. chlorate/antimony sulfide ratio is considered most efficient.

Table 2
Potassium Chlorate – Lead Thiocyanate base priming mixes

		Experimental	
	FA #70	**A**	**B**
Potassium Chlorate	53	35	53
Lead Thiocyanate	25	17	25
Antimony Sulfide	17	30	12
TNT	5	5	-
PETN	-	-	10
Calcium Silicide	-	15	-

Table 3
Lead Styphnate base priming mixes

	PISTOL			**RIFLE (#257)**			**RF**
Mix Type	**Small**	**Large**	**(X)**	**Small**	**Large**	**(Y)**	
Lead Styphnate	37	37	30	37	37	42	45
Tetrazene	4	4	4	4	4	3	4
PBTN	-	-	-	5	5-	-	-
Antimony Sulfide	25	25	31	15	15	-	-
Aluminum	4	4	11	7	7	-	
Calcium Silicide	-	-	-	-	-	5	-
Barium Nitrate	30	30	24	32	32	42	20
Gum solution (ml/lKg)	30	30	30	30	30	30	30
Lead Dioxide	-	-	-	-	-	3	7
Ground glass	-	-	-	-	-	-	22

(X) South European mfg.
(Y) FN - Belgium

Table 4
Lead free priming mixes

Material	Mix (percent by weight)	
	1	2
Diazol	20	17
Tetrazene	5	5
Titanium	5	-
Antimony Sulfide	-	24
Calcium silicide	-	10
Zinc peroxide	50	-
Barium nitrate		34
NG powder	20	-
Lead dioxide	-	10

Note: mix 1 - U. S. Pat. 4,581,082
mix 2 - Winchester mix used in Large Rifle primer during WW II

Factors affecting the choice of priming mix formulation:

- Reduction of barrel bore wear in automatic weapons.
- Sensitivity to available firing pin. Energy levels in specific weapons used.
- Type of foil required in primer assembly (lead foil, paper, etc.).
- Area and type of flash hole vent (single hole of large diameter, twin holes of Berden primer, etc.).
- Muzzle flash with some propellants.

Future trends in primer mix formulations:

- Improved stability.
- Automatic charging suitability.
- New metallic fuels (Titanium, Zirconium, etc.).
- Lead and barium free oxidizers (Zinc peroxide).
- Lead free compositions (return to diazol).
- Search for lower cost materials.

To have a reference point in the laboratory preparations, let's make the following assumptions:

a – Test lot of 1,000 primers is to be loaded

b – Each primer charge will be approximately 60 milligram of mix; hence the total quantity of priming mix will be 60 gram

c – The binder gum solution is the same for all test mixes at 10 ml per batch

d – The percentages of primary explosives in the respective test batches are:

Mercury Fulminate	50%	30 gram
Lead Styphnate *)	40	24 gram
Tetracene	4	2.4 gram
Diazol	20	12 gram
Lead Thiocyanate	25	15 gram
PETN or TNT **)	5	3 gram

*) to prepare 24 gram of Lead Styphnate requires 15.1 gram of TNR, or 8 gram of resorcinol

**)The high explosive boosters (PETN or TNT) may be replaced by a fast burning rate double based ball powder of the type used in rimfire and small pistol cartridges. Such powders have been used in several priming mixes with good results.

Note: for comparison a brief calculation of typical requirements for a commercial installation is included in the Appendix.

The student laboratory preparations will be calculated for the approximate quantities listed above.

Chapter 3

Materials

It is quite difficult to boil a cup of aromatic coffee from beans mixed with an abundance of rat droppings. It is also difficult to make a tasty cake from moldy flour mixed with sawdust. Obviously, to make consistently good priming mix requires good, clean and consistently uniform quality raw materials.

The materials used in priming mixes may be separated into basic groups according to their main purpose, as listed below. Both, military and commercial primer manufacturers have established material specifications for each material used. Sometimes these specifications vary slightly between the two groups. Such variations are usually due to the less expensive, yet adequate, quality of the raw material to produce a good end product.

The list below contains the Merck index number for easy identification of the material by the laboratory personnel. The MIL- specifications may be obtained through the libraries and/or directly from the Naval Publications & Forms Center in Philadelphia ,PA, or from the Department of the Army at Picatinny Arsenal, NJ. Commercial specifications are usually available from the manufacturer and/or distributor of a given material.

The military and commercial specifications are included as reference only. They are of importance for large volume production. For laboratory preparations, due to the small quantities used, it is suggested to use reagent grade materials.

Many of the listed materials are used in preparation of everyday household items dies, food preservative, medicines, etc. As the technology advances, newer materials will be used. So titanium powder, although more expensive than aluminum powder will be used as fuel in more of future mixes. Some other materials have been tried in the past as priming mix components. However, they were found less efficient and/or more dangerous than those currently used. For example, mixes with chlorates and ammonium nitrate (a strong oxidizer and explosive) produced double decomposition and formation of dangerous ammonium chlorate.

MATERIALS USED IN PREPARATION OF PRIMING MIXES

COMMON NAME	CHEMICAL FORMULA	MERCK INDEX#	MIL SPECIFICATION
EXPLOSIVE INITIATORS			
Diazol (DDNP)	$C_6H_2N_4O_5$		D-552
Lead Styphnate	$C_6H_3N_3O_9Pb$		L-757 & L-16355
Mercury Fulminate	$C_2N_2O_2Hg$		M-219
EXPLOSIVE BOOSTERS			
Double base powder (NG fast burning)			
PETN (class 2)	$C_5H_8N_4O_{12}$	6977	P-387
Tetrazene	$C_2H_8N_{10}O$		T-46938
TNT	$C_7H_5N_3O_6$	9534	T-248
Lead Thiocyanate *	$C_2N_2PbS_2$	5268	L-065
FUELS			
Calcium silicide	CaS_2		C-324
Aluminum powder	Al		A-512 A-159 class 2
Antimony Sulfide	Sb_2S_3		
Titanium powder	Ti	9303	T-13405
OXIDIZERS			
Barium nitrate (class 1-A)	$Ba(N03)_2$	983	8-162
Lead Dioxide	PbO_2	5241	L-376
Lead Nitrate	$Pb(N0_3)_2$	5252	L-20549
Potassium Chlorate	$KClO_3$	7494	P-150, grade A
Potassium Nitrate	KNO_3	7528	P-156, class 4
Zinc Peroxide	ZnO_2	9955	

*also called Lead Sulfothiocyanate

Continued on next page.

COMMON NAME	CHEMICAL FORMULA	MERCK INDEX#	MIL SPECIFICATION
BINDERS			
Gelatine	compound	4242	C-D-221 type 1
Gum Arabic	compound		
Gum Tragacanth	compound	4462	G-96
Nitrocellulose lacquer	compound		
Shellac		8315	
Thymol as mildew preventer	$C_{10}H_{14}O$	9246	USP-T-117
INTERMEDIATE PROCESS MATERIALS			
Aminate	$CH_6N_4\ HCO_3$	------	A-60569
Ethanol (alcohol)	C_2H_6O	212	O-E-00760 type III
Hydrochloric Acid	HCl	4682	reagent grade
Mercury metallic	Hg	5742	double distilled
Methanol	CH_4O	5816	
Nitric acid class a	HNO_3	6420	A-183
Picramic acid	$C_6H_5N_3O_5$	7287	
Picric acid	$C_6H_3N_3O_7$	7288	A-187
Potassium Thiocyanate, purified	KSCN	7581	commercial
Resorcinol (type 1, cl.2)	$C_6H_6O_2$	8056	R-60854
Sodium Nitrite	$NaNO_2$	8486	S-24521
Sulfuric acid	H_2S0_4	8854	A-179
Styphnic acid (TNR)	$C_6H_3N_2O_8$	8730	T-50611
Water	H_2O	distilled	

MATERIAL REQUIREMENTS

ITEM	GRAMS TO PRODUCE 1000 G PRODUCT
TNR	1000
Resorcinol	600
Sulfuric acid	6000
Nitric acid	2500
Sodium nitrite	
Lead Styphnate	1000
TNR	620
NaOH	200
Lead nitrate	1060
Acetic acid	205
Tetracene	1000
Aminate	1500
Odium nitrite	1700
Acetic acid	665
Priming mix, 209 shotshell	1000
Lead styphnate	400
Tetracene	40
barium nitrate	300
Antimony sulfite	150
Aluminum powder	60
PETN	50
Gum solution	10

ACETIC ACID, GLACIAL CH_3COOH MW 60.05
Ethanoic Acid **MIL-STD-1217B**
Methane Carboxylic Acid
Vinegar Acid
(HAZARDOUS)

Specifications: O-A-76, Acetic Acid, Glacial, Technical

Technical description: Glacial acetic acid is a clear, colorless liquid with a pungent odor. It is hazardous and is classified as a corrosive material. The commercially available acid is about 99.5%, the balance being water. The freezing point is 16.7°C and the material contracts slightly on freezing. It is an excellent solvent for many organic compounds, also phosphorus, sulfur, and halogen acids. Miscible with water, alcohol, ether, carbon tetrachloride; insoluble in carbon disulfide.

Use: Glacial acetic acid is intended for military use as a general neutralizing and acidifying agent and as a solvent for essential oils, resins, etc. Typical commercial applications include use in the manufacture of various acetates, acetyl compounds, plastics and rubber; in tanning; as a laundry soap; printing calico and dyeing silk; preserving foods; solvent for gums, resins, volatile oils and many other substances. Widely used in commercial organic syntheses.

Packaging data and labeling: For military use glacial acetic acid is packaged in a 5-lb (2.27 kg) unit quantity glass bottle, and a 450-lb (204.3 kg), 55-gallon (206.25 liters) unit quantity drum. Each container-bottle or drum is labeled in accordance with the Federal Caustic Poison Act. In addition, each container shall be labeled arid marked in accordance with MIL-STD-129, Title 49. CFR Parts 100-199 and each container shall bear the following label:

ACETIC ACID, GLACIAL
DANGER! CAUSES SEVERE BURNS

Do not get liquid or vapor in eyes, on skin, on clothing. In case of contact, immediately flush skin or eyes with plenty of water for at least 15 minutes; for eyes get medical attention.

Store in area maintained above 62° F (16.7° C).

If frozen thaw by carefully moving container to warm area. TLV for acetic acid is 10 ppm or 25 mg/m^3.

Storage: Glacial acetic acid should be stored in a warm dry area away from sources of heat or flame. Containers should be kept tightly closed and plainly labeled. When stored under ideal conditions, this material has an indefinite shelf life.

Disposal: For appropriate disposal procedures contact the installation's Environmental coordinator. In all cases every effort should be made to recover usable products from the waste material.

MATERIAL SPECIFICATIONS
Acid, hydrochloric

USE: MF preparation

APPEARANCE: Clear, transparent, colorless liquid, free from visible sediments, suspended matter, separated material

GRADE: Reagent *

*) Because of the relatively small quantities used, it is economically permissible to use reagent grade

Packing: Commercial, glass bottle inside carton

Inspection: Supplier's certificate on label

MATERIAL SPECIFICATION
Acid, nitric

USE: MF Preparation

APPEARANCE: Clear; free from sediments, suspended matter, separated material

GRADE:

REQUIREMENTS:	%
Nitric acid contents, mm.	90
Nitrobodies	none
Chlorides (as HCl), max.	0.01
Sulfates (as H_2S0_4), max.	0.01
Nitrogen oxides (as NO_2), max.	0.02
Lead salts, max.	0.01
Ash, max.	0.01

Inspection: Supplier's certificate

MATERIAL SPECIFICATION:
HNO_3 -Acid, nitric
MIL-A-183
class e

USE: nitration

APPEARANCE: No darker than standard color solution free from sediments, suspended matters and separated materials

REQUIREMENTS:

Specific gravity at 15.5°C, minimum,	1.415 (42° Be')
Nitric acid, minimum	65.50%
Residue on ignition not to exceed	0.01 %
Maximum impurities - chlorides (as HCl)	0.10 %
sulfates (as H_2SO_4)	0.20 %
lead salts	0.10%
ashes	0.10%

Packing: Stainless steel container showing lot number

Inspection: Analysis certificate with each lot

Note: 42^0Be' acid 100 ml contains 95.2 gram HNO_2
100 gram contains 67.5 gram HNO_3

MATERIAL SPECIFICATION
H_2S0_4 Acid, sulfuric
MIL-A-179

USE: Tetracene sulfonation

APPEARANCE: Electrolyte grade

REQUIREMENTS:

Specific gravity at 15.5°C		1.83 (66° Be′)
Maximum impurities	Fe	0.01%
	SO_2	0.01%
	NO_3	0.001%
	Cl	0.01%

Packing: Stainless steel or lead lined containers

Contamination with iron is not important and hence storage of sulphuric acid in iron tank (and resultant-ferrous sulfate) is permissible. The concentration of H_2SO_4 is a factor in crystal control of TNR. Acid with 96-98% concentration yields TNR resulting in lead styphnate crystals of uniform, thin, hexagonal plates.

Note: 66°Be′ = 1.84 Sp. Wt. contains 95.6% H_2SO_4

"66 grade" commercial contains only 93% H_2SO_4
100 ml 1.84 Sp. Wt. contains 174 gram H_2SO_4
100gram 1.84 Sp. Wt. 95.6 gram H_2SO_4

MATERIAL SPECIFICATION
Aminoguanidine bicarbonate

USE: Tetracene preparation

APPEARANCE: White, line powder, free from lumps

REQUIREMENTS:

Moisture	0.25% maximum
Calcium, total	0.01% maximum
Cu	0.005% maximum
Excess acid required for dissolution (as $NaHCO_3$)	0.1 % maximum
Aminoguanidine bicarbonate	98.5 %

PACKING : Maximum 100 Kg net in polyethylene bag in fiber drum

Inspection: 250 gram sample taken from at least two containers in the lot; composite sample is prepared by blending of samples on a sheet of paper.

Moisture - approximately 5 gram of sample is transferred to a tared weighing bottle; heat in oven at 50 C for 18 hrs., loss in weight of sample calculated as percent moisture. Alternate method is to place bottle with sample in a desiccator over sulfuric acid or dry calcium chloride at 65 mm Hg vacuum and ambient temperature for 24 hrs.

Ca total - 5 gram of aminoguanidine bicarbonate is placed in a 500 cc beaker.
Add 30 cc of distilled water and 5 cc of glacial acetic acid.
Allow the acid to react then dilute with distilled water to 150 cc, yielding a clear solution.
Heat to boiling. Add 20 cc of boiling hot 8% solution of ammonium oxalate in a fine stream while stirring.
Place mixture upon a steam batch for 90 minutes filter and wash three times with hot 0.2% ammonium oxalate solution.
Ignite precipitate to redness in an electric furnace, weigh as CaO and calculate to % $CaCO_3$.

Copper - 10 gram of aminoguanidine bicarbonate dissolved in 150 cc of water with sulphuric acid (for slight acidity) and electrolyzed according to standard analytical procedure using platinum electrodes must not yield any visible copper deposit. Percentage of copper is determined by a colorimetric technique

aminoguanicine bicarbonate - 2 gram sample added to 50 cc distilled water in a 500cc beaker.
Add 30 cc HCl (1-3) gradually, stirring the solution; cool in ice bath to 7± 3 C; add titrate, at 7°± 3° C with N/2 $NaNO_2$ solution; first add about 3cc and wait until this nitrite disappears, then add 0.5cc at a time and finally proceed one drop at a time. Use KI-starch paper as indicator.

MATERIAL SPECIFICATION
Aminoguanidine bicarbonate

The addition of $NaNO_2$ is complete when, after a ten minute period, a blue still forms upon the KI paper. This titration results in an accuracy of ±0.1% of aminoguanidine bicarbonate.

$$\text{Percent aminoguanidine bicarbonate} = \frac{A \times N \times 13.612}{W}$$

A = volume of N/2 $NaNO_2$ in cc
N = exact normality of $NaNO_2$
W = weight of sample in grams

Excess acid required for dissolution:

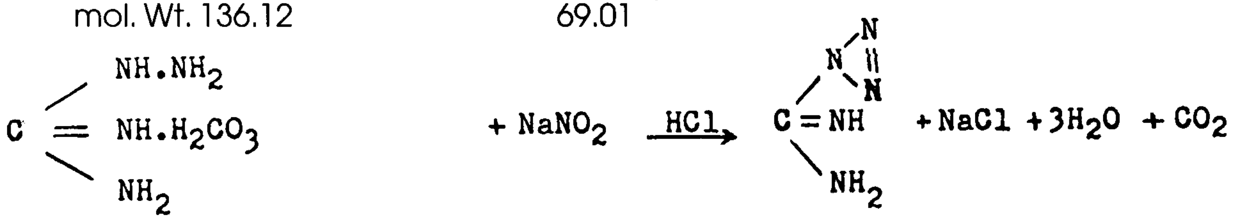
Two (2.000) grams sample is placed in a titrating dish
Add 20 cc of neutral distilled water
Add 50 cc N/3 HCl (accurately measured), stirring
Constantly until solution clears
Into a second titrating dish add 20 cc of the same
Neutral distilled water and 50 cc N/3 HCl, stirring constantly
Using bromphenol blue indicator (0.1% solution in H_2O)
Titrate each solution with N/3 NaOH
First appearance of violet-blue is the endpoint

$$\% \, NaHCO_3 = 0.617 \left(\frac{a-b}{W} \times N \times 13.612 \right) = 0.617 \, M$$

a = Volume of N/3 NaOH blank titration in cc
b = Volume of N/3 NaOH sample titration in cc
N= Exact normality of NaOH
W= Weight of sample in grams
M = Percentage aminoguanidine bicarbonate as determined by actual analysis
0.617 is the ratio of molecular wts. of $NaHCO_3$ to Am. bic.

The chemical reaction in titration of aminoguanidine bicarbonate is described in Lieber, Chem. Reviews 25, p 213.

aminoguanidine bicarbonate $NaNO_2$ guanyl azide
mol. Wt. 136.12 69.01



1 cc N/2 $NaNO_2$ corresponds to 0.136 grams aminoguanidine bicarbonate

MATERIAL SPECIFICATION
Antimony sulfide
MIL-A-159, class 1, 2, 3, or 5

APPEARANCE: Natural stibnite (Chinese Needle Antimony Sulfide) or synthetic antimony sulfide manufactured from pure materials yielding a product with characteristics and structure equal to natural stibnite. Used in "as received" condition.

REQUIREMENTS:

granulation through U. S. Std. sieves	No. 80	100 %
	100	80% mm.
	200	20 % mm.

Composition :	Sb	70.5 % mm.
	S, total	27.5 % mm.
	Free S	0.05 % max.
	Insoluble matter	0.5 % max.
	Acidity as H_2S0_4	0.01 % max.
	Pb	0.15 % max.
	Fe	0.10 % max.
	As	0.01 % max.
	Moisture	0.10 % max.

Inspection: Approximately 100 gram each, samples taken from 10% of containers in shipment; composite sample blended by rolling on paper and tested as per following procedures.

Packing: Polyethylene bag in fiber or steel drum.

MATERIAL SPECIFICATION
Barium nitrate
MIL-B-162, class 1, granulation A

APPEARANCE: Dirt free, white, crystalline powder

REQUIREMENTS:

Granulation through US Std. sieve:

Primer type	*Shotshell with PETN*	*Shotshell without PETN*	**centerfire*
Sieve No. 80	78 % min.	100 %	
100	65 % min.	80 % min.	-
140	60 % min.	-	99.9 % min.
200	30 % max.	25 % max.	40 % min.
230	10 %max.	15 % max.	25 % max.

Composition:	$Ba(NO_3)_2$	99.7 % mm.
	Moisture	0.20% max.
	Matter insoluble	
	In hot water	0.10% max.
	Chlorides as $BaCl_2$	0.007% max.
	Id as Ca or Sr	0
	Heavy metals	0
	Grit	0.05 % max.
	pH value	5.0 - 8.0

Inspection: Samples taken from 10% of containers in shipment; composite sample blended by rolling on sheet of paper and tested as per following.

Packing: Std. commercial

*) The correct granulation of $Ba(N0_3)_2$ is essential; since PETN acts partially as oxidizer it is expected that finer granulation of $Ba(N0_3)_2$ will assist in this respect.

Chlorides (calculated as NaCl)	0.05% max
Total barium (calculated as $Ba(N0_3)_2$	98.5% mm
Total insoluble in water	0.5% max

Barium - Potassium nitrate

Barium nitrate	26 gram
Potassium nitrate	40 gram
Boiling water	100 gram

- Dissolve materials.
- Place beaker with hot solution into cold water bath.
- Stir solution until temperature drops to ambient.
- Decant mother liquor for next batch.
- Transfer precipitate onto paper covered tray.
- Air dry precipitate.
- Screen dry material.
- The double salt should contain 56-58% Barium nitrate note: theoretical value is 56.55%.
- Results of test indicate how much material to add to the mother liquor for the next batch.

MATERIAL SPECIFICATION
Calcium silicide

- Material received from vendor (Electrometallurgical Corp. - NY) is crushed, screened, ground.
- Digested 5 days in hot water, frequently agitated.
- Hot water is changed every 12 hours.
- Digested material is washed by decantation 6 times with cold water.
- Washed material is screened through No. 200 mesh.
- Screened material is dried.

MATERIAL SPECIFICATIONS
ETHANOL

USE: MF preparation (mercury fulminate preparation)

APPEARANCE: clear, transparent, colorless liquid, free from visible insoluble matter, characteristic odor

GRADE: not to contain dye or denaturant

REQUIREMENTS:

Ethanol, by volume	min.	95.0 %
Benzene, by volume	max.	0.01 %
Acidity (as H-acetic)	max.	0.005 gm/100 ml
Aldehydes	max.	20 mg/100 ml
Nonvolatile matter	max.	0.004 %
Oorganic impurities	max.	0.1 %

Inspection: Supplier's certificate

Gum Tragacanth, Specification JAN-G-96

APPEARANCE: White or light tan color,
leaf or flake form, without odor

REQUIREMENTS:

Composition:

Ash	3.5 %max.
Gums, Indian	0
Gums, foreign	0
Acidity, inorganic	0
Acidity, organic	0.40% max.
Cl, or chlorides	0

Inspection: Samples taken from l0% of containers in shipment
composite sample blended, by rolling on sheet of paper

Packing: Std. commercial fiber containers

Gelatine, Specification C-D-221, type 1

APPEARANCE: U.S.P. grade edible gelatine without artificial or natural flavor and/or color

REQUIREMENTS: None specified

Inspection: None, material accepted on supplier's guarantee

Packing : Std. commercial

Thymol

APPEARANCE : U. S. P. T117 grade, crystalline

REQUIREMENTS: Melting point 50° - 51° C

Inspection : None, material accepted on supplier's guarantee

Packing : Std. commercial

Alcohol, Ethyl, denatured (for binder use only)

APPEARANCE: S. D. 1 alcohol

REQUIREMENTS: None specified

Packing: Sd. commercial

Inspection: None, material accepted on supplier's guarantee

MIL-L-10287A
19 AUGUST 1953

SUPERSEDING
MIL-L-10287 (ORD)
2 May 1950

MILITARY SPECIFICATION

LACQUER, CELLULOSE NITRATE FOR IDENTIFICATION OF SMALL ARMS AMMUNITION

This specification has been approved by the Departments of the Army, the Navy and the Air Force.

1. SCOPE

1.1 **Scope.**—This specification covers a lacquer used in the manufacture of small arms ammunition for identification purposes.

1.2 **Classification.**—This specification covers one grade and two types of lacquer as follows:

Type I—Opaque.
Type II—Transparent.

2. APPLICABLE SPECIFICATIONS, AND STANDARDS

2.1 The following specifications and standards of the issue in effect on the date of invitation for bids, form a part of this specification:

SPECIFICATIONS

FEDERAL

TT-P-141—Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling and Testing.
TT-P-143—Paint, Varnish, Lacquer, and Related Materials; General Specification for Packaging, Packing, and Marking.

MILITARY

MIL-T-6094—Thinner; Cellulose Nitrate Dope and Lacquer.

STANDARDS

MILITARY

MIL-STD-105—Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-129—Marking of Shipments.

(Copies of specifications and standards required by contractors in connection with specific procurement functions should be obtained from the procuring agency or as directed by the contracting officer.

Federal Std No 595 - colors

3. REQUIREMENTS

3.1 **Color.**—The color shall be as specified in the contract or order, and of a shade conforming to Fed. Std. No 595

3.2 **Vehicle.**

3.2.1 *Volatile.*—The volatile portion of the lacquer shall consist of any suitable solvents except benzol (benzene), methanol, chlorinated or other known toxic compounds.

3.2.2 *Nonvolatile vehicle.*—The nonvolatile vehicle shall consist of cellulose nitrate, modifying resins, and plasticizers.

3.3 **Pigment.**—The pigments shall be light, fast, and opaque. Extenders of any type shall not be used.

3.4 **Quantitative requirements.**—The lacquer shall conform to the quantitative requirements specified in table I and 3.4.1.

TABLE I.—*Quantitative requirements*

Characteristics	Requirements	
	Minimum	Maximum
Total solids, percent by weight of lacquer:		
Type I		
Black	32	...
All other colors	35	...
Type II	30	...
Pigment, percent, by weight of lacquer:		
Type I		
Black	2	...
All other colors	5	...
Drying time, set to touch, in minutes	...	4

3.4.1 *Viscosity.*—The viscosity of the lacquer, when thinned with thinner conforming to

MATERIAL SPECIFICATION
Lead Dioxide
MIL-L-376, type I, class 2

SPECIFICATIONS:	PbO_2	90.0% mm.
	Moisture	0.2%
	Iron	0.015%
	Chlorine	0.002%
	Granulation	99.8% -325 mesh

PACKAGING: Fiber drum

HANDLING: Inhalation can cause lead poisoning

SHIPPING REGULATIONS: Oxidizer - Yellow Label

Lead Oxide

Typical analysis:

Total lead	92.7%
Lead peroxide	3%
Lead oxide	99.95%

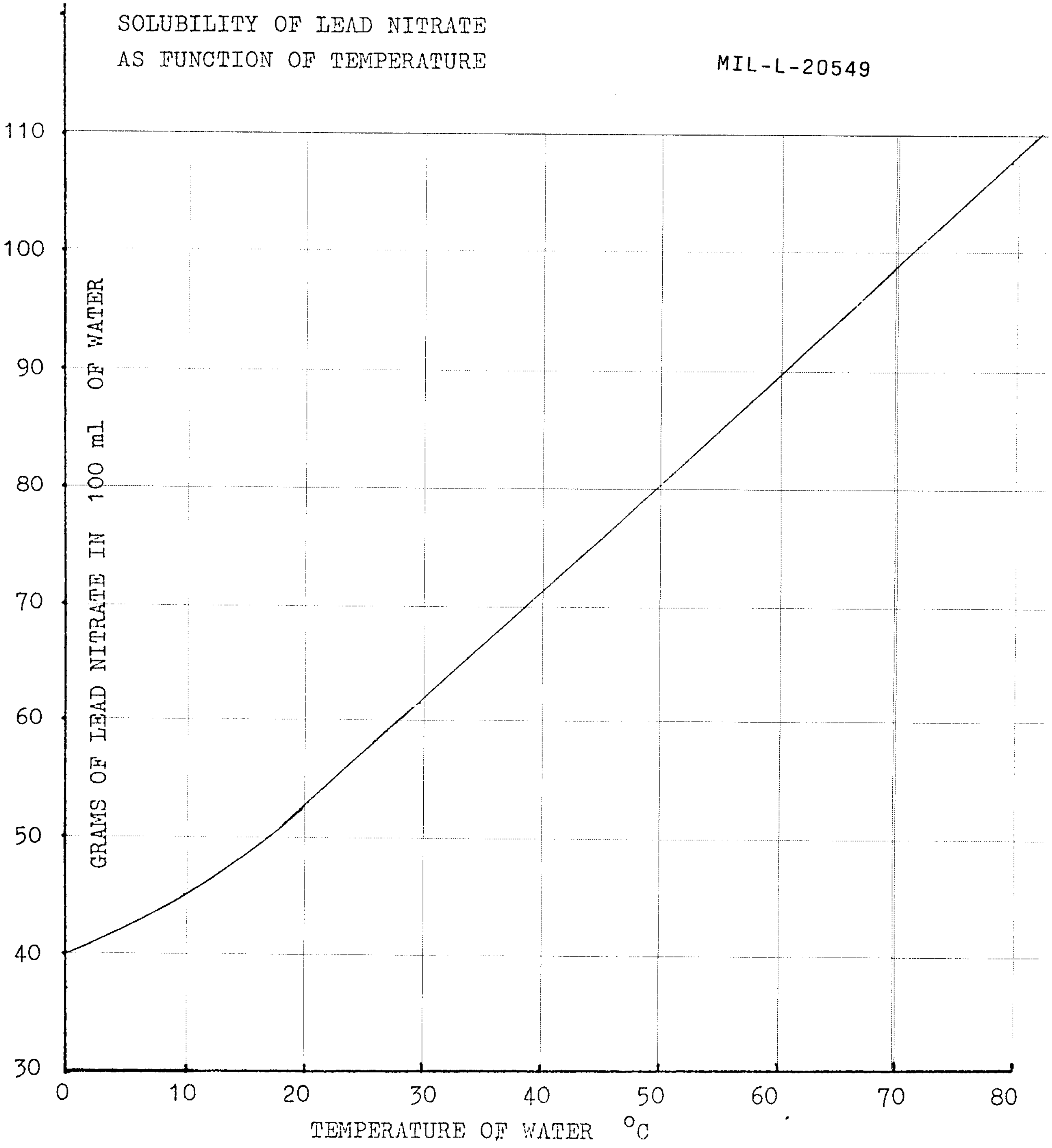
LEAD NITRATE
$Pb (NO_3)_2$

SPECIFICATIONS:	$Pb (NO_3)_2$	97.8% mm.
	Acidity	0.5% max. (as HNO_3)
	Copper	20 ppm
	Iron	100 ppm
	Chloride	250 ppm
	Insolubles	0.10% max. (in 10% Nitric acid)

PACKAGING: Fiber Drums

HANDLING: Oxidizing agent liberates nitrogen oxides when heated. Inhalation can cause lead poisoning.

SHIPPING REGULATIONS: Oxidizer - Yellow Label


SOLUBILITY OF LEAD NITRATE
AS FUNCTION OF TEMPERATURE
MIL-L-20549
GRAMS OF LEAD NITRATE IN 100 ml OF WATER
110
100
90
80
70
60
50
40
30
0
10
20
30
40
50
60
70
80
TEMPERATURE OF WATER °C

LEAD THIOCYANATE
Pb $(SCN)_2$

SPECIFICATIONS:

Lead (as Lead Thiocyanate)	99.5%
Thiocyanate (as Lead Thiocyanate)	99.5%
Moisture	0.10%
Insoluble Matter	0.10%
Sodium	0.10%
Chloride	0.10%
Solubility	pot. thiocyanate, nitric acid
Granulation	100% -140 mesh

PACKAGING:

Level A. Lead thiocyanate: packaged in a heat-sealed polyethylene bag in accordance with PPP-C-3OI inside a lined fiber drum conforming to PPP-D-723, type III, grade A. Each drum not exceed 200 pounds net.

Level B. Lead thiocyanate: packaged in a heat-sealed polyethylene bag in accordance with PPP-C-301 inside a lined fiber drum conforming to PPD-723, type II, grade A. Each drum not exceed 100 pounds net.

Level C. Lead thiocyanate: packed in watertight shipping containers in accordance with PPP-C-301 for shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with Consolidated Freight Classification Rules or other common carrier regulations as applicable to the mode of transportation.

HANDLING: Inhalation and ingestion can cause lead poisoning.

SHIPPING REGULATIONS: ORM-A, m.o.s. Poison.

PICRAMIC ACID

USE: Preparation of Diazodinitrophenol (DDNP)

GRADE: Technical/laboratory grade

APPEARANCE: Dark red powder (needles or prisms, depending on the manufacturing process

REQUIREMENTS

Granulation: 90% Min. % through U. S. Std. Sieve No. 60

Melting point: 169-170°C

Inspection: Supplier's certificate

Packing: Drum or appropriate commercial packing and marking

Note: This material is used in manufacture of dyes; laboratory grade- as reagent for albumin and as indicator.

MATERIAL SPECIFICATION
Potassium Chlorate

USE: Priming mix ingredient

GRADE: Technical

APPEARANCE: White powder or crystals odorless

REQUIREMENTS:

Granulation:

U. S. Std. sieve No.	% THROUGH, MINIMUM
60	99.5
200	92.0

Composition:

$KClO_3$ minimum	99.7
KCl maximum	0.06
Water insolubles, max.	0.01

Inspection: Approximately 100 gram each, samples taken from 10% of containers in shipment at random; composite sample is blended by rolling on paper and tested as specified procedures.

Packing: Polyethylene bag in fiber or steel drum

MATERIAL SPECIFICATION
resorcinol
MIL-R-60864
type 1, class 2

USE: Trinitroresorcinol preparation

APPEARANCE: Colorless, or nearly colorless; needle-shaped crystals or flakes; technical grade

REQUIREMENTS:

Granulation through U.S. Std. sieve No.8		98.0 %	minimum
Setting point		109°C	minimum
Composition:			
	Resorcinol	99.0 %	minimum
	Catechol	0.45%	maximum
	Phenol	1.00%	maximum
	Acids	0.02%	maximum
	Moisture	1.00%	maximum

Packing: In fiber or steel drums (std. commercial)

MATERIAL SPECIFICATION
Sodium hydroxide, solution

USE: Lead styphnate preparation lead styphnate, tetracene, and priming mix scrapping

APPEARANCE: Technical grade solution, colorless, clear liquid free of sediments at 21° C

REQUIREMENTS:

Specific gravity at 20° C	1.525
NaOH concentration	50 ± 1 %
Freezing point	± 12.78° C

Composition:	NaOH	50 ± 1 %
	Na_2O	38.6 ± 1.0%
	Na_2CO_3	0.29 % max.
	NaCl	0.5 % max.
	Fe	0.0015% max.
	Sulfates	0.05 % max.
	Phosphates	0.05 % max.

Packing: 50 gal. steel drum with one bung in drum head

Inspection: Supplier's certificate

TEST:

PURPOSE: Determine NaOH solution concentration

EQUIPMENT:
1 - 50 cc pipette
2 - 500 cc volumetric flask
3 - 50 cc beaker
4 - Ditration unit

MATERIALS:
1 - Methyl orange indicator
2 - Distilled water
3 - HCl
SAFETY PRECAUTIONS: std. laboratory

PROCEDURE:

1 - With a pipette draw 20 cc sample.
2 - Dilute sample to 500 cc in volumetric flask.
3 - Shake flask to obtain homogenous solution.
4 - With clean pipette draw 20 cc of diluted solution to a beaker.
5 - Titrate the diluted sample with standard HCl of 0.4-0.5 normality with methyl orange indicator.

SODIUM HYDROXIDE
Grade: ACS: Technical (Fed. P-S-631. Type I).
Synonym: Caustic soda.

Chemical Formula: NaOH

Characteristics: White deliquescent flakes or pellets; absorbs water and carbon dioxide from the air; soluble in water, glycerol, and alcohol. Very corrosive (caustic), to organic tissue.

Use: Laboratory.

Storage and Handling: Store in a cool, dry place. Keep containers tightly closed.

Storage Compatibility Category: SCC I.

Personnel Hazard: Caustic and corrosive; causes severe burns to the skin and eyes; destroys organic tissue. Avoid contact with the skin, eyes, or clothing. Do not take internally. When handling the material in bulk, wear suitable protective clothing, gloves, respirator, and chemical safety goggles.

Fire Hazard: Noncombustible but reacts violently with water evolving considerable heat.

A. USE: Lead Styphnate manufacture
Neutralization of residual acids
Explosives destruction

B. REQUIREMENTS

Appearance: White flakes or pearls, technical grade

Molecular Weight - 40
Melting point - C°318
Specific Weight Kg/du3:2, 13

NaOH	:	99%
Na_2CO_3	:	0.03%
Na_2SO_4	:	-
NaCl	:	0.0008 %
SiO_2	:	0.0001
Fe_2O_3	:	0.0009%
CaO + MgO	:	0%

C. CONDITION

Packing: In drum or appropriate packing, commercial marking

MATERIAL SPECIFICATION
Sodium Nitrite
MIL-S-24521

USE: Source of nitrous acid in TNR and/or tetracene manufacture

APPEARANCE: Colorless, or white crystals or flakes

REQUIREMENT: U S P grade

$NaNO_2$	99.0 % minimum. (dry $NaNO_2$ - 97 % minimum)
Insoluble matter	0.05% maximum
Chloride	0.05% maximum
Sulfate	0.01% maximum
Na_2CO_3	0.15% maximum
Cu	0.01% maximum

Packing: Polyethylene bag in fiber or steel drum

Inspection: Composite sample taken from 10% of the containers in shipment blended by thorough rolling on sheet of paper

- Assay – 3.40 grams of sample dissolved and diluted with distilled water to 500 cc in a volumetric flask and mixed thoroughly. Add 100 cc of N/10 $KMnO_4$ into 250cc Erlenmeyer flask. Add 20cc of 1-3 H_2S0_4. Add 50 cc of the $NaNO_2$ solution under the $KMnO_4$ solution with agitation. Add 2 cc of N/10 oxalic acid to destroy the excess of $KmnO_4$. Heat to 75° ± 5° C and titrate while hot the excess of oxalic acid with N/10 KMnO until a permanent pink color is obtained.
  Calculation + (cc N/10 $KMnO_4$ - N/10 oxalic acid) x 1.015 equals % $NaNO_2$

- NaCl – 5 grams of sample dissolved in a 200 cc beaker with 50 cc of distilled water. Add 20 cc Fe indicator solution and boil 5 minutes.To the cool solution add 1cc N/2O KCNS, and then add a slight excess of N/2O $AgNO_3$ under constant stirring. Titrate the excess of $AgNO_3$ with N/2O KCNS.
  %NaCl (cc N/20 $AgN0_3$ - cc N/20 KCNS) x 0.2923 and divided by sample weight.

- Na_2CO_3 – 10 gram sample dissolved in distilled water and titrated with N/IO H_2S0_4 using phenolphthalein as indicator. % Na_2CO_3= (cc N/IO H_2SO_4) x 0.106

MATERIAL SPECIFICATION
Tetracene
(guarayl nitrosoamino guanyl tetrazene)

USE: Priming mix ingredient

APPEARANCE: White to slightly yellowish color needle shaped crystals

REQUIREMENTS:

Granulation through U.S. Std. No. 45 Sieve	100 %
Specific gravity	1.65 ± 0.05
Melting and explosive point.	130 ± 2C°
Purity	no evidence of heavy metals
Nitrogen contents	74.5 ± 1.0

Inspection: approximately 250 g each samples from 3 containers in lot are blended to create a homogenous composite sample for testing as follows.

Color and appearance: Transfer a portion of about 10 grams of the wet sample as received to a Buchner funnel and apply suction until the sample is almost dry. Spread the sample on a watch glass and place in a desiccator or sulfuric acid for 24 hours or in an oven at 70° to 75° centigrade for at least 90 minutes. Use this sample for all determinations where a dry sample is required. Take four (4) portions of about 0.5 milligram (mg) each from different places in the sample and spread them over an area of about 2 centimeters (cm) square on a glass microscope slide with reflected light using a magnification of 850 times.

Granulation: Transfer about 80 grams of the wet sample as received, to a Buchner funnel about seven centimeters in diameter and apply suction for two minutes. Weigh a 10 gram portion of this damp sample and transfer it to a tared three inch No. 45 U. S. Standard sieve, which has been placed in a porcelain evaporating dish containing sufficient water to cover the wire portion of the sieve. Shake the sieve for a few minutes in such a manner that the tetracene is below the surface of the water and caught in the evaporating dish. When most of the tetracene has passed through the sieve, run a stream of water through the sieve while brushing the residue lightly with a soft, camel-hair brush. When no more material appears to pass through the sieve, rinse that remaining on the sieve with 96 percent ethyl alcohol and dry the sieve and contents on a steam bath. Place the sieve in an oven at 70° to 75° C for 30 minutes, cool in a desiccator and weigh. Calculate the gain in weight as percent material retained on the sieve.

Specific gravity: Weigh a dry 10 milliliter (ml) pycnometer. Fill it with water at 30° Centigrade and weigh again. Empty and dry pycnometer, add 0.2 to 0.4 gram of dry tetracene and weigh again. Add about 5 ml of kerosene and put under vacuum in a vacuum desiccator for a few minutes to remove air bubbles. Fill pycnometer with kerosene at 30° Centigrade and weigh. Calculate as follows:

Specific gravity of tetracene =

$$\frac{W}{A - \frac{(B - C)}{S}}$$

Where,
W = weight of sample
A = weight of water contained in bottle when filled
B = weight of bottle + tetracene + kerosene
C = weight of bottle + tetracene
S = specific gravity of kerosene

Melting point and explosion point:

Weigh a capillary tube (1.5 to 2 millimeters (mm) in diameter) and add a dry sample of tetracene until about 0.5 to 1.0 mg is present. Put on a safety mask and determine the melting point in a Vanderkamp apparatus (Scientific Glass Apparatus Co. catalogue 59, M-1945, Bloomfield, New Jersey.) Heat the capillary tube in the enclosed air-space. Regulate the electrical heating system so that the rise in temperature is one (1) degree Centigrade © every three (3) minutes. Observe the capillary tube through the magnifying glass, which is part of the apparatus, and note when the substance melts and when the substance explodes.

Purity:

To test for the presence of heavy metals treat a 0.3 gram dry sample of tetracene with 10 milliliters (ml) of nitric acid and 5 ml of sulfuric acid, evaporate the strong fumes of sulfuric acid, and add nitric acid dropwise to the fuming solution until the solution is water white. Cool, dilute to 150 ml with water and treat with hydrogen sulfide. No detectable precipitate shall be present.

Nitrogen content:

A dry sample of 2 mg of tetracene will be used for the nitrogen determination by the Micro-Dumas Method. Detailed instruction for performing this analysis can be found in the following analytical texts:

"Standard Methods of Chemical Analysis," Quantitative Organic Microanalysis" by A. Steyermark.

MATERIAL SPECIFICATION
Trinitrore Sorcinol
MIL-T-50611

USE: lead styphnate manufacture

APPEARANCE: Manufactured by process giving a product with organic impurities derived from resorcinol and capable of producing lead styphnate insensitive when containing at least 20% water

REQUIREMENTS: Granulation through U.S. Std. sieve No. 10 - 100 %
Melting point: 178° ± 2° C

Composition:	TNR	98.0 % minimum
	Chlorides (as HCl)	0.01% maximum
	Iron and/or	0.01% maximum
	Sulfates (as $BaSO_4$)	1.0 % maximum
	Nitric oxide	trace only

Packing: TNR containing 20% water is packed in rubberized cloth bag; each bag containing approximately 10 Kg (dry weight) TNR has a cap of rubberized cloth of the bag diameter placed over the top of the material before being tied up. Five or less of these bags are placed in a steel drum lined with rubberized cloth or polyethylene bag. The large bag is tied securely and the remainder of the drum space is filled with sawdust soaked in water. The steel drum is closed and checked to be watertight.

Inspection:

Fe and/or Al
- 5 gram sample is placed into a 250cc beaker.
- Add distilled water to dissolve the sample.
- Add NH_4OH to precipitate sample.
- Fiter wand wash precipitate in a Gooch crucible.
- Ignite precipitate.
- Weigh as Fe_2O_3 or Al_2O_3.

Nitric oxide
- Prepare approximately 5 cc of saturated solution of sample in distilled water.
- Treat with saturated solution of diphenylamine in concentrate H_2SO_4.
- Result must be free from all odor of nitric oxide and only faint blue color.

Continued on page 28

Sulfates

-10 gram sample placed in a drying dish
- Drying dish placed in drying oven at 55° C for 180 minutes
- 5 gram of dry sample dissolved in 300 cc boiling water
- Cool solution to 21° C
- Filter and wash filtrate
- Determine sulfate in filtrate as $BaSO_4$ following std. analytical procedure.

MATERIAL: Water, distilled

USE: Priming mix manufacture

Description:

PH ≥ 5.5

Resistance $\geq 1 \times 10^6$ Ohms
Chlorides
Sulphates
Nitrates
Iron (traces only)
Copper
Zinc
Aluminum
Evaporation residue <0.001 gram/100 ml

NOTES:
Directly after manufacture pH value is about 7. After contact with air the pH value decreases due to formation of H_2CO_3 from CO_2 in the air. Check pH value after extended storage; prior to use.

ZINC PEROXIDE
ZnO_2

USE: Priming mix oxidizer

APPEARANCE: Yellowish powder

REQUIREMENT: Technical grade

Composition:	ZnO2	55% min.
	ZnO	40-45%

Inspection: Supplier's certificate

Packing: In drum or appropriate commercial packing and labeling

NOTE: Also used in pharmaceutical preparations (creams)

MATERIALS USED IN PRIMER ASSEMBLY

Paper, foiling, "A"	-	Consolidation of priming mix in cup
Paper, foiling, "B"	-	Covering the flash hole in battery cup
Foil, lead	-	Covering the flash hole in battery cup of chlorate mix primers
Solvent, "A"	-	Wetting agent for paper, foiling, "A"
Solvent, "B"	-	Wetting agent for paper, foiling, "B"
Lacquer (MIL-L-10287)	-	Dust proofing and identification of primer type
Compound, tumbling surfaces	-	To remove excess lacquer from primer

MATERIAL SPECIFICATION
PAPER, FOILING – "A"

USE: Foiling primer cups (centerfire and shotshell)

REQUIREMENT: Paper (may be colored for identification) impregnated by passing it through a solution of 5% shellac in alcohol

Item	Sample		Specification	Allowable variation
Grade			*white kraft*	
Density, final	500 sheets 24" X 36"		17.7 Kg	6.5%
Density, paper only	id		16.8	5.5
Shellac	id		0.90 kg	25%
Material thickness	shellacked		0.09 mm	± 0.01mm
Tensile strength	12.7 mm wide x 80 mm long - shellacked		4.75 Kg	35%
Tensile strength	id	unshellacked	4.5 Kg	30%
Elongation	id	shellacked	2.5%	1.0%
Id	id	unshellacked	2.5 %	1.0%
Mullen test (bursting Strength)	id	shellacked	14.5 Kg	30%
Id	id	unshellacked	15.0 Kg	30%
Roll dimensions dictated by foiling press parameters and plate dimensions				
Roll width	(typical)	70.0 mm	± 0.5 mm	

Packing: Material and method must be adequate to prevent apparition of injurious defect during transportation and storage.

Marking: Each coil or lot should be provided with a tag showing the size, grade, order number and the name of manufacturer.

Inspection procedure: 3 m long sample taken from 3 different containers. Test values are mean values of all samples, results are average of 20 repetitive tests. Shellac value is evaporated residue after extraction with alcohol on a composite sample. Density of paper only is the difference between average total density and shellac density. Tolerances for paper before shellacking are not basis for rejection but for vendors information. Representative roll is tested on press for foiling characteristics. Shellac is grade A. (MIL-S-20526)

Note:

primer	**disc dia. (mm)**	
large rifle	4.47	-0.02
50 cal	6.95	-0.02

MATERIAL SPECIFICATION PAPERS FOILING, "B"

USE: Shotshell primer battery cup foiling

REQUIREMENT: White absorbent paper, kraft paper, Aloe No.609 or equivalent

Item	Sample	Specification	Allowable Variation
Density		150g/m^2	4%
Tensile strength	12.7 mm wide by 180 mm long	3.3 Kg mm.	
Elongation	id	3%	± 0.5%
Bursting strength (Mullen test)		0.9 Kg/cm^2 min.	
Paper thickness		0.20 mm	± 0.02mm

Roll dimensions dictated by foiling press parameters and plate dimensions		
Roll width (typical)	77.8 mm	± .5
Roll inside dia. (typical)	25 mm	
Roll outside dia. (typical)	300.0 mm	

Packing: Material and method must be adequate to prevent apparition of injurious defect during transportation and storage.

Marking: Each coil or lot should be provided with a tag showing the size, grade, order number and the name of manufacturer.

Inspection: Sample from each lot tested for requirements: results are average of 20 repetitive tests.

MATERIAL SPECIFICATION
LEAD FOIL

USE: Battery cup foiling of chlorate type primers

REQUIREMENT:

Composition	Pb	98.90 % mm.
	Sb	1.0 %
Foil thickness :	0.12 ± 0.01 mm	
Roll width	77.75 ± 0.75 mm	
Roll inside dia.	46	
Roll outside dia.	115	

Packing: rolls on cardboard core: each roll wrapped in paper.
Approximately 50 Kg of rolls in wooden box or equivalent

Inspection: 150 mm sample taken from 10 % of rolls and tested according to current ASTM "Methods of chemical analysis of metals."

MATERIAL SPECIFICATION
SOLVENT "A"

USE: wetting agent for primer foiling paper

MATERIAL: plasticized ethyl cellulose

viscosity at 25° C	80 ± 8 seconds #4 FORD cup
weight gram/liter	880 ± 10%
weight solids gram	120 ± .5%
volume solids, theoretical	10%
area covered at 0.2 mm dry	± 3.8 m^2/liter
finish	high gloss

NOTE: Dilute with lacquer thinner if required.
Material is equivalent to nitrocellulose lacquer when dry

SOLVENT "B" on following page

SOLVENT "B"

USE: Wetting agent for battery cup foiling paper

REQUIREMENT:
Commercial grade ethyl acetate
Commercial grade butyl acetate

In cold weather use	100 % ethyl acetate
In hot & humid weather use	50 % butyl acetate 50 % ethyl acetate

Packing : Clean, returnable steel drum with bungs on side and end.

MATERIAL SPECIFICATIONS LACQUER

USE: Identification or sealing varnish for Boxer and Berdan or Military primers.

REQUIREMENT:

Composition:	Nitrocellulose 3-40 second R.S. (3O% denatured alcohol SDNol wet)	72 g
	N butyl acetate	1350 g
	Ethyl acetate	3300 g
	Alcohol denatured, SD NO.1	l80 g
	Dye solution (methanol, synthetic 75.5%)	100 g
	% by wt.	N butyl acetate 20.0% Calco fast spirit concentrate color 4.5%

Lacquer solids	1.12 ± 0.12 %
Viscosity	± centipoises
Density	± 1.15 g/cc

Packing: clean, returnable steel drum with bungs on side and end

Remark: Max. temperature for use: 70° C (dry)

Color: Green, red or yellow

MATERIAL SPECIFICATIONS
TUMBLING COMPOUND

USE : tumbling of primers to clean off lacquer

REQUIREMENTS: standard commercial tumbling compounds, based on corn husks.
Wood sawdust etc.
Maize super fine
2540 Super grits
Tumbrite, hardwood saw dust

Chapter 4

Safety

The personnel safety in the preparation and handling of the priming mixes is the most important element of the operation. The safety precautions pertaining to the specific material are in the respective chapter. Some of the precautions may seem repetitive. However, such repetition is intended to remind the student into following the safety instructions. The most common laboratory safety includes:

1 – Eye protection to be worn during presence in the laboratory.
2 – Dust mask to be worn during operations.
3 – Flameproof overalls to be worn during presence in the laboratory.
4 – No eating, drinking, and smoking in the laboratory work area.
5 – Wash hands and face before eating and/or drinking.
6 – Pay full attention to the work being performed.
7 – Keep good housekeeping.
a – Aall materials are kept in appropriate and closed containers.
b – All dirty and/or contaminated containers and equipment, are placed IMMEDIATELY into the sink and filled with water.
c – All spilled materials are IMMEDIATELY wiped up with a wet sponge (hands to be protected with rubber gloves).
d – After completion of work; all containers, equipment and tools are cleaned, dried and returned to the respective holding area.
e – All containers with materials are returned to the storage area.

The safety instructions, guidelines and regulations for the preparation, handling, storage, transportation and destruction of explosives would fill a substantial library.

Following is a list of a few basic publications which are applicable to the subject. They are all available through open sources. Probably the most important from the student and laboratory work aspect are the following:
NOLTR 61 -138

(Oct. 1961)	Naval Ordnance Laboratory, White Oak, MD, Technical report No. 61-138 titled "Explosives, Propellants and Pyrotechnic Safety Covering Laboratory, Pilot Plant and Production Operations" by Russell McGill.
DOD4145.26M (Oct. 1968)	Department of Defense Contractor's Safety Manual, Dept. of Defense, Washington, DC.
TBCML5O (Oct.1961)	Department of the Army Technical Bulletin-(Oct.1961) "Chemicals and Chemical Products" Headquarters Dept. of the Army.
MI - STD - 1218 (June 1969)	"Military Standard ACS Grade Chemicals"

The above publications may be requested from:

Department of the Navy
Naval Publications and Forms Center
5801 Tabor Ave.
Philadelphia, PA 19120
or
U.S. Department of Commerce
National Technical Information Center
Springfield, VA 22161

Further information on state, local and/or federal safety regulations are:

ATFP 5400.7 "Explosives Law and Regulations"
Bureau of Alcohol, Tobacco and Firearms, Dept. of the Treasury, Washington DC 20226
available from Superintendent of Documents
U.S. Government Printing Office
Washington, DC 20402

NFPA#495 "Manufacture, Transportation, Storage and use of
Explosive Materials" available from:
National Fire Protection Association
470 Atlantic Ave., Boston, MA 02210

Additional explosive safety information may also be available upon request from the respective material manufacturers and/or suppliers. For data and addresses contact:

Institute of Makers of Explosives
1120 Nineteenth St. NW, Ste. 310
Washington, DC 20005
http://www.ime.org

Chapter 5

Intermediate Operations

It is advisable that prior to preparation of primary explosives, the student performs the intermediate operations. These include the weighing and blending of dry primer components, washing and/or drying of high explosive components (TNT, PETN) preparation of binders, and making of TNR. Such operations are relatively safe, due to the small quantities of the explosives involved. They are also simple and provide the student with practical experience in handling of priming mix material. The operations are described below in the convenient student laboratory format.

Dry pre-mix

The components of dry pre-mix are the fuels and oxidizers added to the explosive pre-mix components during the final mixing operation.

Since the dry pre-mix components are in dry powder form, they are screened to:

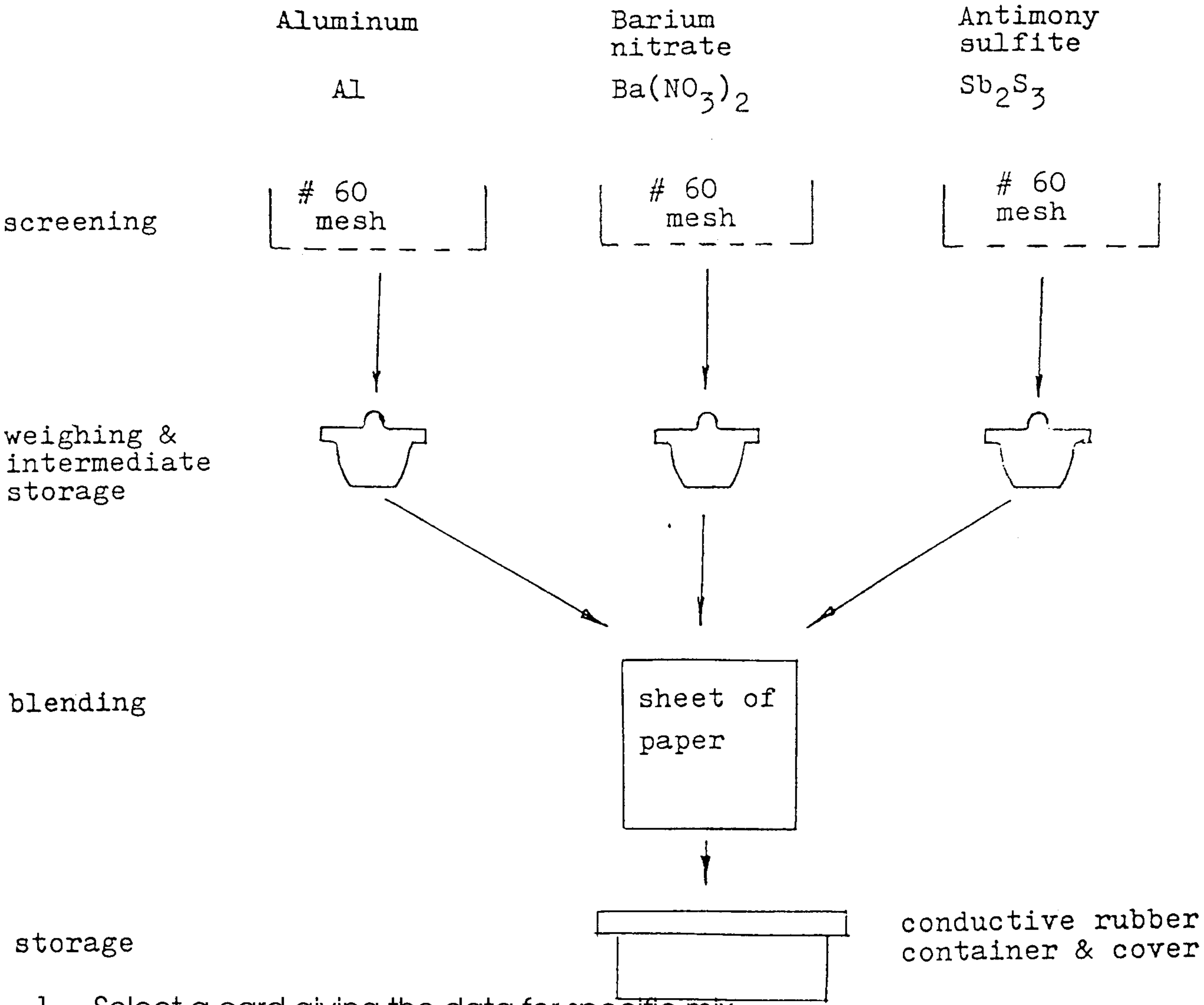
a – Prevent foreign material from entering the final mix

b – Prevent material particles from adhering to each other and forming clusters.

Equipment:

Table, approx. 200 x 30 cm, with conductive top
Cabinet, with shelving, std. commercial
Container, with cover, conductive
Scale, table model, capacity 1500 g
Accuracy ± 1%
Sieve, non-sparking, 60 mesh
Scoop ,hand, non-sparking
Spatula, rubber, commercial std.
Hood, exhaust to outside
Sheet, approx. 80 x 80 cm of brown wrapping paper, Kraft

Dry Component Pre-mix



1. Select a card giving the data for specific mix.
2. Weigh and sift; through a separate sieve into a separate container each material according to the card. Screening is necessary to break up materials and remove dirt. Screening is done on weighed material, not bulk material in storage containers.
3. Place weighed and sifted materials on a sheet.
4. Blend the materials by raising and lowering alternately left and right side of the sheet causing the materials to flow and roll together.
5. When the mixture obtains a uniform (gray) color and there are no patches of unblended material stop the blending.
6. Lifting the sheet dump the blended mixture into the corresponding labeled container.
7. Place container in cabinet.
8. Clean up the area and check that all bulk storage containers are closed.

GUM SOLUTION

The gum solution is added to the final priming mix as a binder for other components. After the primer is assembled and dried, the gum solution prevents the mix from crumbling and/or falling out of the cup.

The gum solution is neither explosive, nor a fire hazard. With exception of thymol and the coloring agent, the solution is essentially non-toxic.

Gum solutions of various formulations and concentrations are used for different primers, as listed below; they all meet the U. S. Mil. specifications.

Equipment:

Table
Scale, 1 Kg capacity
Scoop, stainless steel
Pail, 5 liter capacity, stainless steel
Sink, stainless steel, approx. 50 x 100 cm
Stirrer, electric, laboratory type
Stirrer, glass rod
Jar, 2 liter capacity, stainless steel
Flask, stoppered, cc capacity; required ea.
Shelving, commercial, office type

Materials:

All materials are readily available from commercial sources.

Formulation:

The tabulation below lists typical gum solutions used in priming mixes.
All weights are DRY weight in grams, except where noted.

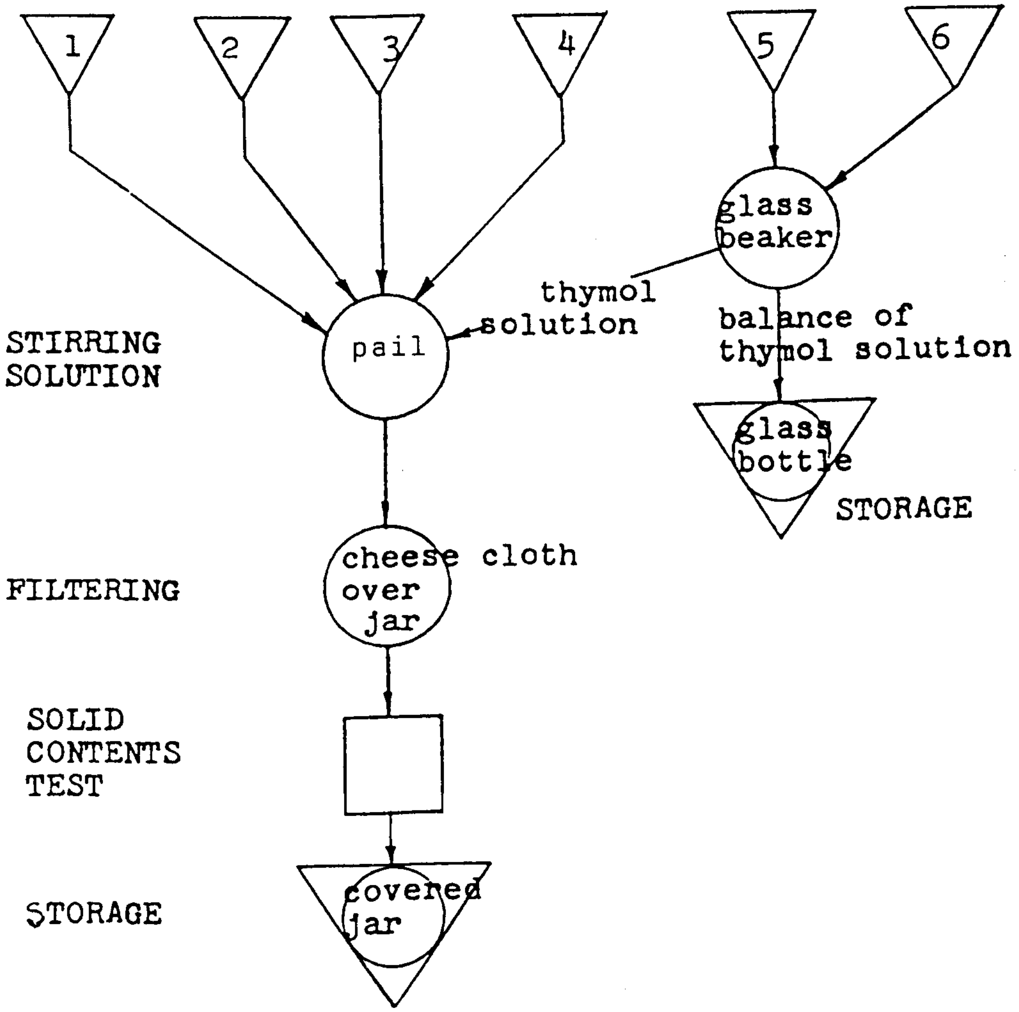
Primer mix	C.F.& S.S.	RF	.50 cal.	primer	F70 (#956)
Gum mix	std.	RF	heavy	foiling	light
Gum arabic	34.3	54.0	92.5	15.0	17.0
Gum tragacanth	17.2	-	46.2	7.2	9.4
Gelatine	3.7	-	-	1.13	1.85
Thymol solution-cc	2.65	1*	4.0	10.0	2.0
Dye**)	-	6.1	-	-	-
H_2O -cc		100			
Total vol/batch	1000 cc		1000 cc	1000 cc	1000 cc
Total wt./batch	1010		1030	1005	1005
Wt. solids/batch	54.2 ± 1	55 ± 2	139 ± 2	23.5 ± 3	28.3 ± 3

**) Dyes are used for priming mix identification only, various manufacturers use Prussian blue, Malachite green, etc. for this purpose. The dry dye is generally dissolved in ethyl alcohol prior to addition to the gum solution.

SAFETY PRECAUTIONS

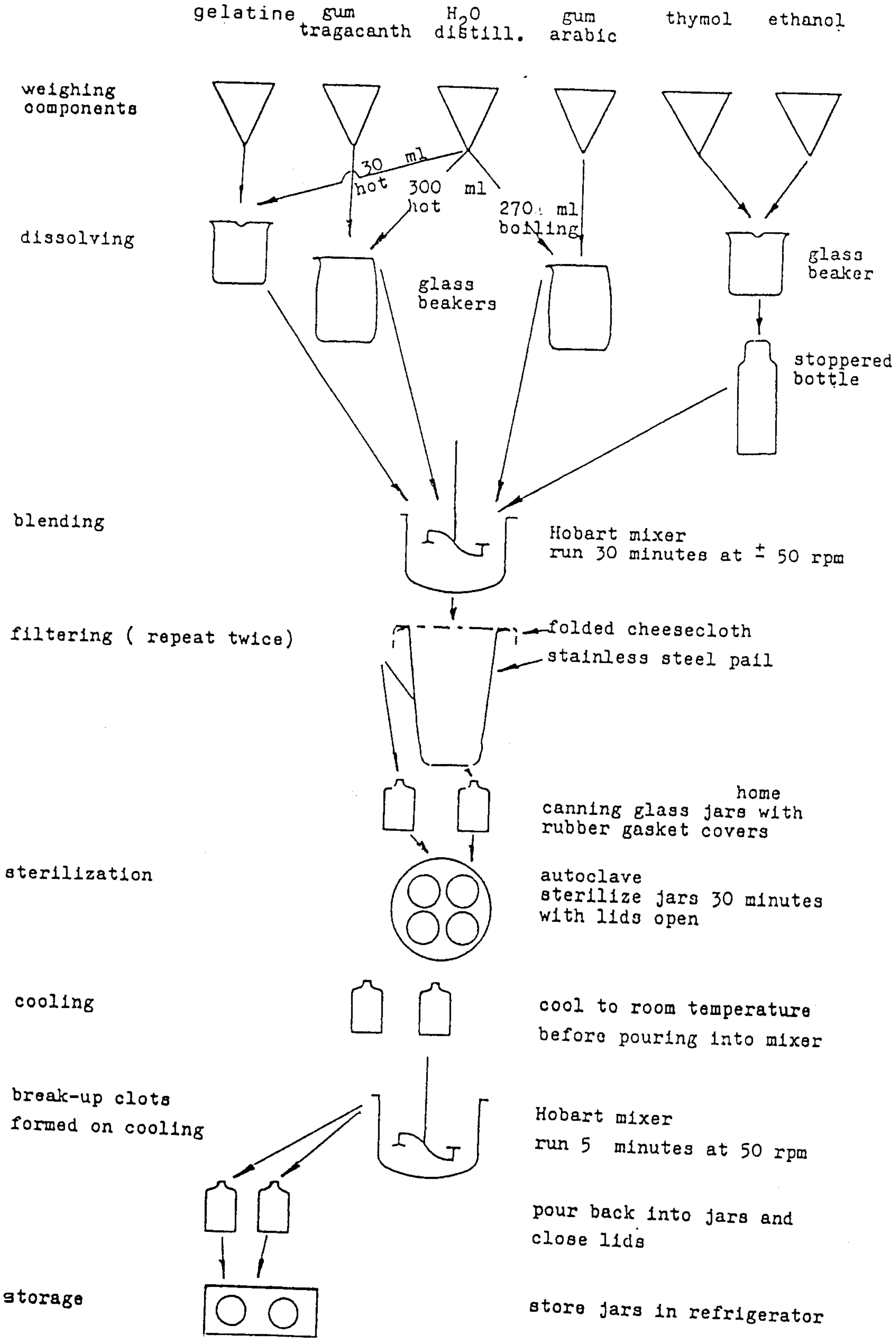
Only basic good housekeeping practices to be followed. All gum solution is to be properly labeled showing the type of mix and date of manufacture.

Process diagram



1 - H_2O
2 - gelatine
3 - gum tragacanth
4 - gum arabic
5 - thymol
6 - ethyl alcohol

GUM SOLUTION



Thymol Solution Preparation

Thymol	2 g
Alcohol, ethyl	10 cc

Pour alcohol into suitably sized glass jar. Add thymol and stir manually at ambient temperature with a glass rod stirrer until dissolved.
Pour solution into screw cap bottle. Label properly.

Dye solution preparation

dye	4 g
alcohol, ethyl	100 cc

Pour alcohol into a suitably sized glass jar
Add dye and stir at ambient temperature using electric stirrer.
Pour solution into glass stoppered bottle.
Label properly.

Procedure, Gum Solution:

1. Determine moist weight of each ingredient.
2. Weigh required amount to get correct DRY weight of each ingredient.
3. Place all weighted ingredients into one glass container properly labeled.
4. Clean & weigh mixing pail.
5. Fill sink with hot water and heat to 75° ± 5° C (not over 80°)
6. Fill pail with 950 cc demineralized water.
7. Place pail with hot water into the sink.
8. Insert and start electric stirrer.
9. Sprinkle dry ingredients into the pail gradually to avoid formation of lumps which will be difficult to dissolve*).
10. Continue stirring the solution 1 hour while maintaining the sink water temperature at 70° ± 5° C.
11. After 1 hour remove pail from sink and wipe outside dry. When stirred for more than 1 hour the solution tends to coagulate and gel. Stop stirrer.

12. Weigh pail with gum solution while still hot and add water to bring it to the values listed in table.
13. Replace pail in sink under stirrer.
14. Drain hot sink water and refill with cold water.
15. Start stirrer and continue stirring until gum solution temperature drops below 36° C.
16. Add thymol solution.
17. Continue stirring five (5) min. more.
18. Filter gum solution through 2 thicknesses of cheesecloth into another clean dry jar.
19. Wring out cheesecloth into the second jar to get all gum out.
20. Cover jar and place in cool place, (sink with running water).
21. Take sample of gum solution and determine solid contents. If gum solution does not conform to specifications, it is scrapped. Gum solution older than 20-22 days is also scrapped.

*)Dye for rimfire mix may be added to the gum arabic in dry state prior to step 9.

PURPOSE: Moisture determination of gum solution ingredients.

EQUIPMENT:

1. Laboratory mortar and pestle
2. Tared weighing bottle
3. Analytical laboratory balance
4. Laboratory drying oven

MATERIALS: none

Note: The gums that are received from vendors contain about 15% moisture.

SAFETY PRECAUTIONS: Standard laboratory

PROCEDURE:

1. ± 10 g. of material is ground so that particle size is about 3mm.

2. Sample is placed in weighing bottle and weighed, data are recorded.

3. Bottle with sample is placed in drying oven at 100° C for 24 hours.

4. Bottle with sample is removed from dryer, weighed and data recorded.

5. Continue trying and weighing sample at 60 minutes intervals until loss is 0.25% or less per 60 minutes of drying time.

6. Moisture percentage is:

$$\%\text{ m.} = \frac{\text{initial wt. (1)} - \text{final wt. (4)}}{\text{initial wt. (1)}} \times 100$$

7. The most weight of the gum to be used is:

$$\text{moist wt.} = \frac{\text{dry wt. (per formula)}}{\text{100\% moisture (6)}} \times 100$$

PURPOSE: Determination of solids percentage in gum solution.

EQUIPMENT:

1. Tared weighing bottle
2. Analytical laboratory balance
3. Laboratory drying oven
4. Desiccator

MATERIALS: None

SAFETY PRECAUTIONS: Standard laboratory

PROCEDURE:

1. ± 20 cc of gum solution is placed in weighing bottle.

2. Sample is weighed and data recorded.
3. Bottle with sample is placed in drying oven at 100° C for 24 hours.

4. Bottle with sample is removed from the oven, covered and placed in the desiccator to cool at ambient temperature.

5. Dry cool sample is weighed and data recorded.
6. Percentage of solids calculated as:

$$\text{wt. of solids/batch} = \frac{\text{wt. of sample dry (5)}}{\text{wt. of sample wet (1)}} \times \text{wt. of gum solution in batch}$$

PETN Preparation

GENERAL:

PETN is added to the priming mix to improve the primer brisance power. It is required by the U. S. Mil. specs of primers and is also used in many commercial primers. Since the PETN is used in relatively small amounts in the primers, it is economically not justifiable to make PETN in the primer plant. It is usually purchased from manufacturers such as Hercules Powder, who make PETN in large quantity for other uses, such as blasting caps, explosives, etc.

PETN is shipped in wet condition to reduce its sensitivity. To prevent freezing of wet PETN in transit and/or storage, alcohol is added to the water.

The PETN soaked with water-alcohol must be washed free, before use in priming mix. Obviously, in mixes not containing PETN, or where it is made in the plant, such washing operation is eliminated.

Large operations requiring several kilograms of PETN per week use a centrifuge capable to process approximately 7 to 10 Kilograms of PETN per load. The washed PETN is then weighed and separated into exact quantity, each corresponding to one wet priming mix batch. Following is a simple way to wash the PETN for laboratory use.

EQUIPMENT:

Beaker, pyrex glass, 1500 ml capacity
Scoop, small, stainless steel
Stirrer, paddle, stainless steel
Funnel filter with aspirator
Wash screen, stainless steel, to fit over beaker
Sink, wash, stainless steel, approx. 100 cm long by 50 cm wide by 25 cm deep
Container, hard rubber or plastic, 250 ml capacity
Cover, hard rubber or plastic, to fit over container
Balance, 1000 gram capacity, ± 0.5 gram accuracy

MATERIALS:

Water, clean, tap
PETN, specification MIL-P-387, class 2

APPEARANCE: White or light buff color powder

REQUIREMENTS:

Granulation through U.S. Std. sieve:	No. 100	94 % min.
	No. 200	80 % max.
	No. 200	65 % min.

Melting point 140° C min.
Stability at 120° C, vacuum 5 cc gas max. in 24 hrs. test

Composition:	Nitrogen contents	17.50% min.
	Acetone insoluble	0.10% max.
	Particle insoluble	0
	Acidity or alkalinity	0.003% max.

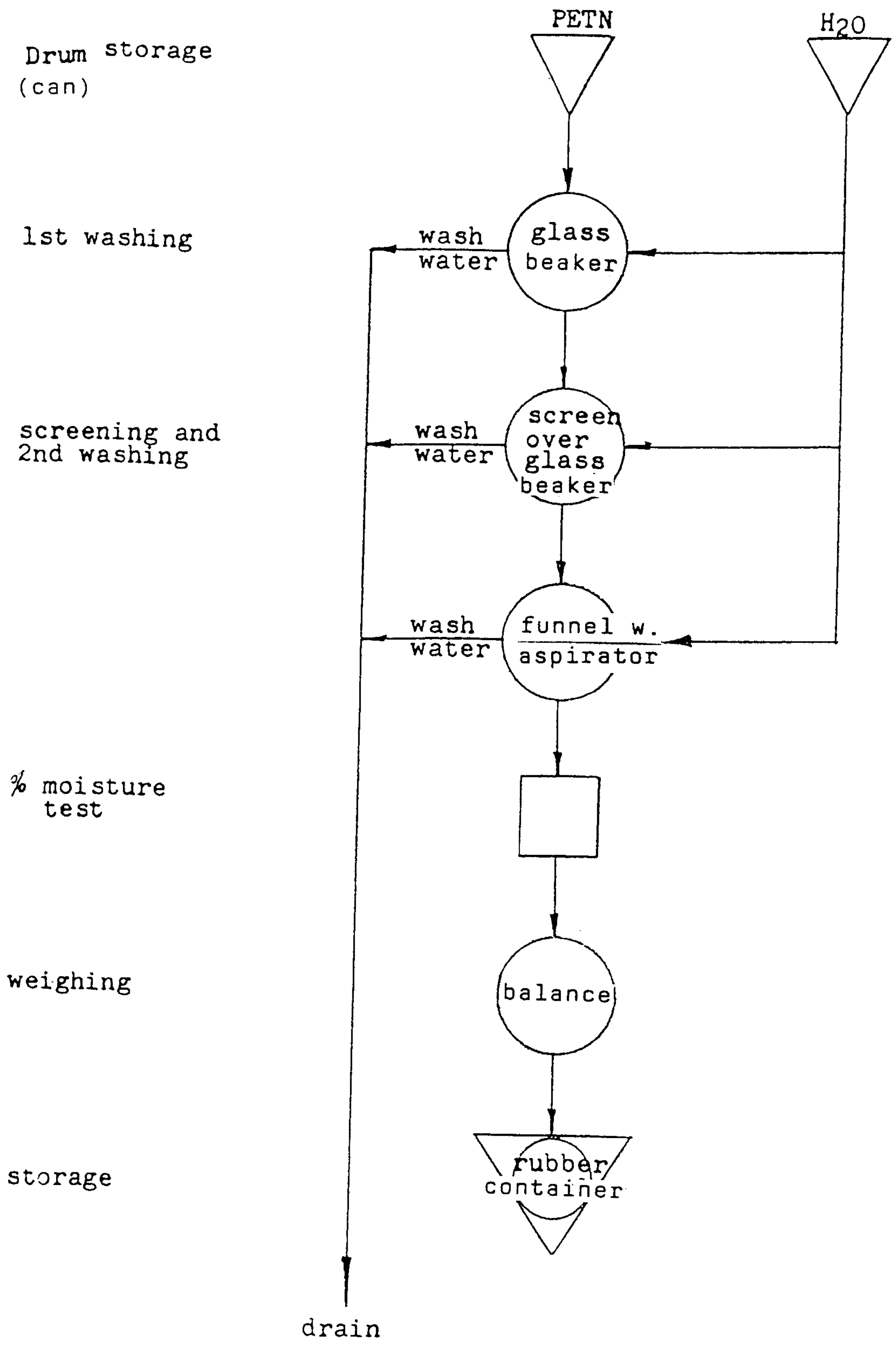
Packing: PETN slurry containing minimum 40% water packed in rubberized cloth bags each containing approximately 20 Kg (dry weight) PETN. For cold weather packing PETN slurry must contain minimum 20% denatured alcohol and 20 ltr. water. Six or less rubberized bags are placed in steel drum lined with rubberized cloth or polyethylene bag and the empty space in the drum is filled with water or 50-50% alcohol-water solution before closing of the drum. Each container labeled in accordance with local regulations on shipment of high explosives.

Inspection: Certificate of analysis to be furnished with each lot by the supplier. 250 g each, samples from five containers in a lot are tested separately as per following procedures.

Safety Precautions:

1 - Eye protection to be worn when handling PETN.
2 - Only personnel trained and approved by explosive engineer may handle PETN.
3 - PETN is a secondary explosive presenting fire hazard in dry condition.
4 - All spilled PETN to be cleaned up immediately before it dries.
5 - Area where PETN is handled to be washed with hose after use.
6 - Primary explosives must not be present in building used for PETN washing.
7 - PETN must always be labeled.

Process diagram



PROCEDURE: Washing PETN

1 - The purpose of the washing is to extract the antifreeze from PETN.
2 - The bulk PETN is shipped in metal cans. Inside the can is a rubber bag. PETN is packed in a separate bag inside this rubber bag. The remaining space in the can is filled with water-methanol mix to prevent freezing and damage to the drum in cold weather.
3 - Open the barrel, untie rubber liner and fold the open ends over the can sides
4 - Remove PETN bag and place it in the wash sink.
5 - Open the bag and place about half of the contents into the pyrex glass beaker.
6 - Fill beaker with PETN with water, using the water stream to stir and suspend PETN.
7 - Allow PETN to settle and decant water off.
8- Transfer PETN with scoop from the beaker onto screen.
9 - Wash PETN through screen into another beaker using water stream.
10 - Fill beaker with water using the stream to stir and suspend the PETN.
11 - Line the bottom of the funnel with filter paper; pour the suspended PETN onto the funnel.
12 - Start the aspirator.
13 - Rinse the beaker and transfer its contents onto the funnel.
14 - Spray a jet of water onto the PETN in the funnel, but carefully, to prevent splashing.
15 - Stop the water spray.
16 - Run the aspirator until the PETN appears only moist (it will contain about 15% water).
17 - Stop the aspirator.
18 - Remove the filter paper with PETN and place into a storage container.
19 - Remove a portion of the PETN from the container and weigh very accurately the quantity required for one batch of wet priming mix. The moisture percentage has been calculated previously in the laboratory and the operator-student has made the corresponding allowance for the moisture contents.
20 - Transfer the weighed quantity into a rubber/plastic container, cover it tightly and label with PETN quantity, date and lot.
21 - The unused wet PETN is replaced in the first container, covered and returned to the storage cabinet/magazine.

Drying PETN

Occasionally dry PETN is required. Following is the procedure for handling and storage of dry PETN.

1 - The PETN from step 18 (previous procedure-washing PETN) is hand carried to a separate and secure location for drying.
2 - PETN is spread, with gloved hand, gently on a shallow tray and covered with a sheet of paper in dryer at 25 - 30° C.
3 - Dry PETN is transferred into a fiber (cardboard) container and left stored in the dry room.
4 - Portion required for mixing is weighed in the PETN weighing area, placed in a rubber/plastic container, covered and labeled.
5 - The fiber-cardboard container with the unused dry PETN is immediately returned to the drying area.

PURPOSE: Moisture determination of PETN after washing

EQUIPMENT:

1 - Tared weighing bottle
2 - Analytical laboratory balance
3 - Laboratory drying oven

MATERIALS:
None

SAFETY PRECAUTIONS: Std. laboratory

PROCEDURE:

1 - ± 10 g sample of PETN is removed from each container and placed in tared weighing bottle.
2 - Sample is weighed and data recorded.
3 - Bottle with sample is placed in drying oven at 55° C for 12 hrs. (overnight).
4 - Bottle with sample is removed from the oven, covered and cooled.
5 - Bottle with dry sample is weighed and data recorded.
6 - Percentage of moisture (as ratio of loss in weight to the original weight) is calculated and recorded.
7 - Operator weighing PETN for explosive premix batches is notified.

Trinitroresorcinol (Styphnic acid) preparation

GENERAL:

TNR is an intermediate material in the manufacture of lead styphnate. Although not a primary explosive, TNR is a relatively strong acid forming sensitive metallic salts upon prolonged contact with various metals. It does not react readily with stainless steel, or copper and therefore these materials are used in TNR manufacture and storage.

TNR is prepared from resorcinol by sulfonation and nitration, then washed and dewatered for storage.

Two types of TNR may be prepared:

a – "Gable" type - light yellow or orange, very pure, melting point 174° ± 1° C. Lead styphnate made from this material has thin and twinned crystals and is more sensitive than LS made from adulterated TNR. The Gable TNR has been used for the manufacture of double salt of styphnate lead hypophosphite.

b – "Adulterated" type formed by addition of sodium nitrite during the sulfonation stage. In all other respects the preparation follows the conventional method of preparation. The Adulterated TNR is cocoa brown in color with melting point of ± 155° C and a melting range of several degrees.

The degree to which the above reactions are formed depends on:

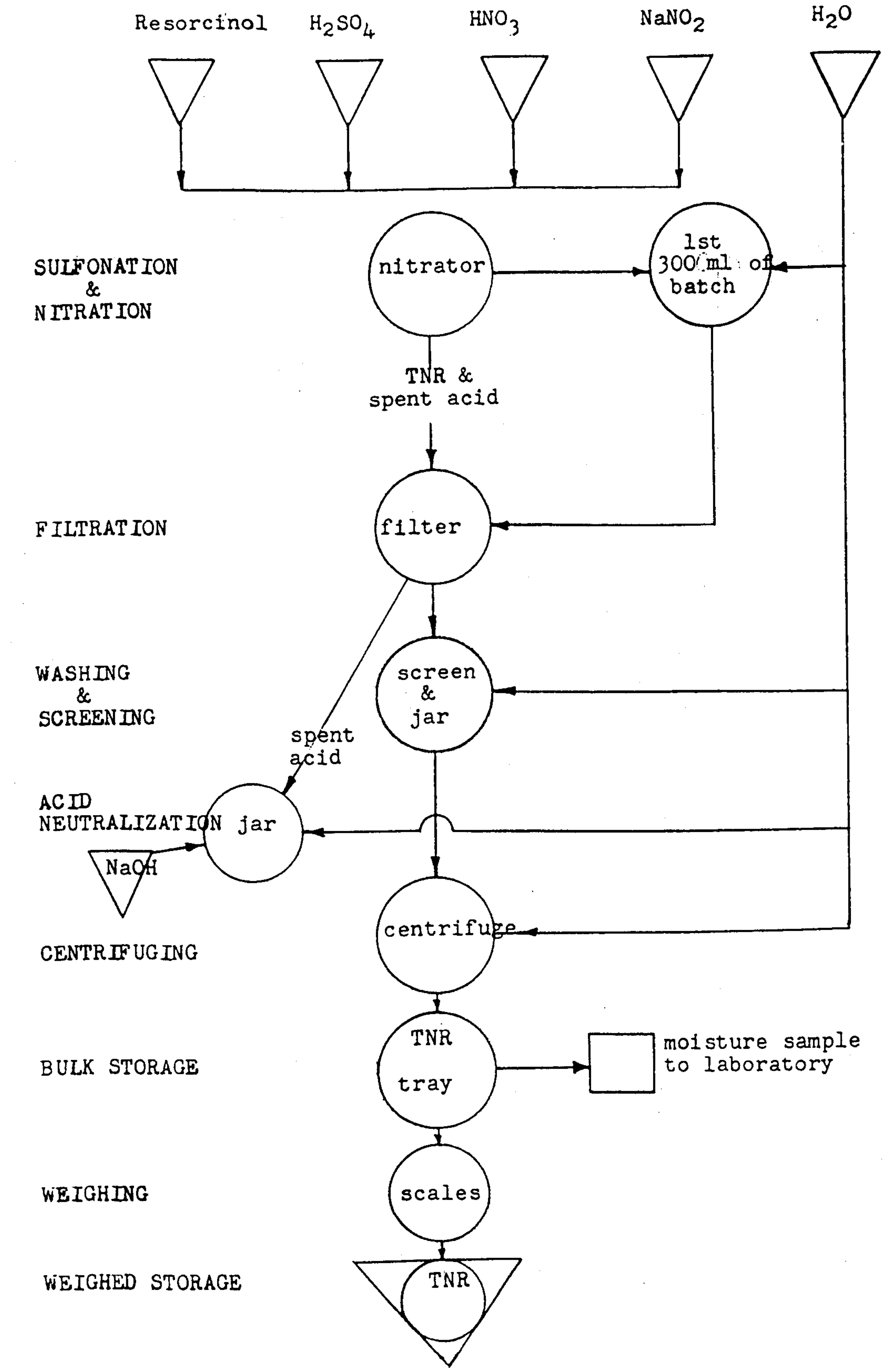
- Temperature and time when $NaNO_2$ is added, as it affects the degree of HNO_2 decomposition.
- Rate of addition of $NaNO_2$; rapid addition causes local concentration of $NaNO_2$; salt and increased loss of HNO_2 due to high local of reaction.
- Temperature of sulfonation and nitration.

The TNR preparation process described is based on the methods developed in the early forties, but is still in use by the military and commercial producers throughout the world.

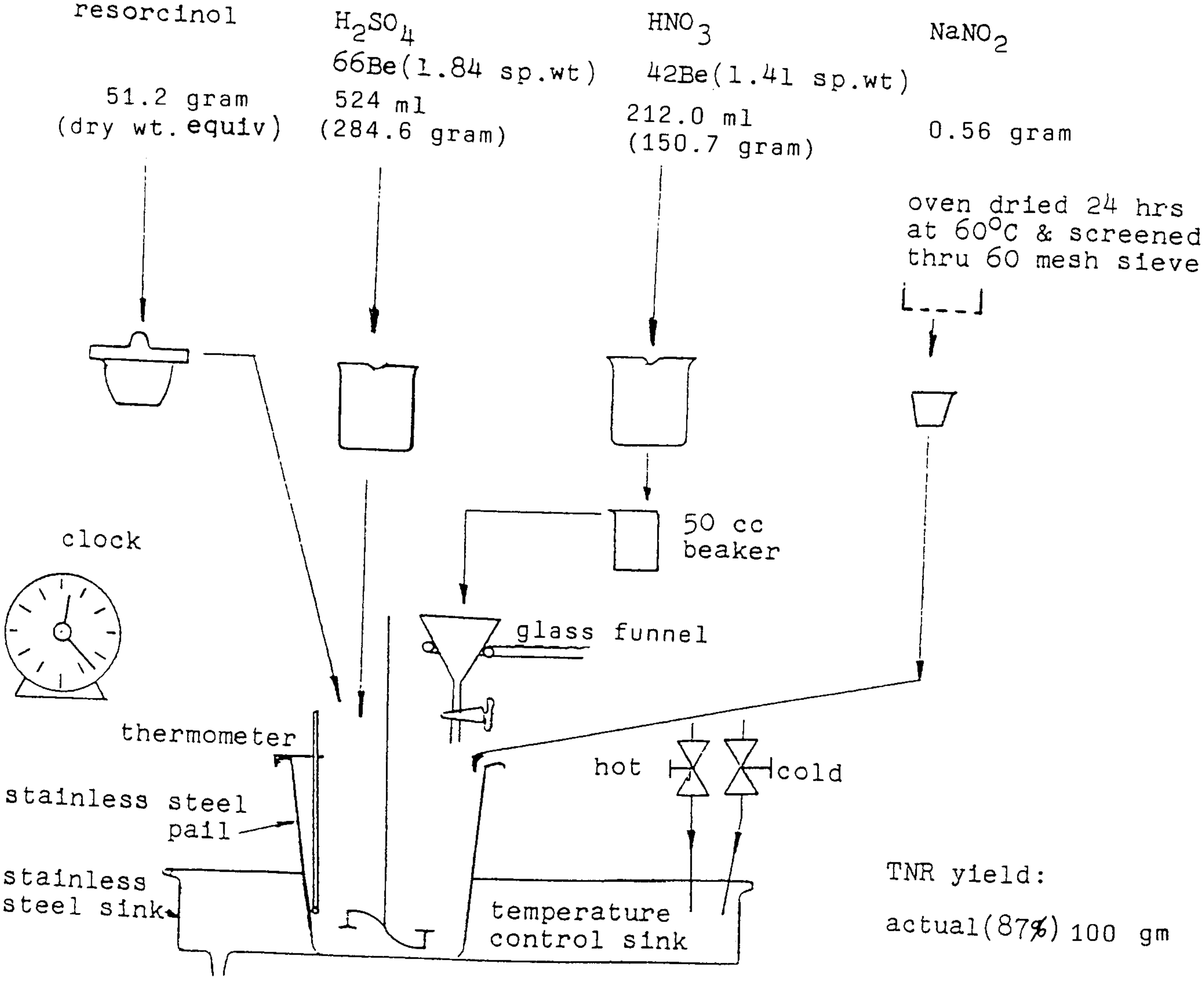
Safety precautions - TNR

1. Eye protection to be worn at all times in the nitration area.
2. TNR is an explosive, even if relatively insensitive, and must be handled carefully.
3. Poisonous nitric oxide fumes are generated during nitration and neutralization. Prolonged exposure to the fumes must be avoided.
4. All materials used in the TNR preparation are irritating to the skin; rubber gloves to be worn when handling the materials.
5. If resorcinol, or caustic get on skin, flush the area involved immediately with plenty of tap water.
6. If acids get on skin, flush the area involved immediately with plenty of tap water, then with sodium bicarbonate solution.
7. Report to infirmary for further care immediately after contact with materials used in TNR preparation.
8. Sometimes nitric acid is delivered in carboys; place rubber cap over the mouth and neck of carboy before moving, to prevent splattering of acid soaked into the stopper seal.
9. A bottle with sodium carbonate solution is to be kept in easily accessible location.
10. Hands and face of personnel in the TNR preparation area must be washed before meals and/or smoking.
11. Nitrator must be cleaned thoroughly after each batch; exposure of sulfuric acid to oxides of nitrogen or nitrous acid causes formation of nitrosyl sulfuric acid which in turn causes formation of crystal control impurities in the TNR.
12. Heating and cooling of the nitrator batch must be done in conformity with the instructions of the nitrator manufacturer to prevent thermal shock of the nitrator material or liner.

Process diagram



TRINITRORESORCINOL
LABORATORY PREPARATION



TNR yield:

actual(87%) 100 gm

TRINITRORESORCINOL
LABORATORY PREPARATION

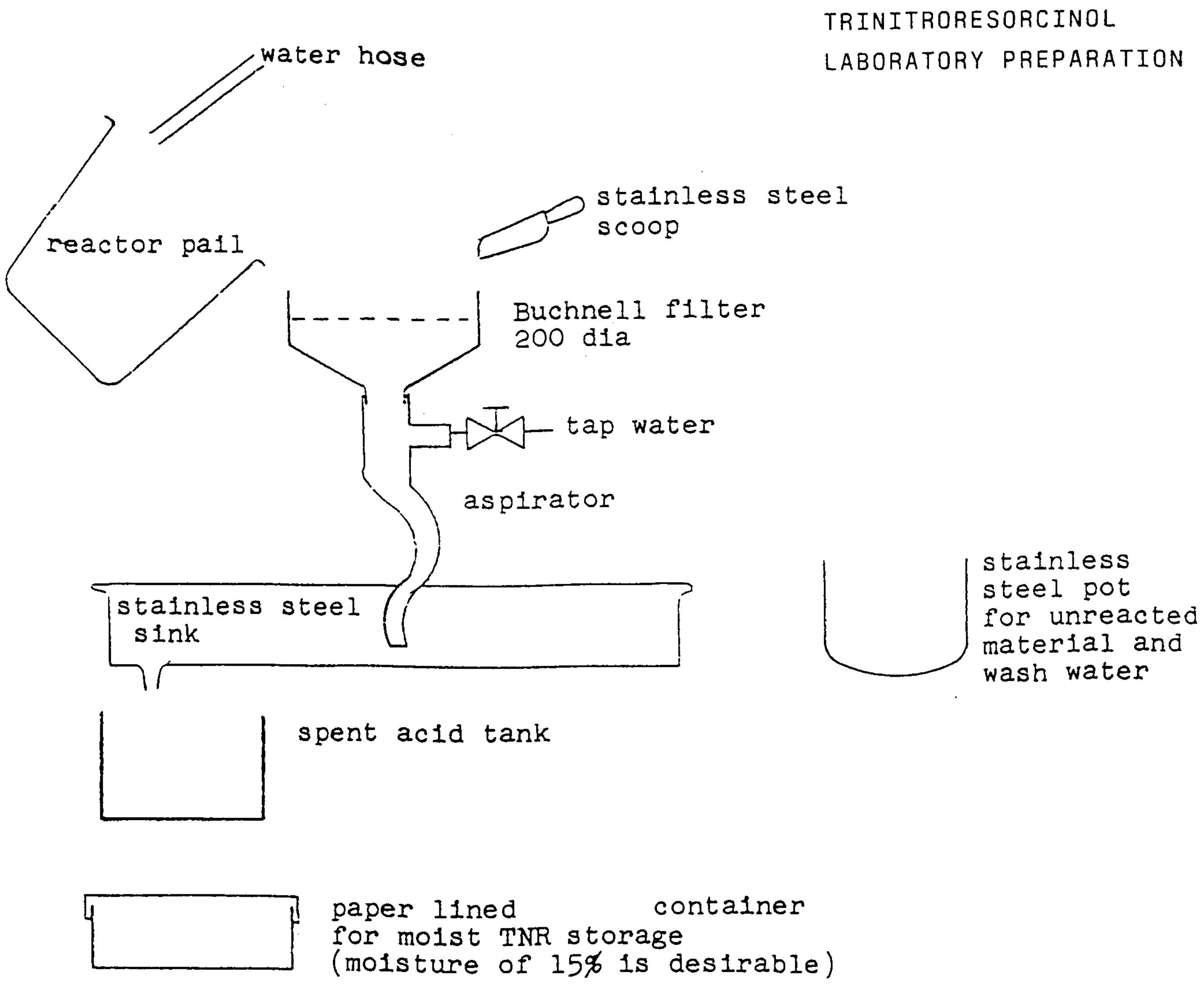
Sulfonation:

1 - Pour the correct amount of H_2SO_4 into the pail.
2 - Start stirrer, checking that it does not touch pail walls.
3 - Fill sink with hot water until the pail contents is 30° ± 5° C.
4 - Add 1 level teaspoon full of resorcinol to the pail contents.
5 - Add a little of $NaNO_2$ from the jar and continue through sulfonation.
6 - Add hot water to sink to raise the pail batch temperature to 63° C.
7 - Resume gradual addition of resorcinol so that the last is added in about 15 minutes from process start.
8 - Allow batch to remain at 60° C for 60 minutes after last resorcinol has been added.
9 - Cool gradually to 30-35° C (about 12 minutes).

Note: The batch is now ready for nitration which may be carried to the point permitted by time. The nitration may be resumed for completion later. However, let stirrer run, wait about 10 minutes after last addition of HNO_3 then change sink water temperature to keep batch at 18° C. Before nitration resumption bring batch to 30° C.

Nitration: (start with batch at 30° C)

1 - Measure 10 cc from glass beaker into the nitric acid reservoir.
2 - Open reservoir valve to get flow rate of about 7 cc/minute.
3 - Repeat step 1 until 100 ml, of HNO_3 has been added. Since the reaction is exothermic, cooling of the batch is necessary to keep the temperature at 35° C.
    a) if during the process, temperature drops below 23° C, the reaction will slow down and concentration of unreacted HNO_3 builds up. Then as the temperature raises this acid will react rapidly, causing foaming over the pail, loss and decomposition of the TRN. Therefore when the temperature drops add hot water to the sink to heat the batch back to 35° C.
    b) If the batch temperature raises over 38° C, stop addition of HNO_3 and let batch to cool down to 35° C before resuming process.
4 - After the 100 ml of HNO_3 have been added, stop batch cooling and allow the pail contents to heat up to 60° C; however,
5 - Resume addition of HNO_3 immediately, without waiting for the batch to reach 60° C.
6 - When all HNO_3 has been added start the cooling of the batch.
7 - When the batch is about 30° C (in about 20 minutes) it is ready for filtration.
8 - Stop the stirrer and remove it from the pai.l


TRINITRORESORCINOL
LABORATORY PREPARATION
water hose
reactor pail
stainless steel scoop
Buchnell filter 200 dia
tap water
aspirator
stainless steel sink
stainless steel pot for unreacted material and wash water
spent acid tank
paper lined container for moist TNR storage (moisture of 15% is desirable)

TRINITRORESORCINOL
LABORATORY PREPARATION

Filtration:
Performed in well-ventilated area or under hood.

1 - Aspirator is turned on and checked for leaks.
2 - Reactor pail contents are slowly ladled onto the filter which draws off the spent acids from the material.

Notes:
The bottom portion of the batch can be blue-purple in color and contains unreacted resorcinol and mixed acids. It will react with the spent acid as it is mixed on the filter and the temperature will rise. Allow this unreacted material to stand and combine with the next batch. If the unreacted blue-purple material is poured on the filter bed the heat of reaction may ignite the TNR.

3 - After the pail has been emptied rinse it with tap water and add the wash water to the unreacted materials. Also, carefully rinse the stirrer shaft and blades and add this water to the wash water.
4 - After all/most spent acid has been drawn off the filter pour the contents of previous batch wash water over the material on the filter, turning and spading the material during the process.
5 - After all wash water has been added, wash the filter contents with vigorous jet of tap water for 3-5 minutes.
6 - Continue aspirator operation for about 30 minutes.
7 - Take 5 g sample of TNR and check for moisture and sulfate. If sulfate contents exceeds 0.5% additional washing (step 5) is required.
8 - Acceptable, moist TNR is ladled into storage contained, dated and labeled.

Notes:
The spent acids are either diluted to 0.5% concentration or neutralized with alkali before dumping into sewer. Approximately 1 Kg of flaked NaOH is required for one batch operation. The neutralized solution has a deep brown color.

TNR ADULTERANT FOR LABORATORY PREPARATION

USES: To introduce impurity into styphnic acid causing crystal growth in the precipitation of lead styphnate

SAFETY PRECAUTIONS: Same as in TNR manufacture

EQUIPMENT: Same as in TNR manufacture

PROCEDURE:

1 – Measure 100 ml of distilled or demineralized water into a jacketed stainless steel kettle.
2 – Heat the water to 25 cc.
3 – Add 33 gram resorcinol.
4 – Add 42 gram $NaNO_2$ and dissolve in solution.
5 – Bring solution to boil and keep simmering 60 minutes.
Note: overheating causes excessive gas evolution.
6 – Transfer solution from kettle into stainless steel pails.
7 – Cool solution in pails at least several hours.
8 – Place 200 g crushed ice into a 3 liter stainless steel pot (in a sink).
9 – Over this ice pour 120 ml 42 Bé HNO_3.
10 – Start the agitator.
11 – Add the solution from step 7 at a rate of approximately 3 ml / minute.
12 – Keep the pot contents temperature at 2 ± 2° C throughout step 11 by adding small quantities of ice.
13 - Heat the solution in pot to 30° C to start the exothermic reaction which continues for approximately 30 minutes with copious gas evolution.
14 – After reaction starts allow temperature drop 5° C from the maximum reached.
15 – Heat solution to 70° C keeping in mind that the pot contents temperature will continue to rise for about 10 minutes after the circulating hot water is shut off.
16 – Continue agitating the solution 15 minutes after the temperature of 70° C has been reached.
17 – Cool the solution in pot to 35° C by circulating cold water in the sink.
18 – Line the filter bed with filter paper, then cover the paper with muslin cloth.
19 – Carefully pour the pot contents over the prepared filter.
20 – Start the aspirator to draw the mother liquor off.
21 – Wash the adulterant on the filter bed with tap water, breaking the caked adulterant with gloved hand in the process.

22 – repeat step 21 three: (3) times starting the step after all wash water from the preceeding operation has been drawn off.

Note: do not break the muslin-filter paper pad during this operation.

23 – Lift the muslin, cloth with the adulterant from the filter and dump the adulterant on stainless steel drying tray.

24 – Dry the adulterant in open air for 4-6 weeks; in emergency it may be dried in a dryer at a maximum temperature of 50° C.

Note: The average adulterant yield is 250 gram/ batch.

25 – Place 200 ml of distilled or demineralized water into a 3 liter capacity stainless steel pot.

26 – Turn on agitator.

27 – Add 2.2 gram 100% NaOH pellets.

28 – Add 11.3 gram of dry adulterant from step 24.

29 – Continue agitation 15 minutes to assure that all materials are in suspension.

30 – Stop agitation and allow foam to clear.

31 – Add distilled or demineralized water to bring the solution volume to 230 ml.

Notes:

– The quality and quantity of adulterants to be added to the pure TNR is determined in a test precipitation of lead styphnate.

– Exercising care in performance of each step above is the only way of assuring uniform performance from batch to batch.

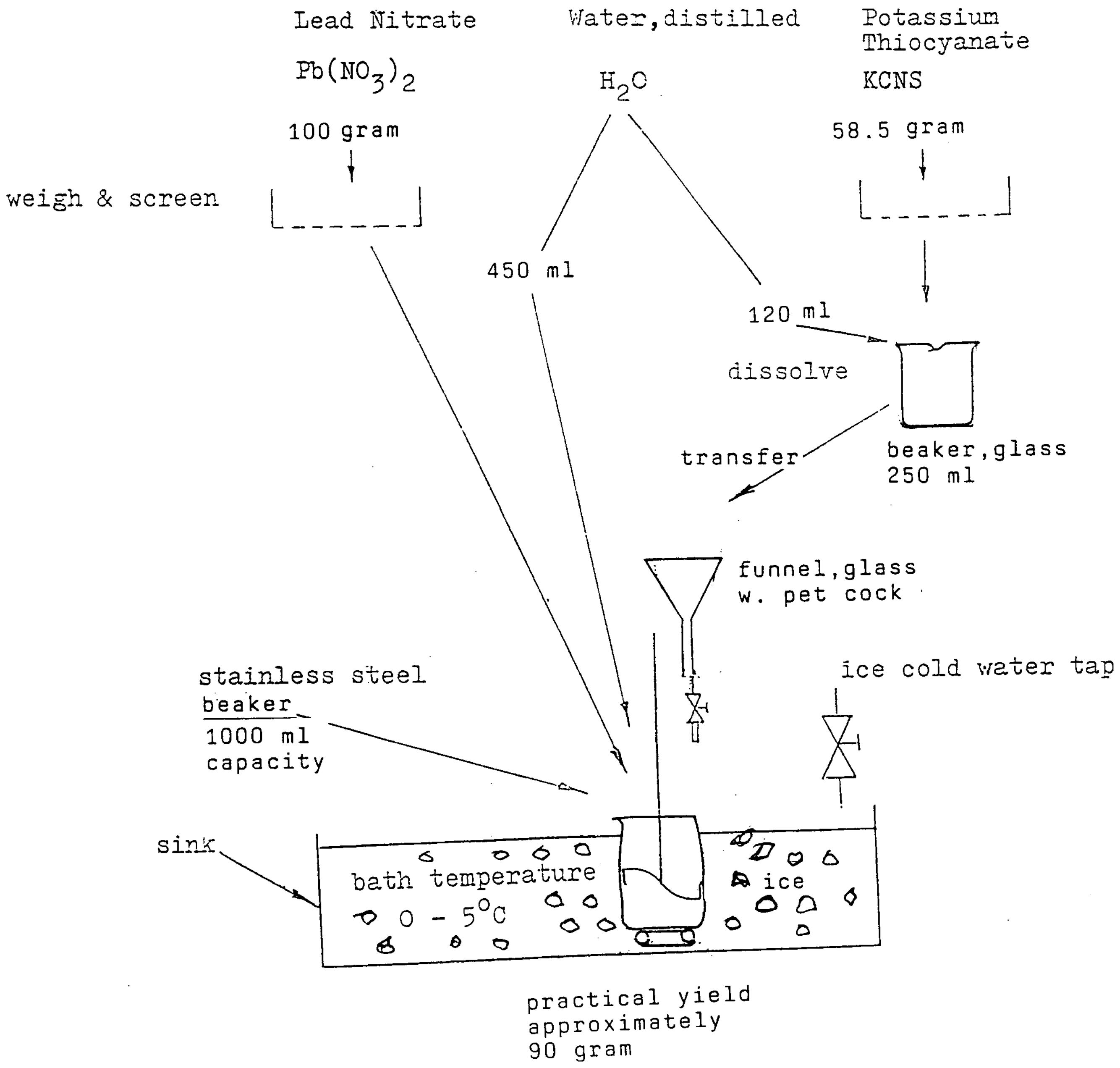
– The adulterant is classified as an explosive and should be handled accordingly .

Addition of the exact amount of $NaNO_2$ at the proper time and rate, and maintenance of correct temperature schedule produce uniform adulteration of TNR from batch to batch.

Lead styphnate made from a new batch of TNR is to be checked under microscope.

U.S. Patent No. 2275172, lists catechol, phenol, tri-oxymethylene, glucose and aluminum powder as materials causing impurities in TNR which control crystal growth in lead styphnate.

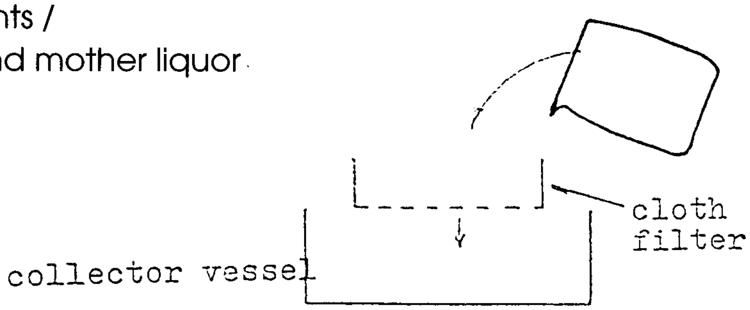
LABORATORY PREPARATION OF LEAD THIOCYANATE



LABORATORY PREPARATION OF LEAD THIOCYANATE

PROCEDURE:

1 - Place the screened lead nitrate and water into the beaker.
2 - Start stirrer and continue until all nitrate is dissolved.
3 - Pour ice cold water into the sink.
4 - Check that the lead nitrate solution temperature drops to 5° C.
5 - start addition of potassium thicyanate solution at a rate of about 3 ml/minute (about 1 drop/second).
6 - Continue process until all materials have been placed into the beaker.
7 - Stop stirrer.
8 - Pour beaker contents / precipitate and mother liquor onto filter.



9 - Rinse beaker with ice cold deionized water and pour rinse water over precipitate on filter.
10 - Wash precipitate twice with small jet of ice cold water.
11 - Place cloth from filter with washed precipitate into DARK and dry in warm air.
12 - The yield is about 94% theoretical, i.e. 90 grams.

13 - Check a dry sample for crystal size and adjust step five (5) for optimum product size.

14 - Mother liquor and wash water are boiled down for recovery of potassium nitrate.

LEAD THIOCYMTATE PREPARATION:

The theoretical equation for the reaction is:

$$\mathrm{Pb(NO_3)_2} \quad + \quad 2\ \mathrm{KCNS} \quad \longrightarrow \quad \mathrm{Pb(CNS)}\downarrow \quad + 2\ \mathrm{KNO_3}$$

$$331.2\ \mathrm{g} \quad + \quad 2 \times 97.2\ \mathrm{g} \qquad\qquad 323\ \mathrm{g} \quad + 2 \times 101\ \mathrm{g}$$

The reaction is carried out at low temperature to reduce the solubility of lead thiocyanate (0.05 g/100 ml at 20° C) and hence increase the yield.

Since the product is light-sensitive, it is dried in a dark area and stored on containers not transmitting light.

The purity of the product meets the specifications

The required granulation may be affected by the concentration of the solution, the rate of potassium thiocyanate addition, and the cooling temperature.

The quantities and process have been adjusted to allow the use of lead styphnate precipitation equipment and methods.

SAFETY:

Lead thiocyanate is relatively safe to make from an explosive point of view; but its preparation and the product are extremely TOXIC.

Utmost protection of personnel from direct contact by use of masks, goggles, gloves, work cloths,etc. is required.

To keep dust and fumes from personnel contact, the work area is equipped with powerful and positive exhaust system with filters and traps to keep the outside area from contamination.

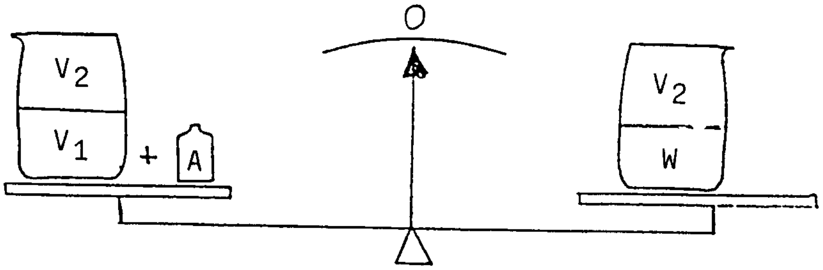
General

The primary explosives of the priming mix are dangerously sensitive. To reduce their sensitivity during processing, explosives are in wet, water saturated state. They are weighed and blended prior to addition to other components of the priming mix.

The technique of wet weighing goes back to antiquity. Every student of high school physics knows about Archimedes weighing of the golden crown.

The wet explosive weighing uses the same method, but since only few components are being weighed time after time, certain simplifications have been introduced into the technique described below:

Using the basic Archimedes law of physics:



V_2 = volume of water over the wet material
V_1 = volume of water equal to volume of wet material
W = weight of wet material = V_1 x Spec. Density of material
A = Archimedes differential weight
Note that Spec. Density of water = 1

then the weights become: $V_2 + V_1 + A = V_2 = V_1 x S$

transposing

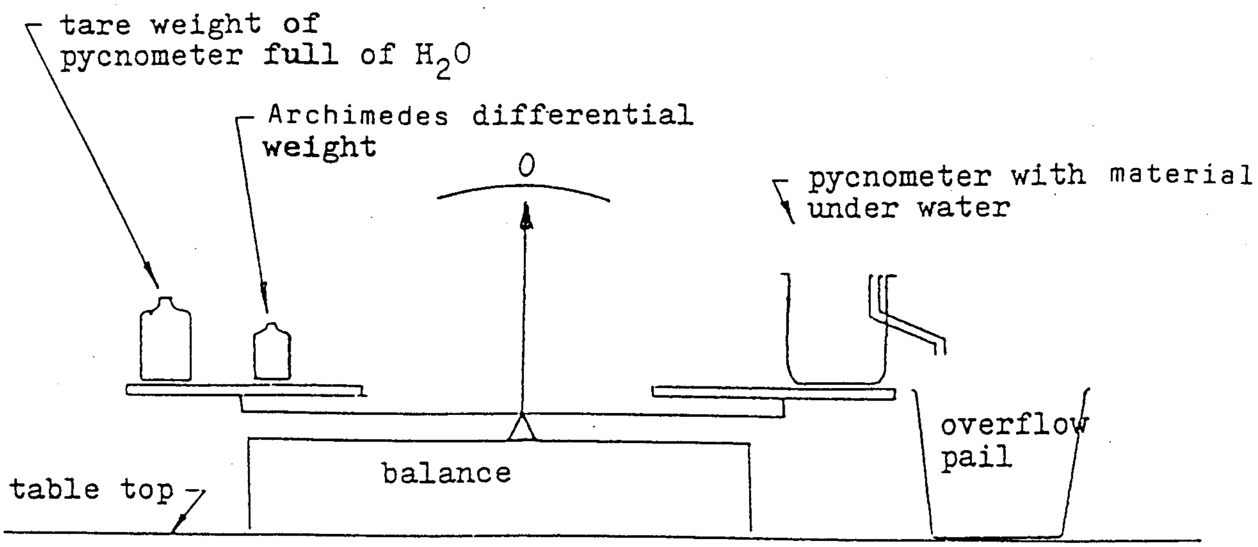
$$A = V_1 x S - V_1$$

$$A = V_1 (S - 1)$$

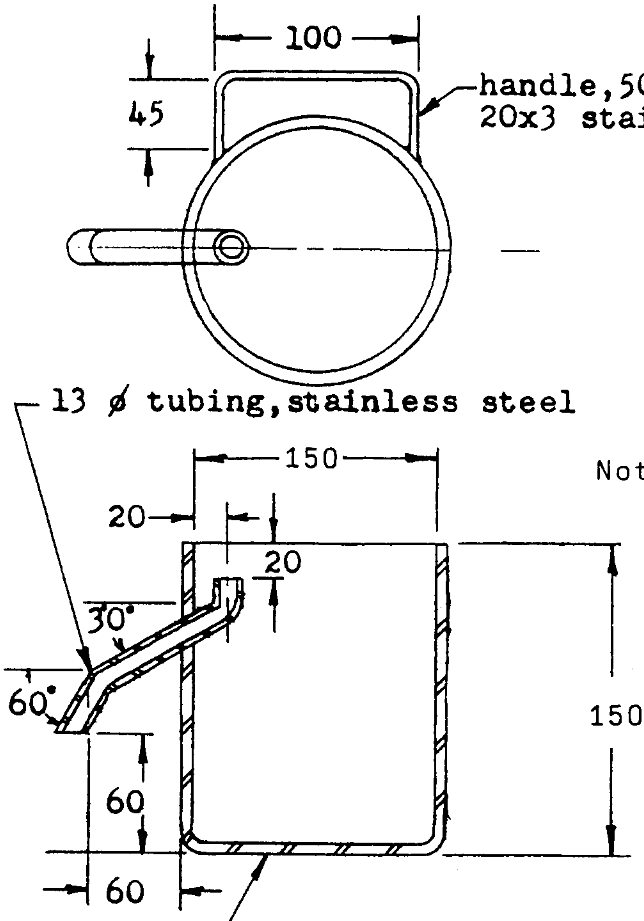
but since $V_1 = \frac{W}{S}$ $\quad A = \frac{W}{S} (S - 1)$

finally $\quad A = W \frac{S - 1}{S}$

In the laboratory work,the special beaker used for weighing of wet materials under water is called pycnometer. The actual laboratory set-up looks like this



PYCNOMETER



handle,50 from top
20x3 stainless steel

13 ø tubing,stainless steel

Note: all dimensions are in millimeters

316 type stainless steel
seamless drawn,satin polished,
commercial type standard size
beaker or pot

all welds with 316 stainless rod containing Columbium or Molybdenum to avoid intergranular corrosion;gas welding preferred,grind and polish all weld beads to eliminate porous surface

Table of Archimedes Differential Weight "A" for some of the primary explosives used in priming mixes

MATERIAL	APPARENT Spec. Density	VALUE OF "A" FOR 1 gram OF MATERIAL
Dinol	1.63	0.39
Lead Styphnate	3.03	0.67
Mercury Fulminate	1.75	0.42
Tetracene	1.63	0.39

Tetracene density is 1.63 thus accurate weighing is required to keep the percent error low. Furthermore, the percentage of tetracene in the primer mixture is small which further complicates the task in large volume operation.

Tetracene is made into a slurry in a separate tank and measured volumetrically into the wet premix. When volume of production is relatively small it is more advantageous to measure tetracene by the pycnometer method.

SAFETY PRECAUTIONS: Pycnometer weighing

1 - Eye protection is worn while working with lead styphnate and tetracene.
2 - Rubber gloves are worn while working with lead styphnate.
3 - lead styphnate and tetracene are sensitive primary explosives and must be handled carefully.
4 - Spilled explosives are wiped up immediately before they dry up.
5 - Floors are kept wet and are washed with hose after each day's work.
6 - Do not allow formation of scale or adherent layer of lead styphnate on pycnometers or other equipment. This scale is extremely sensitive and can explode even when wet. Inspect and clean all pycnometers with hot NaOH.
7 - When lead styphnate scale starts forming on rubber storage buckets clean them with ammonium acetate solution. Stronger reagent would damage the bucket material. Wash and rinse the buckets thoroughly with water after ammonium acetate cleaning.
8 - Do not use any scoops, spatulas or spoons to stir or transfer lead styphnate or tetracene. Use only a stream of water or gloved hand.
9 - Lead styphnate which has settled and formed a solid lump at the bottom of container may be loosened with stream of water only. Do not loosen with gloved hand.
10 - Never leave lead styphnate in pycnometer overnight. The lead styphnate would settle into a firm mass difficult to move and also starts formation of a sensitive scale in the pycnometer walls.
11- Catch basin inspection and scrapping procedures are detailed in chapter on lead styphnate preparation; follow these instructions.

Note:

- Do not weigh lead styphnate by placing the pycnometer first into sink allowing it to drain and then check the weight on the balance.

- When transferring the lead styphnate from the pycnometer to the rubber liner the inverted pycnometer bottom may be hit by the heel of the hand to facilitate the lead styphnate dumping.

Pycnometer weighing

1 - Storage container with tested good product is brought from the magazine.

2 - Swirl explosive into suspension by rotating the container.
3 - Pour suspended explosive into the pycnometer.
4 - Material adhering to the container sides is washed down with small stream of water.
5 - Place the weight of tare of pycnometer plus the Archimedes difference weight on the scales.
6 - Pycnometer with explosive is placed on the opposite pan on scales so that pycnometer overflow can run into the storage container.
7 - The pycnometer stands in the same position on the balance pan during each weighing to reduce error in the pan uneveness. Balance is tared and used on the same spot on the table for the same reason.
8 - Remove overflow stopper and allow pycnometer to drain until water stops running and starts to drip.
9 - At this point the scales should be balanced.
10 - If the pycnometer and contents weigh more, let water run into the pycnometer so as to displace and carry into overflow some explosive until the balance pointer indicates zero.
11 - If the pycnometer and contents weigh less, allow explosive in pycnometer to settle and decant clear water, then refill with slurry.
12 - Correct weight of explosive in pycnometer is transferred immediately to the rubber container.
13 - Rotate pyonometer to bring all explosives into suspension.
14 - Pour suspension into container.
15 - Wash the remainder of explosive from the pycnometer into the container with small stream of water.
16 - Wipe off any explosive splattered on the rim of the rubber container with wet sponge.
17 - Allow explosive in container to settle and decant clear water, leaving about 1 cm of water above.

Vibration to remove excess water from wet explosive pre-mix

In the wet premix preparation first the tetracene is measured (weighed) and placed in the rubber liner ;then the lead styphnate is measured (weighed) in the pycnometer and placed in the rubber liner over the tetracene.

The wet premix weight includes the necessary moisture to make the final primer mix homogenous. Generally, the wetter the mix the more homogenous it is.
However, the wet mix will also depend on the mixing equipment, and the final amount of water may be adjusted to yield the optimum mix for the conditions.

Vibration to remove excess water.

1 - Place liner into corresponding recess in vibrator.
2 - Switch vibrator motor on, the vibration speeds the settling of the lead styphnate and tetracene.
3 - With the liner contents settled, switch vibrator off.
4 - Carefully decant the clear water from the liner, leaving about one (1) centimeter of water above the mix.
5 - Immediately wipe off with wet sponge the solid mix adhering to the liner lip.
6 - Place the liner on balance and check weight, if more water is to be removed repeat steps 1 through 5.
7 - Liner with correct wet premix weight is placed into the corresponding rubber storage pot.
8 - Check that no solid materials are exposed on the liner lip and sides and that the liner contents is completely covered with water.
9 - Cover the storage pot with the cover.
10 - Hand carry the storage pot with liner to the storage magazine and never carry more than two (2) pots at a time (one in each hand).

Chapter 6

Mercury Fulminate Priming Mixes

General:

Although mercury fulminate is prepared and stored wet, it is used in dry priming mixes. It is the oldest practical priming and detonator initiating material. While used first in a priming mix at the beginning of the 18th century, it was still in service during WW II. In spite of such shortcomings as short storage life, (particularly at elevated temperatures), its sensitivity to percussion, powerful ignition, availability of raw materials and ease of manufacture were factors making it popular.

Mercury Fulminate priming mix was used by Western Cartridge Co. (later Winchester-Western) as late as 1947. The mix composition was essentially the same as the Italian Army mix.

The manufacturing method falls into 4 basic groups:

1 – Cold solution of mercury in nitric acid.
2 – Warm solution of mercury in nitric acid.
3 – Use of bleaching materials in addition to the basic raw materials.
4 – Production of pure fulminate by re-precipitation from ammonium hydroxide solution.

The present process has been selected on the basis of simplicity, safety of operation and good quality of product.

During the process evaluation the methods described in the following sources were compared:

Technology of Explosives, Vol. 111, Urbanski, T.
Chemistry of Powders and Explosives, Davis,
German text on chem. technology, 1902
MIL-TM9-1910
Private notes on fulminate manufacture

1 - Equipment - Laboratory Preparation of Mercury Fulminate

1 – Stainless steel sink with cold tap water source.
2 – Hood with exhaust system.
3 – Fume exhaust hose with suction fan.
4 – Balance, capacity 250 gram, accuracy ± 0.1%.
5 – Erlenmeyer flask, capacity 1000 ml.
6 – Beaker, glass, capacity 250 ml.
7 – Balloon, glass, capacity 1500 ml.
8 – Stand, for balloon, allowing balloon tilting.
9 – Pail, stainless steel, capacity 12 liter.
10 – Buchner filter.
11 – Stand for Buchner filter.
12 – Screens, No. 100 and No. 200 mesh.
13 – Container, storage, with cover, material: conductive rubber.
14 –Soft conductive rubber liner to fit inside container 13.

2 - Equipment - Priming mix preparation

1 – Jelly bean bag with remote activation.
2 – Container, storage, with cover, material: conductive rubber.
3 – Screen, drying 14 MF.
4 – Scale, capacity 250 gram, accuracy ± 0.1% 1.
5 – Container, storage, with cover, material: conductive rubber .
6 – Pycnometer.

SAFETY PRECAUTIONS

1 - Eye protection is worn while working with primary explosives.
2 - Rubber gloves are worn while working with primary explosive and must be handled carefully.
3 - Spilled explosives are wiped up immediately before they dry up.
4 - Floors are kept wet and are washed with hose after each day's work.
5 - Do not allow formation of adherent layer of mercury fulminate on pycnometers or other equipment. This scale is extremely sensitive and can explode even when wet. Inspect and clean all pycnometers with hot water.
6 - When mercury fulminate starts forming on rubber storage buckets clean them with hydrochloric acid solution. Wash and rinse the buckets thoroughly with water after acid cleaning.
7 - Do not use any scoops, spatulas or spoons to stir or transfer mercury fulminate. Use only a stream of water or gloved hand.
8 - Mercury fulminate which has settled and formed a solid lump at the bottom of container may be loosened with stream of water only. Do not loosen with gloved hand.
9 - Never leave mercury fulminate in pycnometer overnight. It would settle into a firm mass difficult to move and also starts formation of a sensitive layer on the pycnometer walls
10 - Catch basin inspection and scrapping procedures are detailed in section on mercury fulminate manufacture; follow these instructions.
11 - Dry mercury fulminate is extremely sensitive to electric spark ignition, hence all equipment must be grounded and anti-static clothing and shoes worn by the operators.

Note:

- Do not weigh mercury fulminate by placing the pycnometer first into sink allowing it to drain and then check the weight on the balance.
- When transferring the mercury fuliminate from the pycnometer to the rubber liner the inverted pycnometer bottom may be hit by the heel of the hand to facilitate the dumping.

SAFETY PRECAUTIONS:

1 - **Mercury, metallic.**
Poisonous vapors readily absorbed by respiratory system, intact skin, and gastrointestinal tract. Spilled and heated elemental mercury is particularly harmful. Spills must be cleaned immediately, if not possible, wet area with water or sprinkle calcium sulfide onto affected surface. Small drops of mercury can be picked up using a small steel pipe or glass pipette connected to a suction flask by small bore rubber tubing.
Mercury or its compound are only heated under a hood.
Avoid breathing fumes.
Wash thoroughly after handling.

2 - **Nitric acid.**
Highly toxic when inhaled or ingested.
Causes severe burns on contact with skin; in case of contact wash copiously with water for at least 15 minutes.
In case of contact with eyes, wash with plenty of water, 15 minutes minimum and get medical attention.

3 - **Hydrochloric acid.**
Concentrated acid is highly corrosive and causes burns. Aqueous solutions are corrosive, irritating and poisonous. Fumes are corrosive and irritating to mucous membranes.

4 - **Ethanol.**
Heavy fume concentrations cause headache, irritation of eyes, nose and throat. Dangerous fire hazard, can be extinguished by water or CO_2 extinguishers (vaporing liquid or dry powder).

WASTE TREATMENT:

Waste substances from mercury fulminate manufacture are:

1 - Spent liquor decanted from above the product (step 14).

2 - Sediment (slime) removed from fulminate by washing (step 17-24).

3 - Condensed fumes.

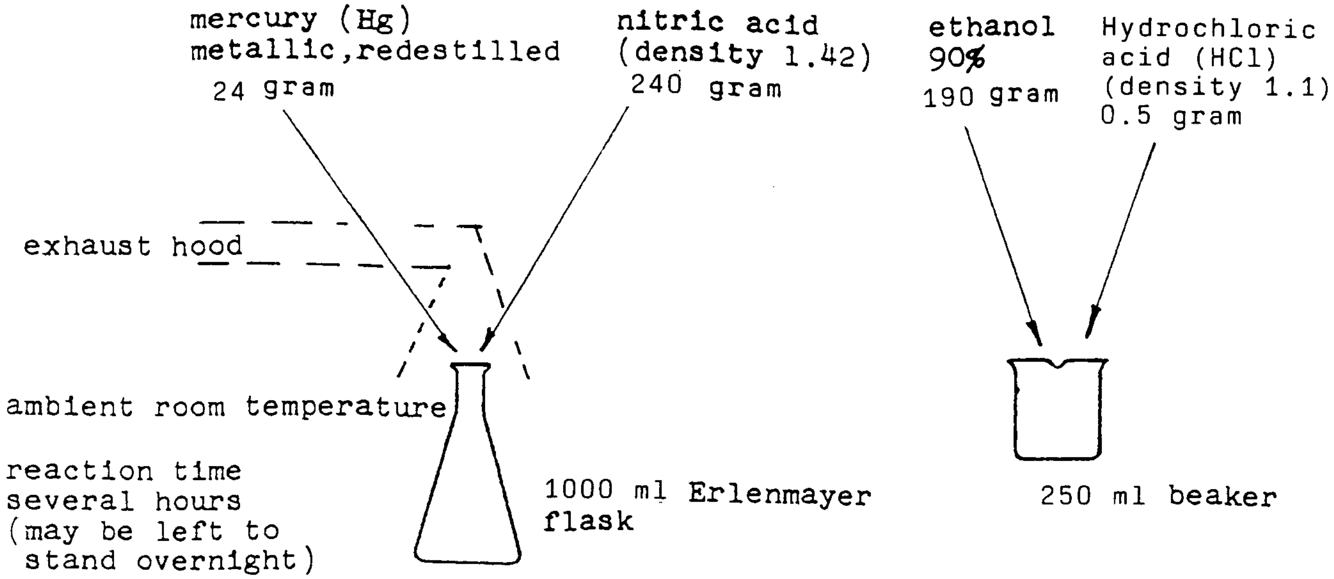
Each step is treated separately as follows:

1 - Spent liquor contains 3% dissolved substances consisting of:
oxalic acid 90-96%
mercurous nitrate 3-6%

- Addition of 100 cc HCl to 5 liters of liquor will precipitate the mercury compound.
- Sediment is separated from the rest of the liquor by decanting.
- Remaining sediment is dissolved in equal weight of concentrated HCl.
- 100 gram metallic Sn dissolved in 600 gram concentrated HCl is added to thin solution causing precipitation of pure mercurous chloride.
- Recovery of mercury in metallic form is done by:
- Neutralization of liquor with milk of lime (causing precipitation).
- Separation of precipitate from the rest of the liquor by decanting.
- Addition of HCl to dissolve the precipitate.
- Addition of Zn to displace the mercury (or by electrolysis of the above solution).

2 - Slime is treated in the same manner as liquor , i.e.
- Dissolve slime in HCl.
- Recover metallic Hg by displacement with Zn.

3 - Condensed fumes contain:
Alcohol ($CH_3.CH_2OH$)
Acetaldehyde ($CH_3.CHO$)
Ethyl nitrate ($C_2H_5\ ONO_2$)
Ethyl nitrite ($C_2H_5\ ONO$)
Nitro ethane ($C_2H_5NO_2$)
- Distillation of the condensate over NaOH allows recovery of the alcohol which is recycled (lime may be used instead of NaOH for the same purpose).



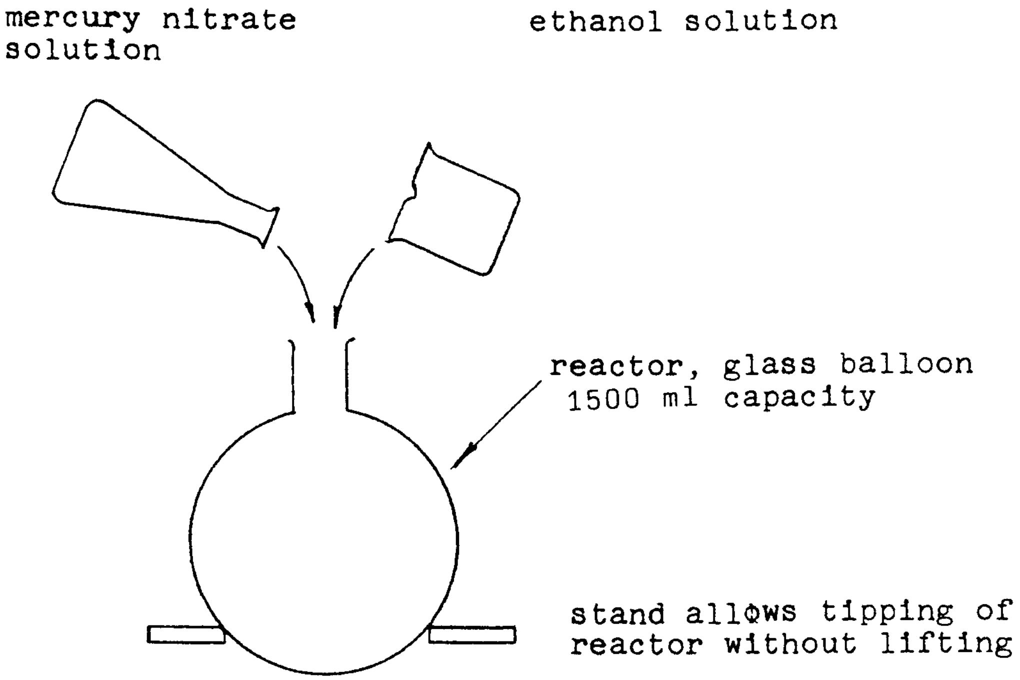
1 - Place tared Erlenmeyer flask on balance.
2 - Add specified quantity of mercury.
3 - Add specified quantity of nitric acid.
4 - Remove flask from balance and place under hood; do not stir or shake unnecessarily.
5 - Place tared beaker on balance.
6 - Add specified quantity of ethanol.
7 - Add specified quantity of hydrochloric acid.

8 - Note: When nitric acid is scarce, the specified quantity of acid per gram mercurys reduced to half and the solution from steps 3 & 4 is left overnight.
In the morning the solution is decanted and new acid added to dissolve the remaining mercury. The combined solution is then added to the ethanol in the reactor.

However, use of water diluted reagents and acids produces dark grey fulminate containing excessive amount of metallic mercury. Similar results are obtained by insufficient use of nitric acid and/or low reaction temperature. Such fulminates are unstable in storage and unusable for commercial and military purposes.

Yield: the mercury fulminate yield is about 90% theoretical:
24 gram Hg yields 0.9 x 34 gram = 30 grams mercury fulimate.

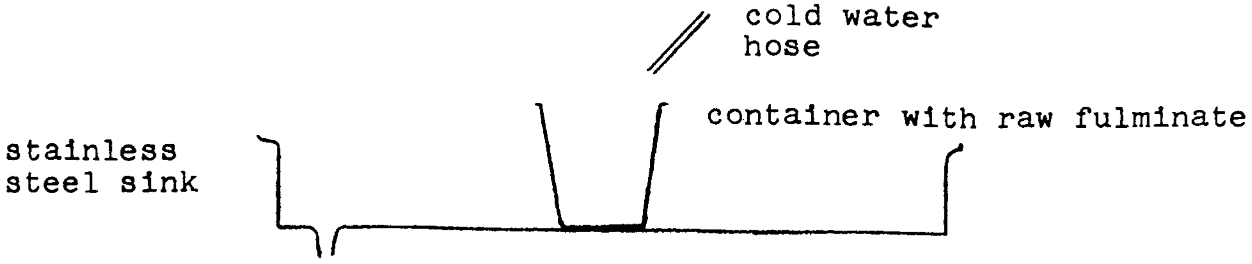
LABORATORY PREPARATION OF MERCURY FULMINATE



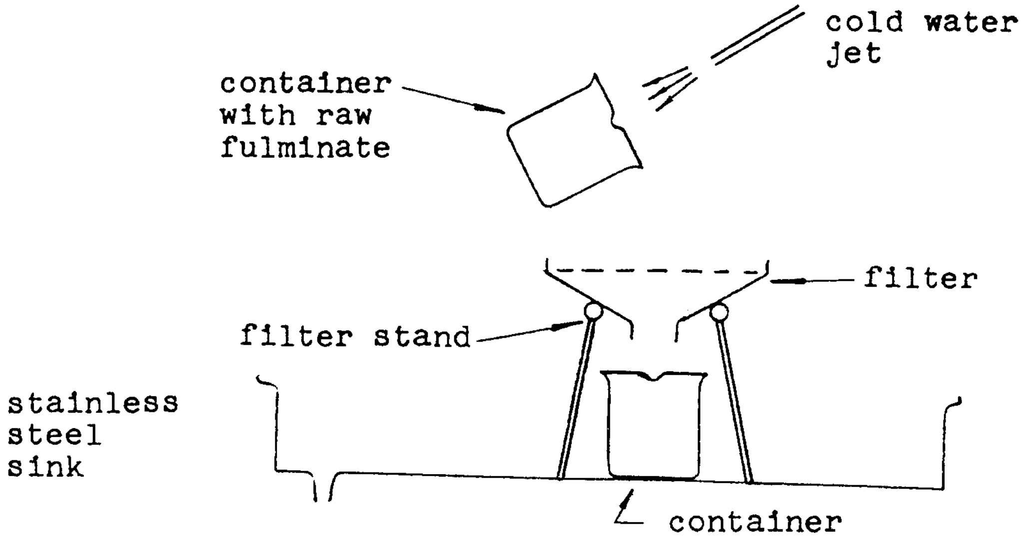
9 - Pour beaker contents into the reactor.
10 - Pour Erlenmeyer flask contents from step 4 into the reactor.
11 - Place the fume exhaust nozzle over the reactor opening; the reaction starts in about 15 minutes with evolution of gas, then boiling and filling the reactor with fumes. The fumes are first white then become brownish red as the reaction heat drives off nitric acid, then white again.
The violence of the reaction may be suppressed by addition of cold ethanol, however, excessive addition will inhibit the reaction and may lead to formation of free mercury contaminating the final product. Mercury fulminate precipitates in the form of greyish yellow crystals of good commercial quality.
12 - After reaction completion leave the reactor standing 30 minutes to allow cooling of the contents to ambient temperature.
13 - Remove the fume exhaust nozzle.
14 - Slowly tilt the reactor to decant the mother liquor into a glass container.
15 - Remove the container with liquor to the reprocessing area.
16 - Place another glass container under the reactor opening.
17 - With a small stream of water wash the precipitated fulminate into the container.
18 - Fill the empty reactor full of tap water immediately, washing the fulminate traces from the opening area.

MERCURY FULMINATE

WASHING:

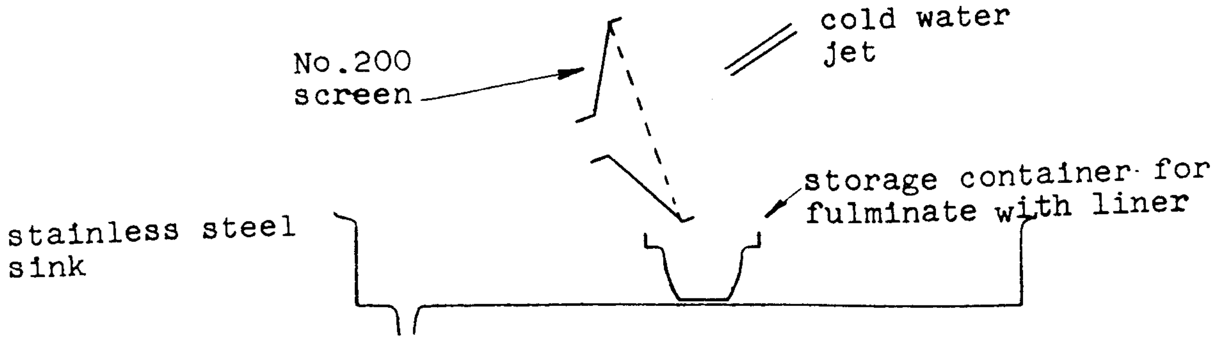


19 - Transfer the container with wet fulminate to the wash sink.
20 - Wash the raw fulminate eight (8) times following the procedure for washing explosives.
21 - Check the last wash water with litmus paper- the reaction should be neutral, if otherwise, continue washing until the fulminate is free of acid.



22 - Container with washed fulminate is placed over a No. 100 mesh screen located over an empty container.
23 - The fulminate is washed onto and through the screen with a stream of demineralized water.
24 - Large crystals retained on the sieve are transferred with a stream of demineralized water to a porcelain bowl and covered with 2-3 cm of excess water in a separate operation under industrial conditions these large crystals are carefully ground under water and then added to the next batch to be screened for size.
25 - Fulminate which passed through the No. 100 screen is next washed onto a screen No. 200 in the same way as described in steps 22 and 23.
26 - The fulminate retained on screen No. 200 is gently washed over allowing small crystals to pass through the sieve.
27 - the container with the small crystals is transferred to scrapping area where the fulminate is scrapped and the metallic mercury recovered for reuse.

MERCURY FULMINATE



28 – Fulminate retained on the screen is transferred with a stream of cold demineralized water to a storage container; contents of several reactors may be stored in one container.
29 – Decant the water used in the transfer process leaving about 5 cm of water over the settled solids in the storage container.
30 – Any solid material adhering to the container side is washed down with a little water.
31 – Container is covered, dated and coded.
32 – Covered container is hand carried to an underground magazine with humid atmosphere and at temperature above, freezing but below 15° C. If there is danger of freezing, store the fulminate under a 50/50 mix of water and alcohol (methanol or ethanol).
33 - As the wet fulminate is being transferred from container to container, fill the used containers with tap water immediately after transfer.
34 – The resulting mercury fulminate is of good military quality (98% pure) and crystal size to obtain fulminate of improved stability and extra pure (99.75%) follow steps below.
35 – To the container with fulminate from step 21 add strong ammonium hydroxide solution to dissolve all fulminate.
36 – Filter the solution through a cloth screen into a clean container.
37 – Cool filtrate to ambient temperature.
38 – Add a solution of 30% acetic acid; the fulminate precipitates.
39 – Repeat steps 20 through 33 above.

Note: During washing and screening of fulminate keep the reaction and water temperature as low as possible to prevent fulminate hydrolization.
Free metallic mercury reacts with copper or copper alloys to form copper fulminate which leads to further reductions; hence use only stainless steel containers, tools, etc.

DRYING MURCURY FULMINATE

1 – Covered rubber container with wet MF is brought from storage.

2 – A clean drying screen is positioned on the work table.

3 – Water collected over the NP is carefully decanted into the collection pail.

4 – Flexing the liner bottom loosens the wet NP.

5 – Tipping the liner deposit NP onto the screen.

6 – With gloved hand gently spread the wet MF more or less evenly over the screen.

7 – Fill the liner with water.

8 – Place the screen into the dryer.

9 – Dry the MP at 30 - 35 ° C for approximately 6 hours.

10 – Shut off the dryer and allow to cool to ambient (or 20° C, before removing dry NP.
Note: the sensitivity of MF increases with higher temperature.

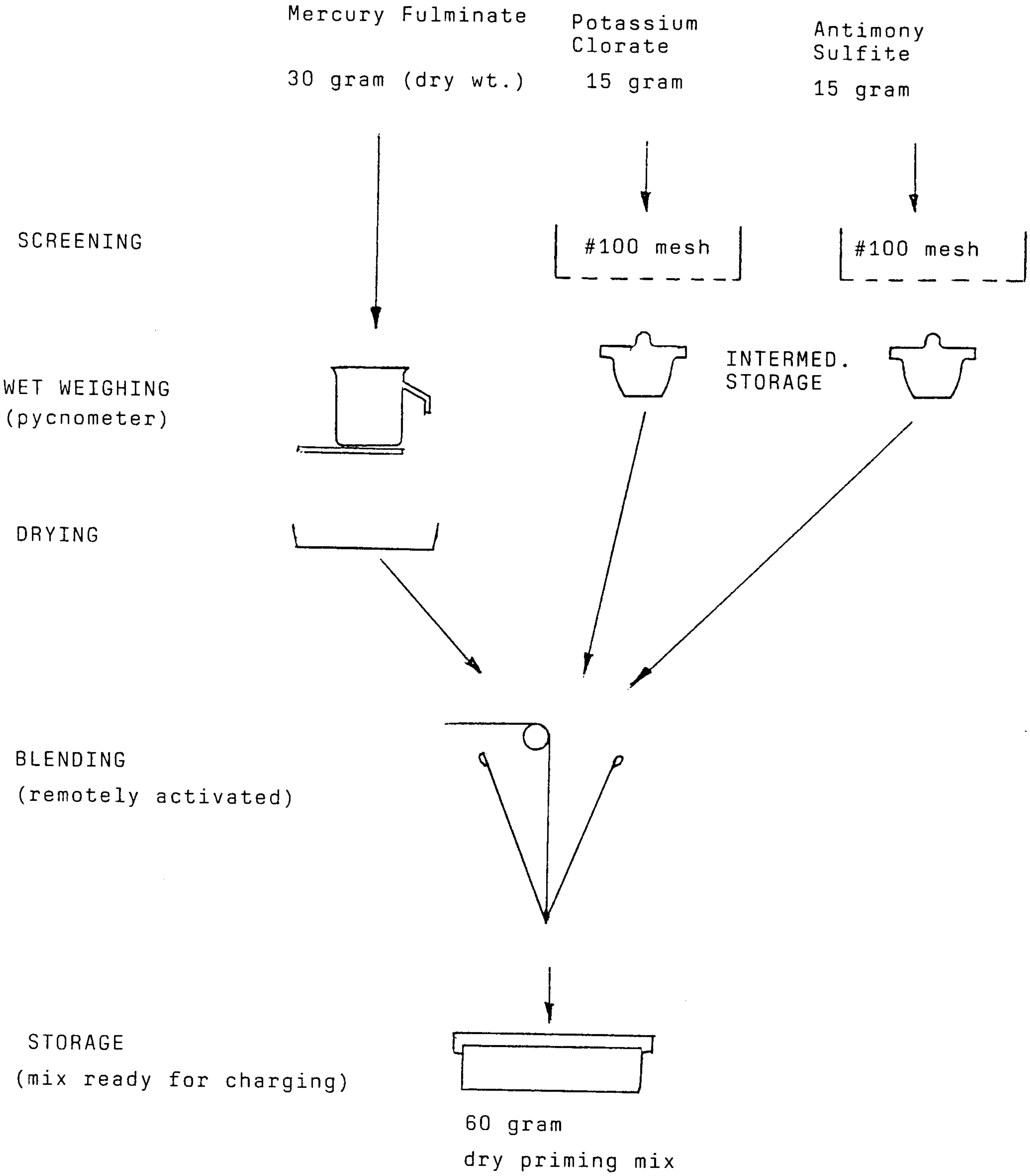
11 – Remove screen with cool, dry, MF and place into container.

12 – Cover container and transfer to mixing room immediately.

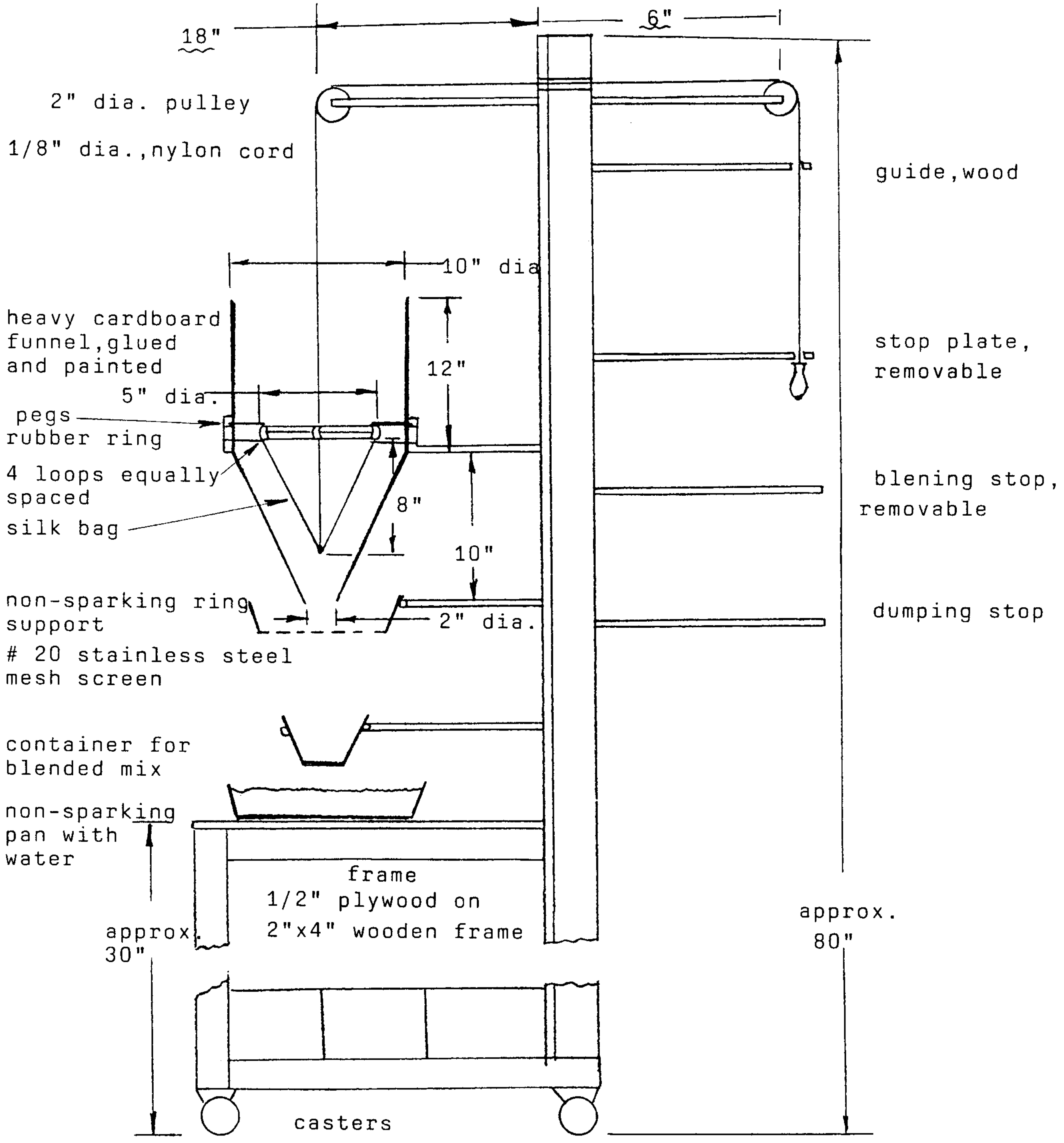
Note: Used, empty screens are tapped in upside-down position over a waste treatment container with water.

At different locations the fulminate was dried by different methods.
a – In vacuum dryer at 35 - 45° C.
b – Natural air drought drier at 45 - 50° C for 8 hrs.
c – Forced air drier at 65 - 70° C for 90 minutes.
Regardless of the type dryer, the wet fulminate is spread in silk screens prior to placement onto the dryer itself.

BLENDING OF DRY MERCURY FULMINATE PRIMING MIX



JELLY BEAN BAG UNIT FOR BLENDING DRY PRIMING MIX



NOTES: 1) the silk bag ring is held in place by six (6) wooden pegs glued into the funnel support ring (3 above and 3 below the bag ring)

2) the complete unit is wheeled outdoors,positioned under a lightly roofed, open end shed.The jelly bean bag is facing towards open end and safe open area.

MANUFACTURING PROCEDURE - Final Mixing MF Priming Composition

1 - Check the jelly bean bag operation:
  a - Bag to be free of foreign matter.
  b - Operating properly when pulling cord is actuated.
  c - Bag inverting properly over the final mix container on support ring.
  d - Scrap pan is full of water.

2 - Containers with the correct quantity of components are placed on the shelf next to the bag.

3 - Place the components into the jelly bean bag in the following order:
  a - Potassium chlorate
  b - Antimony sulfide
  c - Mercury fulminate
4 - Place empty containers into the pail full of water.
5 - Carry the pail out of the mixing area - set the "operation in progress" flag up.
6 - Operate the bag pulling cord 3 minutes.
7 - Dump the bag contents onto the screen.
8 - Return to the mixing area.
9 - Visually inspect the mix, cover the container and transfer the closed container to the primer charging area.

Notes - Mercury Fulminate priming mix

1 - The mercury fulminate priming mix composition may also be mixed in wet, but has the following disadvantages:
  a - Thorough mixing is difficult without a suitable binder.
  b - Addition of binder increases the hygroscopicity of the composition (1% of moisture will cause misfires).
  c - Wet mix in the past was blended on a glass plate (at FA) manually, putting a large quantity of composition in contact with the operator.
  d - The wet mix must be screened through two sets of screens to break up the granules.
  e - The wet material must be finally dried anyhow.
  f - The overall wet mixing process is more complex and offers more personnel exposure than dry mix operation.

.2 The MP priming mix charging into cups is done by volume in a manner similar to the wet LS-T priming mix charging, i.e. priming composition is measured volumetrically into holes in a charging plate, made from stainless steel or bronze, and then transferred into cups kept in a plate with equal number of holes. However, because of the dry composition, the metering, transfer, foiling, anvil seating, etc. is done on hydraulic presses behind barricades.

Chapter 7

Potassium Chlorate Priming Mix

This chapter describes the preparation of the K-chlorate mix, Known as the FA #70. This priming mix replaced (in the U. S. service) the mercury fulminate mixes during WW I and remained in use throughout WW II. The main reason why this priming mix has been replaced by LS-T mixes in 1948, is not so much the performance, as the corrosive effect of K-chlorate mixes on the barrel bore. However, K-chlorate primers are more stable at elevated temperatures, which is one reason why they are still used in special applications.

The manufacturing process of K-chlorate mixes is no more complex than that of LS-T mix, but the same degree of care and respect for the sensitivity and power of the product is required.

This chapter also describes the preparation of Lead Sulphocyanate. This material is also available commercially and since the preparation is very toxic, and the quantities are relatively small it may be preferable to purchase this priming mix component.

The procedures have been calculated so that the equipment used for LS-T operations may be also used to make the FA7O mix.

Equipment:

The same equipment used to make Lead Styphnate - Tetracene priming mix may be used to make FA #70 Potassium Chlorate wet mix.

Materials	**Percent Dry weight**
Potassium Chlorate	53 ± 5
Trinitrotoluene	5 ± 1
Lead Sulphocyanate	25 ± 3
Antimony Sulfide	17 ± 3

SAFETY PRECAUTIONS:

Lead thiocyanate is relatively safe to make from explosive point of view; but its preparation and the product are extremely TOXIC.

Utmost protection of personnel from direct contact by use of masks, goggles, gloves, work clothes, etc. is required.

To keep dust and fumes from personnel contact, the work area is equipped with a powerful and positive exhaust system with filters and traps to keep the outside area from contamination.

All precautions applicable to LS-T wet priming mix apply, except as noted.

The specification calls for destruction of the final mix if not used within six days after preparation. This is due to the presence of moisture which may change the crystal structure of the components and hence their sensitivity. Since the final mixing is normally done daily from the pre-screened, preweighted components, the time limit is only a safety feature.

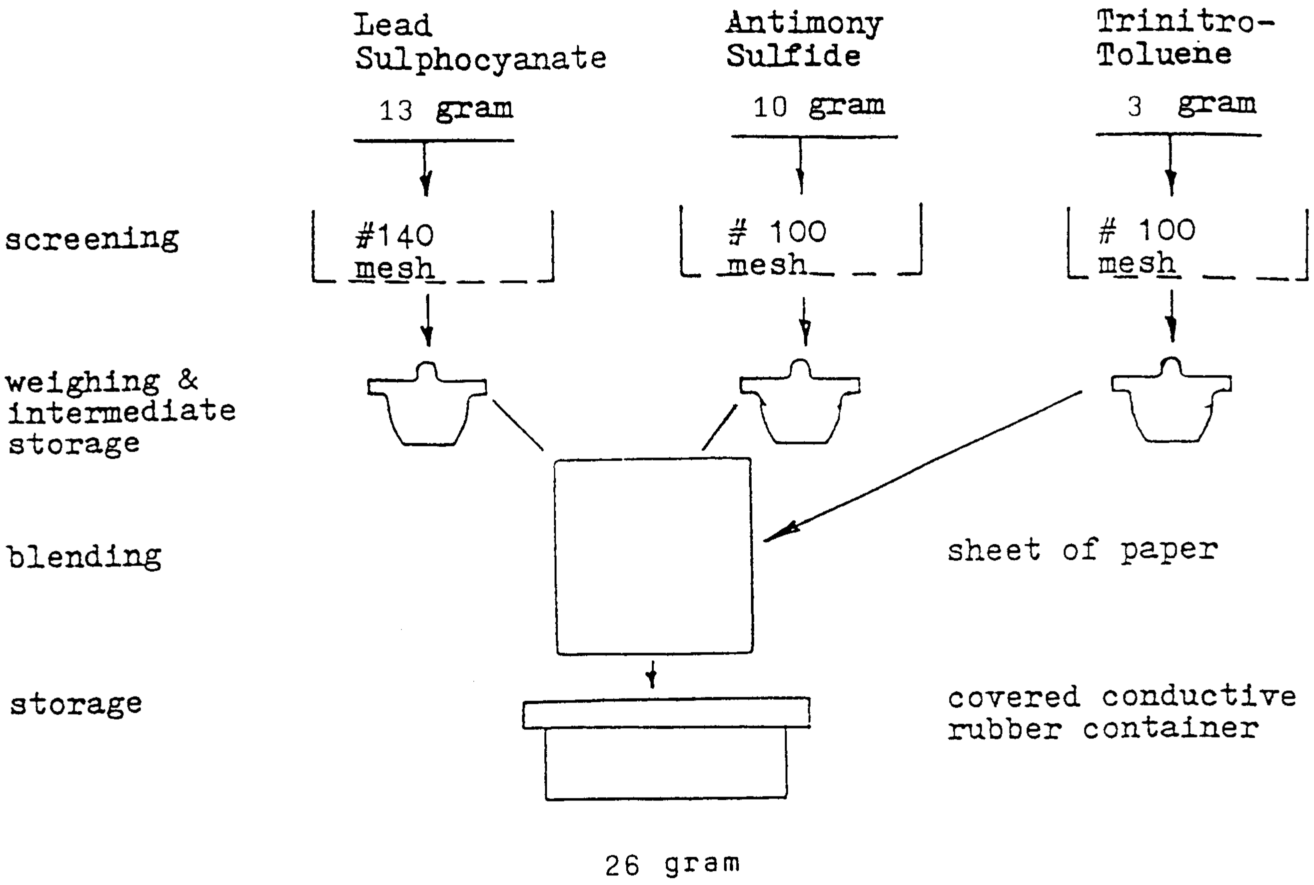
No burning test is required.

Procedure notes:

The proper crystal size of the components is essential for the specified mix sensitivity and performance. The inspection and testing of the priming mix is limited to the chemical and physical tests of the ingredient prior to mixing.

Finished primers are subject to the same tests as the LS-T mix charged primers. The chlorate priming mixes will cause barrel corrosion even if left overnight.

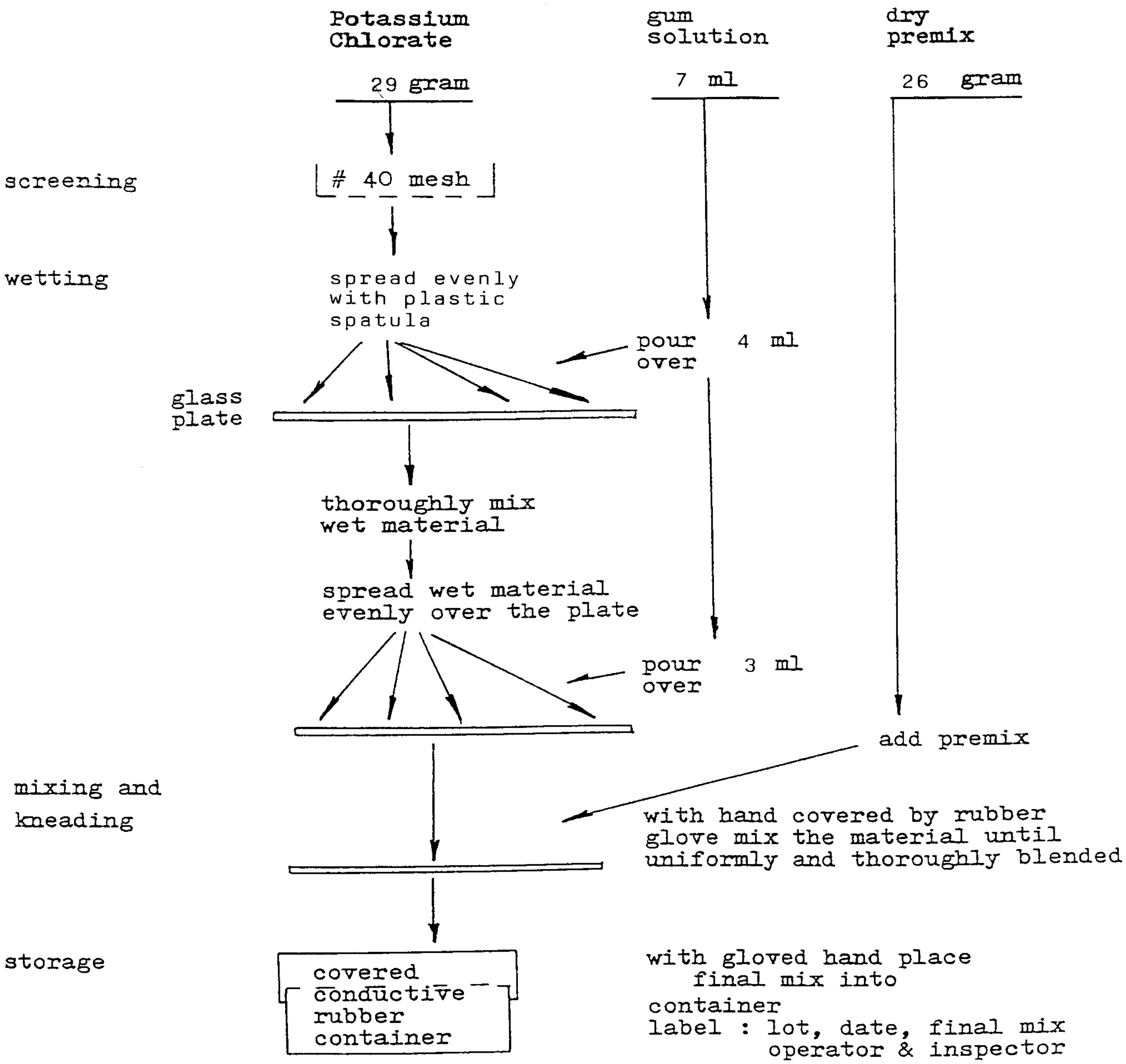
LABORATORY PREPARATION OF FA #70 PRIMING MIX
Dry Premix



Note: In the laboratory preparation the TNT may be replaced by fast burning double base ball powder, as used in rimfire and small pistol cartridges.

The FA #70 dry premix preparation is identical to the LS-T dry premix even though TNT is present. Although TNT is a high explosive, it requires powerful initiator and containment for initiation; hence it is relatively safe in the present situation.

LABORATORY PREPARATION OF FA#70 PRIMING MIX



Note: Final mix is stored at temperature between 4° C and 10° C until needed, but not over 6 days; afterwards the composition is destroyed. Final mix is inspected visually for uniform blending.

The FA #70 mix and its preparation are given in the MIL-F-18601.

Chapter 8

Preparation of Tetracene

Of all priming mix explosive components, Tetracene is probably the simplest to prepare, both in equipment and the process. *Tetracene may be made from aminoguadinine sulfate (generally preferred in Europe), or from aminoguadinine carbonate (preferred in the United States). Both methods are similar and have individual merits, but the detailed discussion is not intended in the scope of this chapter. The method described below has been chosen as the optimum due to its simplicity and good product yield.

Using a 40 liter capacity stainless steel pot (reactor) instead of a glass beaker, with a similar increase of other containers and components, 1,000 grams of Tetracene per batch is easily achieved. The cooling-heating cycle remains essentially the same. Sometimes dextrin is added to the reacting solution to control the crystal structure.

*Tetracene is formed by reaction of aminoguanidine bicarbonate in acetate solution and sodium nitrite.

Safety precautions - tetracene

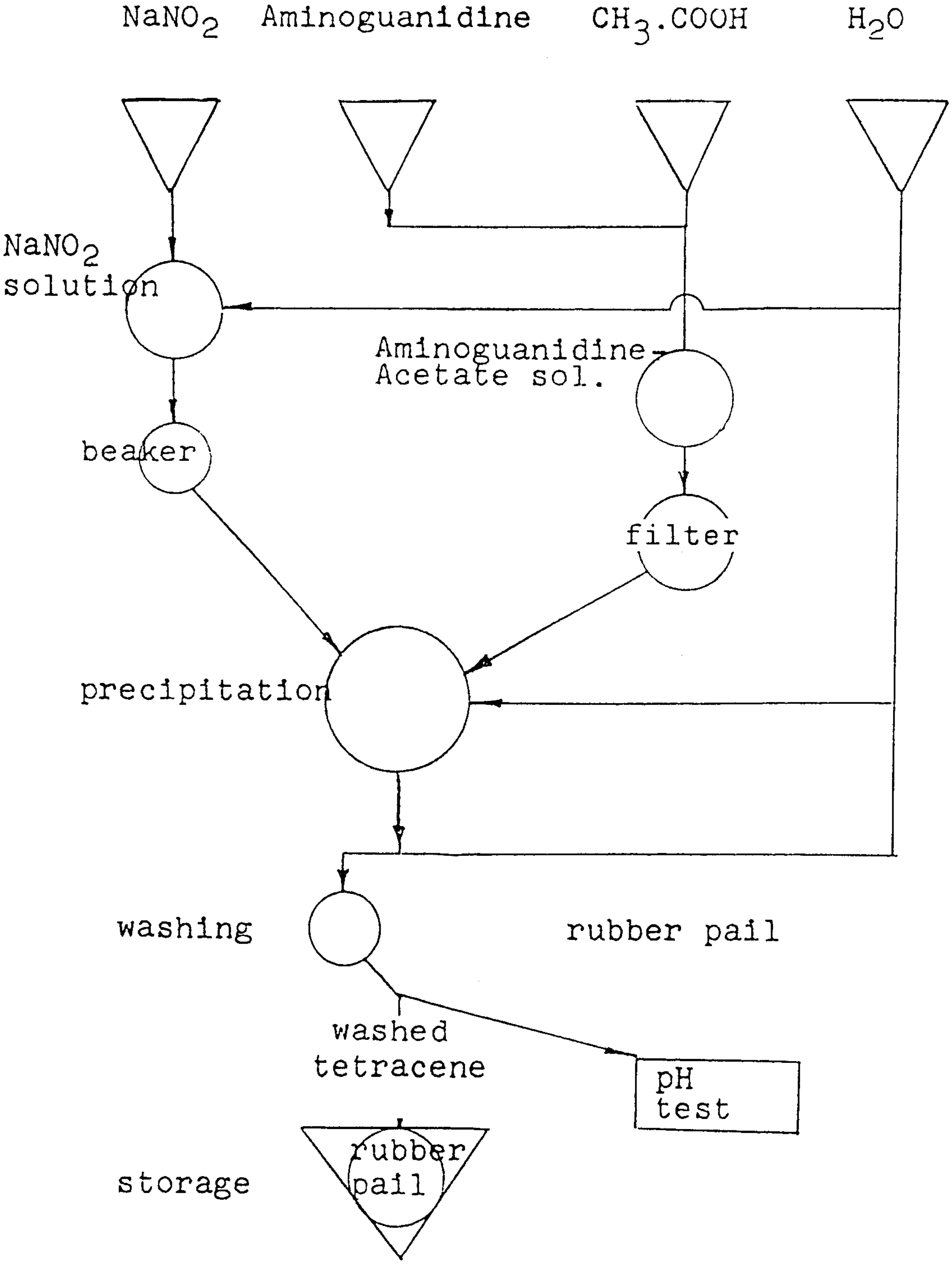
1 - Eye protection is worn at all times.
2 - Spilled tetracene is cleaned up immediately before It dries up.
3 - Nose and mouth mask is worn when working with solid $NaNO_2$. Taken internally $NaNO_2$ is toxic and affects heart functioning.
4 - Body contact with materials in Tetracene preparation is to be avoided.
5 - Working area is kept clean, water and other liquids are mopped up when spilled. Spilled $NaNO_2$ is swept up.
6 - Reaction beaker is washed out with 10% NaCH solution at least once a week. This prevent and/or reduces scale formation as well as keeps wet any scale formed.
7 - Reaction beaker is washed with water afterwards to keep them free of alkali.
8 - Sink in which reaction beaker stands is washed with 10% NaOH solution at least once a week. Remove reaction beaker from sink during this wash.

After washing sink with water to remove the alkali, leave a small stream of water to run into the sink constantly to keep it wet at all times.

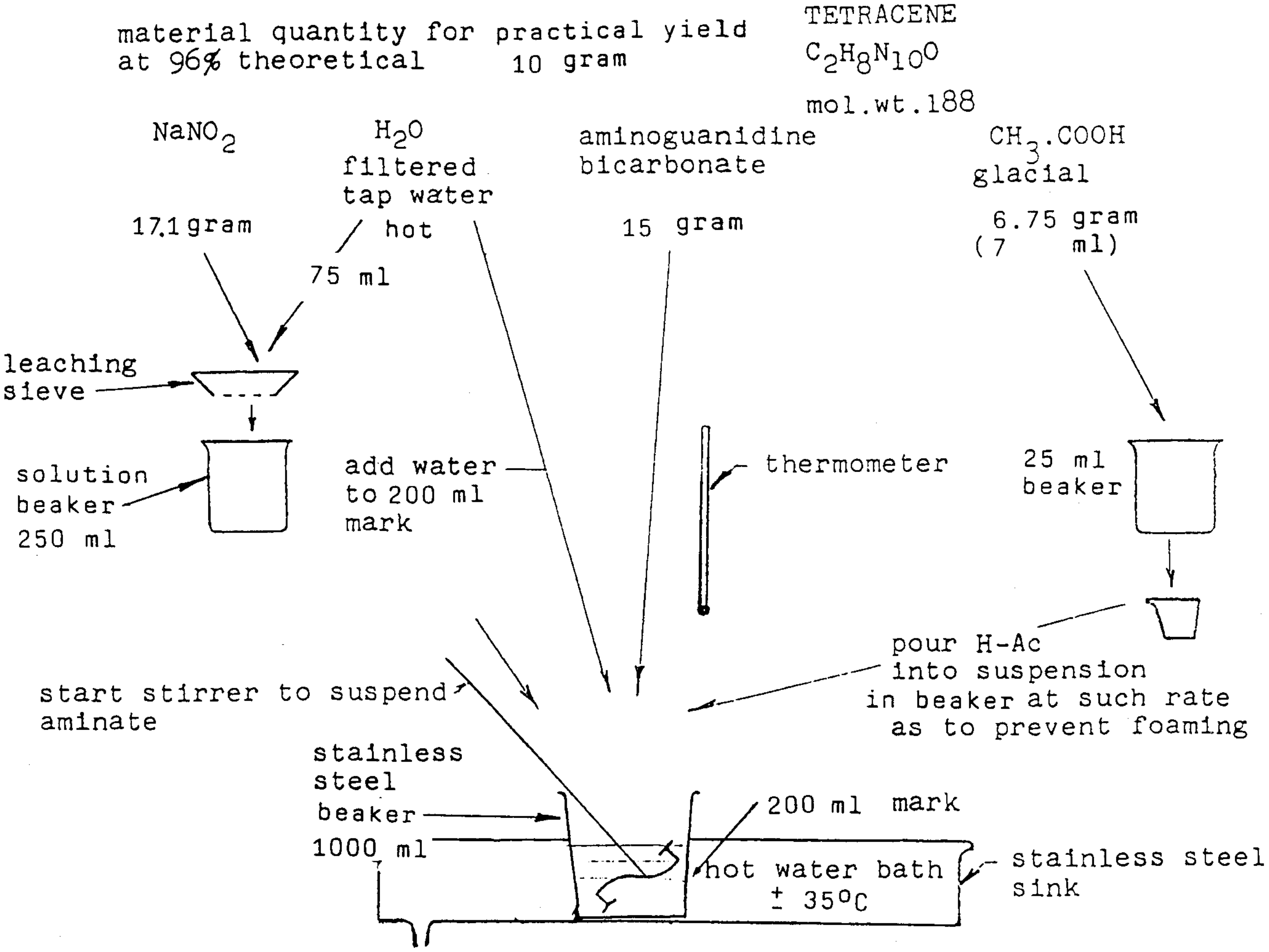
9 - Sink where conductive rubber containers are washed is treated in the same way sink above.

10 - Scrapping Tetracene in the catch basin is done by following the lead styphnate scrapping procedure except that only NaOH is required. Tetracene decomposes in hot alkaline solution into ammonia and other soluble products which are washed down the drain.

PROCESS DIAGRAM FOR PREPARATION OF TETRACENE



PROCESS DIAGRAM FOR PREPARATION OF TETRACENE



1 - Place $NaNO_2$ on leaching sieve and add water to fully dissolve material.
2 - Filter the solution before pouring into beaker since any foreign matter in the solution would end in the priming mix.
3 - Place aminate in beaker add water to 200 ml mark and start stirrer since aminate is insoluble in water; put hot water into sink to heat beaker contents to 30 ± 1° C.
4 - Add H-Acetic into beaker at such rate as to prevent foaming over; the completion of aminate solution is indicated by the clearing of the solution and the end of CO_2 evolution.
5 - Add water to beaker to the 200 ml mark.
6 - Add $NaNO_2$ solution.
7 - Add water to the 300 ml mark.
8 - Maintain temperature of the beaker contents at 30 ± 1° C for 22 - 24 hrs.
9 -- Wash tetracene as per standard washing procedure 8 -10 times.

Note: Presence of Cu ions prevents tetracene formation so that 0.08% Cu in the aminate reduces the yield from 96% to 5%. Fe, Ni, etc. have no appreciable effect even in relatively high concentrations.

10 - Washed tetracene is tested for pH.

11 - Acceptable tetracene is labeled with date and lot number and stored in conductive rubber container.

Note: If aminoguanidine-acetate solution is not complete after addition of H-Ac, heat pail contents to 45 ± 2°C. Afterwards allow time for the solution to cool to 35 ± 1° C before next step.

PURPOSE: Tetracene wash water, pH determination

EQUIPMENT:

1 - 100 ml glass bottle with rubber stopper
2 - Laboratory pH meter

MATERIALS:

none

SAFETY PRECAUTIONS: std. laboratory

PROCEDURE:

1 - 25 ml of the clear water from the storage container of tetracene is placed in bottle and labeled (indicating contents, date and bucket number).
2 - Bottle with sample is transferred to the laboratory and tested during the day sample was taken.
3 - Sample is placed in the pH meter.
4 - The pH of water sample must be within 6.00 - 7.00.
5 - If test results deviate, the tetracene in the corresponding container must be rewashed and test repeated.

Note: Tetracene which does not pass this test must not be used in primer mix.
An interesting footnote to the Tetracene preparation.

The U. S. patent No. 3,707,411 dated December 26, 1972 and assigned to the Dynamit Nobel Co. of Troisdorf, Germany, lists a priming mix for caseless ammunition consisting of:

Tetracene 50% by weight
Nitrocellulos (Gun Cotton 50% by weight

The above mix exhibits bood precussion sensitivity and propelleant ignition characteristics.

Chapter 9

Preparation of Lead Styphnate

GENERAL:

Lead styphnate is the major explosive component of the currently used wet priming mixes.* The method described below details the preparation of normal lead styphnate as used by many commercial and military installations throughout the world. The description is illustrating a one pail/batch method. Again, increasing the number of pail-batches to 6 - 7 pails gives a daily shift volume of 6 final mixes. However, the production capacity must always consider the fact that sensitive primary explosive is being handled. Hence care and safety are most important. Ample time must be given to the operator to perform the required work safely, without urgency, and systematically. Excessive zeal and urgency only lead to fatal accidents.

Systems and methods have been developed for continuous production of lead styphnate. Their advantages are increased productive capacity and safety. Again the safety aspect is the most important, as only a small portion of lead styphnate is in the process at any one time.

However, the penalty is the cost and complexity of the equipment. The system of pumping, metering, accurate and sensitive controls, etc. require extra service and maintenance.

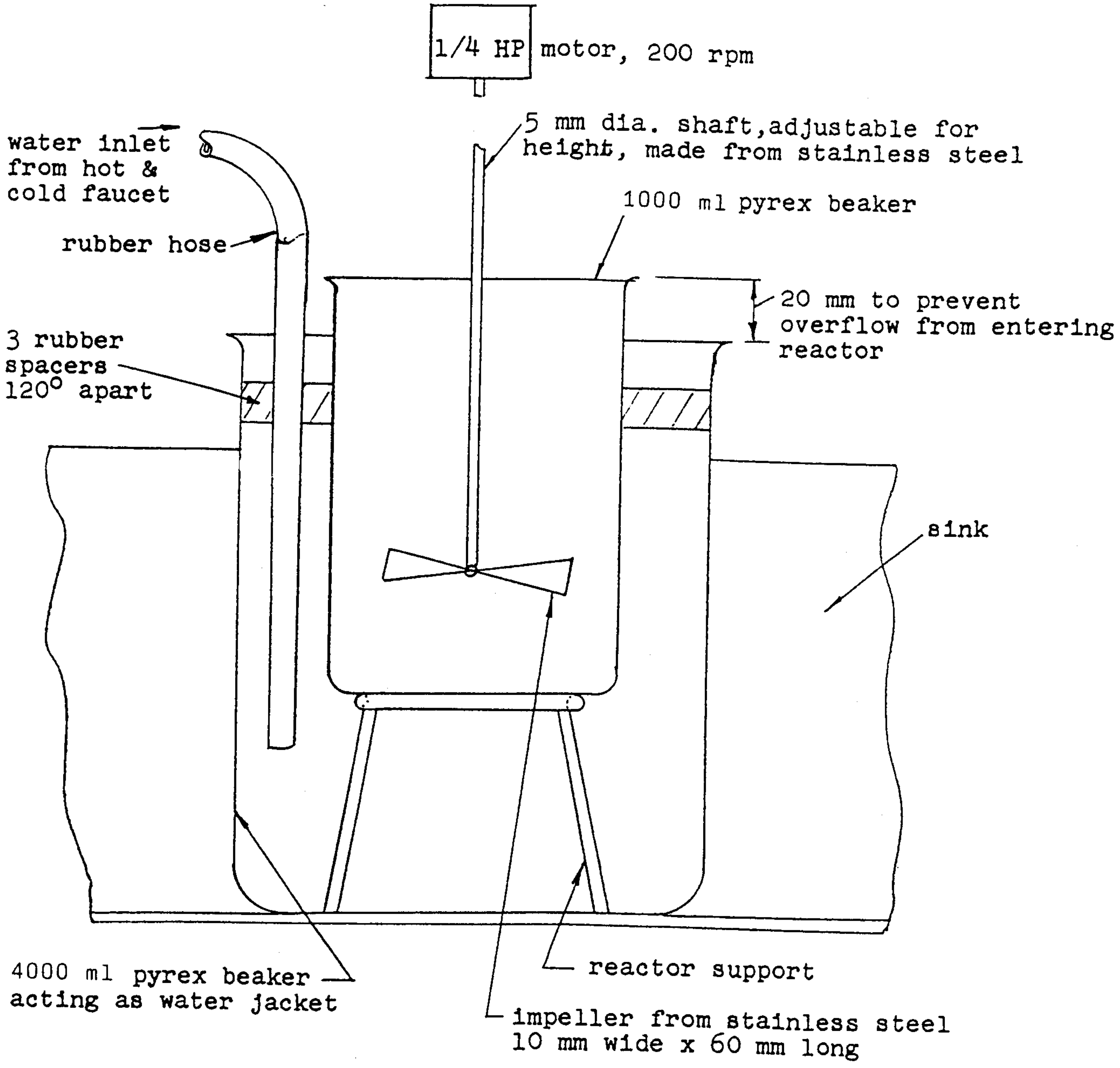
For a production capacity of about 100 million primers per year the 6 pail line is adequate, with minimum equipment requirements.

*The lead styphnate is a lead salt of styphnic acid (trinitroresorcinate; it is prepared by adding lead nitrate to sodium styphnate in the presence of acetic acid.

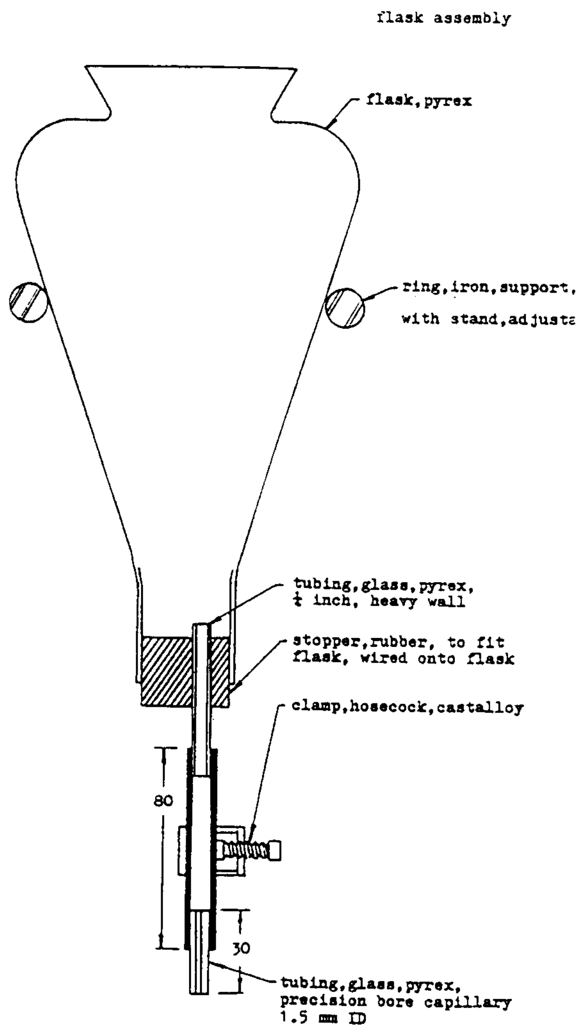
Note: The preparation of lead styphnate and tetracene and the washing of PETN are essentially laboratory type operations.

SAFETY PRECAUTIONS – lead styphnate

LABORATORY SCALE REACTOR MADE FROM OFF-THE-SHELF COMPONENTS



FLASK ASSEMBLY

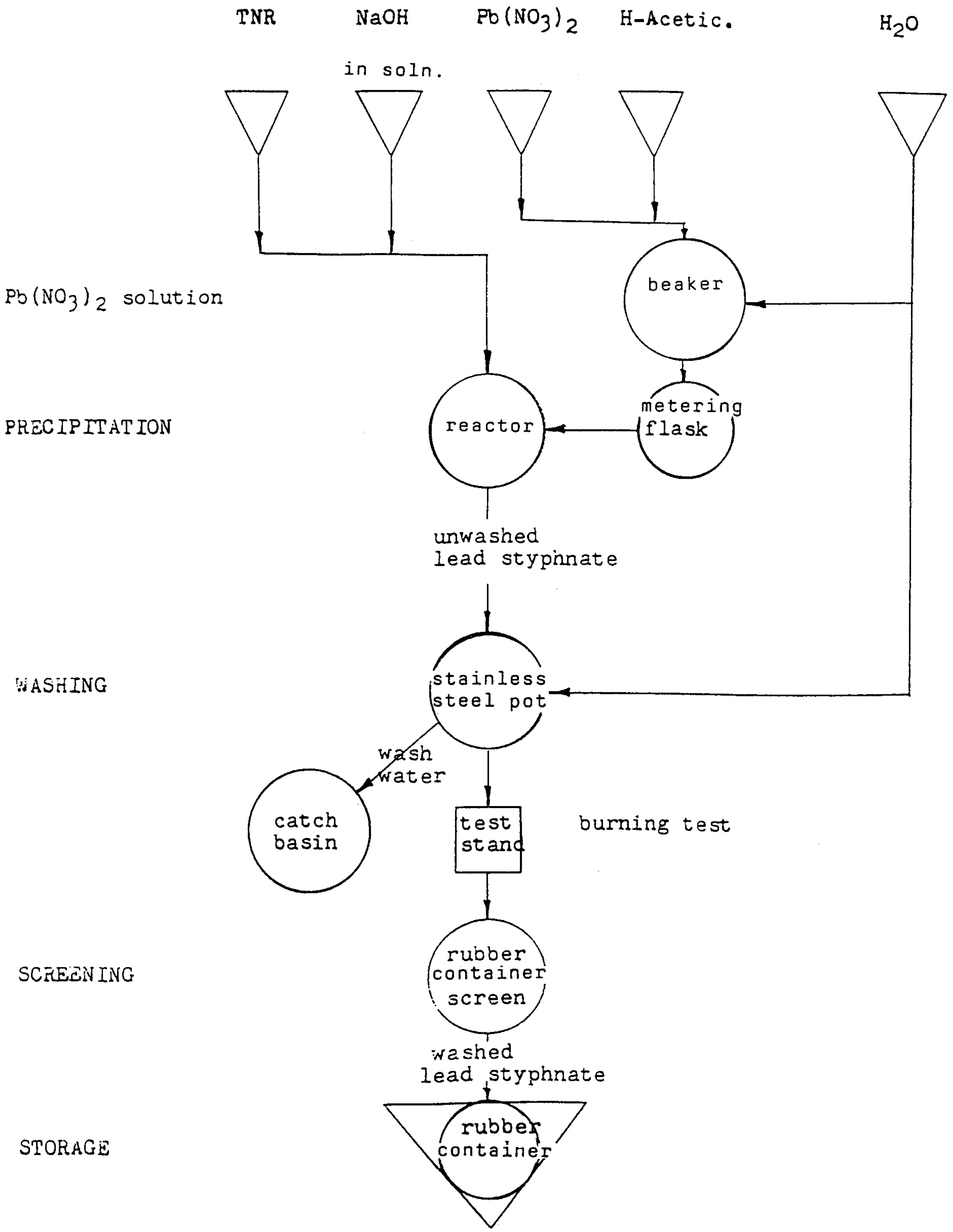


1 - Eye protection is worn while working with lead styphnate, lead nitrate, sodium hydroxide, acetic acid.
2 - Rubber gloves to be worn to minimize staining hands and to protect them from chemicals.
3 - Nose and mouth respirator mask to be worn when weighing lead nitrate.
4 - Lead compounds are toxic, wash hands thoroughly before eating or smoking.
5 - Lead styphnate is a sensitive primary explosive but is safe to handle in wet condition prepared by this process.
6 - Spilled lead styphnate must be wiped up immediately before it dries up. Keep spillage and splash to a minimum.
7 - Keep floors wet continuously and wash them with hose after a day's shift.
8 - During the precipitation stirrers must not strike the side of the reactor. The surface of the stirrer blade and the reactor walls are covered with a very sensitive layer of lead styphnate which would probably explode on impact. Although the quantity is small and would not produce any damage it could initiate the contents of the reactor and produce a substantial explosion.
9 - Wash and clean reactor thoroughly after each precipitation.
10 - The catch basin explosives are to be scrapped at intervals not exceeding two (2) weeks, (sooner if the explosive accumulation in the catch basin warrants it) because it is possible for the explosive in the catch basin to grow the crystal size and become more sensitive.
11 - Since the solubility of sodium styphnate (compound formed when scrapping lead styphnate) is limited, it is important to scrap the catch basin contents as soon as enough explosive scrap accumulates.
12 - Lead styphnate not passing the burning test after repeated washes is scraped immediately in the reactor in which it precipitated.
13 - Place the reactor with lead styphnate to be scrapped under the stirrer in the precipitation sink.
  - Add water until the reactor is half full.
  - Heat water in sink.
  - Start stirrer by hand.
  - When the temperature of the reactor contents reaches 50° - 60° C add 100 ml sodium hydroxide solution.
  - Allow 3 minutes for reaction.
  - Add glacial acetic acid (enough acid to obtain acid reaction).
  - Results a dark colored solution free of suspended solids.
  - Remove reactor to wash sink.
  - Flush pail contents down the drain with ample amount of water (tap).
14 - Scrapping lead styphnate in catch basin.
  - Shut off water flow into the catch basin, and allow contents to settle.
  - Remove baffle plates, pulling upward on rods joining them.
  - Place baffle plates in wash sink.

SAFETY PRECAUTIONS Cont'd.

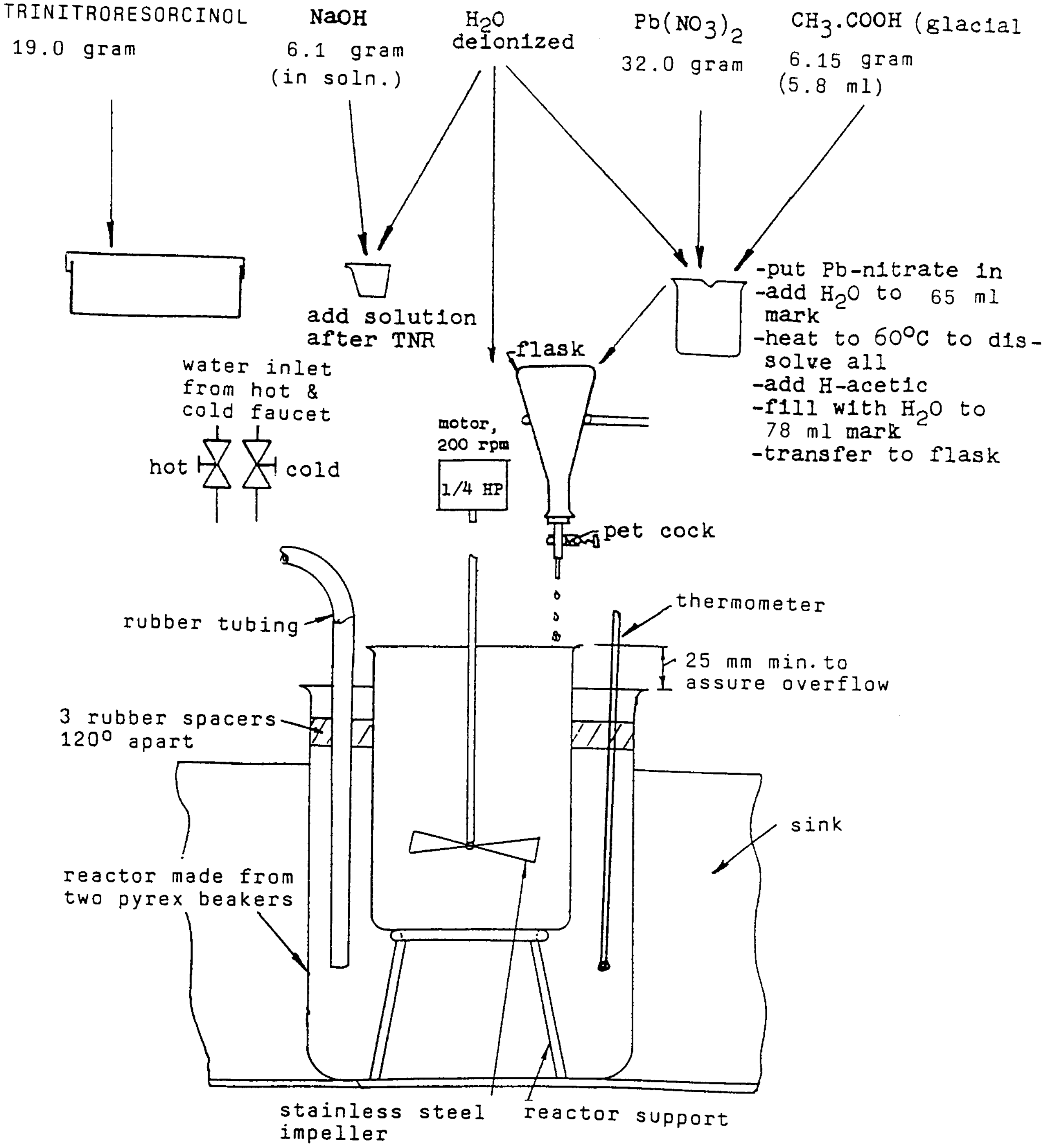
- Carefully, not to stir the settled solids, bail out the water from the basin and pour it into the precipitation sink.
- Plug the catch basin drain with a rubber stopper.
- Rinse off the baffle plates with a little water from a hose.
- Lower the steam pipe outlet into the catch basin so that it extends 5-10 cm below the remaining water surface.
- Open steam valve and allow steam to flow into water.
- Mount a portable electric stirrer on a board next to catch basin so that the blade will be about 2 cm from the bottom on side opposite to steam inlet.
- Manually try rotating the stirrer to check that it clears the catch basin.
- Start the stirrer.
- Continue steam flow until the basin contents heats to about 65° C, this increases the solubility of sodium styphnate.
- Since there is almost no change in appearance of the suspended explosive it is difficult to estimate the amount of sodium hydroxide necessary to scrap the explosive. Therefore estimate the weight of explosive in the basin and add 1000 ml (50 gram solid NaOH) solution for every 100 g of explosive.
- Lead styphnate decomposes even better in presence of tetracene; tetracene decomposes into ammonia and other soluble products. Ammonia in turn reacts with lead styphnate, forming very soluble ammonium styphnate.
- PETN in basin is only saponified by the NaOH and does not decompose lead styphnate.
- Allow the NaOH to act on the explosive for a few minutes.
- Add glacial acetic acid in quantity equal to that of NaOH; the suspended mixture changes from brown color to a solution of almost black color. The addition of H-Ac also raises the temperature of the materials. The odor of H-Ac in the solution is indication that enough acid has been added.
- After no more suspended solids are in evidence, pull out the rubber stopper from the drain and open tap water valve in the wash sink so that water runs into the catch basin to displace the dark solution.
- Keep the steam and stirrer on for 2 minutes after the basin clears up.
- Stop the stirrer, remove from basin and rinse the blades.
- Pull the steam outlet from the water and shut the steam flow, so that solution does not draw into the steam line as the steam condenses.
- If the solution is not run into the drain immediately and is allowed to cool, it will separate yellow, gelatinous sodium styphnate. Should this happen just reheat with steam and dilute with tap water before running it out into drain.
- If the suspension does not turn into solution after addition of H-Ac, repeat the process starting with addition of NaOH.
- If the above process does not dissolve all explosives in the catch basin, then repeat the operation from the beginning.
- If only a small amount remains undissolved, leave it for the next clean-up.
- During the catch basin cleaning avoid excessive friction or hard blows to the explosives in the basin.

PROCESS DIAGRAM - LABORATORY PREPARATION OF LEAD STYPHNATE



LABORATORY PREPARATION OF LEAD STYPHNATE

$PbC_6H_3N_3O_9$
MOL. WT. 468



- put Pb-nitrate in
- add H_2O to 65 ml mark
- heat to 60°C to dissolve all
- add H-acetic
- fill with H_2O to 78 ml mark
- transfer to flask

Lead Styphnate yield 30 gram

LABORATORY PREPARATION OF LEAD STYPHNATE

1 - Place TNR in reactor.
2 - Add water to 135 ml mark.
3 - Place stirrer in reactor, start rotation and check that stirrer blades do not touch reactor.
4 - Heat jacket water to 55°- 60° C.
5 - Fill flask with Pb-nitrate solution and place over reactor.
6 - Check reactor contents temperature with a thermometer periodically.
7 - When reactor contents is at 40° - 45° C add Na0H solution; TNR reacts forming a Na-styphnate and the temperature raises to ± 55° C.
8 - Open petcock on flask and let Pb-nitrate solution in at a rate of 100 cc/minute as the nitrate runs into the reactor the mixture becomes progressively thicker and thicker until a gel forms and mixture movement stops.
9 - At this stage heat jacket: water to 70° C; the formed gel is a highly hydrated form of lead styphnate. Under microscope it appears as a fibrous mass of needle like crystals. Under continuous addition of Pb-nitrate the gel breaks up and the mixture movement resumes. By the time all Pb-nitrate runs into the reactor the mixture becomes a free fluid with suspended dense crystalline monohydrated lead styphnate.
10 - Drain the hot jacket water and fill jacket with cold water as soon as all Pb-nitrate has been added. This increases the LS yield.
11 - Continue stirring the mixture 5 minutes after last Pb-nitrate has been added.
12 - With a clean glass rod place a drop of reactor liquid on a slide and inspect under the microscope:
    a) If gel is present, continue stirring 5-10 minutes more to complete- the conversion.
    b) If no gel is present, the batch is ready for further handling.
13 - Stop and remove stirrer from reactor.
14 - Remove Pb-nitrate flask.
15 - Wash lead styphnate six (6) times as per washing instructions.

LEAD STYPHNATE

Notes:

- TNR sample left drying at 100° C over 12 hours starts sublimation and dry weight calculation error becomes excessive; if necessary to dry TNR over 12 hours keep oven at 55° ± 5° C.
- NaOH solution is 75g NaOH/100 cc.
- Only 25.7 g Pb-nitrate are required for theoretical reaction.
- Excess is used to:
  a- Ensure completion of reaction.
  b- Decrease LS solubility (common ion effect).
  c- Eliminate measuring error in Pb-nitrate weighing.
- Pb-nitrate solution to be kept at 21° C or more to prevent precipitation from saturated solution.
- Acetic acid - is added to:
  a- Speed gel conversion (step 10) from needlelike crystals to the LS suitable for wet priming mix.
  b- Not for acidity, since the mixture is acid throughout the whole reaction already.
- Addition of acetate ions in equivalent quantity or other acids do not produce the desired results. The gel formation can also be eliminated by higher reaction temperature or lower reagent concentration, but the resulting product is not as safe to handle in wet mix as when following the described method.
- The above described process of reaction of sodium styphnate and lead nitrate yields a relatively safe product of uniform quality.
- The lead styphnate sensitivity depends on the shape and size of the crystals formed, as well as on the relative dimensions of the crystal axes. The adulterant material formed in the styphnic acid changes these dimensions by restraining growths along what would be the longest axis. This factor greatly reduces the wet lead styphnate sensitivity.

- **Lead Styphnate Crystals:**
  Shape - From the user's point of view the optimum form is a thin regular hexagon; long hexagons are due to shortage of adulterant (typical when using Gable type TNR). Excessive amount of adulterant in the TNR produces LS crystals where the two sides parallel to the long axis disappear and a cube or rectangle is formed. Process conditions such as rate of lead nitrate addition, reaction temperature etc. do not change the relative axial dimensions, only the overall crystal size.

  Size - Although the above process is relatively simple, the conditions and temperatures should be followed carefully for correct crystal size. Fine LS crystals make the priming mix look wetter while large crystals give it a sandy texture.

MATERIAL SPECIFICATION
Lead styphnate, normal

USE: Priming mix ingredient

APPEARANCE: Light brown color, dense crystalline monohydrated material free from foreign matter and fused clumps of individual crystals; material is also free from needlelike crystals of hydrated lead styphnate.

REQUIREMENTS:

Apparent Density : 1.6 ± 0.1 g/cc
Crystalline Structure: Hexagonal plate type crystals with length and width almost equal; longest dimension of any crystal 0.2 mm max.
Granulation through U.S. Std. No. 45 sieve, in wet condition : 100%

Heat Sensitivity:

Results	*Action*
"OK"	accepted
"pitted slightly"	accepted
"pitted badly"	rework
"burned"	scrapped

Composition:

Pb contents	44.0 ± 0.4 %
pH of saturated Solution	5.0 -7 .0
N contents	8, 97 ± 0.1 %

Inspection: See tests

Packing : Lead styphnate slurry containing minimum 10% denatured ethyl alcohol and minimum 10% water is packed in rubberized cloth bag; each bag containing approximately 10 Kg (dry weight) normal lead styphnate has a cap of rubberized cloth of the bag diameter placed over the top of the slurry before being tied.
Five or less of these bags are placed in a steel drum lined with rubberized cloth or polyethylene bag. The large bag is tied securely and the remainder of the drum space is filled with sawdust saturated with 50-50 solution of denatured ethyl alcohol and water. The steel drum is closed and checked to be watertight.
Each container is labeled in accordance with local regulations on shipment of initiating explosives.

Preparation of Lead Styphnate

PURPOSE: Determination of relative sensitivity of wet lead styphnate.

EQUIPMENT:

1 - 30 ml pyrex glass or polyethylene beaker.
2 - Burning test stand.
3 - Brass teaspoon or scoop.

MATERIALS:

1 – Paper towels, commercial grade.
2 - Black powder, FFG granulation.

SAFETY PRECAUTIONS: The same as for Lead Styphnate preparation.

PROCEDURE:

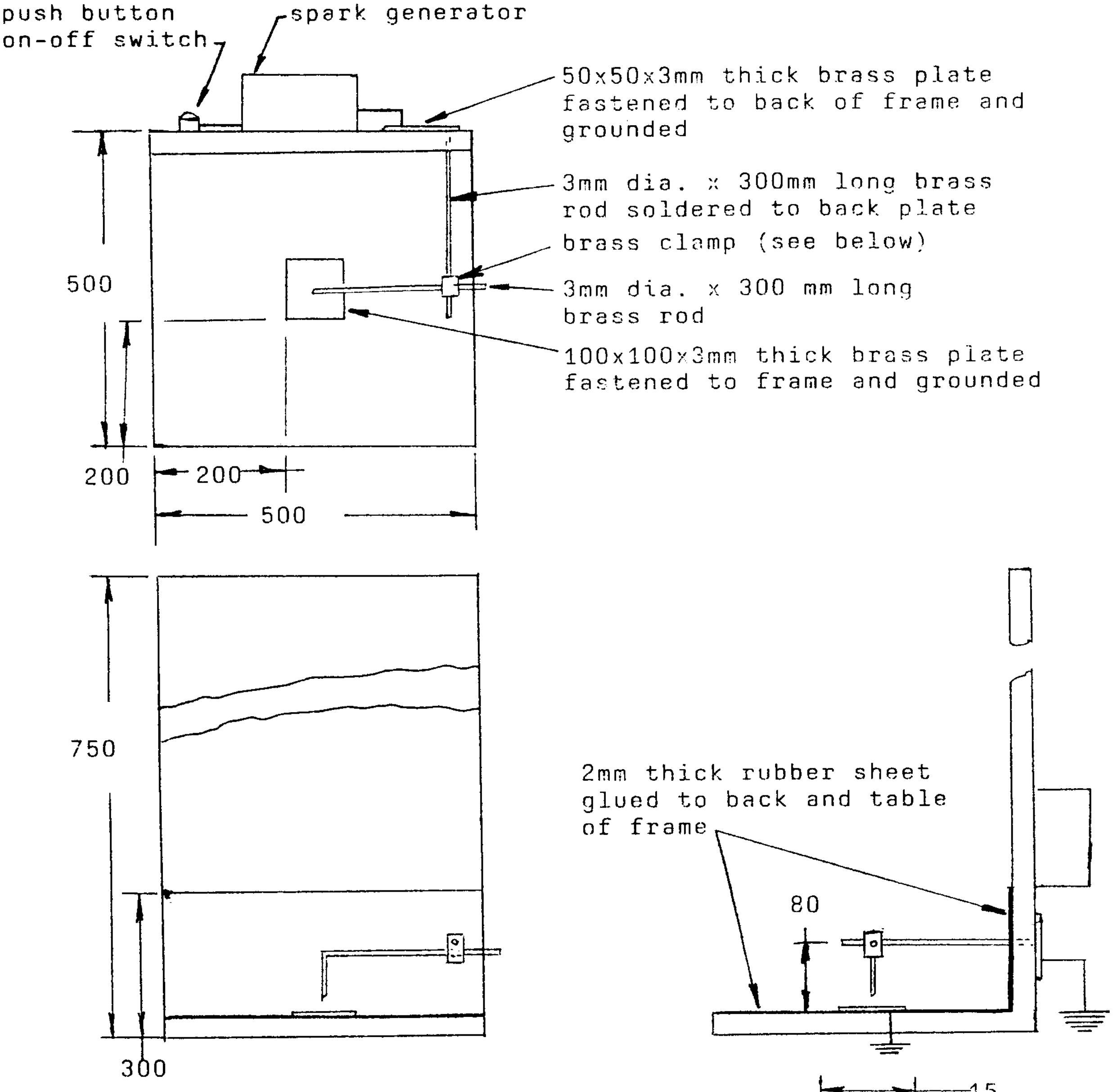
1 - ± 3 gm sample of washed lead styphnate is transferred from the precipitation vessel into a 30 ml beaker, using gloved finger.
2 - Number beaker to identify the vessel from which sample taken.
3 - Place a small lump (about pea-size, ± 0.3 g dry weight) of lead styphnate in the center of the brass plate on the burning test stand.
4 - With a strip of paper towel blot the lead styphnate; apply pressure with the thumb to paper to remove as much water as possible.
5 - Repeat step 4 with a new portion of the paper strip until the paper no longer appears wet; at this stage the sample moisture contents is ± 20 %.
6 - With brass spoon pile 2 spoonfuls of the black powder on top of the lead styphnate lump and on the brass plate immediately adjacent to it.
7 - Swing the unit rod so that the point is over the brass plate; and the black powder between the plate and the point.
8 - Step behind the partition of the unit and turn on the switch; line current flows into the transformer and activates the induction coil. A spark jumps from the rod to the plate igniting the powder.
9 - Turn off the switch.
10 - Examine carefully the lead styphnate lump surface and record results:

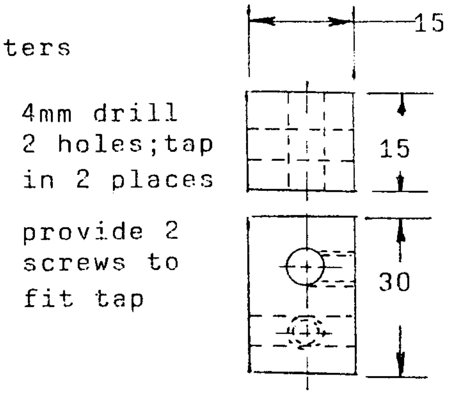
See Next Page

OK – when the only change in surface is blackening due to powder smoke.
Lightly pitted - Surface erupted in a few places only.
Badly pitted - Surface erupted in several places, but no fragments broke away from the lump burned.
- Lump surface is entirely erupted and particles of lead styphnate are scattered around the plate surface.
- sample burned and/or detonated so that nothing is left of it.

11 – OK and lightly pitted lead styphnate is acceptable.
Badly pitted " is reworked and retested.
Burned " is scrapped.

BURNING TEST STAND





NOTES:
1) all dimensions are in millimeters
2) the unit is constructed from 5mm (¼") plywood on 50x50mm (2"x2") wooden frame,glued and screwe together
3) test stand may be placed on laboratory bench

PURPOSE: Lead styphnate inspection.

EQUIPMENT:

1 – Microscope with 150X magnification and graduated eyepiece and stage.
2 - Laboratory drying oven.
3 – Analytical laboratory balance.
4 - Laboratory size pycnometer.
5 –U.S. Std. screen No. 45.

MATERIALS:

1 – Concentrated sulfuric acid
2 – Concentrated nitric acid
3 – Ammonium acetate
4 – Distilled water

SAFETY PRECAUTIONS:

The same as for Lead Styphnate preparation.

PROCEDURE:

A) Crystal shape and size:
1 – A drop of lead styphnate slurry is spread in a thin layer on a microscope slide.
2 – Place slide under microscope and inspect sample for impurities and crystal form.
3 – Using the millimeter scale on the stage determine the two largest dimensions of the largest crystal.

B) granulation:
1 – Using laboratory pycnometer (6.7 g pycnometer difference) weigh 10 g dry weight of lead styphnate.
2 – With a stream of water wash the lead styphnate from the pycnometer onto the No. 45 screen.
3 – The material is collected in a rubber pail below the screen.
4 – When no more lead styphnate passes through the screen allow screen to drain.
5 – Place the drained screen into dryer oven at 50° C for 60 minutes.
6 – Dry screen is examined under strong light; presence of visible amounts of lead styphnate on the mesh is cause for rejection of the lot.

C) Pb contents:
1 – Using H_2SO_4 and HNO_3 convert lead styphnate into $PbSO_4$ (water insoluble lead sulfate).
2 – Filter off and wash the lead sulfate.
3 – Dissolve lead sulfate in ammonium acetate.

4 - Adding potassium chromate, precipitate lead as $PbCrO_4$ (yellow lead chromate).
5 - Filter $PbCrO_4$ and wash.
6 - Dry the lead chromate.
7 - Weigh the lead chromate and calculate % Pb.

D) Apparent density:
1 - Place 3 cc of n-butyl alcohol into a 5 cc graduate (7.5 mm internal diameter) graduated in 0.05 cc.
2 - Add 2.00 g (dry weight) of lead styphnate in small portions and wash down the walls of the graduate with butyl alcohol.
3 - To assure thorough wetting, shake the graduate carefully by inverting it at least five times.
4 - Add butyl alcohol to the 5 cc mark.
5 - Allow the graduate to stand 180 minutes.
6 - Record the volume occupied by the lead styphnate.
7 - Apparent density is:

$$\text{a.d.} = \frac{\text{sample weight (gm)}}{\text{sample volume (cc)}}$$

E) Material insoluble in ammonium acetate:
1 - Accurately weigh a sample of one (1) gram dry lead styphnate.
2 - Add sample slowly to 200 cc of 10% $NH_4C_2H_3O_2$ at ambient temperature, stirring constantly.
3 - Stir until no more of the sample dissolves (± 15 minutes).
4 - Filter solution through a tared, fritted glass filtering crucible of porosity F (Gooch crucible).
5 - Wash all particles from the beaker into the crucible with jet of distilled water from a wash bottle.
6 - Wash the crucible until the wash water comes through colorless.
7 - Dry crucible and contents in oven for 40 minutes at 102° ± 2° C.
8 - Place crucible and contents in desiccator at ambient temperature to cool.
9 - Weigh and calculate the weight difference as percent material insoluble in ammonium acetate.

F) Acidity:
1 - Accurately weigh one (1) gram of lead styphnate and transfer into a 150 cc beaker.
2 - Add 100 cc of freshly boiled and cooled distilled water having a pH of 6.0 ± 0.5.
3 - Leave mixture standing 15 minutes, stirring occasionally.
4 - Filter and determine pH of the filtrate at 25° ± 2°C using meter which reads directly in pH units with accuracy of ± 0.1 pH. The meter shall have glass electrodes and shall be calibrated with standard buffer solution.

PURPOSE: Lead styphnate, pH determination.

EQUIPMENT:

1 - 30 ml bottle with rubber stopper
2 - pH indicator with glass electrode, reading directly in pH units and accurate 0.1 pH

MATERIALS:
None
SAFETY PRECAUTIONS:
Std. laboratory

PROCEDURE:

1 - Collect sample ± 25 ml of supernatant saturated solution from the rubber bucket of lead styphnate in storage.
2 - Cover sample bottle with rubber stopper and label, indicating bucket number, date and test.
3 - Make the test according to the instructions by the pH indicator manufacturer
4 - pH to be 5.00 - 6.00 for acceptance of lot.
5 - pH less than 5.00 indicates definitely poor wash.
6 - If pH of sample is out of acceptance limit (above or below) rewash the lead styphnate in the corresponding bucket.
7 - Retest sample of solution from the rewashed bucket of lead styphnate. contents not passing acceptance test will not be released for primer mix.

PURPOSE: Determine NaOH solution concentration

EQUIPMENT:

1 - 5 ml pipette
2 - 50 ml volumetric flask
3 - 5 ml beaker
4 - Titration unit

MATERIALS:

1 - Methyl orange indicator
2 - Distilled water
3 - HCl

SAFETY PRECAUTIONS: Std. laboratory

PROCEDURE:

1 - With a pipette draw 2 ml. sample.
2 - Dilute sample to 50 ml. in volumetric flask.
3 - Shake flask to obtain homogenous solution.
4 - With clean pipette draw 2 ml. of diluted solution to a beaker.
5 - Titrate the diluted sample with standard HCl of 0.4-0.5, normally with methyl orange indicator.

PURPOSE: Scraping lead styphnate

EQUIPMENT:
1 - Container for H-acetic
2 - Container for NaOH solution
3 - Sink with hot and cold running water

MATERIALS:
1 - Acetic acid, glacial or 56% commercial (whichever is less expensive)
2 - NaOH solution (400 g of technical NaOH flake in 705 ml. H2O)
3 - Water, tap, hot and cold

SAFETY PRECAUTIONS: The same as for Lead Styphnate preparation.

PROCEDURE:
Lead styphnate dissolves in action of sodium hydroxide and acetic acid as follows:

$$\text{Styphnate: Pb} + 2\,\text{NaOH} \rightleftharpoons \text{Styphnate : Na}_2 + \text{Pb(OH)}_2$$

The equilibrium tends to follow the left side of the equation as seen by the scarcity of $Pb(OH)_2$ which is white in color. Availability of NaOH allows conversion to the right side of the equation, but great excess of NaOH again decreases the solubility of Na-styphnate. Addition of H-acetic produces irreversible process: *)

$$\text{Styphnate : Na}_2 + \text{Pb(OH)}_2 + \text{CH}_3\text{COOH} \Rightarrow \text{Pb(CH}_3\text{COO)}_2 + \text{styphnate.O.Na.O. Na}$$

Lead acetate and Na- styphnate are soluble in water (especially hot) and may be run into the drain. The combination of NaOH and H-acetic works better than Na-acetate because:

a) Heat of neutralization raises the temperature, thus increasing the reaction and solubility of Na-styphnate.
b) Excess of H-acetic makes the solution acid increasing the solution of lead.

*) The final product of the reaction is a complex lead-aceto-styphnate, water soluble and safe for disposal into drain.

Chapter 10

Diazol Preparation

Lead has been identified as one of the key problems in environmental pollution. Lead-free automotive fuel, lead-free paint, iron shot, are some of the examples of how to eliminate this pollutant. Lead styphnate base primers fired in relatively large numbers in the indoor ranges used by military and police are also considered as sources of lead pollution.

Studies have been conducted to develop a lead-free priming mix which would solve this problem. One such mix is based on the use of Diazodinitrophenol (1JDNP)*

Following is the description of a manufacturing process of a typical DDNP/Tetracene type priming mix.

*The preparation, physical and chemical characteristics of DDNF are given in detail in *Davies, Meyer and Enc.Expl.*

PREPARATION:

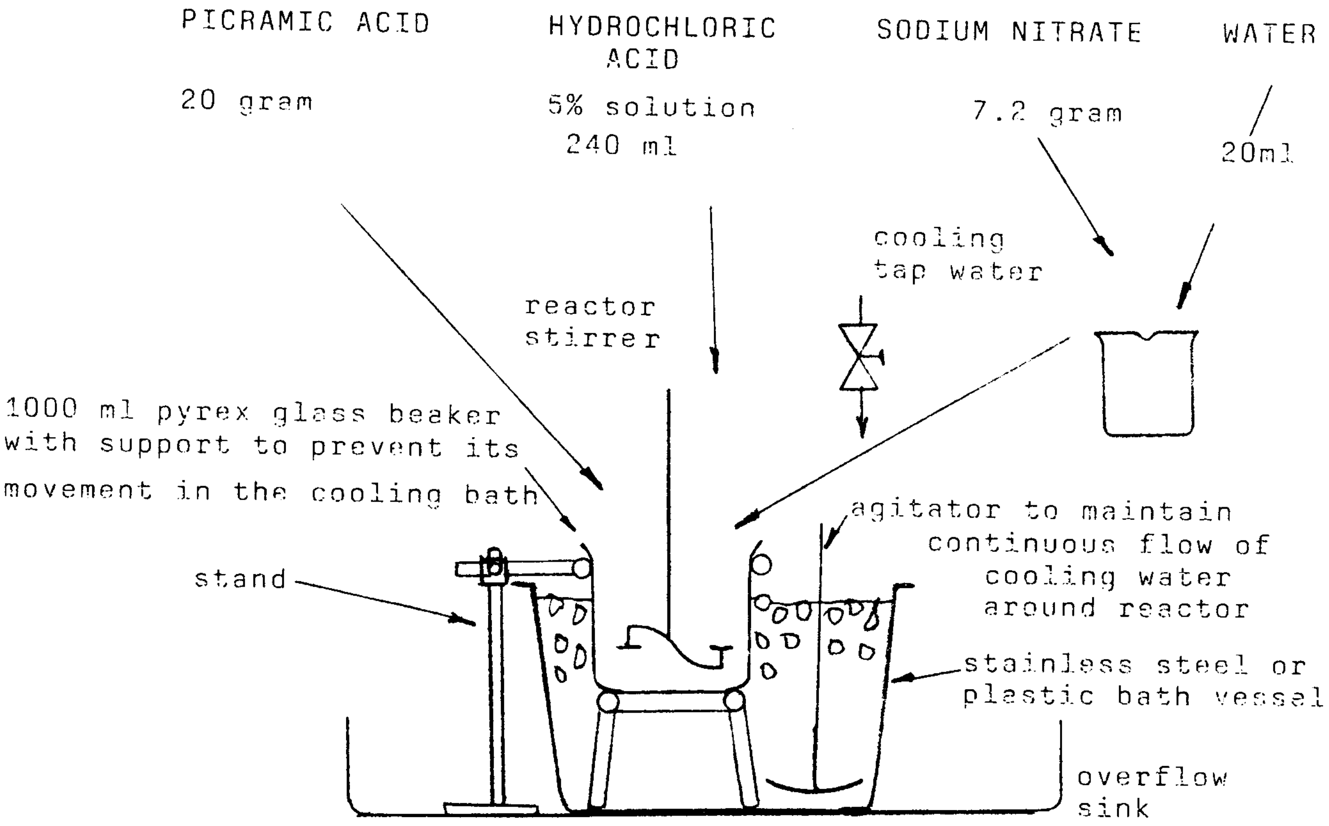
Diazodinitrophenol

- Material purchased from vendor (Hercules Powder, Trojan Powder).
- Screened through No. 60 mesh with stream of low pressure water.
- Stored under water in saturated condition.
- Prior to use place material in suction funnel and remove excess water.
- Transfer material onto paper covered tray and dry at 4000.
  Note: Safety and handling procedures of dry DDNP is the same as for dry mercury fulminate.
- Maximum 400 grams of dry material are kept in a container for transfer to weighing area.

Tetrazene

- Material received from storage is dried and handled in the same manner as diazodinitrophenol.

LABORATORY PREPARATION OF DIAZODINITROPHENOL (DDNP)



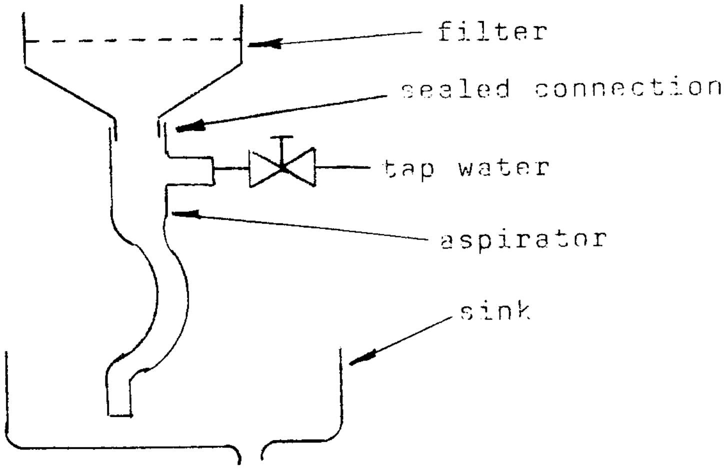
PROCEDURE:

1 - Pour hydrochloric acid solution into the reactor.
2 - FIl cooling bath vessel with crushed ice and cold (or chilled) water.
3 - Start cooling water agitator.
4 - Adjust small cooling water overflow.
5 - Add picramic acid to the cold reactor contents.
6 - Pour ALL AT ONCE the previously prepared sodium nitrate solution into the reactor.
7 - Start reactor stirrer and gradually increase its speed to 150 RPM.
8 - Continue stirring 20 minutes.
9 - Stop reactor stirrer.

LABORATORY PREPARATION OF DIAZODINITROPHENOL (DDNP)

PROCEDURE: (continued)

10 - Start filter aspirator.
11 - Transfer reactor contents onto the filter with a small jet of ice cold water.
12 - Rinse the reactor with ice cold water and pour the rinse over the material on the filter.
13 - With ice cold water wash the material an the filter six or more times.



14 - With a small jet of ice cold water transfer the washed material from the filter into the storage container.
15 - Allow the DDNP to settle at the bottom; then decant the top water allowing about 10 mm (1/4 inch) water over the DDNP.
16 - Cover the container, label with date and lot and transfer to explosive storage.

Note: If dry DDNP is needed, it is dried in the same manner as other high explosives (such as PETN, TNT, Lead Styphnate or Mercury fulminate).

PREPARATION PROCEDURE:

1 – Weighed nitrate, antimony sulfide are blended on a sheet of paper and rescreened through No. 60 mesh, in a manner described.

2 – Blended materials are placed in jelly bean bag.

3 – Dry tetracene is added to the jelly bean bag.

4 – Calcium silicide is added to the jelly bean bag.

5 – The bag contents is blended 5 minutes.

6 – Contents from the jelly bean bag is placed into a wetted mixing machine.

7 – Gum solution is poured over the contents.

8 – Machine is run forward one (1) minute.

9 – Machine is stopped.

10 – Diazodinitrophenol is added to the mixing machine contents.

11 – Machine is run forward 5 minutes.

12 – Machine is stopped.

13 – Machine is run reverse 2 minutes.

14 – Stop machine.

15 – Run machine forward.

16 – While the machine is running dump the priming mix onto a prepared tray by inverting the mixing machine drum.

17 – The machine is run for 2 minutes in upside-down position.

18 – Repeat steps 32 through 61 of chapter eleven (11).

E. Tabular diazodinitrophenol shall be of but one grade.

E–1. *Acidity (as HCl).*—Maximum, 0.01 percent.

E–2. *Color.*—Green.

E–3. *Form.*—Tabular crystals having a maximum length of 0.2 mm.

E–4. *Granulation.*—100 percent through a No. 100 U. S. Standard sieve. Sieves shall conform to Federal Specification RR–S–366.

E–5. *Bulk density (dry).*—Minimum, 0.4 gm. per cubic centimeter.

E–6. *Sand test requirement.*—Weight of sand crushed, minimum, 33 gm.

F. METHODS OF SAMPLING, INSPECTION, AND TESTS

F–1. *Size of lot.*—A lot shall consist of not more than 300 pounds of diazodinitrophenol (dry weight).

F–2. *Sampling.*—By means of the hand or a horn spoon, sufficient material to form a primary sample of approximately 100 gm. (dry weight) shall be removed from each barrel or drum in the lot. Primary samples shall be made up of equal portions taken from each bag in the barrel or drum. Each primary sample shall be blended on a smooth surface by mixing with a horn spatula. Water shall be added in case the sample appears to be dry. The sample shall be spread out and divided into squares approximately ½ inch on a side by means of the spatula. Small portions shall be taken from each square to make a retained portion of the primary sample of approximately 20 gm. (dry weight). The retained portion of each primary sample shall be placed in a tightly stoppered bottle so labeled that the barrel or drum from which the sample was taken can be easily identified. The remaining portions of the primary samples shall be thoroughly mixed, spread out, marked into squares and small portions taken from each until a composite sample of approximately 50 gm. (dry weight) is obtained. This composite sample shall be placed in a smooth-necked bottle with a tight-fitting stopper and labeled to show the name of the material, manufacturer, plant, contract or order number, lot number and the number of pounds in the lot. All acceptance tests shall be made on the composite sample representative of the lot. The primary samples shall be held for possible future examination should the composite sample fail to meet the requirements of this specification.

F–3. *Inspection.*—

F–3a. *Army.*—Inspection shall be made in accordance with the requirements of U. S. Army Specification 50–0–1 and shall be made at the point of delivery, unless otherwise specified in the contract or order.

F–3b. *Navy.*—Inspection shall be at the point of delivery, unless otherwise specified in the contract or order.

F–4. *Tests.*—The laboratory tests shall be made in accordance with the following paragraphs. For Navy purchases, the tests shall be made at a Government laboratory, unless otherwise specified in the contract or order.

F–4a. *Preparation of dry sample.*—Since the sample as received will contain approximately 40 percent water, the following procedure shall be followed to prepare dry material for those tests where it is called for: Place a piece of filter paper, cut to proper size, in a Gooch crucible held in an adapter on a suction flask. Transfer approximately 3 to 5 gm. of the sample to the crucible with a stream of water from a wash bottle. Air dry the sample by means of suction and then transfer the material to a paper tray. Spread the material to a thin layer, thereby breaking up lumps and then heat in a hot air oven at 60° C. for a minimum of 16 hours. Hold the dried sample in a desiccator or in a rubber stoppered bottle until used.

F–4b. *Acidity (as HCl).*—By means of a hard rubber spatula transfer approximately 4 to 5 gm. of the sample (as received) to a 250-m*l.* beaker. Add 25 m*l.* of distilled water to the beaker, and swirl until the mixture becomes homogeneous, then allow the material to settle out. Decant the supernatant liquid through a tared Gooch crucible (designated as A) containing an asbestos mat. Repeat with a second 25 m*l.* of water. Transfer the filtrate to a 100-m*l.* beaker and add, with constant stirring, 10 m*l.* of approximately 0.1 N silver nitrate. If a turbidity results filter through a tared fritted glass crucible of fine porosity. Wash with alcohol, then with ether, and dry 1 hour at 100° C. Cool and weigh. Transfer the diazodinitrophenol from the beaker to the tared Gooch crucible (A). Wash with 3 successive other washes. Suck until free from ether, then dry for 1 hour at 50° C. Cool and weigh. Calculate the weight of AgC*l* to percentage of acidity as HC*l* as follows:

$$\text{Percentage of acidity (as HC}l\text{)} = \frac{25.5W}{B}$$

where

W = weight of AgC*l*
B = dry weight of sample.

F–4c. *Color.*—The color shall be determined by visual inspection.

F–4d. *Form.*—Spread a thin layer of the sample over approximately a 0.5 square inch area of the central surface of a 1-inch by 3-inch glass microscope slide, in such a manner that the individual crystals are discernible. Measurement of crystal length shall be made by means of a micrometer eye-piece using a magnifi-

F–4e. *Granulation.*—Place approximately 10 gm. of wet dia zodinitrophenol on a No. 100 U. S. Standard sieve and was through with a jet of water.

F–4f. *Bulk density.*—

F–4f(1). *Apparatus.*—Smooth-wall hollow metal cylinder close at one end. The cylinder shall have an inside diameter of 0.21 inch and a length of 1.650 inches, with a capacity of 1 m*l.*

F–4f(2). *Procedure.*—Fill the tared cylinder with dry dia zodinitrophenol by pouring the material into the cylinder whil it is held in a vertical position. The dry material is to be poure into the cylinder from a piece of glazed paper held at the mouth of the cylinder. The surplus material is removed from the top of the cylinder with a piece of paper or other suitable safe object. After leveling the material in the cylinder, tap gently with the finger to cause settling, and weigh. Report the final weight minus tare weight (in grams) as the bulk density of the material under test.

F–4g. *Sand test.*—Transfer to each of two empty No. 6 blasting caps 0.400 ± 0.001 gm. of dried diazodinitrophenol. Each blasting cap shall be composed of gilding metal, and be approximately 1.56 inches long by 0.217 inch inside diameter. Insert a reinforcing capsule (ferrule), with outside diameter of approximately 0.217 inch, completely open at the lower end, and with a hole 0.13 ± 0.01 inch diameter at the top end. Apply a pressure of 3,000 pounds per square inch for 3 seconds. Crimp each cap to one end of a piece of miner's fuse 8 or 9 inches long, taking care that the end of the fuse is held firmly against the charge in the cup. Crimp near the cap mouth so as to avoid squeezing the charge. Pour into the cavity of the sand test bomb 80.0 ± 0.1 gm. of Standard Ottawa sand, which passes through a No. 20 U. S. Standard sieve and is retained on a No. 30 U. S. Standard sieve and level the sand by striking the bomb two or three times. Insert the fuse through the hole in the cover of the bomb, and lower the cap into the bomb cavity so that it is in the center of the cavity and just touching the sand. Pour 120 ± 0.1 gm. more of the sand around the cap and tap bomb as before to level the sand. To avoid possible loss of sand caused by the explosion blowing the burned fuse through the hole in the cover, a piece of rubber tubing about ½ inch long and of such inner diameter that it fits the fuse snugly, is slipped over the fuse and adjusted at a point on the fuse so that the rubber will be against the inner side of the bomb cover when the cap is in position. Fasten the cover securely to the bomb, taking care not to displace the cap in the sand. Light the fuse, and after the explosion has taken place, empty the sand onto a sheet of smooth (glazed) paper taking care to remove any sand which may adhere to the side of the bomb, or to any pieces of the detonator shell or burnt fuse. Empty all the sand on the No. 30 sieve fitted with a pan, and weigh the sand which passes through the sieve after shaking 3 minutes, in a Ro-Tap sieve shaker, or equivalent.

F–5. *Rejection and resubmission.*—If the composite sample representative of the lot fails to pass the inspection tests, the entire lot shall be rejected. However, the manufacturer shall have the option of having an analysis of each primary sample made at no expense to the Government. The manufacturer may then remove or replace defective portions of the lot represented by the primary samples which fail to meet the requirements, and resubmit the lot for acceptance, provided that the markings on the container are such that complete removal or replacement of the defective portions of the lot can be made to the satisfaction of the Government inspector. New samples shall be taken from the entire resubmitted lot and subjected to all of the inspection tests required by this specification. If the resubmitted lot fails to pass the inspection tests, the lot shall be finally rejected.

G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G–1. *Packing.*—Diazodinitrophenol in bulk shall contain when packed, not less than 40 percent of water and in this wet condition shall be packed in accordance with the requirements of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc.

G–2. *Marking.*—

G–2a. Each barrel, drum, or keg must be plainly marked *INITIATING EXPLOSIVE—DANGEROUS—DO NOT STORE OR LOAD WITH ANY HIGH EXPLOSIVE,* in accordance with Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc.

G–2b. Unless otherwise specified, each container shall be marked "DIAZODINITROPHENOL (TABULAR)" with quantity contained therein as defined in the contract or order under which the shipment is made, the name of the contractor, month and year of manufacture, and number of contract or order. In addition, shipments for the Army shall be marked in accordance with the requirements of U. S. Army Specification 100–2; for the Navy in accordance with the requirements of the Navy Shipment Marking Handbook.

H. NOTES

H–1. *Use.*—The material covered by this specification is intended for use in loading fuse detonators and the manufacture

PURPOSE: Analysis of diazol-type priming mix.

EQUIPMENT:

1. Beakers (50, 100, 150, 250, 500 cc)
2. Gooch crucible
3. Filter funnel with suction attachment
4. Meker burner
5. Vacuum drying oven
6. Desiccator
7. Analytical balance
8. Photoelectric colorimeter

MATERIALS:

1. Distilled H_2O
2. Ethanol
3. Filter paper
4. H_2SO_4 (normal sol.)
5. Acetone
6. Acetic acid
7. Alpha napthylamine
8. Ttetracene

PROCEDURE:

A. $Ba(NO_3)$ 2 determination

1. 0.5 gram of mix in 100 cc beaker is moistened with ethanol.
2. Add 50cc H_20 and allow soaking for 12 hrs.
3. Filter on tared Gooch crucible into a 500 cc beaker.
4. Wash with hot H_2O to final filtrate volume of 150 cc.
5. Heat filtrate to boiling.
6. Add gradually 20 cc H_2SO_4 (1-1).
7. Continue boiling 15 minutes.
8. Keep hot 3 hours min. (preferably overnight)
9. Filter on tared and ignited Gooch crucible.
10. Wash twice with hot H_2O.
11. Supporting crucible on a triangle so that it is within but does not rest on a larger crucible.
12. Ignite precipitate to a dull red heat using Meker burner.
13. Place crucible with precipitation desiccator and close.
14. Allow precipitate to cool to ambient temperature.
15. Weigh crucible and precipitate.

$$\% Ba(N0_3)2 = \frac{\text{wt. } BaS0_4 \times 1.11976 \times 100}{\text{wt. sample}}$$

B. PbO_2 determination

1. Crucible with residue from procedure A is placed upright in standard 600 glass funnel.
2. Wash with acetone without using suction until solvent is clear colored (to remove diazo-dimitro-phenol).
3. Wash with H_20 using suction, to remove acetone.
4. Treat the residue on crucible with solution of 25 cc H_2O, 3 cc HNO_3 (conc.), 5 cc H_2O_2 (3%).
5. Allow 20 minutes for dissolution of PbO_2 and tetracene.
6. Apply suction to crucible.
7. Wash residue in crucible with H_2O.
8. Save residue for procedure C.
9. Transfer filtrate to 250 cc beaker.
10. Add 10 cc H_2SO_4 (conc).
11. Evaporate until start of sulphur fumes.
12. Cool residue to ambient temperature.
13. Add 50 cc 20.
14. Add 25 cc ethyl alcohol.
15. Transfer and filter residue on tared and ignited Gooch crucible using solution of 80 cc H_20, 10 cc H_2S0_4 (conc.) 10 cc ethyl alcohol.
16. Repeat steps A-12 through A-15.

$$\% PbO_2 = \frac{\text{wt. } PbSO_4 \times 0.78875 \times 100}{\text{wt. sample}}$$

C. Sb_2S_3 determination

1. Crucible; with residue from B-8 is dried in oven at 100° C for 60 minutes.
2. Dry residue and crucible are placed in covered desiccator and cooled to ambient temperature.
3. Residue and crucible are weighed.

$$\frac{\text{wt. } Sb_2S_3 \times 100}{\text{wt. sample}} = Sb_2S_3$$

D. Diazo-dinitro-phenol determination

1. Weigh 100 milligrams of sample on tared Gooch crucible.
2. Place crucible upright in a glass funnel.
3. Wash sample with small portions of dry acetone (total 5 cc) until acetone is not colored.
4. After wash, apply suction for a short time to remove remaining solvent on asbestos mat.
5. Place crucible with undissolved sample in vacuum oven for 60 minutes at 55° C.
6. Place crucible with dry sample residue in covered desiccator and let cool to ambient temperature.
7. Weight and record.

$$\% \text{ diazo-dinitro-phenol} = \frac{\text{wt. Loss} \times 100}{\text{wt. sample}}$$

Note: Evaporation of acetone solution and subsequent weighing yield incorrect results because DDP is oxidized.

Chapter 11

Wet Priming Mix Blending

The wet priming mixing is the final operation in the priming mix preparation. This operation requires a special mixing machine. This unit is constructed in such a way that the bowl and the rotary blades do not touch and/or grind against each other. Yet the space between them should be as close as practically possible, to assure uniform blending of the mixed materials. The rotating elements should not have any backlash and should stop if encountering too much resistance. Furthermore, the rotary blades should be removed from the bowl by remote control. The bowl, in turn, should be pivotal by remote action to dump the priming mix onto a tray.

Sometimes the priming mix is blended in mixers similar to those used in food processing industry for mixing dough.

Yet the requirements of safety in operation applicable to the Sygma blade mixer applies to these mixers as well.

The remainder of equipment used in the final blending operations is of the commercial laboratory type, i.e. beakers, balances, rubber (conductive or stainless steel) containers, etc.

MATERIALS:

The following table lists two typical priming mixes. All weights are DRY weight in grams, except where noted.

Type of priming		**#956 (Mil)**		**(shotshell)**	
Dry Weight of Mix, Total, Grams		60		60	
COMPONENT	**SPECIFIC**	**%**	**Gram**	**%**	**Gram**
Lead styphnate	MIL-L-757A	37 ± 5	22.2	40	24.0
Tetracene	MLL-T-46938	4 ± 1	2.4	4	2.4
Antimony sulfide	MIL-A-159	15 ± 2	9.0	15	9.0
Barium nitrate	T4IL-B-162	32 ± 5	19.2	30	18.0
PETN	T4IL-P-387	5 ± 1	3.0	5	3.0
Aluminum	MI-A-512	7 ± 1	4.2	6	3.6
Gum solution	Drw.B10522388	-	2 ml	-	2 ml

Unfortunately, a suitable and safe mixing machine is not readily available in most laboratory preparations of wet priming mixes. However, since the quantities of explosives are relatively small, the final blending of the wet mixes may be done in the following manner.

The required quantity of the primary explosives, i.e. Lead Styphnate, Tetracene, DDPH, may be slowly and carefully dried in a secure area similar to the dry mixes. The dry explosive components are then carefully deposited into the jelly-bean-bag. The fuels and oxidizers are also added and the dry mix is carefully and thoroughly blended. After blending the mix is deposited into a shallow soft rubber bowl. The required quantity of binder solution is poured over the mix. A small stream of distilled water is also poured over the mix. Several minutes (10-15) are allowed for the liquid to wet the mix through. The wet mix is gently blended with a soft plastic kitchen spatula into a uniform consistency similar to wet clay.

The wet mix is then covered up and stored in a cool magazine, ready for primer cup charging.

If a proper mixing machine is available, the final blending of the priming mix follows the procedures described below.

EQUIPMENT:

Items

Sink, stainless steel, with platform to hold mixing machine
Tray, stainless steel, with 1 inch lip
spatula, plastic or rubbe
Commercial sponge, plastic
Large table, commercial with splash board, rubber mat top, conductive and grounded, 1 m x 1.8 m
Beaker, glass, capacity 100 ml
Bowl, conductive rubber
Cover, conductive rubber
Carrier, stainless steel or wood construction, to hold bowls with cover
Gloves, rubber, commercial
Hose, rubber, std. garden hose type, for cleaning the work area, 15 ft. length
flask, filtering, glass, capacity, 1 liter
Water aspirator
Buchner funnel, large, with rubber stopper
Cheese cloth, std. commercial
Catch basin, std.
Clock, manual setting, 1 hour dial

SAFETY PRECAUTIONS - Wet Priming Mixing

1 - Eye protection to be worn while working with primer mix and/or components.
2 - Rubber gloves to be worn while working with primer mix or lead Styphnate.
3 - Clean, washable overalls to be worn while working with primer mix. Overalls are Changed at least once every week.
4 - Floor in mixing machine area is kept wet.
5 - Mixture spillage to be kept at minimum.
6 - Spilled mixture is cleaned up immediately.
7 - Tray receiving mix from machine is kept wet.
8 - Mixing machine is wetted and excess water wiped off with sponge prior to use.
9 - Whenever machine is stopped during the mixing operation, the inside of machine must be scraped down with a soft rubber spatula. This prevents mixture from drying out on the machine sides and assures thorough mixing.
10 - Machine is washed out with hose between mixing batches and excess water is sponged off.
11 - If mixing operations are temporarily halted during the day, the empty machine is left standing filled with water.
12 - Machine is taken apart and cleaned thoroughly at the end of day's work.
13 - Machine is inspected by qualified mechanic every two weeks or whenever mechanical difficulties are reported (see step 24).
14 - Mixing batch is inspected after dumping from the mixing machine to check that it is mixed thoroughly.
15 - Unsatisfactory primer mixture scraping follows procedures of lead styphnate scrapping except that sodium hydroxide solution reacts with the aluminum powder. Hydrogen, released by the reaction reduces nitro groups of the lead styphnate into corresponding amines.
16 - Allow 30 minutes for the sodium hydroxide solution to act.
17 - Add glacial acetic acid (enough acid to obtain acid reaction.)
18 - Some reagents, such as ground glass, antimony sulfide, calcium silicide, etc. do not dissolve and are removed manually after all explosives have dissolved and rundown the drain.
19 - Scoop out the solid materials with a scoop into a pail partially filled with water so that the scooped materials are kept wet.
20 - Carry the pail to the burning area.
21 - Dump contents of pail into incinerator and allow to dry.
22 - Primer mixture collected in the catch basin is scrapped by the procedure described under "lead styphnate scrapping in catch basin" except because of other components in the mix besides lead styphnate.
23 - Follow above steps 15 through 21.
24 - Mixing machine inspection:
  a - All bearings for end and side play.
  b - Drum walls and blades for scratches and dents, polish off with air grinder and emery cloth.

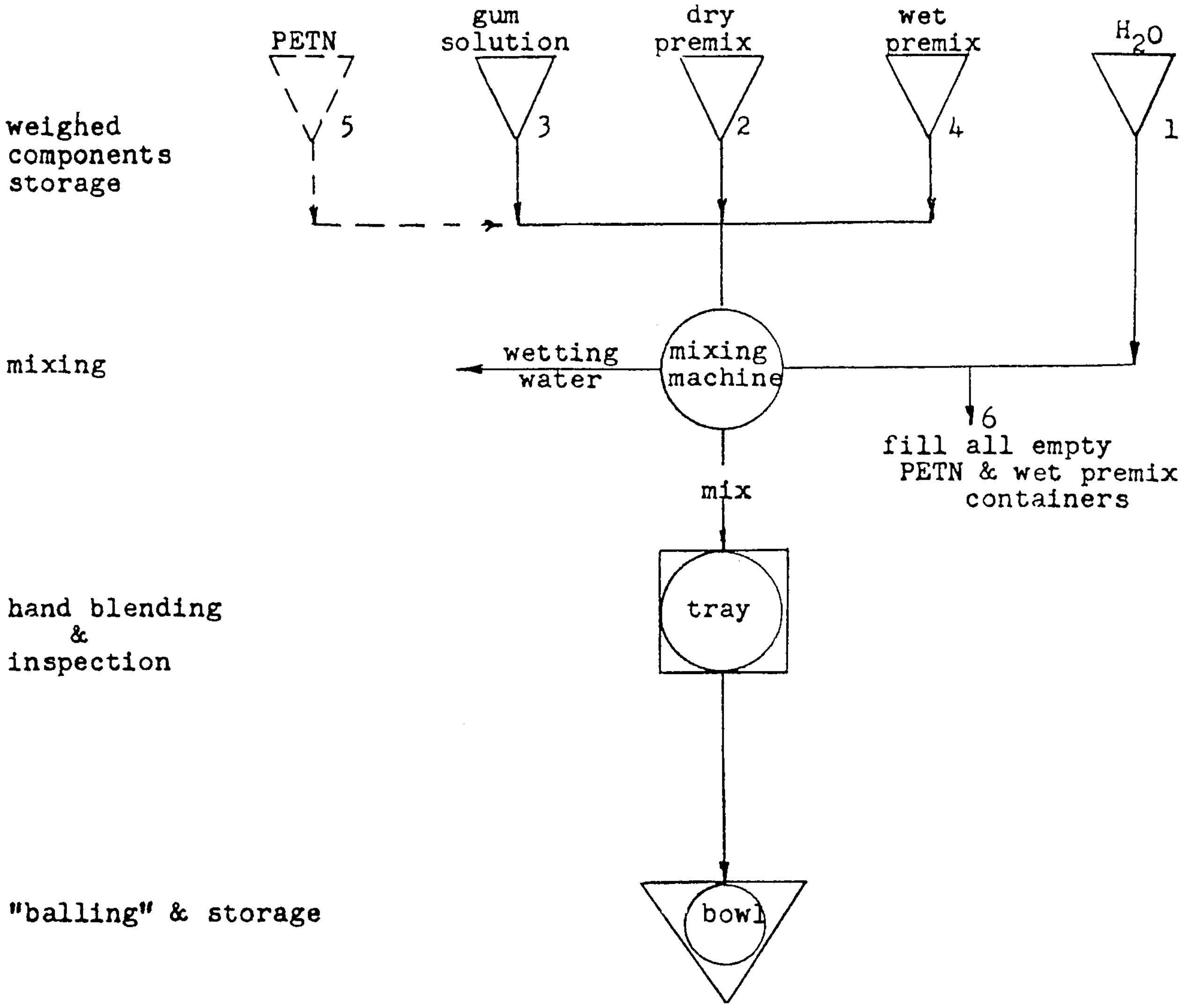
c -Rubber seals between shaft and drum for leaks and wear.
d -Chrome plating on all surfaces, worn plating to be replaced.
e - Blade concentricity with drum insides. Using 0.1 mm brass shims rotate blades and check.
f - Clearance between blades and drum bottom. Std. clearance is 0.5 - 0.6 mm. If clearance is less than 0.5 mm lower drum bottom by grinding off drum surface.

25 -Walls and floors to be wiped or mopped clean at least once a week.

26 -Explosive limits of one mixing batch or, 3500 gram, whichever is less, shall NEVER be violated.

27 -The personnel limit of two persons present in the mixing area shall NEVER be violated.

WET PRIMING MIX BLENDING PROCESS DIAGRAM



Wet Priming Mix Blending

The following procedure is taken from the ordnance procedure (DRWG. B10522388 (8 PAGES) MIX #954) applicable to the Sygma mixer. When using other type mixers, the procedure may be modified to assure consistently good quality of the final mix.

All components are brought from storage and placed on the work table in the mixing room; the gum solution is measured off into the appropriate containers.

1 - Mixing machine drum is filled with water to wet all parts.
2 - Water is dumped out and excess water in drum is sponged out.
3 - Dry, nonexplosive mix is poured into the drum so as to cover the bottom uniformly.
4 - Gum solution is poured evenly over the dry mix so that the gum does not collect in one spot.
5 - Decant the water collected over the surface of the wet mix from the rubber liner into the graduate which contained the gum solution. Discretion is left with the operator regarding the amount of water used. If the primer mix runs wet, not all water will be used to rinse the bowl. If the primer mix runs well then use all the wet premix water. Remember, the wetter the mix the more homogeneous it is.
6 - Flexing the liner bottom, break and loosen the wet premix.
7 - Deposit the wet premix over the gum solution by tipping the liner upside down.
8 - Rinse the liner with the water decanted into the graduate.
9 - Pour the water from the liner over the mix in the drum.
10 - Add the weight d portion of FETN over the wet mix.
11 - Fill the PETN and the wet mix liner with water.
12 - Check that the tray is correctly placed for the dumping of the mixing machine drum.
13 - Check the dumping mechanism.
14 - Return to control room.
15 - Check the clock and start mixing machine.
16 - Forward 2 minutes.
17 - Reverse 1 minute.
18 - Forward 2 minutes.
19 - Stop the machine, walk to the mixing room and with wet rubber spatula, scrape the primer mix down the drum walls. This is a very important step and great care is to be exercised at this point.
20 - Return to control room after immersing the dirty spatula in a water filled liner.
21 - Start mixing.
22 - Forward 3 minutes.
23 - Reverse 1 minute.
24 - Forward 3 minutes.
25 - Stop the machine and repeat steps 19 through 20.
26 - Start mixing.
27 - Forward 3 minutes.
28 - Reverse 1 minute.
30 - Forward 3 minutes, and while the machine is running dump the primer mix onto the prepare tray by inverting the mixing machine drum.

31 –The machine is run forward (upside down) 2 minutes (no reversing in upside down position).
32 – Return the machine to upright position.
33 – Stop the machine and return to the mixing room.
34 – With gloved hand remove most of the mixture left clinging to the blades and drum walls, adding this to the mix on tray.
35 – Do not rotate the machine manually to get the last bit of mix out. What is left in the drum is used in the next batch if started immediately after step 40.
36 – Move tray to the other side of sink.
37 – Fill machine with water to wet all parts.
38 – Position Buchner filter on the 4 liter flask.
39 – Turn on water aspirator.
40 – Place one filter paper and double thickness of cheese cloth on the funnel.
41 – Transfer approximately one half of the priming mix from the tray to the Buchner funnel by molding a portion with both hands and transferring a solid mass.
42 – Filter until no visual water drops are apparent (about 5 minutes); this yields a priming mix with approximately 12-15 % moisture.
43 – Shut off aspirator.
44 – Lift the cheese cloth from the funnel in a bag form and place on separate part of the tray.
45 – Replace cloth for filtering operation of the remainder of the batch.
46 – The primer mix is inspected and unmixed portions are blended by gloved hand.
47 – Thoroughly blended primer mix is made into "snowballs" and placed into bowl.
48 – Wipe edge and outside of bowl clean with wet sponge.
49 – Bowls are covered and covers labeled with white chalk, indicating mix type and batch number.
50 – Labeled bowls are placed in carrying tray and transported to priming mix magazine.
51 – Mixing machine is hosed and drained and is ready for the next batch.
52 – When mixing operations are completed the machine is disassembled and all components cleaned thoroughly with water and brush.
53 – All machine parts are wiped off with sponge soaked in diluted nitric acid.
54 – All machine parts are then rinsed with water.
55 – Machine is reassembled and put in upright position.
56 – Fill machine with water.
57 – Dry run machine to check that everything is in order.
58 – When changing the mixing formulation the machine is cleaned as per steps 52-57.
59 – Wet premix liners, PETN containers and gum solution graduates are transported to wash room.
60 – Dry mix containers are returned to dry mix room.
61 – Mixing room is cleaned up and wetted.

Notes:

a – The sequence of placing the components may be changed, if the mixing is improved; i.e. placing the explosive pre-mix into the bowl before the dry mix.

b -Since the machine is operated in both forward and reverse directions it is essential that the controls be set at zero and the machine be fully stopped before changing the motion.
c -Steps 0 through 51 inclusive complete one mixing cycle (batch).
d -Experienced operators who can judge fairly accurately the final mix moisture, sometimes omit steps 38 though 44.

Washing priming mix containers

All containers, covers, liners and handles from wet pre-mix, PETN and gum solution as well as priming mix containers and covers from charging room are transported to the wash room.

They are placed into the sink, partly filled with water, and filled with water immediately.

The washing procedure is described below:

1 - Eye protection worn during washing.
2 - Rubber gloves worn during washing.
3 - Overalls or rubber coat worn during washing.
4 - Washing area floor is wet and washed down with hose at end of shift.
5 - Empty containers are either washed immediately or immersed in water in the washing sink until cleaning time.
6 - Wash containers inside and outside with sponge under running water.
7 - Rinse containers with running water.
8 - Place containers upside down on the table to dry.
9 - Check that only clean and dry containers are taken out for reuse.

FINISHED PRIMER QUALITY CONTROL

The final quality control is performed by the ballistic laboratory, since the majority of primer tests consists in their being fired.

The following table lists types of tests and the typical frequency:

Test	**Time/Test Minutes**		**Frequency Test/Shift**	
Primer Type	**CF**	**SS**	**CF**	**SS**
Pellet weight	12		8	2
Pellet height	7		2	2
External dimensions	5		8	2
100% firing	25		2	2
Sensitivity gradient	30		1/5	1/3
Functioning (a)	25		1	1/2
Functioning (b)	15		–	I

Chapter 12

Loading and Assembly of Primers

OBJECTIVE:

- To charge and assemble primers in small quantities with a minimum expenditures, using materials readily obtained and with equipment available in school laboratory (small drill press, skill saw, jig saw, sanders, etc.).
- The required procedures to be simple (even if labor intensive), not requiring special skills.

EQUIPMENT:

1. Primer component recondition punches and plates
2. Small pliers
3. Small mix charger unit
4. Set of plates
5. Anvil seating press (automotive hydraulic jack)
6. Bench-mounted barricade

There are some important aspects to consider when selecting the equipment for laboratory operation. Namely, each different primer type to be tested, i.e. small pistol or large rifle, requires its own set of plates. Similarly, different priming mix compositions may require different thickness charging plates. To avoid unwanted costs it is recommended that the serious student considers first the one-at-a-time approach. Using a plate holding only 10 cups and not requiring a charging unit but using only a slow, simple, and careful manual charging method. Such an approach not only is inexpensive, but also provides one with the feel and practice in the required operations.

All sketches and data given are indicative only of the types and dimensions of tools used by the military and commercial manufacturers. The exact tool dimensions may vary depending on the component source and processes used.

SAFETY PROCEDURES:

All general and specific safety precautions applicable to priming mix preparation must be rigidly followed.

After all materials, equipment plates, fixtures, etc. have been assembled, make a dry run using nonexplosive material such as flour, sugar, talcum ,etc. for the mix substitute. Having satisfied yourself that the system plates mate and work properly, you may proceed to work with the priming mix.

MATERIALS:

The laboratory requirements are for only a small quantity of primer cups and anvils. Hence it is not practical to acquire the presses, dies and tooling needed to make these metallic components. The obvious option would be to purchase the necessary cups and anvils from the commercial manufacturer.

Unfortunately, this is not always possible for a variety of reasons. The only recourse then is to recover and recondition fired primers. The Berdan type primers (anvil integral with the cartridge case) require special tooling for removal from the case, which is not readily available. Since this type of primer is not commonly used in the United States it may be omitted from further discussion. The Boxer type primer (anvil in the primer cup) is fortunately quite easily removed from the cartridge case with a simple punch. The punch can be made from a common 2-3 inch long nail, as shown on XII-16.

After removal from the cartridge case, the primer is gripped by small pliers (for convenience) and the anvil is pried out with a hardened steel pin. The primer anvil is usually undamaged and requires only cleaning before reuse.

The primer cup is reconditioned in that the firing pin indent is flattened out. This is done by placing the cup, opening side up, on a steel surface and hammering out the dimple. The flattening punch is easily made by grinding a one-inch long masonry nail shown on XII-16. The working end of the punch should fit the cup inside, have the bottom surface perpendicular to the nail axis and have a rounded edge to match the cup inside radius if possible. A small bench vise or a steel plate, and an 8-oz. hammer are all the additional equipment required.

The reconditioned cups are inspected visually, then cleaned and tumbled dry before reuse. The cleaning, drying and tumbling of anvils and cups may be done by hand using a one quart mason jar and a kitchen sieve (to separate the components from tumbling material such as grits).

To assure good assembly, it is recommended that the reconditioned primer components be of the same type/source. Cups from one source and anvils from another source may not fit together properly.

PLATES:

In the industrial operations the primer charging and assembly plates are precision made on carefully controlled milling machines to assure maximum alignment of all holes, pins, fixtures, etc. Furthermore, the industrial operations require multiple plates of each type to assure a steady and high speed production flow. Such plates are made from steel, and although expensive, last for many years.

For laboratory processes, with a limited number of primers, such plates may not be necessary. Practically, one set of plates of each kind may be adequate. While it is highly advisable to make a steel template of a plate, so that all holes match properly; another solution is possible. To assure correct relation of holes, a sandwich is made of the required plates. Such "sandwich" is rigidly clamped (or screwed) together and all holes are predrilled with a small diameter drill through the hole "sandwich." Marking two of the adjacent sides of the "sandwich" assures correct plate orientation. Even if a given hole is slightly off, it will be consistent with the same hole in all plates. A simple method of marking and locating the holes in the "sandwich" is by gluing a paper copy of the pattern over the top.

Although it is desirable to make the plates from steel, they may be made from other non-sparking materials such as brass, aluminum, plastic, or hard wood. A convenient size plate for laboratory use is shown below.

There is one essential requirement. The charger plate must be made from stainless steel with reverse taper holes to allow positively free fall of the priming mix. Plates made of plastic, or wood will have holes not smooth enough for this operation. Although, a gentle tapping on the charger frame will facilitate the dropping of mix, it may cause some dispersion of the mix over the cup plate. This may be hazardous and undesirable.

CHARGING AND ASSEMBLY PROCEDURE:

1 - Place and secure (fasten) the laboratory charger onto the bench barricade.
2 - Fill the charger pan with water.
3 - Fill the cup plate with clean cups.
4 - Place the cup plate into the charger.
5 - Deposit a necessary quantity of mix on the charger plate.
6 - With a rake made from stiff-hard paper (such as post card) move the mix over the plate so that all holes are full.
7 - Push the charger bottom plate in, to allow the mix to drop into respective cups.
8 - Position the pin plate over the charger plate to push the mix out of the charger plate.
9 - Remove the cup plate with charged cups from the charger and transfer to the next operation.
10 - Return all unused mix to the storage container.
11 - With a wet sponge thoroughly clean all equipment (charger plate, charger frame and any item in contact with the mix).
12 - Wash the sponge and neutralize the wash water to decompose the priming mix
13 - Place a quantity of previously punched out foiling discs, on a wetted sponge or cloth (see note No. 4).
14 - With tweezers place the moist foiling disc over each cup.

15 - With a single brass or wooden pin pre-seat the foiling disc into the cup while pre-compressing the mix at the same time.
16 - Position the pin plate over the cup plate.
17 - Place the assembly into the hydraulic press.
18 - Actuate the press to finish seating the mix.
19 - Remove the pin plate and transfer the cup plate to the next operation.
20 - Holding the anvil shaker plate at a slight incline, sprinkle the anvils over, by slight tapping of the plate assist the anvils to fall into the holes. Most anvils fall head in first. Inspect the plate and check that all anvils are head-in (tines up).
21 - Transfer the anvils into the anvil seating plate by placing the anvil plates together and then inverting them.
22 - Remove the anvil shaker plate. The anvils should all be head-up in the anvil seating plate.
23 - Bring the cup plate and anvil plate together by "booking" them.
24 - Lift the anvil plate and check that all anvils are in the respective cups.
25 - Place the anvil seating punch plate over the cup plate and seat the assembly into the hydraulic press; actuate the press slowly so that all anvils are seated properly. See Note 3.
26 - Remove the assembly from the press, lift off the seating punch plate and visually inspect all assembled primers.
27 - Using an eye dropper, deposit a small droplet of sealing lacquer into each primer.
28 - Place a flat cardboard plate over the cup plate and invert the assembly.
29 - Lift the cup plate off, leaving the primers on the cardboard.
30 - Place another cardboard plate over the primers on the first cardboard plate and again invert the assembly; lifting the top cardboard leaves the primers anvil up on the second cardboard plate at ± 40° C.
31 - Place the cardboard with the primers into a dryer for 24-48 hours; the dry primers are ready for testing.

WET MIX PRIMER LOADING AND ASSEMBLY UNDER LABORATORY CONDITIONS

This procedure is essentially the same as applicable for the dry mix process, except for the actual mix loading (steps 6 thru 8).

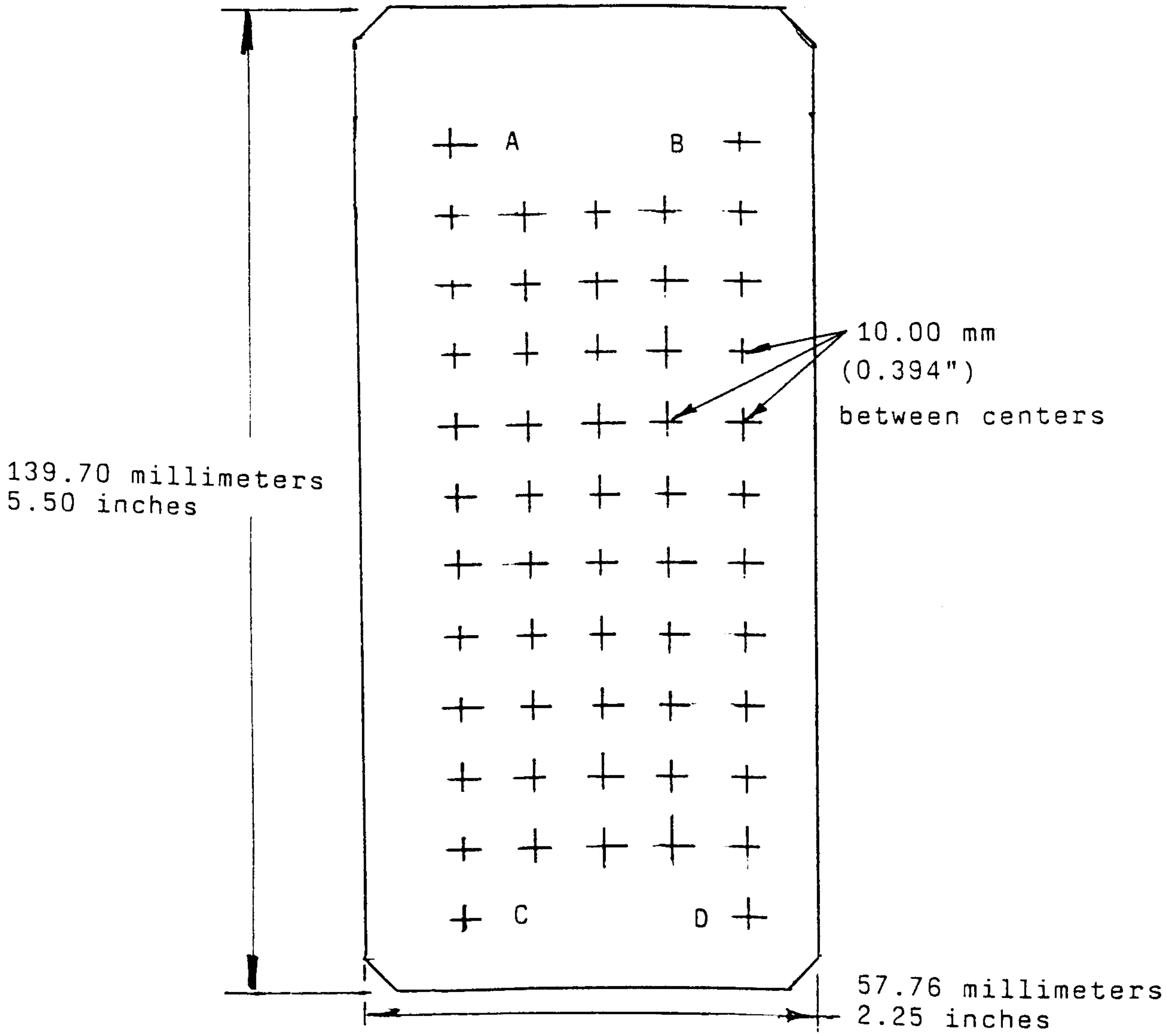
In the wet mix, loading is as follows:

6a - Position the charging plate on the charging table.
6b - With a plastic (kitchen type) spatula place a small quantity (like a small cherry) of the wet mix on the charging plate; cover the mix storage container.
6c - With the spatula spread the mix over the charging plate so that all holes are filled uniformly.
6d - Scrape the excess mix off the plate, return to the storage container and cover.
6e - Drop the spatula into a pot full of water.

7 - Invert the charger plate over the cup plate.
8 - Pivot the pin plate over the charger plate so that each pin pushes the priming mix from the hole in the charger plate into the corresponding cup in the cup plate. See Note 5.
9 - Through 31 follow the dry mix procedure.

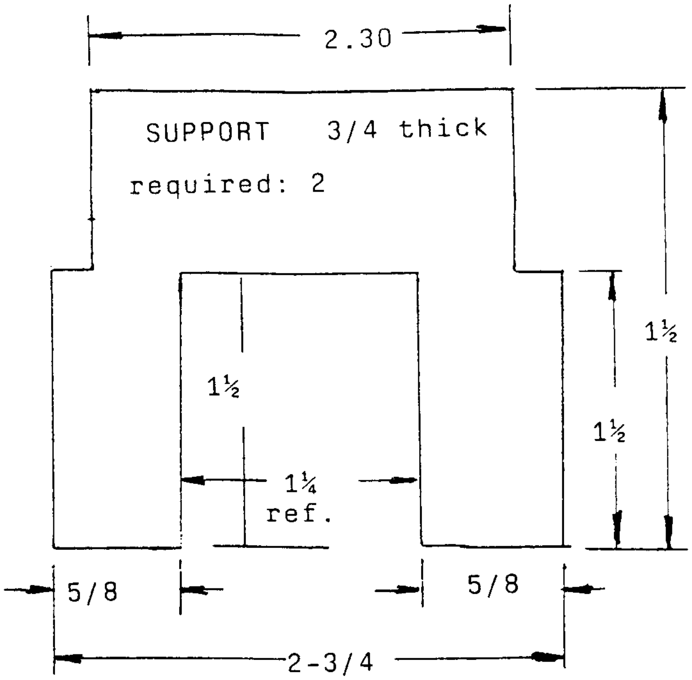
NOTES:

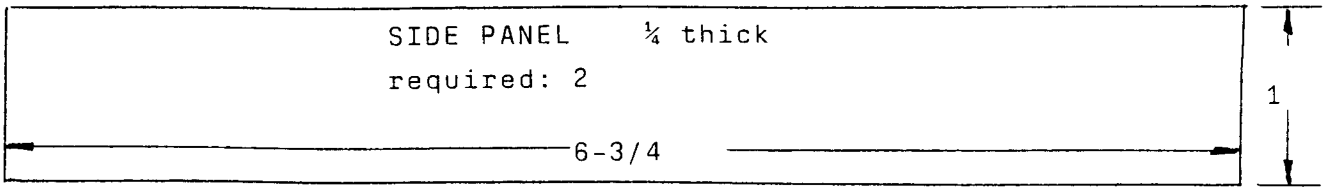
1 - If the proper charging plate is not available, it is possible to spread the dry mix directly over the cup plate being careful to fill the cups evenly and to remove all remaining mix from the cup plate.
2 - If a hydraulic press and/or the anvil seating punch plate is not available, each individual anvil may be seated in a simple reloading press equipped with single anvil seating punch.
3 - Anvils of the "U" shape (CIL-Canada, Aquila-Mexico) are easier to seat than the 3-prong anvils (Olin-WW, Remington).
4 - The least expensive, yet efficient foiling material is the newsprint paper, this material is of uniform quality, practically free, and burns well.
5 - For good housekeeping: deposit the charging plate into a pot full of water, with a wet sponge wipe off the pins in the pin plate, the charging table, and then place the sponge onto the pot with water. After all charging equipment has been washed, destroy the mix in water by neutralization as given in the respective mix composition. If more than one cup plate is to be charged with wet mix at the same time, it is not necessary to wash the charging plate and the pin plate immediately. It is sufficient to only wipe off the charging table before resuming repeated mix charging.



Template suitable for drilling holes in the primer assembly plates.
MATERIAL: 10mm (approx. 3/8") steel

SMALL CHARGER UNIT FRAME COMPONENTS





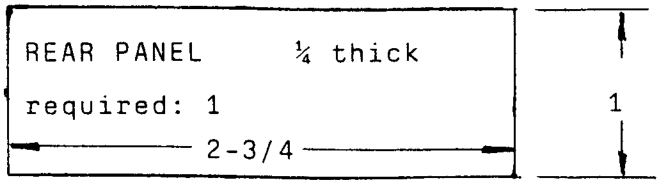


PLATE GUIDE: 3/16 dia x
7 long
required: 4

MATERIALS: Wood and adhesive

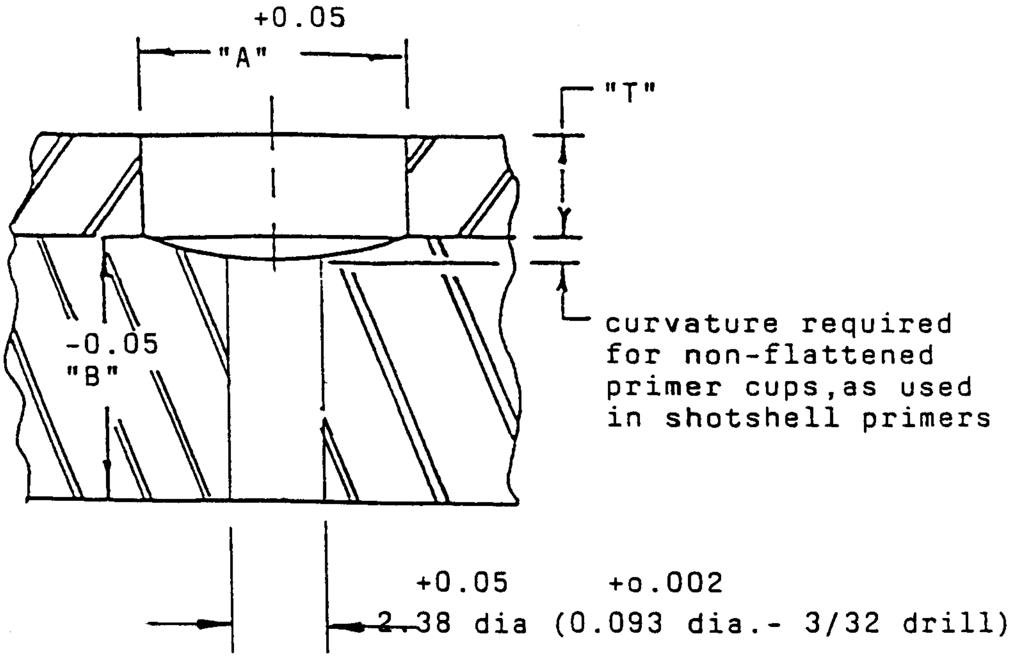
DEMINSIONS: Inches

PRIMER LOADING - ASSEMBLY - PACKING PLATES DIMENSIONS

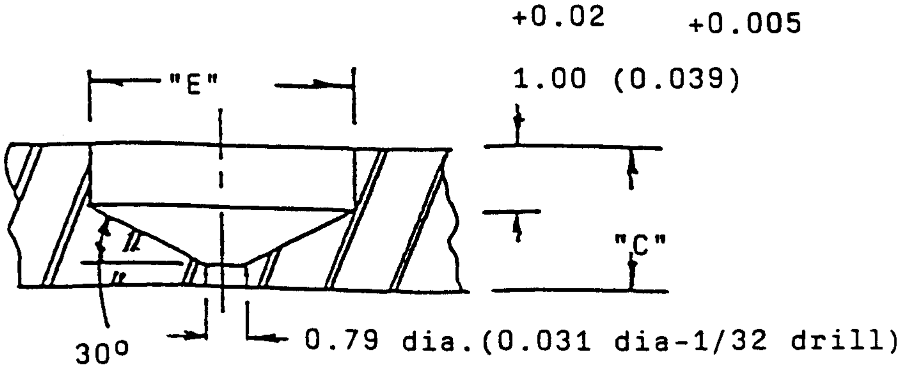
PRIMER TYPE	PISTOL		RIFLE		SHOTSHELL
	SMALL	LARGE	SMALL	LARGE	
PLATE					
MIX CHARGING					
thickness	1,27 (0.050)	1.27 (0.050)	1.52 (0.060)	1.42 (0.056)	1.42 (0.056)
top dia.	3.76 (0.148)	3.76 (0.148)	3.25 (0.128)	3.76 (0.148)	3.76 (0.148)
bottom dia. (rubbing side)	3.81 (0.150)	3.81 (0.150)	3.30 (0.130)	3.81 (0.150)	3.81 (0.150)
pellet ejection pin dia.	0.25 (0.010) smaller than the respective plate rubbing side hole diameter				
CUP PLATE "T"	2.41 (0.095)	2.79 (0.110)	2.41 (0.095)	3.05 (0.120)	3.70 (0.120)
"A"	4.55 (0.179)	5.28 (0.208)	4.70 (0.185)	5.70 0.224)	5.70 (0.224)
"B"	5.51 (0.217)	5.13 (0.202)	5.51 (0.217)	4.87 (0.192)	4.24 (0.167)
ANVIL SHAKER PLATE					
"C"	2.10 (0.083)	2.13 (0.084)	2.03 (0.080)	2.13 (0.084)	see drawing
"E"	4.04 (0.159)	4.50 (0.177)	4.00 (0.157)	4.50 (0.177)	
ANVIL SEATING PLATE					
"C"	2.25 (0.088)	4.76 (0.169)	3.17 (0.125)	4.76 (0.187)	
"E"	3.97 (0.156)	4.60 (0.181)	4.00 (0.157)	4.60 (0.181)	

Note: 1 - Dimensions are in millimeters (inches).
2 - Dimensions are for a typical wet priming mix; final plate thickness depends on the specific mix composition.
3 - Tolerances of charging plate dimensions are 0.01 (0.0005).

PRIMER CUP PLATE



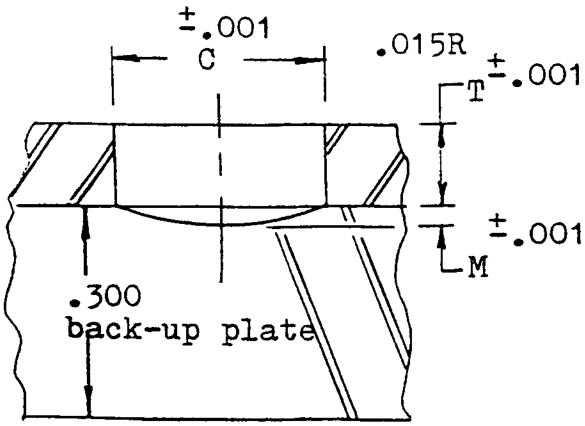
PRIMER ANVIL PLATE



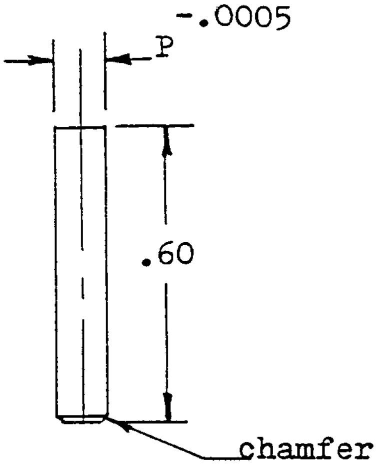
Note: 1 – Plate material - 303 stainless steel.
2 – Dimensions are in millimeters (inches).

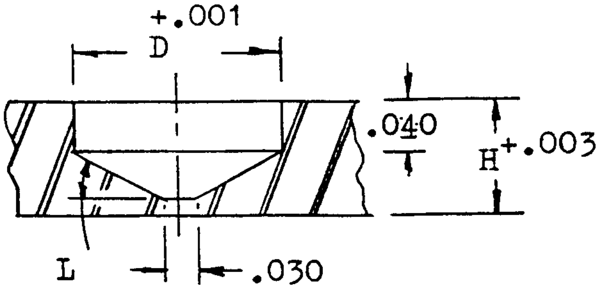
Dimensions of Cavities in Plates Used for Primer Charging and Assembly

Primer cup plate



knock-out plate pin





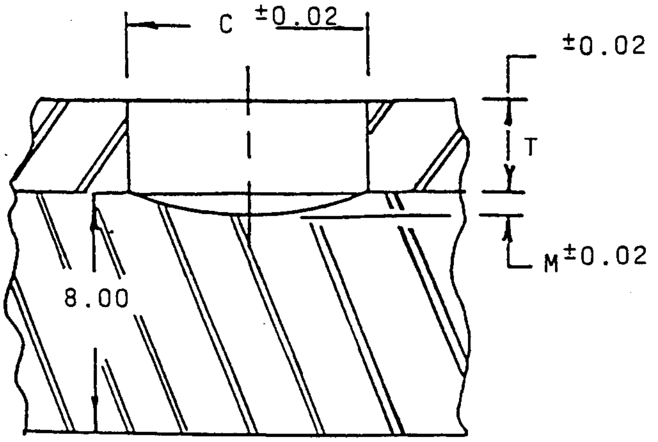
anvil shaker plate

DIMENSION (inches)			**PRIMER**		
	SMALL PISTOL	**SMALL RIFLE**	**LARGE PISTOL**	**LARGE RIFLE**	**SHOTSHELL 209 TYPE**
Primer cup plate					
C	.179	.179		.214	.214
T	.085	.085		.090	.135
M	.010	.010		.012	.015
Knock-out plate pin					
P	.097			.130	.120
Anvil shaker plate					
D	.155		.177	.196	-
H	.086		.086	.084	-
L	33°		33°	29°	-

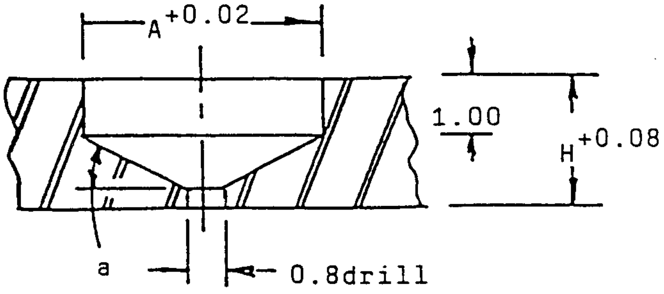
Note: For flat bottom primer cups the dimension M is zero.

Dimensions of Cavities in Plates Used for Primer Charging and Assembly

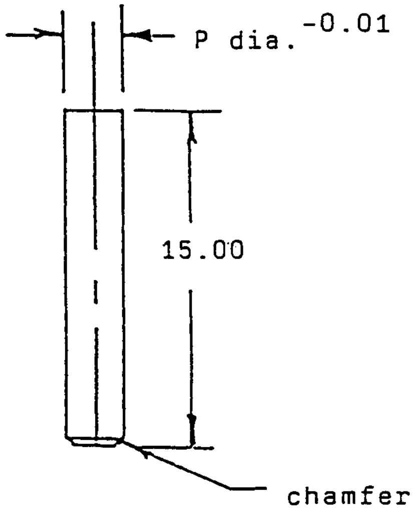
Primer cup plate



anvil shaker plate



knock-out plate pin

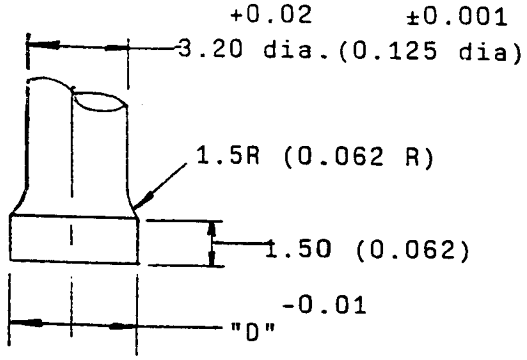


PRIMER TYPE		**SMALL PISTOL**	**SMALL RIFLE**	**LARGE RIFLE**	**SHOTSHELL 209 TYPE**
Primer cup plate					
	C	4.55	4.55	5.44	5.44
	T	2.16	2.16	2.29	3.43
	M	0.25	0.25	0.30	0.38
Anvil shaker plate	A	4.00	4.00	5.00	-
	H	2.18	2.03	2.13	-
	a	33°	33°	30°	-
Knock-out plate pin dia.	P	2.46	2.46	3.30	3.05

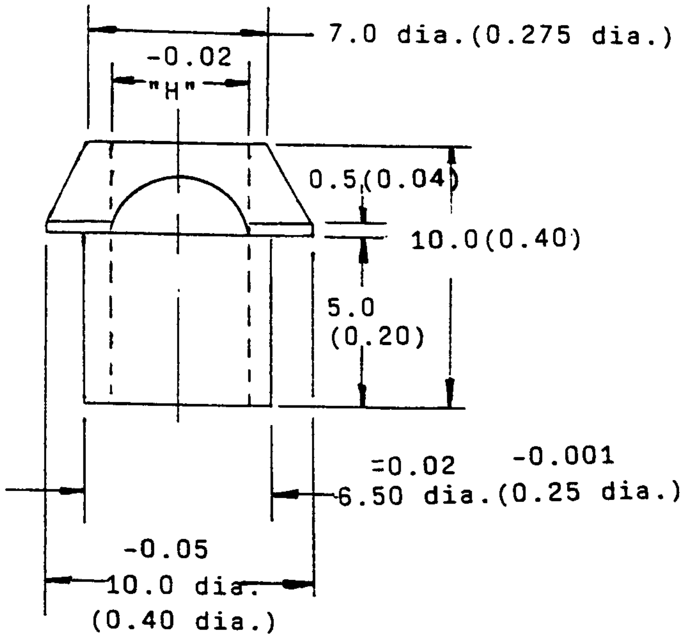
Note: All dimensions are in millimeters.

PRIMER FOILING PUNCH AND DIE

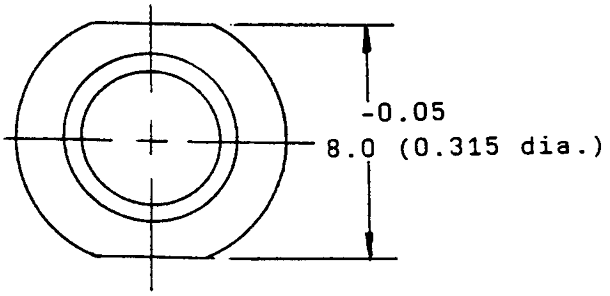
PUNCH
material: drill rod
heat treat: Rc 58-60
flash chrome plate working surfaces
0.01 thick



DIE
material: AISI W 1
harden and draw
flash chrome plate working
surfaces 0.01 thick



DIAMETER	-0.01 "D"	+0.02 "H"
PRIMER TYPE		
small primer	3.63 (.1430)	3.63
large rifle	4.47 (0.1760)	4.47
shotshell barrery cup	5.13 (0.202)	5.12 (0.202)



Notes: 1 - dimensions are in millimeters (inches)
2 - sliding fit for punch "D" in die "H"
3 - for smooth finish components are made on hand screw machine

PRIMER FOILING PUNCH AND DIE

MATERIAL: AISI W1
HEAT TREAT: Rc 58-60
FINISH: Flash chromium plate 0.01 mm (0.0005 inch) thick, approx.
10 mm (0.4 inch) of working end

DIMENSIONS **PRIMER TYPE**	**PUNCH** **DIAMETER "D"**	**DIE** **DIAMETER "H"**
Small pistol	3.63 - 0.01 (0.1430 - 0.0005)	3.63 + 0.02 (0.1430 + 0.0005)
Large Rifle	4.46 - 0.01 (0.1760 - 0.0005)	4.47 + 0.01 (0.176 + 0.0005)
Shotshell		
Primer cup	same as large rifle primer	
Battery cup	5.12 – 0.01 (0.201 – 0.0005)	5.12 + 0.01 (0.201 +0.0005

Dimensions in millimeters (inches)

ANVIL SEATING PIN

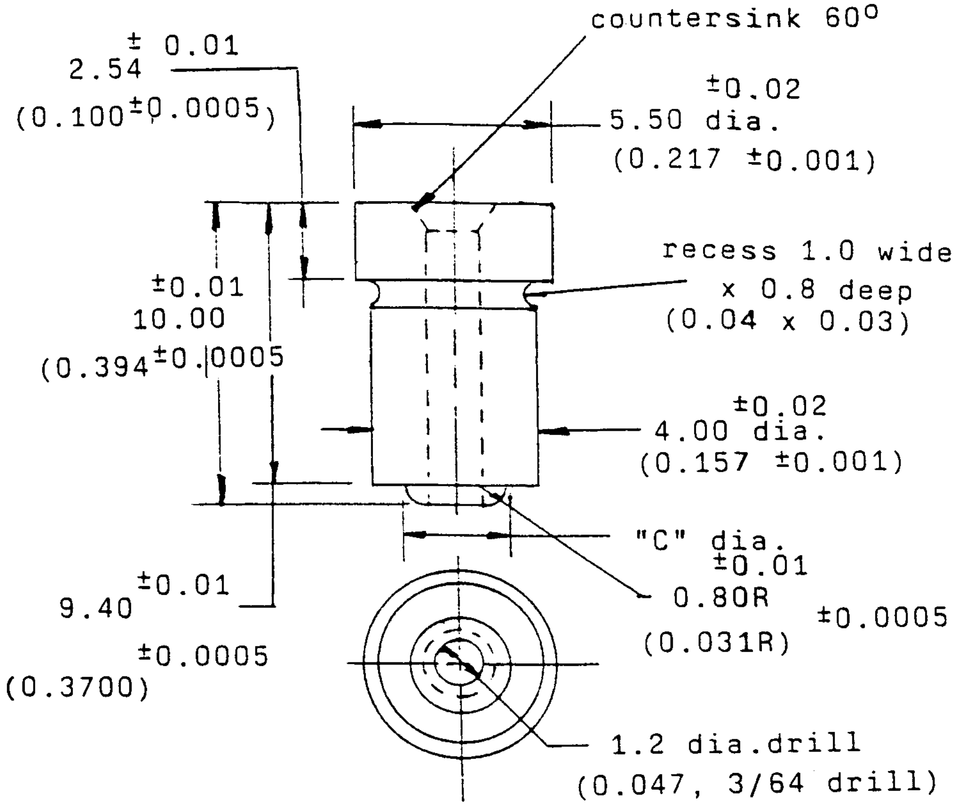


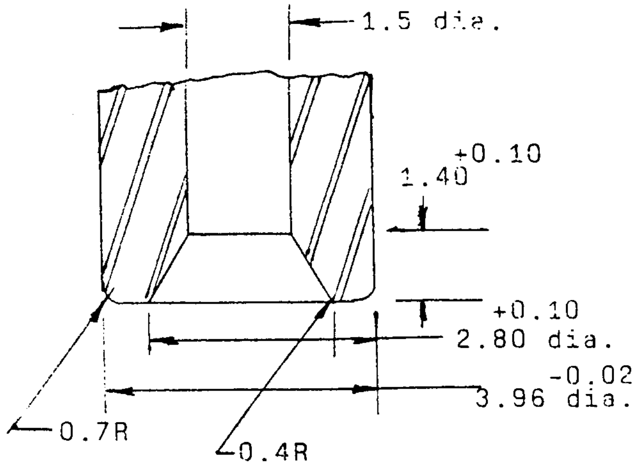
FIG. 12-16

DIMENSIONS	**"C"**	
	MM	**Inches**
PRIMER TYPE	±0.02	±0.001
Small pistol	1.80	0.710
	±0.02	±0.001
Large Rifle	2.54	0.100

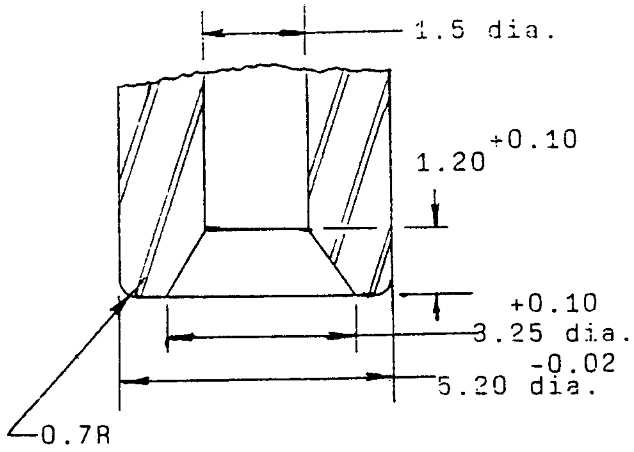
Notes: 1 – Pin top surface ground perpendicular to axis.
2 – Pin is made on hand screw machine for smooth finish.
3 – Pin length is critical for uniform anvil seating depth when used on press fixture.

ASSEMBLY PUNCHES FOR BATTERY CUP TYPE SHOTSHELL PRIMERS

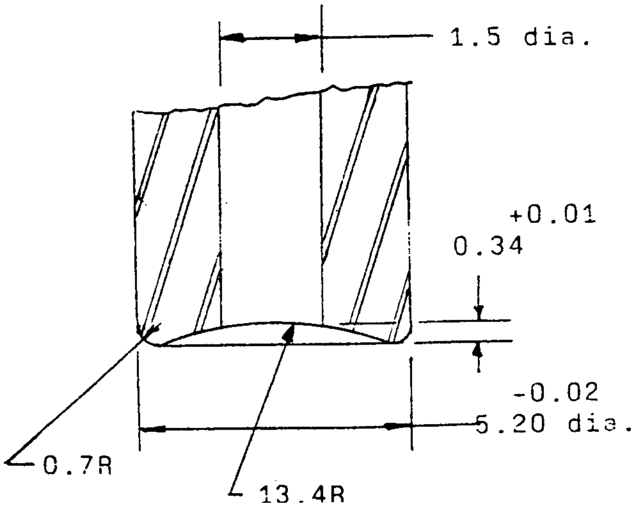
ANVIL SEATING (1ST)



ANVIL SEATING (FINAL)

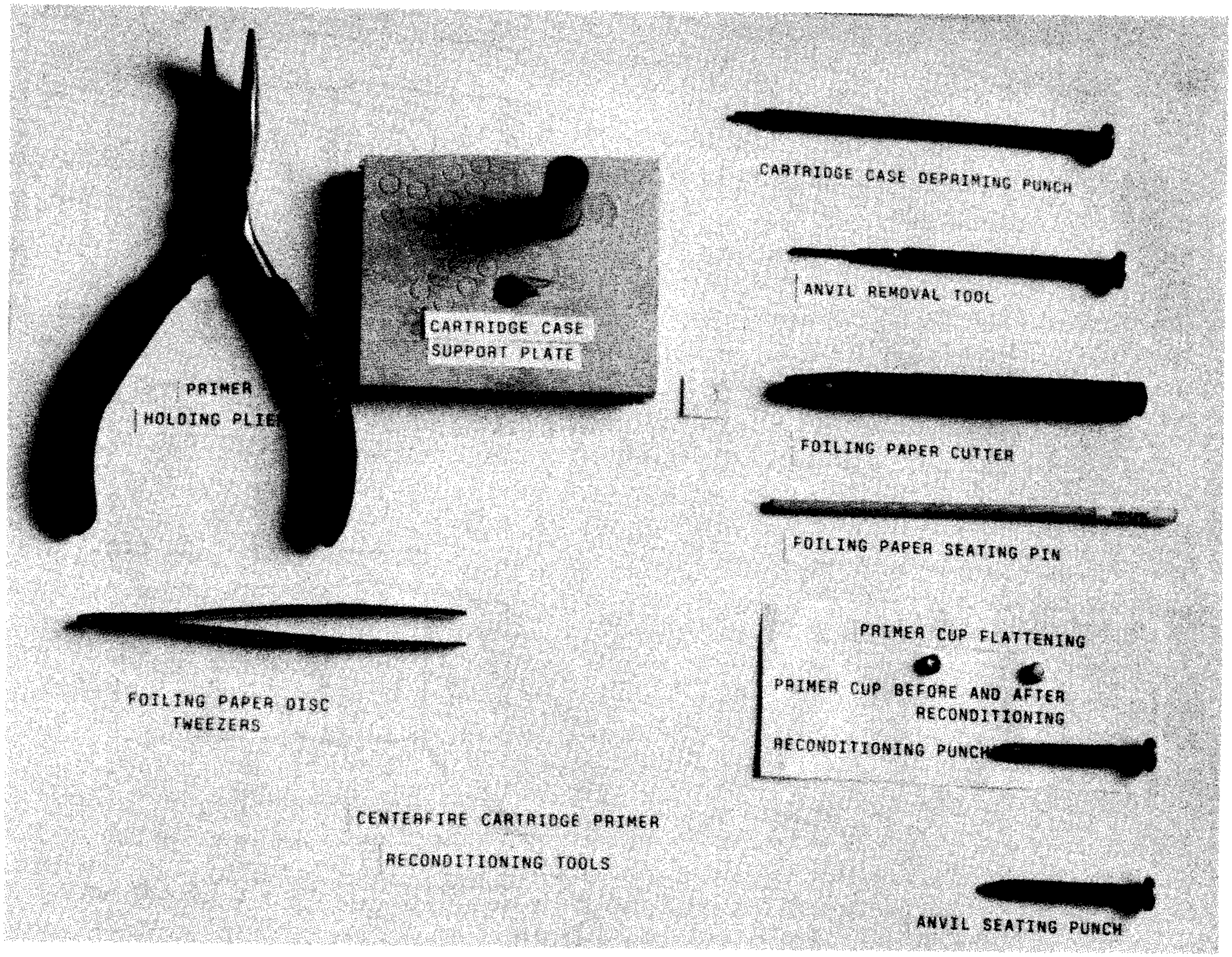


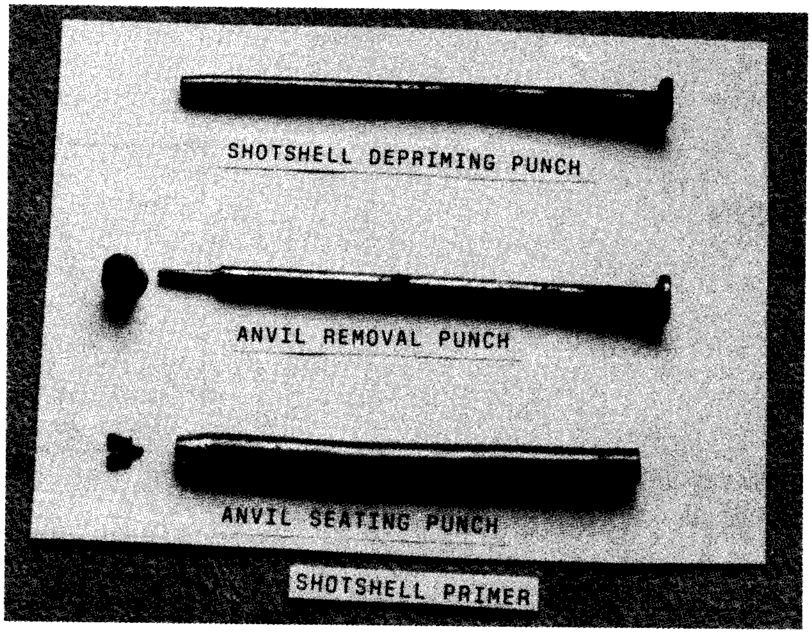
PRIMER CUP SEATING



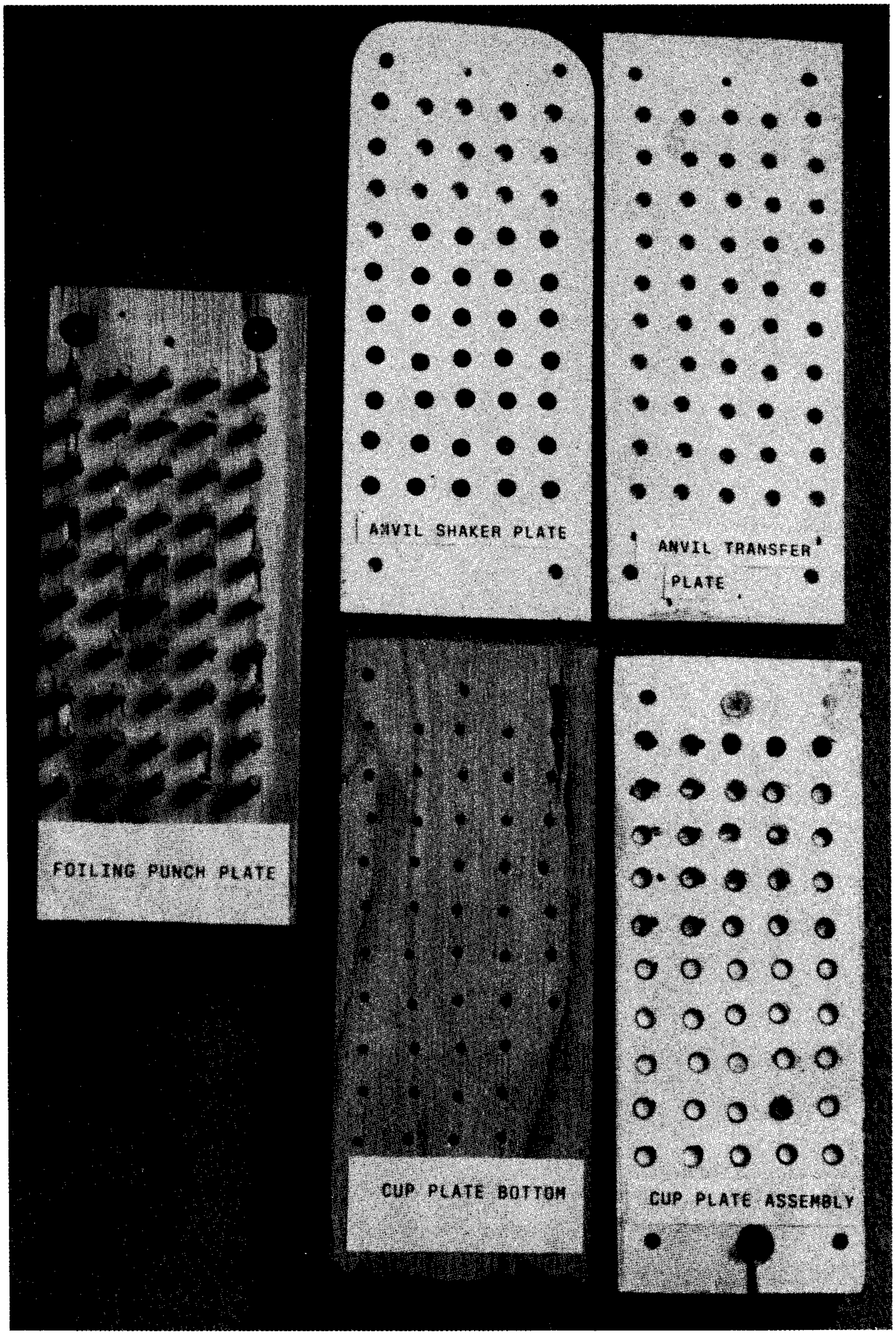
Notes: 1 – Dimensions are in millimeters.
2 – Do not scale drawings.

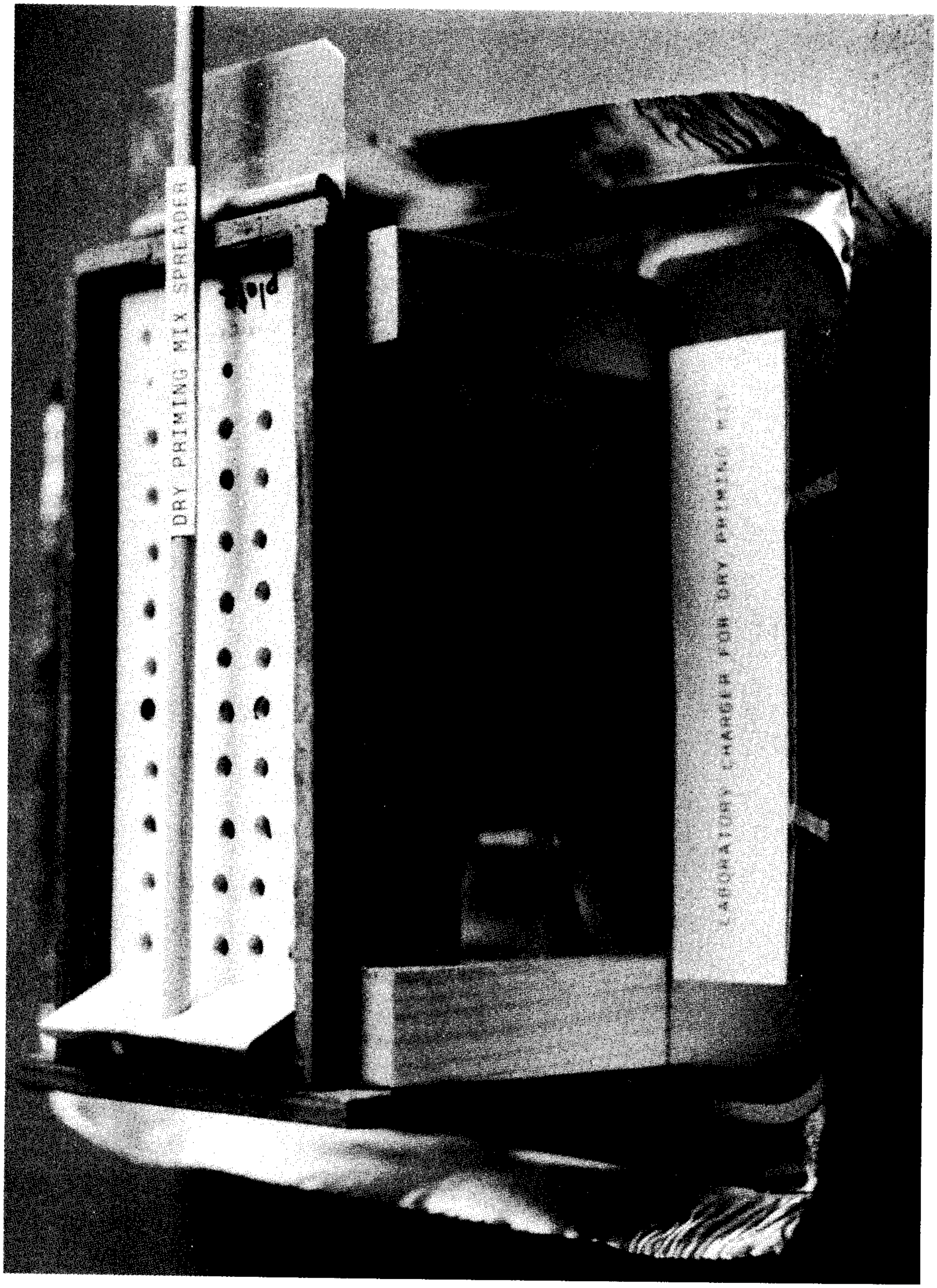
PRIMER RECONDITIONING TOOLS





PRIMER PLATES





LABORATORY CHARGER FOR DRY PRIMING MIX

It is appropriate at this time to mention briefly the quality control and testing of primers. During the manufacture in the arsenals and/or large commercial plants, the primers as all components of small arms ammunition, are subject to rigorous and periodic visual inspection, gauging and testing. This includes not only the finished product, but also metallic components, chemical semi-products, etc. Since this text is concerned primarily with the laboratory preparation of priming mixes and their performance, such rigorous testing as mentioned above may be dispensed with.

The testing of laboratory prepared and assembled primers, particularly with reconditioned cups and anvils, is tested for ignition of propellant in a loaded cartridge only.

The technique of priming and reloading of small arms cartridges has been amply described in detail in many ammunition catalogues, periodicals, and books. These publications are readily available through the public libraries. Hence, it would be redundant to do it in this text.

Appendix A

Abbreviations, Bibliography and Production Data

ABBREVIATIONS USED IN TEXT

DDNP	Diazodinitrophenol
HE	High Explosive
LAP	Loading-Assembly-Packing operations
LS	Lead Styphnate
MF	Mercury Fulminate
NC	Nitrocellulose (gun cotton)
NG	Nitroglycerine
PETN	Pentaerythrite Tetranitrate
T	Tetracene
TNR	Trinitroresorcinol
CTG	Cartridge
CF	Center fire (cartridge or primer)
RF	Rimfire cartridge
SS	Shotshell
LR	Large rifle primer
LP	Large pistol primer
SP	Small pistol primer
SR	Small rifle primer

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Following are some data on typical manpower and material requirements for a primer manufacturing facility. The values are based on general type conditions and will vary slightly from one specific plant to another. However, they are good, reliable starting point for economic evaluations of projected facilities.

ASSUMPTIONS:

Production: capacity - 40 million shotshell primers per year
40 million large rifle primers per year

Production time*) 1 work day = 8 hours
1 work week = 40 hours
I work month = 165 hours
1 work shift/year = 2000 hours

*) These values are based on experience of different plants, they include such variants as holidays, down time, etc. The actual value of operator hours per year are 1980 hrs. for first shift; 1940 hours for 2nd shift, and 1920 hours for 3rd shift, However, for general work the above assumptions may be used.

Priming mix requirements:
40,000,000 shotshell primers x 0.060 g mix = 2400 Kg/year = 200 Kg/mos
40,000,000 large rifle primers x 0.042 g mix 1680 Kg/year = 140 Kg/mos

340 Kg/mos

340 Kg/ mos at 3000 g/batch = 116 batches
of the 340 Kg the component breakdown is:

Material	**% in mix, approx.**	**Kg/month required**
Lead styphnate	40	136.0
Tetracene	4	13.6
PETN, washed and weighed	5	17.0
Dry mix	51	173,4

Production rates required to meet preceeding assumptions: See next page.

PRODUCTION RATE

MATERIAL and/or OPERATION		TOTAL HOURS/MONTH
TNR	735 g TNR to make 1174 grams Lead Styphnate 85 Kg id 136 Kg Lead Styphate since each lot of Resorcinol yields 26 Kg TNR; approximately 4 batches per month are needed (4 x 2 days x 8 hrs/day)	64
Lead Styphnate	1175 g/pail x 6 pails/day = 7050 g/day; hence 20 days are needed	160
Tetracene	1000 g/pail x 5 pails/batch; 3 batches/months	24
PETN	850 g/day x 20 days = 1 hour x 20 (washed with a funnel and flask)	20
Wet pre-mix	6 batches/day (the same as Lead Styphnate)	160
Dry mix	3 batches/hour x 8 hours = 24 batches/day	40
Final mix	116 mixes/month at 6 mixes/day = 19.2 days	160
Cleaning outside catch basins		12
		640

Note: with exception of the nitrator and centrifuge for TNR, and the blender for final mixing, all equipment is off-the-shelf laboratory material available through variety of supply catalogues.

The personnel required for the above production consists of:

Chemist - 1 (Supervision, office work, quality control, burning tests)

Operators - 4
- a – TNR preparation
- b – Lead styphnate and washing
- c – Tetracene and dry pre-mix
- d – Wet pre-mix final mix

PRIMER MANUFACTURING LINE

DIRECT LABOR REQUIREMENTS

Primer type	**SS**	**SS & RF**	**SS & RF & CF**
Production rate 100/min.	4	4 5	4 5 4
Metallics manufacture			
Foreman	1	1	1
Adjustor *	1	2	
Operator	3	5	7
Material handler	1	1	2
Explosive manufacture			
chemist	1	1	1
operator		1	2
LAP			
Foreman	1	1	1
Adjustor	*	1	2
Operator	3	4	6
Material handler	1	1	1

* Because of the small volume operation, the adjustor functions are performed by the foreman.

MATERIAL REQUIREMENTS

Item	**grams to produce 1000 g product**
TNR	1000
Resorcinol	600
Sulfuric acid	6000
Nitric acid	2500
Sodium nitrite	
Lead Styphnate	1000
TNR	620
NaOH	200
Lead nitrate	1060
Acetic acid	205
Tetracene	1000
Aminate	1500
Sodium nitrite	1700
Acetic acid	665
Priming mix, 209 shotshell	1000
Lead styphnate	400
Tetracene	40
Barium nitrate	300
Antimony sulfite	150
Aluminum powder	60
PETN	50
Gum solution	10

MATERIAL REQUIREMENTS

Item	**material**	**grams/1000 pcs.**
209 primer cup	brass 70/30	220
209 primer anvil	brass 70/30	215
209 battery cup	brass 70/30	600
LR primer cup	brass 70/30	225
LR anvil	brass 70/30	75
Foiling paper	paper	7
209 primer	priming mix	55
LR primer	priming mix	40

Appendix B

Loading, Assembly and Packing Wet Priming Mix

The third major phase of primer production, after manufacture of metallic components and priming composition, is the primer loading with mix, assembly with anvil and/or battery cup, and packing into appropriate containers.

Because of the small individual size and large volume requirements, the small arms primer is well suited for plate loading.

The classic method of using plates in loading ammunition has been known for over two centuries, both in technical literature and industrial practice. The following description of plate loading and assembly of primers is based on the present day practices and experiences in this field.

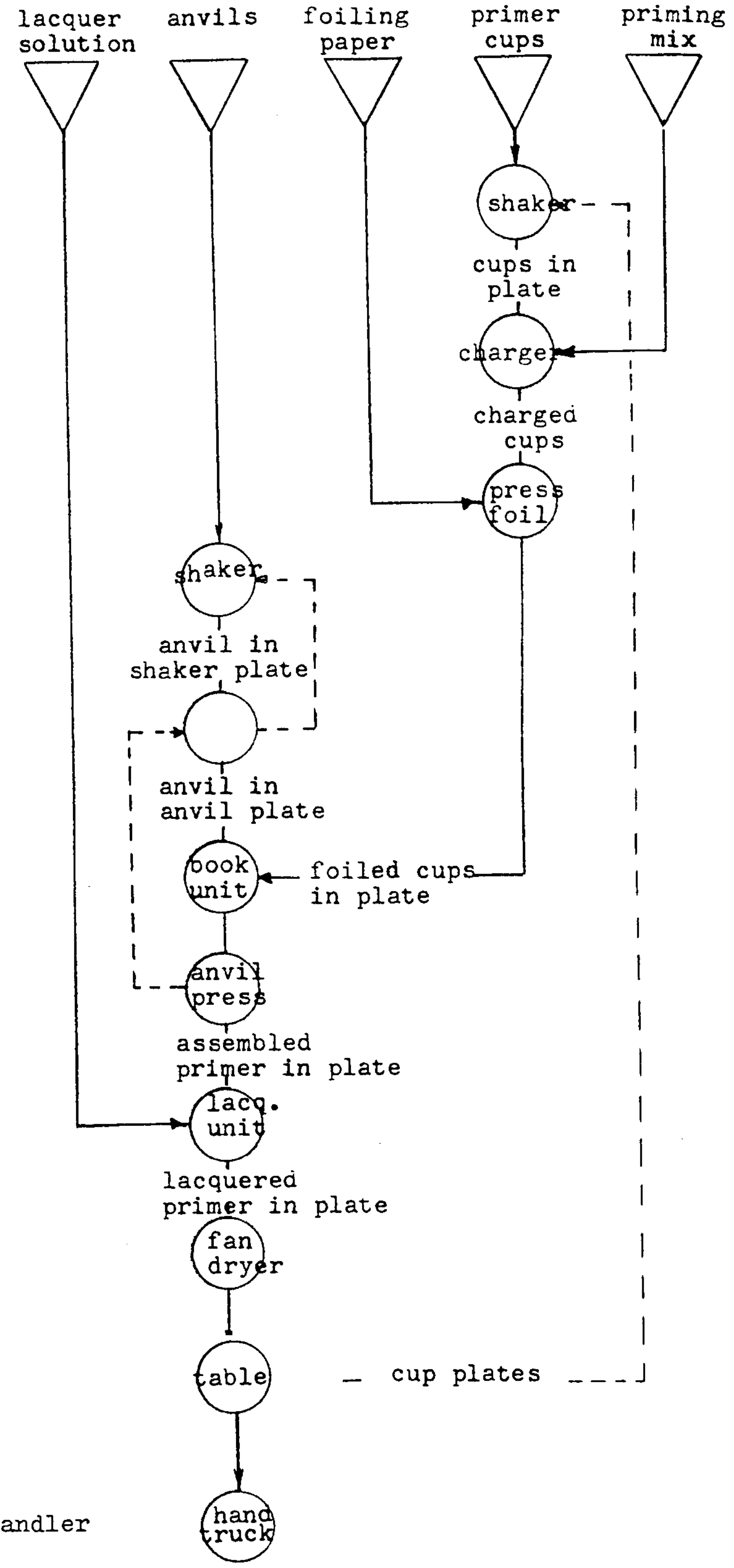
As the name implies, plate loading uses plates, each containing a number of identical cavities, ranging in number from 500 to 1000, for a given component and/or operation. A complete set of plates is made from a jig plate to assure that all cavities in all plates match perfectly.

Using a plate, the operator, instead of performing each operation, such as cup orienting, pellet metering and inserting, etc. on every individual component, performs each operation on a large number of identical components. By this method, a given operation, even if requiring one minute to perform, will yield 500 - 1000 pieces per minute. To duplicate such production rate without plates requires a complex and expensive continuous motion assembly machine. Following text describes the plate loading method. For clarity's sake, each operation is described and illustrated separately. In actual practice some operations may be eliminated. For example, commercial primers are generally not lacquered. The use of integral anvil-battery cup also eliminates several operations. The use of automated cup feeders, use of fiber optics in inspection operations, automatic conveyor belts, etc. reduce further and eliminate the work load of the individual operators.

However, each improvement in efficiency has its cost in capital investment, space and energy requirements. It is not our objective to design the ultimate primer LAP line, but to show the basis for a simple and reliable system, using the maximum of "off-the-shelf" industrial elements. The system is optimum for a production capacity of 40 - 50 million units/1 shift-year with 4 operators. Smaller production still requires the same equipment. Reducing the number of operators to two or three, reduces the efficiency due to increased legwork and extra motions

CHARGING AND ASSEMBLY

Operation	operator number
material from stores	mat'l handler
shake cups into plates	2
charge cups with mix	1
foiling	3
shake anvils into shaker plates	3
transfer anvil to anvil plate	3
book cups and anvils	3
anvil seating	3
lacquering	2
drying lacquer	2
finisher primer to storage pad transfer	4
transferring tray carriers to	mat'l handler



Operation 1.

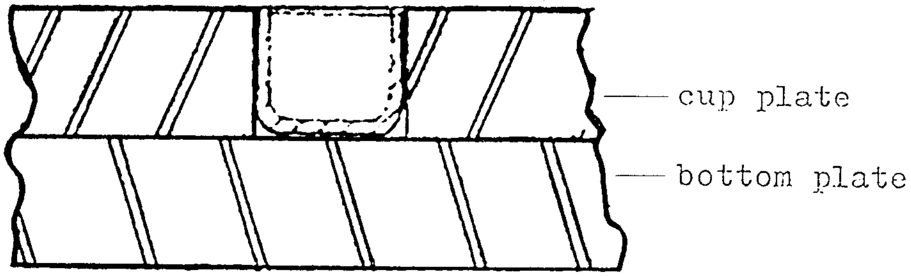
Primer cups are dropped by hand over the plates vibrating on the shaker frame. The plates are positioned on an incline so that the cups travel gently over the plate surface until finding an open cavity. The cups fall into the cavities face up. Occasionally a cup will fall into the cavity bottom up. It is picked up by a wooden pointed pencil-like instrument with tacky wax on the tip, and replaced face up in the cavity.

Cups which miss the open cavities fall off the lower edge of the plate into the shaker pan to be again scooped up and dropped over the plate.

The maximum efficiency is achieved by adjusting the shaker frequency and amplitude, and the plate inclination for a given cup type.

To assure an uninterrupted flow of components, approximately 25 plates are needed for each cup shaker. Skilled operators will fill two 1000 hole-plates per minute.

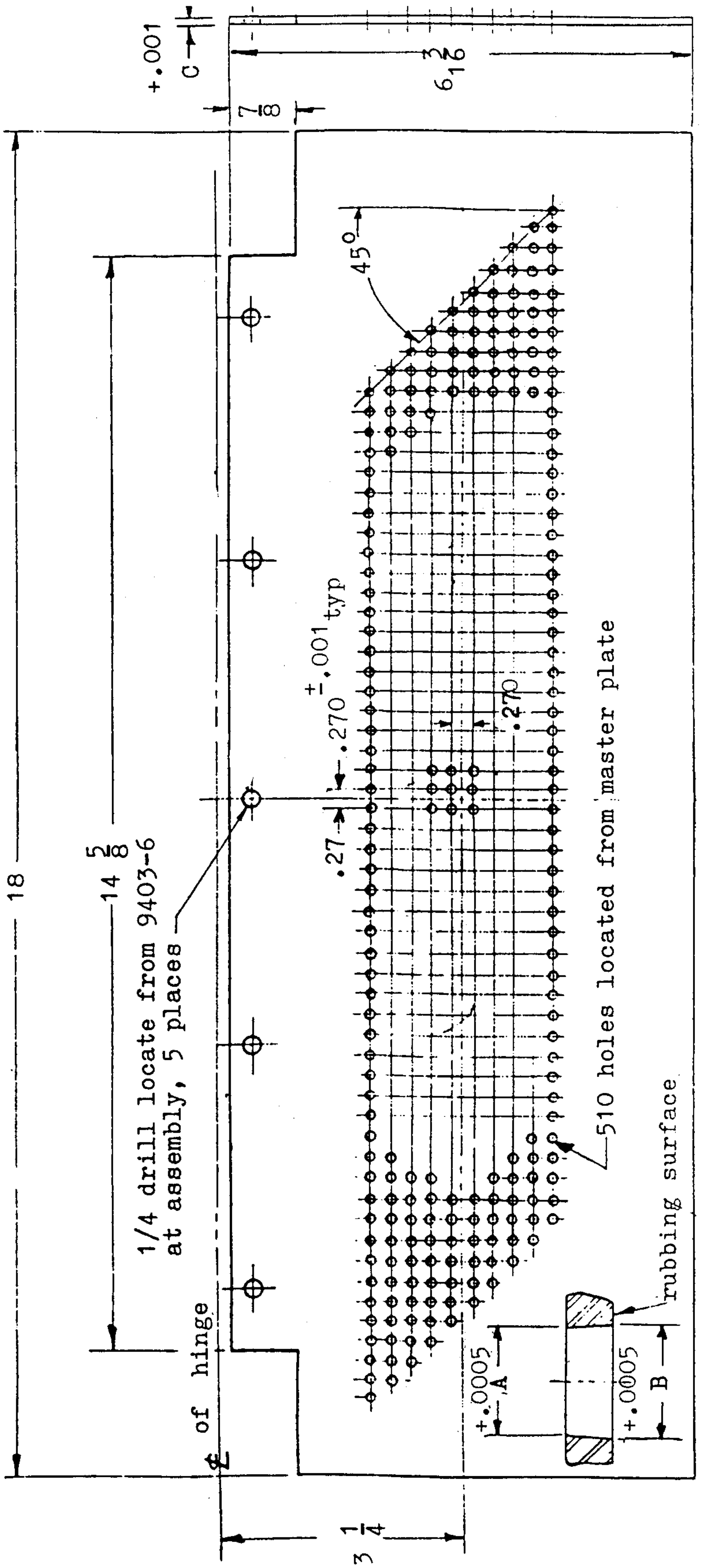
The filled plates are placed on a conveyor.



Part 9402-1

matl: stainless steel
303
finish: grind top &
bottom surfaces

tolerances:
on decimals ± .005
on fractions ± 1/64
angles ± 0° 30'



enlarged view of hole

primer type	A	B	C
.30 cal.	.148	.150	.055
7.62MM (34 & 36)	.135	.137	.075
.45 cal.	.148	.150	.049
5.56 MM	.128	.130	.060

Operation 2.

A batch of priming mix in a rubber container is brought from storage and placed on the charging table. The charging plate is pivoted onto the table (and sometimes is clamped by a magnet to the table surface). The priming mix is deposited over the charging plate and manually rubbed into the cavities in the charging plate. Excess priming mix is scraped off to the side of the table, ready for reuse.

The plate full of cups, from previous operation, is picked from the conveyor and placed into the slot in the charging table. The charger plate is then rotated over the cup plate so that each primer mix pellet in the plate is located directly over the respective cup in the cup plate. A pin plate containing a number of pins to match the holes in the charger plate is brought over and down on the charger plate. The pins knock out the primer mix pellets into the primer cups.

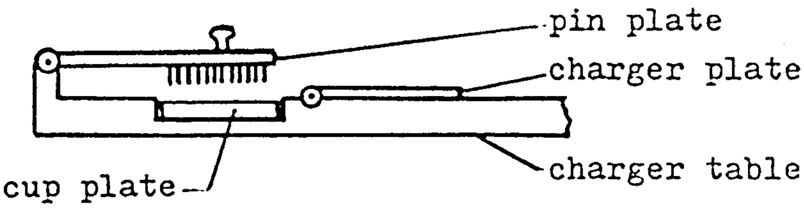
The pin plate is then rotated back to its starting position, the empty charger plate, is brought back to rest on the charge table, and the cup plate with charged cups is picked up by the charging operator.

The cups are visually inspected for correct charge and the full plate is placed in the foiling machine.

One operator with one charging table processes about one plate per minute. Some new charging tables have the holes in the charging plate staggered so that each rotation of the charger plate indexes the plate and aligns a new set of holes over the cups. Thus a plate with 3 sets of holes will charge with mix 3 cup plates with only one priming mix rubbing in.

The production rate depends on the number of cavities in each plate and the operator skill.

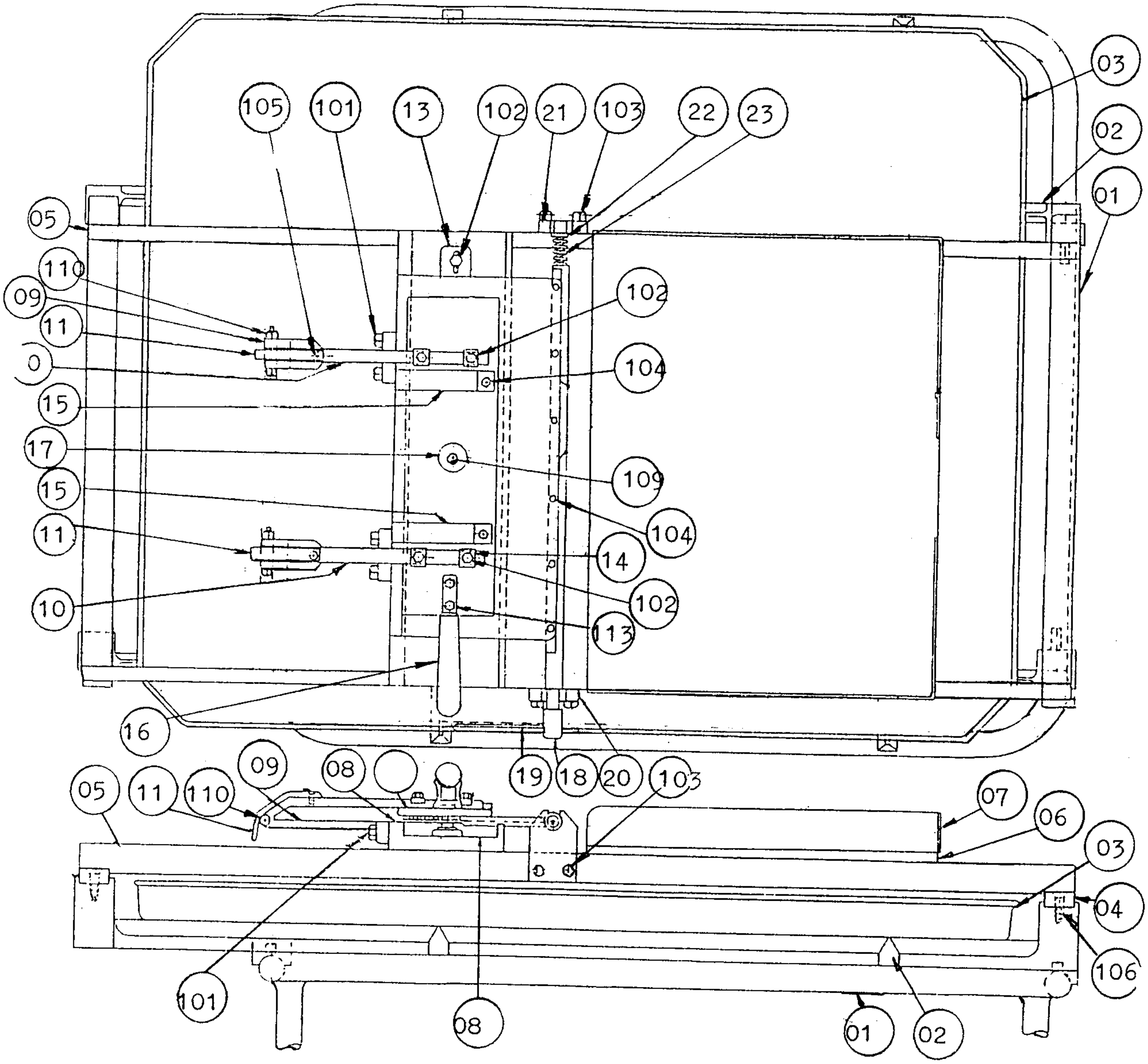
Operation 3.



A typical charging table consists of a bronze or stainless steel frame mounting a surface plate, charging plate and a hinged pin plate. The frame is surrounded or mounted over a water filled pan.

TYPICAL WET PRIMING MIX CHARGING UNIT

(Top View)



Operation 3.

The cup plate, full of cups with priming mix pellet in each, is placed in the foiling machine. This machine has the upper die portion equipped with a row of pins matching a series of holes in the cup plate. A strip of paper is fed perpendicular to the plate motion, over the cup plate. The pins descend, blank out a small disc of paper and push it into the primer cup. The priming mix is compressed with a force of about 45 Kg by the punch and is spread evenly inside the cup. The foil paper not only prevents the mix from sticking to the punch tip, but also acts as a barrier preventing the dry mix (of assembled primer) from falling out. After one row of cups is foiled, the plate is indexed and a new row lines up under the punches. The machine stops automatically after the last row has been foiled.

To assure better adhesion of the foil to the cup mix and to keep the paper from sticking to the punch, the paper roll sometimes passes through a bath of alcohol base solvent.

In large scale operations, the cup plates, after charging, are placed on a conveyor. Another operator then feed the plates to a battery of 4 foiling machines.

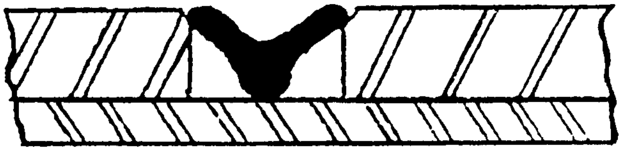
The foiled cup plates are removed from the machine, visually inspected for defects (such as no foil, incomplete foil, double foil, etc.) and placed on conveyor which brings them to the anvil assembly.

Operation 4.

The anvils, like the cups, are oriented by dropping into a plate. These plates, with corresponding cavities, are positioned on an inclined frame in the anvil shaker.

As the anvils slowly move down the anvil shaker plate, they drop point down into the respective cavities. Only an occasional anvil drops into the cavity point up. Such anvil is reversed by use of the same tool as used on the cups.

However, for primer assembly, all anvils must be in a plate with the point up. This reversing is done in the next operation.



anvil shaker plate

Operation 5.

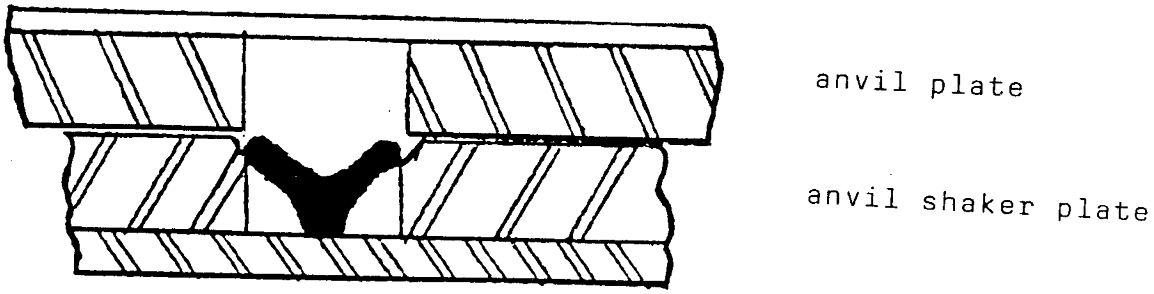
A full anvil shaker plate is removed from the shaker frame and placed on a flat surface.

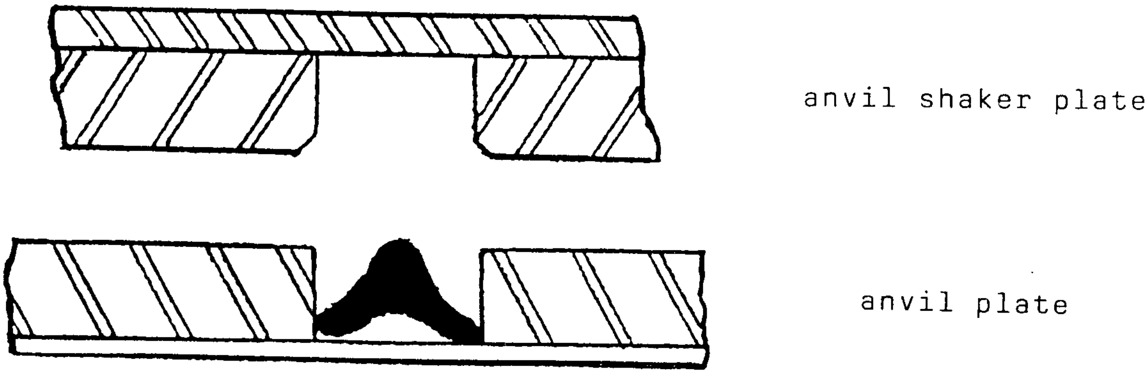
An empty anvil plate is positioned on the shaker plate.

The assembled "sandwich" is reversed 180° and lightly tapped on the flat surface (usually a block of wood). The anvils are thus transferred into the anvil plate point up.

The shaker plate is removed and returned to the shaker frame. The anvil plate is visually inspected that all anvils are in place, and is transferred to the next operation.

Note: Usually, the anvil plates are placed on a conveyor to avoid operator's movement. However, to maintain a steady and uniform flow of components, the number of anvil plates should be the same as the number of cup plates. For lacquered primers, the number of anvil plates is less than the cup plate number.





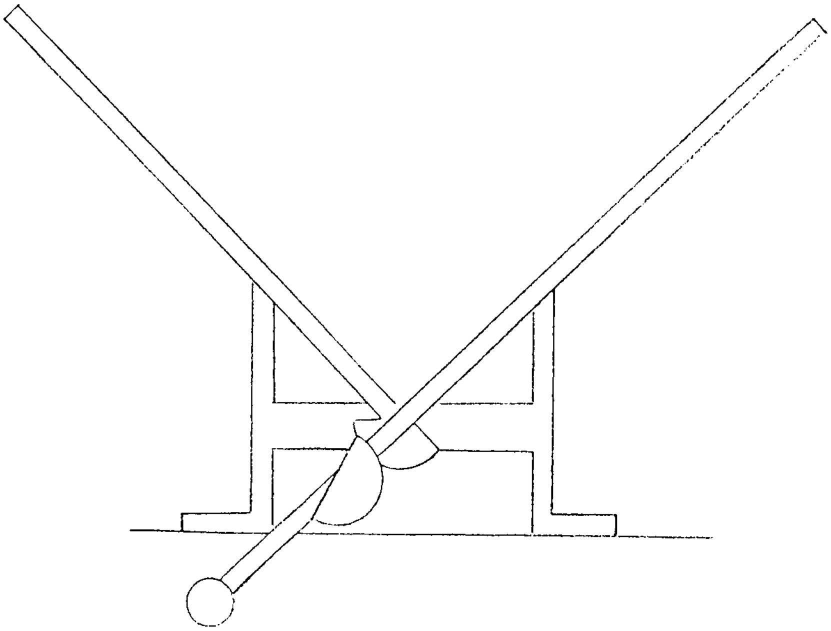
Operation 6.

The plate with foiled cups and a full anvil plate are placed into the corresponding sides of a fixture shaped like a large "V." A movement of a handle brings the sides of the "V" together. The fixture is opened, the sandwich removed and placed on a flat surface with the anvil plate on top.

The anvil bottom plate (which is now the uppermost) is removed and the remaining plates are passed to the next operation.

Note:

Skilled operators do the bringing of the anvil and cup plates together by hand without the use of the above fixture. Such operation is faster and less cumbersome. The plates, held in hand, are placed edge-to-edge at about 45 degrees, then swiftly brought together.



Operation 7.

The assembled plates are placed into a press. This anvil seating press is usually a small hydraulic unit containing a die set with the appropriate stops for the plates on the bottom platen. The upper platen contains a number of punches equal to the number of cups and anvils. Operation of the press brings the punches down, seating the anvils in the respective cups.

The plate assembly is removed from the press, the anvil plate is returned to operation 5 and the plate with the assembled primers moves to the next operation.

Note: The older type of anvil seating presses were mechanical, similar to the foiling presses, with only one row of punches and an indexing mechanism for the plates. Both the hydraulic and the mechanical single row model have their own merits and disadvantages.

Operation 8.

The assembled primers are visually inspected prior to placement into the lacquering unit.

This unit consists of a lacquer holding pan on the bottom, a frame to hold the primer plate, and an upper plate with pins corresponding to the number of primers in plate.

This upper pin plate is first brought down so that the pins are dipped into the lacquer, then brought up again.

Next, the plate with primers is slid into the frame and the pin plate is brought down on the primers, depositing a small drop of lacquer into each cup.

The pin plate is released and the primer plate removed, visually inspected and transferred to the next operation.

Note:

The lacquering unit described is a manually operated one, with the pin plate counterbalanced and spring loaded to the proper seating depth. The primer plate is slid into the unit from the side. The unit is usually designed around an existing, off-the-shelf die set.

A fully automatic system can be built, but then the visual inspection before and after lacquering must be either eliminated, or replaced by an optical inspection system. Again, the cost of equipment vs. labor is the determining factor.

Operation 9.

The plate with lacquered primers is pushed through a lacquer drying unit. This unit consists of a roller conveyor passing through a box with infrared heaters on top and an exhaust fan in the side. The plates are pushed through by the next following plate, thus maintaining a continuous flow. The temperature inside the box thermostatically controlled.

Operation 10.

The primers in plate emerging from the lacquer dryer are transferred to cardboard or plastic storage plates by first being inverted into an intermediate plate (where they are located cup-up) and then into the storage plate (anvil-up). The transfer is done in the conventional "sandwich -180 degree " rotation.

The primers in the storage plates may be passed through a final drying unit similar to the lacquer dryer, or they are placed into dryer trays (stacked up 3-5 high) and dried in a dryer room. The final choice depends on the manufacturer, inventory requirements, and final use of the primer (loading into cartridge cases, packing for sales, etc.)

NOTES

1 - Commercial primers are generally not lacquered, hence operations are eliminated as well as the possibility of lacquer smearing on the primer cup.

2 - The present setup is optimum for a production capacity of 40-50 million units/year shift with 4 operators. Smaller production requires the same equipment. Reducing the number of operators from four to 3 or 2 will reduce the efficiency due to increase leg work and motions.

3 - Introduction of continuous drying process after the inspection operation would reduce the primer handling but would increase considerably the hazard of primer explosion and propagation of same. See note on primer drying and storage method.

4 - Primers with lacquer smeared on the cup outside are cleaned immediately after assembly (before drying). About 10,000 primers are tumbled with 1 Kg of Maizo or hardwood sawdust in the tumbling barrel and separated from the cleaning material on a screen sieve on barricaded shaker.

5 - Lot size, one unit's shift work (200 -230 M primers).

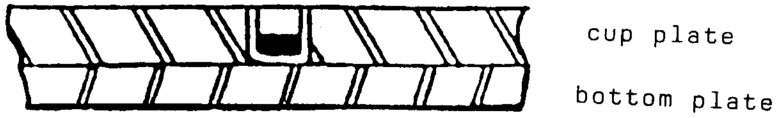
The LAP of 209 type shotshell primers is quite similar to the centerfire primer operations, but because of the third element (i.e. battery cup) involves additional operations.

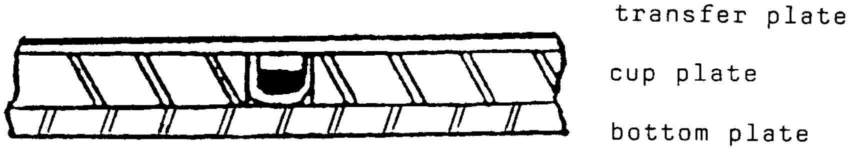
The equipment used in the shotshell primer assembly is essentially the same as used in the centerfire primer. The plate dimensions and the number of component holes will be slightly different due to the fact that the battery cup flange diameter is larger than the primer cup dia.

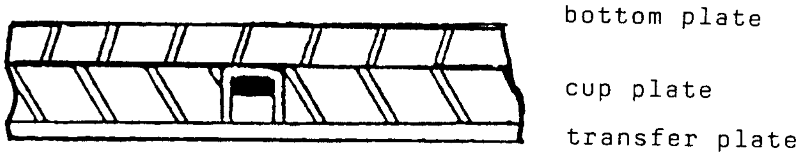
The Operations 1 through 3, i.e. primer cup orienting, charging and foiling, are identical for both centerfire and shotshell primer. The following operations describe the differences of the shotshell primer LAP.

Operation 4S.

For assembly with the anvil-battery cup, the foiled primer cup has to be positioned in the cup plate bottom-up, i.e. reversed. To accomplish this a thin steel plate without holes, called the transfer plate, is placed over the foiled primer cups in the cup plate. The "sandwich" of the transfer-cup-bottom plates is inverted upside down. The bottom plate, which is now located on top of the sandwich, is removed and returned to the corresponding operation. The cup and transfer plate assembly is now ready for the next operation.



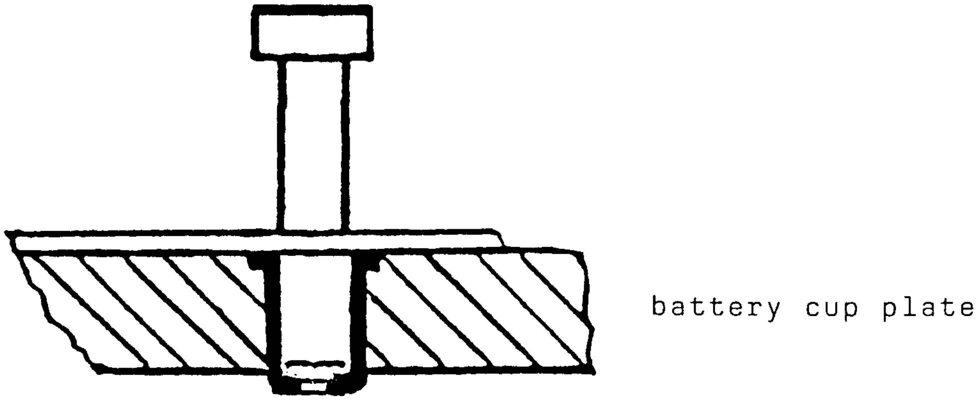




Operation 7S.

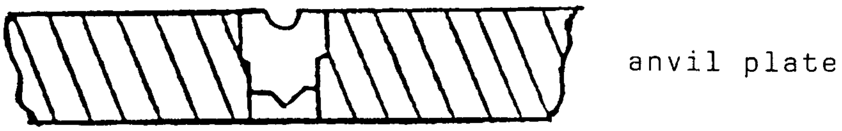
The inspected battery cup plate is inserted into the foiling press. This press is very similar to the cup foiling press with the upper platen holding a row of punches matching a row of cavities in the battery cup plate. A strip of foiling paper, passing through a wetting bath is fed at 90 degrees to the plate's longitudinal axis. As the punches descend, they blank out a small disc from the paper strip and push it to the bottom of the battery cup. After the punches return to the upper position, the plate indexes. When all rows of the plate have passed through and all battery cups have paper discs, the press stops automatically. The wetting of the foiling paper assists in a clean and burr free blanking operation.

Since the press runs by itself, one operator can easily serve four presses with plates.



Operation 8S.

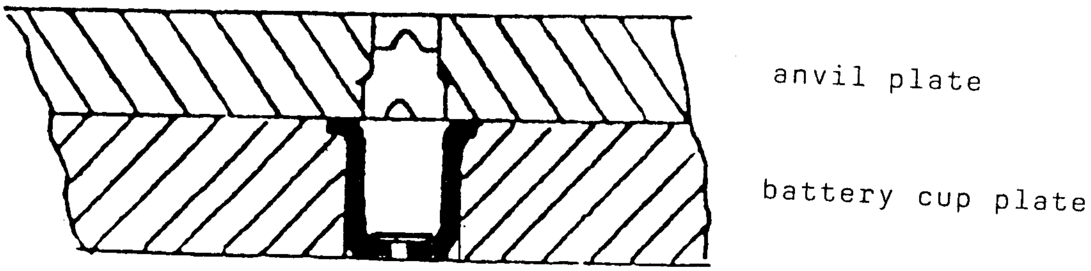
The anvils are dropped manually over anvil shaker plates on the shaker frame. As they move down the inclined plate, the anvils fall point down, into the plate cavities. The full plates are transferred to the next operation.



Operation 9S.

A plate full of battery cups and a plate full of anvils are placed into the corresponding wings of the "V" shaped unit identical to that used in assembly of centerfire cups and anvils described previously. A movement of the handle brings together the two plates. Now the anvils, point up are aligned with the flanged, open, ends of the battery cups.

The plate assembly is removed from the "V' unit and transferred to the next operation.

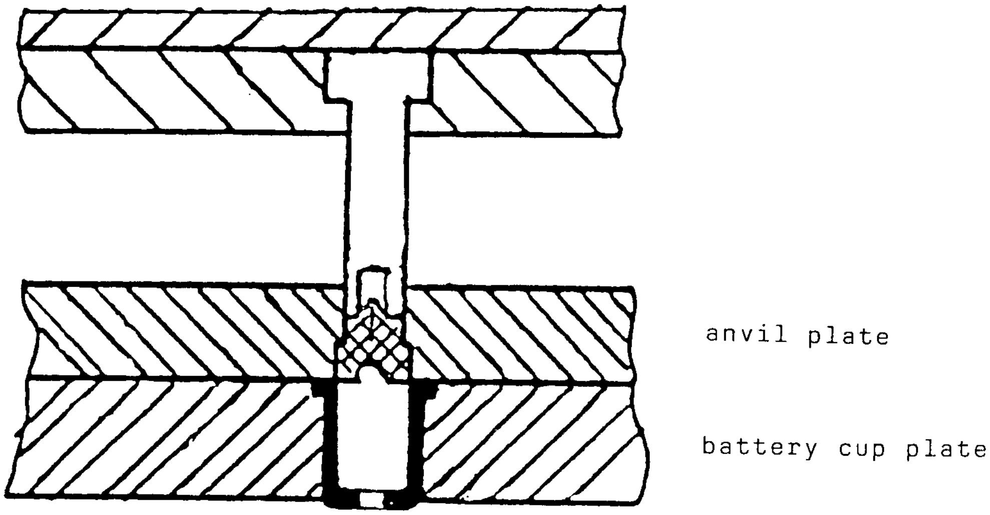


Operation 10S.

The assembled plates are placed into a small hydraulic press similar to that used in the centerfire primer anvil seating operation. The upper platen with the seating punches seats the anvils during its downward stroke.

After the cycle is completed, the plate assembly is removed from the press, the anvil plate is returned to the respective anvil shaking operation, and the plate with the battery cups (and seated anvils) is transferred to the next operation.

Note: the old presses used a weight control instead of hydraulic system to force the punches down.

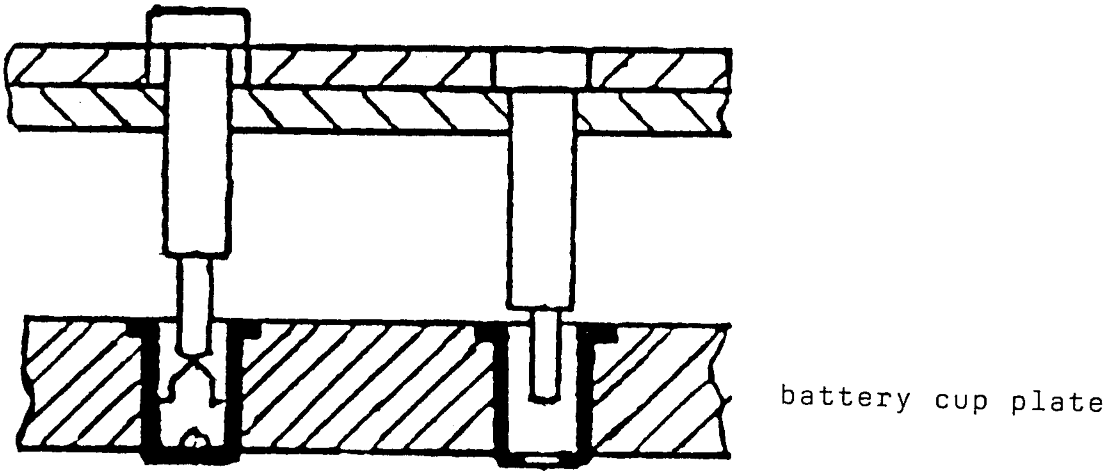


Operation 11S.

The plate with battery cup & anvil seated is placed into an inspection unit similar in construction to the lacquering fixture. The pan holding the lacquer is removed and the top of the pin plate is open to inspection.

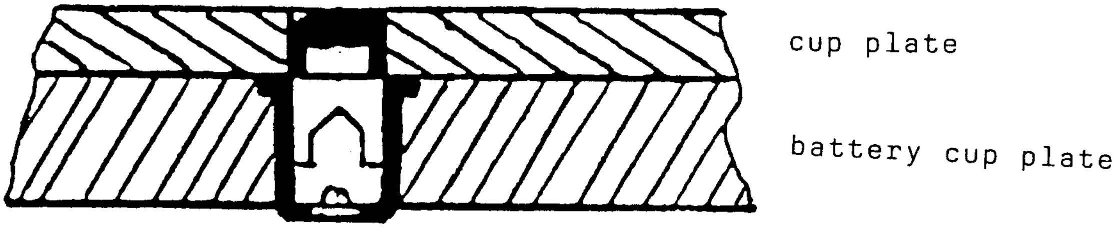
Then the pin plate is lowered into the battery cups without anvils and/or tipped and defective anvils. The respective pins will stay down as shown on the diagram.

This method allows a rapid and positive identification of defective components without dependence on visual inspection only. Defective parts are removed from the plate and replaced by good parts. The inspected battery cup plate is then transferred to the next operation.



Operation 12S.

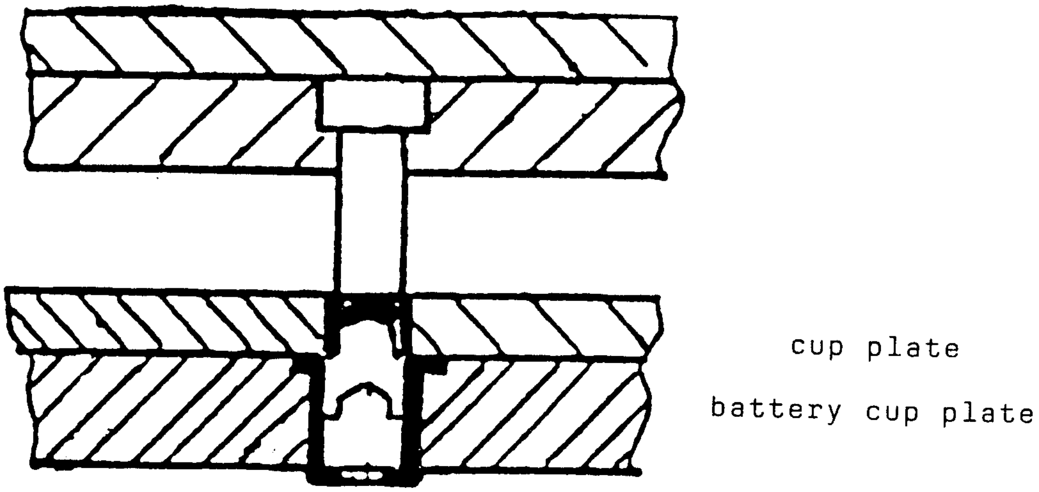
The battery cup plate and the primer plate (with transfer plate under) are placed into a "V" unit. This unit is the same construction as that used in centerfire primer assembly operation. By manual motion of the handle, the two sides are brought together so that the primer cups are now on top of the battery cups, with the priming mix facing the anvil point. The whole plate assembly is removed from the "V" unit and the transfer plate, which is now on top, is returned to Operation 4S. The battery cup and the primer cup plate assembly is transferred to the next operation.



Operation 13S.

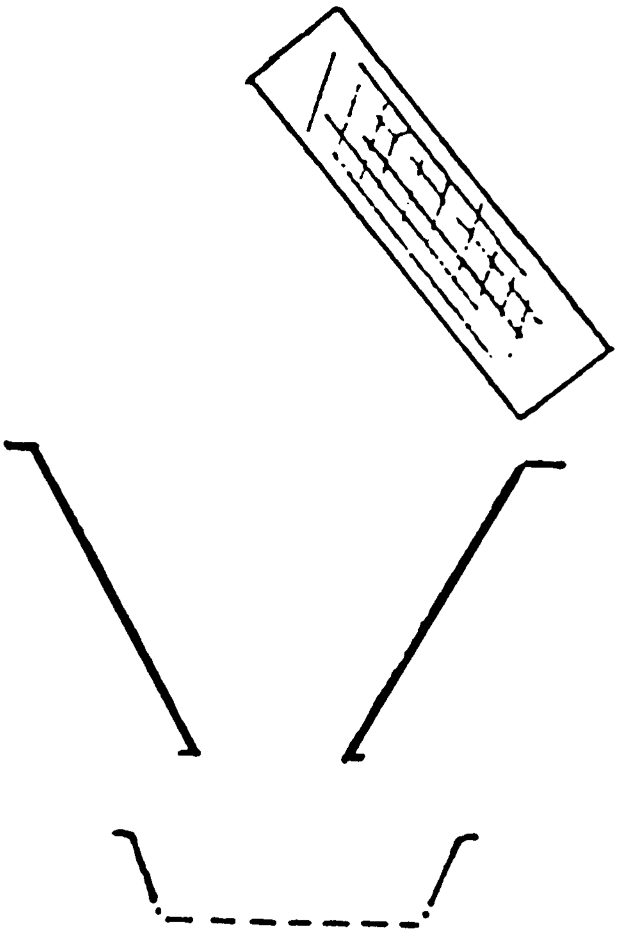
The battery cup plate and primer cup plate assembly are placed into a press similar to the anvil seating press. The upper platen contains a punch plate which matches the primer cup plate holes. As the platen lowers, the punches seat the primer cups in the battery cups, completing the primer assembly.

The whole plate assembly is removed from the press, the cup plate is lifted off and returned to Operation 1 and the exposed primers are visually inspected.



Operation 14S.

The battery cup plate with the inspected primers is inverted over hopper, dumping the primers into a drying can placed underneath. The empty battery cup plate is returned to Operation 5S. The drying cans, made from copper or conductive plastic, each contain about 2,000 primers. When the can is full, it is placed in a wooden can carrier holding 6 - 8 (depending on design) cans. The can carrier with full load is placed on a hand truck.



Note: a typical drying can is approximately 10 x 15 cm x 8 deep.

Operation 15S.

The loaded hand truck is transported to the drying room. The carrier with the cans is placed on a shelf and kept there seven days (one week) at 70° C and 20 % relative humidity.

The dry primers remain in storage until required by the cartridge loading department.

Note: To reduce inventory and work in process, sometimes a continuous drying process after the final visual inspection may be used. However, the hazard of a primer explosion and propagation of same is increased. Furthermore, the dry primer is still kept in dryer room during the ballistic testing of the primer lot, etc.

PRIMER CHARGING & ASSEMBLY SAFETY PRECAUTIONS

GENERAL

1 - All personnel in primer manufacturing area wears eye protection at all times.
2 - Priming mix carrier and worker trucking loaded trays to and from dryer room must wear prescribed safety shoes.
3 - Female operators must wear hair nets or scarves.
4 - Only wet priming mix in the proper bowl is allowed in the wall magazine.
5 - Only one tray carrier is lifted and handled at a time.
6 - Scrap and clean up pails are never placed inside each other even when empty and washed.
7 - Working area floor is regularly inspected to be free from cracks and coated with good floor paint.
8 - Floor in working area is swept thoroughly every day.
9 - Floors and walks adjacent and around primer charging and assembly area are swept thoroughly once every week.
10 - Primer charging and assembly area floor is washed with soda ash solution (25 g/ 1000 cc of water) once every week.
11 - All other floors are washed with plain water at foreman's discretion.
12 - Catch basin of washing sink is cleaned at least every two (2) weeks in accordance with the scrapping procedure described in the priming mix manual.
13 - Any unusual condition is reported to the supervisor immediately.

WALL MAGAZINE

14 - Bottom tray in wall magazine is always filled with water.
15 - Bottom tray In wall magazine is cleaned at least once a week and refilled with water.
16 - Priming mix in rubber bowl is handled only in wooden carrying tray provided for this purpose.
17 - Only one (1) carrying tray with eight (8) bowls is hand carried at any one time.
18 - Maximum allowed capacity of a single wall magazine is five bowls with priming mix.
19 - All priming mix (from the wall magazine and the charger unit) is returned to the storage magazine at the end of work day, or when the charging operations are closed for more than 30 minutes.

CHARGING

20 - All charging equipment is inspected and cleaned before work startup.
21 - Reservoir of the charging unit is filled up with water.
22 - Maximum amount of priming mix on the bed plate is 350 g.
23 - When the operator leaves the charging unit or is not charging for less than 5

minutes, all priming mix is collected into one lump on the bed plate and covered with wet cloth.

24 - When the operator leaves the charging unit or is not charging for more than 5 minutes the priming mix is returned to the rubber bowl and the charger unit cleaned up.

25 - At the end of each shift and before each rest period all priming mix is returned to the wall magazine and the charging unit cleaned up.

26 - At the end of each shift the reservoir of the charging unit is to be drained and washed. The wash water is poured into sink (not drain) and the stainless steel pail is also washed in the sink.

27 - Good housekeeping to prevent spillage of primer mix on the floor must be observed.

28 - Rubber bowls containing priming mix are kept free from smears and dried spots of priming mix, both inside and outside. Bowl must be wiped clean every time it is handled.

29 - Empty rubber bowls and covers are submerged in water in the pail near the charging unit.

ASSEMBLY

30 - Whenever the foiling press and/or the anvil seating press are stopped, the charging must be also stopped immediately to prevent drying out of mix in cups.

31 - Maximum two (2) charged plates are allowed between charging and foiling (or spinning) at any one time to prevent excessive drying out of primer mix.

32 - Defective primers picked during assembly operation are put in corresponding containers with water.

33 - Primers dropped on floor during operation are swept up and put in corresponding container with water once every hour.

34 - Dry room door is kept locked at all times.

35 - Only authorized personnel is allowed to enter the dry room.

36 - Dry room floor is sprinkled with water and swept once a week.

PRIMER CHARGING AND ASSEMBLY SAFETY PRECAUTIONS

Cleaning lacquer off primers

1 - No personnel may enter the cleaning room while the tumbling barrel is in operation as indicated by red lights.
2 - Doors to the cleaning room are closed during tumbling barrel operation.
3 - Maximum 20,000 primers are placed in the tumbling barrel at any one time.
4 - Maximum 100,000 primers are allowed in cleaning room at any one time.
5 - When the shaker separating primers from cleaning chips is in operation, only operator behind the steel plate is allowed in the room.
6 - Primers brought in for screening are not left exposed near the shaker.
7 - Floor in and around the cleaning room is swept daily.

Scrapping of assembled primers

1 - Primers charged with priming mix and rejected because they do not pass inspection are placed in a container filled with water.
2 - Scrapped primers are collected in bucket with water until about 5,000 primers accumulate.
3 - Hand carry bucket with primers to the burning station.
4 - Decant water from the bucket.
5 - Dump primers into the furnace.
6 - Open gas valve and ignite gas.
7 - Allow primers to burn.
8 - After all primers have burned and the metal cooled, clean out the scrap from the furnace.

DRY PRIMING MIX PROCESS

Loading - Assembly - Packing

The classic/traditional dry priming mix is the Mercury fulminate mix described in Chapter 6. This LAP process is similar to the wet mix LAP except that all operations are performed mechanically by remote control and automatic detectors, all placed behind heavy barricades. Furthermore, since the majority of primers with dry mix is of the Berdan type (i.e. the anvil is integral with the cartridge case), the anvil plates and the anvil seating units are not used. During the dry LAP process the plate filled with the primer cups is inserted through a gate in the barricade into the charging unit. This charger is a conventional type powder charger consisting of a fixture for holding the primer plate, with two plates positioned over it. The upper of these plates is the charging plate and the lower plate acts as the bottom. A "V" shaped hopper opened at the bottom (and full of dry priming mix) is pushed across the charging plate surface. The dry mix falling through the trough bottom fills the cavities in the charging plate. After all cavities in the charging plate have been filled the bottom plate is displaced slightly so that its holes line up with the cavities in the charging plate and allow the dry mix to drop into the primer cups.

The plate with the charged cups is then pushed along a track to a foiling and compacting unit similar to the wet mix operation. The plate with the primers is next positioned in a lacquering unit where a small drop of sealer is deposited into each primer. The plate with finished primers passes through a drier tunnel. All above operations are remotely controlled and automated.

PACKING OF PRIMERS

Since the centerfire primers are generally dried in individual nests inside special cardboard trays; they are transferred in these trays to the cartridge priming and loading operations. The shotshell primers, which are dried in bulk, are also transferred to the shotshell priming and loading in this form.

The reloading market packaging is done separately. The early primers were all packed in bulk into small metallic boxes (the percussion caps are still packed in this manner) .Next, as a convenience feature for reloaders, the primers were packed oriented, on edge in ten rows of ten primers each (100 primers) into small plastic trays with cardboard covers. To prevent possible ignition propagation from one primer to another, the present packing consists of a plastic tray in which each primer sits in its own individual cavity. The tray is slid inside a cardboard cover. The accidental ignition and/or propagation is reduced to a minimum. However, it is not recommended to store unused primers in bulk. Unused primers should be returned to the same storage box from which they were removed.

Storing primers in bulk (particularly centerfire type) in a tightly closed strong metallic container makes it into an unwanted and dangerous device.

Appendix C

Small Arms Primer Cups and One Pail Batches

The primer cups are produced on small double acting presses with automatic strip feed and scrap cutters. Depending on the press size and force, from one to five cups are blanked and cupped with each stroke of the press.

Finished cups are usually broken into lots; for example, one shift run from one press (about 100,000 pieces).

The anvils are produced by small blanking presses. The battery cups are made either by small double action presses with flanging done as a secondary operation, or on larger presses with progressive die sets forming the battery cup in a series of gradual operations.

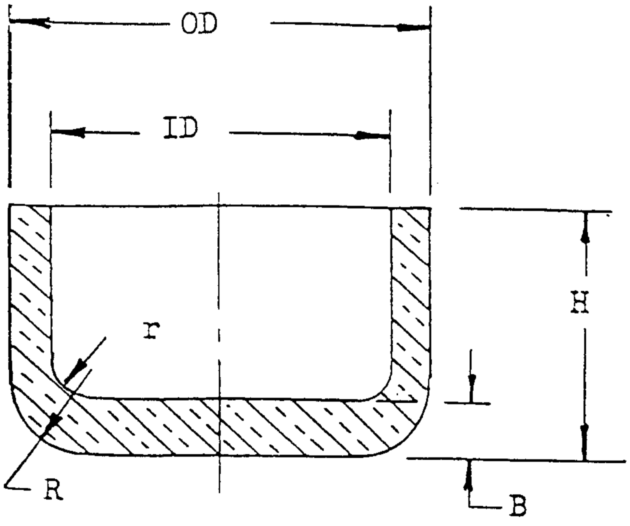
The advantage of the double action presses is the relative low cost of the equipment and tooling. However, the disadvantages are the labor intensive operation and the large floor space required.

The additional advantage of a progressive die system is the possibility of forming the anvil as an integral part of the battery cup. This method not only eliminates separate anvil manufacture, but also reduces the possibility of missing anvil at the primer assembly.

The washing and drying equipment for the metallic components is conventional type. The final choice is dictated by the production volume, labor cost, water and cleaning materials availability. This equipment may vary from modified small cement mixing machines, through fully automatic continuous operation degreasing, washing, rinsing and drying units. The washing (degreasing) of components is carried out in rotary barrels, the components are placed inside the barrel, a predetermined quantity of soap is added to the barrel contents, hot water is poured in and the barrel is rotated for certain time.

After the required time the wash water is drained off by decanting or opening the valve in the drum bottom, they are rinsed with hot water and then dried by tumbling in the same wash barrel. The hot rinse water transfers enough heat to the components so that they dry by themselves. External heat is sometimes applied to speed the cycle.

Dry components are checked by random quality control sample and if accepted, are transferred to intermediate storage.

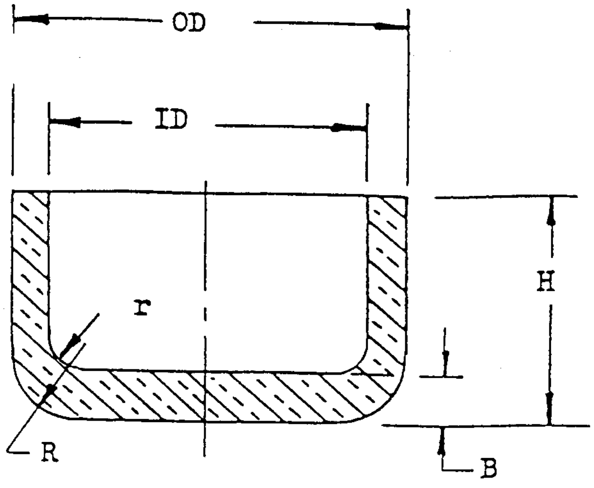


PRIMER TYPE (dimensions in mm)		**large rifle**	**small rifle**	**large pistol**	**small pistol**	**209 type shotshell**
Outside dia.	OD	$5.34^{+.02}$	4.43^{+002}	$5.33^{+.002}$	$4.45^{+.02}$	$5.34^{+.02}$
Inside dia.	ID	$4.64^{+.01}$	$3.70^{+.01}$	$4.63^{+.01}$	$3.80^{+.01}$	$4.60^{+.02}$
Height	H	$3.00^{+.15}$	$2.80^{+.15}$	$2.80^{+.12}$	$2.80^{+.15}$	$3.60^{+.20}$
Bottom thickness	B	$0.64^{+.03}$	$0.64^{+.03}$	$0.43^{+.05}$	$0.42^{+.02}$	$0.47^{+.02}$
Outside radius	R				0.76	
Inside radius	r		0.38			0.38

Notes:

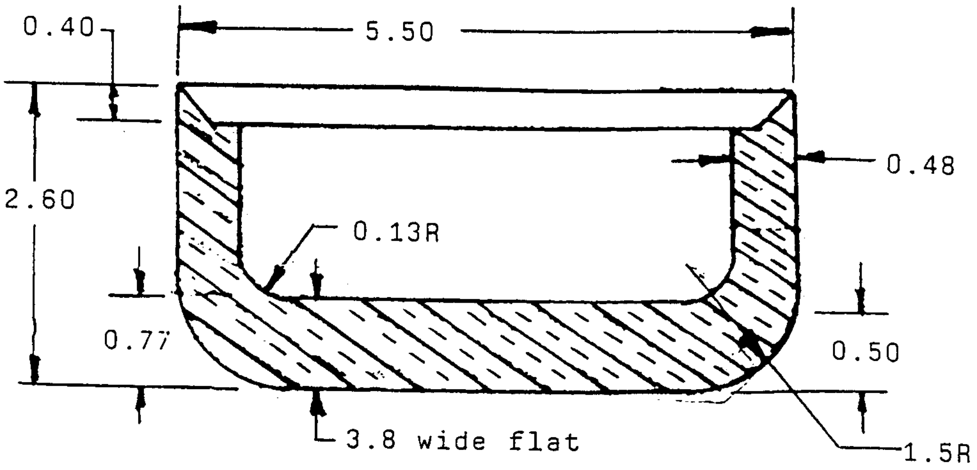
1 - Cup has no inside taper, to give the anvil the greatest amount of support at the upper edge of the cup.
2 - Waviness of cup rim not to exceed 0.03 mm.
3 - Wall thickness variation not to exceed 0.025 mm.
4 - Cup top not more than 2 degrees out of square.
5 - Outside finish commercially clean and bright, free from acid, oil, dirt, etc.
6 - Cup not annealed after draw.
7 - Dimensions are for manufacturing purposes only, acceptance is governed by functioning and sensitivity tests.

CUP, PRIMER, PERCUSSION, SMALL ARMS



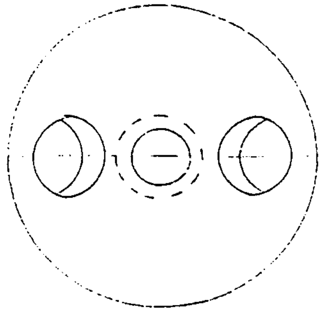
PRIMER TYPE (dimensions in mm)		.50 cal Browning
outside dia.	OD	$8.03^{+.02}$
inside dia.	ID	$7.02^{+.01}$
height	H	$5.37^{+.15}$
bottom thickness	B	$0.87^{+.03}$

7.62 x 39 Russian
primer cup
dimensions in millimeters



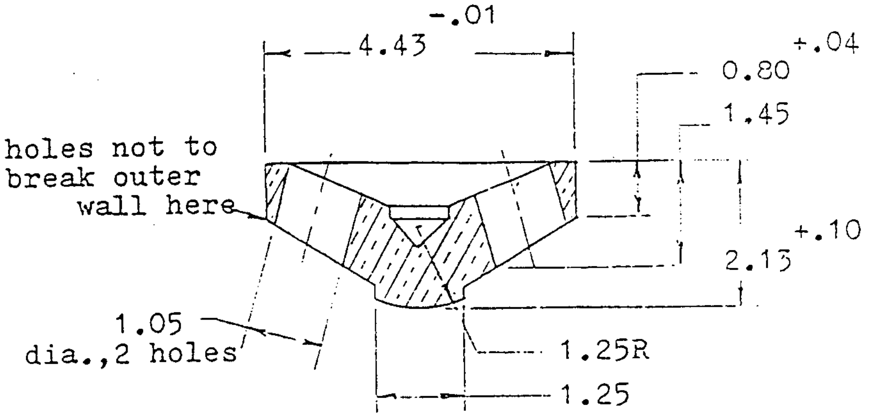
The anvil is probably the most difficult metallic primer component to produce. Its small size, complex form and close dimensional tolerances require exceptionally good tooling and quality control.

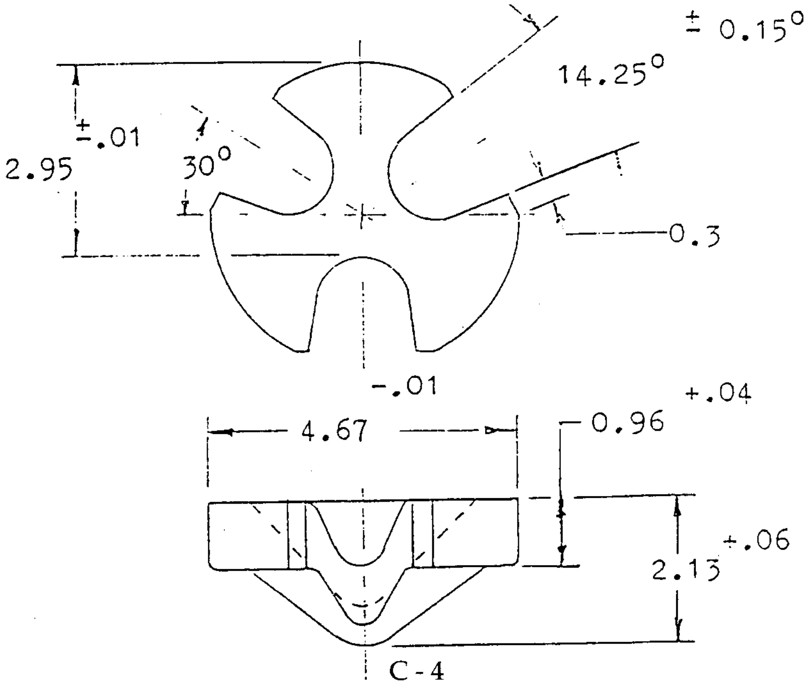
Following are drawings and typical dimensions of some anvils.



ANVIL, PRIMER, PERCUSSION, LARGE RIFLE SIZE

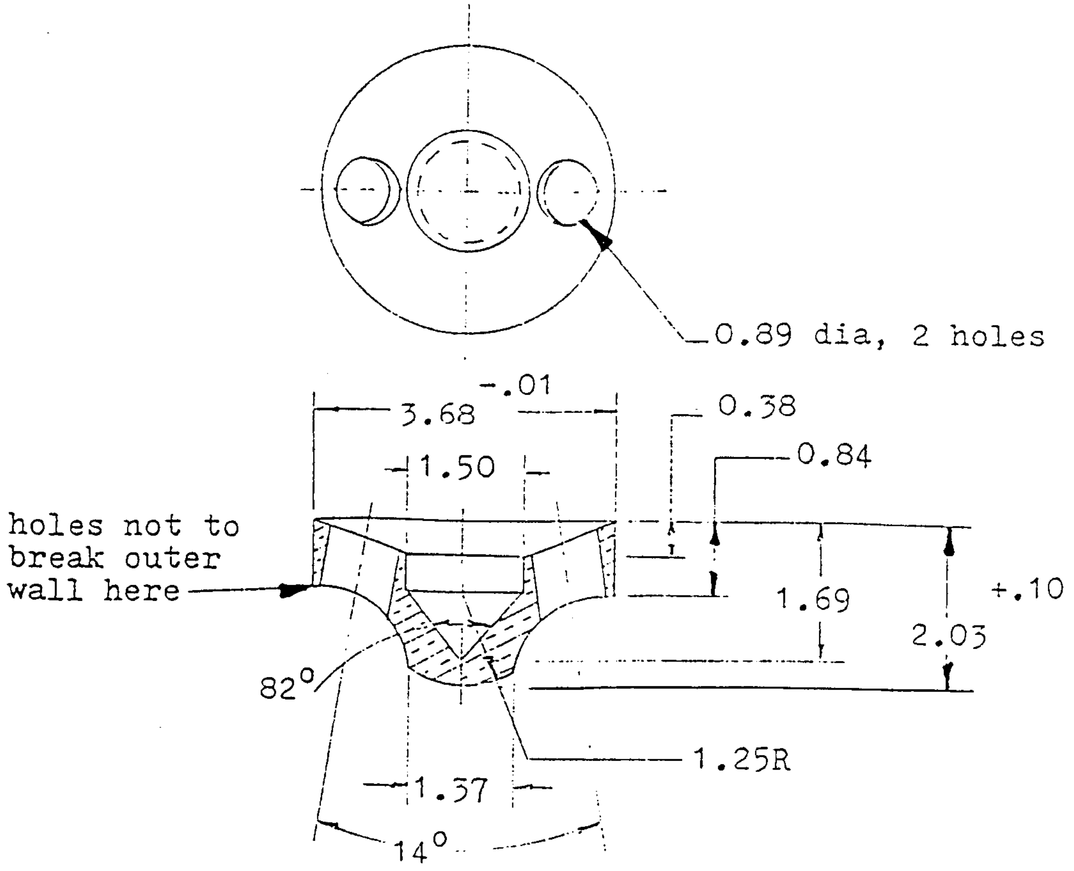
(dimensions in millimeters)

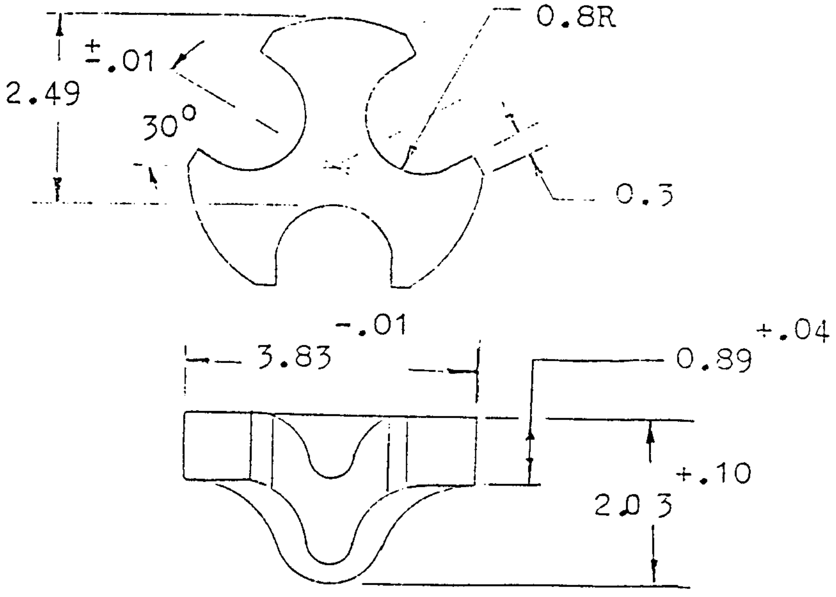




ANVIL, PRIMER, PERCUSSION, SMALL PISTOL SIZE

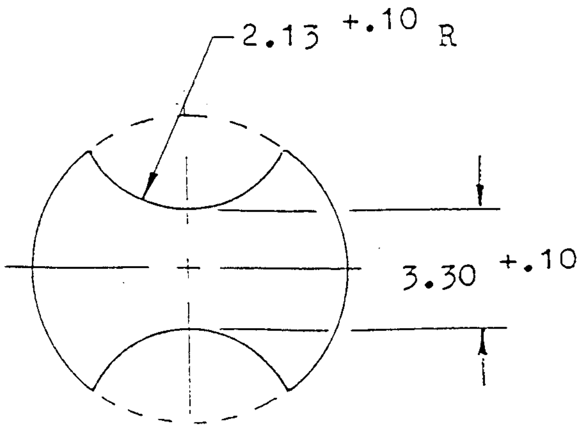
(dimensions in millimeters)



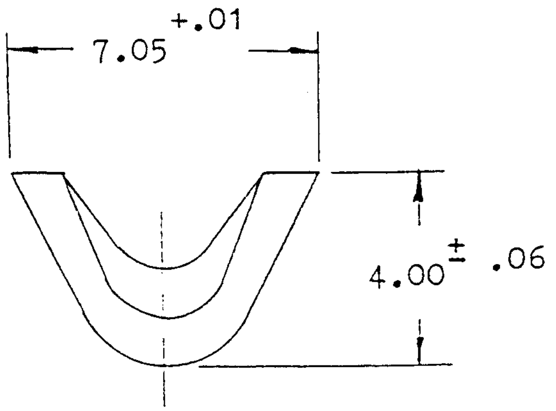


ANVIL, PRIMER, PERCUSSION, .50 CAL. BROWNING

(dimensions in millimeters)

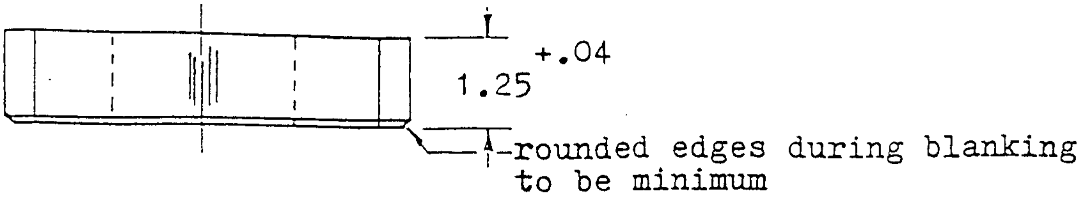


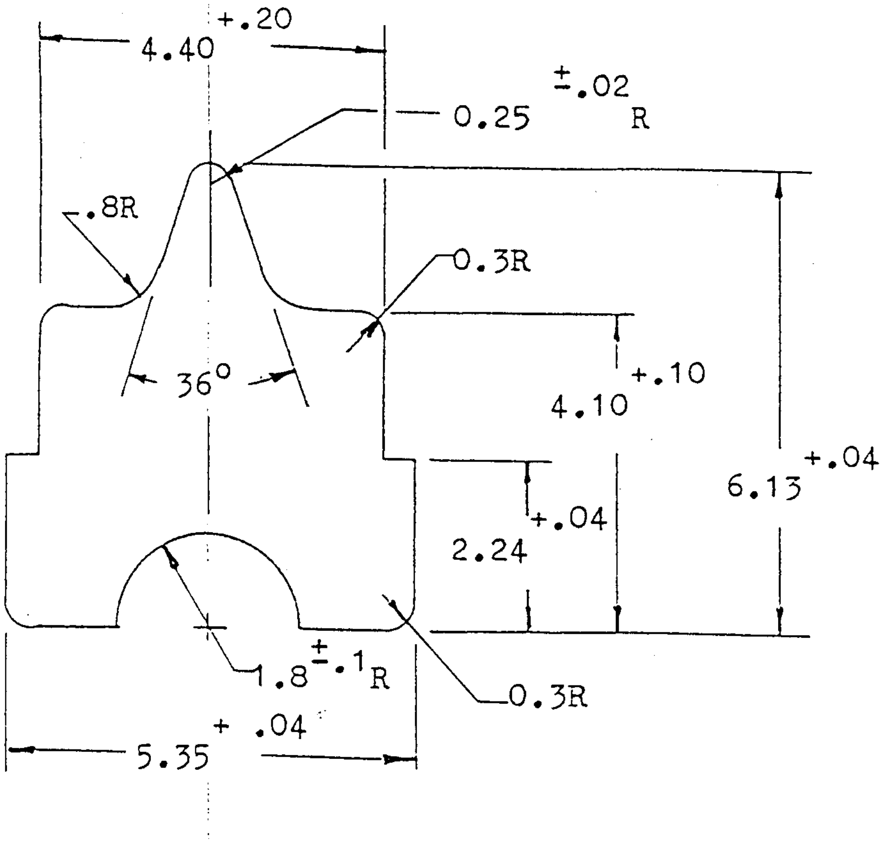
strip thickness 1.20 + .04



ANVIL, PRIMER, PERCUSSION, 209 SHOTSHELL TYPE

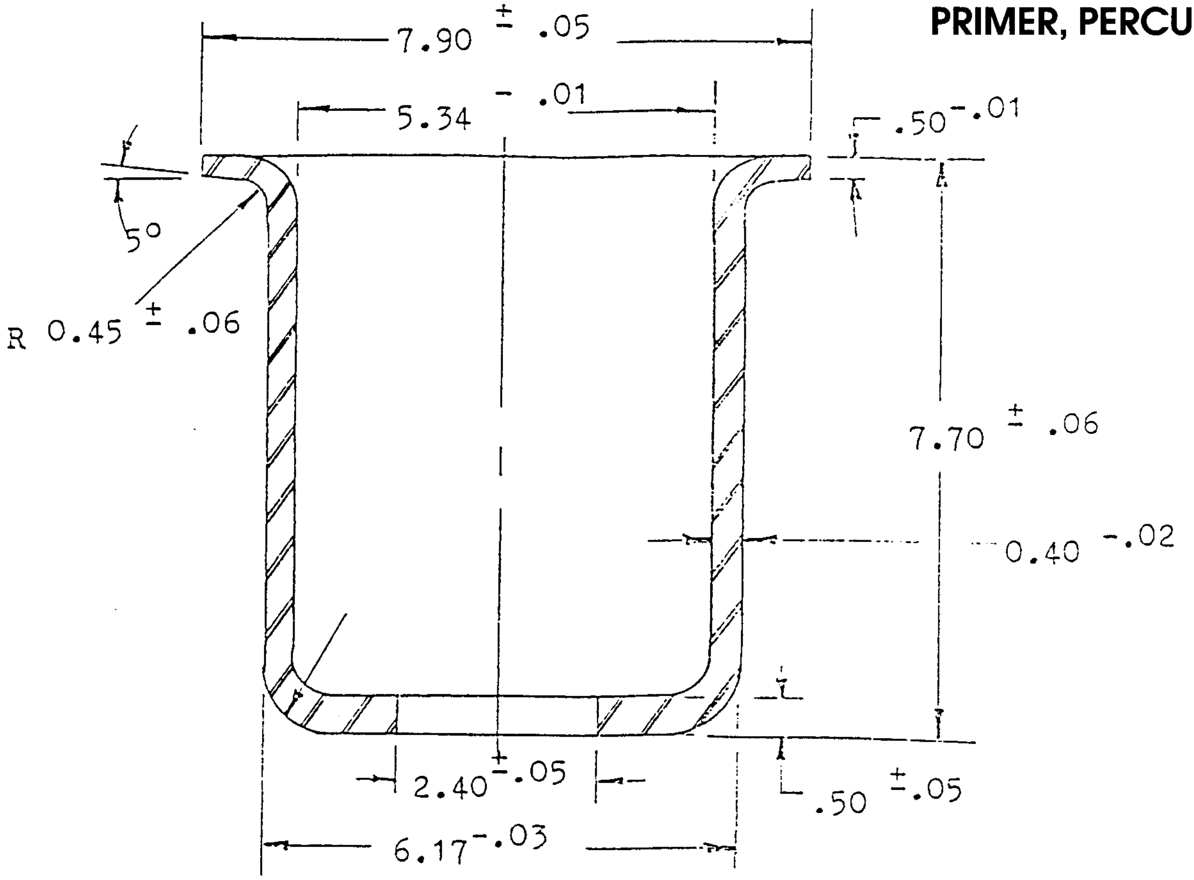
(dimensions in millimeters)



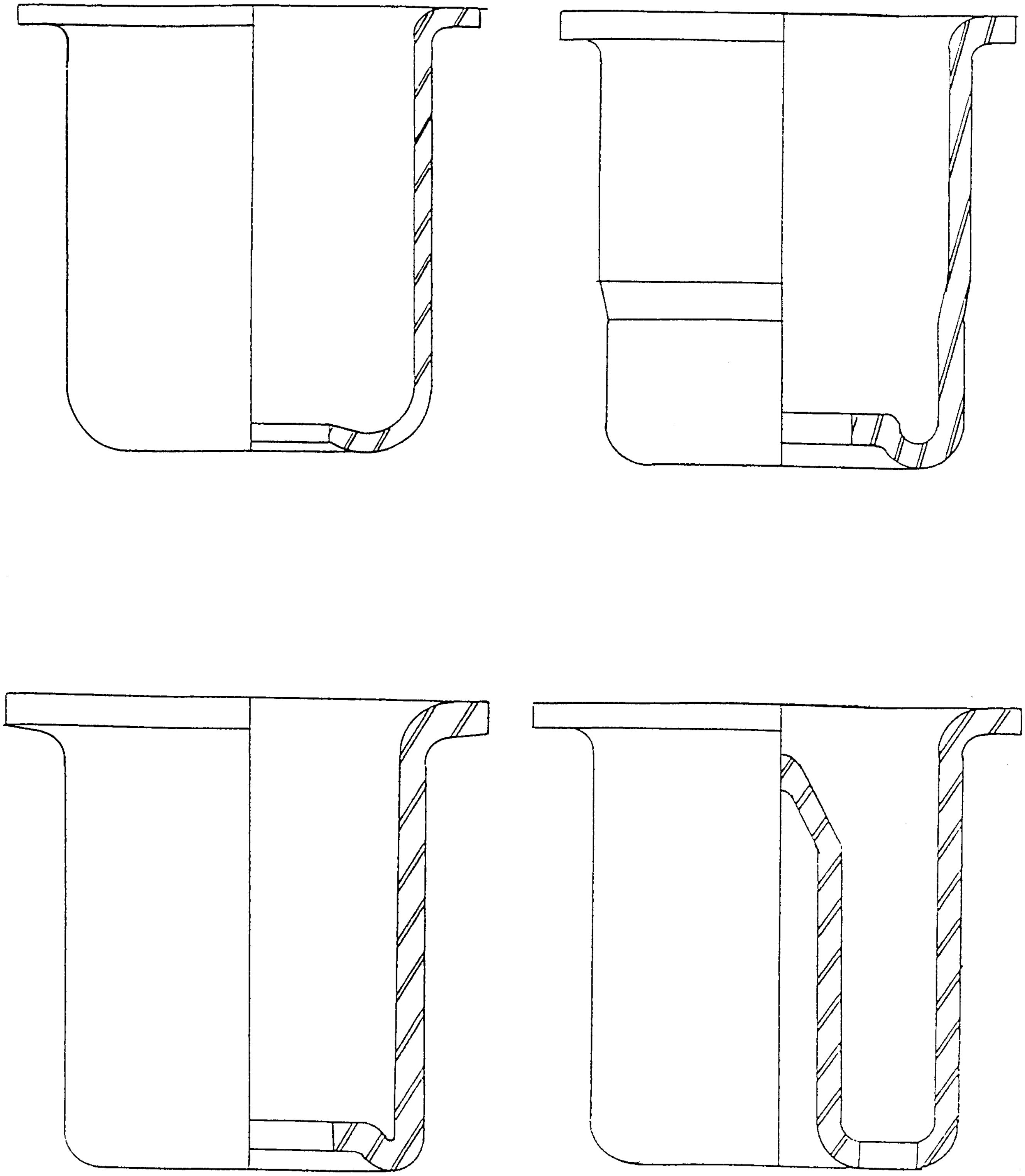


The typical battery cup dimensions and the various forms of battery cups will vary from manufacturer to manufacturer. The reasons for such variations may be the need for more positive anvil and primer cup support, better seat in the shotshell case, improved gas leak obturation, etc.

BATTERY CUP, PRIMER, PERCUSSION



typical battery cup dimensions (in millimeters)



Examples of battery cups used by different primer manufacturers

From the above it would appear that all primers of a certain type, for example "large rifle," would have the same external dimensions. However, in practice there are minor variations from manufacturer to manufacturer as well as between various plants of the same company. These variations are due to:

a - Tools used

b - Process

c - Raw material characteristics and strip thickness

d - Anvil geometry (2-prong, 3-prong, conical, flat, anvil in cartridge case, etc.)
e - Dimensions of the primer pocket in the cartridge case

f - Fnal use of the primer (military case load, commercial case load, reloader market, etc.)

The enclosed tabulated dimensions are for reference only. They are based on measurements of one line of primers. To try developing dimensions and tolerances of cups and anvils which would be acceptable in all combinations would be meaningless. The components could fit, but would not perform as required.

The dimensions of the various primer cups tabulated show that the dimensions of the large rifle cup are the same as the shotshell cup.

The small rifle primer cup and the small pistol primer cup are dimensionally the same except for the material thickness of the cup bottom. The small rifle cup is thicker since it is used in cartridges with heavier firing pin force.

The reasons for the above differences in primers are:

a - The primer cup must contain a sufficiently large charge to positively ignite the propellant even under -40° C conditions.

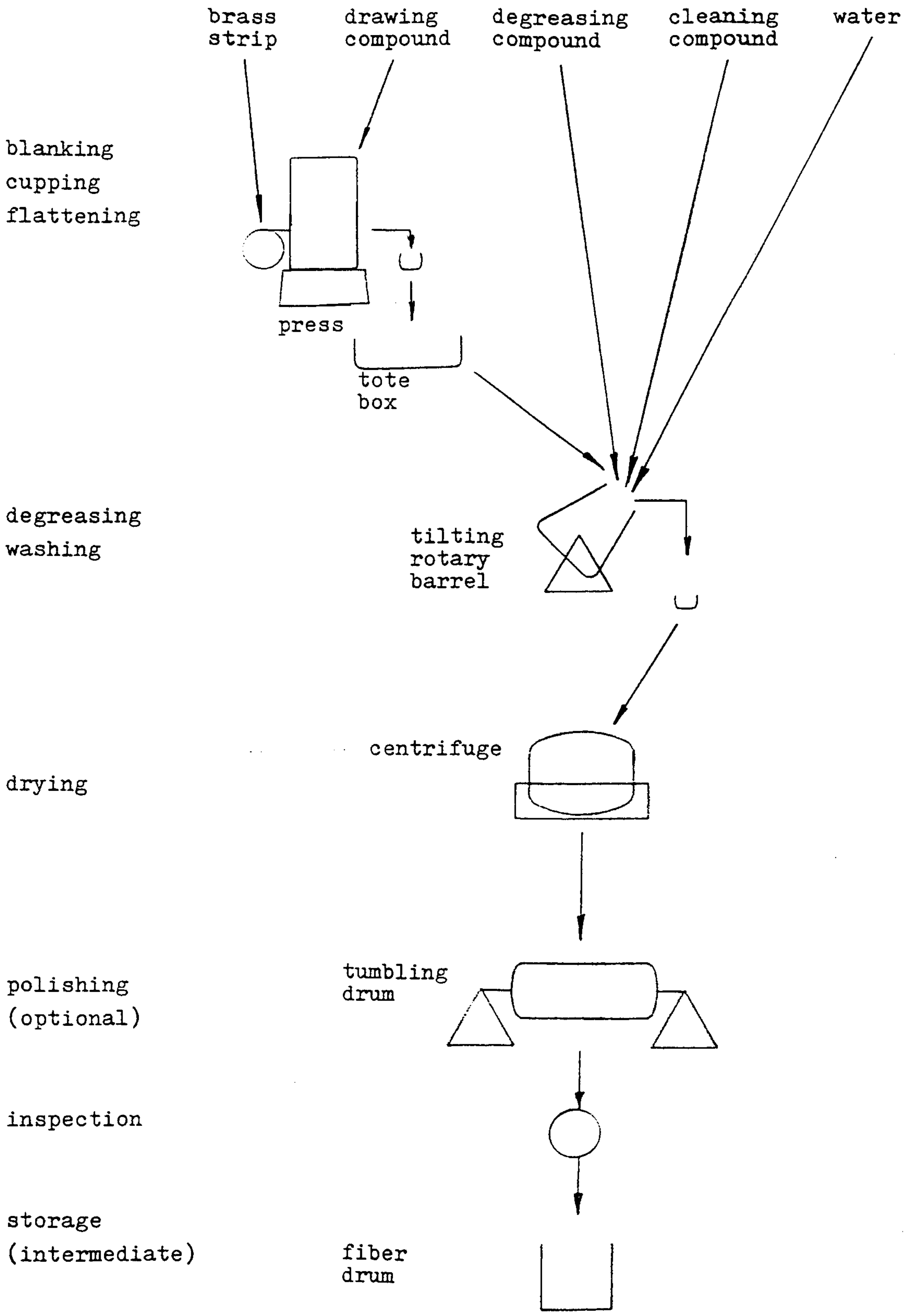
b - The cup must be small enough to fit into the cartridge case head without weakening it.

c - The cup must be soft enough to deform and fire even with a weak firing pin indent; this is especially important for commercial ammunition expected to function even in old and semi-defective arms.

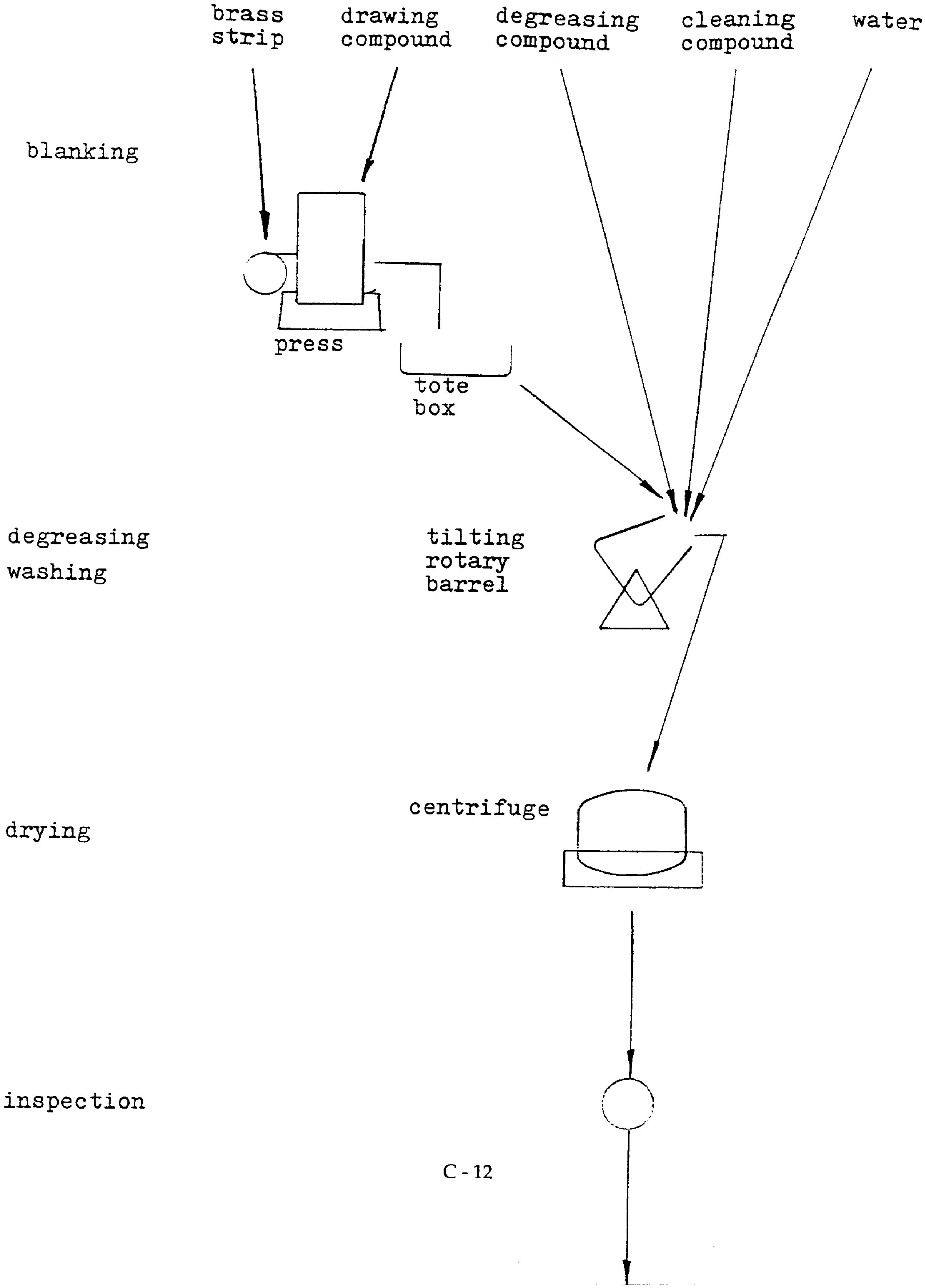
d - The cup must be hard enough not to fire when impacted on by a firing pin during bolt closing of a semiautomatic or automatic arm.

It is obvious from the above that the primer cup will satisfy such conditions only if kept within a very narrow band of production specifications. Close tolerance of the cup strip material thickness, metallurgical composition, grain size and metal hardness are essential.

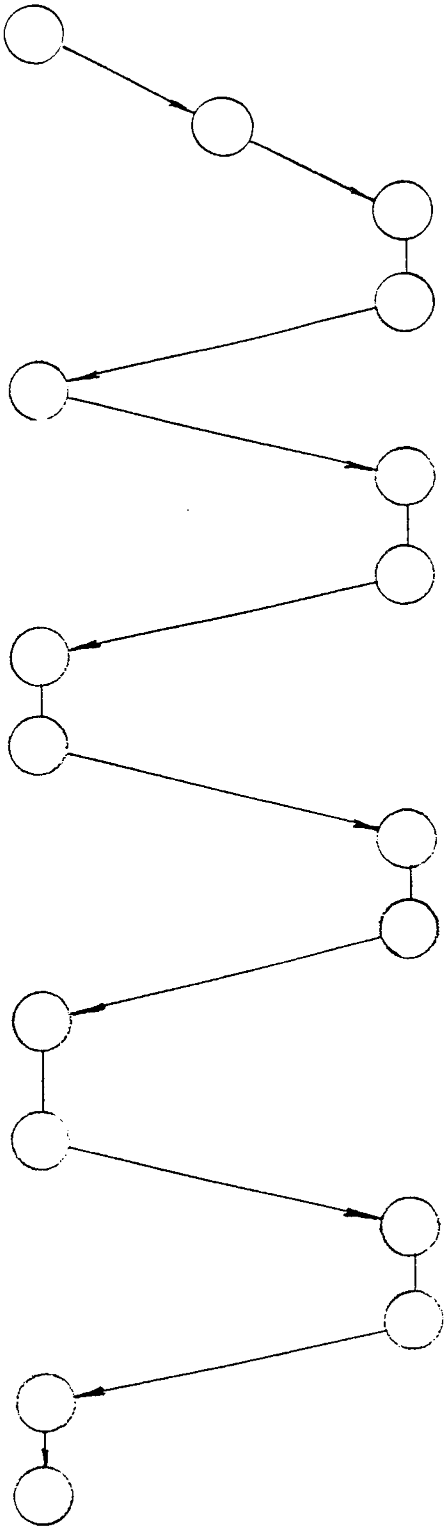
PROCESS DIAGRAM, PRIMER CUP MANUFACTURE



PROCESS DIAGRAM, PRIMER ANVIL MANUFACTURE

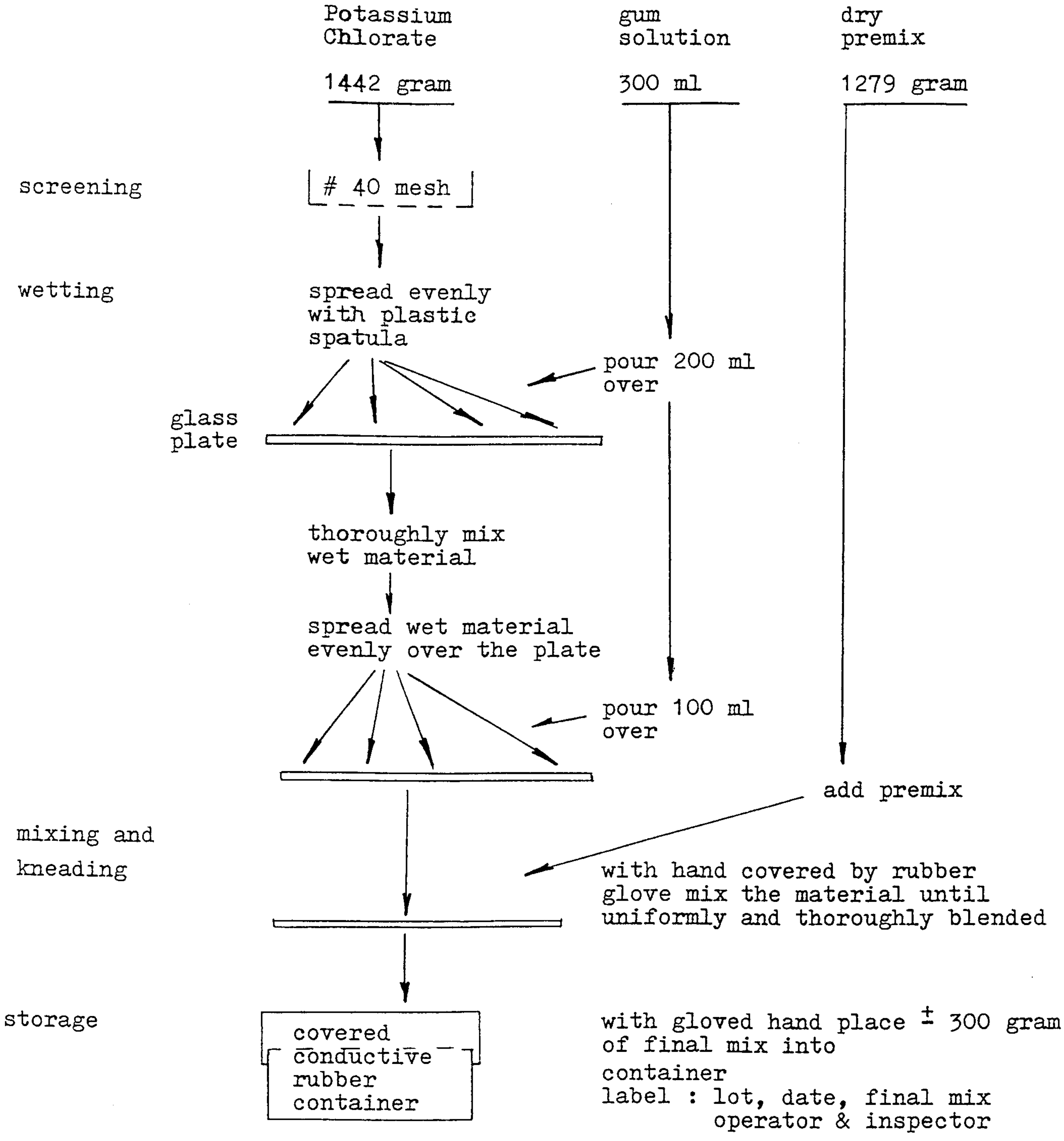


PROCESS DIAGRAM, BATTERY CUP MANUFACTURE
(Conventional method)



1 - blanking and cupping	press
2 - annealing	furnace
3 - washing	tilting rotary barrel
4 - drying	hot air rotary dryer
5 - drawing	press
6 - washing	tilting rotary barrel
7 - drying	hot air rotary dryer
8 - trimming	trimming machine
9 - chip separation	rotary perforated drum
10 - washing	tilting rotary barrel
11 - soap dipping	tilting rotary barrel
12 - flanging and piercing	press
13 - gauging	rotary perforated drum
14 - washing	tilting rotary barrel
15 - drying	hot air rotary dryer
16 - inspection	visual & hand held gages
17 - storage	fiber drum

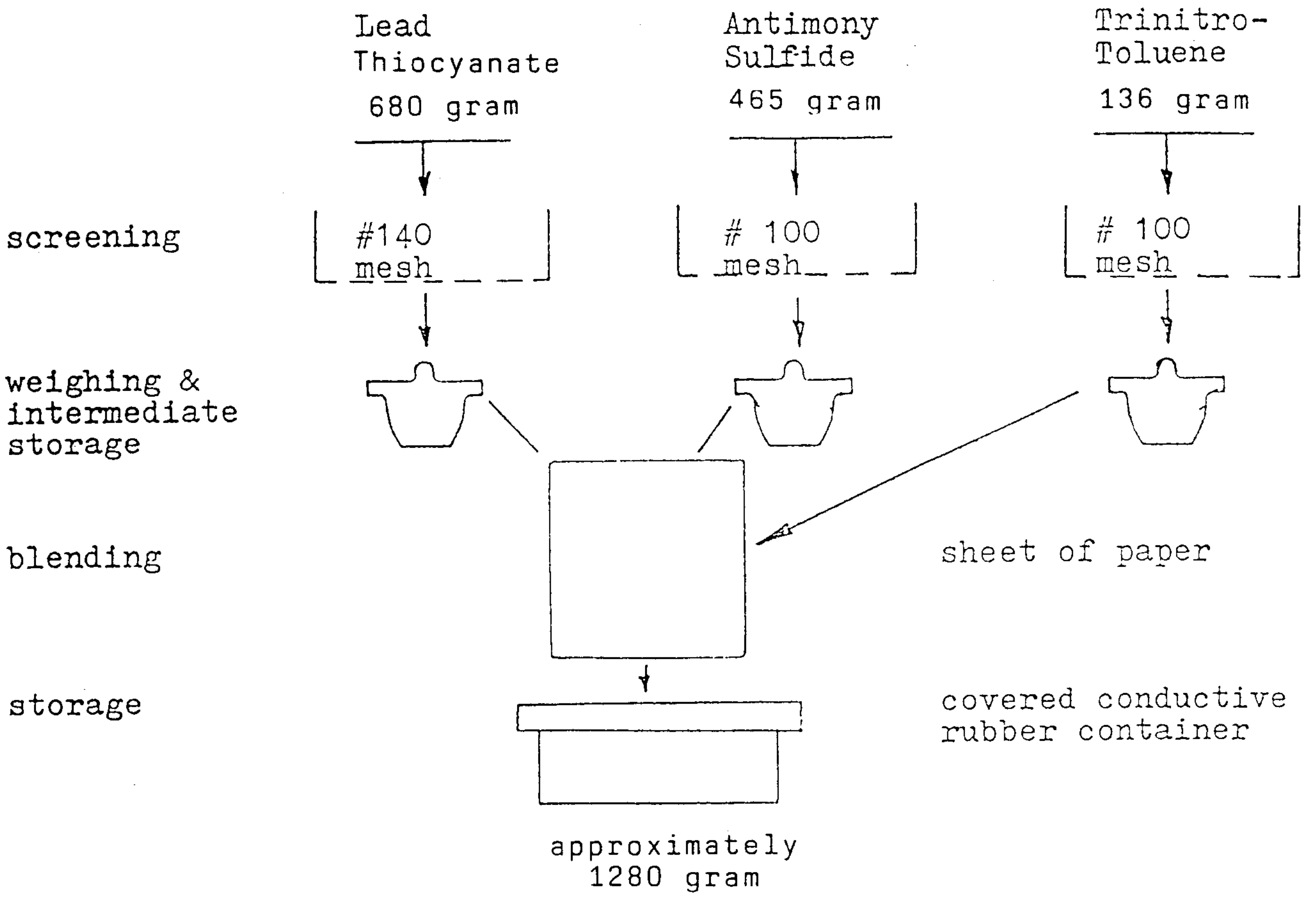
ONE PAIL BATCH PREPARATION EXPLOSIVE MIX MANUFACTURE, FA #70



Note: final mix is stored at temperature between 4°C and 10°C until needed, but not over 6 days; afterwards the composition is destroyed

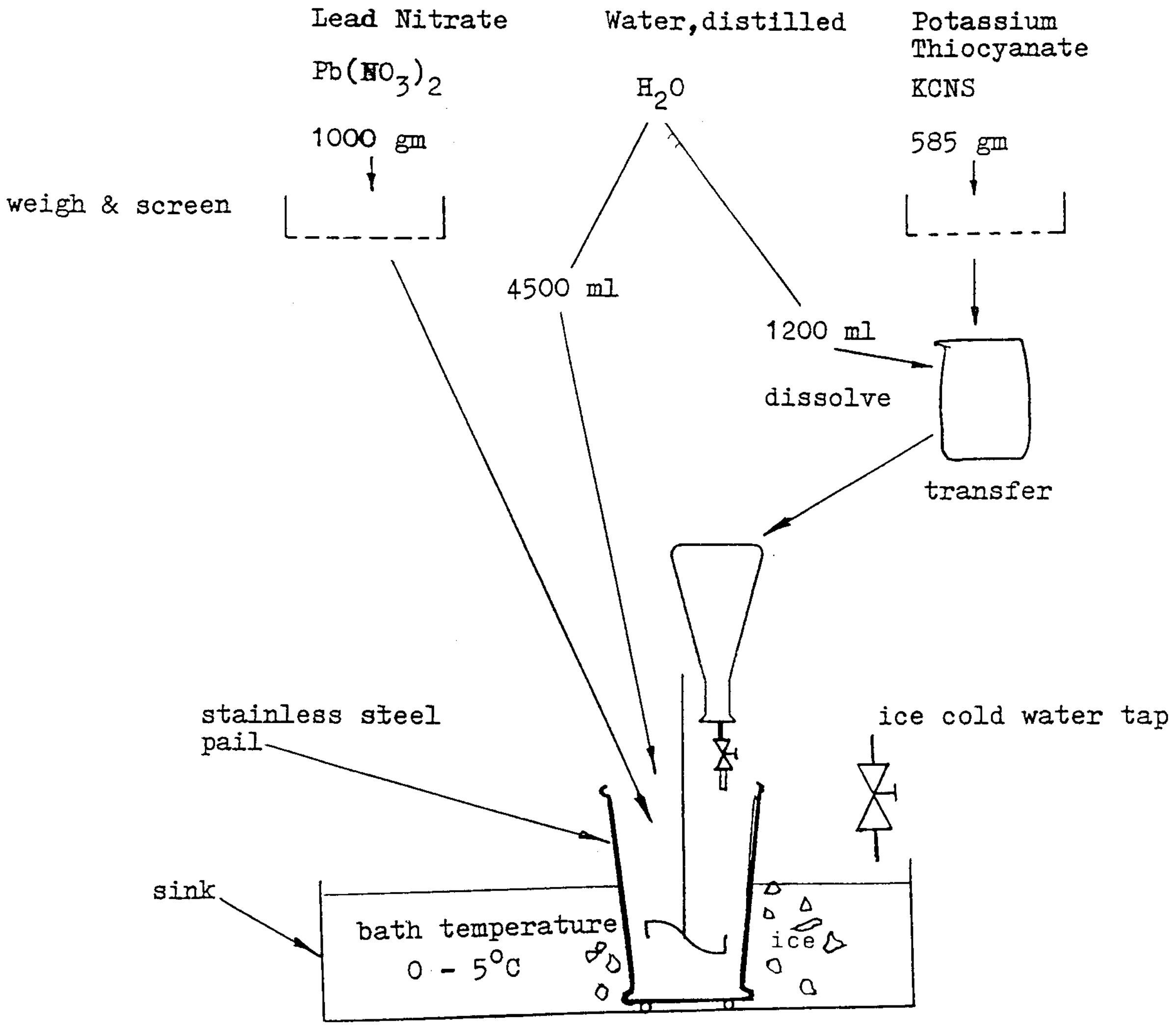
final mix is inspected visually for uniform blending

ONE PAIL BATCH PREPARATION OF FA #70
DRY PREMIX

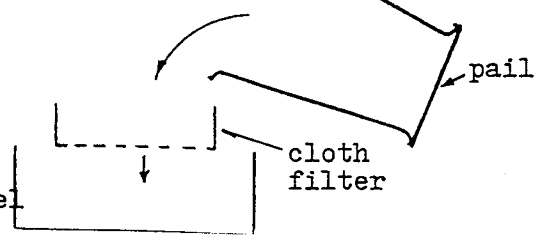


The preparation is identical to the LS-T dry premix even though TNT is present. Although TNT is a high explosive, it requires a powerful initiator and containment for initiation; hence it is relatively safe in the present application.

ONE PAIL BATCH PREPARATION OF LEAD THIOCYANATE



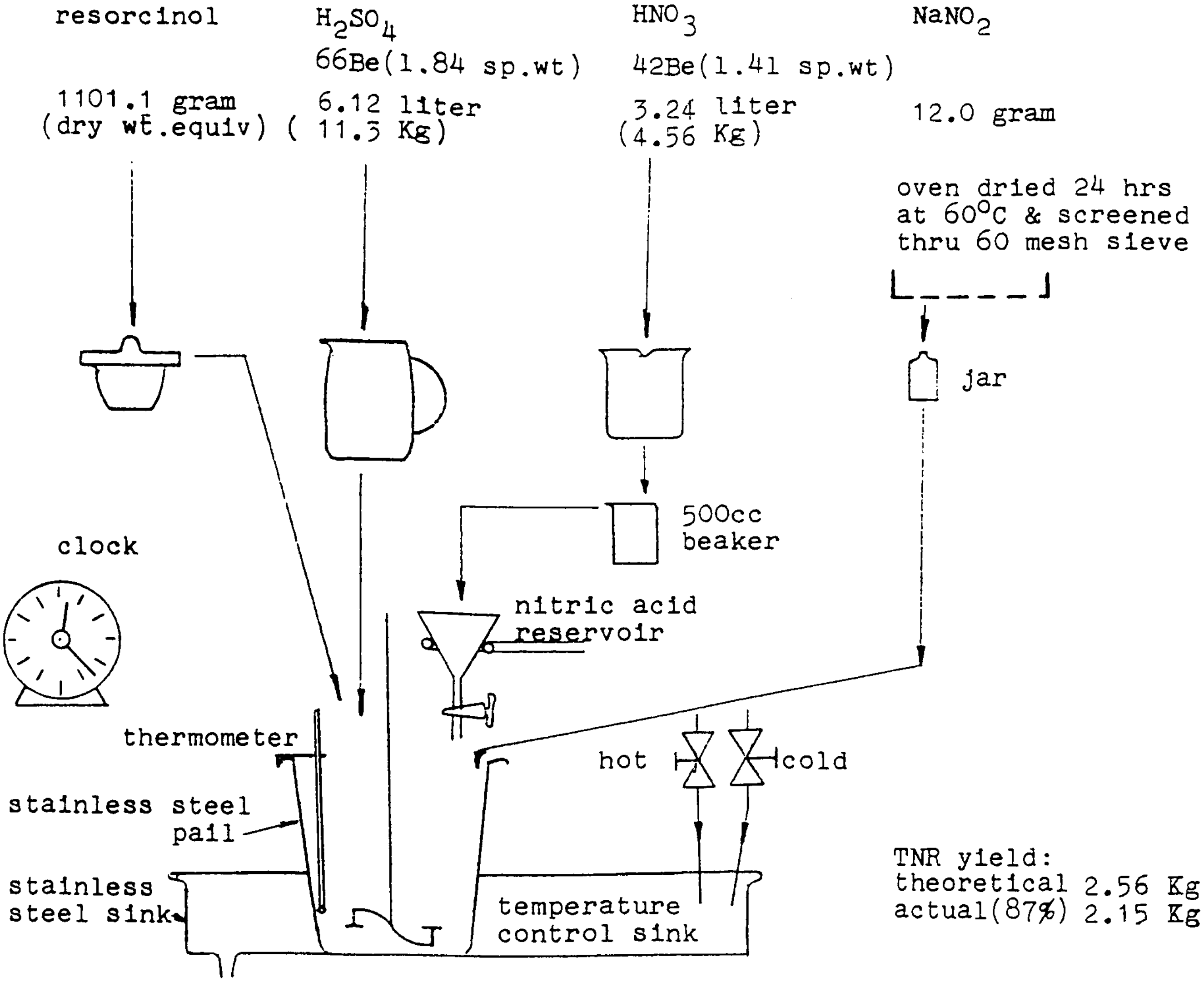
1 - place the screened lead nitrate and water into the pail
2 - start stirrer and continue until all nitrate is dissolved
3 - pour ice cold water into the sink
4 - check that the lead nitrate solution temperature drops to 5°C
5 - start addition of potassium thicyanate solution at a rate of about 40 ml/minute
6 - continue process until all materials have been placed into the pail
7 - stop stirrer
8 - pour pail contents (precipitate and mother liquor) onto filter



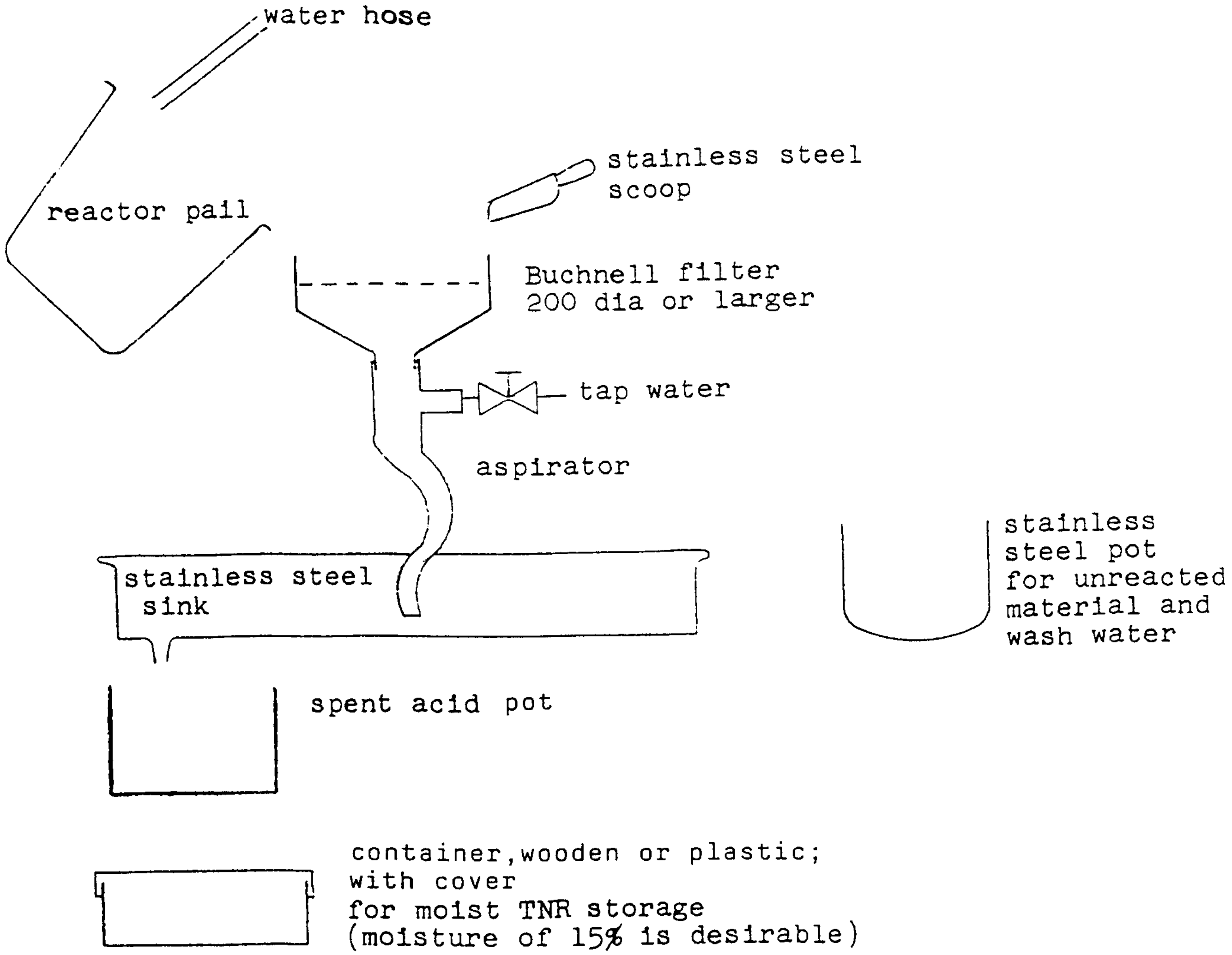
ONE PAIL BATCH PREPARATION OF LEAD THIOCYANATE, cont'd.

9 - rinse pail with ice cold deionized water and pour rinse water over precipitate on filter.
10 - wash precipitate twice with small jet of ice cold water.
11 - place cloth from filter with washed precipitate into dark and dry in warm air.
12 - the yield is about 94% theoretical, i.e. 900 gram.
13 - check a dry sample for crystal size and adjust step five (5) for optimum product size.
14 - mother liquor and wash water are boiled down for recovery of potassium nitrate.

ONE PAIL BATCH PREPARATION OF TRINITRORESORCINOL



ONE PAIL BATCH PREPARATION OF TRINITRORESORCINOL



TRINITRORESORCINOL
ONE PAIL BATCH PREPARATION

Sulfonation:

1 - Pour the correct amount of H_2S0_4 into the pail.

2 - Start stirrer, checking that it does not touch pail walls.

3 - Fill sink with hot water until the pail contents is 30 ± 5° C.

4 - Add 4 level tablespoons full of resorcinol to the pail contents.

5 - Add a little of $NaNO_2$ from the jar and continue through sulfonation.

6 - Add hot water to sink to raise the pail batch temperature to 63° C.

7 - Resume gradual addition of resorcinol so that the last spoon is added in about 90 minutes from process start.

8 - Allow batch to remain at 60° C

(about 30 minutes).

Note: The batch is now ready for nitration which may be carried to the point permitted by time. The nitration may be resumed for completion later. However, let stirrer run, wait about 10 minutes after last addition of HNO_3 then change sink water temperature to keep batch at 18° C. Before nitration resumption bring batch to 30° C.

Nitration: Start with batch at 30° C.

1 - Measure 100 cc from glass beaker into the nitric acid reservoir.

2 - Open reservoir valve to get flow rate of about 7 cc/minute.

3 - Repeat step 1 until 2000 ml of HNO_3 has been added. Since the reaction is exothermic, cooling of the batch is necessary to keep the temperature at 35° C.

a) If during the process temperature drops below 32 ° C, the reaction will slow down and concentration of unreacted HNO_3 builds up. Then as the temperature raises this acid will react rapidly, causing foaming over the pail, loss and decomposition of the TNR. Therefore when the temperature drops add hot water to the sink to heat the batch back to 35° C.

b) If the batch temperature raises over 38° C, stop addition of HNO_3 and let batch cool down to 35° C before resuming process.

4 - after the 2000 ml of HNO_3 have been added, stop batch cooling and allow the pail contents to heat up to 60° C.

5 - resume addition of HNO_3 immediately, without waiting for the batch to reach the 60° C.

6 - when all HNO_3 has been added start the cooling of the batch.

7 - when the batch is about 30° C (in about 50 minutes) it is ready for filtration.

8 - stop the stirrer and remove it from the pail.

Filtration

Performed in well ventilated area or under hood

1 - Aspirator is turned on and checked for leaks.

2 - Reactor pail contents is slowly ladled onto the filter which draws off the spent acids from the material.

Note: the bottom portion of the batch can be blue-purple in color and contains unreacted resorcinol and mixed acids. It will react with the spent acid as it is mixed on the filter and the temperature will rise. Allow this unreacted material to stand and combine with the next batch. If the unreacted blue-purple material is poured on the filter bed the heat of reaction may ignite the TNR.

3 - After the pail has been emptied rinse it with tap water and add the wash water to the unreacted materials. Also rinse carefully the stirrer shaft and blades and add this water to the wash water.

4 - After all/most spent acid has been drawn off the filter pour the contents of previous batch wash water over the material on the filter turning and spading the material during the process.

5 - After all wash water has been added, wash the filter contents with vigorous jet of tap water for 3-5 minutes.

6 - Continue aspirator operation for about 30 minutes.

7 - Take 5-10 g sample off TNR and check for moisture and sulfate. If sulfate contents exceeds 0.5% additional washing (step 5) is required.

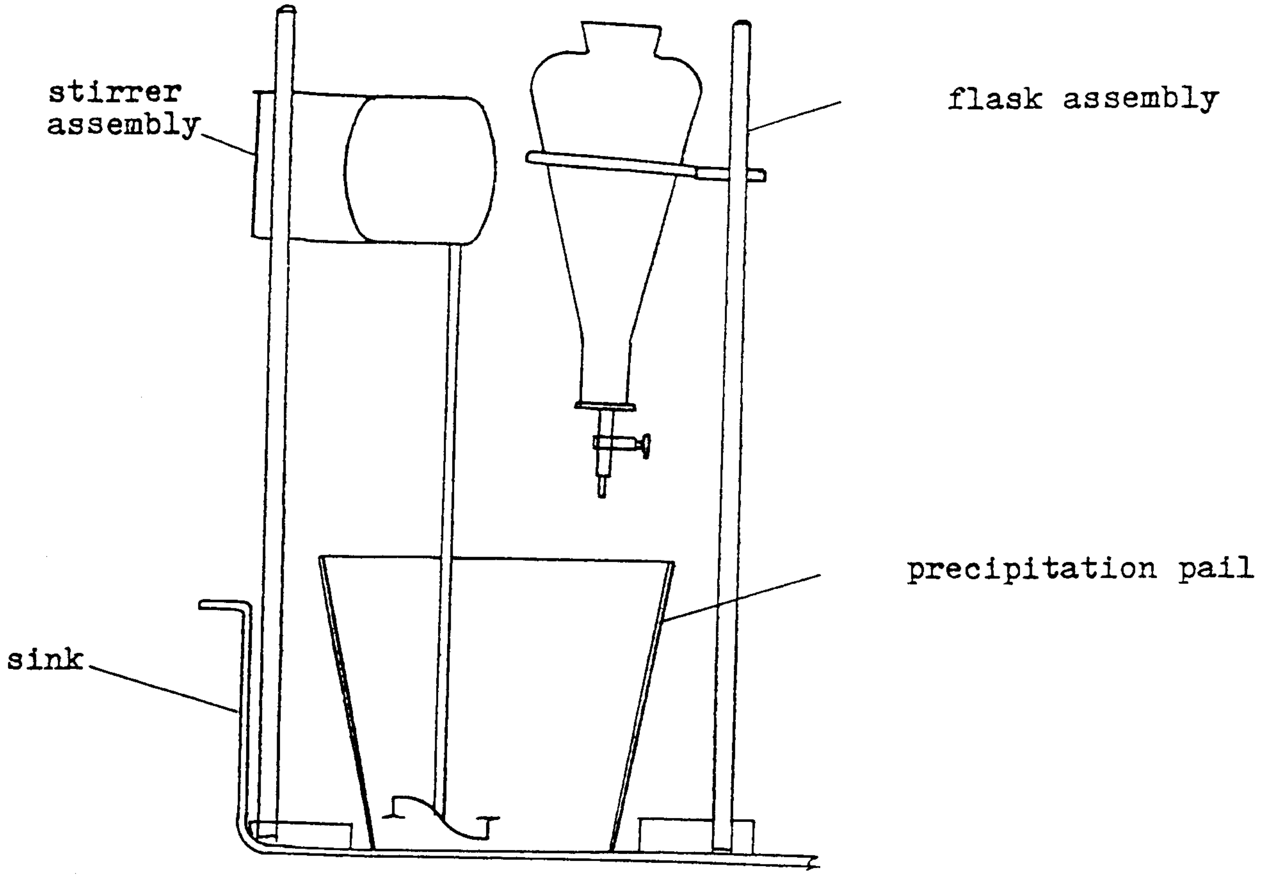
8 – Acceptable,moist TNR is ladled into storage container,dated and labeled.

Notes: The spent acids are either diluted to 0.5% concentration or neutralized with alkali before dumping into sewer. Approximately 10 Kg of flaked NaCH is required for one batch operation.The neutralized solution has a deep brown color.

ONE PAIL BATCH PREPARATION OF LEAD STYPHNATE
Equipment

Number	Item	Purpose
S 1	Table, 150 cm x 200 cm x 75 cm high, rubber or formica covered top, preferably with splash board	52, 55, etc.
S 2	Balance, capacity 2000 gram x 5 gram	
S 3	Weights, set 1 Kg x 5 gram	
S 4	Scoop, stainless steel, 5 each	ladling materials
S 5	Container with cover, stainless steel or conductive rubber, capacity 1500 ml	weighted TNR
S 6	Beaker, glass, capacity 1000 ml, required 3	weighted materials
S 7	Pail, stainless steel, capacity 10-12 liter	Lead nitrate sol.
S 8	Cylinder, glass, graduated,. capacity 1000 ml required - 2	measure NaOH sol. & Acetic acid sol.
S 9	Thermometer, glass, 0° - 100° C , required - 3	
S 10	Microscope, with mirror illumination, 10X eyepiece, 10X objective lense	inspect LS crystals
S 11	Beaker, glass, pyrex, capacity 30 ml	LS burning test sample
S12	Sink, stainless steel, 25 cm deep, bottom 60 cm from floor, with overflow plug, hot and cold water taps	for precipitation pail
S 13	Hood with exhaust fan (about 1 HP)	over sink
S 14	Water deionizer	reaction water
S 15	Pail, stainless steel, capacity 15 liter	LS precipitation
S 16	Flask assembly	Lead nitrate and acetic acid solution
S 17	Stirrer, motor 1/4 HP, 300 rpm, explosion proof, impeller and shaft from stainless steel; with adjustable stand	mixing LS slurry
S 18	Pail, with handle and cover, conductive rubber, capacity 12 liters	LS storage
S 19	Sieve, stainless steel, No 50 mesh (may be built from a suitable ss pail)	filter LS into storage pail

ONE PAIL BATCH PRECIPITATION UNIT FOR PREPARATION OF LEAD STYPHNATE



ONE PAIL BATCH PREPARATION OF LEAD STYPHNATE

MATERIAL PREPARATION:

TNR

The material, as received from storage is in wet condition. It can be dried in oven and weighed directly, however dry TNR creates a dust problem as well as extra work. Therefore it is customary to use wet TNR and follow the procedure:

- TNR contents of the storage container is thoroughly mixed with a paddle to make it uniformly moist throughout the batch.
- 10 gram sample is placed in a covered tared weighing dish.
- Sample is weighed and weight recorded.
- Sample is dried overnight at 100° C.
- Dry sample is weighed and weight loss calculated as % moisture. Storing sample dry at 100° C longer than 12 hrs. some TNR will sublime and the error in dry weight calculation will be excessive; if sample is to be dried longer than 12 hrs. keep the oven at 55° ± 5° C.
- Wet weight of TNR = $\dfrac{735}{100 - \%\ \text{moisture}}$
- After the wet weight is calculated the entire container contents is weighed into the 735 dry weight portions and placed in storage containers.

LEAD NITRATE SOLUTION PREPARATION

To precipitate 3 moles of TNR requires theoretically only 993 gram of lead nitrate, but the excess is used to:

a - Ensure completeness of reaction.

b - Decrease solubility of lead styphnate (common ion effect).

c - Eliminate need for extreme accuracy in measuring lead nitrate.
because of the pail volume limitations, lead nitrate is used in saturated solution and must be kept at 21° C or more, to prevent precipitation.

To prepare solution:

- Weigh 1250 gram lead nitrate into stainless steel pail.
- Add 2500 ml hot (60° C) water.
- Stir until all material dissolves.
- add 238 gram (226 ml) glacial acetic acid.

- add cold water to bring the solution volume to 3000 ml.
- transfer solution into metering flask.

Sodium hydroxide solution preparation

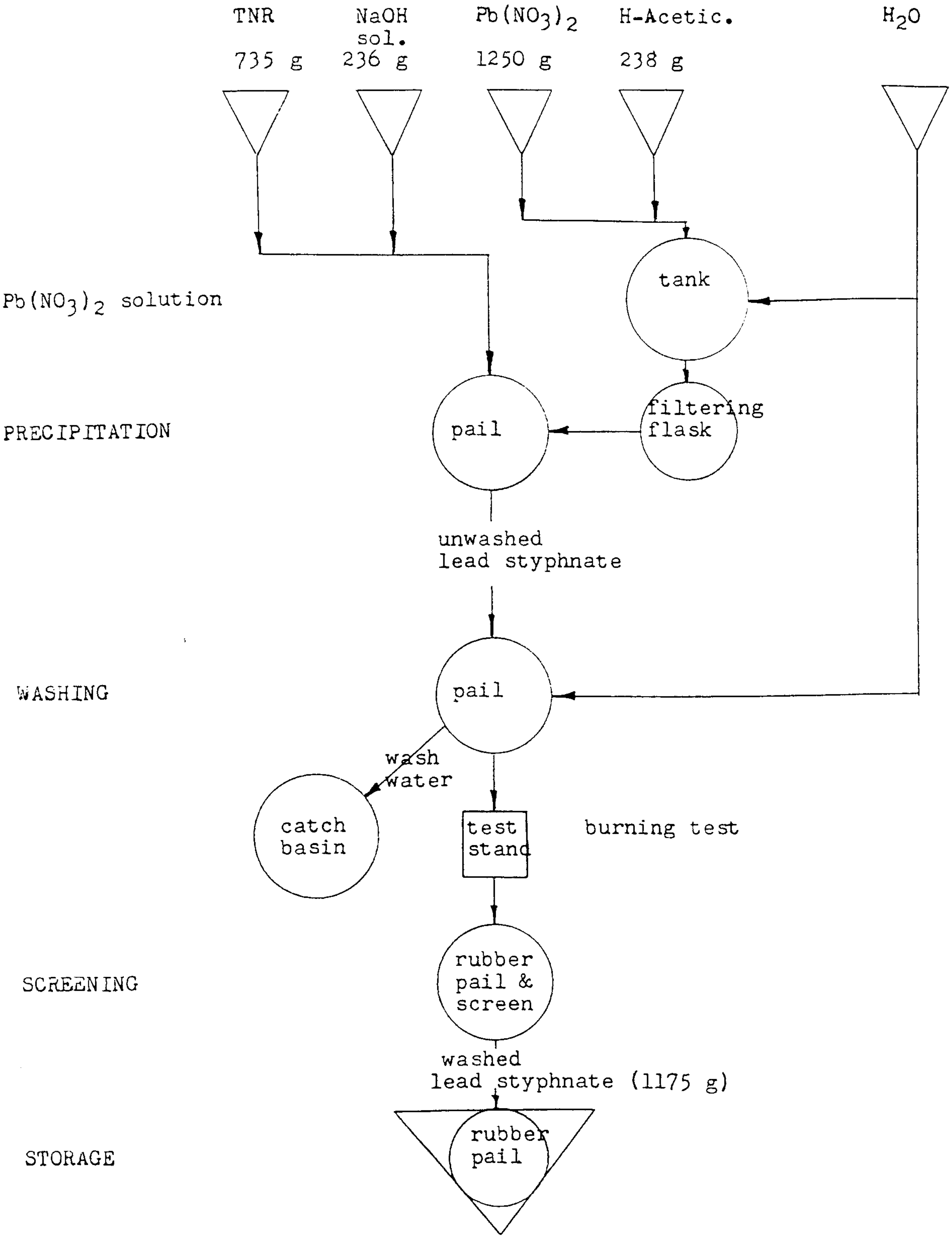
- Dissolve 250 gram NaOH in 250 ml water.
- Add water to 360 ml volume.
- Stir and cool thoroughly to ambient temperature before drawing a titration sample.
- The volume of NaOH solution for one pail batch of lead styphnate is:

$$\text{volume NaOH solution (ml)} = \frac{59000}{n \times V_a}$$

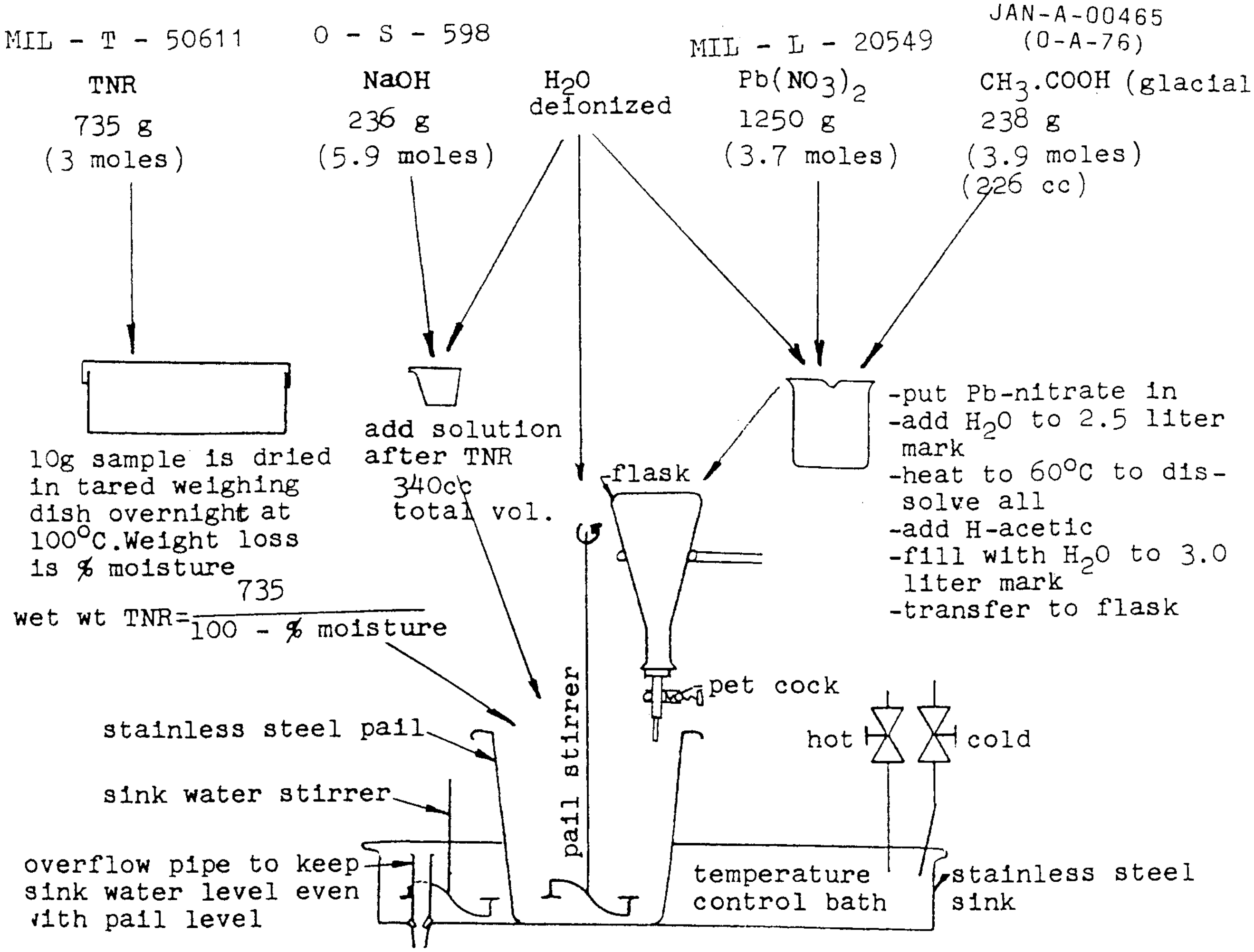
n = normality of standard acid
V_a = volume of standard acid in ml

Note: allow 3-5 ml extra over calculated volume to compensate for loss of solution on the beaker.

PROCESS DIAGRAM - ONE PAIL BATCH PREPARATION OF LEAD STYPHNATE



PROCESS DIAGRAM - ONE PAIL BATCH PREPARATION OF LEAD STYPHNATE



quantities for one pail batch
avg. yield is 1175 g LS

ONE PAIL BATCH
PREPARATION OF LEAD STYPHNATE

PRECIPITATION PROCEDURE

1 - Place 735 gram of TNR in clean stainless steel pail.
2 - Add cold tap water until the volume is at the 5200 cc mark.
3 - Recheck the volume with calibrated glass stirring rod.
4 - Place pail under the stirrer in the precipitation sink.
5 - Lower stirrer into pail and engage belt on pulley.
6 - Check that stirrer does not contact the bottom or walls of the pail.
7 - Place overflow tube into sink drain to maintain sink water level approximately. even with the pail contents level.
8 - Run hot water into the sink.
9 - Heat sink water to 55° - 60° C by opening steam valve and letting live steam into the sink water.
10 - Check that the sink water agitator is working, circulating the water through the large diameter pipe along the sink wall.
11 - Fill the flask lead nitrate solution to the 3000 cc mark by opening and closing the hose cock.
12 - Check the pail contents temperature periodically with the thermometer.
13 - When the pail contents temperature reaches 40° - 45° C, add the measured portion of NaOH solution. TNR reacts with NaCH forming a solution of Na-styphnate and the temperature rises to ± 55° C due to the heat of reaction.
14 - Open the hose cock on the flask outlet and let a small steady stream of lead nitrite solution into the pail. Approximately 30 minutes should lapse before the 3000 cc run in. As the lead nitrate solution runs into the pail the mixture becomes progressively more viscous until a gel forms and movement of the mixture caused by the stirrer stops.
15 - At this stage open the steam valve, heat the sink water to 70° C and close the steam valve.
16 - The gel formed is a highly hydrated form of lead styphnate. Under a microscope it appears as a fibrous mass of needle like crystals. With continuous addition of lead nitrate the gel breaks up and the mixture movement is resumed. By the time when all 3000 cc of lead nitrate solution runs into the pail, the mixture will be a free fluid with suspended dense crystalline monohydrated lead styphnate.
17 - Drain the hot water from the precipitation sink and fill sink with cold tap water as soon as all lead nitrate solution has been added. Such cooling increases lead styphnate yield which averages 1175 grams per pail batch.
18 - Continue stirring the mixture 5 minutes after all lead nitrate solution has been added into the pail.
19 - With a clean glass rod place a drop of pail liquid on a slide and examine under the microscope.

a) If gel is present, continue stirring 5-10 minutes longer, until the conversion is complete.
b) If no gel is present, the batch is ready for further handling.
20 - Lift the stirrer above the pail rim and engage in holding position.
21 - Lift the pail carefully from the precipitation sink and transfer to the wash sink.
22 - Allow the lead styphnate to settle at the bottom of pail.
23 - Carefully decant off the mother liquid.
24 - Refill the pail with a vigorous stream of water from the hose and agitate the settled solid with gloved hand at the same time so that all solids are in suspension.

PRECIPITATION PROCEDURE

25 - Repeat steps 22, 23, and 24 six (6) times.
26 - With gloved hand (NEVER use steel spatula, scoop or spoon) place a sample approximately 1-2 cc of lead styphnate into a labeled 30 cc beaker.
27 - Carry sample to laboratory for burning test.
batch in pail which passed the burning test is subjected to further handling.
Batch in pail which has been rejected in the burning test is rewashed several times and steps 26 and 27 repeated.
29 - Batch of lead styphnate rejected after repeat wash is scrapped immediately in accordance with safety procedures.
30 - Place a screen over clean empty rubber bucket.
31 - Acceptable lead styphnate from stainless steel pail is washed with a stream of cold tap water through the screen into the rubber storage bucket. Contents of four (4) steel pails is transferred into one rubber storage bucket. In this step any foreign matter or lead styphnate scale are removed from the lead styphnate.
32 - After the lead styphnate is transferred to the rubber storage bucket decant the water used in the transfer process leaving about 10 cm of water over the settled solids in the rubber bucket.
33 - Any solid material adhering to the side of the bucket is washed down with a little water.
34 - Cover the rubber bucket with the cover provided and label it with chalk indicating serial number, date and operator.
35 - Covered and labeled bucket is hand carried to lead styphnate storage magazine in building.
36 - As the lead styphnate is being transferred from the stainless steel pails to the rubber bucket, fill the pails with tap water immediately after transfer.

Notes:

- In practice only 2% of LS produced is scrapped after rewash.
- Low yield during process is mostly due to shortage of some reagent. Since lead nitrate is used in excess the shortage is either NaOH or TNR. The acetic acid will not affect the yield. In case of low yield check that the volume of NaOH is correct and all solutions are at 18^{0}C to prevent precipitation from saturate solution.

ONE PAIL BATCH
PREPARATION OF LEAD STYPHNATE

PRECIPITATION PROCEDURE

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EQUIPMENT - LEAD STYPHNATE PREPARATION

Notes:

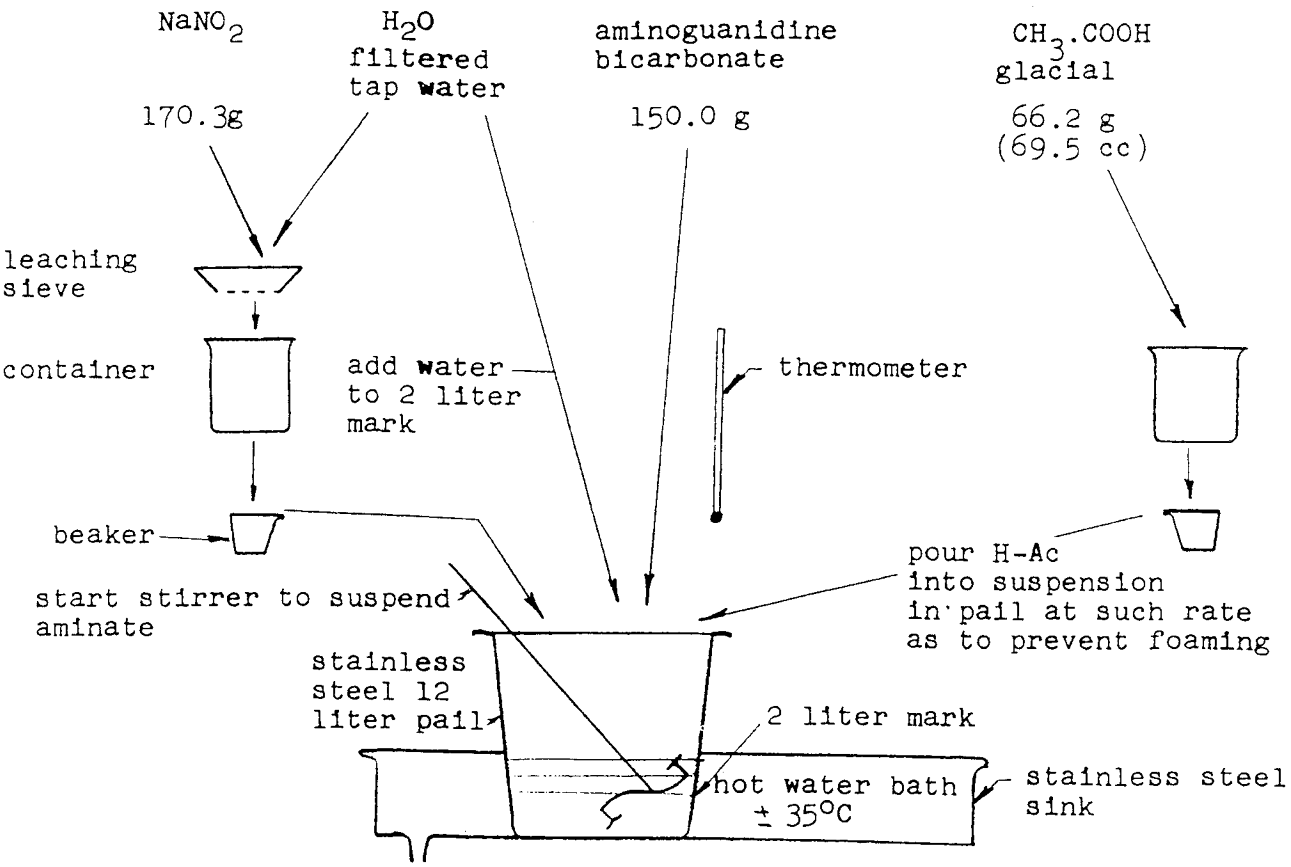
- When more than one pail/batch of product is precipitated at any one time, the corresponding required items should be multiples of the pail/batches.
- If the multibatch operation is to be continuous, the stirrer stands and the flask assembly stands should be located on the outside of the sink back wall;*). Furthermore, the sink itself should have a series of longitudinal baffles so that the temperature of the water in the sink will be uniform around all pail/batches. Additional stainless steel sink with hot and cold water taps will be needed for the washing of lead styphnate (the precipitation sink would be too crowded for safe operation).

*) To keep pails in proper location a set of 3 - 4 studs may be spot welded for each pail at the sink bottom.

ONE PAIL BATCH PREPARATION OF TETRACENE

material quantity for 100 g tetracene
at 96% theoretical yield

$C_2H_8N_{10}O$
mol.wt.188



1 - Place $NaNO_2$ on leaching sieve and add water (approx. 380 cc) to fully dissolve material.
2 - Filter the solution before pouring into beaker since any foreign matter in the solution would end in the priming mix.
3 - Place aminate in pail, add water to 2 liter mark and start stirrer since aminate is insoluble in water; put hot water into sink to heat pail contents to 30° ± 1° C.
4 - Add H-Acetic into pail at such rate as to prevent foaming over. The completion of aminate solution is indicated by the clearing of the solution and the end of CO_2 evolution.
5 - Add water to pail to the 2.5 liter mark.
6 - Add $NaNO_2$ solution.
7 - Add water to the 3.0 liter mark.
8 - Maintain temperature of the pail contents at 30 ± 1° C for 22-24 hrs.
9 - Wash tetracene as per standard washing procedure 8-10 times.

CONTINUED ON PAGE 33

Note: Presence of Cu ions prevents tetracene formation so that 0.08% Cu in the aminate reduces the yield from 96% to 5%. Fe, Ni, etc. have no appreciable effect even in relatively high concentrations.

10 – Washed tetracene tested for pH.
11 – Acceptable tetracene is labeled with date and lot number and stored in conductive rubber container.

Note: If aminoguanidine-acetate solution is not complete after addition of H-Ac, heat pail contents to 45° ± 2° C. Afterwards allow time for the solution to cool to 35° ± 1° C before next step.

The
Poor Man's Primer Manual

by

George Dmitrieff

George Dmitrieff, noted author of firearms and munitions publications has created another masterpiece for the discriminating hobbyist and munitions technicians. This work details the production of primers from a commercial prospective and then in the final chapter gives comprehensive instructions on how to produce small quantities of primers in a laboratory or home environment.

The book covers the process of creating the necessary materials (priming mixes), primer component reconditioning, and development and procurement of the basic tools. These topics are detailed in a manner that the student can readily understand both on a practical and academic level.

The author emphasizes that the processes described herein are dangerous due the sensitive nature of materials used. Furthermore, construction of these products may be subject to federal, state and local laws and regulations which require special licenses.

A Must Have Manual!!

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