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The Manufacture of Silver Azide R.D. 1336

G. W. C. Taylor

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February, 1950
MINISTRY OF SUPPLY

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT.


The Manufacture of Silver Azide R.D. 1336.

by

G. I. C. Taylor.

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Abstract.

A previous report R.R.D.L. No.17/R/49 "Silver Azide as a replacement for lead azide in detonators" described experiments carried out on the preparation and properties of this substance and discussed its possible development as an initiator for Service detonators. The results justified the recommendations for further trials on performance and stability and also for the continued development of the method of preparation.

As a result of these further investigations it is now possible to describe a detailed manufacturing process for silver azide; this is the object of the present report.

The original R.D.1336 method has been modified to give a product of larger primary crystal size in the form of free-flowing aggregates. The process is essentially the simultaneous addition of sodium azide and silver nitrate solutions to a vigorously stirred base solution of dilute ammonia; this is followed by the slow addition of dilute nitric acid until the mother liquors are almost neutral. The crystals as formed in the first stage are reproduced in the second stage giving a virtually unchanged product. By this means a yield of over 94 per cent is obtained. The temperature of all solutions need not exceed 20°C.

The manufacturing procedure has been evolved to use, with the minimum of modifications, the facilities already existing in the filling factories for the production of lead azide. The batch size is 3 lb. and the arrangements for washing, drying and sieving the product are similar to those in use for lead azide. A method is described for the safe chemical destruction of silver azide with recovery of the silver as the chloride.

Some of the properties of R.R.D.L.1336 are given. It is highly resistant to hydrolysis and has superior initiating properties to lead azide. The product as made by this process is silver azide of high purity as no additive is incorporated.

This report is confined to the manufacturing method and brief outline of properties; an account of the development of the process will be given in a complementary report.

Introduction and Object of Investigation.

The evidence in support of the active development of silver azide as a replacement for lead azide in detonators was set out in the previous report (B.R.D.L. No. 17/3/49). It was shown that silver azide could be prepared in a granular free-flowing form which was markedly resistant to hydrolysis in the presence of water and water vapour; this product was less sensitive than mercury fulminate. Recommendations were (1) performance and stability trials to be carried out, (2) the special explosive properties of silver azide to be considered for Service application, (3) a manufacturing process to be developed. The present report is concerned with the latter recommendation and details a method of preparation suitable for adoption in the filling factories. An account of the stages in the development of the method will be given in a following report.
Methods employed.

Silver azide has a very low solubility in water and therefore precipitates in the form of a voluminous and unsatisfactory product when prepared by ordinary methods such as direct interaction of sodium azide solution with a soluble silver salt. The previous report described the simultaneous addition of sodium azide and silver nitrate solutions to a vigorously stirred base solution of dilute sodium hydroxide in order to obtain a free-flowing granular form of silver azide. The granules consisted of a large number of very small crystals, it being intended to limit the primary crystal size and thus avoid highly sensitive material. Sensitiveness tests showed that this had been achieved but performance tests for initiating power carried out by C.S.A.K. were unsatisfactory. It was therefore decided to modify the process to give rather larger crystal size, subject to a close check on sensitiveness.

By substituting ammonium hydroxide for sodium hydroxide in the base solution the necessary increase in solubility to give larger crystals was obtained. The factors influencing crystal size and habit and aggregate formation were studied. The increased solubility imparted by the use of ammonium hydroxide resulted in a proportion of the silver being retained in solution at the end of the simultaneous addition. This gave rise to two problems, (1) the relatively low yield and (2) the treatment and disposal of ammonical silver solutions which are known to constitute explosion hazards under certain conditions. Finally these objections were overcome by following the simultaneous addition stage by the slow addition of dilute nitric acid to the mother liquor until nearly neutral.

The ultimate 3 lb. process was thus evolved and confirmed by a series of 6 batches manufactured on the experimental initiator plant at Woolwich.

The essential features of this process are good agitation and relatively slow development of the crystals; no heated solutions are necessary and 20°C. is the effective temperature at all stages. This avoids possible difficulties due to the volatility of ammonia, hydrazoic acid and ammonium azide.

Every effort has been made to adopt the facilities and follow the procedure in use in the filling factories for the production of service lead azide; in the detailed Appendices of this report attention is drawn to the differences from this procedure.

The marked chemical stability of silver azide caused some difficulty in evolving a method for its safe destruction; the standard lead azide method using sodium nitrite and acetic acid was unsuitable because of the low solubility of silver nitrite which is freely formed. By substituting nitric acid for acetic acid, which decomposes silver nitrite much more readily, a satisfactory process was worked out. It should be noted however that chemical destruction of silver azide has less latitude than that of lead azide. By treatment of the liquors with sodium chloride the silver may be recovered as silver chloride.

Results and conclusions.

By using the process described silver azide is produced in the form of free-flowing aggregate approximately 0.1 mm. in size. Silver azide has a strong tendency to crystallise in acicular habit.
habit but by this method the individual crystals in the aggregates are evenly developed to approximately 0.02 mm. with no pronounced growth on any face. Reproduction of the process and the product has been good over the series of six plant batches.

It is proposed to retain the designation of R.D. 1336 for the silver azide made by this method and sufficient results are now available to give its main properties. Its marked resistance to hydrolysis has been confirmed and chemical analyses indicate that R.D.1336 is virtually pure silver azide which is to be expected in a process where an additive is not included in the final product. Its initiating efficiency is superior to that of Service lead azide. Preliminary results of compatibility trials indicate no reaction with copper, aluminium, tin or nickel under dry conditions and under moist conditions only copper exhibits adverse effects. Sensitiveness tests on R.D. 1336 show, in general, that its characteristics are similar to those of lead azide. Electrostatic tests indicate that a relatively high energy spark is required for ignition, thus R.D. 1336 is not considered to present undue risks in handling. It is emphasized that considerable work is in progress in E.R.D.L. and A.R.L. on these and other properties of R.D. 1336 which cannot therefore be authoritatively expressed in the present report.

Although the standard lead azide plant can be used for the production of R.D. 1336 the process time is longer because of the necessity for slow crystallisation to avoid very small and also acicular crystal formation. This time could only be reduced at the sacrifice of yield or by precipitating at a higher temperature; both expedients are considered inadvisable. It is also possible to improve further the running properties of R.D. 1336 by more rapid drying and also by washing the product with a hydrophobic solvent such as petroleum ether; it is thought that the filling factories would wish to avoid these procedures and they have therefore been omitted. With the recommended processing the bulk density of the product approximates to 1.6 gm/ml, but it is possible that continued manufacture may extend the upward trend which has been observed in the trial runs.

From the viewpoint of crystallisation theory and practice the most important conclusion relates to the reproduction of the crystal growth pattern in the second stage of the precipitation. This occurs rather remarkably despite the decreasing solubility of silver azide in the mother liquor as the ammonia is converted to ammonium nitrate. This will be discussed more fully in the development report.

Recommendations.

It is recommended that the process as described should be adopted for the manufacture of silver azide to give a detonating composition, designated R.D. 1336, of superior initiating power and greater resistance to hydrolysis than lead azide.

Acknowledgments.

To all members of the Initiator Development Group S.E.I., E.R.D.L. Woolwich, who have worked as a team to develop R.D. 1336 to the manufacturing scale in as short a time as possible.
Appendix I. Description of Plant.

1. Plant for solutions.

(a) Silver nitrate. Silver nitrate solution is affected on storage particularly in the presence of light; and it is therefore proposed that the required quantity of silver nitrate crystals should be weighed out and made into solution before each batch of silver azide is prepared. The solution should be filtered into an aspirator made of brown glass or suitably darkened before use.

(b) Sodium azide. The solution is prepared in a 25 gallon stainless steel tank, fitted with a stirrer operated by a compressed air motor. A streamline filter and 25 gallon storage tank is required. An exhaust fan adjacent to the tanks is necessary. This equipment is as for lead azide production.

Note: The storage and handling of silver nitrate and other chemicals such as nitric acid should be carried out in a building away from the possibility of contact with sodium azide.

(c) No special equipment is necessary for the preparation of ammonia and nitric acid solutions. Strong ammonia should only be handled under conditions of good ventilation. The use of glass measuring vessels and containers necessitates great care to avoid breakages and contamination by glass fragments. All solutions including distilled water and methylated spirit must be filtered before use; hardened fast filter papers of the Whatman No. 841 type are suitable for general use with gravity filter funnels.

2. Plant for precipitation.

The service lead azide building unit is suitable i.e. fore-room separated by blast walls from precipitating chamber adjacent to motor house. Except for special operations, manufacture is carried out from behind the blast walls, with observation by means of suitably placed mirrors. An exhaust fan must be fitted to the precipitation chamber. Precipitation is carried out in a 10 gallon stainless steel tilting pan fitted with a rubber covered stirrer rotating at a fixed speed of 77 ± 2 r.p.m., the clearance below the stirrer being 2 inches. In use the pan is kept tilted approximately 5° away from the vertical, corresponding to one half turn of the tilting wheel.

The contents of the pan are maintained at 20°C (± 2°) by water circulated by a rotary pump through the pan jacket from a steam heated and thermostatically controlled calorifier. A thermometer is fitted in the circulating stream with its diaphragm so that the temperature of the water entering the pan throughout the precipitation is recorded. The precipitating building should be provided with a sump large enough to take the mother liquor and wash liquors, i.e. approximately 50 gallon capacity with filter before discharge into the drain. The reacting solutions are contained in glass aspirators fitted with constant head devices, and situated in the fore-room. The solutions are run into the pan through cloth filters and gutta percha tubes passing through the blast wall leading into gutta percha feed funnels in the precipitating pan. The feed funnels are mounted in the pan with their lower outlets just touching the surface when 10 litres of liquid are in the pan, and located 3/4 inch from the pan wall at the opposite ends of a diameter 90° from the pan lip. For silver azide it is recommended that the feed funnels should have a bore
of $\frac{3}{8}$" (for lead azide the bore is only $\frac{1}{4}$").

After the final washing, the pan is tilted to its full extent and the precipitate is discharged into gutta percha drying pots, by water from a gutta percha spray tube fitted inside the rim of the pan 2 inches below the top. The two-handled drying pot has a perforated false bottom covered with a cambric disc and held in position by a gutta percha ring. The R.D. 1336 is retained on the cambric, and the liquors pass through into the lower part which is provided with an outlet leading to a suitable trap.

3. Plant for drying and sieving.

The cold air drying method as used for Service lead azide is employed. Approximately $1\frac{1}{2}$ lb. of wet R.D. 1336 is washed with industrial methylated spirit and cold air is passed through by means of a water operated ejector from a train of sodium hydroxide drying towers. The volume of air is checked by a gas-meter.

The standard aluminium alloy sieving machine as used for Service lead azide is employed, substituting a 40 B.S.S. stainless steel mesh for the silk mesh of 100 B.S.S. aperture.

/Appendix II.
## Appendix II. Materials required.

<table>
<thead>
<tr>
<th>Material</th>
<th>Spec. No. or description.</th>
<th>Qty. for one 3 lb. batch</th>
<th>Approx. quantities for 12 batches.</th>
<th>Remarks.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Sodium Azide</td>
<td>CS778A</td>
<td>650</td>
<td>7.8</td>
<td>17.2 As for lead azide.</td>
</tr>
<tr>
<td>2. Silver nitrate</td>
<td>Pure</td>
<td>1700</td>
<td>20.4</td>
<td>45</td>
</tr>
<tr>
<td>3. Ammonia</td>
<td>368</td>
<td>4.42</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4. Nitric Acid</td>
<td>1.42 pure</td>
<td>1155</td>
<td>13.4</td>
<td>30.5 As for lead azide drying.</td>
</tr>
<tr>
<td>5. Ind.-Methylated spirit</td>
<td>1128 litres</td>
<td>2.0</td>
<td></td>
<td>6</td>
</tr>
<tr>
<td>6. Distilled water</td>
<td>200</td>
<td>2400</td>
<td>585</td>
<td></td>
</tr>
<tr>
<td>7. Sodium Hydroxide sticks</td>
<td>5 lb.</td>
<td></td>
<td></td>
<td>20 For drying towers.</td>
</tr>
</tbody>
</table>

For destroying 1 lb. of silver azide.

<table>
<thead>
<tr>
<th>Material</th>
<th>Spec. No. or description.</th>
<th>Qty. for one 3 lb. batch</th>
<th>Approx. quantities for 12 batches.</th>
<th>Remarks.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Sodium nitrate</td>
<td>As for lead azide destruction</td>
<td>600</td>
<td></td>
<td>Small quantities of solutions of these used for destroying liquors etc. after precipitation.</td>
</tr>
<tr>
<td>2. Nitric acid</td>
<td>1.42 pure</td>
<td>560</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3. Sodium chloride</td>
<td>pure</td>
<td>200</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

In the above tables no attempt has been made to estimate the cost of these materials. Except for silver nitrate, information from the filling factories is available from D.O.F.(F) and the Chief Chemical Inspector. Silver nitrate purchased by C.S./E.R.D.E. cost 90/- per 500 gms. (November 1949).
1. Precipitation of AgNO₃

Ammonia
368 gms. NH₃ (98% solution estimated and calculated volume used)

Water to 16.87 litres

Temp. 20°C.

Sodium Acetate Solution
10 litres of 68 gms/litre
Temp. 20°C.

Nitric Acid Solution
10.0 litres of 110 gms/litre
Temp. 20°C. added in 15 mins.

Distilled Water
1st wash 35 litres

Distilled Water
2nd wash 35 litres

Distilled Water
3rd wash 35 litres

Water Spray
Batch divided between 2 pots

Silver Nitrate Solution
10 litres of 170 gms/litre
Temp. 20°C. added in 9 hr 140 mins. simultaneously with sodium acetate
Stirring continued a further 5 mins. at 20°C
Stirring continued a further 5 mins.

Decant mother liquor
NH₃ Content = 17 gms.
Ag Content = 22 gms.
Stirred 5 mins.
sediment 2 mins.

Decant

Distilled Water

Stirred 5 mins.
sediment 2 mins.

Decant

Distilled Water

Stirred 5 mins.
sediment 2 mins.

Decant

Water Spray

Batch divided between 2 pots

Sodium Nitrite
150 gms/litre.
2 litre of solution.

Nitric Acid
80 gms/litre.
2 litre of solution.

FILTRATE

FILTER

DRAIN

--- Appendix III (cont) ---
Appendix III. Flow Sheet (Cont)

2. Drying and Sieving of EDL336

\[ \frac{1}{2} \text{ batch (approx. 1 lb.)} \]

Industrial methylated spirit
200 ml.
(2 washings)

\[ \text{Contaminated solns.} \quad \text{Trap} \quad \text{destroyed} \]

Drying pet placed on tilting arm of sieve
EDL336 tipped on to sieve and collected
in six receiving pots after vibrating approx.
10 seconds.

\[ \text{Sieving} \]
\[ 40 \text{ B.S.} \]

Final Product EDL336
Yield 1,400 gms. (4 lb. 2 ccs.)
(94% theoretical)
A. Preparation of sodium azide solution.

1. Add 24 lb. of sodium azide to 95 litres of distilled water and stir until dissolved.
2. Draw through streamline filter into the storage tank.
3. Adjust the concentration by the addition of distilled water to a density of 1.0644 gms./ml. (108 gms./litre).
4. Dilute to a concentration of 65 gms./litre for use.

B. Preparation of silver nitrate solution.

1. Weigh out 2C4G gms. of silver nitrate to ± 1 gms.
2. Dissolve the solid silver nitrate in about 3 litres of warm water (35°C) and filter.
3. Make up the volume to 12 litres with distilled water, giving a concentration of 170 gms./litre. This allows 10 litres to be run off for the batch of silver azide.

NOTE: As soon as the silver nitrate solution has been made it should be kept in a brown glass vessel.

C. Preparation of ammonia solution.

The stock .880 ammonia is estimated in the following manner:

1. Dilute 10 mls. of stock solution to 250 mls. with water.
2. Take 5 mls. of the diluted solution and add 50 mls. of N/10 hydrochloric acid solution.
3. Back titrate the excess hydrochloric acid with N/10 sodium hydroxide using methyl orange as indicator.

1 ml. N/10 HCL = 0.0017 gms. NH₃

1 litre of .880 ammonia usually contains 250 - 280 gms. NH₃.

D. Preparation of nitric acid solution.

1. Dilute 1.35 litres of nitric acid of specific gravity 1.42 to a volume of 12 litres.
2. Take 20 mls. of this solution and titrate with N. sodium hydroxide solution using phenol phthalein as indicator.
3. Adjust the concentration by the addition of distilled water to 110 gms. HNO₃/litre.

/End Precipitation
1. Add stock .850 ammonic solution and distilled water to the precipitating pen to give 366 gms. NH₃ in a final volume of 16.57 litres.

2. Tilt the pen 1/2 turn of the tilting handle.

3. Start stirrer at 77 ± 2 r.p.m. and adjust the temperature to 20°C. (± 2°C) by circulating water from the colorifier at 20°C. through the jacket.

4. Adjust the temperature of the sodium azide and silver nitrate solutions to 20°C. (± 2°C).

5. From calibrated aspirators add simultaneously 3.0 litres of 170 gms./litre silver nitrate solution and 3.0 litres of 65 gms./litre sodium azide solution, through the gutta percha feeds during 9 minutes.

6. Then add simultaneously during 140 minutes the remaining 7 litres of each solution.

7. Continue stirring for a further 5 minutes.

8. Add 10 litres of 110 gms./litre nitric acid solution adjusted to 20°C. (± 2°C) at the rate of 1 litre during 15 minutes (i.e. during approx. 150 minutes). At the end of this addition, the pan liquor should be slightly alkaline when tested with a litmus paper.

9. Continue stirring for further 5 minutes.

10. Stop stirrer and allow to settle for 2 minutes.

11. Tilt pen and decant mother liquor through a filter box fitted with a cambric filter cloth into the sump.

12. Add 35 litres of distilled water to the precipitating pen through the gutta percha spray. Stir 5 minutes, settle 2 minutes, and decant the wash liquor through the filter box into the sump.

13. Repeat the washing twice more.

14. Replace filter box by a gutta percha drying pot fitted with a large gutta percha funnel and tilt pen to its full extent.

15. Wash as nearly as possible one half of the R.D.1336 into the drying pot by means of the water spray, the wash water running through a tube into the sump. Draining is completed by vacuum, not exceeding 10 cms. of mercury drawing excess water into a trap.

16. Wash the precipitate with 500 mls. filtered industrial methyl ted spirit, connecting the gutta percha tube from the drying pot to an aspirator outside the precipitating chamber, and using a vacuum of not more than 10 cms. of mercury to remove the contaminated methyl-iod spirit. Repeat washing with a further 500 mls. meths.

- 10 -
17. Remove the drying pot to the drying compartment.

18. Repeat process washing the other half of the R.D. 1336 into a second drying pot, and washing with meths.

F. **Drying of R.D. 1336.**

1. Connect drying pot to drying train in order of (i) gas meter, (ii) tower containing sodium hydroxide solution, (iii) two towers containing sodium hydroxide sticks, (iv) tower with cotton wool filter, (v) drying pot, (vi) gutta percha trap, (vii) vacuum connection.

2. Pass a minimum of 300 cubic feet of dry air at a rate of not more than 20 cubic feet per hour through each pot.

G. **Sieving of R.D. 1336.**

1. Arrange six papier machie receiving pots on the sliding tray.

2. Withdraw the pots under the sieve and transfer an open drying pot to the rack of the sieving unit.

3. Invert the drying pot rack from the foreroom by giving the tilting arm a sharp jerk, completely emptying the R.D. 1336 on to the sieve.

4. Operate the vibrator from the foreroom and sieve for 40 secs.

5. After 5 minutes, the operator may enter the sieving compartment and observe whether all the composition has passed through the sieve.

6. If any composition is still left on the sieve, repeat the vibration (normally one period of vibration will be sufficient).

7. When all the composition has been sieved, the operator must allow at least 15 minutes before touching any part of the apparatus or pots.

8. The pots are taken off one at a time, brushed gently to remove any powder on the outside, placed in a carrying box and removed to the magazine.

9. Place a further six receiving pots on to the receiving tray and repeat the operations with the second drying pot.

10. After all the composition has been removed from the sieving room the operator may remove the residue on the sieve by means of a low vacuum through a rubber ended gutta percha tube connected to a gutta percha trap containing water.

11. Remove any spilt composition by means of damp cotton wool swabs.

/Appendix 5.
The Chemical Destruction of Silver Azide.

Silver azide is more difficult to destroy chemically than lead azide. Firstly, it is more resistant to acid hydrolysis and secondly, when treated with sodium nitrite solution a soluble silver salt will give a flocculent yellow precipitate of silver nitrite. Thus when sodium nitrite and acetic acid, as used for lead azide destruction, are added to silver azide, the insoluble silver nitrite is formed. Silver nitrite is, however, soluble in nitric acid and by using nitric acid in place of acetic acid it is possible to destroy silver azide satisfactorily.

During the course of this destruction silver nitrite is formed and then decomposed by excess nitric acid and this process must be carefully controlled to avoid excess nitric acid being added to the unchanged silver azide which might result in the liberation of hydrazoic acid. An excess of sodium nitrite is therefore maintained during the breaking down of the azide radicle: the subsequent addition of the excess of nitric acid converts the silver nitrite to silver nitrate, leaving a clear solution.

The silver in the solution is recovered by treatment with sodium chloride to convert to the insoluble silver chloride which is then filtered off. The stages of destruction may be carried out in stainless steel vessels as used for lead azide but chlorides should not be allowed to be in contact with this metal.

It is emphasized that in destroying silver azide there is considerably less latitude in the operation as compared with lead azide destruction. This is particularly important when bulk quantities are being handled. If the following process instructions are carried out there should be no difficulty in avoiding both incomplete destruction of silver azide and the liberation of hydrazoic acid.

It is also important to note that the physical form of the silver azide can affect the ease of chemical destruction. For example, if silver azide is dissolved and precipitated out from ammonia solution it may be destroyed more readily. The process instructions refer particularly to silver azide in the physical form of R.D. 1336.

Because of the considerably higher commercial value of silver as compared with lead, it is recommended that waste silver azide should be chemically destroyed rather than burnt in order to recover the silver.

A. Solutions required.

Nitric Acid: 200 ml. of nitric acid e.g. 1.42 is diluted for each litre as used for destroying.

Sodium Nitrite: As for lead azide destruction. Used at 150 gm/litre. This solution should be filtered before use.
B. Destruction of remaining silver azide in the pan after the removal of the batch.

1. Add about 45 litres of distilled water through the feed tubes and start stirring. The fan must be on throughout this operation.
2. Add 2 litres of sodium nitrite solution through the central destroying feed as used for lead azide.
3. Add 2 litres of nitric acid through the same feed during a period of about 15 minutes.
4. Inspect after 30 minutes when all solid should have disappeared.
5. When solution clear, discharge into sump.

C. Treatment of mother and wash liquors.

1. After the previous operation add 2 litres of sodium nitrite solution to the sump followed by the gradual addition of 2 litres of nitric acid.
2. After standing for at least 1 hour the liquor may be discharged through the filter into the drain.

D. Destruction of R.D. 1336 in bulk.

The following refer to the destruction of 1 lb. of R.D. 1336 in the precipitating pan. The quantities may be modified as required for more (up to 3 lb. in the case of a complete batch) or less, as when using a smaller vessel such as the lead azide type destroying pan. There is some liberation of nitrous fumes and good ventilation is essential.

1. Pour 15 litres of distilled water into the precipitating pan and carefully add the R.D. 1336.
2. Retire behind the baffle wall, and start stirrer, and after 5 minutes add 2 litres of sodium nitrite solution.
3. Add simultaneously, through the feed tubes, 2 litres each of sodium nitrite and nitric acid solutions. This operation should take 40 minutes.
4. Continue the addition of a further 2 litres of nitric acid only, during 40 minutes, and continue stirring for 30 minutes.
5. A clear solution should then be obtained, if not, stirring is continued.
6. Filter the solution through a fine cambric filter cloth into an earthenware or other vessel (not stainless steel).
7. Add with hand stirring one litre of sodium chloride solution containing 200 grm. NaCl.
8. Allow to settle, preferably for at least 4 hours, and filter through a fine filter cloth. The filtrate should show no precipitate on the addition of further sodium chloride solution.
9. The filtrate is washed into the drain and the silver chloride on the filter is washed with water and dried. A sample should be tested in a flame to confirm complete absence of explosive material. The yield is 400 grams equivalent to 92 per cent on the silver.

E. Washing of utensils etc., after preparation of R.D. 1336.

1. Funnels, filter cloths and swabs may be soaked in weak destroying solution, washed with distilled water and used again.

2. The methylated spirit washes are mixed with excess aqueous liquors for destroying with solutions as for these liquors. Alternatively the spirit washes may be collected for burning.

3. Drying pots are treated with weak destroying solution then thoroughly washed before use again.

4. It should be noted that the use of tap-water should be avoided throughout the manufacture and destruction of silver azide because of the formation of insoluble silver salt precipitates.

Appendix 6.
Summary of Properties of R.D. 1336.

Some of the properties of silver azide were described in the previous report, and, in general, this data can also refer to R.D. 1336 prepared by the present method. It should be remarked that considerable work is at present in progress (January 1956) in E.R.D.E. and ...R.E. on the examination of typical R.D. 1336 plant products and the results indicated in this appendix should therefore be regarded as preliminary.

1. Physical Properties.

R.D. 1336 is in the form of white granules consisting of irregular aggregates of a particle size of the order of 0.1 mm. The individual crystals in the aggregates are approximately 0.02 mm. in size. Typical appearance is shown in Figs. 1 - 6 which also indicate the growth in the two stages of precipitation. The crystals are orthorhombic and are free from pronounced growth on any faces, for example, the product is free from acicular crystals. It is important that this general crystal size and shape should be maintained in production as it is known that very small crystals and also acicular habit will result in lower initiating efficiency.

R.D. 1336 has a density of 4.98 gm./ml. This is based on a series of 17 determinations giving results ± 0.03, thus indicating consistency of the product and freedom from voids and mother liquor inclusions. The apparent bulk density of the latest plant batch (XT49) is 0.96 gm./ml. as determined by the standard lead azide/butyl alcohol method. R.D. 1336 has therefore a slightly higher true density and appreciably lower apparent bulk density than Service lead azide. It is possible that on continued production higher bulk density would be achieved; this in fact was the experience when Service lead azide was introduced.

R.D. 1336 flows freely but owing to its aggregated nature its manipulation appears different from that of single crystal forms of lead azide and lead styphnate. Its flowing properties are rather more critical than lead azide, for example, more rapid drying or washing with a hydrophobic solvent such as petroleum ether will give the best flowing material. Using the procedure given in this report, however, the typical product may be handled satisfactorily in the smallest scoops likely to be encountered in filling.

R.D. 1336 is discoloured by exposure to strong light as is lead azide, but there is no evidence that this has any deleterious effect.

2. Chemical Properties.

R.D. 1336 is a very pure form of silver azide as shown by the following analytical results: --
The compilation of a chemical specification is in progress by S.P.R.I., E.R.D.E.

Its chemical destruction has already been described in the previous appendix.

The results of hydrolysis tests on R.D. 1336 have followed those given in the previous report. Under very drastic test conditions Lot XT.44 has shown no hydrolysis with the copper strip method, namely, sample moistened with 60ºC water and subjected to 80ºC for 3 days (S.P.R.I. test). Under milder test conditions representation samples kept either moistened with water or in 100% relative humidity for 7 weeks over fluctuating 10ºC - 22ºC, showed no sign of hydrolysis. Under similar test conditions the above lead azide gives pronounced deposits of copper azide.

Compatibility tests in progress on silver azide indicate no reaction with copper, aluminium, tin or nickel under dry conditions. Under moist conditions only copper exhibits adverse effects. These tests are being continued by S.P.R.I. and also in connection with filled detonators by ...R.E.


R.D. 1336 is of the same order of sensitivity to impact as service lead azide. Figure of insensitivity of typical batch (Lot XT.44) = 25. R.D. 1336 does not appear to be more sensitive to friction than lead azide.

Tests in progress by S...E., A.R.E., indicate that R.D. 1336 has unusual insensitiveness characteristics when wetted with water. Thus although its impact sensitivity is not reduced when wet, its friction sensitivity is decidedly less and it will not ignite on soft wood. Its propagating powers are less when wetted and R.D. 1336 therefore appears to be safer for transport in the wet condition. On the conclusion of the present tests a decision will be made regarding recommended transport procedure.

Silver azide of the R.D. 1336 crystalline type presses well into sleeves and has superior initiating power as compared with service lead azide. This superiority increases with increasing diameter of the column. Tests are in progress by S...E., A.R.E. Tests on electrostatic properties have been carried out in E.R.D.E., ignition only occurs with sparks of high energy and it is considered that there are no exceptional electrostatic risks associated with this material.

During the course of development and manufacture of R.D. 1336
many batches have been prepared and handled without incident. It is therefore considered that the production and use of this initiator can be confidently embarked upon with this experience already available. At the same time it is to be remembered that R.D. 1336 is a new composition which belongs to the most sensitive class of explosives and only continued production for a long period may reveal all its characteristics in manufacture and application.