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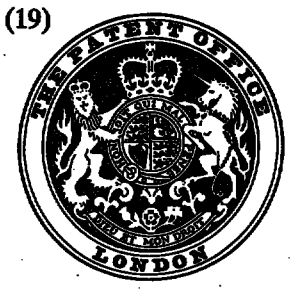

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(54) METHODS OF PRIMING EXPLOSIVE DEVICES

(71) We, IMI KYNOCH LIMITED, formerly known as IMPERIAL METAL INDUSTRIES (KYNOCH) LIMITED, a British Company, of Kynoch Works, Witton, Birmingham B6 7BA do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to the priming of explosive devices, that is to say the incorporation of priming explosive with elements of priming-explosive utilizing devices.

In current practice, primary explosive for use in priming of explosive devices (e.g. ammunition cartridges and detonators) is manufactured away from the priming zone in a large bath or at the maximum possible continuous rate and is then stored. In the case of ammunition, at the priming stage, primary explosive is drawn from store and is mixed with one or more other ingredients of the priming explosive in a substantial batch which is then distributed between elements of the devices (e.g. cap shells, cartridge cases or detonator cases). This sequence involves bulk storage, transport to a mixing station, the mixing operation itself, transport of the mixture to the priming zone, and distribution between said elements. Since primary explosives are inherently dangerous to manufacture and handle, each stage of the sequence is hazardous and needs special precautions, particularly where explosive is present in bulk. However, in the case of detonators, certain primary explosives may be used alone as the priming explosive, in which case mixing of the primary explosive with one or more other ingredients is unnecessary. Nevertheless, the bulk storage and distribution aspects alone are very hazardous.

According to the present invention there is provided a method of priming a plurality of explosive devices by incorporating a discrete body of priming explosive with an element of each device wherein the priming explosive is produced as hereinafter defined, at a rate that is, as hereinafter defined, continuous with the incorporation thereof with the elements of said devices.

By the term "continuous", there is meant that the priming explosive is produced at such a rate that there is substantially no off-line storage of bulk priming explosive.

The priming explosive may be produced in a batch sufficient for a plurality of individual utilisation demands, but small relative to conventional batches. In order to satisfy total output demand over a period, a series of such batches may be produced in the period and these batches can be separated by a distance and/or in a manner facilitating isolation of an explosion in any one batch. The batches can be produced in regular succession, for example at predetermined time intervals, thereby facilitating adequate spacing and subsequent handling.

The maximum permissible size of each batch will depend upon the type of priming explosive and the conditions under which it has to be produced. The sensitivity, explosive energy, required production conditions and divisibility for use of the explosive will all affect the batch size. The total output demand per unit time will also influence the batch size, as will the precautions taken to isolate individual batches. Conventional prim-

ing explosive for rimfire cartridges includes lead styphnate as primary explosive: a batch of styphnate sufficient for, say, 20 priming charges for rimfire cartridges could be made and divided between the rimfire cases in a continuous operation: corresponding arrangements could be made for priming shotgun cap shells and detonator cases. Larger batches will obviously involve extra precautions, but batches as large as several ounces could be produced at intervals of about one minute.

Batches may also be combined for an individual utilisation demand. However, neither combination nor division of batches is preferred, since both incur additional hazards. The preferred arrangement is one in which a batch is matched to an individual utilisation demand, for example each batch in a series is just sufficient for a priming charge for one rimfire cartridge, shotgun cap or detonator and in this arrangement the priming explosive is preferably produced in situ, for example in an element of a utilizing device.

For the avoidance of doubt, the following explanation is given for the terms "utilising device" and "production in situ":-

Utilising device - refers to a combination of at least two elements of an explosive device, at least one of which elements is a discrete body of said priming explosive. The other element may be a mere container or carrier for the explosive, such as a rimfire cartridge case, a detonator case or a cap shell. The utilising device may be a finished explosive device, but it is more likely to be only partly finished; for example a) further elements may have to be added to it to make up the explosive device, such as propellant and a bullet for a rimfire cartridge, or an electric match or a fuse for a detonator; b) the elements may have to be reshaped, or relocated relative to each other as when priming explosive in a rimfire cartridge case is forced into the rim of the case by a conventional spinning punch.

Production in situ - means that the priming explosive is produced in relation to at least one other element so as to provide said combination. Where the other element is a container, the priming explosive will normally be produced within it. If the other element were a carrier, the priming explosive could be produced as a body around a portion of the carrier. As indicated above, producing in situ does not necessarily imply that the combination is immediately ready for final use.

The explosive may be produced in a stream and the stream may be separated into successive "lengths" by explosion barriers.

The term "continuous" as defined above does not necessarily imply immediate utilisation of the priming explosive in priming; in practice, there will inevitably be a degree of "live storage" in cases where production is not effected in situ and this live storage can be adjusted as required to fit production circumstances. However, there will be a continuous flow path between the production and utilisation stages, so that off-line storage of dangerous substances is substantially eliminated. To this end, the production rate and the utilisation rate are matched or substantially matched, at least on average over a period.

A production process in accordance with the invention is preferably automated. The materials required for production of the priming explosive may be automatically metered into a stream or into batches under controlled thermal conditions, even where small quantities are required. Preferably, too, an automated production process is integrated with an automated utilisation stage producing at least a partially finished explosive device; for example, in the case of rimfire cartridge priming, priming explosive production can be continuous with an automatic line for receiving primed cases, loading propellant, and inserting the bullet.

The priming explosive should be sensitive to each or any of heat, friction, flame, electric spark, percussion or other predetermined initiating event. The explosive may be designed to produce heat or flash, rather than substantial quantities of gas, or a detonating shock. Heat and flash producing explosives may be required for initiation of propellants, while shock producing explosives will generally be required for initiation of secondary high explosives such as base charges of detonators.

The following explanation is given of terminology used in this specification hereinafter:-

a) the word "material" is used in a general sense,

b) the word "ingredient" is used to indicate a part of a composition in which the ingredient remains individually identifiable,

c) the word "component" is used to indicate a material which reacts with another component or other components to produce a further material.

The priming explosive may be in any required physical form. It may consist of a primary explosive compound, for example a salt, or of a priming composition containing a primary explosive compound which may be produced in a mixture of some or all of the

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other indredients of the composition: for example, an explosive compound may be produced in a mixture which comprises a fuel and/or an oxidiser and/or a frictionator: such a composition is disclosed in US Patent Specification No 2239547 to Brun.

In any process according to the invention, the materials which are brought together to produce the priming explosive are preferably comparatively insensitive, thereby mitigating storage, mixing and charging problems. However, it may be necessary to use some sensitive starting materials: for example, a priming composition may comprise a plurality of sensitive ingredients, and it may be difficult or impossible to produce all of the sensitive ingredients simultaneously in the composition: for example it would be difficult to produce lead styphnate and tetrazene simultaneously in a rimfire ammunition priming composition, although both these ingredients may be required. Accordingly the invention includes within its scope the use of sensitive starting materials, but where these are used they preferably constitute a minor proportion, preferably a small proportion, of the starting materials.

It is within the broad scope of the invention to form a sensitive explosive, say from relatively insensitive starting materials, and then to combine it with additional ingredients of a composition: this may be necessary if the additional ingredients would interfere with the formation of the sensitive explosive. However, it is preferred to avoid such addition, where possible, as it clearly introduces additional hazards.

Production of the priming explosive may be effected in a liquid medium, preferably water. The liquid medium may be driven off or otherwise removed after formation of the explosive. The liquid medium may perform either or both of the following functions:-

1. it may act as a desensitiser for solid sensitive ingredients, which should be damp until the composition has formed

2. it may act as a reaction medium enabling components to form a new material.

A component which is soluble in a reaction medium may be taken into solution by the medium and thereafter brought together with another component. Alternatively, the components and medium may be independent of each other before being brought together. Further, where components will not react dangerously in the absence of a medium, they may be brought together before being brought together with the medium.

Production of priming explosive in accordance with the method of the invention involves a chemical reaction forming a primary explosive. The desirable characteristics of such a reaction for the purposes of the present invention are set out below:-

a) Simplicity: The reaction should preferably involve only a single stage, without prolonged stirring (preferably requiring none at all) and without requiring closely defined critical conditions such as temperature or pH.

b) Speed: The reaction should preferably be completed in a short period. However, this will not be essential if an incomplete reaction does not prevent further processing, for example spinning, drying, addition or propellant and bullet in production of ammunition, and provided the reaction has been completed by the time the product is required for final use.

c) Compatibility with containers: The solutions or other materials involved in the reaction should clearly be compatible with the material of the container in which the reaction takes place. Some acids will be excluded from reactions which occur in metal containers such as brass rimfire cartridge cases.

d) By-products: There should be no unduly deleterious by-products. In some cases it may be possible to use by-products of a reaction, for example as oxidisers in a priming composition. Where this is not possible, the by-products may be inert, gaseous, volatile or vapourisable, or at least not seriously deleterious.

e) Starting materials: These should be easily and safely handlable in bulk form. Small proportions of sensitive materials, for example tetrazene, may be included in the starting materials, preferably in a desensitized form.

A metathetic reaction will normally best satisfy requirements a) and b) above. The formation of covalent bonds is usually a relatively slow process. Suitable metathetic reactions will be double decomposition reactions and acid base reactions. In addition to metathetic reactions, however, the formation of mixed crystals has also been found to be a suitable reaction under criteria a) and b) above. Priming explosives comprising mixed crystals are already well known in the explosives art.

The required characteristics of the priming explosive are in particular, the following:-

a) it must be effective for its required application - for example ignition of propellant or initiation of a secondary explosive,

b) it must satisfy special requirements dependent upon its specific use, for example ballistics tests in production of ammunition.

c) it must provide the required sensitivity, which will clearly have both upper and lower required values (in ammunition production these are represented by "all fire" and "no fire" heights for sensitivity drop tests of primed cases),

d) it must be compatible with its surroundings - the container and any adjacent explosive such as propellant or secondary explosive, and

e) it must be reasonably stable in storage in the expected conditions of use which may involve high or low temperatures and/or humid conditions.

Where an explosive compound is produced in situ in a method of the invention it is not necessary to produce a so called "free flowing" crystal form. The provision of such a crystal form is a long standing problem in the explosive art and can be avoided by the in situ technique. Further, by using that technique, the nature and sensitivity of the priming explosive may be chosen solely independance upon its final use rather than, as hitherto, upon the technique used to load the explosive into devices. In the past, many suitable initating compounds had to be rejected because they proved too dangerously sensitive for use in conventional priming techniques involving distribution of priming explosive from a batch between explosive devices. Examples of suitable materials are cited below.

According to the preferred method in accordance with the present invention, predominantly relatively insensitive materials are brought together to produce a priming explosive in a quantity suitable for priming an individual explosive device. Preferably the quantity is one of a series of such quantities. Production of the explosive is preferably effected wholly in situ in a device. However, it is within the broad scope of the invention to complete production of the explosive away from the device, the product thereafter being supplied to the device, or to bring the materials together away from the device and to complete production of the explosive in situ.

Apparatus for use in the method of the invention may comprise means for bringing together materials, which together will form said explosive, at a low rate and/or in a succession of small quantitues preferably appropriate to individual utilisation demands, and/or on a flow path continuous with means for supplying partially or completely formed explosive to a succession of other elements of utilising devices.

Where it is desired to produce explosive in a plurality of small quantities, the apparatus may comprise a plurality of dispensing means, each adapted to dispense predetermined doses of material to respective receivers therefor so that each receiver will receive a dose from each dispensing means of said plurality.

The materials dispensed may comprise components of an explosive and a medium in which said components can interact.

Where production is not effected wholly in situ, starting materials may be fed continuously to a mixing zone. The starting materials may comprise some ingredients of an explosive composition and components which will react forming an explosive compound. In the mixing zone, they may be brought together in small quantities or at a low rate. The mixture may leave the zone as a series of small discrete quantities, or as a stream of small dimensions. Such a stream may be interrupted at intervals to reduce explosion hazards; for example, there may be explosion barriers so that an explosion at a particular location in the stream will be limited to the region between successive barriers.

The mixture may be transferred continuously from the mixing zone to a utilisation zone. Where the mixture leaves the mixing zone as a series of small quantities, each quantity may be of a size appropriate to an individual utilisation demand, for example shotgun cap, rimfire cartridge or detonator. The series of quantities may therefore be dispensed directly into a corresponding series of containers for the explosive devices. It would be possible to produce a series of small quantities each of which represents a plurality of utilisation demands, with a subsequent division of each quantity between those demands. However, this is not preferred as it involves an extra processing stage. Where the mixture leaves the zone as a stream of small dimensions, those dimensions may be selected to facilitate division of the stream into quantities appropriate to individual utilisation demands. For example, the cross-section of the stream might be appropriate to an individual rimfire cartridge, cap shell or detonator.

Preferably the materials are mixed in substantially predetermined proportions in said mixing zone.

The mixing zone may be such that it is freely accessible to flow paths by which the materials are supplied to it. Alternatively, there may be means for controlling access to the mixing zone from such flow paths. For example, the materials may be circulated through closed flow paths normally separated from the mixing zone, there being tapping means to tap off quantities of materials to the mixing zone.

Mixing in the zone may be effected by any convenient means for example by turbulence, by mechanical intervention or by passing gas bubbles through the mixing zone.

The following are examples of explosives which can be made by methods in accordance with the invention as applied to priming rimfire cartridge cases.

In these examples, which refer to production of rimfire ammunition, reference is made to sensitivity tests. These involve dropping a 2oz ball onto a bar striker which then indents the rim of a cartridge case. The results are quoted in terms of the "mean fire height" - that is, the release height of the ball above the striker required to give a 50% chance of firing a cartridge in a given sample, usually of 50 cartridges: this is obtained by a statistical calculation, and the associated standard deviation is also quoted. Occasionally the "all fire height" is quoted - this is the release height of the ball at which all cartridges in the sample fired.

EXAMPLE 1 STYPHNATES

a) by double decomposition:

The following materials were used in the indicated proportions by weight:—

Sodium styphnate	27 parts)	
Lead hypophosphite	7 parts)	DRY
Grit	25 parts)	
Lead nitrate	31 parts)	
Tetrazene	3 parts)	WET
Gum Arabic		
Lissapol (Registered Trade Mark)		

The first three materials are relatively insoluble in water when compared with lead nitrate, and they were provided in powder form in a rimfire cartridge case, in a predetermined dose. The dose required depends upon the quantity of initiating composition required to ensure ignition of the propellant. In a cartridge designed to contain about 80 mg. of nitrocellulose base powder as propellant, the quantities of the reacting components were such as to produce about 20 mg. of initiating composition. This can be adjusted as required to give designed ballistic characteristics for the combination.

Lead nitrate is soluble in water, and was added in the form of a solution thereof to the dry materials. The tetrazene was dispersed in the lead nitrate solution, this being a dangerous material to handle dry. The gum arabic and Lissapol were present in small proportions for reasons well known in the art.

The reaction between the lead nitrate and the sodium styphnate then occurred in the cartridge case, giving lead styphnate and sodium nitrate in the resultant mixture. The product was dried out after the reaction, and then approximately 10% by volume of water was added to the dried mixture to render it mouldable. The rimfire case containing the mouldable composition into the rim of the case in the conventional manner. The moulded composition was then passed through a conventional drying arrangement and the primed case was subsequently handled in the conventional fashion.

In order to prime cap shells, antimony sulphide, in dry powder form may be substituted for the grit at least partially.

The double decomposition reaction described above was carried out at room temperature. Increased temperature may lead to larger crystal sizes for the lead styphnate and this may affect sensitivity of the composition. Increase of temperature runs the risk of decomposition of the tetrazene, this being a particular problem above about 70°C. However, the highest possible temperature, subject to other restraints, is advantageous in facilitating crystallisation of the lead styphnate from the gel which forms in the early stages of the double decomposition reaction.

No steps were taken to control the pH of the mixture in the cartridge case. This would be slightly acid because of the presence of the lead nitrate solution, and slight acidity is necessary for crystallisation of the lead styphnate. A pH in the range 3 to 6 is suitable.

The quantity of water used was just sufficient to take the lead nitrate into solution. This gives a paste consistency to the mixture after addition of the solution. It is desirable to minimise the quantity of water used since it has to be driven off after the lead styphnate has formed.

The bulk of the starting materials referred to above are insensitive when compared with the lead styphnate. However, tetrazene is a primary explosive and dry sodium styphnate can be caused to explode if ignited by a black powder fuse.

It may therefore be necessary to keep these materials wet in store and in feeding them to the cartridge case. It will be noted however that the proportion of preformed primary explosive in the starting materials is very small - well under 10% even if the indicated proportion is increased slightly. Further, only small quantities of materials are involved in each individual reaction, so that it is possible to use a starting material having quite substantial sensitivity, although preferably less than the eventual primary explosive. For example, in United States Patent Specification No. 2239547 to Brün, there is described a method of making normal lead styphnate by admixture of reacting quantities of basic lead styphnate, styphnic acid and other priming composition ingredients, the normal lead styphnate being formed in the mixture. The process described in that specification therefore involves the conversion of one explosive, basic lead styphnate, into a more sensitive explosive, normal lead styphnate, in a mixture of other ingredients. Such a process could be adapted to the present invention.

Where the heavy metal is lead, the process conditions are preferably controlled so as to result in formation of normal lead styphnate, but a proportion of basic lead styphnate may be found acceptable depending upon the required circumstances of use. The yield of lead styphnate can be improved by thorough mixing of the components, so as to minimise the proportion of unreacted feedstock remaining in the composition. Where the process is carried out on a small scale, as in the example cited above of priming of rimfire cases by reacting components in situ, mixing can be effected by vibration of the reacting components. The yield of normal lead styphnate can be improved by control of the pH, and it may be necessary to add free acid to ensure the required acidity in the reacting mixture.

The first stage of the double decomposition reaction is the formation of a gel from which the heavy metal styphnate crystallises. The time required for crystallisation from the gel stage depends upon the temperature and the concentration of the mixture, the time being longer for lower temperatures and higher concentrations. Where the composition is being produced in situ, maximum concentration is desirable to avoid having to drive off the solvent, usually water. Thus maximum permissible temperature is also desirable, but this will be limited by the tendency towards thermal decomposition of reacting components and resulting products, and possibly also by the effects of increasing temperature upon crystal size of the styphnate.

b) by reaction with styphnic acid:

A mixture was produced of the following materials in the stated proportions by weight:-

a) styphnic acid 100 parts
b) white lead 100 parts
c) ground glass 50 parts
d) barium nitrate 50 parts

All of the materials were in dry powder form, and all powder particles passed through a 100 mesh (British Standard 410) sieve. When distributed between rimfire cartridge cases, the mixture was moistened with water and permitted to react in situ in the cases. It was found that the resulting primed case could be made to explode satisfactorily with a substantial flame. The quantity of priming composition in each case was of the order of 14 to 15 mg.

The styphnate route is not limited to the production of lead compounds. Other heavy metal styphnates might be produced in a similar manner, and have previously been suggested for use in initiating compositions. Further, the acid reaction is not limited to the use of white lead (lead carbonate) or lead oxide (PbO). An alternative possibility is lead hydroxide. The use of lead oxide is particularly desirable, however, in that there are no resulting by-products, the lead oxide and styphnic acid combining exactly to produce lead styphnate. The use of lead hydroxide is also acceptable on this ground, however, since the only resulting by-product is water, which is in any event present as an ionising medium. A further possibility would be a reaction between lead acetate and styphnic acid. In this case, the expected by-product would be acetic acid, which would be volatile and could be driven off during the reaction. The by-product of white lead is CO_2 which is driven off. Further information on production of styphnates is contained in US Patent 2295104 and German Offenlegungsschrift 2531997.

EXAMPLE 2 MULTIPLE SALTS ESPECIALLY NITRATO-HYPOPHOSPHITES

The double salt lead nitrato-hypophosphite is described in German Patent Specification No. 289016, and the use of it in a priming composition is discussed in US Patent Specifications Nos. 2160469 and 2116878.

In one test, the following materials were used in the indicated percentages by weight:-

Lead nitrate	40%)	
Gum Arabic		WET
Lissapol		
Lead hypophosphite	40%)	
Grit	20%)	DRY

The last two materials are relatively insoluble in water, and are provided in the rimfire case in the form of mixed powder. A concentrated aqueous solution of lead nitrate, incorporating small proportions of gum and Lissapol is then added to the powder in a predetermined dosage. The double salt lead nitrato-hypophosphite will separate from the solution at ambient temperature. The product can then be dried until it is in a mouldable form, whereupon the case can be passed to a conventional spinning punch to compact the composition into the rim. The Lissapol functions as a surfactant in this example, but may be found unnecessary.

Drying may be effected at a temperatures up to at least 100°C, to produce a product containing approximately 10 to 12% water, which will be suitable for compaction. The product may then be dried completely. If preferred, the initial product can be dried completely, and a predetermined dosage of water added to produce the mouldable composition. A further drying step is needed after compacting as in the alternative process.

In an alternative method of forming a priming composition, the lead nitrate, lead hypophosphite and grit are mixed as dry powder, and a predetermined quantity of dry powder is inserted in the rimfire case. About 10 to 12% by weight of water, together with the Gum Arabic and Lissapol, is then added to the mixed powders, and the mixture forms the double salt generally as described above. Since the mixture now includes the required percentage of water to render it mouldable, there is no need for a drying operation before the case is passed to the spinning punch, or other device, for compacting the mixture into the rim of the case. In this method, the powders may be pre-mixed before they are inserted into the case, or they may be inserted independently and the case may then be vibrated to mix the powders therein. The mixing step has been omitted from some tests and a satisfactory product was nevertheless obtained.

It will be noted that in a method as described above, whether or not powders are premixed or the slurry is agitated during reaction, the double salt is permitted to crystalise substantially freely, that is without precautions to control crystal sixe as described in US Specification 2160469. The formation of "extended crystals" referred to in that patent can be permitted in situ in an explosive device.

It is most convenient to form the composition at ambient temperature, and this has been found satisfactory. However, the method is not to be limited to such temperatures; it may be desired to control the temperature at which the product forms, and possibly to supply heat to raise the temperature above ambient. Temperatures up to 45°C have already proved satisfactory; higher temperatures may be used, subject to decomposition of the compounds.

It has also been found possible to form the double salt with a pH value in the region 1-3, although this is believed more acid than is necessary. Undue acidity is undesirable because of the possibility of an attack upon the material of the case by the solutions therein. On the other hand, undue alkalinity of the solutions may cause stress corrosion of the case. A pH of 3 to 5 is believed suitable. The pH will usually be determined by the pH of the lead nitrate component.

The molecular weights of lead nitrate and lead hypophosphite are approximately the same; the powders are therefore preferably used in approximately equal weight proportions. However, a slight excess of either powder may be found desirable in practice depending upon the circumstances. The invention is not to be limited to substantially equal weight proportions, however, since an excess of up to 100% of either component has been found to produce a product having satisfactory sensitivity and initiating power.

As the excess of either product increases, however, adequate mixing of the components may become a problem, and "patches" of unreacted component may be found in the case rim.

Where a solution of lead nitrate is to be added to lead hypophosphite powder, it is desirable to make the solution as concentrated as possible, to minimise the amount of water to be driven off before compaction.

A series of rimfire cartridges primed with lead nitrato-hypophosphite produced by the first method outlined above has been subjected to a series of tests, the results of which are summarised in the following paragraphs:

Sensitivity

Mean fire height - 4.71 ± 1.18 inches

All fire height - 9 inches

This indicates a sensitivity greater than that of conventional priming compositions comprising lead styphnate and tetrazene.

The sensitivity was found to depend on the proportion of the frictionator, i.e. grit in the example quoted above. If the frictionator was not provided, it was found that the product would not fire even in a gun breech. Alternative frictionators comprise powdered glass and carbon particles (coke). The sensitivity was also found to be dependent to a certain extent upon the proportions of the lead nitrate and lead hypophosphite, slightly lower sensitivity being found with an excess of hypophosphite.

Barrel time

This is the time between the fall of the striker of the gun and the emergence of the bullet from the barrel. The time was measured at 2.59 ± 0.13 milliseconds. The range of measurements was 0.58 millisecond. This is satisfactory in comparison with the conventional priming compositions mentioned above.

The barrel time will be dependent to some extent upon the relative proportions of the priming composition and the propellant. In the tests mentioned above, the propellant was the disc-type single base propellant sold by ICI Limited under the name "Acurex" (Registered Trade Mark). In the tests the cartridges contained about 80 mg of propellant, and the quantity of priming composition was about 20 mg in each case. This is within the range of quantities of conventional priming composition.

Pressure and Velocity

The driving pressure produced by the tested cartridges averaged 5.78 tons/sq. in, giving a velocity of approximately 1056 feet/second. This is slightly lower than the pressure and velocity found with conventional priming compositions, but is satisfactory. After storage in humid conditions, the cartridges were found to give a pressure of about 5.68 tons/sq in and a velocity of 1038 feet/second.

Mass explosibility

This is the percentage of cases initiated by an explosion of one case in a group. It was found that 90 + % of the group could be initiated in this manner, possibly because of the very high sensitivity of the priming composition. This may be dealt with to some extent by using additives, such as glass "flour" and other inert substances (see US Patent Specification 2356210) or polyvinyl alcohol (see US Patent Specification 2341262), or by adding a layer of varnish over the priming composition in the primed cases. An alternative method of dealing with the problems would be to produce cartridges in a continuous line, avoiding groups of primed cases at any point along the line.

The priming composition may include further additives to give additional properties or modify the properties of the composition. For example, additives may be included to reduce mass explosibility as referred to above, or to improve workability as described in US Patent Specifications 2327867, 2377670 and 2662818. Other additives may provide fuel: for example, antimony sulphide may be included for this purpose and results in a bigger flame. Silicon and calcium silicide both give sparks. Fuels may be particularly important in priming of caps. The double salt lead nitrato hypophosphite can be formed in the presence of each of the fuels referred to, and it has been found that each of these fuels tends to increase sensitivity of the composition, reducing the proportion of frictionator required. The composition may also include small proportions of other primary explosives, if required.

In the method of producing the multiple salt, it is not necessary to perform the operation wholly within the container of the device, that is the rimfire case, cap or the detonator case. The components may be brought together outside the device, formation being completed in situ. Alternatively, the formation may also be completed outside the device, and the formed product may be charged into the device. It is preferred to produce the multiple salt in small quantities, preferably appropriate to individual explosive devices, because of its high sensitivity and the explosion risk associated with a large bath.

The use of multiple salts in a method according to the invention is not limited to the double salt lead nitrato hypophosphite. Similar multiple salts are referred to in US Patent Specifications 2175826, 2292956 and 2352964 and others may also prove suitable.

EXAMPLE 3 AZIDES

All solutions referred to in this example are in water. Unless otherwise indicated, all tests involved formation of composition in a cartridge case, complete drying of the composition, re-wetting to mouldability, compaction into the case rim and re-drying. Unless otherwise indicated, reacting components react in stoichiometric proportions and mixing of materials was effected in the case, usually by vibration thereof.

Test 1: 5.4 mg. of sodium azide, of particle size lower than 100 mesh (British Standard 410), were mixed in powder form with 5.4 mg. of powdered glass, and this dose was inserted into a .22 rimfire cartridge case. 10.8 μl. of lead nitrate were added to the case in a 50% solution. The sodium azide and lead nitrate reacted in the case to produce lead azide and sodium nitrate. The resulting composition was dried but was not spun into the rim of the case.

This mixture involved a substantial excess of sodium azide over lead nitrate relative to stoichiometric proportions. Nevertheless, in sensitivity tests, the mean fire height for primed cases was 6.2" ± 0.7".

Test 2: 3.5 mg. of sodium azide mixed with 3.5 mg. of powdered glass were provided in a rimfire cartridge case and dosed with 17.8 μl. of a 50% solution of lead nitrate. The mean fire height was 4.63" ± 0.7".

Test 3: 7.7 mg. of a powder having the following proportions by weight were located in a rimfire cartridge case:-

Sodium azide	50%
powdered glass	25%
antimony sulphide	25%

20 μl. of 50% lead nitrate solution were dosed into the case. The resulting mean fire height was 6.4" ± 1.78", and it was noted that the composition produced more flame than that given in Test 2. This latter result is to be expected because of the addition of the antimony sulphide. This type of composition would be suitable for use in a shotgun cap shell.

Test 4: 4.5 mg. of powdered glass were fed into a rimfire cartridge case. 12.5 μl. of a 28% solution of sodium azide were then dosed into the case, and were followed by 18 μl. of a 50% solution of lead nitrate. The case was vibrated during addition of the solutions, but the resulting composition was not spun into the rim. The composition was thoroughly dried.

The resulting mean fire height was 7.1" ± 2.3". However, after addition of 4 μl. of water, the resulting mouldable composition was spun into the rim of the case head, and the mean fire height, after drying, was measured at 5.1" ± 0.89". Cases treated in this way gave an "all fire" height of 9" for a batch of 50 cases.

Test 5: 16 mg. of a dry powder of the following weight proportions were inserted into a rimfire cartridge case after thorough mixing:-

lead nitrate	53%
sodium azide	20%
powdered glass	27%

This powder was wetted with 2.4 mg. (15 weight%), of water and the resulting composition was spun into the rim of the head, and then dried. The composition was observed to be somewhat powdery, and it is believed that insufficient water had been added to ensure complete reaction. Nevertheless, cases primed by this method could be fired and the mean fire height was measured at 10.2" ± 1.28".

Test 6: 4 mg. of powdered glass were metered into a rimfire cartridge case, and the following solutions were added in the order indicated:-

a) 12 μl. lead nitrate solution containing 6 mg. of solid, and
b) 24 μl. barium azide solution containing 4 mg. of solid.

The mean fire height was calculated at 6.4" ± 1.08".

Test 7: 9.3 mg. of powder, comprising 6.7 mg. of lead hypophosphite and the remainder powdered glass, were metered into a rimfire case. 9.5 μl. of a 28% solution of sodium azide were added to the powder, giving a total priming weight of 12 mg.

The mean fire height was calculated as 9.35" ± 3.34". It was observed that this composition gave a large flame, indicating that it may be suitable for use in priming of cap shells for shotgun cartridges.

Test 8: As Test 7, but with 26.5 μl. of barium azide solution (concentration 166 grammes per litre) substituted for the sodium azide, giving a total priming weight of 14 mg.

The mean fire height was calculated at 6.95" ± 1.26". Again, a large flame was observed on initiation of this composition.

Test 9: 13 mg. of a powder, comprising a 50:50 mixture of lead nitrate and powdered glass, were dosed into a rim fire case, and 9 μl. of a 28% solution of sodium azide were added. The total priming weight was 15½ mg.

The mean fire height was calculated at 5.2" ± 0.99".

Test 10: As Test 9, but with 4.4 mg. of barium azide, in the lowest quantity of water capable of taking the azide into solution, substituted for the sodium azide.

The mean fire height was calculated at 4.5" ± 1.59".

In Test 6 above, barium azide and lead nitrate will react to produce lead azide and barium nitrate. The latter is the oxidiser used in conventional priming compositions, but lead azide does not require an oxidiser. A diluent may be required to reduce the explosive violence of the azide, and barium nitrate can function in this fasion. Sodium nitrate produced in the reaction of lead nitrate with sodium azide can function similarly to barium nitrate. The oxidiser can also supply oxygen to subsidiary fuels such as antimony sulphide.

Useful azides are not limited to these cited above. Theoretically, alternative heavy metals may be used, although lead is virtually universal in practice. Alternative lead salts may be used as a reaction component, and soluble lead salts are preferred. In particular, lead acetate is a possible alternative to lead nitrate, since it is conventionally used in production of lead azide. The resulting sodium acetate would not function as an oxidiser, but it would function as a moderator in the manner discussed above. Other soluble lead salts are:- chlorates, citrates, isobutyrates, lactates, nitrites, peroxydisulphates and dithionates. However, lead chlorate should not be used in production of priming composition for ammunition, since combustion of a chlorate would produce chloride ions which would cause rusting of a gun barrel.

It is most convenient to form the priming explosive in an aqueous medium, but this is not essential. In the Tests given above, the reactions occurred at room temperature, but this is also not essential. However, it is preferred not to reduce reaction temperatures since this lowers solubility of the components in water and necessitates extra water in the case. In the above Tests, no particular steps were taken to control the pH of the reacting mixture and a suitable pH value can be established empirically.

In Tests 7 and 8, it is believed that the sodium and barium azides undergo double decomposition with the lead hypophosphite to produce lead azide and sodium or barium hypophosphite. However, it is possible that a sodium or barium azide: lead hypophosphite double salt is formed and provides the or an explosive ingredient in the composition. The formation of such a double salt falls within the scope of the present invention as exemplified by Example 2 above.

By way of example, embodiments of apparatus in accordance with the invention will now be described with reference to the accompanying diagrammatic drawings in which:

Figure 1 is a plan view of an ammunition priming apparatus in accordance with the invention, and

Figures 2 to 4 are diagrams of different feed systems for use in continuous production techniques according to the invention.

Figure 1 illustrates a series of rotatable, pocketed modules, three of which are shown at 10, 12 and 14 respectively. The use of such modules enables different process times to be accommodated on different modules while permitting uniform motion of transported articles through the transport system as a whole, i.e. without acceleration and deceleration.

Module 10 accepts rimfire cartridge cases from a suitable feed indicated by arrow 16 and passes them at a predetermined rate to module 12. Here, each case receives a predetermined dose of a first component required for a chemical reaction to produce a primary explosive, as indicated by arrow 18. The cases are then passed in succession to module 14, where they receive a predetermined dose of a second component required to produce the explosive, as indicated by arrow 20. At least one of these modules will dis-

pense a liquid, preferably comprising water. One or both of the modules may dispense ingredients of a priming composition in addition to components which are to react in the case to form a primary explosive.

Further processing depends on the chemistry of the reaction involved. If required, mixing means may be used to mix the materials in the case. There may be means for removing excess liquid in the case when it is no longer required for the reaction. In any event, the cases are finally passed to an oven, diagrammatically indicated at 22, where the composition in the cases is dried out. The dried primed cases may then be passed to a line, preferably automated, for propellant charging and insertion of the bullet, if the cartridge is not a blank.

Figures 2 to 4 show diagrammatically possible general layouts of feed systems for use in continuous production techniques according to the invention.

In the first embodiment shown in Figure 2 ingredients that are to form part of a priming composition are fed along paths 110 and 112 to a mixing zone 114. Each ingredient is relatively insensitive. Relatively insensitive components that will react to produce a primary explosive are included respectively in path 110 and path 112. It will be understood that two paths are shown only as an example: there may be any number of incoming flow paths, for example, one for each ingredient of the mixture other than the sensitive explosive, and one each for the components required to produce the explosive. Preferably, however, the ingredients and components on the flow paths have been premixed where that is possible. The ingredients and components are mixed in small quantities in the zone 114, and in the illustrated example they are immediately dispensed from that zone in predetermined small quantities into a series of containers such as the rimfire cartridge case indicated at 116.

There may be a suitable dispenser diagrammatically indicated at 118 for controlling the quantity of mixture passed from the zone 114 to each case 116. Alternatively or in addition there may be control gates 120, 122 in the flow lines 110, 112 for controlling feed of materials to the mixing zone.

In an alternative arrangement shown in Figure 3, the materials are circulated continuously on closed flow paths 124, 126. The mixing zone is indicated at 128 and there are controllable tapping devices 130, 132 for tapping controlled quantities of ingredients from the closed paths 124, 126 into the zone 128. Zone 128 has a dispensing outlet 134 for supplying mixture to a cartridge case, cap shell or detonator case. There must be suitable means for feeding material into each closed path to make up that tapped off.

This aspect of the invention is not limited to immediate dispensing of the formed mixture, nor to a "localised" mixing zone such as that shown in Figures 2 and 3. Instead, ingredients may be fed on flow paths 136, 138 shown in Figure 4 to one end of a tubular mixing zone 140. Tube 140 may be formed as a "Static Mixer" as supplied by Kenics Corporation of Danvers, Massachusetts, USA. Such a mixer is described in the June 1970 issue of Chemical and Process Engineering in an article entitled "Static Mixer".

In any process in which the mixture is not to be supplied immediately to containers such as cases or cap shells, it may be pumped along a ducting system. Such a system may have barriers at intervals along its length dividing it into predetermined regions so that an explosion in the system will be localised between explosion barriers. The cross-section of the stream may be such that it can be divided transversely for dispensing into containers. However, division of the stream into predetermined quantities may be effected by any suitable dispensing device at the output end.

In Figure 2, relatively insensitive materials may be gated into the mixing zone in predetermined proportions appropriate to an individual utilisation demand. The mixture may be immediately dispensed and further similar quantities gated into the mixing zone. The embodiment of Figure 3 may be operated in a similar manner. Alternatively, the dispenser at the outlet from the mixing zone in either embodiment may be arranged to divide the mixture between a series of devices. In any of the embodiments illustrated in Figures 2 to 4, the output rate is substantially matched to the demand rate on a production line for explosive devices continuous with the illustrated apparatus.

The invention is not limited to details of the illustrated methods and the explosives referred to in the EXAMPLES.

In some instances, it may be found that deliberate mixing of materials is unnecessary. For example, if at least one material goes into solution which will penetrate a body of another material without mixing, then a deliberate mixing step can be omitted. In general, however, it will be preferable to include deliberate mixing to ensure complete reaction.

Regarding the selection of a primary explosive, this must depend upon the required conditions of production and use, bearing in mind the criteria already set out above. The

double salt lead nitrate hypophosphite is a particularly desirable ammunition priming explosive from the point of view of most of the criteria for both reaction and product. It can be produced by a simple, speedy reaction between lead nitrate and lead hypophosphite, both of which are compatible with a brass rimfire cartridge case. There are no by-products, and the starting materials are both non-explosive. The product is sensitive, and compatible with the container and propellant. It is capable of initiating propellant powders. It has been found to give some difficulties in connection with humid storage conditions. However, these can be dealt with by application of a sealing material, e.g. a bituminous material, to the join between the bullet and a cartridge case. A suitable material has been found to be "Ritolastic" (Registered Trade Mark) produced by Lancashire Tar Distillers Building Products of Church Road, Litherland, Liverpool.

The acid-base reaction for production of lead styphnate also satisfied the above requirements, and has the advantage that it produces a product in close accordance with conventional priming compositions and which is known to be relatively insensitive to humid storage conditions. This route is therefore preferable to the lead nitrate/sodium styphnate route since the sodium nitrate produced by the latter route is hygroscopic.

There is one difficulty in the production of styphnates by either of the chemical routes referred to, namely the necessity to incorporate tetrazene to obtain required sensitivity in the product. Tetrazene is itself a sensitive primary explosive, and must therefore be used in very low quantities in the feed stocks. Where one of these feed sticks is in the form of a liquid, the tetrazene may be included in that liquid as a dispersion and suitable dispersing agents may be included in the solution to aid this. It would however be desirable to substitute an alternative sensitiser for the tetrazene if possible. Lead nitrate hypophosphite could be produced simultaneously with lead styphnate, particularly where a double decomposition reaction involving lead nitrate is used.

Lead azide satisfies most requirements, but is known to give corrosion difficulties when used in copper alloy containers such as brass cartridge cases. It then produces a copper azide through reaction with the copper in the alloy case, and this is itself a sensitive initiating material.

It should be mentioned however that a reaction in accordance with this invention does not have to take place in the final container. It could occur in another type of receiver which may be designed to apply less stringent requirements on the reaction conditions. The formation of explosive could even be completed in such a receiver and the resultant "pellet" passed on for charging to the final container. Where explosive is produced on or around a carrier, for example the bridgewire of a detonator matchhead, the carrier could project into a receiver of this type. In some instances, it may be possible to remove by-products which would otherwise be deleterious, for example if they were soluble in the reaction liquid which could be decanted, drained away or sucked out of the receiver.

There are further aspects of certain embodiments described above which distinguish them from the prior art referred to throughout the specification.

In a first aspect, an explosive is permitted to form freely, particularly, but not essentially, without interference with its crystal form. We have already referred above to the free formation of lead nitrate hypophosphite crystals, and compared this with the stirring suggested in US Specification 2 160 469. We have also referred to the avoidance of the necessity to form "free flowing" and "reduced sensitivity" crystal forms of azides and styphnates - for example as described in British Patent Specifications 1 336 561 and 519 340. Such crystal modification techniques are not excluded from the present invention, but they may prove unnecessary if the present proposals are adopted.

The priming of explosive devices according to the method of the invention may, therefore, involve producing a priming explosive comprising a "multiple" salt of a hypophosphite, particularly where the multiple salt has been permitted to crystallise freely during its formation. In the present specification the term "multiple" salt is used to indicate a salt produced by co-crystallisation of two or more component salts. Such a multiple salt may be used with another explosive, for example a styphnate which may be formed simultaneously, for example in situ, with the multiple salt.

We have established that it is not essential to mix the components of lead nitratohypophosphite to obtain a satisfactory product, although mixing will give added assurance of a uniform product in large scale production of explosive devices. It appears that a solution containing at least one component can diffuse through a body of undissolved material to produce the double salt. Since only small quantities of material are involved in the preferred embodiment the degree of diffusion is usually sufficient to give the required result.

The priming explosive may be a composition including other, preferably non-

explosive, materials, for example a frictionator and/or a fuel. The multiple salt preferably comprises a nitrate component and a hypophosphite component. Preferably both components are lead salts.

Such a priming explosive may advantageously be used with a nitrocellulose-based propellant. The propellant may be in the form of discs, for example as supplied by Imperial Chemical Industries Limited under the name "Acurex". The propellant may, however, alternatively be in the form of ball, flake or military powders. Alternatively, such a priming explosive may be used in priming detonators and may then be used with a secondary charge of a detonator. This may be a base charge, for example tetryl or PETN.

In a second aspect, at least some by-products of an interaction which produces an explosive are retained in an explosive composition. We have referred above to the ability to retain by-products in some circumstances, particularly where they can act as an oxidiser or at least as a desired diluent.

The by-product preferably forms a substantially anhydrous crystal, and preferably has a low degree of hygroscopicity. It is preferably a nitrate where it is required to act as an oxidiser. However, there are other requirements which may have to be met. For example, in the double decomposition reaction for production of styphnates the styphnate feedstock should be as soluble as possible. Sodium styphnate has an acceptable solubility, and sodium nitrate performs satisfactorily as an oxidiser in a mixture containing lead styphnate, replacing the conventional barium nitrate. Potassium and ammonium styphnate are also satisfactory on the basis of solubility, but potassium and ammonium nitrate have not performed satisfactorily as oxidisers. Magnesium styphnate is the most soluble, but magnesium nitrate includes a substantial quantity of water of crystallisation, which is difficult to remove.

It is not necessary for the double decomposition reaction to provide all of the oxidiser required in a composition. For example, a conventional oxidiser such as barium nitrate could be added to a composition after formation of a heavy metal styphnate therein. Addition of barium nitrate at an early stage in such a reaction may result in the formation of barium styphnate which is an insensitive explosive relative to lead styphnate.

An explosive and by-product may be produced in a mixture of other ingredients, which may include each or either of a frictionator and a fuel. Alternatively, other ingredients may be added to the composition after the explosive and by-product have been produced.

The nature of the explosive will depend to some extent upon the intended use. For example, where it is required to initiate a low explosive, including propellants such as ball, flake, disc and military powders which usually include nitrocellulose and may be of single, double or triple base type, the priming explosive is required to ignite the low explosive and not to cause it to detonate. The priming explosive is therefore required to produce flame, and/or heat and/or sparks, and may not be itself a detonating explosive. Where the priming explosive is intended to initiate a high explosive, for example the base charge of a detonator, it will usually be required to produce a shock, and a detonating initiator will then be required. An actual detonator may have two or more primary charges, for example a matchhead sensitive to heat produced by electric currents and a priming charge sensitive to a spit from the match head. Both of these primary charges may be produced by techniques in accordance with the invention.

In any event, priming explosive produced in a method of the invention should be capable of initiating finished explosive devices. Such devices will usually be arranged to produce substantial quantities of energy, for example in the form of heat and/or gas and/or shock. All reasonable precautions must be taken to ensure that such devices are not initiated except in response to a predetermined event or events, and this can be done by suitable choice of priming explosive and/or suitable choice of container therefor.

The invention is intended for use in manufacture of finished explosive devices and partly finished devices for example initiating devices such as caps. Manufacturing processes will usually involve continuous or semi-continuous repetitive operation over a substantial period, with a substantial total output demand over that period. The invention enables production of priming explosive to be spread over the relevant period instead of concentrating it, thus avoiding production and storage hazards. Thus, the rate of production of priming explosive in a method in accordance with the invention is substantially matched with the demand, although it may be acceptable if there is a small deficit or a small surplus which could be handled by a relatively simple explosive magazine. For manufacturing purposes, it is desirable that chemical reactions involved should be easily reproduceable with consistently reliable results.

WHAT WE CLAIM IS:

1. A method of priming a plurality of explosive devices by incorporating a discrete body of priming explosive with an element of each device wherein the priming explosive is produced, as hereinbefore defined, at a rate that is, as hereinbefore defined, continuous with the incorporation thereof with the elements of said devices.

2. A method as claimed in Claim 1 wherein said explosive is produced in a plurality of small quantities.

3. A method as claimed in Claim 2 wherein each quantity corresponds to the quantity required for each body of explosive.

4. A method as claimed in Claim 2 wherein each quantity is a batch sufficient for a plurality of such bodies of explosive.

5. A method as claimed in Claim 3 wherein said explosive is produced in situ (as hereinbefore defined).

6. A method as claimed in any one of Claims 1 to 5 wherein production of the priming explosive comprises production of a styphnate primary explosive.

7. A method as claimed in Claim 6 wherein the styphnate is produced by double decomposition.

8. A method as claimed in Claim 6 wherein said styphnate is produced by reaction between styphnic acid and a compound including a heavy metal.

9. A method as claimed in any one of Claims 1 to 5 wherein production of the priming explosive comprises production of an azide primary explosive.

10. A method as claimed in any one of Claims 1 to 5 wherein production of the priming explosive comprises production of a multiple salt.

11. A method as claimed in Claim 10 wherein said multiple salt is lead nitratohypophosphite.

12. A method as claimed in any one of Claims 1 to 11 wherein production of the priming explosive comprises reacting components to produce a primary explosive and a by-product which remains in the primary explosive.

13. A method as claimed in Claim 12 wherein said by-product will function as an oxidiser.

14. A method as claimed in Claim 12 or Claim 13 wherein said by-product is a nitrate.

15. A method as claimed in any one of Claims 12 to 14 wherein said primary explosive is a heavy metal styphnate.

16. A method as claimed in any one of Claims 12 to 15 wherein said by-product forms a substantially anhydrous crystal.

17. A method as claimed in Claim 16 when appendant to Claim 15 wherein said styphnate is lead styphnate and said by-product is sodium nitrate.

18. A method as claimed in any one of Claims 1 to 17 wherein production of the priming explosive is effected by taking a substantially dry premix comprising components that will react together in the presence of a liquid medium to form a primary explosive compound and combining said liquid medium therewith.

19. A method as claimed in Claim 18 for forming said priming explosive in situ wherein an appropriate amount of said premix is dosed into an element of each device and the liquid medium is combined, in the element, with said premix.

20. A method as claimed in Claim 19 wherein said premix is dosed into said element and the liquid medium is then added thereto.

21. A method as claimed in any one of Claims 18 to 20 wherein the premix additionally comprises one or more ingredients of a priming composition.

22. A modification of a method as claimed in any one of Claims 18 to 21 wherein at least one of said components and/or ingredients is initially contained in the liquid medium.

23. A method as claimed in Claim 1 conducted substantially as hereinbefore described with reference to the accompanying drawings.

24. A method as claimed in Claim 1 wherein said priming explosive is produced by a method substantially as described in any one of Examples 1 to 3 herein.

25. Explosive devices whenever primed by a method as claimed in any one of Claims 1 to 24.

26. Explosive devices as claimed in Claim 25 which are primed rimfire cartridge cases, detonator cases or shotgun cartridge caps.

27. Rimfire cartridges, shotgun cartridges or detonators including devices as claimed in Claim 26.

28. A method of incorporating a body of priming explosive within each of a plurality of containers therefor, each container forming, or intended to form part of a unit of ammunition, the method comprising the steps of dosing an amount of a substantially dry

premix into each container, said premix comprising components that will react together in the presence of a liquid medium forming a primary explosive compound, and combining, in each container, an amount of said liquid medium with the premix.

29. A method as claimed in claim 28 wherein said amount of liquid medium is dosed into each container after the premix has been dosed thereinto.

30. A method as claimed in Claim 28 or claim 29 wherein said premix additionally contains one or more ingredients of a priming composition.

31. A container, or unit of ammunition comprising such a container, within which there has been incorporated a body of priming explosive by a method including that claimed in any one of claims 28 to 30.

32. A container as claimed in claim 31 which is a rimfire cartridge case or a cap.

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