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MANUFACTURE OF COMPACT COMBUSTIBLE EXPLOSIVE CHARGES

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The present invention relates to a new or improved method for the manufacture of combustible explosive charges in compact form, characterised by a high gas yield and capable of production in forms of considerable web thickness, for the generation of gas pressure, and to compact combustible gas producing explosive charges thereby obtained.

Compact gas producing charges composed of a combustible explosive material are frequently employed for the actuation of gas pressure operated mechanical devices and gas escape reaction actuated devices, for instance rockets.

Although it is known to use pellets of black-powder, and although pellets of compositions made from similar ingredients modified by the presence of inorganic or organic diluents have been suggested as charges for the production of gas pressure for such purposes, extruded charges made from smokeless powders consisting essentially of gelatinised nitrocellulose compositions are now commonly employed on account of their markedly superior gas yield and other advantages. It is, however, difficult to produce charges of gelatinised nitrocellulose compositions of any considerable web thickness if a volatile solvent is used in their production, since the solvent evaporates only very slowly from the interior of the charge, and in practice it is usually necessary to employ a composition containing nitroglycerine as gelatinising agent in order to make and extrude the composition without using a volatile solvent.

It would, however, be desirable to produce compact charges for the generation of gas pressure for the aforesaid purposes without necessitating the use of pressing apparatus and also without consuming the organic nitrates required for the manufacture of smokeless powder.

It has been proposed in British specification 453,210 to provide a charge for gas-pressure operated devices consisting essentially or largely of ammonium nitrate and containing as sensitiser of its thermal decomposition a chromium compound adapted to generate chromic oxide on heating in the presence of ammonium nitrate, so that the gas evolving composition is capable of undergoing a self sustained decomposition without detonation when ignited locally and at ordinary pressure by a non-detonating igniting element insufficient of itself to effect any substantial general rise in temperature of the charge.

According to this previous proposal it is desirable to include in the composition a material oxidisable by the ammonium nitrate so as to yield gaseous products, this material being present in

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a proportion sufficient to obviate the formation of oxides of nitrogen, and, in the case of charges required for operating mechanical devices, sufficient to yield a high proportion of permanent gases in the gases formed. Suitable chromium compounds are stated to include ammonium, alkali metal, and certain other chromates, bichromates and polychromates, the ammonium and potassium salts being particularly effective. These are referred to hereinafter as chromate compounds. According to the aforesaid proposal it is stated that the ingredients may be admixed by a milling operation, and compact compositions prepared by pressing mixtures containing ammonium nitrate and ammonium bichromate with mineral jelly, bitumen, or paraformaldehyde as the oxidisable material are exemplified.

Notwithstanding the sensitising effect of the chromate compounds generally on the thermal decomposition of ammonium nitrate we have now found that compact ammonium nitrate charges containing an oxidisable material and sensitised with a chromate compound and suitable for the generation of gas pressure can be produced by a casting operation involving at least partial fusion of the charge provided that there is included in the composition sufficient of at least one ammonium nitrate fusion promoting ingredient solid at ordinary temperature to render the composition pourable at a temperature not exceeding approximately 110 to 115° C. Preferably the chromate compound is introduced into the composition only after a mixture at a temperature at least approximating to pouring temperature has been formed, comprising at least the ammonium nitrate and the fusion promoting ingredients. The oxidisable material may or may not comprise an ammonium nitrate fusion promoting ingredient. Other constituents can be introduced before or after the chromate compound, but the maintenance of compositions containing the chromate compound at high temperature should be avoided by casting the composition without unnecessary delay, otherwise it may show evidence of exothermic reaction.

The chromate compound may advantageously be an alkali metal or ammonium bichromate or polychromate, and the proportion employed may conveniently amount to from about 2 to 20 per cent of the weight of the ammonium nitrate used.

By ammonium nitrate fusion promoting ingredients, solid at ordinary temperature, we mean solid materials (other than chromate compounds) adapted when heated with ammonium nitrate to temperatures substantially below the fusion point

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of the latter to form liquid or partly liquid melts in which there is only one liquid phase and ammonium nitrate is a constituent of the liquid phase.

Water is a liquid that promotes the fusion of ammonium nitrate, but unless sufficient hydratable salts or the like to fix the water as water of crystallisation when the melt is cooled, are present, only a very limited amount of water can be included in the composition. The quantity of the fusion promoting ingredients included in the composition, including any water not capable of being bound as water of crystallisation must be sufficient to render the mixture pourable at the aforesaid temperature, since above that temperature the thermal decomposition of the ammonium nitrate by the chromate compound may occur at an appreciable rate, and it will be understood that there must be used only such fusion promoting ingredients as are capable of being heated with ammonium nitrate and also with the chromate compound alone without undergoing any substantial decomposition leading to the formation of gaseous products, at a temperature not exceeding the aforesaid pouring temperature. Olive colouration indicates chromate destruction.

Subject to this proviso there may be used as fusion promoting ingredients a wide variety of organic or inorganic substances having the aforesaid properties, whether or not of oxidising or oxidisable character. It is frequently desirable to employ a plurality of fusion promoting ingredients and these may if desired be of differing chemical character.

It is often necessary that the amount of oxidisable material in the cast charge should be sufficient not merely to prevent the formation of any appreciable quantity of oxides of nitrogen, but also to yield a high proportion of permanent gases, more particularly carbon monoxide and hydrogen, in the products of its combustion. On the other hand, the formation of an unoxidised carbon smoke is seldom desirable. The employment of ammonium nitrate fusion promoting ingredients of oxidisable organic or inorganic character in conjunction with ammonium nitrate fusion promoting ingredients of an oxidising character facilitates the production of cast charges of desirable oxygen balance at a low pouring temperature.

One useful group of ammonium nitrate fusion promoting ingredients includes saline and non-saline compounds capable of forming eutectic mixtures with ammonium nitrate, whether or not of oxidising character, for instance alkali metal nitrates, alkaline earth metal nitrates, alkali metal chlorides, alkaline earth metal chlorides, magnesium sulphate and ammonium chloride, or other non-alkaline anhydrous salts. Likewise there may be used various non-saline organic compounds. A useful group of ammonium nitrate fusion promoters of oxidisable character is provided by weakly basic or non-basic organic amino derivatives, for instance dicyandiamide, guanidine nitrate, nitroguanidine, acetamide, guanidine and like compounds.

An especially useful category of fusion promoting ingredients is also provided by highly soluble non-alkaline salts containing water of crystallisation, whether or not of oxidising character, for instance magnesium nitrate hexahydrate, Epsom salts, zinc nitrate hexahydrate, or calcium nitrate tetrahydrate, microcosmic salt and the like. The water of crystallisation liberated by such hydrated salts at raised temperature

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has the desirable effect of retarding thermal decomposition of the ammonium bichromate in the melt. Instead of the hydrated salts containing water of crystallisation the anhydrous salts or salts of a lower degree by hydration and water in amounts up to that capable of being bound as water of crystallisation when the melt is cooled may alternatively be employed.

As oxidisable ingredients organic polyhydric compounds are frequently too easily oxidised by the polychromates or bichromates to be of service according to the present invention, but in addition to the fusion promoting ingredients there may be included in the cast composition suspended solid combustibles such as lamp black, graphite, resins or the like, and the cast composition may also include fusible oxidisable ingredients such as nitro-hydrocarbons, hydrocarbons, waxes or the like emulsified in the melt with the assistance of emulsifying agents such as bentonite or kieselguhr.

The invention is illustrated by the following examples, in which the parts are parts by weight.

Example 1

10 parts magnesium nitrate hexahydrate are fused in a kettle having a stirrer and a previously made mixture of 67 parts powdered ammonium nitrate, and 15 parts dicyandiamide is then introduced into the kettle, which is heated to 90 to 93° C. and stirred at this temperature until as much of the material as possible has fused. 8 parts finely crystalline ammonium bichromate are then stirred into the mixture, and the resulting mixture is poured at 90 to 95° C. into wooden moulds of the required shape, in which it is allowed to solidify. The resulting cast charge is thereafter removed from the moulds before it has completely cooled.

Example 2

10 parts magnesium nitrate hexahydrate are fused in a heated kettle fitted with stirring apparatus and a previously prepared mixture of 56 parts finely crystalline ammonium nitrate, 6 parts potassium nitrate and 8 parts dicyandiamide is then introduced and heated to 100° C. 2 parts bentonite and 11 parts trinitrotoluene are then introduced into the mixture which is stirred until the molten trinitrotoluene has been well emulsified. 6.5 parts finely crystalline ammonium bichromate are then stirred into the mixture, which is poured at 95 to 100° C. into wooden moulds of the required form, from which it is removed after solidification, but before it has completely cooled.

Example 3

A mixture of 71 parts finely crystalline ammonium nitrate, 10 parts nitroguanidine, and 7 parts acetamide are melted together with stirring in a kettle at 100° C., 8 parts finely crystalline ammonium bichromate are then introduced into the mixture, whereupon 2 parts bentonite and 2 parts carnauba wax introduced, and the mixture is stirred until the carnauba wax has been well emulsified. The resulting mixture is poured into wooden moulds of the required form from which it is removed after it has solidified, but before it is quite cold.

Example 4

A mixture of 69 parts finely crystalline ammonium nitrate, 10 parts nitroguanidine, 2 parts magnesium nitrate hexahydrate and 7 parts acetamide are melted together with stirring in a

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kettle at 100° C. 8 parts finely crystalline ammonium bichromate are then introduced into the mixture, whereupon 2 parts bentonite and 2 parts carnauba wax are introduced, and the mixture is stirred until the carnauba wax has been well emulsified. The resulting mixture is poured into wooden moulds of the required form, from which it is removed after it has solidified, but before it is quite cold.

Example 5

56 parts finely crystalline ammonium nitrate, 15.2 parts guanidine nitrate, 11.6 parts nitroguanidine, 2 parts magnesium nitrate hexahydrate and 7.2 parts dicyandiamide are fused together at a temperature of 105° C., whereupon 8 parts finely crystalline ammonium bichromate are introduced into the melt with stirring. The melt is then cast into cardboard moulds of the required form, in which it is allowed to solidify, and from which it is removed before the cast has completely cooled.

Example 6

58 parts finely crystalline ammonium nitrate, 15.2 parts guanidine nitrate, 11.6 parts nitroguanidine and 7.2 parts dicyandiamide are fused together at a temperature of 105° C. whereupon 8 parts finely crystalline ammonium bichromate are introduced into the melt, with stirring. The melt is then cast into cardboard moulds of the required form, in which it is allowed to solidify, and from which it is removed before the cast has completely cooled. Examples 1-6 give orange coloured casts.

Example 7

62 parts finely crystalline ammonium nitrate, 10 parts sodium nitrate, 8 parts guanidine nitrate and 7 parts nitroguanidine are heated and stirred together at a temperature of 102 to 103° C., whereupon 8 parts finely crystalline ammonium bichromate are stirred in, 1.7 parts lamp black and 3.3 parts graphite are next stirred in to the mixture which is poured at a temperature of about 100 to 102° C. into moulds of the required form in which it is allowed to solidify and from which it is removed before it has completely cooled.

It is sometimes convenient to cast the compositions into Celluloid containers which are left in place, and are burned off in the operation of igniting the charge, instead of casting them into moulds from which they are removed. Charges of any desired web thickness in tubular or solid forms may be made according to the method of the present invention. Not only are the novel charges of the invention highly compact or dense as a result of having solidified from fused condition, but in addition they possess substantial homogeneity or uniformity of composition throughout, in degree characteristic of solidification from such fused condition. The chromate sensitizer is at least in part dissolved in the fluid ammonium nitrate phase at the temperature at which the melt is cast, and in any case becomes uniformly distributed throughout the melt in extremely intimate and effective association with the other components of the mixture, and continues to be so distributed upon cooling and solidification of the cast mass. Such intimacy and uniformity of distribution is impossible to attain by mechanically mixing the solid materials.

The cast charges described in the foregoing examples are all capable of burning when ignited,

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yielding a high gas evolution. In using them for the generation of pressure it is frequently desirable to employ a relatively small quantity of smokeless powder made from nitrocellulose of small web thickness, desirably in conjunction with a small quantity of blackpowder, to assist their ignition. It is not, however, essential to use smokeless powder. For instance, a cast tubular pellet having an external diameter of say 2 inches and an annular thickness of $\frac{3}{4}$ inch, and weighing 2 to 3 lbs. could be ignited by means of an electric powder fuze containing about 15 to 20 grams of blackpowder inserted into the tube.

Having now particularly described and ascertained the nature of our said invention, and in what manner the same is to be performed, we declare that what we claim is:

1. A combustible explosive charge in compact form for the generation of gas pressure, which comprises ammonium nitrate in preponderating proportion, in intimate commixture with an oxidizable material, a chromate compound adapted to sensitize the thermal decomposition of the charge, and an ammonium nitrate fusion-promoting ingredient solid at ordinary temperatures and adapted to render the charge fluid at a temperature not exceeding 115° C.; said charge being compact and homogeneous due to solidification of the mixture from fused condition.

2. A process for the production of combustible explosive charges in compact form and suitable for the generation of gas pressure which comprises mixing ammonium nitrate with at least one fusion promoting ingredient solid at ordinary temperature, said ingredients being adapted to render the charge pourable at a temperature not exceeding about 115° C., rendering said mixture pourable by at least partial fusion at a temperature not exceeding about 115° C., thereafter adding from 2 to 20% based on the weight of ammonium nitrate of a chromate compound adapted to sensitize the thermal decomposition of the charge, adding at some point during the mixing operation an oxidizable carbonaceous material sufficient in amount to bind a preponderant proportion of the oxygen present in the combustion products of the charge in the form of carbon monoxide, and casting the charge by pouring without appreciable delay after addition of the chromate compound.

3. A process for the production of combustible explosive charges in compact form and suitable for the generation of gas pressure which comprises mixing ammonium nitrate with at least one oxidizable carbonaceous fusion promoting ingredient, such ingredients being sufficient in amount to bind a preponderant proportion of the oxygen present in the combustion products of the charge in the form of carbon monoxide and adapted to render the charge pourable at a temperature not exceeding about 115° C., rendering said mixture pourable by at least partial fusion at a temperature not exceeding about 115° C., thereafter adding from 2 to 20% based on the weight of ammonium nitrate of a chromate compound of the charge, and casting the charge by pouring without appreciable delay after addition of the chromate compound.

4. A process as defined in claim 2 in which one fusion promoting ingredient consists of an organic amino derivative which is not more than weakly basic.

5. A process as defined in claim 2 in which one

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fusion promoting ingredient consists of magnesium nitrate hexahydrate.

6. A process as defined in claim 2 in which one fusion promoting ingredient consists of an alkali metal nitrate.

7. A process as defined in claim 2 in which the fusion promoting ingredients comprise a mixture of (a) an organic amino derivative which is not more than weakly basic and (b) magnesium nitrate hexahydrate.

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