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PROPELLENT POWDER

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This invention relates to propellent powders, and more particularly to progressive-burning smokeless powders characterized by surface treatment with new and improved deterrent materials.

It has been known in the art of propellent explosives that increased projectile velocities could be secured, while maintaining safe breech pressures in the firearm, by surface treatment of the powder grains with deterrents, this result being obtained by decreasing the combustion rate of the initially burning portion of the powder grains. Many materials have heretofore been proposed for use as smokeless powder deterrents, such as aromatic nitrohydrocarbons, dialkyl phthalates, dialkyldiarylureas, and a variety of waxes, fats, and resins.

Despite the many proposals which have been made in this art, improvements have been desirable in several aspects, notably in providing deterrent materials which may easily be applied as a coating on powder grains without undesirable agglomeration of the grains, which result in ballistically as well as chemically stable grains, and which act with greater efficiency.

It is therefore an object of this invention to provide improved progressive-burning smokeless powder grains.

Another object of this invention is to provide surface-treated smokeless powder grains having a desirable combination of ignitibility, reduced initial combustion rate, appropriate density, and ballistic stability.

Other objects will appear from the following detail description.

In accordance with this invention, generally stated, the foregoing objects are accomplished by employing a diaryl phthalate as the surface-treating agent for smokeless powder grains. This class of phthalate esters has been found to possess particularly advantageous properties for such use. The solvent and plasticizing properties are conducive to rapid absorption at the surface of the smokeless powder grains and the resulting product is characterized by a uniform, stable, and readily ignitible surface layer. Due to the relatively high melting point, substantially higher concentrations can be tolerated in the surface layer of powder grains without causing tackiness than is the case with liquid deterrents previously in use, and accordingly greater deterrent effect may be secured. Furthermore, by reason of their extreme insolubility in water, the novel deterrents enhance the water resistance of the treated powder grains.

Representative diaryl phthalates, which are suitable for use in the surface-treatment of powder grains in accordance with this invention, (the approximate melting points in degrees C. being indicated in parentheses) are: diphenyl (73°), di-p-cresyl (84°), di-o-cresyl (110°), di-2,4,5 trimethyl phenyl (119°), and di-α-naphthyl (155°) phthalates.

The foregoing and other like esters may be applied as the deterrent coating on single- or multiple-base powder grains of the desired shape and size in accordance with any suitable method. The preformed grains of smokeless powder may be composed of nitrocellulose or other gelatinizable nitrated carbohydrate smokeless powder base, for example nitrostarch, alone or in combination, and, if desired, may contain in addition one or more nitrated polyhydric alcohols, for example, nitroglycerin, nitroglycol, and the like and/or an aromatic nitrohydrocarbon such as di- or trinitrotoluene. The surface-treatment may advantageously be effected as described in the following specific examples, in which detailed directions are supplied in order fully to illustrate the invention.

In one method, the grains may be surface-treated while suspended by means of vigorous agitation in an excess of a non-solvent liquid. For example, the smokeless powder grains may be vigorously agitated in about six times their weight of water, at a temperature of about 65° C., contained in a suitable jacketed vessel. The diaryl phthalate may be emulsified in a separate vessel in ten to fifteen times its weight of hot water containing about 0.1% (based on the weight of ester) of gum arabic or other suitable protective colloid such as starch, methyl cellulose, gelatin, or the like, and the emulsion added to the suspension of the powder grains. The temperature may be maintained at about 65° C. and agitation continued for a suitable period, for example 2½ hours when the deterrent coating amounts to 5% of the weight of powder being treated. If desired, for insuring stability of the coating, the grains may be maintained at an elevated temperature of 50°–70° C., with or without agitation, for an additional period up to about 24 hours. The suspension is then cooled during agitation, the excess liquid removed by filtration or centrifuging, and the grains washed with water. The product may then be dried, as by treatment with warm air heated to 50° C. for several days, graphited, brought to equilibrium with the desired relative humidity, and stored ready for use.

In accordance with another well-known type

of coating process, the treatment may be carried out in a "sweetie barrel," consisting of a rotatable cylinder partly immersed in a temperature controlling bath or provided with other suitable means for heating and cooling the contents. The powder grains, containing from about one fourth to one half their weight of water, are placed within the treating cylinder, which is then rotated while the temperature is raised. When the grains have become heated to a temperature of about 70° C., the diaryl phthalate is added, rotation of the treating cylinder being continued. This rotation and the consequent agitation and mixing of the grains is maintained for a period of about 15 minutes after the addition of the deterrent has been completed. The mixture is then cooled to room temperature by continuing the rotation while cold water is circulated through the temperature-controlling bath. If additional heat treatment of the grain is desired, the grains may be maintained submerged under hot water in a suitable vessel for the required period. After removal of the excess water by filtering or centrifuging, the product may be dried, graphited, brought to equilibrium at the desired relative humidity, and stored ready for use.

Aryl phthalates which are characterized by a melting point between 60° C. and about 100° C. may be applied in accordance with the above procedures, since the treatment of the powder grains can then be carried out at a convenient temperature, somewhat above the melting point of the particular deterrent employed. However, aryl phthalates having melting points above 100° C. may also be utilized in the surface treatment of powder grains at convenient temperatures substantially below 100° C. (that is, at temperatures at which the deterrents themselves are solid) either by employing them in admixtures which are liquid at the desired temperature, or by applying the deterrent in the form of a solution in a suitable solvent.

Where a solvent is thus employed, it should be sufficient in amount to render the deterrent substance liquid at the temperature of the treatment and should be sufficiently volatile so as to be readily removed from the powder grains during the subsequent drying treatment. Preferably, a solvent is employed for the deterrent which is not a solvent for the smokeless powder base, for example, aromatic hydrocarbons such as benzene or toluene, chlorinated hydrocarbons such as ethylene dichloride, trichloroethylene, or carbon tetrachloride, alcohols such as ethyl or isopropyl, or a solvent naphtha of suitable volatility. However, it may be advantageous at times, in order to facilitate the coating treatment and/or secure greater penetration into the powder grains, to add to the deterrent solution a volatile solvent for the powder grains, for example, low boiling esters or ketones such as ethyl or isopropyl acetate, acetone, or ethyl methyl ketone.

The amount of deterrent employed may vary in accordance with the type of base powder being treated and the ballistic characteristics desired. In general, however, an amount between ½ and about 5% by weight of the powder grains will provide the desired results, it being understood however, that somewhat higher proportions may be employed when necessary.

As illustrative of the advantages which may be secured by use of the deterrents in accordance with this invention, the following table contains ballistic test data obtained with a given base powder coated in identical manner in accordance with the first specific example described above, (*a*) with diphenyl phthalate exemplifying the new and improved deterrent substances, and (*b*) with symmetrical diethyldiphenylurea, exemplifying one of the most extensively used deterrents of the prior art. The base powder employed consisted of gelatinized spherical grains, as described in U. S. Patent 2,027,114, consisting of nitrocellulose, 2% incorporated dinitrotoluene and 15% nitroglycerin, having a gravimetric density of 0.84 and average grain diameter of 0.019 inch. The deterrent coating amounted in each case to 5% by weight of the powder. The resulting powders, having gravimetric densities of (*a*) 0.86 and (*b*) 0.84, were loaded and fired in .30 caliber-1906 ammunition having a bullet weighing 150 grains. In this ammunition, a 36.8 grain charge of the uncoated powder produced a muzzle velocity of 2470 feet per second at a pressure of 45,000 pounds per sq. in. The comparative test data are listed in the following table:

	Deterrent	
	(*a*) Diphenyl phthalate	(*b*) Diethyldiphenylurea
RESULTS OF FIRING AT NORMAL TEMPERATURE		
Powder charge ..... grains	46.5	47.5
Pressure ..... lbs./sq. in.	43,900	45,000
Muzzle velocity ..... ft./sec.	2,682	2,680
RESULTS OF FIRING AT —40° C.		
Pressure ..... lbs./sq. in.	38,200	35,100
Pressure loss ..... do	5,700	9,900
Velocity ..... ft./sec.	2,559	2,516
Velocity loss ..... do	123	164

The diaryl phthalate deterrent in accordance with this invention displayed superior results particularly with respect to efficiency (a smaller charge of the powder treated therewith being required to provide the desired ballistics) and in the ignitibility of the coated powder (shown by smaller changes in the ballistic properties on lowering the firing temperature from about 25° C. to —40° C.).

Microscopic examination of thin sections of grains of the diphenyl-phthalate-coated powder revealed the presence of a uniform surface layer in which the novel deterrent was concentrated. Similar study of the grains after storage of the powder at 50° C. for 30 days showed that no appreciable alteration of this structure had occurred.

Firing tests carried out after this period of storage at 50° C. likewise substantiated the absence of any significant change in the ballistic properties of diaryl-phthalate-coated powder grains on being subjected to elevated temperatures, this result representing a considerable improvement over liquid ester deterrents such as diethyl phthalate heretofore in use.

It will be understood that the invention is not to be limited to the specific details stated above but that variations may be made, as will be realized by those skilled in the art, which are contemplated within the scope of the following claims.

Having now described the invention, what is claimed as new and is desired to be secured by Letters Patent. is:

1. Propellent powder grains surface-treated with a diaryl phthalate.

2. Propellent powder grains surface-treated with diphenyl phthalate.

3. Single-base propellent powder grains surface-treated with a diaryl phthalate.

4. Single-base propellent powder grains surface-treated with diphenyl phthalate.

5. Multiple-base propellent powder grains surface-treated with a diaryl phthalate.

6. Multiple-base propellent powder grains surface-treated with diphenyl phthalate.

7. Nitrocellulose-nitroglycerin propellent powder grains surface-treated with a diaryl phthalate.

8. Nitrocellulose-nitroglycerin propellent powder grains surface-treated with diphenyl phthalate.

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