# Chapter IV TAGGANT SAFETY AND COMPATIBILITY REVIEW

# Chapter IV. --- TAGGANT SAFETY AND COMPATIBILITY REVIEW

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## COMPATIBILITY OVERVIEW

The explosives and gunpowders communities operate under a particularly severe constraint. Their products must work, essentially 100 percent of the time, under a wide range of user conditions. At the same time, the products must remain inert, or not "work," during the manufacturing process, in storage, during transportation, and until initiated at the site of end use. Thousands of people come into contact with explosives and gunpowders every day; an accident can have extremely severe consequences to those people, including injury and death. The consequences of an explosive or gunpowder not functioning when properly initiated are somewhat less severe, although misfires can result in considerable safety hazard to those who must remove or work around the nonfunctioning material.

A good deal of analysis and testing is required to ensure proper operation of a particular explosive material; proper operation in this context means the material will remain reliably inert until initiated, at which point it will reliably detonate (explosives) or burn (gunpowders). Over the years, qualification procedures have been developed to evaluate the reliability and safety of operation of explosive materials. These procedures vary with the organization involved, but generally combine analysis of the fundamental chemical properties of the material, appropriate testing, development of manufacturing control mechanisms, quality control of ingredients and the finished product, and long-term experience in manufacture, storage, transportation, and use. These qualification procedures are used when a new product is developed or when a significant change is made to an existing product.

The addition of identification and detection taggants to explosive materials would constitute a significant change to the material qualification program is therefore necessary to investigate the compatibility of the explosive materials with the taggants. This chapter briefly discusses the involved in compatibility, describes qualification procedures in industry and for defense applications, suggests the form. that a quification program should take to demonstrate the compatibility of taggants with explosives and gunpowders, and describes the compatibilitytesting that has been reported to date.

### EXPLOSIVE MATERIALS COMPATIBILITY PARAMETERS

Explosive materials are chemical systems that I iberate a large amount of energy in an extremely short time. The detailed physical and chemical behavior of these reactants is not well-understood, due to the complexity of some of the reactants and the very short reaction time scale. However, the principal measurable parameters of the materials and their reactions are well-known. To demonstrate compatibility of the explosive materials with the taggants, it is necessary to show that there is no significant change in these parameters as a result of the addition of taggants. The principal parameters include:

- energy density and rate of release,
- sensitivity,
- chemical stability,

- electrical properties,
- generalized mechanical properties, and
- toxicity.

### Energy Density and Rate of Release

The energy density and rate of energy release are the two most important performance attributes of commercial explosives and gunpowder. Energy density is a fundamental chemical property of the explosive material formulation. The available energy of a given explosive material is well-understood, and it can be measured with a high degree of accuracy and reliability. It can also be calculated quite accurately from the basic chemical knowledge of a particular formulation. The presence of the small amounts of taggants that are currently recommended should have only a minute effect. Limited testing has borne out this conclusion. <sup>12</sup>

Generally speaking, the higher energy density explosives tend to be easier to initiate and tend to progress to a fast energy release or detonation more quickly. Primary explosives used in caps are an exception. They are easy to initiate, and build to detonation very rapidly, but do not always have a high energy density.

The rate of energy release is a function of the materials involved and the physical proximity of the fuel and oxidizer components. When the fuel and oxidizer are in the same molecule, as in nitroglycerine, the explosive can release its energy on a millionth of a second time scale. Ammonium nitrate/fuel oil mixtures, on the other hand, contain rather large, separated fuel and oxidizer components and thus release their energy on a much slower time scale. The physical proximity of the components also tends to affect sensitivity; the intimately connected materials are generally more sensitive than the gross mixtures. The balance of fuel to oxidizer directly affects the energy density and sensitivity of the explosive material. The balance that yields idealized combustion products generally yields the highest energy and most sensitive explosives.

The rate of energy release cannot be predicted quantitatively from basic physical and chemical considerations but it can be estimated in a qualitative way. Energy release rate can be measured accurately but the test methods can be quite expensive and difficult. A few hundredths of a percent by weight of taggants should not affect the energy release rate.

### Sensitivity

Sensitivity is an ill-defined term which has meaning in a safety sense, but is not definable with simple direct physical constants. One relative sensitivity scale can be developed from impact and friction tests, another scale from electrochemical reactions, and still another from thermal considerations. All aspects of reactions to external stimuli must be considered and judged with respect to practical experience. Then with a variety of "sensitivity " numbers and functions a systems safety estimate is made — not always totally scientifically but with an additional input from experience and common sense.

Sensitivity tests are referenced and discussed in other sections of this report, but the individual numbers are not in themselves the final criteria. It is their sum total plus experience which determines sensitivity.

### Chemical Stability

Chemical stability is a critical safety parameter, of paramount importance in the handling, transportation, and storage of the raw materials that go into making explosives and gunpowder and in the manufacture, handling, transportation, storage, and use of the final explosive product. The stability of the explosive products cannot be adequately predicted ana-

<sup>&#</sup>x27;1 etter,R E Lunn (Du Pent) to C Boyars (Aerospace), "Tagging — Du Pent PilotTestSatety and Stability Tests, " Mar 6, 1978,pp 5-17, 5-41, 5-42

<sup>&</sup>lt;sup>4</sup>C Boyars, Compatibility of Identification Taggants W ith Explosives, Aerospace report No AT R-78( 1860-02)1 ND, August 1978

<sup>&#</sup>x27;Safety and Performance Tests for Qualification of Explosives, /Kabik, (NSWC, WO), R Stresau (Stresau Laboratories, Enc.), K R Hamilton (NWC), JJones, (NWC), NavordOD 44811, vol.1, January 197.2

lytically, but must be confirmed by tests that demonstrate the stability behavior of the products, such as long-term rates of decomposition, interact ions between the explosive components, and reaction with materials into which they are likely to come into contact during manufacture, packaging, and end use. As an example, picric acid and ammonium picrate, rather powerful high explosives, which are insensitive and generally quite safe, were once used extensively. When these explosives come into contact with copper or copper salts, however, they become quite sensitive; their use is, therefore, now quite limited.

### Electric Properties

The sensitivity of initiation of explosives by static electricity and/or induced currents has always been a major concern. There are several modes of initiation due to electrical energy. One, inductive coupling, is serious enough to preclude the use of electric blasting caps in some operations. Direct initiation by static spark discharges is another mode, The energy of an electric field can be coupled to an explosive device in other ways, for example, by thermal heating of a wire or capacitance effects, The primaries, lead styphnate and lead azide, are extremely sensitive to electric effects. Dry nitrocellulose and black powder are also very sensitive. Most cap-sensitive high explosives and generally used blasting agents are not particularly sensitive to electric forces. Addition of taggants to the explosive materials could cause a change in their electrical properties; buildup of a static charge during the addition of the taggant to the mix could be one mode. As analytical methods are not adequate to handle the problem, tests are normally conducted.

### Generalized Mechanical Properties

The relationship of mechanical properties to explosive safety has only recently been understood to be of paramount importance, Experience and intuition led the industry into explosive formulations that were not ideal chemically, but have proven safe and economical. Most, but not all, commercial explosives are rather soft granules, rubbery or gelatinous substances, or sometimes liquid-like.

When soft substances are subjected to impact the mechanical forces are not concentrated in a small volume and they dissipate as low-level thermal waves. Stiff, brittle materials experience strong fast compression or shock waves under impact conditions that locally produce high-energy concentrations. Local high-energy concentrations create hot spots. This means that a hot spot can be a center of intense chemical reaction and therefore, in an explosive composition, a region of fast energy release. Thus, an initiation center is created when the rate of energy release exceeds its dissipation. Grit or hard substances can create local hot spots under handling conditions present in the mixing and packaging processes, and especially in operations such as explosive tamping in the bore hole. As an example, a small number of hard particles has been demonstrated to critically sensitize certain military explosives in United Kingdom la boratories.4 The danger of hot-spot creation may be even greater for more, brittle explosives, such as those used in cast boosters.

The effects of adding taggants to explosives could be simulated using complex hydro-elastic-plastic computer codes, but the calculations would be quite expensive. In addition, lack of sufficient data on the detailed physical properties of the various materials would tend to limit the reliability of such calculations. Experimental testing must therefore be undertaken.

### Toxicity

The decomposition products of explosive reactions are generally toxic; standard precautionary measures must be taken to avoid excessive exposure. The materials used in the taggants are generally not mutagenic or carcinogenic. Tests must be conducted to evaluate the toxicity of any taggant materials

<sup>&</sup>lt;sup>4</sup>C. Bean (Atomic Weapons Research Establishment, Alderwington, U. Va.), private communication to D-E Laboratories, May 1979.

whose properties are not well-known, and to determine if the end-product gases show addi-

tional toxicity as a result of the addition of taggants to explosive products.

### QUALIFICATION OF EXPLOSIVES

A new explosive compound or formulation must be subjected to an extensive series of tests before it can be qualified for use and manufacture. The number and nature of the tests differ between various manufacturers of commercial explosives and between commercial manufacturers and Government developers such as the Department of Defense (DOD) and the Department of Energy (DOE). Tests are specifically designed for the explosive product, the environment it will be subjected to, and its end use. It follows that an extensive battery of tests are required for each explosive. Interpretation of the tests, including the validity of some prescribed ones, is not straightforward and a single number derived from a test or tests cannot alone define its safety. The closest that one can come to a measure of explosive safety is the long-term accident record. [t is important to realize that experience plays a role equal to good scientific understanding and execution of prudent, conservative practices. The decisionmaking process as to whether or not the new explosive and process of manufacture are safe is therefore unique to each organization.

In general, the qualification procedures described in this section are those followed by agencies or companies that routinely develop new explosives or significant modifications of existing explosives, including Government agencies such as DOD and DOE and some manufacturers of commercial explosives. Companies that rarely develop new products do not generally need a comprehensive qualification program. Within those organizations that do have a comprehensive program, the complexity, qualification time, and cost vary considerably, due to differing manufacturing procedures and end uses. As an example, complete qualification of a new military explosive can take several years with a total cost of many m i I I ions of dollars.

NAVORD Report OD 44811 specifies safety and performance tests for qualification of explosives for the Navy. There is also a Joint Service Safety and Performance Manual used by all three services. The DOE procedures are similar to the DOD ones but are not documented in a single manual. Each plant and laboratory has its own rules and specifications approved by the director. There are certain procedures and test methods that are common to all, however, which are briefly discussed in this sect ion.

The initial testing is done on small quantities on a laboratory scale, usually less than a gram. Drop weight impact tests are always done, followed by friction and thermal test such as DTA, DSC, Taliani, or others. The results of a statistically significant number of tests are then compared with known standard explosives. If the tests give satisfactory results, then a laboratory or plant level management decision, usually backed up by a safety committee review, will give a go ahead to make limited quantities sufficient to do the preliminary performance tests such as detonation velocity, detonation pressure, and shock sensitivity. These tests usually require several pounds of the new explosive to complete. At this stage more elaborate chemical compatibility and thermal stability tests are also run along with some accelerated aging tests. The small-scale laboratory tests are repeated at this stage and compared with the original results. Unless all test results are satisfactory, further work on the new explosive will be stopped.

If results are satisfactory and if the performance is as desired then a management decision beyond the laboratory level will generally be made to proceed with limited pilot production. As much as several hundred pounds may be involved. It is at this stage that manufacturing hazards are assessed. Special tests will usually evolve at this stage that will relate to the actual manufacturing equipment such as pipe diameter in which a liquid explosive or slurry will or will not propogate a detonation. Exact details of equipment and controls are then reviewed. In the case of addition of taggants there is the possibility of buildup of the material in some part of the mixing or cartridge-loading machinery. Consideration is given to fail-safe controls in the event of power failures or other equipment failures. Transportation of raw materials and finished product within the plant is planned. Barricades and remote control are planned where required. For example, the pressing of booster pellets of Tetryl or PETN is a hazardous operation and must be done by remote control and the press itself barricaded so that no personnel are exposed in case of an accidental explosion. Storage in magazines must also be planned.

If the new product has passed its performance and safety requirements in the pilot study, a parallel effort of evaluating the new explosive in its use environment is made. Here DOD and DOE differ significantly from industry. Military weapons are subjected to many extreme environments and the finished weapon with the new or modified explosive must undergo special safety testing to qualify it. Commercial explosives generally are used in somewhat more benign environments and the end-use safety testing is more limited and less expensive. End-use testing is required for permissible explosives (i. e., explosives that have been approved by the Bureau of Mines for use in underground coal mining operations). Their cap sensitivity, toxic fume production, and failure diameter must be established. For example, the minimum size bore hole required for a particular permissible explosive to function properly must be determined, as well as the safety of use in the underground coal environment (incendivity testing).

Samples from pilot production must, at this stage, be submitted to the Department of Transportation (DOT) for determination of shipping category. DOT has stated that addition of taggants does not change the shipping

category of the explosives used in the program.  $^{\scriptscriptstyle 5}$ 

The aspects of quality control are addressed during the pilot phase of development. Chemical and physical test specifications are established to control all component raw materials. Incoming taggants must be examined for foreign material and their code verified. If the taggants are gritty, such as the Westinghouse ceramic particles, there must be assurance that each taggant is properly coated with the desensitizing polyethylene or wax. Similarly, sampling and test schemes for product quality assurance are set up at this stage.

In some cases a company's management may decide that the change involved in the new explosive is small and complete requalif ication is not required. The extensive experience the management has developed in the history of its plant and products makes this, in many cases, an acceptable procedure. Although taggants would be added in only a smal I amount by weight, their use in explosives is sufficiently different from other constituents that it is the general consensus of manufacturers and other parties that addition of taggants will require complete requalification of al I tagged explosives.

### Description of Qualification Tests Normally Performed

Testing of explosives involves a wide variety of tests which must ascertain chemical composition, performance, sensitivity, and stability. Chemical composition analysis is a dominating factor since it is obvious that the manufacturer and user must know what he is using and what he has made. Chemical analysis methods are not the direct concern here, as taggants change the composition little, but it is to be emphasized that knowledge of the chemical composition must be a part of qualification assessment.

<sup>&#</sup>x27;Letter, P J Student (As\$oc of Amer Railroads) to R B Moler (Aerospace), June 27,1977

There is a large number of tests that are specific to evaluation of an explosive product. The details of these tests are given in several sources. <sup>610</sup>The most commonly used tests are briefly described below.

### Performance

Performance is determined by measuring detonation velocity, detonation pressure, pressure rise rate, shock sensitivity, and failure diameter in explosives and ballistic properties such as burn rate, muzzle velocity, and chamber pressure in gunpowder. The addition of small amounts of inert material to an explosive probably will not effect its performance significantly; however, performance must be demonstrated. Detonation velocity measurements consist of placing electric probes in precisely measured positions, detonating the explosive, and measuring the time that it takes the detonation front to pass between the probes with high-speed electronic equipment.<sup>11 12</sup> Initiation or shock sensitivity tests are done by separating a donor explosive from the test acceptor explosive by a measured gap. The gap is varied until a 50-percent probability of explosion of the acceptor explosive is estab lished.

Detonation pressure and pressure rise rate are measured by inserting transducers into the explosive material and recording the resultant pressures on fast response rate electronic equipment. Critical diameter testing, to establish the failure diameter of an explosive material, is accomplished by attempting to detonate varying diameters of the explosive. The

' 'Safety and Performance Tests, opcit

diameter at which 50 percent of the tests propagate to a high-order detonation is the critical or failure diameter.

The chamber pressure of gunpowder is measured by the use of spherical copper crush gauges or by transducers placed in the chamber. Burn rate is measured by a variety of methods, often by placing the powder in aVgroove, igniting one end, and measuring the velocity by high-speed camera, thermocouple, or pressure transducers. The muzzle velocity of the propelled projectiles can be measured by a variety of methods, including photography and make or break switches.

### Impact

Impact tests, although variable in nature and sometimes difficult to interpret, are critically important; their relationship to safety is obvious. They quickly provide information that categorizes the level of hazard of an explosive composition. They normally are used to tell if significant differences exist between explosive samples. Impact tests are not infallible and the results must be considered in relation to other type testing.

Impact tests range from laboratory-scale tests involving less than 35 mg to large-scale drop tests amounting to as much as 50 kg. As indicated previously, the initial tests would be laboratory-scale tests.

All laboratory impact machines are similar in principle. The energy source is a free-falling weight which impacts the explosive sample through a mechanical linkage. Criteria are established for distinguishin between positive and negative responses. The criteria differ for various laboratories so comparisons are only valid when made in a single laboratory. The tests consist of dropping the weight from varying heights onto samples of test explosives placed between them - sample weights are usually about 50 to 100 mg. The results are recorded as a go or no-go. A statistical analysis of the data determines the relative stimulus level corresponding to a chosen level of probability that the explosive will react to give a positive result according to the arbitrary cri-

<sup>6&</sup>quot;Safety and Performance Tests, opent

<sup>&</sup>lt;sup>2</sup>Joint Service Safety and Performance Manual tor Qua/l/l/cat/on of Explosives for Military Use (China Lake, Ca I If Nava I Wea pens Center, September 1971)

<sup>&</sup>lt;sup>4</sup>G R Walker, CARDE, Canada, E G Whitbread, ERDE, United K ingdom; D C Horning, NSWC/WO, U S A, *The Technical Cooperation Program* Manual of Sensitiveness Tests, TTCP Panel 0-2, February 1966

<sup>&</sup>quot;K R Becker, C M Mason, and R W Watson, Bureau of MinesInstrumentedImpactTester(Bureau of Mines) RI 7670, 1972

<sup>&</sup>lt;sup>10</sup>R W Watson, Card-Gap and Projectile Impact Sensitivity Measurements, a compilation, 1 C 8605, 1971

<sup>&</sup>lt;sup>12</sup>C M Mason and t G Aiken, *Methods for [ valuating Explo*sives and Hazardous Materials (Pittsburg Mining and Safety Research Center, Bureau of Minel), report No 1 (" 8541, 1971

teria, <sup>13 14 15</sup> Some manufacturers report a **50**percent probability height, but most report a threshold height.

Bullet tests are done by firing bullets or projectiles, usually .22, .30, or 50 caliber, into the test explosive. Powder loads are varied to obtain a range of projectile velocities. The test explosive may either be essentially unconfined in an ice cream carton, or highly confined in a heavy steel pipe, The minimum velocity required to obtain a reaction is reported, <sup>6</sup>

### Friction

In the manufacture, handling, and use of explosives there are many situations where frictional forces either are or could be present. Several test methods have been devised over the years and two of them have been used extensively in evaluating the taggants. In the Bureau of Mines tester a sample is placed on an anvil and subjected to the glancing, rubbing motion of a weighted shoe attached to the end of a pendulum that swings freely over the anvil. The shoe is either mild steel or a specified phenolic resin-bonded composite. The other test, developed by commercial industries, utilizes a 2-kg torpedo which is released to slide down a V track and obliquely impact the test sample. Both the height and angle of impact are independent variables, 17

A new precision instrument developed in West Germany and known as the BAM (after the Bundesanstalt fur Material prufung which developed it) seems to demonstrate improved discrimination. Some of the permissible will be tested on this new machine at the Bureau of Mines. <sup>18</sup>The friction surfaces in this device are ceramic. The load on the moving friction surface is varied until a response level is established.

### Stability

Stability testing may be divided into two general categories. One is simply long-term storage in which samples are removed periodically and retested to see if a significant change has occurred. The second category involves accelerated aging, which generally means subjecting the test sample to extreme temperature environments and then measuring the effects of the environment. Stability tests normally conducted include the above-described friction and performance tests, plus tests which are basically thermal in nature. These thermal tests provide a measure of some physical chemistry parameters of the explosive as well as being measurements of stability.

Among the stability tests widely used are:

Differential thermal analysis (DTA) in which identical containers, one containing the sample and the other a standard reference material, are set up in identical thermal geometries with temperature sensors arranged so as to give both the temperature in each container and the difference in temperature between the containers. The data are displayed as a DTA thermogram in which this temperature difference is plotted against the temperature of the sample. Such a plot is almost a straight line if the sample has no rapidly changing thermal behavior. Excursions below or above the baseline are due to endothermic, that is heat absorbing, or exothermic, that is heat releasing, reactions. The DTA analysis permits the interpretation of phase changes, decomposition, and melting points; from these, some kinetic information on thermal stability can be obtained. Sample sizes are in the order of 20 mg. Since the temperature of the thermal event is dependent, to some extent, on the heating rate, various heating rates are normally used. The standard rates are 100 C/rein and 20 C/rein.

Differential/ scanning calorimetry is very similar to DTA except the energy difference (calories) between the standard reference material and the explosive is recorded during the time-temperature program.

Vacuum stability is measured by placing a 5mg sample in a gas burette and then evacuat-

<sup>&</sup>lt;sup>13</sup>Safety and Performance Tests, op. cit.

<sup>&</sup>lt;sup>14</sup>Joint Service Safety and Performance Manual, op. cit

<sup>&</sup>lt;sup>15</sup>G. R. Walker, et al., op. cit.

<sup>&</sup>lt;sup>16</sup>R. W. Watson, op. cit.

<sup>⊡</sup>lbid.

<sup>\*</sup>Instruction Manual, Friction Tester, Bundesanstalt fur Materialprufung (BAM).

ing the burette. The flask containing the sample holder is then heated to an appropriate temperature for 20 to 48 hours. The gas evolved is measured by the manometer connected to the sample flask and then normalized to standard temperature and pressure. Test temperatures specified for military explosives are 1000 C and 1200 C. Dynamites and slurries are less temperature-resistant and usually contain volatile compounds; therefore, the test is really only useful for candidate booster materials, gunpowders, and explosive components of detonating cord.

The *Taliani* test is almost exactly the same as the vacuum stability test except that the test is usually run in a nitrogen atmosphere at **750** C at some laboratories and 93.30 C at others; taggant tests in one laboratory were run at 1200 C. At the end of 1 or 2 hours, the apparatus is vented to 1 atmosphere to eliminate the effect of the vapor pressure of water and the expansion of the original gas. The pressure change between 2 and 5 hours is measured.

In the *chemical reactivity test* (*CRT*) a sample of the explosive, approximately 0.25 g, is usually heated under a helium blanket at 1200 C for 22 hours. Tests have been conducted at other temperatures and times; tests with the Westinghouse taggants in dynamites were run at 1000 C for **4** hours. A cryogenic gas chromatography unit is then used to measure the individual volumes of the product gases, including such species as nitrogen oxide, carbon monoxide and dioxide, water, and other gases as may be determined necessary. This test is used principally to determine the reactivity of explosives with other materials, i.e., a compatibility test.

In the hot *bar* test a bar is heated to 2500 C and test samples of explosive are dropped on it. In the hot *tip* test, a 7\8-inch square by  $1\8$  inch-thick piece of steel is heated to white heat by means of a Presto-Lite torch and dropped on a test sample.

The stability bath test measures an exotherm and, therefore, decomposition at elevated temperatures. It is similar to the DTA, but uses larger samples. The sample is generally heated to a predetermined temperature and retained there for a number of hours. Visual evidence of decomposition is sought as well as the measurement of endothermic and exothermic reactions.

The *abel heat* test consists of heating samples in contact with methyl violet paper, usually at 71 °C. The elapsed time before the paper changes color is recorded. The test is applicable only to explosives containing nitrate ester. A similar test, the *German test* is done at 1200 C and a minimum time of 40 minutes allowed before a color change.

When the stability of an explosive is being compared to the stability of that explosive after an additive (such as the taggant) has been incorporated, the tests are normally conducted with significantly increased concentration of -that additive. Thus, while only 0.05 percent by weight of taggants is proposed to be added to explosives, stability tests are conducted with taggant concentration as high as so percent.

### Incendivity Testing (The Gallery Test)

Incendivity testing is done to certify explosives and blasting assessories for use in underground mines. Permissible explosives are those that pass the proscribed incendivity test. An explosive charge, which is loaded into a steel cannon (mortar), is fired directly into the gallery chamber containing a flammable mixture of natural gas and air or natural gas, air, and coal dust. There are two large gallery tests for explosives. on one test the incendivity is measured in mixtures of coal dust and natural gas in which the gas concentration (4 percent) is below the explosive limit of the mixture. In the other, the incendivity of explosives is measured in the presence of an 8-percent natural gas-air mixture.

The gallery represents a coal mine face, and is a 6-ft, 4-inch diameter steel tube, 80 ft long. The first 20 ft are charged with the flammable air/gas mixture and isolated by a thin membrane from the remaining 60 ft of tube which is filled with air and acts as an expansion volume. In the 4-percent concentration test, 1'A lb charges of the explosive are fired in the cannon under specified conditions. Ten trials are made; if any explosion occurs the explosive has failed the test. In the 8-percent concentration version, the amount of explosive that is being treated is varied from shot to shot to establish the weight required to cause a 50-percent probability of ignition. 19

### Cap Sensitivity

This test provides a simple means for differentiating an explosive from a blasting agent. A No. 8 detonator is inserted into a sample of given size and fired. If the sample is initiated to detonation, the material is classified as an explosive. A material that is not initiated to detonation is classed as a blasting agent. The test is used by the Bureau of Explosives to establish its shipping classification. The sample is put into a container at its approximate packaged density and a No. 8 detonator is inserted through the cover. The assembly is placed on soft ground in an isolated, safeguarded area, and the detonator is fired. If a crater is formed, the sample is considered to be cap-sensitive. The sample container is a 1qt, spiralwound, paperboard cylinder with cover, of the type used commercially for food packaging. Any commercial No. 8 blasting cap may be used as the detonator.

### Spark Sensitivity

The method of determinin<sub>g</sub> sensitivity to spark initiation is to subject the material to single discharges from a capacitor charged to a high voltage. The maximum energy of the spark discharge to which the material can be subjected without being ignited is a criterion of its sensitivity. Results are expressed as the maximum energy, in joules at 5,000 v, at which the probability of an ignition is zero. 20

### Charge Generation

Taggants are electrically nonconductive. A charge can be generated on them by pouring the taggant into the mixer; a charge generation test was therefore devised by one manufacturer. The test apparatus consists of an angled

chute (grounded stainless steel, 2 ft long), and an ungrounded stainless steel catch container with a known capacitance connected to an electrostatic volt meter. The taggants were poured from a polyethylene container, down the chute into the catch container. The charge developed is calculated from the voltage. The relaxation time is determined by the time required for the charge to dissipate. The charge generated, and relaxation time, can then be compared to materials commonly added to explosive materials, such as aluminum powder.

### Elements of a Taggant Compatibility Qualification Program

Taggants are a sufficient departure from the materials normally used in explosives and gunpowder to require full qualification of the new taggant-explosive material composition. While the taggants are fabricated from quite inert materials and are to be added in amounts of only a few hundredths of a percent by weight, the conservative safety philosophy of the explosives industry makes requalification necessary. As the detailed physical chemistry of the explosive reactions is not completely understood, it is not possible to safely conduct a few spot tests and generalize to all explosive materials from these tests. Table 23 outlines the elements of the type of qualification test program considered adequate by the OTA study team.

In principle, the manufacture of explosive materials consists simply of adding together the fuel, oxidizer, sensitizers, and stabilizers, mixing the components and packaging them in

### Table 23.–Elements of a Suggested Compatibility Qualification Program

- Unique with each manufacturer.
- Analysis to define the new explosive or ingredient
- Laboratory testing+impact, friction, thermal, chemical composition, electrical aging, chemical Interaction, performance
- Pilot production
- . Commitee and management review
- Early production and review
- Special tests.

lker, et al 01) cit

<sup>&</sup>lt;sup>20</sup>R W Watson, op cit

<sup>•</sup> Experience

SOURCE Off Ice of Technology Assessment

a casing (most explosives) or granulating the mixture (gunpowder). I n practice, however, each explosive mixture of ingredient is combined and processed in ways that differ significantly for each manufacturer. The number of ingredients used can vary from 2 (for ANFO) to 10 or more for some explosives and smokeless powders. The mixing process used can vary from the simple mixing of ammonium nitrate and fuel oil to form ANFO to a complex process involving preparation of the basic ingredients (one manufacturer grinds all ingredients to a 300 mesh powder for instance) and several mixing and processing stages. The equipment used also varies widely, from the wooden mixing equipment used by one manufacturer of nitroglycerine-based dynamites to the complex continuous process equipment used by one manufacturer of emulsions. End uses also vary; soft dynamites are often dropped or otherwise subjected to impact forces which would be unsafe if used with more brittle explosives such as TNT boosters. For these reasons, the qualification program must be unique to each manufacturer, and must reflect the exposure expected during the manufacture, storage, transportation, handling, and use of that particular product.

While it is true that the state of the art and laboratory instrumentation of physical chemistry are not sufficiently advanced to provide a detailed understanding of the process involved in all explosive reactions, it is certainly true that a careful and thorough analysis of the probable effect of adding taggants to explosive materials can provide a great deal of information. This information can be used as a preliminary screen to eliminate obviously dangerous explosive-taggant combinations, such as taggants placed directly in primary explosives or the use of gritty taggants. In addition the analysis can suggest critical tests and provide insight into the expected result and their interpretation. Proper analysis must therefore be considered the first element of any compatibility qualification program.

Laboratory testing must obviously play the central role in a qualification program. The exact tests to be performed are a function of the manufacturing process and end use, the results of the analysis, and the standard procedure of the manufacturers. At a minimum, tests must be conducted to demonstrate that the addition of taggants to explosive materials does not increase their impact and friction sensitivity; does not detrimentally alter the thermal, chemical, electrical, or storage properties of the materials; does not decrease stability; does not alter the chemical interactions involved (by eliminating interactions originally present or by introducing new interactions); and does not adversely affect the performance of the explosive material,

After the small-quantity laboratory tests and the analysis are successfully completed, pilotplant scale production should be initiated to investigate potential problems involved in the manufacturing, packaging, and storage of the tagged explosives and gunpowder. This testing should simulate, as nearly as possible, the actual manufacturing processes to be used if tagged explosives were to be produced.

Reviews, both technical and managerial, are an integral part of the qualification process. Substantive special reviews would probably be held at the end of the small-scale laboratory testing phase and at the end of the pilot production.

Through their qualification process the manufacturer would gain a great deal of experience in handling and workin, with the tagged explosives. This experience, and the general experience gained by working with the untagged explosives, and with other explosives, represent an important, although qualitative, part of the qualification evaluation process. For this reason, it is desirable for the manufacturers to conduct at least a large part of the qualification process. Some manufacturers do not have the requisite facilities and personnel to conduct the initial analyses and laboratory testing. This testing can be accomplished by outside agencies. It is obviously necessary for the manufacturer to participate in the pilotscale testing phase.

In the taggant compatibility testing which has taken place (presented below), the manu-

facturers were asked to suggest critical tests that were required before the pilot test manufacturing and distribution program could take place. That process is not sufficient for a formal compatibility qualification program. A minimum program, such as described above, must be conducted; additional tests, suggested by the manufacturer, may be made a part of the program.

### TAGGANT COMPATIBILITY TESTING ACCOMPLISHED TO DATE

Several hundred individual tests have been conducted in an effort to define the compatibility of identification taggants with explosive materials. These tests have generally been paired tests in which the reaction of a specific explosive material to a specific test is compared to the reaction of that material when identification taggant have been added. Materials tested include dynamite and other capsensitive high explosives, cast boosters, black powder, and smokeless powder.

Several varieties of identification taggants have been tested, including the current 3M baseline taggant in both encapsulated (type C) and unencapsulated (type A) form; a harder, more highly cross-1 inked variety of the taggant (type B); a higher melting point variety (type D); the Westinghouse ceramic taggant; and the Curie-point taggant.

No tests have shown increased explosive sensitivity due to the addition of the baseline 3M taggant (either encapsulated or unencapsulated). Similarly, no changes in electrical, general mechanical, or toxicity characteristics have been noted, Decreased chemical stability was noted, however, for one type of smokeless powder (Herco<sup>®</sup>);<sup>21</sup>22 decreased stability was also noted in one type of booster material (Composition B). The tests conducted to date clearly show that some chemical reaction takes place when Herco<sup>®</sup> powder or Composition B is mixed with a high concentration of 3M taggants and then heated to a high temperature; further research is required to deter-

mine the nature and cause of the reaction, the extent of the safety hazard created, and what remedial steps may be feasible. Extremely limited testing has indicated no significant change in ballistic velocity or chamber pressure when the 3M taggants are added to smokeless powders, even at extremely high taggant concentrations.

The hard 3M taggants (types B and D) did cause significantly increased sensitivity in capsensitive explosives, as did the Curie-point taggant and the unencapsulated Westinghouse taggant.

Compatibility testing for the detection taggant materials has been recently initiated with black powder and cap-sensitive high explosives. No data has been formally reported; toxicity and mutogenacity tests of the materials themselves have been negative.

The following paragraphs briefly summarize the tests so far conducted. The extent of testing described in the tables includes those whose results had been formally reported by March 1, 1980. However, OTA has reviewed all testing about which information was received, whether or not formal reports have been issued. Tests are continuing.

### Dynamites

The paired compatibility tests conducted with dynamite and with EDCN are summarized in table 24, In this table and those which follow in this section, an asterisk by the taggant type indicates a sensitization or other indication of noncompatibility The other symbols are defined in the legend. As can be seen from the table, no significant differences in response to the various tests evaluated were ob-

<sup>&</sup>lt;sup>21</sup>Letter, W. O. Cashin (Hercules) to S. E. Salvers (Aerospace), "Tagging: Program—Smokeless: Powder," Aerospace purchase order W-0214, Nov. 7, 1979.

<sup>&</sup>lt;sup>24</sup>Letter, D. Seaton/A. Payne (LLL) to E. James (OTA), "Compatibility Screening of Various Taggants With Hercules Corp. "Herco<sup>w</sup>" Propellant," Dec. 7, 1979.

				1	Test type				
					Electro-				
	Drop		Sliding	5 - k g	static			Chemica	11
Type of dynamite	weight	Friction	rod	impact	discharge	e Heat	(PaTbAo I )	reactivity	
Vibrogel	A, C	A, C		С	С				
R e d H	A A,C	A,C		A,C	С				
	0 % A , C	A,C		A, C	с				
Unig e	I A, C	A,C		A,C	с				
Gelobel AA	A , B *		Х *	A, B*, W*					
EGDN	C,W, X*		A, B*, C,	C , W *	C, \	N, X,D, E			W.x "
			W, X,D, E	х					Α΄
Nitroglycerin.	С	С	Α΄	С		Α ′			
90/10 EGDN/NG : :			С,Ү,Ζ*	D *		С			
60% ammonia gelatin	W			W		W			
60% semigelatin	W			W			W	W	
40% specia	i I			W			W		
85% hydrive				W				W	
850/o gelatin	W	W	W	W					
Gelatinous permissible	W	W	W	W					
60/40 NG/EGDN		W	W	A* 0.5*					A'
Power Primer		A',C'	Y*, E,A*, C	A", C,D^	A'				

Table 24,–Summary of Compatibility	Tests Conducted With	Dynamite and Dynami	te Ingredients
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X-unencapsulaled Westinghouse ceramic taggant

B-unencapsulated hard cross.linked 3M taggant C-encapsulated 3M laggant D-encapsulated higher melting point 3M taggant

A- unencapsulaled 3M laggant

Y-encapsulated Curie-point laggant Z-unencapsulah?d Curepoint faggant

' indicating Irradiated taggant "-md[caled noncompatibility

E –unencapsulated higher melting point 3M taggant

W-encapsulated Westinghouse ceramic taggant

SOURCE Office of Technology Assessment

served for any of the dynamites into which either the encapsulated or unencapsulated baseline 3M taggants were added. Unencapsulated hard or gritty taggants of various sorts caused sensitization under impact testing.

In addition to those tests shown in the table, a small number of drop weight tests were conducted in which the 3M taggants (both baseline and the cross-linked varieties) were encapsulated in several high melting point resins. Sensitization of both Power Primer and 90/10 EGDN/NG were noted for most combinations tested.

A final series of tests examined the stability of tagged Power Primer, Coalite-8S, and EC DN under both accelerated aging (higher temperature) and ambient aging conditions. The Power Primer showed a significant decrease in stability as measured in the Abel test after 2 months aging at 400 C. Unfortunately, no control test was conducted with untagged Power Primer, so no compatibility judgment can be made. No other signs of decreased stability appeared in the other tests.

### Gels and Slurries

A smaller number of tests was conducted to compare the response of tagged and untagged gels, slurries, and emulsions. These tests are summarized in table 25. In no case tested was there an indication of changes in sensitivity or stability due to the presence of taggants. Tests were also conducted to determine if the addition of taggants to the gels and slurries would affect performance as the explosive materials aged. Tests included initiation sensitivity and detonation velocity as well as visual observation of gel quality. Both ambient and accelerated aging tests were conducted. No changes in these properties were observed. Cap-sensitivity tests at low temperature were also conducted with special sensitized emulsions containing a combination of the baseline 3M and the Westinghouse taggants. The performance

									Test	type				
				Drop	Sliding	Projectile		Chemica			Weight loss under	Hot	Hot	Electro- static
Type gel or slurry				weight	rod	impact	Friction	stability	stability	Taliani	heat	tip	bar	disch
Gel-po ● H20, MMAN	wer N, SN, AN	Α-	2	A,C			A,C	А	С					С
Mixture of tovex		300, t	ovex	320 C	С	С			Ũ			С	С	
Gel-coal				С			С			С	С			С
Gel-powder				С			С			С	С			С
Permissible	(unspecified)			W	W		W							

Table 25.–Summary of Compatibility Tests Conducted With Gels and Slurries

A-unencapsulated 3M taggant B-unencapsulated hard cross linked 3M taggant C-encapsulated 3M taggant D-encapsulated higher metting point 3M taggant E-unencapsulated higher metting point 3M taggant W-encapsulated Westing ceramic taggant X -unencapsulated Westinghouse ceramic taggant

' indicating iradiated taggan "MMAN –monomethylamme nitrate SN –sodium nitrate

Y-encapsulated curie-poml taggant

Z-unencapsulated curie point taggant

AN-ammonium nitrate

SOURCE Office of Technology Assessment

of the tagged explosives was superior to the untagged control samples. It should be noted that the reason for any change in performance should be carefully investigated.

### Cast Boosters

The tests comparing the sensitivity and stability of tagged and untagged cast boosters are summarized in table 26. The 3M taggant did not affect the sensitivity of any of the cast boosters explosives in any of the paired testing. Evidence of decreased stability was observed in tests conducted of molten booster material to which 3M taggant had been added. I n a series of tests, Goex heated booster explosives to temperatures between 1200 and 1650 C for a period of 16 hours. " Evidence of decomposition of the explosives occurred, including bubbling, dislocation, and the appearance of voids. Pentolite (50/50 PET N/TNT), Octol (25/75 TNT/HMX), and an explosive mixture similar to Composition B were tested. The only paired test was with the Composition B-like material. Composition B normally contains just under 30 percent TNT and just under 60 percent RDX, with the rest being wax. The Goex mixture used A-3 instead of pure RDX. As A-3 contains approximately 9 percent wax, the composition of the Goex Composition B differs from standard Composition B. Ignoring 💛 Letter J W Heron (Goex, I nc. ). to S Derda (Aerospac e), "Status of Tagging Program, " Aerospace purchase order W-025, Tab\_reptDTD10/4/79

this nomenclature difference, the tagged composition B showed significantly more severe degradation at the 120° C test temperature than did the untagged composition B at a 1300 C test temperature. As no control tests were conducted with an untagged batch of explosives for the Octol and Pentolite tests, it is impossible to ascertain if the taggants were responsible for the observed reactions. While testing is often conducted at temperatures above those encountered in normal use, it is extremely dangerous to heat common booster materials to temperatures above 1200 C. The test serves as an indication of a potential compatibility problem. More carefully controlled tests are currently underway at the Naval Surface Weapons Center, White Oak, Md. Preliminary indications are that a 50-50 mixture of unencapsulated taggants and TNT undergoes a chemical reaction at 1200 C; research is continuing to determine the nature, cause, and safety significance of this apparent incompatibi l it y.

On July 15, 1979, an explosion and fire occurred at the Goex factory in Camden, Ark., causing damage which Goex has estimated at \$2 million. The explosion took place in a meltpour operation in which scrap high explosives were being melted. Goex, inc., asserts that the scrap materials available for melting down included some materials containing 3M identification taggants. Goex further asserts that the explosion began in a way that resembled the

Type of booster	Drop weight	Vacuum stability	BAM friction	Pendulum friction	Sliding rod	Therma stability	
PETN ,	A, B. C,X*,W		A,B, X	w	C,W		
Pentolite	A, B,X*	A, C,Y,Z	A.B, X		W		
50/50 pentolite	W			W	W		
Composition B	. W			W	W	С*	
T N T	W			W	W		
R D X	W			W	W		

### Table 26.-Summary of Compatibility Tests Conducted With Cast Boosters

B-unencapsulaled hard cross linked 3M taggant C -encapsulated 3M taggant Y-encapsulated Cune-point laggant

X- unencapsulaled Westinghouse ceramic taggant

Z-unencapsulated Curie-poml taggant

D-encapsulated higher melting point 3M Liggant '-idicating Irradiated taggant E unencapsulated higher melting point 3M taggant '-Indicated noncompatibility

w-encapsulated Westinghouse CETAMIC taggant

SOURCE Off Ice of Technology Assessment

A-unencapsulated 3M taggant

reaction of tagged booster material in the above tests. Goex claims that the explosion must have been caused by the taggants. The Aerospace Corp. asserts that no tagged booster material was located at the Camden factory at this time, and that furthermore the low concentrations which Goex asserts were present could not have initiated an explosion; the tests to which Goex refers involved extremely high taggant concentrations, OTA is not familiar with the facts regarding the possible presence of taggants, and is not aware as the report goes to press of any experimental data on the possible destabilizing effects of low concentrations of taggants mixed with TN T/RDX mixtures.

As would be expected, the more gritty taggants clearly showed evidence of sensitizin, the booster explosives. In the case of the Curiepoint taggant, sensitization occurred even for encapsulated taggants; these are the only tests showing sensitization with encapsulated taggants.

### Black Powder

The black powder compatibility test results are summarized in table 27. Neither the black powder nor the black powder tailings are sensitive to either the friction or impact tests conducted, even for the gritty taggants, However, no stability tests were conducted.

### Table 27.–Summary of Compatibility Tests Conducted With Black Powder

	Test	type
Type of powder	Drop weight	BAM frlctlon
FFFg Tailngs	A,B, X A.B, X	A,B, X A,B, X

A - unencapsulated 3M tagant

B-unencapsulated hard cross-linked 3M taggant

X –unencapsulated Westinghouse ceramic taggant

SOURCE Off Ice 01 Technology Assessmem

### Smokeless Powders

The compatibility tests conducted with smokeless powders are summarized in table 28. Only the encapsulated 3M taggant (type C) was tested. Tests were originally conducted by Hercules, Olin, and Du Pent on their own smokeless powders.<sup>2425</sup> No evidence of sensitization or change in electrostatic properties was observed. I n the case of the Herco<sup>®</sup> powder, however, the Taliani and German heat tests both indicated a significant decrease in stability due to the addition of the taggants (in a 50-percent concentration) to the smokeless powder. (Although Hercules tested only Herco<sup>®</sup> powder, Hercules believes that their

<sup>&</sup>lt;sup>24</sup>W. O. Cashin letter, op. cit.

<sup>&</sup>lt;sup>12</sup>Letter, A. B. Opperman (Du Pont) to S. Derda (Aerospace), "Process and Product Laggant Compatibility Demonstration Lest for DuPont Smokeless Powder, Phase 1," Aerospace purchase order W-2030.

					Test t	уре				
<u>Type of</u> powder	Impact	Friction	Electro- static discharge	Impmge- ment	Critical height to explosion	DSC	Tallanl	German heat	BallIstic velocity	BallIstic pressure
Hercules HPC Hercules bullseye Hercules Herco <sup>rr, r</sup>	с	с	с	с	с	с	с*	с с,		
Du Pent H1-skor	С	с	С					С	С	С
Du Pent PB	С	С	С					С	С	С
Du Pent IMR 3031	C	С	С					С	С	С
Du Pent IMR 4064	С	С	С					С		
DIIN 231								С		
Diln 296								С		
DIIN 452								С		
Olln 540								С		
Olln 473								C		
Dlin 571								C.		
Olln 680								C		
Olln 748								C		
DIIN 760.								° C		
Olln 785								c		
Olln W C 571								Ū	с	с

### Table 28.–Summary of Compatibility Tests Conducted With Smokeless Powders

C-encapsulated 3M taggant

"-Indicaled noncom pall blirly

SOURCE Off/cc of Technology Assessment

other brands of powder designed for the reloading market are so similar to Herco" that similar test results could be expected. OTA believes that this is highly likely for the four other Hercules brands that are chemically identical to Herco"; it may not be the case for the three Hercules brands with different compositions.) As no changes were noted for the Du Pent or Olin Abel tests, the Herco<sup>®</sup> tests were repeated at the Naval Ordnance Station. Indian Head, Md. The decreased stability was confirmed. A more carefully controlled series of tests was then conducted by the Lawrence Livermore Laboratory (LLL) for the Aerospace Corp. in an attempt to isolate the element or elements of the taggant materials which are responsible for the incompatibility. <sup>26</sup> Briefly, the tests indicated that there exists an incompatibility between something in the Herco" and the melamine/alkyd which forms the basic matrix of the 3M taggants. It may be a basic reaction with the melamine/alkyd or with the catalyst used to speed up the cure time. There may also be reactions occuring between the taggant pigments and the Herco® powder. The LLL tests are continuing in an attempt to resolve the issue.

At the present time, there appears to be an incompatibility between the 3M taggants and the Herco" smokeless powder. Hercules has indicated that it does not consider the combination safe and has stopped all work on it. OTA feels that, on the basis of the tests just described, the conclusion must be drawn that the 3M taggants cannot be safely added to the Herco<sup>®</sup> powder unless the present incompatibility is resolved. Some justification exists for questioning the validity of tests using severely increased concentrations of the taggant materials (50 percent in the tests v. 0.05 percent of encapsulated material in the proposed taggant program), but it has not been demonstrated that there is a threshold concentration below which the problem disappears, and that such a threshold would never be exceeded in practice.

Preliminary ballistic tests have been conducted on tagged WC 571 shotgun powder manufactured by Olin. Ballistic velocity, chamber pressure, and time to initiate burning were measured. Tests were conducted at three temperatures ( $-30^{\circ}$  C,  $20^{\circ}$  C, and  $50^{\circ}$  C) and four taggant concentrations (2, 4, 10, and 20 times the recommended concentrations), both with the taggants mixed in the powder and

<sup>&#</sup>x27;" [| 5f',]ton A l',]vne lettt'r, OIJ C lt

with the taggants separated and placed directly over the primer flash hole.

The Olin rationale for such extreme tests condition (up to 20 times the nominal concentrations, 100-percent segregation) was an attempt to evaluate the worst-worst case conditions that might appear due to segregation of the taggants from the powder during manufacture, transportation, and storage.

No deviation from acceptable ballistic performance was noted for the ambient- and hightemperature tests. A steady decrease in velocity and pressure was noted with increasing taggant concentration. The practical signifance of this depends on the extent to which taggant concentration would vary in actual use by handloaders, which can and should be established by careful testing and statistical analysis. At the low-temperature condition two anomalous test results occurred. Evidence of improper ignition occurred in 1 of the 20 firings at the 20 times normal concentration, 100percent segregation condition. Improper ignition would constitute a safety hazard as the round might not clear the barrel, Significantly reduced bal I istic performance occurred on 1 of the 20 tests at 4 times nominal taggant concentration, with the taggants and powder mixed. No other performance degradation was noted, even under conditions of higher taggant concentrate ion.

### DISCUSSION OF COMPATIBILITY TEST RESULTS

Several hundred tests have been conducted to investigate the compatibility of explosive materials with identification taggants. Most of the tests have been conducted with the baseline 3M taggants and variations of these taggants; a large number of tests, however, have also been conducted with several other candidate taggant materials. Compatibility tests have included those designed to indicate insensitivity, decreased creased stab i l it y, changed electrical properties, and changed performance, Explosive materials have included dynamites, gels, emulsions and slurries, cast boosters, bldck powder, and smokeless powders. A full set of qualification tests has not been completed on any single explosive product and only a small fraction of the hundreds of products has had any testing. Given these limitations, it is still possible to draw some tentative conclusions on the compat ibi I ity of taggants with explosive materials (which may change as more data becomes available) and to discuss the implications of these results for the taggant program,

First, it is important to realize the purpose of ~ compatibility qual if i cat ion testing program. In brief, a set of tests is established on the basis of analysis, the projected manufacturing, storage, transportation, and end-use processing of the material, and the normal procedures and experience of the organization conducting the tests. If the candidate explosive product fails to pass any of the critical tests in the series, it is judged to have failed the qual ification test program. If a flaw can be corrected, then the tests can continue, but the material must pass all of the critical tests, not just a majority or a certain fraction.

There is no indication that the 3M taggants are incompatible with dynamites, gels and Slurries, or black powder.

Composition B booster material and Herco" smokeless powder do show significantly reduced stability in the presence of the 3M identification taggants. Furthermore, careful testing appears to indicate that the incompatibility is with the basic melamine/alkyd material of the taggants, rather than with a particular pigment or the polyethylene encapsulate. Tests, similar to those conducted with Herco", were conducted with other smokeless powders; no loss in stability was noted for other Hercules powders, or for the Olin or Du Pent smokeless powders. The reaction, therefore, probably is between the melamine/alkyd and one of the sensitizers or stabilizers of the Herco<sup>®</sup>. As the formulations of both Herco<sup>®</sup> and the 3M identification taggants currently stand, the two are not compatible. Further investigation may isolate the element of incompatibility, and it may be possible to replace elements in either the Herco<sup>®</sup> or the taggants to remove the incompatibility. It is not yet possible to tell whether the booster material incompatibility is with the basic melamine/alkyd or with one of the components of the taggants.

Both the smokeless powder and booster material tests took place at high temperatures, and, in most of the tests, at high-taggant concentrations. The temperature used for the smokeless powder test was higher than would be expected in actual manufacture, storage, or use; the temperature used for the cast booster is sometimes reached in manufacturing processes. In each test, a taggant concentration of so percent was used rather than the 0.05-percent tagging concentration suggested for routine use. The tests, nonetheless, indicate that the stability of the materials has decreased, due to the addition of taggants, and that a reaction is taking place between elements of the taggants and elements of the explosive material. Standard qualification test procedure requires that such evidence be considered a sign of an existing incompatibility between the materials. Careful ly controlled testing, and extensive analysis must be completed before it can be determined if the observed evidence of incompatibility does, in fact, indicate a potential safety problem during the manufacture, storage, transportation, and use of the tested materials. Unless demonstrated otherwise, it must be assumed that it is unsafe to add the taggants to that smokeless powder or the booster material. Until the elements of the incompatible lity have been identified, a question remains as to the safety of adding the taggants to similar smokeless powders and booster materials, although tests with other smokeless powders and boosters have shown no evidence of incompatibility.

The significance of the 01 in ballistic property tests cannot be fully assessed at this time. The Olin tests indicated that increasing taggant concentrations lead to a reduction in velocity and pressure, and this could create a problem if and only if it proves impossible to mix taggants with smokeless powder in such a way as to avoid extreme variations in taggant concentration from one round to the next. Testing is required to establish how great a variation in concentration could be expected using reasonable manufacturing methods, and normal transportation, storage, and loading procedures. The Olin tests did show one case of poor performance (at four times the suggested taggant concentration), but performance anomalies sometimes occur without taggants, and a single anomaly is not enough to justify a prediction as to whether taggants would increase the frequency of such occurrences. The segregation tests were conducted with 100-percent segregation, which appears quite unreal istic. Testing is needed to establish the extent of segregation which might occur before a realistic worst case can be defined. Unlike the Herco<sup>®</sup> and Composition B cases, the Olin ballistic property tests do not appear to OTA to constitute sufficient evidence to require presumption of an incompatibility. It remains true, however, that no presumption of compatibility can be made until adequate ballistics tests have been conducted.

This raises the question of the value of a taggant program from which smokeless powders and cast boosters were excluded. As noted in chapter VI, smokeless powders are used in a significant percentage of criminal bombings (approximately 20 percent) and cause 10 to 20 percent of deaths and injuries. As also noted in chapter VI, criminal bombers are likely to react to a taggant program, If smokeless powders are not tagged, then a logical reaction would be for a large number of bombers to switch to the use of smokeless powders. Although bombs using smokeless powder are considerably less efficient (lower specific energy) than those using cap-sensitive high explosives, smokeless powder bombs are responsible for a considerable number of injuries and deaths. Effective controls over smokeless powder by means other than taggants may be possible but appear unlikely. Booster material is rarely used as a bomb filler. It is used, however, to initiate blasting agents. The current BATF plan would be to not directly tag blasting agents, but to tag the booster and detonators used to initiate the blasting agent. Exclusion of boosters from the taggant program may well require an alternate control mechanism for blasting agents. Given the extremely large quantity of blasting agent produced (3.4 billion lb annually), any other control mechanism may have serious cost consequences.

The above discussion concerned the results of the tests to investigate the compatibility of the baseline 3M taggants with explosive materials. Tests were also conducted using hard or gritty taggants, In all cases, the unencapsulated hard taggants caused increased sensitivity to the drop weights, and, in most cases, to the sliding rod tests. The ceramic Curie-point taggants caused increased sensitivity in some cases even when encapsulated, although no incompatibility was noted for the Westinghouse or hard-core 3M taggants when encapsulated with polyethylene. When a hard resin was used as an encapsulant, the 3M taggants showed a clear sensitization of PETN. The implications of these tests are obvious. I-lard or gritty taggants must be encapsulated. The encapsulated material should not only be soft but it should also be a heat sink. The use of a soft additive is a common desensitizer in military explosives. Composition B and other RDX-based explosives include approximately 1 percent wax with a softening point in the 800 F range.

The tests show that encapsulated gritty taggants, such as the Westinghouse ceramic taggant, may be alternatives to the baseline 3M taggant. As even a small amount of the unencapsulated material (0.01 percent) causes increased sensitivity, however, great care must be exercised to ensure essentially 100-percent" encapsulation; this may seem to create an impossible quality control problem. However, the problem may not be as difficult as it first appears. If 99 percent of the taggants are encapsulated, then unencapsulated taggants would constitute only .00025 percent by weight of the explosive, almost two orders of magnitude less than the amount demonstrated to cause increased sensitivity. Tests of those extremely low levels might well show no increased sensitivity.

As noted above, much compatibility testing remains to be accomplished. Identification taggants have undergone comprehensive testing with a representative sample of dynamites, gels, slurries, cast booster materials/smokeless powders, and black powder; even after the resolution of the compatibility questions which testing so far has revealed, it would eventually be necessary to test taggants with all such materials before instituting a comprehensive tagging program. In the case of detonators and detonating cord, compatibility testing has not been completed even with a representative sample. Compatibility testing of detection taggants started only recently, and with the exception of testing with detonators it is less far advanced than compatibility testing of identification taggants.

It is necessary to resolve the incompatibility observed between the 3M identification taggants and the Composition B booster material as well as the Herco<sup>®</sup> powder however, before it makes any sense to finish the rest of the tests with other materials. The resolution of the smokeless powder incompatibility could take any of several forms, including:

- Reformulation of the 3M taggant- this could require starting essentially from scratch in the taggant-testing program, as the reformulated taggant would undoubtedly exhibit different compatibility, as well as survivability properties.
- It might be possible to develop a different taggant that proved compatible with smokeless powders, and to use the existing 3M taggant for explosive materials with which it is compatible.
- Reformulation of the Herco" powder this may or may not be easily accomplished, once the element or elements that react with the taggant are isolated. This option would only be viable if no other smokeless powder were found to be incompatible.
- Exclusion of Herco" from the taggant program —the economic effects on competition would need to be carefully considered, as would alternate control mechan isms.

- Exclusion of smokeless powders from the identification taggant program such an exclusion would rely on the fact that smokeless powders would be less effective than cap-sensitive high explosives and that the detonators would be tagged. OTA believes that this last approach may not be viable- too many people are currently killed or injured using smokeless powders and the numbers would almost certainly increase if that approach were adopted. Alternate control mechanisms for smokeless powders would be required,
- Demonstrate ion that the observed stability problem does not constitute a safety hazard. The observed decreased stability occurs at elevated tern peratures and at more than two orders of magnitude higher taggant concentration, As the decomposition

rate is both temperatu r-e and concentration sensitive, it may be that **no** safety hazard exists under realistic conditions If it could be positively demonstrated that the decomposition rate was within the normal I I y accepted range for temperature regimes and concentrations which reflect worst case actual use conditions, then it may be possible to add taggants to the smokeless powder, particularly if no furincompatibilities surface. ther However, demonstrate ion of safety would have to be quite convincing to overcome the currently perceived incompatibility.

A resolution of the booster incom pat bility problem could be accomplished by a s milar set of methods, once the elements of t leincompatibility have been identitied.