The results for the quality of vapor determined in this manner are compared with the experimental results, and those computed by the theoretical method, in Table V. The agreement in this case is better than may usually be expected.

OTHER APPLICATIONS

These principles and methods are of wide application to all types of vaporization processes, only a few of which have been demonstrated. The application of the thoretical methods to natural gasoline absorption and fractionation has been treated (13). The same theoretical methods as applied to complex mixtures may be used for studies in fractionation of petroleum products at all pressures. The extremely simple application of the equilibrium curves (56), Figures 9 and 10, to distillation problems of complex mixtures, making possible all the simplifications of the graphical method of McCabe and Thiele (50) for binary mixtures may not be theoretically exact but is justified in many cases, and appears satisfactory for fractionation even at high pressures.

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Analogs of Tetryl Trinitrophenylnitraminoethyl Nitrate (Pentryl) I.

LE ROY V. CLARK, Bureau of Mines Experiment Station, Pittsburgh, Pa., and Explosives Experiment Station, Bruceton, Pa.

THE safety of permissible explosives depends not only upon the explosive but also upon the effectiveness of the detonator used to initiate its detonation. The use of ineffective detonators-that is, low-grade detonators (17) or detonators that have become phlegmatized because of deterioration of the detonant under unfavorable storage conditions--greatly increases the chances of misfire, delayed shots, imperfect explosion, or simple ignition, with all the attendant jeopardy to personnel and property.

An investigation is made to determine certain of the chemical, physical, and explosive properties of sym-trinitrophenylnitraminoethyl nitrate (pentryl). Tests show that it can be used as a base charge in "compound" detonators for the initiation of detonation of industrial and military explosives. For this purpose it may be used to replace picric acid, tetryl, lead styphnate, or various of the nitrated polyhydric alcohols commonly used as base charges. Numerous tests show pentryl to be an appreciably more powerful explosive than TNT and point to its value for those uses in which TNT has been of proved value.

In pursuance of the campaign waged by the Bureau of Mines for safety and efficiency in mining operations, it is the continuing policy of the Explosives Division to investigate the chemical and explosive properties of all substances that give promise of use either as initiating agents or as base charges in detonators. In this paper the writer presents the results of an investigation of sym-trinitrophenylnitraminoethyl nitrate, hereafter termed "pentryl," and gives comparison with other compounds previously investigated by the Explosives Division.

Moran (16) in 1925 prepared pentryl by the nitration in mixed acids of 2,4-dinitrophenylaminoethanol which is obtained by the action of 2,4-dinitrochlorobenzene on aminoethanol. E. von Herz (11) in 1931 was granted a German patent for the use of pentryl for certain military purposes; he prepared pentryl by the nitration of β -hydroxyethylaniline (10).

Davis (4) in 1926 was granted a patent for the preparation of sym-trinitrophenyl-N-butylnitramine, which compound will be investigated at a later date by the writer. This compound, as well as pentryl, may be regarded as an analog of tetryl, as shown by the following formulas:



PREPARATION OF PENTRYL

PREPARATION OF 2,4-DINITROPHENYLAMINOETHANOL. The preparation of phenylaminoethanol and substituted phenylaminoethanols has been studied by numerous investigators, notably Ladenburg (15), Demole (5), Knorr (14), Otto (20), Badische Anilin- und Sodafabrik (1), Gault (7), and von Herz (10).

The method used by the writer for the preparation of 2,4-dinitrophenylaminoethanol was that proposed by Moran (16):

One hundred parts of 2,4-di-nitrochlorobenzene are dissolved

nitrochlorobenzene are dissolved in 510 parts of 95 per cent ethyl alcohol, 30 parts of amino-ethanol are added, and the solu-tion is warmed to 70° C.; 20 parts of sodium hydroxide dissolved in 30 parts of water are added slowly to the rapidly agitated alcoholic solution, the temperature of the mixture being maintained at 70° to 80° C. from 30 minutes to one hour after addition of the sodium hydroxide is completed. The mixture is then cooled and filtered free of the precipitated sodium chloride and a by-product of the reaction to be mentioned sodium chloride and a by-product of the reaction to be mentioned later. With the filtrate concentrated to about 600 cc., the 2,4dinitrophenylaminoethanol, after standing a few hours, is thrown out in the form of large orange-yellow crystals which cling tenaciously to the sides and bottom of the vessel. During the concentration of the filtrate, about 1100 cc. of alcohol may be recovered for re-use.

The yield, after washing with alcohol to remove adhering mother liquor, was found by the writer to be 70 per cent of the theoretical. The crude product contains a small amount of occluded sodium chloride. After repeated recrystallization from alcohol, a melting point was established at 92° C.

As a by-product of this reaction there is precipitated with the sodium chloride a fine powdery product, lemon-yellow in color and melting at 222° C. This compound, to be studied later, is the bis-dinitrophenylaminoethanol:



NITRATION OF 2,4-DINITROPHENYLAMINOETHANOL (PEN-TRYL). Pentryl may be prepared as follows:

One hundred grams of 2,4-dinitrophenylaminoethanol are dis-solved in 1000 grams of 95 per cent sulfuric acid, keeping the temperature between 20° and 30° C. Since there is no marked tendency for the temperature to rise, it is easily controlled with a bath of cold water. The sulfuric acid solution is then added during 12 to 15 minutes to 300 grams of nitric acid (47° Bé.), keeping the temperature at 20° to 30° C. The addition com-pleted, the temperature is held at 30° C. during a half-hour period, then slowly raised to 40° where it is again maintained constant for a half-hour. At the end of this second period of heating, the temperature is raised to 50° and maintained constant over a third half-hour period. third half-hour period.

During the later stages of nitration, pentryl separates out as a fine powdery suspension in the spent acid from which it may be recovered by drowning in ice water. The yield was 90 per cent of theoretical. The acidic product is stabilized by washing with water and cold sodium bicarbonate solution. If heating is too rapid during the early stages of the nitration, the product has a tendency to form into small, hard granules from which it is extremely difficult to effect complete removal of spent acid.

The stabilized crude product may be recrystallized in benzene, from which the pure product separates in the form of minute cream-colored crystals. Repeated recrystallization established the melting point at 128° C. The loss during purification amounts to about 6 per cent.

Tests made to determine the effect on yield of the use of smaller quantities of sulfuric acid showed that the yield might be increased as much as 3 per cent by decreasing the quantity of sulfuric acid, but the increasing difficulty encountered in effecting solution of the 2,4-dinitrophenylaminoethanol and the increasing viscosity of the nitration mixture make such a reduction of questionable value.

PHYSICAL PROPERTIES OF PENTRYL

Pentryl, prepared according to the above method, is in the form of microscopic crystals, nearly white in color with a slight cast of yellow.

The density of pentryl under various conditions and its solubility in various solvents have been determined by the writer. The true density is 1.82, yet its apparent density is only 0.45. When compressed in a detonator shell at a pressure of 3400 pounds per square inch (239.0 kg. per sq. cm.), it has an apparent density of 0.74.

The solubility in certain of the more common organic solvents is as follows:

| Solvent | Soly, in 100 Gr. At 25° C. | AMS OF SOLVENT At 50° C. |
|----------------------|-------------------------------|-----------------------------|
| | Gram | Grams |
| Toluene | 0.63 | 1.70 |
| Benzene | 0.70 | Very soluble |
| Ethanol | 0.11 | 0.48 |
| Methanol | 0.67 | 2, 14 |
| Chloroform | 0.07 | 0.26 |
| Carbon tetrachloride | Trace | Trace |
| Ethylene dichloride | 0,72 | 2.68 |
| Ether | 9.16 | |

pact tests on pentryl compared with similar tests on tetryl, picric acid, and TNT are as follows:

| Explosive ^a | MAX. HEIGHT OF DROP OF 2000-GRAM Weight for "No Explosions" in 5 Trials |
|------------------------|---|
| | Cm. |
| Pentryl | 305 |
| Tetryl | 27.5 |
| Picric acid | 42.5 |
| TNT | >100 |
| | |

^a 0.02-gram samples were used.
 ^b 100 cm, when 500-gram weight was used.

Results of tests with the type A pendulum friction device are as follows:

| Explosive ^a | MAX. HEIGHT OF FALL OF PENDULUM ^b FOR "NO EX- PLOBIONS" IN 3 TRIALS | WEIGHT Added TO Steel Shoe | Swings of Pendulum in Each Trial |
|------------------------|---|--|--|
| | Cm. | Kq. | |
| Pentryl | 25 | 20.0 | 9-11 |
| Tetryl Pieric acid | | 20.0 | 27~30 4547 |
| TNT | >150 | 20.0 | 62-65 |
| | • • • • • • | | |

^a 5.0-gram samples used in each trial.
^b Type of shoe, steel.

The results of these tests indicate that the sensitiveness of pentryl to impact is about the same as that of tetryl, is somewhat greater than that of picric acid, and is much greater than that of TNT. The sensitiveness to friction is somewhat greater than that of tetryl, and is much greater than that of either picric acid or TNT.

PREPARATION OF TEST DETONATORS. After Wöhler and Matter (27), the experimental detonators tested during this investigation have been prepared in the following manner unless otherwise specified: A base charge of 0.50-gram of the explosive to be tested is weighed into a No. 8 detonator shell, and a priming charge of 0.20 gram of mercury fulminate is added. Both base and priming charges are then compressed under a reënforcing capsule at a pressure of 3400 pounds

> per square inch (239.0 kg. per sq. cm.). This pressure has been adhered to in order that results may be on a comparable basis with those from a recently completed investigation of diazodinitrophenol (DDNP) (2).

> EFFECT OF PRES-SURE ON EXPLOSI-BILITY. Pentryl, both when unconfined or after having been compressed at high pressures in a detonator shell (either with or without a reenforcing capsule),

> is ignited by the spit

of a fuse. On being

thus ignited, it burns

rapidly with a brilliant white flame.





FIGURE 1. LEAD PLATE TESTS OF EFFECT OF PRESSURE ON PENTRYL Numbers indicate pressure in pounds per square inch (kg. per sq. cm.).

EXPLOSIVE CHARACTERISTICS OF PENTRYL

SENSITIVENESS TO IMPACT AND FRICTION. Pentryl detonates when struck a sharp blow. Several samples were tested for sensitiveness to impact on the Bureau of Mines' small impact device (19), and for sensitiveness to friction on the type A pendulum friction device (19). The results of imNo detonations occurred during these tests.

The lead plate test (9) is used by manufacturers to ascertain the optimum compression, as indicated by the degree of penetration of the plate, to be employed in the preparation of detonators.

Figure 1 shows the results of lead plate tests. It is ap-

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parent that pentryl produces an excellent lead plate when compressed at 2320 pounds per square inch (163.1 kg. per sq. cm.) Increasing the pressure from 2320 to 5830 pounds per square inch (163.1 to 409.8 kg. per sq. cm.) showed no significant effect. The degree of fineness of the striations on the plates in Figure 1 indicates that pentryl possesses a high degree of brisance.



Pierie acid TNT FIGURE 2. LEAD PLATE TESTS OF PENTRYL COMPARED WITH OTHER NITROAROMATIC EXPLOSIVES

Tests in the No. 2 sand test bomb have shown that increasing pressures from 1160 to 5830 pounds per square inch (81.5 to 409.8 kg. per sq. cm.) have no effect on the sandcrushing strength of pentryl. In this series of tests the average weight of sand crushed was 56.4 grams. This value was 0.6 gram lower than the highest value and 0.7 gram higher than the lowest value.

SENSITIVITY OF PENTRYL TO DETONATION BY INITIATING AGENTS. The sensitivity of an explosive to detonation by a given initiating agent is expressed in terms of the minimum weight of the initiating agent which causes complete detonation. The minimum weight of initiator required varies with the relative initiating power of the different initiators. Complete detonation is indicated when there is no further increase of sand crushed in the Bureau of Mines No. 2 sand test bomb (21, 23, 24) with an increased weight of initiator, except that resulting from the additional weight of initiator. Results of tests are as follows:

| | MINIMUM DETONATING CB | | | | |
|--|-----------------------|-----------|-------|--|--|
| | | Mercury | Lead | | |
| EXPLOSIVE | DDNP | fulminate | aziae | | |
| | Gram | Gram | Gram | | |
| Pontmilh | 0.095 | 0.150 | 0.025 | | |
| Pierie acid | 0.115 | 0.225 | 0.12 | | |
| TNT | 0.163 | 0.240 | 0.10 | | |
| Tetrvl | 0.075 | 0.165 | 0.00 | | |
| Trinitroresorcin | 0.110 | 0.220 | 0.05 | | |
| Trinitrobenzaldehyde | 0.075 | 0.175 | 0.05 | | |
| Tetranitraniline Hevanitrodiphenylamine | 0.075 | 0,165 | 0.05 | | |

Minimum weight of priming charge which causes complete detonation

of base charge. b When no reënforcing capsule is used, there is required 0.035 gram of lead azide to detonate pentryl, whereas more than 0.4 gram of fulminate is required. DDNP will not cause detonation unless confined by a reenforcing capsule (2).

These data show that the sensitiveness of pentryl to detonation by initiating agents is similar to that of tetryl, trinitrobenzaldehyde, tetranitraniline, and hexanitrodiphenylamine, and is greater than that of TNT, pieric acid, or trinitroresorcin.

A comparison of the sand-crushing strength of pentryl with

that of other explosives in the No. 2 sand test bomb is as follows:

| x | WT. OF SAND (THAN (| BRIATISE | |
|--|---|--|--|
| Explosive | By total $charge^{a}$ | By base charge | $\frac{\text{STRENGTH}}{(\text{TNT} = 1)}$ |
| | Grams | Grams | |
| Pentryl Tetryl DDNP Picric acid TNT | $\begin{array}{c} 67.5\\ 65.9\\ 63.9\\ 57.0\\ 55.3 \end{array}$ | $56.0 \\ 54.2 \\ 52.2 \\ 45.3 \\ 43.6 \\ 100 \\$ | $1.29 \\ 1.24 \\ 1.20 \\ 1.04 \\ 1.00 \\ $ |
| Trinitroresorcin Trinitrobenzaldehyde Tetranitraniline Hexanitrodiphenylamine | $52.9 \\ 62.8 \\ 56.3 \\ 60.2$ | $ 41.2 \\ 51.1 \\ 44.6 \\ 49.5 $ | $0.94 \\ 1.17 \\ 1.02 \\ 1.14$ |
| · · · · · · · · · · · · · · · · · · · | 0.20 | moury fulming | te and a base |

cury fulminate and a ^a Using a priming charge of 0.30 gram merc charge of 0.50 gram of the explosive under test.

These data show the sand-crushing strength of pentryl to be 29 per cent greater than that of TNT, 4 per cent greater than that of tetryl, 7 per cent greater than that of DDNP, and 24 per cent greater than that of pieric acid.

EFFECT OF ADDITION OF OXIDANT ON SAND-CRUSHING STRENGTH OF PENTRYL. The decomposition of pentryl to carbon dioxide, nitrogen, and water may be represented as follows:

$$\begin{array}{r} C_{a}H_{6}O_{11}N_{6} \,+\, 4O_{2} \longrightarrow \, 8CO_{2} \,+\, 3N_{2} \,+\, 3H_{2}O \\ 362.1 \,\,+\,\, 128 \end{array}$$

Thus, 362.1 parts of pentryl require for complete combustion 128.0 parts of oxygen, and one part of pentryl requires 128.0/362.1 or 0.353 part of oxygen. The oxygen value per gram of pentryl is then -0.353 gram. The oxygen value per gram of potassium chlorate is +0.392 gram. It is apparent that a mixture containing 52.6 per cent of pentryl and 47.4 per cent of potassium chlorate will be exactly balanced with respect to oxygen content.

To compare the sand-crushing strength of pentryl mixed with potassium chlorate, mixtures of the two in various proportions were prepared and detonators containing these mixtures were tested in the No. 2 sand test bomb (Table I). Two series of tests were run, one with detonators containing a reënforcing capsule and using 0.20 gram of mercury fulminate as a priming charge, and a second using detonators without a reënforcing capsule and containing a priming charge of 0.10 gram of lead azide.

TABLE I. SAND TEST OF PENTRYL-POTASSIUM CHLORATE

| | | Mix | TURES | | |
|--------------|---------|------------------------------------|-----------------------|---|---|
| | | WEIGHT OF | SAND CRUSHI | D FINER THA | N 30 Мезн |
| | | DETONATOS WITH BEË CAPSULE A | PREPARED INFORCING | DETONATOR WITHOUT RE CAPSULE AN ING A PRIM | PREPARED ENFORCING D CONTAIN- ING CHARGE |
| | | CHARGE OF A | 1.40 GRAM OF | AZI | DE |
| | | Sand crushed | Sand crushed | Sand crushed | Sand crushed by |
| m - C | | by total | base | total | base |
| BASE U | HARGE K | chargea | charge | chargeb | charge |
| oz. | 0% | Grams | Grams | Grams | Grams |
| 100 | ,° | 62.5 | 56.0 | 56.8 | 54.6 |
| 100 | 5 | 63.3 | 56.8 | 56.4 | 54.2 |
| őő | 1ě | 63.3 | 56.9 | 58.0 | 55.8 |
| 85 | îš | 63.9 | 57.4 | 57.5 | 65.3 |
| 80 | 20 | 64.4 | 57.9 | 57.7 | 59.5 |
| 75 | 25 | 63.7 | 57.2 | • • | • • |
| źŏ | 30 | 62.5 | 56.0 | • • | •• |
| • • | | | | 4 0 50 gram | of nentryl- |

^a Total charge consisting of a base charge of 0.50 gram of pentryi-ptassium chlorate mixture plus a 0.20-gram priming charge of mercury fulminate. b Total charge consisting of a base charge of 0.50 gram of pentryl-b Total charge consisting of a base charge of 0.50 gram of pentryl-potassium chlorate mixture plus a 0.10-gram priming charge of lead azide.

These data indicate that the addition of an oxidant greatly enhances the strength of pentryl and is therefore to be recommended as of distinct economic advantage.

SURVEILLANCE TESTS. An explosive, if it is successfully to withstand storage under all conditions, must be resistant to moisture, to light, and to moderately high temperatures.

Pentryl has been stored in distilled water at room temperature for 15 weeks with no loss in strength, as indicated by the sand test. No visual signs of decomposition were apparent at the end of this period, nor had any acidity developed.

Samples of pentryl exposed to light in the laboratory for several months have developed only a slight discoloration.

Pentryl has been subjected for 48 hours to the International 75° C. heat test (22) with no loss in weight. When the temperature was elevated to 120°, oxides of nitrogen appeared in 4 hours.

ever, was not sufficiently far-reaching to be manifest in the results of the sand test.

STRENGTH BY THE SMALL TRAUZL BLOCK METHOD. The Trauzl lead block test (19) offers a means of ascertaining the comparative disruptive power of explosives. Results of tests in the small Trauzl block are shown in Table II.

These data show that the disruptive power of pentryl is almost 30 per cent greater than that of TNT or picric acid and

about 16 per cent greater than that of tetryl or DDNP.

Munroe and Howell (18) have determined the lead block (large Trauzl block) expansion of eight explosives (Table III).

It may be deduced from Tables II and III that pentryl possesses nearly the same relative disruptive power as blasting gelatin.

Tolch and Perrott (25) have shown that the relative order of strength of different types of commercial explosives is not changed greatly whether determined in Trauzl blocks or by the ballistic pendulum through which the relative propulsive strength of explosives is ascertained (19). It may safely be assumed, then, that the propulsive strength of pentryl is greater than that of the other nitroaromatic explosives tested by the writer, and, except for blasting gelatin, greater than that of any of the commercial explosives given in Table III.

TABLE III. LEAD BLOCK EXPANSION OF EIGHT EXPLOSIVES (18)

| Explosive | EXPANSION OF BLOCK Cc. | Relative Strength (TNT = 1) |
|-----------------------|---------------------------------|-----------------------------------|
| Blasting gelatin | 400 | 1.32 |
| 60% straight dynamite | 321 | 1.06 |
| Granulated TNT | 302 | 1.00 |
| 50% straight dynamite | 281 | 0.93 |
| 40% straight dynamite | 267 | 0.88 |
| 40% ammonia dynamite | 202 | 0.67 |
| 40% gelatin dynamite | 195 | 0.65 |
| Black blasting powder | 28 | • • |

LEAD PLATE TESTS. Figure 2 shows results of lead plate tests with which to compare pentryl with tetryl, TNT, and picric acid. It is apparent in Figure 2 that, tested under similar conditions, pentryl, tetryl, and pieric acid produce

TOTAL

PERCENTRAGE

TABLE IV. PROPAGATION TESTS

| GRADE | Type of Detonator Classified according to Base Charge | WEIGHT OF CHARGE | VELOCITY OF SHOCK WAVE IN TUBE (Paper) | VELOCITY OF PARTICLES IN TUBE (Paper) | Lengte (Receiver, 40%) Paper tube | i of Gap Straight Dyr Ifon pi | vamite) pe |
|-------|--|---------------------|---|--|---|-------------------------------------|---------------|
| | | Grams | Meters/sec. | Meters/sec. | Meter | Meters | (feet) |
| | Pentryl | 0.50 | | | 4.00 | 16 59 | (54) |
| • | DDNP | 0.50 | | | 3.50 | 10.0 | (01) |
| | Mercury fulminate | 0.50 | | | 1.50 | •• | •• |
| | Mercury fulminate | 1.00 | | | 3.00 | •• | •• |
| 8 | Tetryl primed with mercury fulminate | 1.34 | 1065 | 1200 | 2.504 | 12 5 | (AII) |
| 6 | Tetryl primed with lead azide | 0.66 | 1185 | 1405 | 4 005 | 14.0 | (**) |
| 6 | Picric acid primed with mercury fulminate | 1.25 | 1105 | 1340 | 3 005 | 12 5 | (41) |
| 8 | 80:20 mercury fulminate-KClO | 2.12 | 965 | 1010 | 0.005 | 2.5 | (11) 51 |
| 6 | 80:20 mercury fulminate-KClO ₂ | 1.05 | 965 | •• | 1 004 | 0.0 | (11.0) |
| 7 | Lead styphnate | 1.74 | 970 | •• | 0 504 | 2 50 | (11 EX |
| 8 | Nitromannite-mercury fulminate | 1.16 | 1030 | •• | 0.905 | 12.5 | (41) |
| | | | | | | | (**) |

Burning of receiver occurred in four trials at 18.5 meters, Data by Gawthrop from commercial electric detonators. Established by one trial only.

TABLE V. FRAGMENTATION TESTS

| Explosive | WEIGHT OF CHARGE | WEIGHT OF TEST GRENADE | 2 | PARTICLES | CAUGHT OF | N SIEVE No.: | 10 | TOTAL PARTICLES RECOVERED | WEIGHT OF PARTICLES RECOVERED | OF TOTAL WEIGHT RECOVERED |
|--------------------|---------------------|---------------------------|-----------------|-----------|-----------|--------------|----|---------------------------------|-------------------------------------|---------------------------------|
| | Grams | Grams | | | | | | | Grams | |
| Pentryl Tetryl | $16.5 \\ 16.5$ | 431 405 | $\frac{35}{32}$ | 44 23 | 20 | 21 | 30 | 150 | 427 | 99.0 |
| Pieric acid TNT | 16.5 16.5 | 466 430 | 36 25 | 48 14 | 19 6 | 10 1 | 10 | 123 | 465 | 99,8 99,8 |

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1 2 3 4 5 6 FIGURE 3. LEAD BLOCK COMPRESSION TESTS

> Original block DDP TNT 4. 5. 6. Pierie acid Tetryi Pentryi

To ascertain the effect of long exposure to moderately high temperatures, a quantity of pentryl was stored for 40 days at 75° C. (167° F.), a temperature higher than is ever encountered under normal storage conditions. No decrease in the sand-crushing strength of pentryl was caused by this treatment.

| TABLE II. COMPARISON OF | f Tests in | SMALL | TRAUZL | BLOCK |
|-------------------------|------------|-------|--------|-------|
|-------------------------|------------|-------|--------|-------|

| Explosive ^a | Expansion of Cavity in Block | $\begin{array}{l} \text{Relative} \\ \text{Strength} \\ \text{(TNT = 1)} \end{array}$ |
|--|------------------------------------|---|
| | Ce. | |
| Pentryl | 15.8 | 1.30 |
| Tetryl | 13.8 | 1.13 |
| TNT | 12.2 | 1.00 |
| Pierie acid | 12.4 | 1.02 |
| DDNP | 13,4 | 1.10 |
| Pentryl 90% } KClO ₃ 10% } | 16.0 | 1.31 |
| Pentryl 80% } KClO ₈ 20% } | 16.2 | 1.33 |
| Pentryl 70% KClO ₃ 30% } | 15.6 | 1.28 |

^a Base charge, 0.50-gram; priming charge, 0.30-gram fulminate.

It is apparent that pentryl is quite resistant to pyrolitic decomposition at 75°. However, at the end of 40 days of exposure to this temperature, the original cream color of the crystals had deepened to a somewhat more pronounced yellow. The melting point was lowered from 128° to 127.5° C., indicating a very slight amount of decomposition, which, howsimilar plates of a superior type. The plate produced by TNT is somewhat inferior.

LEAD BLOCK COMPRESSION VALUE OF PENTRYL. The capacity of an explosive to produce compression (as in a lead block) when exploding under atmospheric confinement only is designated as the percussive force of the explosive and is manifest only in the brisant explosives. Kast (13), Hess (12), and Vennin, Burlot, and Lécorché (26) regard the compression value of an explosive as an indication of relative brisance.

The tests were carried out in the manner described by Munroe and Tiffany (19). A No. 7 reënforced mercury fulminate detonator, the base of which rested centrally on the surface of a 50-gram charge of the explosive under test, was used in each shot to effect detonation.

Figure 3 illustrates an unused lead block and blocks showing the different degrees of compression caused by diazodinitrophenol, TNT, picric acid, tetryl, and pentryl. The following table shows the numerical values of the compression caused by these explosives:

| | WEIGHT | Density | HRIGHT O | F BLOCK | Com- pression of | RELA- TIVE PER- CUSSIVE |
|-------------|--------|----------|----------|---------|------------------------|----------------------------------|
| | OF | OF | Before | After | LEAD | Effect |
| EXPLOSIVE | CHARGE | CHARGE | firing | firing | BLOCK | (TNT = 1) |
| | Grams | Gram/cc. | Mm. | Mm | Mm. | |
| Pentryl | 50.0 | 0.75 | 64.0 | 45.5 | 18.5 | 1.25 |
| Tetryl | 50.0 | 0.75 | 64.0 | 47.4 | 16.6 | 1.12 |
| Picric acid | 50.0 | 0.75 | 64.0 | 47.6 | 16.4 | 1,11 |
| TNT | 50.0 | 0.75 | 64.0 | 49.2 | 14.8 | 1.00 |
| DDNP | 50.0 | 0.75 | 64.0 | 53.5 | 10.5^{a} | •• |

* Limited quantity of DDNP permitted only one test.

These data show that the relative percussive effect produced by the detonation of pentryl is about 25 per cent greater than that of TNT, 12 per cent greater than that of tetryl, and 13 per cent greater than that of picric acid.

RATE OF DETONATION. The velocity at which the detonation wave travels through a charge of explosive, or the rate of detonation, is determined on the Mettegang recorder, a description of which has recently been supplied by Munroe and Tiffany (19).¹ Rates were determined over half-meter lengths, the explosive being contained in extra light lead tubing having an inside diameter of 0.5 inch (1.27 cm.) and weighing 12 ounces (340 grams) per foot (30.5 cm.):

| Explosive | DENSITY OF CHARGE | RATE OF DETONATION |
|-------------|-------------------|-----------------------|
| | | Meters/sec. |
| Pentryl | 0.80 | 5000 |
| Tetryl | 0.90 | 5400 |
| Picric acid | 0.98 | 4970 |
| TNT | 0.90 | 4450 |

PROPAGATION TESTS. A modified gap test has been used by the writer (2) and others (3, 8)to ascertain the relative ability of the disturbance sent out by the explosion of a shielded detonator to transfer detonation over an air gap to a receiving charge of explosive.

A detailed description of the method of carrying out this test has been given by the writer in an earlier paper in which DDNP was compared with experimental straight fulminate detonators and commercial electric detonators in the ability of each to transmit detonation across an air gap.

¹ For a further description and results of tests with this device see Mcttegang, Rept. of 5th Congr. Applied Chem., 2, 327 (1903); Glückauf, 40, 1046 (1904); Kast, "Sprengund Zundstoffe," p. 1025, Fredr. Vieweg & Sohn, Braunschweig, 1921; Bichel, "Testing Explosives," Chap. 5; Z. ges. Schiess-Sprengstoffw., 3, 403 (1908); Hall, Bur. Mines, Bull. 15 (1912); Marshall, "Explosives," 2nd ed., Vol. II, p. 476, J. & A. Churchill, London, 1917. In Table IV, results of tests with experimental pentryl detonators and tests with certain commercial electric detonators are compared with previous tests.

It is apparent from these data that a detonator containing a 0.50-gram base charge of pentryl primed with 0.20 gram of mercury fulminate (reënforced) causes the propagation of detonation to a receiving charge of Pittsburgh Testing Station Standard 40 per cent straight dynamite over a distance which is 37 per cent greater than that distance over which the tested commercial No. 8 tetryl, No. 6 picric acid, or No. 8 nitromannite detonators were effective, and nearly 500 per cent greater than that distance over which the No. 8, 80:20 fulminate-chlorate, or the No. 7 lead styphnate detonators were effective.

IGNITION TEMPERATURE. Ignition temperature of pentryl was determined by dropping 0.02-gram charges on a molten metal bath and noting the interval of time between the moment of contact of the explosive with the hot molten mass, and the moment of explosion. Results of these tests are as follows:





FRAGMENTATION TESTS. Figures 4 to 7 show results of fragmentation tests with pentryl, tetryl, pieric acid, and TNT to determine the relative shattering power of these explosives.

For these tests 16.5 grams of the explosive under test were detonated in a malleable-iron hand-grenade body by a No. 6 tetryl electric detonator, the detonator having been substituted for the usual bouchon assembly. The grenade body was closed at top and bottom with pipe taps, the larger (top) bearing a hole just large enough to permit passage of the detonator leg wires.

The grenade was fired in a large volume of sand, and the sand sieved to recover the metal fragments. Results of tests are shown in Table V.

SUMMARY

1. 2,4-Dinitrophenylaminoethanol (melting point, 92° C.) may be prepared in 70 per cent yield by condensation of 2,4dinitrochlorobenzene with β -aminoethanol.

2. By nitration of 2,4-dinitrophenylaminoethanol, symtrinitrophenylnitraminoethyl nitrate (pentryl) may be produced in 90 per cent yield (melting point, 128°C.).

3. Pentryl has a true density of 1.82, an apparent density of 0.74 when compressed in a detonator shell at a pressure of 3400 pounds per square inch (239 kg. per sq. cm.), and a loose density of 0.45.

4. Pentryl is soluble to some extent in most of the common organic solvents, and very soluble in glyceryl trinitrate.

5. The sensitiveness of pentryl to impact is similar to that of tetryl, somewhat greater than picric acid, and much greater than TNT. Sensitiveness to friction is somewhat greater than tetryl, and is much greater than picric acid or TNT.

6. Pentryl produces an excellent lead plate when compressed at 2320 pounds per square inch (163.1 kg. per sq. cm.).

7. The sensitivity of pentryl to detonation by such initiating agents as mercury fulminate, lead azide, or DDNP, is similar to that of tetryl, trinitrobenzaldehyde, tetranitraniline, and hexanitrodiphenylamine, and is greater than that of picric acid, TNT, or trinitroresorcinol.

8. Compression in a detonator shell at pressures up to 5830 pounds per square inch (409.8 kg. per sq. cm.) has no apparent effect on sand-crushing strength of pentryl.

9. The sand-crushing strength of pentryl is 29 per cent greater than that of TNT, 4 per cent greater than that of tetryl, 7 per cent greater than that of DDNP, 24 per cent greater than that of picric acid, 13 per cent greater than that of hexanitrodiphenylamine, and 10 per cent greater than that of trinitrobenzaldehyde.

10. Addition of oxidants such as potassium chlorate greatly increases the sand-crushing strength of pentryl.

11. Pentryl is not affected in its development of energy either by dry or wet storage at normal temperatures over a period of 4 months.

12. Dry storage for 40 days at 75° C. did not affect the sand-crushing strength of pentryl. The sample, during this period, became slightly discolored and the melting point decreased 0.5°.

13. The disruptive power of pentryl as measured in the small Trauzl block is 30 per cent greater than that of TNT, 15 per cent greater than that of tetryl, 18 per cent greater than that of DDNP, and 27 per cent greater than that of picric acid. By the same type of test it is deduced that pentryl is of about the same strength as blasting gelatin.

14. Under similar conditions of test, pentryl, picric acid, and tetryl develop very similar and excellent types of lead plates, which are much superior to the plates produced by TNT.

15. The lead block compression value (regarded as a measure of relative brisance) of pentryl is 11 per cent greater

than that of tetryl, 13 per cent greater than that of picric acid, and 25 per cent greater than that of TNT.

16. The rate of detonation of pentryl (density, 0.80) is 5000 meters per second.

17. A shielded detonator containing a base charge of pentryl, when fired in galvanized pipe, caused transfer of detonation to a receiving charge of 40 per cent straight dynamite over a distance of 54 feet (16.5 meters). Commercial tetryl detonators (No. 8), No. 6 commercial picric acid detonators, and No. 8 nitromannite-mercury fulminate detonators transfer detonation over a distance of 41 feet (12.5 meters). With a No. 8, 80:20 fulminate-chlorate and a No. 7 lead styphnate detonator, detonation is propagated over a distance of only 11.5 feet.

18. Pentryl explodes when heated to 235° C.

19. Fragmentation tests in hand-grenade bodies show that, under the conditions of test, the relative shattering power of pentryl is somewhat greater than that of picric acid, and much greater than that of TNT or tetryl.

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Correction

In the article on "Studies in Distillation. Liquid-Vapor Equilibria in Ethyl Alcohol-Water Mixtures" [IND. ENG. CHEM., 24, 883 (1932)], in Table II the values of y corresponding to the values of x of 0.1000 and 0.1200 are in error and should read: y = 0.4410 and 0.4665, respectively.