## MILITARY MEDIA INC.

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TNT (Trinitrotoluene)

Preparation of Trinitrotoluene (Three Stages).

A mixture of 294 grams of concentrated sulfuric acid (density 1.84) and 147 grams of nitric acid (density 1.42) is added slowly from a dropping funnel to 100 grams of toluene in a tall 600-cc. beaker, while the liquid is stirred vigorously with an electric stirrer and its temperature is maintained at 30 to 40 degrees Celsius by running cold water in the vessel in which the beaker is standing. The addition of acid will require from an hour to an hour and a half. The stirring is then continued for  $\frac{1}{2}$  hour longer without cooling; the mixture is allowed to stand over night in a separatory funnel; the lower layer of spent acid is drawn off; and the crude mononitrotoluene is weighed. One-half of it, corresponding to 50 grams of toluene, is taken for the dinitration.

The mononitrotoluene (MNT) is dissolved in 109 grams of concentrated sulfuric acid (d. 1.84) while the mixture is cooled in running water. The solution in a tall beaker is warmed to 50 degrees and a mixed acid, composed of 54.5 grams each of nitric acid (d. 1.50) and sulfuric acid (d. 1.84), is added slowly drop by drop from a dropping funnel while the mixture is stirred mechanically. The heat generated by the reaction raises the temperature, and the rate of addition of the acid is regulated so that the temperature of the mixture lies always between 90 degrees and 100 degrees. The addition of the acid will require about 1 hour. After the acid has been added, the mixture is stirred for 2 hours longer at 90-100 degrees to complete the nitration. Two layers separate on standing. The upper layer consists largely of dinitrotoluene (DNT), but probobly contains a certain amount of TNT. The trinitration in the laboratory is conveniently carried out without separating the DNT from the spent acid.

While the dinitration mixture is stirred actively at a temperature of about 90 degrees, 145 grams of fuming sulfuric aced (oleum containing 15% free SO3) is added slowly by pouring from a beaker. A mixed acid, composed of 72.5 grams each of nitric acid (d. 1.50) and the 15% oleum, is now added drop by drop with good agitation while the heat of the reaction maintains the temperature at 100-115 degrees. After about three-quarters of the acid has been added, it will be found necessary to apply external heat to maintain the temperature. After all the acid has been added (taking 1 1/2 to 2 hours), the heating and stirring are continued for 2 hours longer at 100-115 degrees. After the material has stood overnight, the upper TNT layer will be found to have solidified to a hard cake, and the lower layer of spent acid to be filled with crystals. The acid is filtered through a Buchner funnel (without filter paper), and the cake is broken up and washed with water on the same filter to remove excess of acid. The spent acid contains considerable amounts of TNT in solution; this is precipitated by pouring the acid into a large volume of water, filtered off, rinsed with water, and added to the main batch. All the of the product is washed three or four times by agitating it vigorously with hot water under which it is melted.

After the last washing, the TNT is granulated by allowing it to cool slowly under hot water while the stirring is continued. The product, filtered off and dried at ordinary room temperature, is equal to a good commercial sample of crude TNT. It may be purified by dissolving in warm alcohol at 60 degrees and allowing to cool slowly, or it may be purified by digesting with 5 times its weight of 5% sodium hydrogen sulfite solution at 90 degrees for half an hour with vigorous stirring, washing with hot water until the washings are colorless, and finally granulating as before. The product of this last treatment is equal to a good commercial sample of purified TNT. Pure ALPHA-TNT, melting point 80.8 degrees, may be procured by re-crystallizing this material once from nitric acid (d. 1.42) and once from alcohol.