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1945-1985
Processes
NRDES and
Pre NRDES

Los Alamos

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memorandum

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DATE: January 6, 1993

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SUBJECT: CONTAMINATION FROM THE RECRYSTALLIZATION OF PETN ON TWO MILE MESA 1945 - 1985

History

Almost all of the EBW detonators made at Los Alamos have used PETN in the explosive component initiated by the exploding bridgewire - called the "initial pressing." In the beginning, attempts were made to use commercially available PETN, but it was soon realized that significant physical impurities - threads, wood chips, metal turnings - were contained in the powder. In addition, the crystals were too coarse for application in EBWs. Thus, recrystallization was carried on to clean up the material and also to adjust the crystal size. Some experiments were made to see what process should be used, but quickly recrystallization from acetone solution by the addition of water was selected. A "recipe" was fixed upon as a way of assuring reasonable control of the crystal size and form. The Fisher Subsieve Sizer (Permeameter) was used for acceptance of the material. The original recipe yielded powder with readings of 3500 cm^2/g. The same powder was used in all detonators until about 1960, when finer (smaller crystals) were desired. A number of different recipes came into use then, with variants such as "reverse recrystallization" and the use of ethyl alcohol along with acetone. Basically, however, the process was one of water addition to the PETN/solvent solution. In the estimates made here, the original recipe is used as the basis for calculation, since the later variants did not change the amount of PETN left in the filtrate very much. Some recipes used temperature variations (such as using ice water), but at the end of the process the solutions were always near room temperature.

Recipe

Early batches were termed "1/2 pound." The original recipe was recorded in a memorandum from Ken Greisen to E. Lofgreen, dated June 18, 1945: 240 g of PETN were dissolved in 1400 ml (1100 g) of acetone. The mixture was then filtered and the crystals washed with more distilled water. The filter cake was at first air dried but later was oven dried at about 70°C. The filtrate was, of course, a saturated solution; washing of the crystals knocked more of the PETN out of the solution but that did not affect the amount going down the drain much. Later a larger, "two pound" batch was adopted but the proportions were the same as in the original recipes. Manual specification MD 9601 gives the proportion for all types of PETN.

The solubility of PETN in acetone and acetone/water was reported by Roberts and Dinegar in the Journal of Physical Chemistry, 62, 1009 (1958). It shows that the PETN/acetone starting solution is not saturated at room temperature but becomes so with only a little water added. At the end of the water addition, the water content is about 72% by weight. Roberts and Dinegar did not carry their study beyond 55.8%, necessitating an extrapolation to arrive at an estimate. The solubility is a function of temperature; for purposes of estimating, the table for 298.2 A (77 F) was used. Our estimate of the amount of PETN in the filtrate from a half pound batch is 0.3 gram; from a two pound batch, 1.2 grams.

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