Verified efficient method of preparing PETN from diluted HNO3

use
32,7 ml of 70% HNO3 (75% excess)
18,9 ml of 96% H2SO4
10,0 g of Pentaerythrite
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or
34,0 ml of 65% HNO3 (66% excess)
24,8 ml of 96% H2SO4
10,0 g of Pentaerythrite
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or
34,8 ml of 58% HNO3 (48% excess)
36,3 ml of 96% H2SO4
10,0 g of Pentaerythrite

Nitration:
1. Cool nitration mixture during mixing of acids, thus minimalize even minimal decomposition of HNO3. Nitration mixture must be cooled before nitration process to temperature of 10°C.
2. While stiring, add pentaerythritee in small portions (1-2g) to nitration mixture, always after previous batch is dissolved. Nitration mixture gradually thicken as PETN forming in solution.
3. Constantly monitor reaction temperature and maintain it in 10-15°C range. Interval of adding pentaerytritol conform to reaction temperature, must not rise over 15°C, leave beaker in cold water.
4. Stir with mixture for next 5 minutes after all pentaerythrite is added and dissolved. Mixture is now thick, but stirring is going well. During nitration process must not be developed any brown fumes of NOx!
5. Now put beaker with mixture into water bath and maintain temperature at 50°C, continuously stir with mixture. During 20 minutes at this temperature, all of possible sulfoesters come into PETN for maximal yield of nitration.
6. While maintaining mixture at higher temperature, mixture must be monitored for developing of NOx fumes. Only light brown colour can be in shrouded beaker. Raised development of NOx pointing to higher temperatures used (even during previous nitration) or insufficient chmemicals purity and further heating may end up in uncontrolled reaction and oxidation of formed PETN. In this case it is better to do not heat at all and end just after nitration (if brown fumes apperas during nitration), the yield will be lower. In case of accidentally runaway reaction during heating, immedialtely pour mixture into cold water, don't try stop reaction by cooling beaker, it will not help.
7. When heating after 20 minutes pass off, pour reaction mixture into cold water and follow standart procedure of filtration, neutralization and purifying of PETN.

Yield was 22,1g of PETN from 10g of pentaerythrite, ~95% of theoretical yield (with 65% HNO3 used).

This procedure is result of my research of most effective method preparing PETN from diluted HNO3. Acid ratios are precisely calculated on data from PETN nitration graph published by T. Urbanski in his book vol. IV. Generaly said, PETN is forming to maximum 30% of water portion in nitration mixture. But, when also H2SO4 is contained in nitration mixture, minimum ammount of water must be keep, or oxidation with small yields occur. With this acid ratios 20% of H2O is minimum. So, this nitration mixture has 20% of water on nitration start and 30% at the end, area for most effective nitration, but again only with this ratios. For other acid ratios must be all recalculated. Excess of HNO3 is used to controll ammount of reaction water. Only ammount of nitration mixture can be extended for lower mixture thickness, but it lower utilization of acids and overall efficiency. But it isn't necessary.