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Technical Paper submitted to USA Department of Defence Explosives Safety Board (DDESB) Seminar at Las Vigas, Nevada on 20-22nd August 1996.

Topic: Nitroglycerine (NG) Stability Test. Its function and issues a dilemma of the producer. Can we circumvent it for better safety ?

1 INTRODUCTION

In Nitro Glycerine production, due to its sensitiveness there is need for its immediate disposal. The only test NG is being subjected to, before its use is Abel Heat Test and it is confirmed that the NG is passing this stability test. Due to various reasons, the test values have become incomprehensible, failing occasionally, irrationally denying all logic, making it a dilemma for the producer. A lot of anxiety and helplessness are created due to delay in consumption of NG, increasing the exposure time. Can we find out a solution?

2 NITRO GLYCERINE STABILITY TEST

Stability test for an explosive is conducted during the manufacture to assess its shelf life to ensure it's safe handling over a specified period during storage of the ammunition. There are a number of such tests available for different explosives but for NG the one and the only one quick, qualitative test conducted is Abel Heat Test. This test consists of heating about 2 ml of sample NG in a test tube at a constant temperature at 180 degree Fahrenheit on a water bath. A Potassium Iodide Starch Paper (Heat Test Paper) partly wetted in a Glycerine Water mixture is hanged in a hook in the test tube. The Nitrous and Nitric vapours liberated from the test sample during heating reacts with the KI solution and liberates Iodine. A brown tinch is formed on the test paper. The time taken for the sample for the tinch is called its heat test value. The more the time a sample takes, the more is its stability and a minimum of Ten minutes is specified for the Nitro Glycerine for the Military use in India. The test though appears to be very simple and forthright, a lot of ambiguities arises over the values, much to the anxiety and discontentment of the producer. The Heat Test values often turns unreliable.

3 FACTORS AFFECTING THE HEAT TEST VALUES OF THE NG

Many factors known to have a direct bearing on the quality of the NG and hence on the Heat Test value. The quality of the raw material Glycerine has its major share and other important items to be mentioned are the Oleum/Sulphuric Acid and the Nitric Acid used for nitration, composition of the Nitrating Acid, Soda solution and water used for process. Apart from raw materials the process conditions, method of manufacture and washing methods etc also affects the heat test values. There are some other elements which are purely dependant on the frame of mind of the individuals who do the test that influence the heat test values. These are phsycological influences.

4. Heat Test values inconsistent, uncontestable and biased.

4.1 Factory X grumbles that they get heat test values just near 10 minutes but they are not worried as not single batch reported failing. i.e. < 10 minutes.

Factory Y got results which are varying over a range from 7.5 minutes to 13 minutes though the average values are similar to Fy X. A good number of batches of NG were reported

as failing requiring disposal differently. Though process capability or process inconsistency can be reason for this kind of values, considering the restrictions imposed on the raw material standards and process parameters such variations are not generally acceptable or attributable to the set of conditions. Fy Z says that they are well above the passing standards.

4.2 For the same NG, when independently tested by more number of individuals, from the different Factory Quality Control staffs or Quality Assurance Establishment Staffs gave different values differing by 3.5 minutes.

4.3 For the same NG, different set of heat test values reported when tested using different batches of heat test papers.

4.4 The same bulk of NG, when sampled at the first instance, gave passing value but subsequent samples reported failing values. On 9-8-95, the results are A = 8' B = 11' A1 (B) = 8.5', A2 (B) = 10'

23-8-95 A = 11' B = 7' B1(A) = 7' C = 8'

4.5 For the same glycerine one factory accepted as the NG produced is said to be 11.5 minutes in Lab Scale Nitration. The same glycerine was rejected as the NG produced is said to be failing with 9 minutes heat test in 1996 January.

4.6 In the continuous process plants where the process conditions remain the near same there is wide variation in the heat test values from the first hour and second hour products. On 2-9-94, A = 13' B = 8.5' (difference 4.5'). This kind of variation is not unfrequent.

4.7 When a bulk quantity of NG is washed there used to be increase in the heat test value upto 4 minutes.

4.1.95 B = 7.5' --> B1 = 11.5'

But on some days there is no improvement given by washing.

4.8 A steady minded man D give an uniform value regularly. A disturbed or slightly excentric man (R) gives results varying from one batch to the next in a day or one day to the next day for the similar conditions.

4.9 A set NC-NG blends made using NG of failing Heat test value reported higher heat test values (20 +) while the blends made using passed NG batches reported lower HT (14+minutes). Propellant made with Low HT NG Blends were submitted to CHT trials and accepted on par with the normal batches.

4.10 NG sample when tested in one place (Laboratory) is reported 6.5' while the same was reported as 12.5' when tested at another lab by the same set of persons.

4.11 There are similar controversial values often the producer is coming across. when the values reported are above the passing standard, there is no concern and it is digested. But when the reported values are hovering around the minimum, the variations will result in some passing and some failing causing irritation and nervousness for the plant operator. The operator thus has reasons to doubt the values and forced to think that the values are un-reliable and does not truly represent quality of NG.

5 ENLARGED NG HANDLING TIME

Nitro Glycerine - the high friction and impact sensitive explosive has to be consumed as and when produced in order to minimise the risk of exposure and potential. Frequent unfamiliar failures lead to holding of NG for long durations occasionally for a whole day that too in large quantities. The risk become enormous.

6 LABORATORY NITRATION OF GLYCERINE

Glycerine the raw material for Nitro Glycerine is first tested for chemical impurities and accepted. Then, as per the traditionally accepted practice, the sample of Glycerine thus passing in chemical test is nitrated in Laboratory using the nitrating acid mixture containing Oleum and Nitric acid. This is to confirm that the Glycerine does not contain any impurity which can cause instability during nitration or separation of the NG from the emulsion. This provides confidence for using the Glycerine in the Plant. A small bulk of Glycerine is nitrated further in the Plant and NG tested for stability and confirmed its passing standard before the entire bulk is taken to use for production. Thus a mandatory laboratory nitration of glycerine provides an opportunity to know the quality of Nitro Glycerine likely to be produced with a set of raw materials to be used in the plant and in the process conditions going to be adopted in the plant. Heat test of laboratory nitration sample gives a fair indication of the quality of NG produced in the plant.

7 LAB TO PLANT ADOPTION

A sample of Glycerine drawn from the specified bulk can be nitrated in the laboratory with mixed acid of identified bulk, maintaining the plant nitration and washing conditions and the Heat Test values can be determined for the Nitro Glycerine. The similar conditions and raw materials can be adopted for the plant and the Nitro Glycerine produced can be considered as given the same Heat Test values as given in the lab scale. Sampling of NG for Heat Test everyday at hourly intervals making this into batches can be withdrawn. NG can be consumed by mixing with the Nitro Cellulose to convert it to the required, stable, safe gelatinised form. The NG-NC paste formed can be tested for the heat test or other stability tests and quality of the paste and hence the NG can be confirmed. This procedure if introduced can save a lot of anxiety to the NG plant personnel who always remain on nerves due to the always uncertain and ambiguous heat test values reported time to time. Also this procedure will bring down the holding time of NG in bulk and thus will bring down the risk of exposure of human to potential hazard. It will also lead to better mental stability of the NG plant personnel and increased attention to the running of the plant and enhance safety further.

8 CONCLUSION

Plant personnel due to their commitments to lot of their personal activity and associated problems, we nowadays unable to work just like machines without much conscious interactions or applications and slowly turning incapable of reacting to abnormal situations. Hence, there is need for keeping the conditions of working as much a routine without putting severe mental strain to the operators. The suggestion offered is in this direction and will be of much use to the mankind.

Concluded