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TEST METHODS AND CRITERIA FOR AMMONIUM NITRATE FERTILIZERS

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***ABSTRACT

This project was aimed at determining whether agricultural grade ammonium nitrate can be reliably and reproducibly excluded from UN Class 1 on the basis of the UN Test Series 1 and 2 protocols. Comparisons were made with industrial grade ammonium nitrate prills. Six fertilizer grade prill samples and three industrial grade samples gave negative results in the Series 1 Gap Test; in the Test Series 1 Internal Ignition Test, all the samples gave negative results with the exception of an agricultural grade prill which yielded a "weak" positive result. All of the samples passed the Test Series 2 Internal Ignition Test. On the basis of this data, all nine samples would be judged to be too insensitive for acceptance in UN Class 1. The test results were reproducible and there was no reason to question the applicability of the UN test protocol to the classification of agricultural grade (or industrial grade) ammonium nitrate prills.

***INTRODUCTION

This is the final phase report on the project "Development of Test Methods and Criteria for Ammonium Nitrate Fertilizer and Materials" covered under Interagency Agreement No. DTRS56-95-X-0037 between the U.S. Department of Transportation and the U.S. Bureau of Mines. The project was aimed at determining whether agricultural grade ammonium nitrate can be reliably and reproducibly excluded from UN Class 1 on the basis of the United Nations Test Series 1 and 2 protocols. Comparisons were made with industrial grade ammonium nitrate prills.

Materials Tested

Six separate samples of fertilizer grade ammonium nitrate (AN) prills and three industrial grade AN prill samples were obtained from various commercial suppliers. They were assigned test control key numbers according to the following list.

Key No. 4568 - Fertilizer grade ammonium nitrate prills obtained locally from an Agway distributor; manufactured by Arcadinia Corp., Memphis, TN.

Key No. 4594 - Industrial grade ammonium nitrate prills obtained from the Donora Plant of Dyno Nobel, Inc.

Key No. 4595 - Industrial grade ammonium nitrate prills obtained from Senex Explosives, Inc., Cuddy, PA; manufactured by Nutrite Inc., Ontario, Canada.

Key No. 4598 - Fertilizer grade ammonium nitrate prills obtained from La Roche Industries, Cherokee, AL.

Key No. 4599 - Fertilizer grade ammonium nitrate prills obtained from La Roche Industries, Atlanta, GA.

Key No. 4600 - Fertilizer grade ammonium nitrate prills obtained from Unocal Corp, Los Angeles, CA.

Key No. 4620 - Fertilizer grade ammonium nitrate prills obtained from Simplot Canada Ltd.

Key No. 4629 - Fertilizer grade ammonium nitrate prills obtained from Terra International (Canada) Inc.

Key No. 4638 - Industrial grade "low density" prills obtained from Senex Explosives, Inc., Cuddy, PA; manufactured by Nutrite Inc., Ontario, Canada.

Physical and Chemical Characteristics

No attempt was made to determine the chemical purity of the ammonium nitrate prills tested under this project. However, simple measurements of average pouring density, diesel oil retention, and particle size distribution were made. The particle size data are presented in Table 1; the density and oil

retention values and average particle size estimates are presented in Table 2. Oil retention values represent the percentage weight of No. 2 Diesel fuel retained by the prills after being saturated with the oil and allowed to drain for 4 hours; the values are taken as estimates of prill porosity on a relative scale.

The most obvious physical difference between the industrial grade prills and the agricultural grade prills was their pouring density. The three industrial grade prills, Key Nos. 4594, 4595, and 4638, had pouring densities ranging from 0.79 to 0.86 g/cm^3 while the six agricultural grade prills had pouring densities ranging from 0.91 to 1.02 g/cm^3. There also appeared to be a significant difference in oil retention between the two grades of prills. Oil retention for the industrial grade prills ranged from 8.7 to 10% while the six agricultural grades had retention values ranging from 4.2 to 7.0%. The average particle size of the industrial grade prills deduced from the particle size distributions ranged from 2.06 to 2.19 mm; these values were on the small end of the range of 2.08 to 2.90 mm calculated for the agricultural grade prills.

Experimental Procedures and Results

The nine samples of ammonium nitrate prills were subjected to the United Nations Test 1(a)(iii), the U.S. version of the Gap Test, and to Test 1(b)(ii) the Internal Ignition Test. If indications of positive response were obtained in the Series 1 Tests, additional tests were conducted using the Test Series 2 versions of the Gap Test and Internal Ignition Tests [Tests 2(a)(iii) and 2(b)(ii)]. Velocity probes were not used in the Gap Tests except when necessary to clarify test results. With this exception, the tests were conducted as prescribed in the UN Manual of Test and Criteria (1). All tests were conducted with the ammonium nitrate prill samples at or near pouring density i.e. untamped. The experimental results are presented in Table 2.

In the Gap Tests at 0 in gap [Test 1(a)(iii)], the six samples of fertilizer grade prills produced completely negative results with tube fragmentation lengths ranging from 8.0 to 12.0 inches and no examples of witness plate puncture. The three samples of industrial grade prills exhibited somewhat

higher shock sensitivity in the Gap Test as judged by tube fragmentation length. The sample designated Key No. 4594 produced tube fragmentation ranging from 13-3/4 in to 14-3/4 in but no instances of witness plate puncture; these results are interpreted as negative (–) according to the test criteria. The industrial prill sample designated Key No. 4595 produced complete tube fragmentation (16 in) in two cases out of three but again no instance of witness plate puncture. To clarify these results, repeat trials were conducted with sample tubes instrumented with velocity probes. The three repeat trials produced tube fragmentation lengths of 14-1/4, 14-1/4, and 16 in, domed witness plates and non-steady reaction velocities. According to the test criteria, these results are interpreted as negative (–). The industrial prill sample designated Key No. 4638 was apparently the most shock sensitive producing complete tube fragmentation (16 in) in all three trials but no instance of witness plate perforation. Velocity probe records showed decaying velocities ranging from 2400 m/s at the booster end to about 1200 m/s at the downstream end. Based on the criteria for the Gap Test, these results would be considered negative (–).

In view of the borderline results with the industrial grade prills in the Test Series 1 Gap Test, the prills were also tested in the Test Series 2 version of the Gap Test which utilizes a 2.0 in plexiglass gap. With prills designated Key Nos. 4594 and 4595, there was no damage to the tube or witness plate. However, with the industrial prills designated Key No. 4638 there was some damage to the downstream end of the tube and some damage, but no perforation of the witness plates. This behavior is not clearly understood but has been observed before with certain propellant sensitized blasting agents. In any case, the results of the Test Series 2 Gap Test with the three industrial grade prills were all negative (–) according to the test criteria.

In the Internal Ignition Tests with a 20 g igniter [UN Test 1(b)(ii)], all of the samples with the exception of the two fertilizer grade prills designated Key Nos. 4599 and 4600 gave completely negative results, i.e. no damage to the heavy wall steel confinement tube or end caps. The tests with prills designated Key No. 4600 resulted in significant pipe damage in one trial out of three. In this case, the pipe was split open but remained in one piece, a

negative (–) result according to the test criteria which requires the pipe to be split into at least two pieces for a positive (+) result.

The 20 g Internal Ignition Tests with the fertilizer grade prills designated Key No. 4599 gave one result in three trials where the pipe was split into two pieces. This would be interpreted as a positive (+) result according to the test criteria.

In view of the near positive response of the prills designated Key No. 4600 and the positive response of the prills designated Key No. 4599 in the Test Series 1 version of the Internal Ignition Test (20 g igniter), additional tests were made using the Test Series 2 version of this test using a 10 g igniter. In these trials, completely negative results were obtained with no damage to the confinement tube or end caps.

Discussion and Conclusions

The six fertilizer grade ammonium nitrate samples and the three industrial grade samples gave negative (–) results with zero gap in the UN Gap Test for Solids and Liquids [Test 1(a)(iii)]. With the exception of one fertilizer grade sample, all of the samples gave negative (–) results in the Internal Ignition Test using a 20 g igniter [Test 1(b)(ii)]. The fertilizer grade prill sample designated Key No. X-4599 gave a marginally positive (+) result with a 20 g igniter but gave completely negative results when retested with a 10 g igniter in Test 2(b)(ii). On the basis of these tests, all nine samples would be judged to be too insensitive for acceptance in UN Class 1 and designated Not Class 1 according to the United Nations Procedure for Provisional Acceptance of a Substance in Class 1.

The three industrial grade AN samples appeared to be slightly more sensitive in the gap test than the fertilizer grade samples in that they produced somewhat more tube damage. This is probably attributable to the lower density and higher porosity of the industrial grade prills. On the other hand, two of the fertilizer grade samples (Key Nos. 4599 and 4600) were slightly more reactive in the Internal Ignition Test than the three industrial grade prill samples. The reason for this is not known but it should be noted that the two

prill types that "reacted" in the Internal Ignition Test had the highest average particle size. This could allow for more ready igniter flame penetration in the internal ignition test leading to greater total energy release. Historically, low density, porous materials have proven to be the most reactive in the Internal Ignition Test.

In conclusion, all nine samples of ammonium nitrate prills examined here could have easily passed the Gap and Internal Ignition Test requirements of UN Test Series 2 and be considered too insensitive for acceptance into UN Class 1. The three industrial grade prills appeared to be slightly more sensitive in the Gap Test than the six fertilizer grade prills; two of the fertilizer grade prills were somewhat more reactive in the Internal Ignition Test. With the exception of the somewhat puzzling results with the prills designated Key No. 4638 in the Series 2 Gap Test, the test results were about as expected on the basis of experience with many other materials and there is no reason to question the applicability of UN Test Series 1 or 2 to the classification of agricultural grade (or industrial grade) ammonium nitrate prills.

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***REFERENCES

1. United Nations, Recommendations on the Transport of Dangerous Goods, Tests and Criteria, Second Edition, Part 1 - Tests and Criteria for the Classification of Explosive Substances and Articles, Test Series 1 and 2, New York, 1990.

Table 1 - Particle Size Distribution of AN (Using 500 g)									
Sieve No.	Key No.								
	X-4568	X-4594	X-4595	X-4598	X-4599	X-4600	X-4620	X-4629	X-4638
5	0	0.1	0	0	0	0.5	2.7	0	0
6	0.2	0.7	0.3	3.1	35.5	19.6	44.4	9.2	0
8	69.1	118.3	119.1	287.6	454.5	449.9	307.5	402.4	87.9
10	259.0	236.2	204.4	96.7	9.4	25.0	123.3	87.8	215.1
14	166.2	125.3	120.1	94.0	0.5	4.7	21.9	0	154.5
18	3.1	15.1	20.0	18.3	0.1	0.3	0.1	0	38.0
35	0.5	1.1	10.2	0	0	0	0	0	2.7
Fall through	0.1	0.5	22.2	0	0	0	0	0	1.5
Total	498.2	497.3	496.3	499.7	500	500	499.9	499.4	499.7

Sieve No. 5 = 4.0 mm; Sieve No. 6 = 3.35 mm; Sieve No. 8 = 2.36 mm; Sieve No. 10 = 2.0 mm; Sieve No. 14 = 1.41 mm; Sieve No. 18 = 1.0 mm; Sieve No. 35 = 0.5 mm

Table 2. - Summary of Test Results with Various Ammonium Nitrate Prills

Key No.	Mfr.	Type	Density g/cc	Oil Retention (%)	Average particle Size, mm	Results (1)			
						0 in Gap	20 g Igniter	2 in Gap	10 g Igniter
X-4568	Arcadinia (Agway)	Fertilizer	1.02	4.8	2.11	11.0; d 9.75; d 10.5; d	No damage No damage No damage		
X-4594	Dyno Nobel	Industial	0.85	8.7	2.19	14-3/4; d 13-3/4; d 13-3/4; d	No damage No damage No damage	No damage No damage No damage	
X-4595	Nutrite	Industrial	0.86	10	2.08	16; d 16; d 14-3/4; d	No damage No damage No damage	No damage No damage No damage	
X-4595	Nutrite	Repeat trials with Velocity Probes				14-1/4; d 14-1/4; d 16; d	All three trials, gave decaying velocities ranging from 2500 m/s at booster end to ~ 1000 m/s at down-stream end		
X-4598	La Roche	Fertilizer	0.99	5.8	2.46	10; d 9-1/2; d 9-1/4; d	No damage No damage No damage		
X-4599	La Roche	Fertilizer	0.91	7.0	2.90	10-3/4; d 9-1/2; d 13-1/2; d	No damage No damage Pipe split open 2 pieces		No damage No damage No damage
X-4600	Unocal	Fertilizer	0.92	4.8	2.85	11-3/4; d 11-1/4; d 10; d	Pipe split open 1 piece No damage No damage		No damage No damage No damage
X-4620	Simplot	Fertilizer	0.97	6.4	2.72	12; d 11; d 11-3/4; d	No damage No damage No damage		
X-4629	Terra	Fertilizer	0.94	4.2	2.76	8; d 8-1/2; d 10-1/4; d	No damage No damage No damage		
X-4638	Nutrite	Industrial	0.79	9	2.06	16; d (2) 16; d 16; d	No damage No damage No damage	6.0; d (3) 8.5; d 6.0; d	

(1) Gap test results are given in terms of tube fragmentation length (in) and witness plate damage (d = dome).
(2) All three trials gave decaying velocities ranging from 2400 m/s at the booster end to about 1200 m/s at the down stream end.
(3) Pipe laid open at down stream end of tube for distances noted; observed velocities about 700 m/s.

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