STABILIZATION OF DYED AND UNDYED BULK RDX

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MASTER

DEVELOPMENT DIVISION

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ABSTRACT

Particle characterization of dyed and undyed RDX stored at elevated temperatures for various durations has been completed. Gas adsorption and microscopy analysis were performed on various samples heat treated at 100, 120 and 140 C for times up to 912 hours. Particle size and surface area remained essentially constant; however, surface decomposition apparently occurred as indicated by particle color changes.

DISCUSSION

Heat treatment of dyed and undyed RDX was conducted to determine particle size changes at elevated temperatures. Heat treatment was conducted on 3-gram samples stored in a closed glass vial. Each sample occupied approximately three-fourths of the total vial volume. Elevated temperatures of 100, 120 and 140 C were employed for times ranging up to 912 hours. Simple linear regression analysis was performed on each sample to determine if a particular treatment did change surface area or particle size. The results for all samples showed no significant change. Table I gives the surface area and microscopy analysis for various heat treatments. Surface area was determined by adsorption isotherms of nitrogen at liquid nitrogen temperature. Microscopy analysis was used to measure particle size in order to closely observe each physical property of the RDX crystals. Particle irregularity and size does not hinder this analysis; thus, a complete distribution analysis was performed to observe minimum size changes. The microscopy technique developed for the Zeiss analyzer measures the dimensions of a large quantity of particles. From dimensional measurements physical parameters of volume, surface area, cross-sectional area, etc., can be calculated when shape is considered(1); repeatability of the technique is $\pm 0.25 \mu\text{m}$ for the measurement of length. Table I includes the arithmetic mean particle length determined from a population of approximately 1000 particles.

The following samples were analyzed and were coded as follows:

- A₁ - RDX 4RCG-110, Undyed
- A₂ - RDX 4RCG-110, Class H Treated with 200 ml 2.8 g/l DuPont G, 0.5 g/l Rhodamine B
- A₃ - RDX 4RCG-110, Class H Treated with 200 ml 2.8 g/l DuPont G
- A₄ - RDX 4RCG-110, Class H, Dyed with 200 ml Rhodamine B 0.5 g/l
- B₅ - RDX 4RCG-110, "Ground," Untreated
- B₆ - RDX 4RCG-110, "Ground," Dyed with 200 ml 2.8 g/l DuPont G 0.5 g/l Rhodamine B
- C₇ - RDX Purified RDX from EB-E-08, Lot 102
- C₈ - RDX Purified RDX from EB-E-08, Lot 102, Dyed with 200 ml 0.5 g/l Rhodamine B, 2.8 g/l DuPont G
- P₅ - RDX 4RCG-110 Ground, Untreated
- P₆ - RDX 4RCG-110 Ground, Dyed with 200 ml 2.8 g/l DuPont G, 0.5 g/l Rhodamine B

(1) A. A. Duncan, *Particle Characterization* (July - September 1971 and October - December 1974).

Samples denoted P_5 and P_6 were used after it was found that lots B_5 and B_6 had in fact not been ground.

Figs. 1 and 2 are photographs of the 120 and 140 C treated samples taken at various times throughout the treatment. Surface decomposition appears to be the most significant change observed. Both undyed and dyed RDX samples changed from either a white or the dyed pink color to dark brown. Samples aged up to 912 hours at 120 C were similar in color to those aged 120 hours.

RDX isothermal weight loss with time at 120 C (see Fig. 3) was conducted on undyed samples A_1 (unground) and P_5 (ground). Approximately 20 grams of RDX was loosely spread in an uncovered 10 cm diameter petri dish. After 1680 hours at 120 C and ambient pressure $\sim 95\%$ of each sample remained. Particle size and/or surface area did not appear to influence weight loss. There was only about 0.5% difference between the ground and unground samples.

COMMENTS AND CONCLUSIONS

Apparent surface decomposition is the only observable effect of elevated temperature on bulk RDX. Sublimation and recrystallization do not appear to have occurred, possibly due to the decomposition of surface molecules instead of mobilization.

Surface area fluctuations, which are at times erratic, are caused by the increase or decrease of surface imperfections and/or volume changes of individual crystals in a critical size range. (Critical size ranges are in this case normally associated with the finer particles.)

Table I. Specific Surface Area* of Bulk RDX at Various Temperatures

Sample Identification	Initial Properties	456 Hours At 100 C	Oven Temperature 120 C (hrs)								Oven Temperature 140 C (hrs)			
			2	6	24	48	72	96	120	912	24	48	96	120
A ₁	4580	4390	4115	4750	4890	4510	3820	3330	3290	3230	2780	2945	3450	2630
A ₂	4670	5125	4580	5230	4755	4650	3885	2595	3540	2560	2865	2755	2840	3375
A ₃	3815	4790	4670	4910	4160	3710	2850	1215	2875	2435	2585	2405	2910	2515
A ₄	2460	3030	2570	2870	4295	2090	4085	2900	2850	3230	2870	2930	2740	2850
B ₅	2965	-	2120	-	-	3078	-	2940	2425	3315	-	-	-	-
B ₆	4130	-	3305	-	-	3780	-	3920	3120	1915	-	-	-	-
C ₇	3080	2210	2750	2860	2655	2915	4175	2145	1715	2945	3090	2835	2035	1325
C ₈	2385	2295	2500	2430	2520	2845	4440	2170	1490	2155	2414	3240	3686	1490
P ₅	8530	9315	-	-	9630	10490	9925	-	8295	-	9325	8015	7900	7620
P ₆	7245	8270	-	-	9775	7830	9210	-	10475	-	7785	7755	7455	8410

Mean Particle Length**

A ₁	64.53	68.06	69.08	68.14	64.53	66.00	66.33
A ₂	61.60	65.14	61.87	67.77	55.35	64.07	68.46
A ₃	73.10	68.27	67.57	66.18	56.93	62.73	70.34
A ₄	68.86	64.08	68.57	65.85	56.40	61.46	71.07
B ₅	74.34	69.22	67.77	66.30			
B ₆	76.71	67.78	69.52	62.58			
C ₇	122.63			120.05	103.04	133.22	167.73
C ₈	122.01			135.56	122.54	127.66	146.04
P ₅	5.75						9.95
P ₆	7.84						10.21

*Surface area in cm²/g

**Mean length in μm

Fig. 1. Stabilization of Dyed and Undyed Bulk RDX
Heat Treatment 120 C, Ambient Pressure

Fig. 2. Stabilization of Dyed and Undyed Bulk RDX
Heat Treatment 140 C, Ambient Pressure

Fig. 3. RDX Isothermal Weight Loss With Time at 120 C.