MASTER

FORMULATION OF CUSTOM SIZED LX-15 GRANULES

Ted W. Stull

DEVELOPMENT DIVISION

APRIL 1980

Process Development
Endeavor No. 101

IT IS UNLIMITED

Mason & Hanger-Silas Mason Co., Inc.
Parkex Plant

OPERATED FOR THE
Department of Energy

UNDER
U. S. GOVERNMENT CONTRACT DE-AC04-76DP-00487

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights.

Printed in the United States of America

Available from

National Technical Information Service

U. S. Department of Commerce

5285 Port Royal Road

Springfield, VA 22161

Price: Printed Copy ~~\$3.50~~ ^{5.00}; Microfiche ~~\$3.00~~ ^{3.50}

FORMULATION OF CUSTOM SIZED LX-15 GRANULES

Ted W. Stull

DEVELOPMENT DIVISION
(April 1980)

MASTER

Process Development
Endeavor No. 101

ABSTRACT

LX-15 is a booster explosive formulation consisting of 95% HNS I and 5% Kel F-800 developed by Lawrence Livermore Laboratory. The purpose of this effort was to develop formulation techniques for the production of custom size granules that are amenable for processing in automatic weighing equipment. This report details processes whereby 0.4 and 1.5 kg size batches are produced, meeting those requirements. Efforts to date have found that granule size is dependent on batch/vessel size, water-to-solvent ratio and the degree of vessel agitation.

INTRODUCTION

LX-15 (95% HNS I and 5% Kel F-800) is a booster explosive formulation developed by H. A. Golopol, et. al., of Lawrence Livermore Laboratory. A report(1) describing their work provides information showing the improved slapper initiation sensitivity of LX-15 over the conventional booster explosive PBX 9407 (94% RDX and 6% Exon 461).

For the fabrication of booster pellets, it is desirable to have material with a granule size that can be processed on automatic weighing equipment. A suitable size is that which passes through a USS No. 16 sieve (1.19 mm opening) and is retained on a USS No. 60 sieve (0.25 mm opening). This work was initiated to develop formulation procedures that would reproducibly provide such material.

(1) H. A. Golopol, D. B. Fields, and G. L. Moody, UCRL-52175, Rev. 1;
"A New Booster Explosive LX-15 (RX-26-AS)."

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

104

DISCUSSION

LX-15 formulation work was originally initiated in a 30-litre reactor to fulfill a specific purchase order requirement. The procedure used was a two-solvent system patterned after that referenced in the LLL report. This procedure provided a larger granule size than desired. During the course of this work, process changes were incorporated in an attempt to reduce the granule size. These changes included the use of a single solvent system (n-butyl acetate), increasing agitator speed and adjusting the water/solvent ratio. After completing the purchase order work, two additional batches of LX-15 were produced using the approximate conditions previously found to yield the best results. Table I details the conditions and results of these two batches. Note that 85 to 90% of the material produced was the desired granule size with a water-to-solvent ratio of approximately 4.5/1.

The effect of process variables on granule size were more thoroughly evaluated. Table II contains results from seven batches. The initial batch (0025-170-01) was made using the approximate conditions of 950 RPM and a water-to-solvent ratio of 4.5/1 that had given the best results in the 30-litre reactor. Only 41.5% of resulting material was within the desired granule size. The next two batches (0028-170-01 and 02) were made using water-to-solvent ratios significantly different than the previous ratio of 4.5/1. Batch 0028-170-02 with a water-to-solvent ratio of 6/1 provided 87.4% of suitably sized material. To determine the effect of agitator speed on granule size, the agitator speed was reduced in steps of 300 with other conditions the same as the initial batch (0025). Two such runs were made, one at 350 RPM and the other at 650 RPM. These two runs showed that as agitator speed is decreased, granule size increases.

The last two batches were made with modifications (accomplished prior to heating the slurry) to the general formulation procedure (formulation procedure detailed in Appendix A of this report). These modifications were:

1. An additional 0.5 litre of solvent was added to the slurry of HNS, water defoamer, lacquer, and wetting agent to improve the homogeneity of the slurry.
2. After slurring the above mixture for approximately 5 minutes, 3 litres of water were added (making a total of 7.5 litres) to maintain the desired water-to-solvent ratio.

Batch No. 0030-170-01 was made with a water-to-solvent ratio of approximately 5/1 and the 72% yield of desired granule size reflected the less than optimum water-to-solvent ratio.

The last batch (0031-170-01) was made to reproduce batch no. 0028-170-02 and to assure that the modifications to the formulation procedure had little effect on granule size. The increased percentage (91.2) of suitably sized granules indicates: (1) the process is reproducible, and (2) the yield may be further increased by adjusting the ratio of solvent volume to the amount of HNS being processed. Due to the high percentage of correct granule size obtained, it was decided at this time not to investigate this ratio effect.

Table I. Formulation of LX-15 (30-Litre Reactor)

<u>Detail</u>	<u>Batch No. 0011-170-01</u>	<u>Batch No. 0014-170-01</u>
H ₂ O Volume (litres)	11.6	18.1
n-Butyl Acetate (litres)	2.5	4.07
H ₂ O/NBA Ratio	4.64/1	4.45/1
Agitator Speed (RPM)	950	950-1000
Yield (g)	890	1385
Yield (%)	98.8	93.8 ^a
Sieve Fractions		
Thru USS 8 (%)	100	100
Thru USS 12 (%)	94	95.9
Thru USS 16 and on USS 60 (%)	85	89.8
Fines (thru USS 60 (g))	81	0 ^a

^aFines not retained or included in yield calculation.

Table II. Formulation of LX-15 (10 Litre Reactor)

Detail	Batch Numbers						
	0028-170-01	0025-170-01	0028-170-02	0029-170-02	0029-170-01	0030-170-01	0031-170-01 ^a
H ₂ O Volume (litres)	2.65	4.55	6.15	4.55	4.55	7.55	7.55
n-Butyl Acetate (litres)	1.02	1.02	1.02	1.02	1.02	1.52	1.28
H ₂ O/NBA Ratio	2.58/1	4.44/1	6/1	4.44/1	4.44/1	4.95/1	5.88/1
Agitator Speed (RPM) ^b	950	950	950	650	350	950	950
Yield (g)	379	383	385.4	382.9	382.6	380.5	384
Yield (%)	97.4	98.5	99.1	98.4	98.4	97.8	98.7
Sieve Fractions							
Thru USS 8 (%)	100	100	100	100	25.9	100	100
Thru USS 12 (%)	96.3	98.7	100	60.9	4.3	99.9	100
Thru USS 16 and on USS 60 (%)	50.6	41.5	87.4	19.0	0.8	72.0	91.2
Fines (thru USS 60 (g))	10.0	6.0	3.6	7.0	6.4	8.5	5.0

^aThese two batches were made with modifications to the basic formulation procedure.

^bAgitator speed in all cases was held as indicated until individual granules were formed. At that time, speed was reduced to approximately 200 RPM and cooling initiated.

CONCLUSIONS

LX-15 batch sizes of 0.4 to 1.5 kg can reproducibly be made with a granule size suitable for processing on automatic weighing equipment. Granule size is influenced by agitator speed, vessel/batch size and the ratio of solvent-to-water in the process.

Although not investigated in this study, the ratio of HNS to total solvent volume may also be a factor that influences granule size.

This study provides guidelines for further scale-up of the process.

ACKNOWLEDGEMENT

The author is indebted to John R. Thurston for his help on this project and suggestions that significantly reduced the time necessary to perform the study.

APPENDIX A

GENERAL FORMULATION PROCEDURE

Formulation Vessel: 10 or 30-Litre Reactor

Defoamer: Hercules (approximately 0.03 wt. % of deionized water used)

Wetting Agent: Elvanol 52-22 (approximately 0.01 wt. % of deionized water used)

Solvent: n-Butyl Acetate

Water/Solvent Ratio: 10-litre reactor (approx. 6/1)
30-litre reactor (approx. 4.5/1)

HNS/Water Ratio: 10-litre reactor (approx. 1/16-20)
30-litre reactor (approx. 1/12-14)

Binder: Kel F 800 (5 wt percent of HNS used)

PROCEDURE:

The volume of deionized water desired (according to batch size) is added to the reactor and an agitation of 200 to 300 RPM established. The defoamer and wetting agent are then added to the reactor and this mixture agitated for approximately 5 minutes before adding the dry HNS. (The defoamer and wetting agents are pre-prepared in water solutions prior to admission to the reactor vessel.)

After the HNS becomes completely wetted, the pre-mixed solution of Kel F in n-butyl acetate is slowly added to the reactor contents. After the mixture appears homogeneous, agitation is continued for approximately 5 minutes then increased to approximately 950 RPM and steam applied to the reactor jacket. (The heating rate depends on batch size, but was approximately 1 to 2 C/minute with these batch sizes.)

As the mixture is heated, the material goes through an initial bead formation, to a stringy agglomerate and finally, to an individual bead. The temperature at which the final bead formation occurs is 85 to 87 C. When the final bead formation occurs, the agitator speed is immediately reduced to approximately 200 and cooling water applied to the reactor jacket. When the temperature of the reactor contents drops below 48 C, the reactor is emptied, the PBX is separated from the liquid and washed several times with cold water. After the wash cycles, the material is placed in an oven and dried at approximately 100 C.

DISTRIBUTION

DOE

Ralph E. Caudle
Assistant Director of Operations
Military Application
Attn: Robert E. Clough
Washington, DC

ALO

H. N. Meyer, Director
Weapons Development Division

R. R. Fredlund, Jr., Director
Classification & Technical
Information

AAO

G. W. Johnson
Operations Branch

Mound Facility

R. T. Braun

SNLL

R. D. Cozine - Org. 8180

LLNL

G. S. Root (15 Copies)
Attn: A. C. Van Dyk

C. T. Brockett, Technical
Information Dept.

LANS

E. H. Eyster - WX-DO
Attn: J. J. Wechsler

J. Aragon - WX-3

Report Library - ISD-4

SNLA

B. E. Arthur - Org. 4370

J. C. Crawford - Org. 2500/
D. H. Anderson - Org. 2510

C. B. McCampbell - Org. 2300

Central Technical Files - Org. 3141

TIC

Technical Information
Center (27 Copies)
Oak Ridge, TN

PX

Division Manager, Mfg. Engineering
Division Manager, Quality
Division Manager, Development
Division Manager, Manufacturing
Division Manager, Safety & Fire
Protection
Technical Library

Circulation Copy:

- (1) Plant Manager
- (2) M&H-SM Co., Inc., Lexington, KY