

## PROCESSING OF PBXN-111 ON A TWIN SCREW EXTRUDER

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### ABSTRACT

Techniques were developed to process PBXN-111 on a 40-mm W&P co-rotating twin screw extruder (TSE). This included developing a feed stream scenario to introduce the nine ingredients of PBXN-111 by way of four feed streams, conducting feeder/pump testing to determine the minimum and maximum throughput, and determining the screw and barrel configuration. During the final processing trial, a sufficient amount of PBXN-111 was produced to load two 20-pound generic test charges. Test charges of PBXN-111 processed using traditional batch technology with the same lot set of ingredients were also manufactured. All generic charges were tested in the same underwater quarry test series. Results indicate that TSE processed material performs comparably to that of batch processed material. In addition, tests were performed to determine the mechanical properties and chemical composition of the explosive loaded into the test charges. Both the TSE processed and batch processed PBXN-111 met specification. However, the TSE processed material was lower in density and had a lower stress at maximum strain. The TSE processed material contained less aluminum, which would account for the lower density. Also, it was cast at a higher vacuum level than the batch processed material (~60 mm Hg versus ~15 mm Hg) which may have caused micro-porosity in the samples.

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## INTRODUCTION

This report describes the continuous processing techniques that were developed to produce PBXN-111 charges for underwater testing, the chemical analysis and mechanical property test results, and the results of the quarry tests. The purpose of this study was to determine how Twin-screw extruder (TSE) manufactured PBXN-111 performed in underwater testing compared to traditional batch manufactured PBXN-111. Therefore, TSE processed generic charges were manufactured with the same lot set of ingredients that were used to produce traditional batch-processed charges manufactured at Yorktown, NSWC and tested "side-by-side". Chemical analysis of the resultant explosive and quarry testing of the generic units show that PBXN-111 processed using a 40-mm W&P co-rotating TSE performs comparably to test units manufactured by the traditional batch process.

There are several steps needed to develop a process to manufacture a formulation on a TSE. These include developing a feed stream scenario, conducting feeder/pump testing, and determining the screw and barrel configuration. All TSE facilities contain a finite number of solid and liquid feeders. Therefore, depending on the formulation to be processed, pre-blends may have to be prepared. In determining the feed stream scenario, attention must be paid not only to the number of feeders/pumps in the facility, but also to the accuracy of these units. Cast/cured energetic materials, such as PBXN-111, typically contain small amounts of various ingredients. In order to feed them accurately, they must be blended with other ingredients. Once the feed stream scenario has been developed, the accuracy of the feeders must be determined. This may determine the minimum and/or maximum throughput that can be attained. In conjunction with the feed stream scenario development, the barrel configuration must be determined. Dependant on where the individual ingredients (or blends) are introduced into the extruder, the screw must be configured to perform a particular task. At different points in the TSE, material must be conveyed and/or mixed. The intensity of mixing must be considered – this determines the type and number of screw elements used and their placement. Also, if vacuum is to be applied, a dynamic seal must be formed. The elements needed to form this seal are dependant on the properties of the formulation.

## RESULTS AND DISCUSSION

### FEEDER STUDIES

The explosive, PBXN-111, contains nine ingredients. The ingredients and weight percentages are based on the requirements of MIL-E-82902 (OS) [1]. The explosive was formulated with an NCO/OH ratio of 1.0, based on the manufacturers' certifications of these ingredients, and a polymer to plasticizer ratio (R45HT to IDP) of 1.0. The feed stream scenario (Table I) was based on work conducted by William Newton and Robert Thompson [2, 3, and 4]. The majority of the binder (minus the curative and a small portion of the plasticizer) and all of the aluminum were blended in a Cowles dissolver. The remainder of the plasticizer and the curative was blended by hand. The two classes of RDX were blended in a V-Cone blender. Details of the material preparation and feeder set-up are described by Prickett, et al [5].

Feed Stream	Weight Percent
RDX Blend	20.00%
AP	43.00%
Aluminum Slurry	35.73%
IDP/IPDI	1.27%

**Table I.** The feed stream scenario developed for this project.

The aluminum/binder slurry was fed using the Seepex Progressive Cavity Pump, Model MD 003-24; the 200 $\mu$  AP was fed using the T-37 K-Tron Loss-in-Weight (LIW) feeder, the blended RDX was fed using the C&M Brabender LIW feeder; the IDP/IPDI blend was fed using the 0.167 cc/rev Zenith B-series gear pump.

Initially, minimum and maximum rates were tested at total throughputs of 30 and 50 pounds per hour. However, the C&M LIW feeder could not feed the RDX at a rate of 6 pounds per hour (30 lb/hr total throughput). The minimum amount of blended RDX that could be fed, accurately, using this feeder was 7 lb/hr. Therefore, testing of the remaining feed streams was at total throughputs of 35, 40, and 50 pounds per hour. The feeder test results are shown in Tables II – V. Unlike other feed systems used during this work, the output of the Seepex pump, used to introduce the aluminum slurry does not include a mass control feed back loop. The pump is controlled by setting its frequency; therefore, during feeder testing a calibration curve was produced. The pump frequency was varied and the flow rate measured. The results are listed in Table V. In addition, due to safety concerns, a pressure transducer was connected to the feed line immediately prior to introducing the feed stream to the extruder.

Set Point (lb/hr)	Actual (lb/hr)	$\pm$ Target wt%	15s Coefficient of Variation	30s Coefficient of Variation	60s Coefficient of Variation	Amount of AP in Hopper (lb)
12.90	13.23	2.56%	0.04	0.02	0.00	31
17.20	17.34	0.81%	0.04	0.02	0.01	46
21.50	21.68	0.84%	0.03	0.02	0.01	35

**Table II.** Test results of 200 $\mu$  AP fed in the T-37 K-Tron LIW feeder.

Set Point (lb/hr)	Actual (lb/hr)	$\pm$ Target wt%	15s Coefficient of Variation	30s Coefficient of Variation	60s Coefficient of Variation	Amount in Hopper (lb)
7.00	6.98	-0.29%	0.05	0.04	0.03	22
8.00	8.18	2.25%	0.06	0.05	0.02	40
10.00	10.04	0.40%	0.05	0.04	0.03	30

**Table III.** Test results of the RDX blend fed in the C&M Brabender LIW feeder.

Set Point (lb/hr)	Actual (lb/hr)	$\pm$ Target wt%	15s Coefficient of Variation	30s Coefficient of Variation	60s Coefficient of Variation
0.44	0.41	-6.82%	0.09	0.07	0.06
0.64	0.62	-3.13%	0.06	0.05	0.03

**Table IV.** Test results of the IDP/IPDI blend fed with the 0.167 cc/rev Zenith B-series gear.

Pump Frequency (Hz)	Transducer Pressure (psi)	Flow Rate (lb/hr)	15s Coefficient of Variation	30s Coefficient of Variation	60s Coefficient of Variation
35.00	53	10.80	0.02	0.01	0.01
45.00	65	13.70	0.02	0.01	0.01
60.00	75	17.97	0.04	0.02	0.01

**Table V.** Test results of the aluminum/binder slurry with the Seepex pump.

### TSE PROCESSING TRIAL

Three extruder trials were conducted during the execution of this project. The barrel temperature, total throughput, screw speed, and screw configuration were varied during these trials. Only the final processing trial (IHR1106G-N111D-0288) will be discussed here.

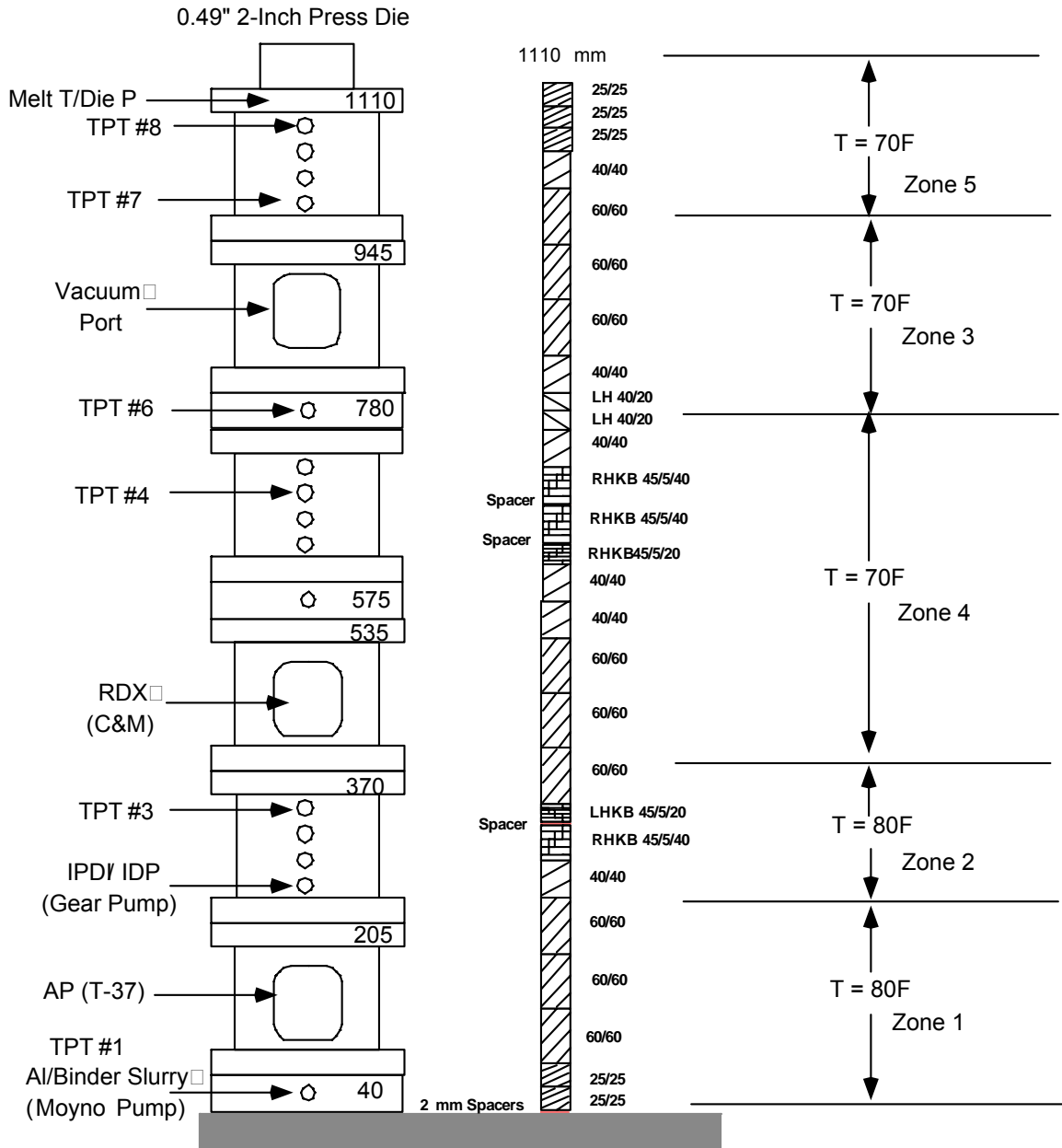
The screw and barrel configuration for the final processing trial (IHR1106G-N111D-0288) is shown in Figure 1. Temperature-pressure transducer (TPT) #1 was used to monitor the pressure of the aluminum/binder slurry feed line. As shown in Figure 1, TPT #3 monitored the pressure/temperature responses at the first mixing section (Al/binder slurry, AP, and IDP/IPDI blend). TPT #4 measured the temperature and pressure at the second mixing section, after the RDX addition. TPT #6 monitored the pressure at the "dynamic seal" area. TPT #7 was located in the portion of the extruder at which vacuum was pulled. It was "zeroed" to a value of about 40 psig; in this way the amount of vacuum being pulled could be monitored and recorded. TPT #8 was placed at the end of the extruder in an attempt to determine the amount of "back fill" along the screw elements at the end of the extruder. A transducer was also placed in the die area. The 0.491" diameter die with a length to diameter ratio of 1.5 was used.

A rotating index table (RIT) was set up to collect material exiting the extruder. Five stock pots were placed on the RIT: one collected "start-up" material, three were used to collect PBXN-111 explosive at the correct formulation and 50 mmHg of applied vacuum, while the last one was used to collect material exiting the extruder during "ramp down".

Material at full formulation was processed through the 40-mm TSE at a vacuum level of 50 mmHg, a die temperature of 70 °F, a total throughput of 50 lb/hr, and a screw speed of 40 rpm for two hours. The explosive was "extruded" into three stock pots. This material was then transferred to another facility where it was vacuum cast (at 60 mmHg) into two 20 pound (nominal weight) charges and four "sample pans" using conventional loading techniques.

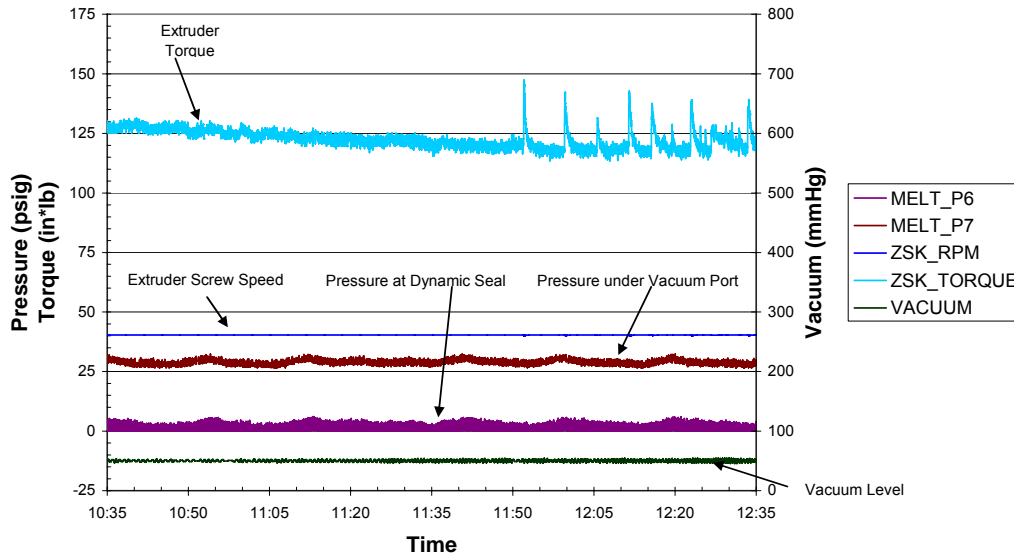
Figure 2 shows the extruder torque, pressure at the vacuum port and dynamic seal, extruder screw speed and vacuum level recorded during steady state. The traces indicate that the process was stable except for small torque spikes after 1-1/2 hours of processing. The cause of these spikes is unknown.

PBXN-111 Barrel Configuration  
Screw Configuration # 77



**Figure 1.** The screw and barrel configuration used during PBXN-111 Processing Trial IHR1106G-N111-0288.

**IHR1106G-N111D-0288**  
**Pressures & Processes**  
**at Steady State**



**Figure 2.** Pressure and process responses during steady state of Processing Trial IHR1106G-N111D-0288.

CHEMICAL AND MECHANICAL PROPERTY TESTING.

Chemical analysis and mechanical property testing were conducted on TSE processed PBXN-111 by T311 personnel at IHDIV NSWC, Yorktown Detachment. This testing was also conducted on standard batch processed explosive produced at IHDIV NSWC, Yorktown Detachment (Lot IHY06G30GBP-354). The results are shown in Table VI. The TSE processed PBXN-111 met the specification requirements. However, the density and stress were considerably lower than that of the batch processed material. Apart from the processing method, the other main difference in how the samples were prepared is the vacuum level during casting. Typically, explosive is cast at a vacuum level 5 – 10 mmHg higher than the vacuum level that was applied during mixing. In general, batch processed material is mixed at 5 – 10 mmHg and cast at 10 – 15 mmHg. The TSE processed material was subjected to 50 mmHg. Thus, there may have been some microporosity in the samples causing a decrease in the density and stress. Also, there was some settling of aluminum in the slurry holding tank. Clearly, this would result in a lower density sample. As can be seen in TableVI, the amount of aluminum detected in the TSE processed material is only 23.9% - the nominal amount is 25%, and the batch processed material contained 24.9%. However, the chemical analysis of the batch processed material was conducted on uncured material while the TSE processed material was cured prior to testing. To determine if this affected the results, the analysis was performed on both cured and uncured PBXN-111 from the same mix by IHDIV NSWC, Yorktown Detachment personnel. These results are shown in Table VII. There is about a 0.5% increase in the measured amount of AP and aluminum in the uncured sample compared to the cured sample.

Lot	IHY06G30GB P-354 <sup>a</sup>	IHR1106G- N111D-0288 <sup>b</sup>	Spec Min	Spec Max	Test Method MIL-E- 82902(OS)
Density, g/ml	1.78	1.74	1.74	1.82	4.6.1
Stress	71.9	47.6	40		4.6.2
Strain	12.5	12.3	8		4.6.2
Hardness	40	35	20		4.6.3
Vacuum Stability, ml/g	0.14	0.12		0.5	4.6.4
AP, wt%	43.1	42.4	40.0	44.0	4.6.5
Al, wt%	24.9	23.9	23.0	26.0	4.6.5
RDX, wt%	20.0	20.4	18.0	21.0	4.6.5
AP+Al+RDX, wt%	88.0	86.7	86.0	89.0	4.6.5

a) Chemical analysis conducted on an uncured sample.

b) Chemical analysis conducted on a cured sample.

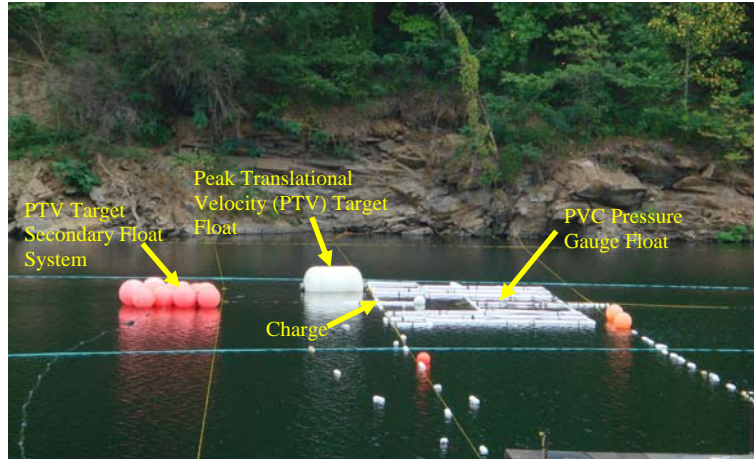
**Table VI.** The results of chemical analysis and mechanical property testing conducted at NSWC/IHDIV, Yorktown Detachment on batch processed PBXN-111 (IHY06G30GBP-354) and TSE processed PBXN-111 (IHR1106G-N111D-0288) using the same lot set of ingredients.

	Analysis on Uncured PBXN-111	Analysis on Cured PBXN- 111	Nominal
AP, wt%	42.2	41.6	43.0
Al, wt%	24.3	23.8	25.0
RDX, wt%	20.5	20.7	20.0
AP+Al+RDX, wt%	87.0	86.1	88.0

**Table VII.** Chemical analysis of the same mix of uncured and cured PBXN-111 processed and tested at NSWC/IHDIV, Yorktown Detachment.

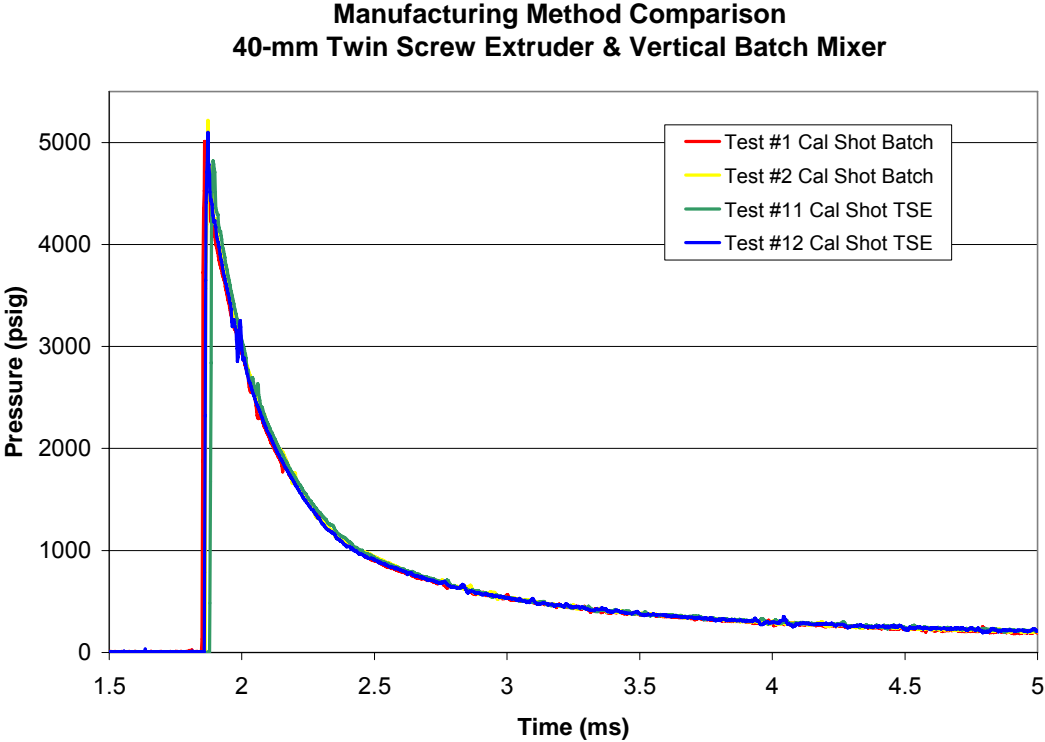
#### QUARRY TESTING

The quarry test layout, showing the free field gauges and peak translational velocity (PTV) target, for all tests conducted during this series is shown in Figure 3. Tourmaline piezoelectric pressure gauges were used to evaluate the performance of each explosive charge configuration. The pressure gauges are located along seven radial lines at ranges of 10 feet, 15 feet and 20 feet from the center of the charge. Each of these gauge positions had two gauges to provide redundancy in data collection. For these particular charges the 90-degree radial had an additional two gauges located at a 5-foot range. These gauges allow the calculation of similitude parameters for the charges. In order for the pressure gauges to be positioned accurately, anchor points were mounted into the quarry walls near the water level. The pressure gauges were supported (floated) using 4" PVC pipes. One-half inch diameter poly lines that are pulled tight with turnbuckles or a Come Along are used to secure the PVC floats into the correct positions. A full description of the test series can be found in the Test Plan [6].



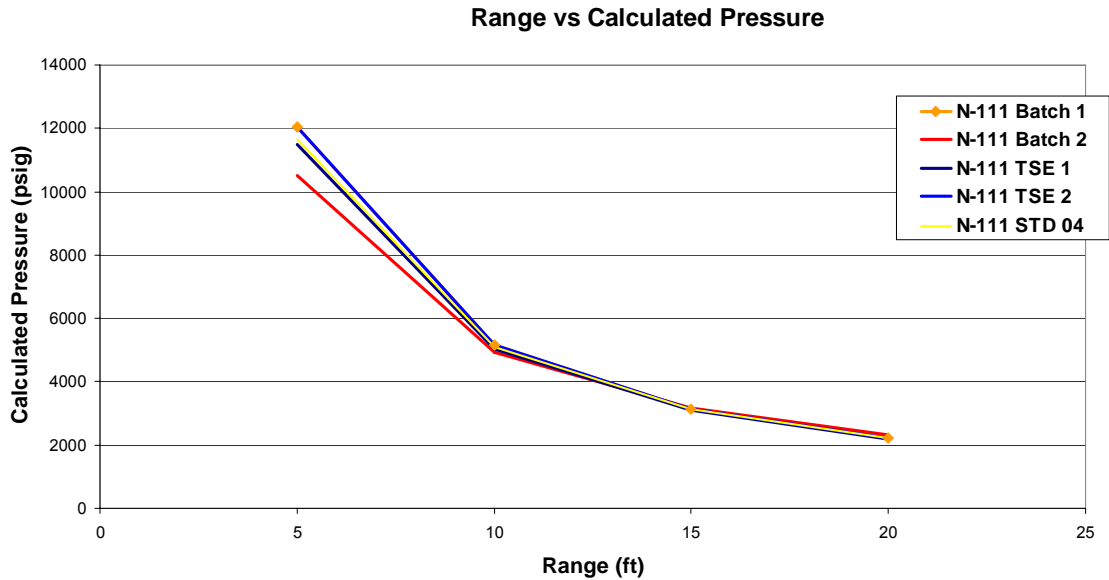
**Figure 3.** The quarry test layout, showing the free field gauges and peak translational velocity (PTV) target, for all tests conducted during this session.

When reviewing the pressure data obtained from the tests [7], traces for the batch and the TSE manufactured charges are nearly identical (Figure 4). The problem in comparing these traces directly is that each charge has a small variation in weight and possibly a small variation in gauge location. In order to account for these variations we use similitude equations to compare the output of each charge. Details of this data reduction technique are described by Swisdak [8] and Lawrence, et al [9].



**Figure 4.** Pressure trace for twin screw extruder (TSE) and batch mixer calibration shots @ 10 feet.

Similitude curves for the TSE and batch processed PBXN-111 charges calculated with 20 pounds net explosive weight are shown in Figure 5. There is a small spread in the pressure data at the shorter ranges because the PBXN-111 Batch 2 test did not have a functioning gauge at the five foot range. Thus this trace is shifted slightly due to the missing piece of data. The calculated pressure at the 5-ft range for PBXN-111 Batch 2 is lower, as expected.



**Figure 5.** Similitude curves for twin screw extruder (TSE) and batch mixer calibration shots calculated with 20 lbs net explosive weight.

Table VIII shows the difference between the vertical batch mixer and the 40-mm TSE processed explosives at 20 feet was approximately 2 percent, which is nearly the same as the overall deviation between all the shots. This result indicates that the manufacturing method does not significantly impact the explosive output of the charges.

Weight (lb)	Range (ft)	Description	Press (psig)	Relative Press	Relative Energy 1Theta	Relative Energy 5Theta	Relative Impulse 1Theta	Relative Impulse 5Theta
20	20	N-111 Batch Averaged	2259	1.019	0.996	1.019	1.000	0.986
20	20	N-111 TSE Averaged	2209	0.997	1.008	1.001	1.038	1.006
20	20	N-111 STD 04	2216	1.000	1.000	1.000	1.000	1.000

**Table VII.** The results of the quarry testing relative to a PBXN-111 standard. Theta, the decay constant, is the time at which the pressure has decreased to 1/e (or 36.8%) of its maximum value [8].

## SUMMARY AND CONCLUSIONS

A process was developed to produce PBXN-111 on a 40-mm W&P co-rotating TSE. A feed stream scenario was developed to feed the nine ingredients in four feed streams. These feed streams were accurately fed to the TSE to manufacture PBXN-111. Material was processed through the 40-mm TSE at a vacuum level of 50 mmHg, a die temperature of 70 °F, a total throughput of 50 lb/hr, and a screw speed of 40 rpm.

The TSE processed PBXN-111 is of comparable quality to the traditional batch processed material. Underwater test results indicate that the maximum pressure, energy, and impulse are the same, whether the material is batch processed or manufactured using a twin-screw extruder. Chemical analysis indicates that the density and stress of the TSE processed material is significantly lower than the batch processed material. This is probably due to processing and casting at a higher vacuum level (introducing microporosity in the material) and settling of aluminum in the slurry holding tank prior to its introduction into the TSE.

#### ACKNOWLEDGMENTS

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