Red Phosphorus Butyl Rubber (RPBR) Process Development

for the MK 34 Mod 2 Smoke Warhead for the 5" rocket

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NSWC Indian Head Technology Development

Established Tasks

1) Develop a continuous RPBR production process on a 20 MM Twin Screw Extruder (TSE)

2) Produce a minimum of 125 lbs of RPBR pellets as a representative Qualification Lot

Experts Consulted

- Chilworth Technology, Inc.
  - Fire and explosion hazard assessment
  - Red phosphorus test data
- Applied Ordnance Technology, Inc.
  - Operating and support hazard analysis
- Dr. Bill Hubble, NSWC Crane
  - Red phosphorus handling expertise

20 MM Twin Screw Extruder

Existing Manufacturer

- Department of the Army, Pine Bluff Arsenal
  - Pine Bluff, Arkansas
- Batch process
- Problems with their methylene chloride emissions
- Developed a solvent recovery system

Product Specification

- Department of the Navy, drawing No. 318AS176
  - 93.75% ± 1.0 % red phosphorus
  - 4.50% ± 1.50% butyl rubber
  - 1.25% ± 0.25% silica
  - 0.50% maximum total volatiles
  - 50% must be between 0.19 - 0.38 of an inch in length
  - Extrusion must be through a 0.249 - .255 inch die

Indian Head Process Challenges

1) Protecting operator from phosphine gas (PH_3)
   - Poisonous
   - Highly flammable
2) **Red phosphorus dust electrostatic discharge (ESD) sensitivity**
   - Minimum ignition energy (MIE) < 3mJ
   - Eliminating the possibility of a fire
3) Isolating the 20 MM TSE operating bay
   - Minimize red phosphorus contamination
4) Red phosphorus preparation
   - Screening and feeding

Indian Head Process Challenges Cont:

5) Binder (butyl rubber gel) preparation
   - Replacing the traditional process solvent
6) Process development on the 20 MM TSE
   - Extruder configuration
7) Product collection
   - Face cutter design and fabrication
8) Finishing stages
   - Drying and silica coating the pellets
9) Red phosphorus waste storage and disposal
   - Locating a disposer

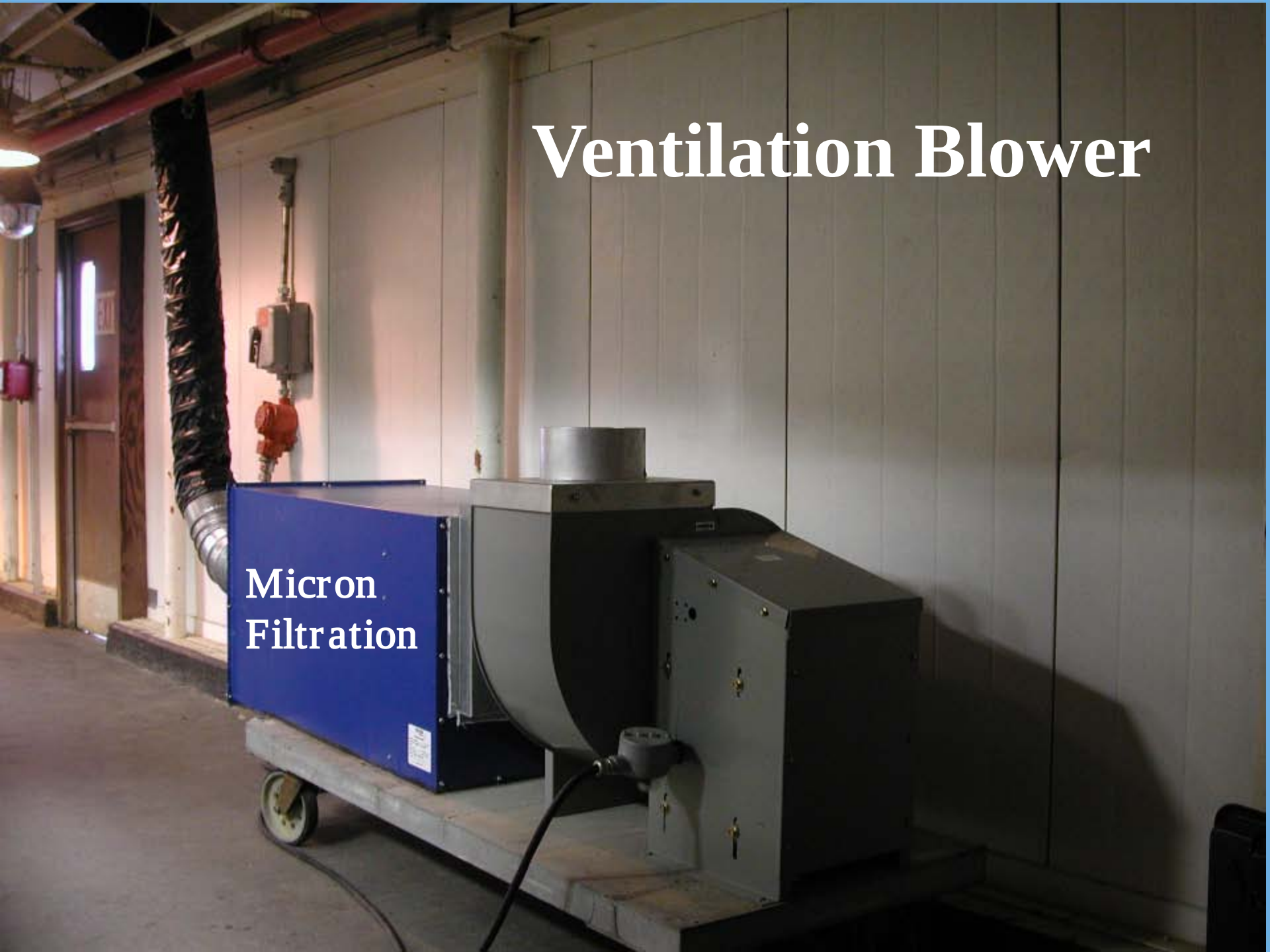
Protection from Phosphine Gas

- Phosphine detectors installed in the bay
  - Manufactured by Drager Safety Inc.
  - Range 0-20 ppm
- Operators required to wear supplied air respirators
  - Required during all operations in the bay
- Operators required to wear flame suits
  - Required during all operations in the bay

Eliminating the Dust Hazard

- N_2 Purging in all red phosphorus handling operations
  - Chilworth recommendation
- Keep all red phosphorus containers closed
- Operators required to wear flame suits
- Isolate the screening operations
- Separate ventilation system
  - Also helped to minimize building contamination

Ventilation Blower



N_2
Red Phosphorus
Feeder

Screening Operation

- Screening solid feedstock is a standard practice
- Sieve red phosphorus through 12-inch diameter x 14 ASTM mesh sieve
  - Particle size range ≈ 20 - 60 micron
- Repackage the screened powder in 10 lb bags
- N_2 purge of 2.0 LPM during screening

Screening Operation

Binder Solvent Determination

	Solvent to Butyl Rubber Ratio Solvent			Hazard Air Pollutant
	7.5:1	4:1	3:1	
*Methylene Chloride	Solid Gel, 50% excess solvent	Solid gel	Solid gel	Yes
**Pentane	Not Tested	Fluid gel w/ excess solvent	Thick gel, Pump-able	No
**Hexane	Not Tested	Fluid gel	Thick gel, Pump-able	Yes
****Heptane**	**Not Tested**	**Fluid gel**	**Heavy gel, Pump-able**	**No**
**Octane	Not Tested	Heavy gel, excess solvent	Heavy gel, Pump-able	No
Tetra-Hydro-Furan (THF)	Not Tested	Unsuitable solvent	Unsuitable solvent	N/A

Notes:

*Methylene Chloride was the specified solvent; it was used as the baseline solvent
*7.5:1 was the specified solvent to butyl rubber ratio
**The manufacturer of the butyl rubber suggested the following solvents.

Binder Formation

- Butyl rubber preparation
  - Cut into 1-inch cubes
- Heptane and butyl rubber added to pressure pot w/ stir
  - Final ratio 2.5:1
- Mixes made in batches
  - 20lbs of binder per batch
  - 36 hour stir time @ 45 RPM

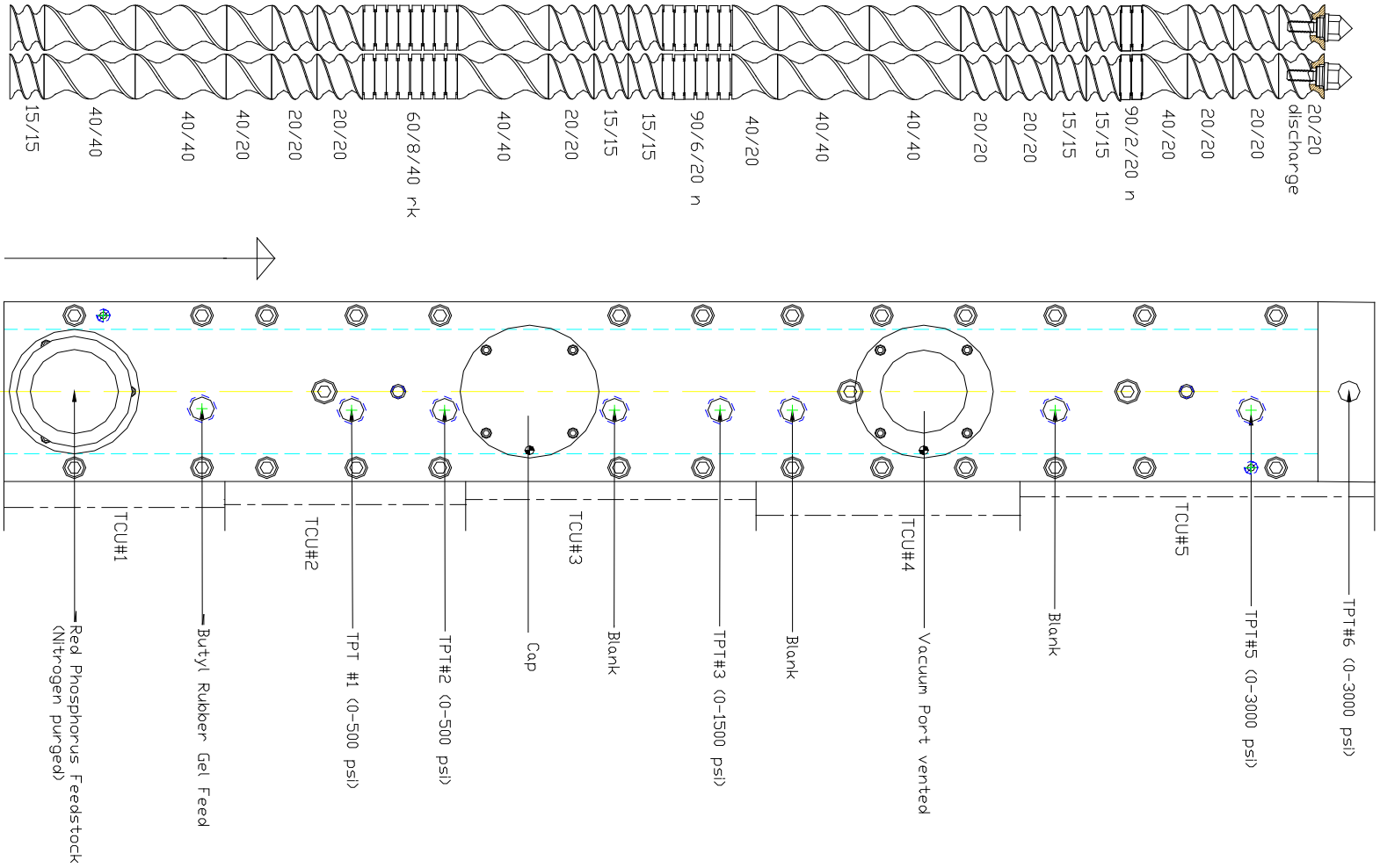
Production Scenario

- Maximize extruder throughput at 8-10 lbs/hr
  - Maintain solids loading in the range of 80-85%
- Complete the Qual. Lot in a minimal number of runs
  - Achieve 60 lbs of wet RPBR per run
- Process will be semi-attended
  - Solid feeder was refilled every hour

Screw/Barrel Configuration

- Screw designed with 3 sets of kneading blocks
  - 1 set of right handed
  - 2 set of neutrals
- Temperature/Pressure transducers placed over the kneading block sections.
- Ambient processing temperature
- Vacuum port was used for venting

Screw Build and Barrel Configuration



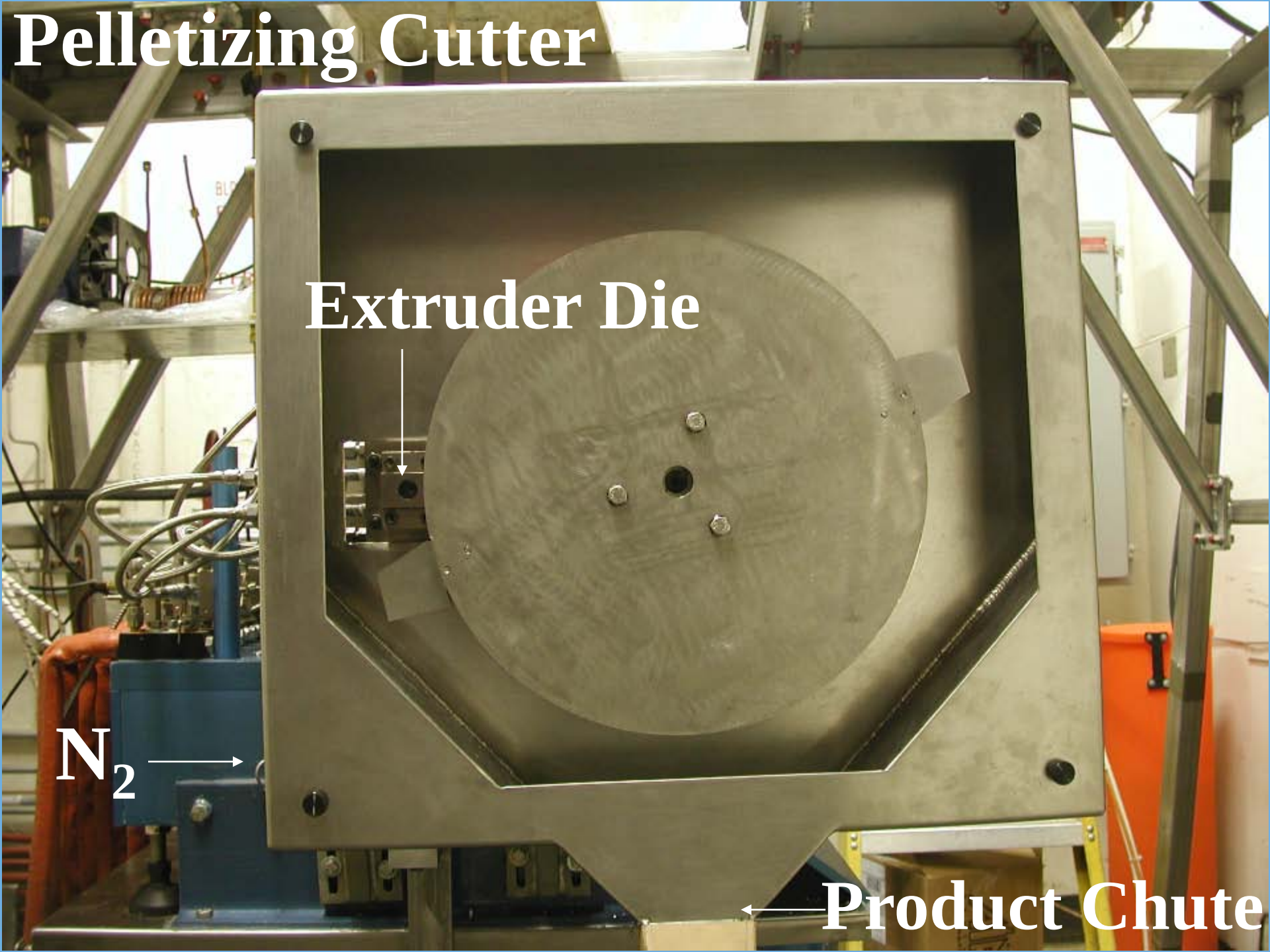
Process Responses

- Extrusion rate maximized at 9.0 lbs/hr
  - Red phosphorus fed at 7.54 lbs/hr
  - Binder fed at 1.5 lbs/hr
  - Extruder speed set at 85 RPM
- No significant pressures at the kneading blocks
- Maximum constant die pressure of 400psi
- Pellets appeared to be soft, but well mixed
- Die and end barrel sections lowered below ambient temperature to firm pellets

Product Collection

- Pellets were cut by face cutter
  - Cutter was tuned to cut 0.25-inch pellets
- Fines reduced to 10% of total extrudate
  - 25 thousandths clearance between die face and blade
- Cut and collected pellets on trays while extruding
- Cutter housing purged with N_2 at 2.0 LPM

Pelletizing Cutter

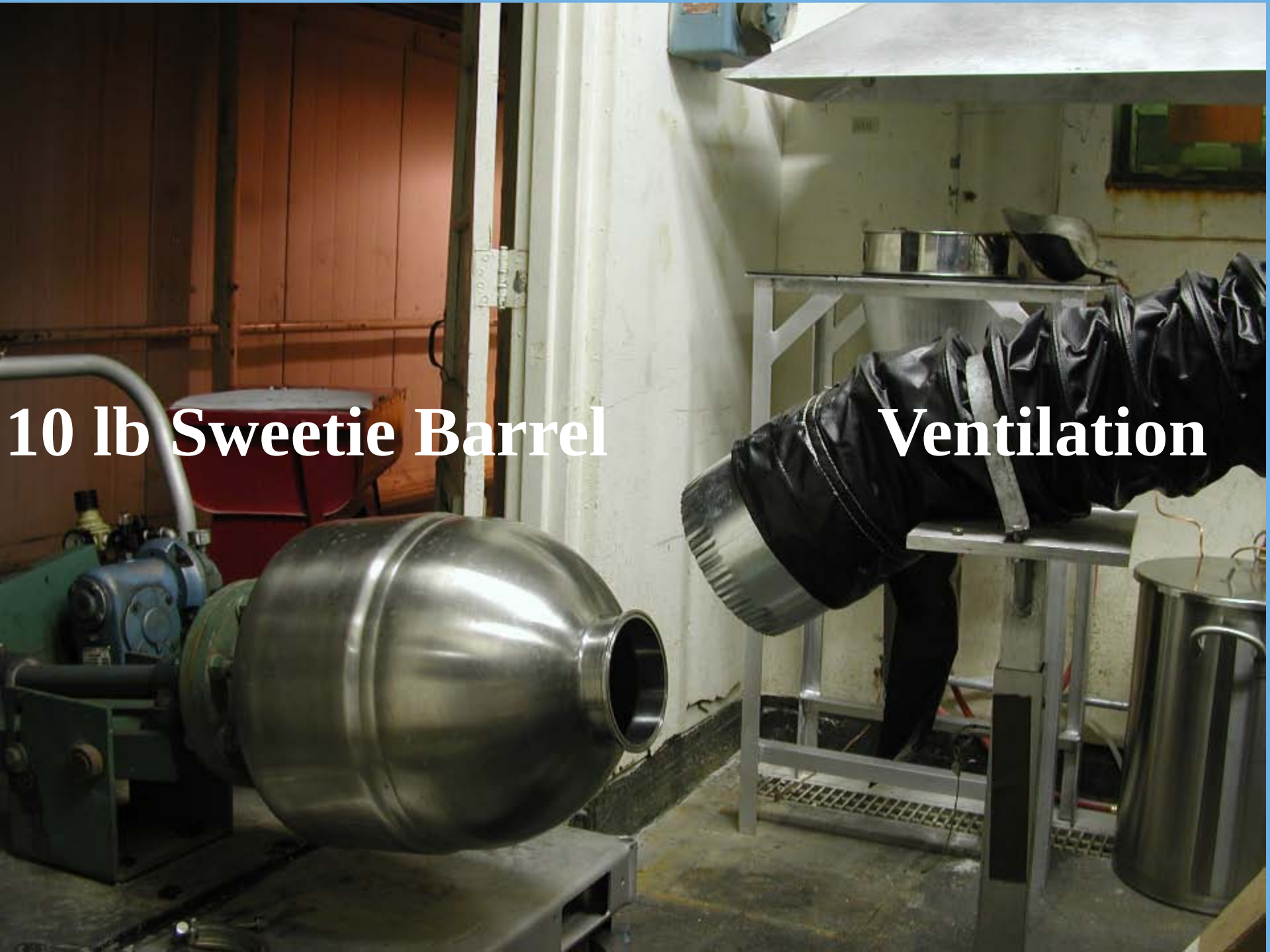


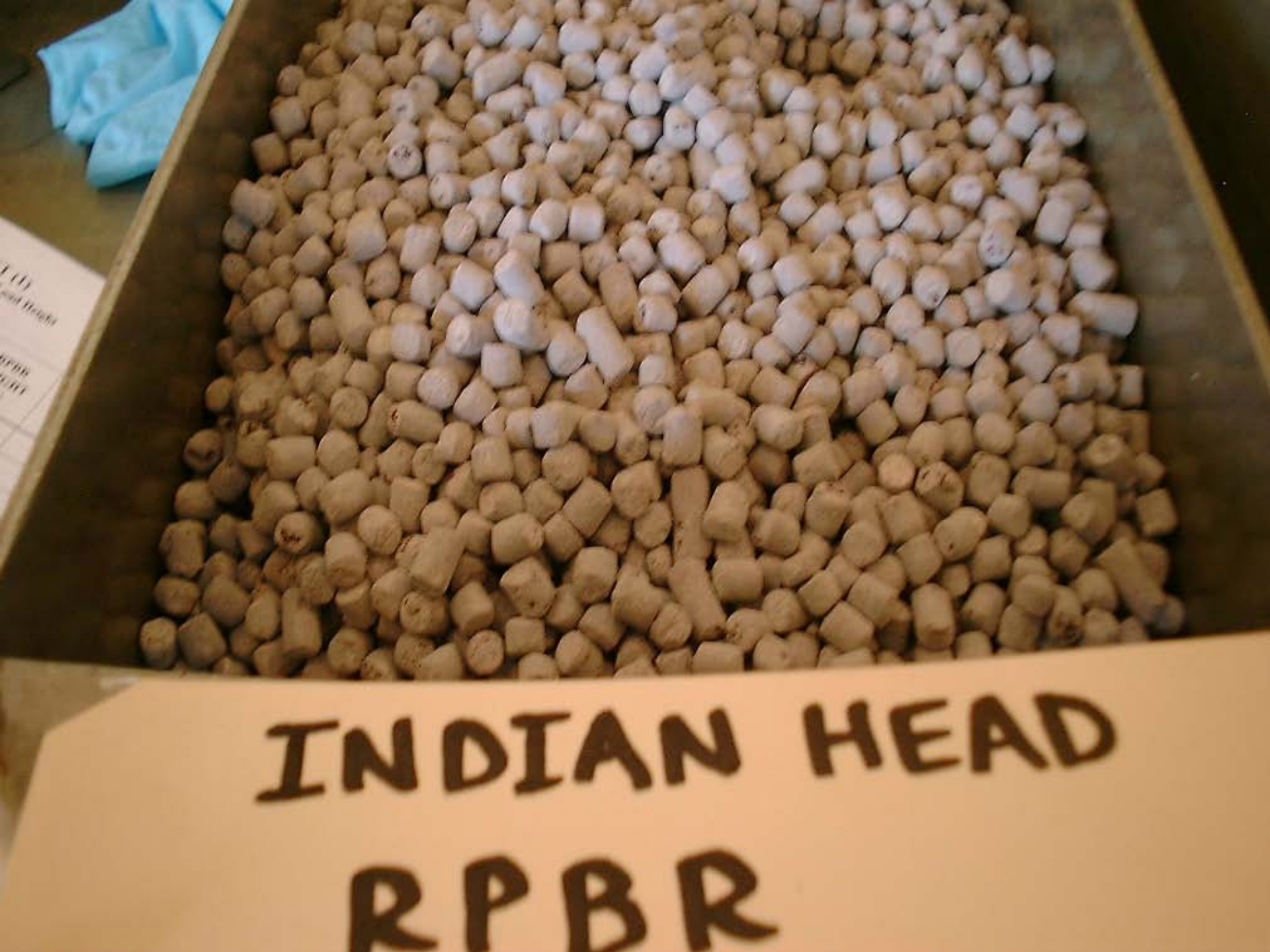
Drying the Pellets

- Pellets were initially dried at 110°F for 2-3 days
- Pellets were finally dried at 130°F for 24 hours

Pellet Coating and Pack-out

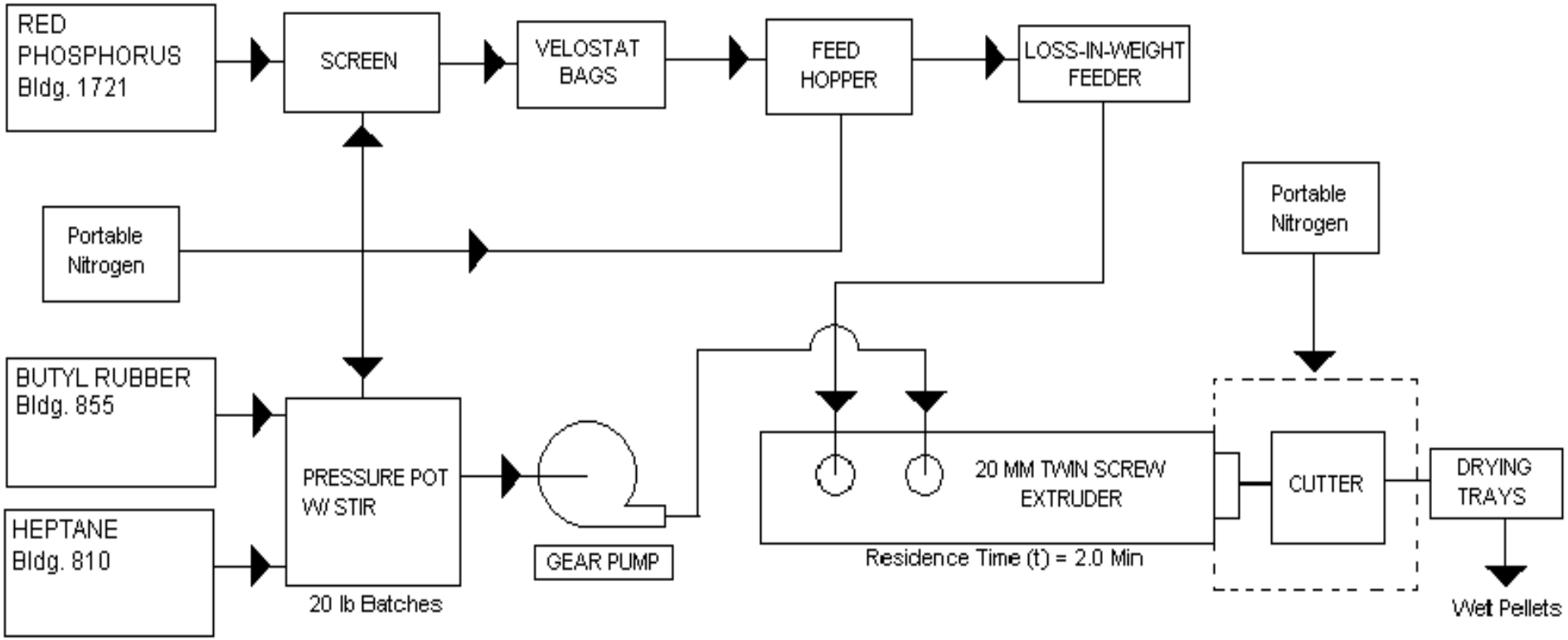
- All of the dried pellets were blended into one lot
- Pellets coated using a 10 lb sweetie barrel
- Pellets coated in 10 lb batches
  - 10 lbs of pellets w/ 0.10 lbs of silica
  - Barrel tumbled at 20 RPM for 10 minutes
- Coated pellets were screened to remove fines
- Pellets were packaged in 10 lb bags


10 lb Sweetie Barrel
Ventilation

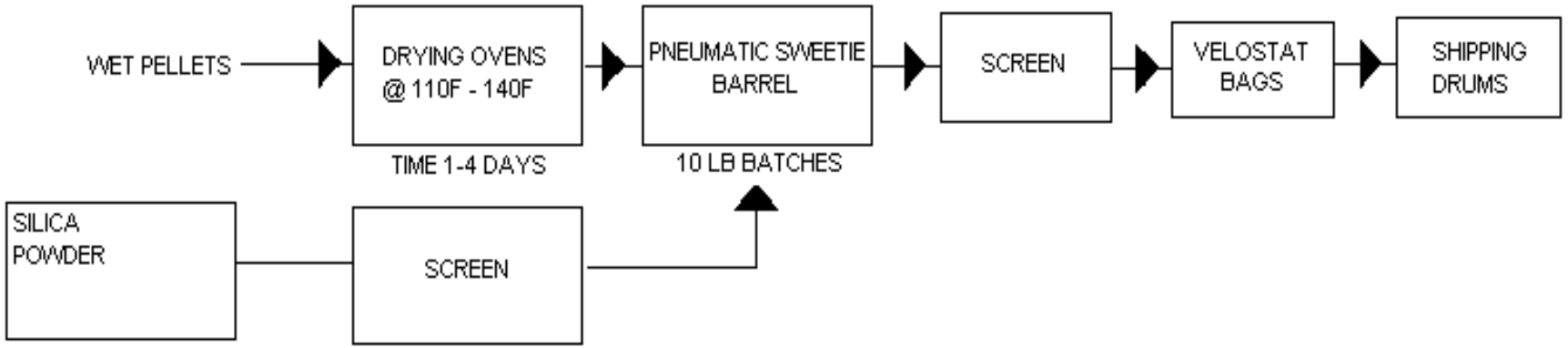

INDIAN HEAD
RPBR

INDIAN HEAD
RPBR
PINE BLUFF
RPBR
LOT: PB-03M001-002

WET PELLET PRODUCTION



FINISHING



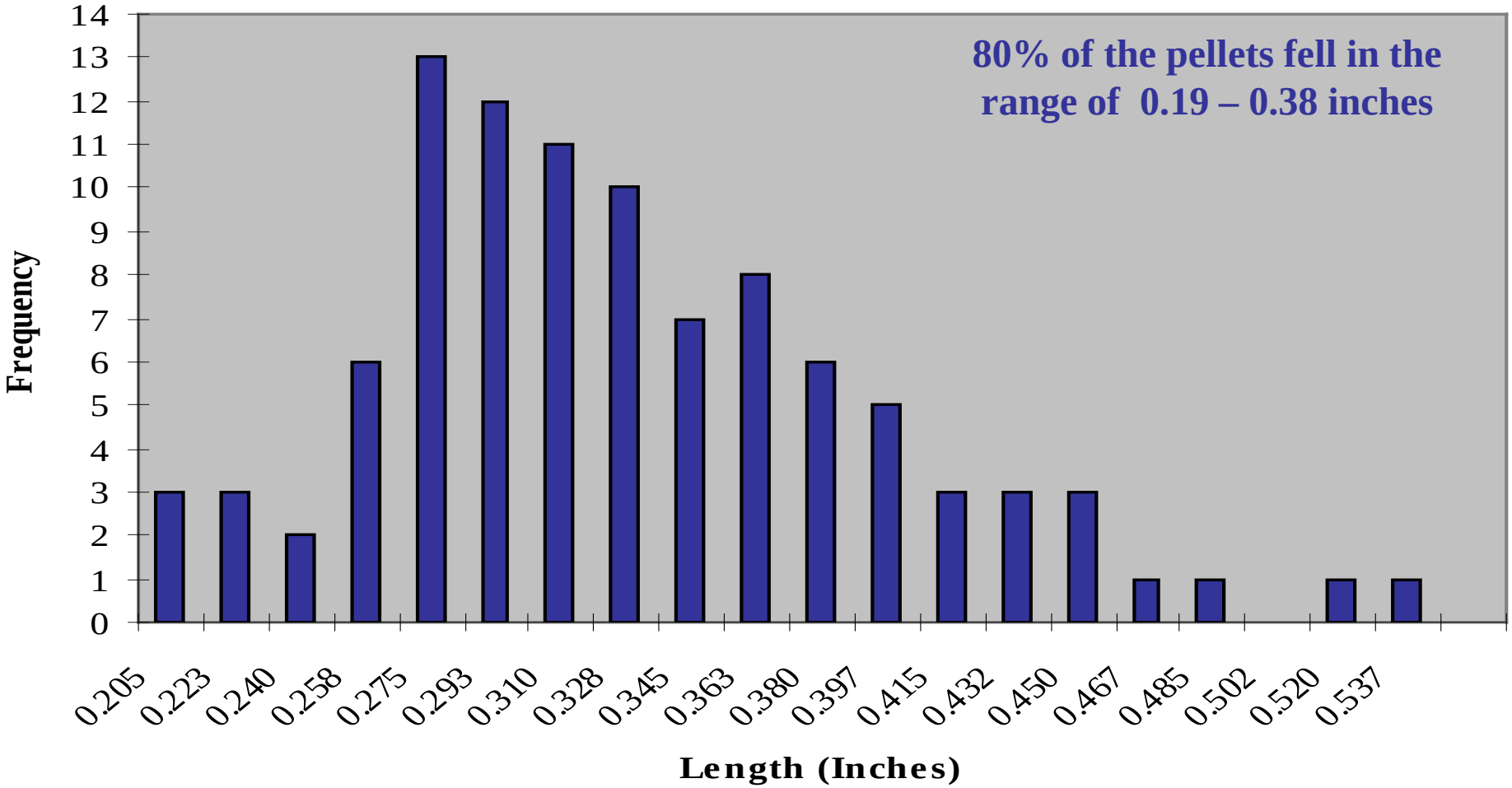
Material Response

Pellet Composition		
Component	Actual Value (%)	Accepted Range (%)
Red Phosphorus	94.2	92.75 – 94.75
Butyl Rubber	4.4	3.0 – 6.0
Silica	1.0	0.75 – 1.25
Total Volatiles	0.4	0.5 Max

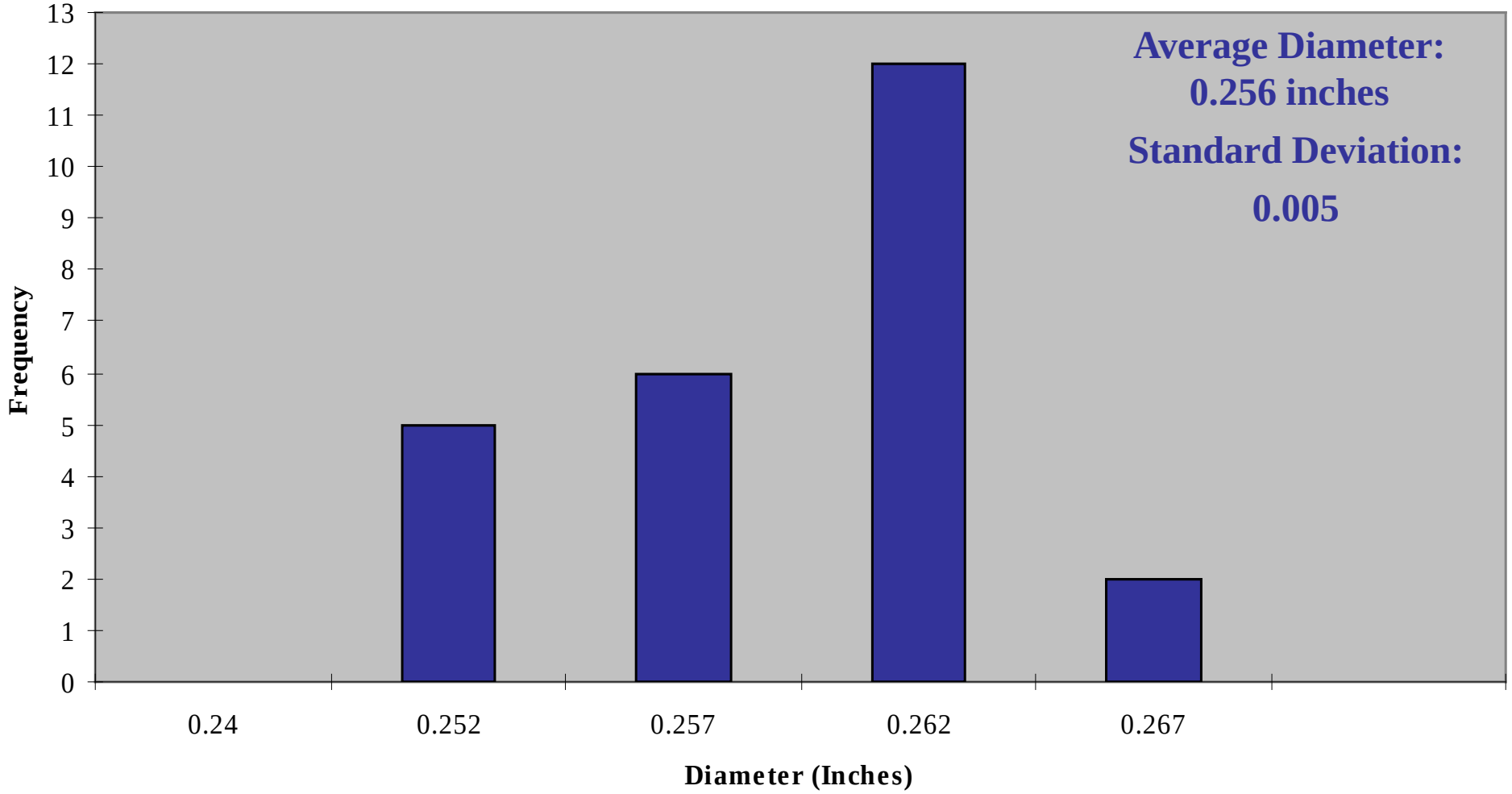
Material Response Cont:

- BAM friction test
  - Tested at 360 N and no reaction occurred
- Impact test
  - 10 drops at a height of 202 cm with no reaction
- ABL ESD test
  - Medium sensitivity at a test result of 0.037J
  - RDX ‘A’ Standard 0.326 J
  - Both are in the medium sensitivity range
- Candle burn test
  - Awaiting result from NSWC Crane

Length Distribution



Pellet Diameter Distribution



Conclusion

- Pellet composition specification was met
- Pellet dimension requirement was met
- Burn time and quality of smoke is unknown
  - Awaiting test results from NSWC Crane
- RPBR process development was a success