PX-70-50

~~*PX-70-52-U~~

19

Particle Characterization

A. A. DUNCAN

OCTOBER, NOVEMBER, DECEMBER

1 9 7 0

Development Division

Mason & Hanger-Silas Mason Co., Inc.

PANTEX PLANT

Amarillo, Texas

For

U.S. Atomic Energy Commission

Albuquerque Operations Office

PARTICLE CHARACTERIZATION

A. A. Duncan

October, November, December, 1970
Endeavor No. 117
Acct. No. 22-2-44-02-317

ABSTRACT

The study of the effects of roll milling on Extex was started; the visual results are indicative that a considerable reduction in crystal size occurs during the formulation process.

DISCUSSION

In the manufacture of Extex (XTX 8003, 80% PETN, 20% Sylgard silicone rubber), the formulation is mixed for about five hours. Freon MF is added in the proportions of four pounds Extex to one quart Freon, to enhance the mixing at lower power than would otherwise be the case. After mixing, the formulation is milled on a three-roll rubber mill for a total of 25 passes.

It has been found that extrudability is related to milling: it seems that an optimum number of passes produces maximum extrudability. This is shown in Fig. 1 which is a plot of mill passes versus extrudability. Extrudability is expressed as an integral number found by the trapezoidal rule of the Rossi-Peakes extrusion test conducted at 125, 150, 175, 200, 225, 250, 275, and 300 psi. In one case (batch 1069-133), two samples were milled six days apart: the results indicate in both cases that extrudability is improved through 20 passes and that an additional five passes was not beneficial; in the unstored sample, a reversion in extrudability occurred. In sample 1069-154, the results were similar to the stored 1069-133 in that 20 passes improved extrudability and additional five passes were not beneficial. Sample 1069-160 exhibited an improvement in extrudability for every case tested, including 25 passes.

It has been concluded from these milling tests that grinding occurs, producing a distribution that enhances extrudability up to a point; additional grinding produces a finer particle size which can be detrimental to extrudability.

Additional milling parameters (temperature, roller spacing, and total milling time) were also evaluated by an extrudability correlation study of 28 samples.

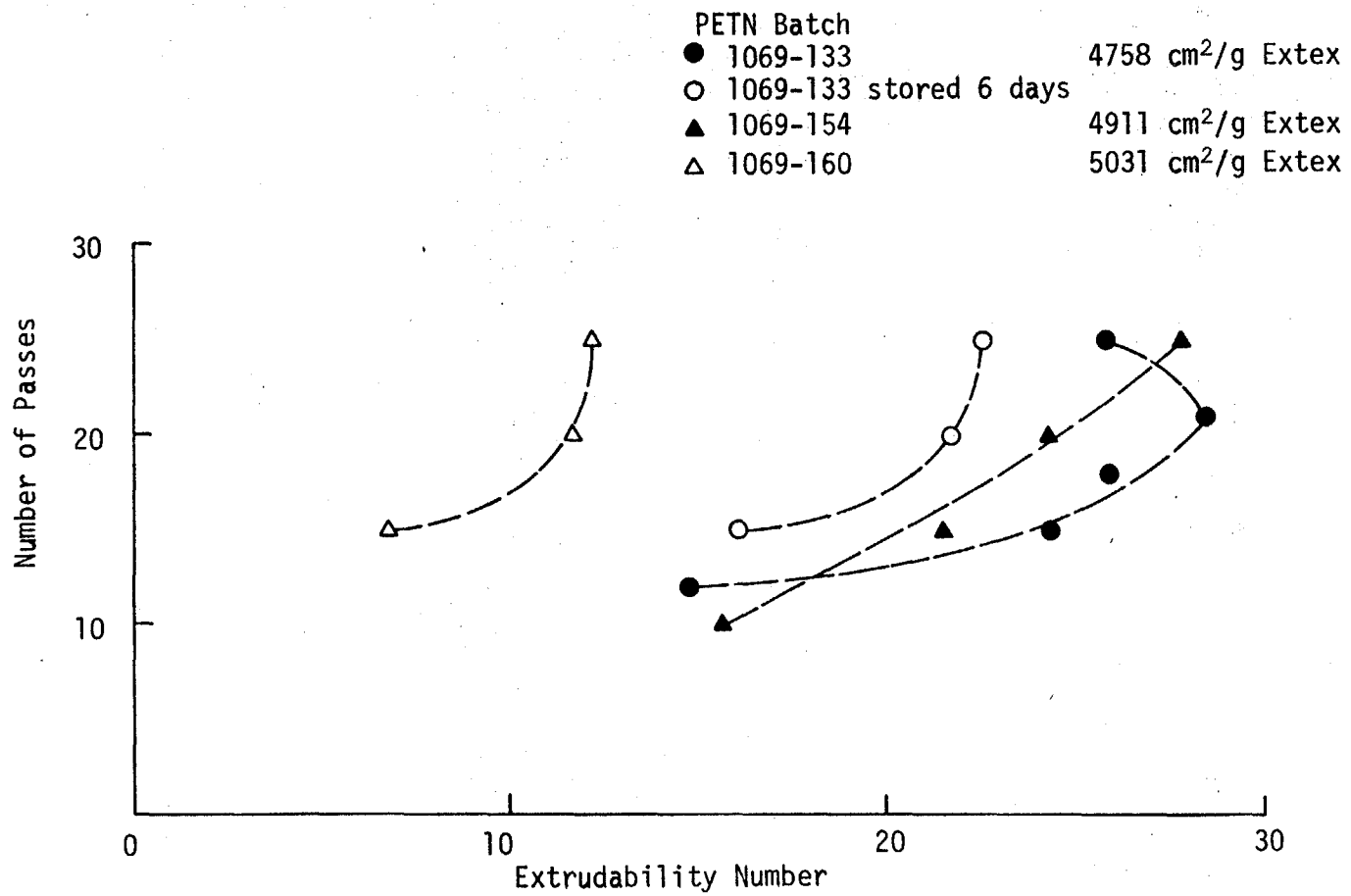


Fig. 1. Extrudability versus mill passes.

The following correlations were found:

Test	Partial Correlation Coefficients	Significance Level
Mill Temperature	+ .52130	.01
Feed Roller Gap	+ .45982	.02
Blade Roller Gap	- .70182	.001
Number of Passes	+ .56014	.001
Total Milling Time	- .24017	<.1

From these results, it can be seen that as the blade roller gap is reduced, extrudability is increased. As the number of passes increases, so does extrudability. With the reduction in blade roller gap and increase in number of passes, there should be an increase in crystal comminution and possibly a tendency for the particle size distribution to peak more sharply at a single mode. Thus a study to evaluate PETN particle changes during milling was started.

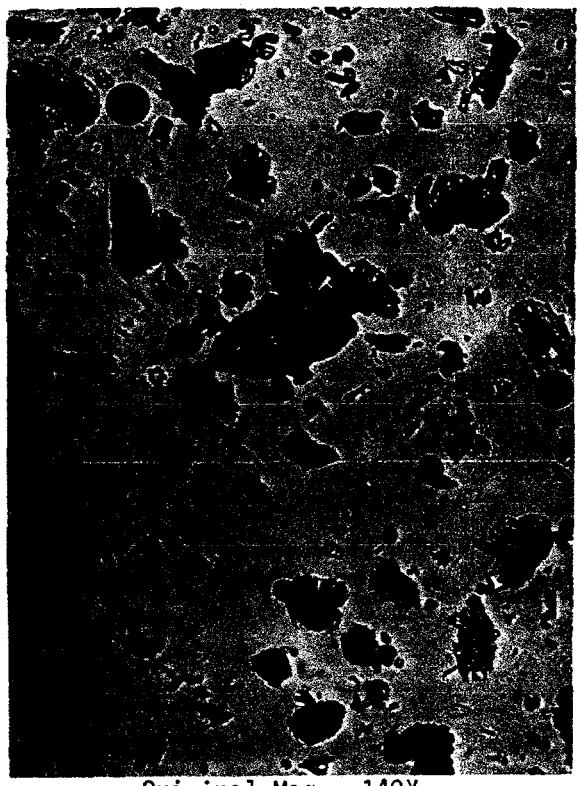
Two methods of evaluating particle size after milling are being studied:

1. Dissolving away the uncured Sylgard and analyzing surface and particle size changes.
2. Dilution of the Sylgard by the addition of Sylgard, Freon MF, or index of refraction oils and then measuring particle size by Zeiss analysis.

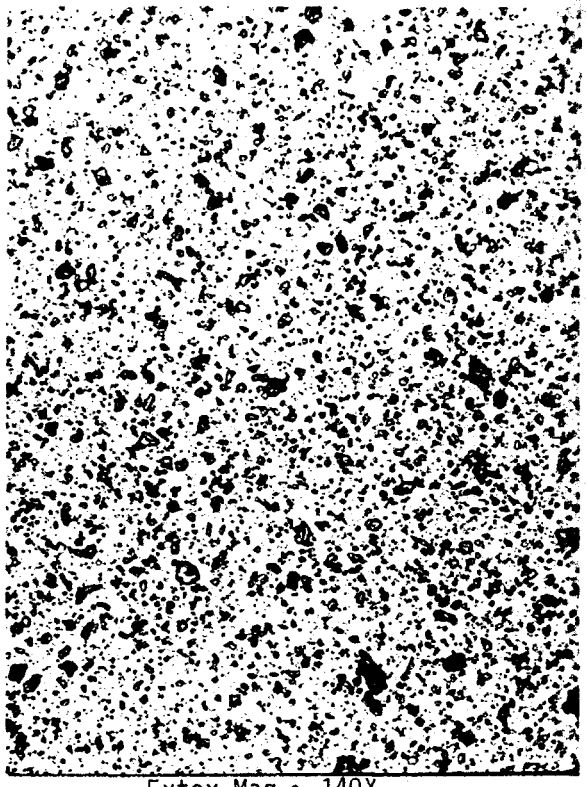
The dilution method appears to be the better method in that particle size reduction or loss does not occur as it does in the solvent method. The use of Freon MF was not satisfactory in that the separation of Sylgard and PETN

was not achieved without harsh treatment. Dilution by adding additional Sylgard is satisfactory; however, the use of index of refraction oils which sharpen the contrast of PETN (those lower than the index of refraction of PETN, 1.554) enhance photomicroscopy measurement. Figs. 2 thru 6 are photomicrographs of PETN before formulation and after 25 mill passes. It can be observed that considerable reduction in particle size has occurred.

Fig. 2 Extex 2070-215-01
PETN 1070-203-01

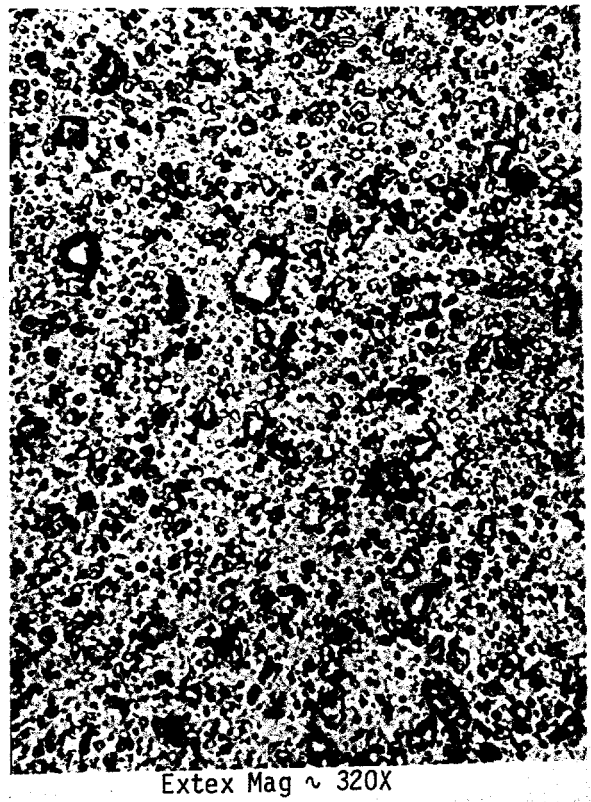


Original Mag ~ 140X



Extex Mag ~ 140X

Original Mag ~ 320X

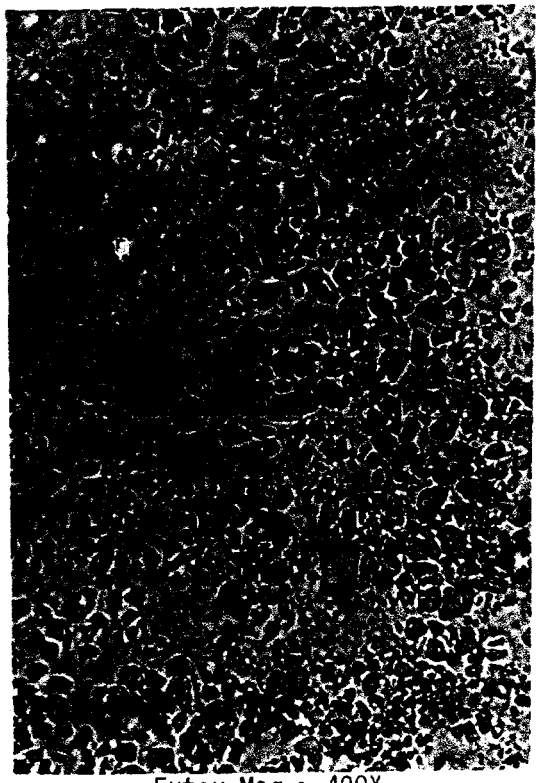


Extex Mag ~ 320X

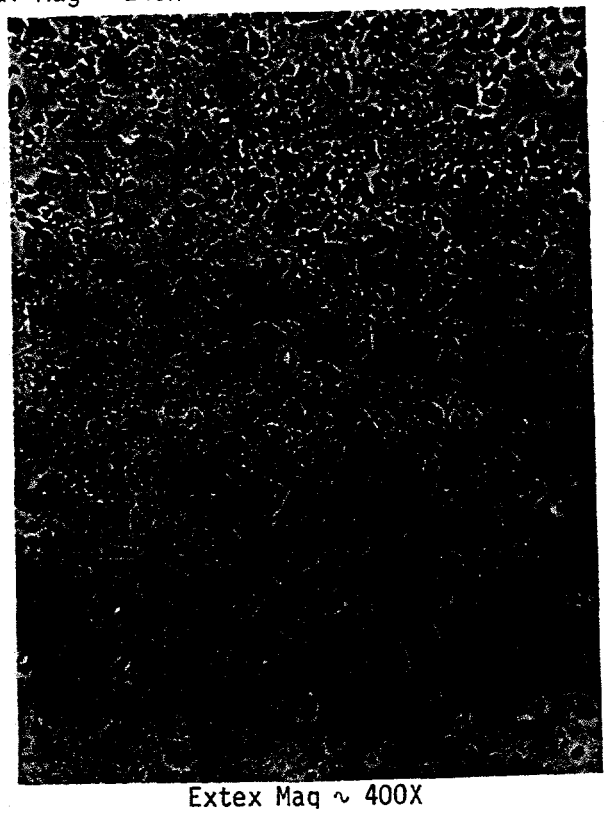
Fig. 3 Extex 8309-301-01 P

PETN 8306-304-01 P

Original Mag ∿ 240X



Extex Mag ∿ 400X

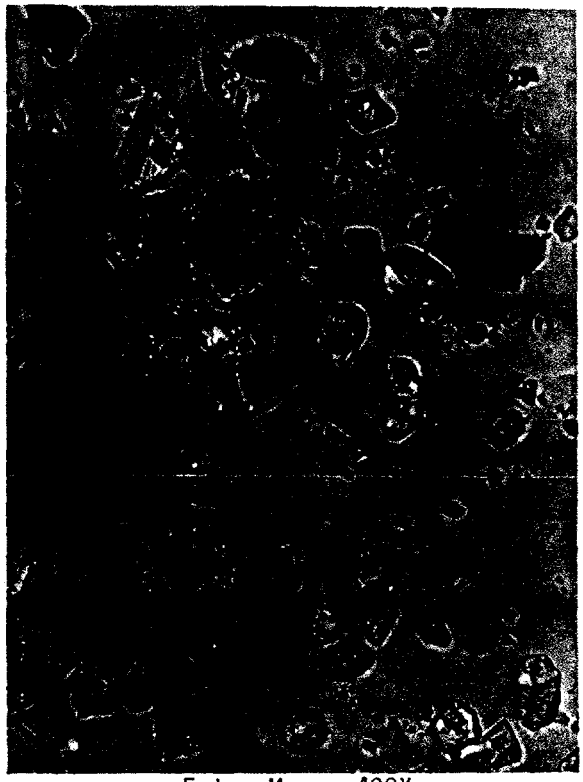


Extex Mag ∿ 400X

Fig. 4 Extex 8309-301-01 D

PETN 8306-304-01 D

Original Mag ~ 145X

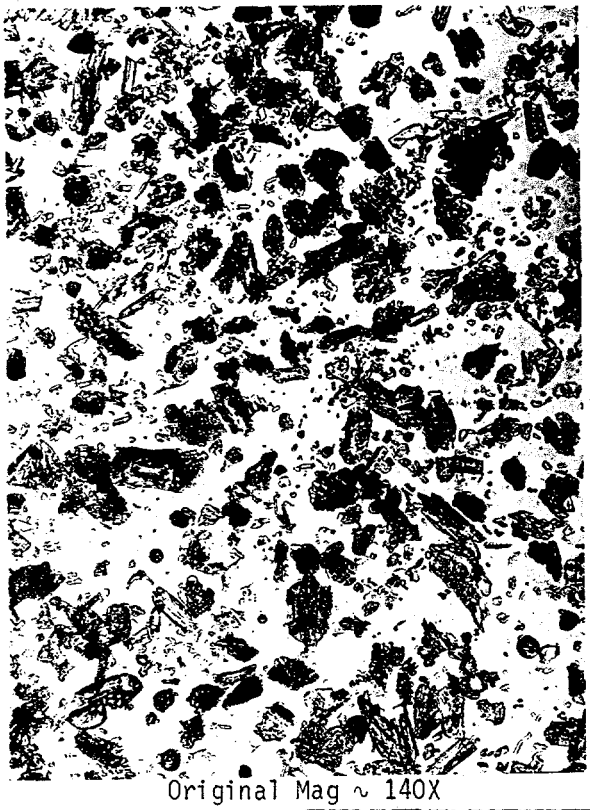


Extex Mag ~ 400X

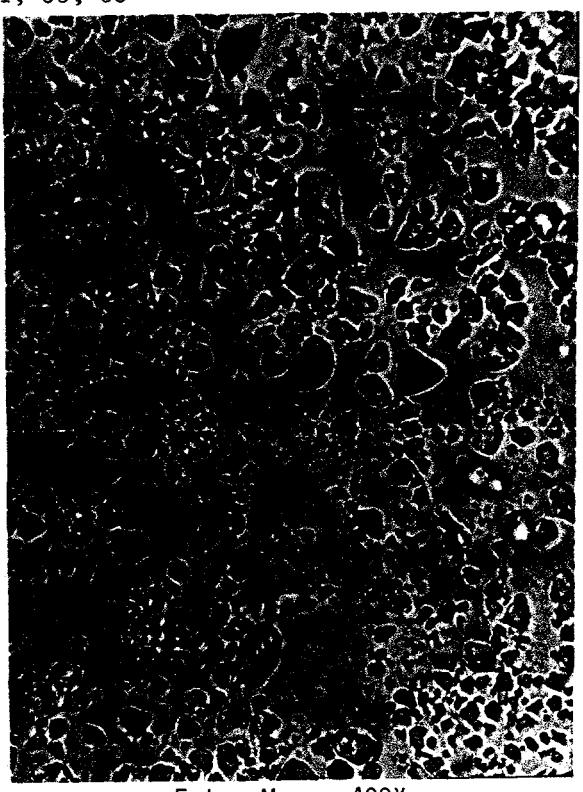
Extex Mag ~ 400X

Fig. 5 Extex 8296-301-01

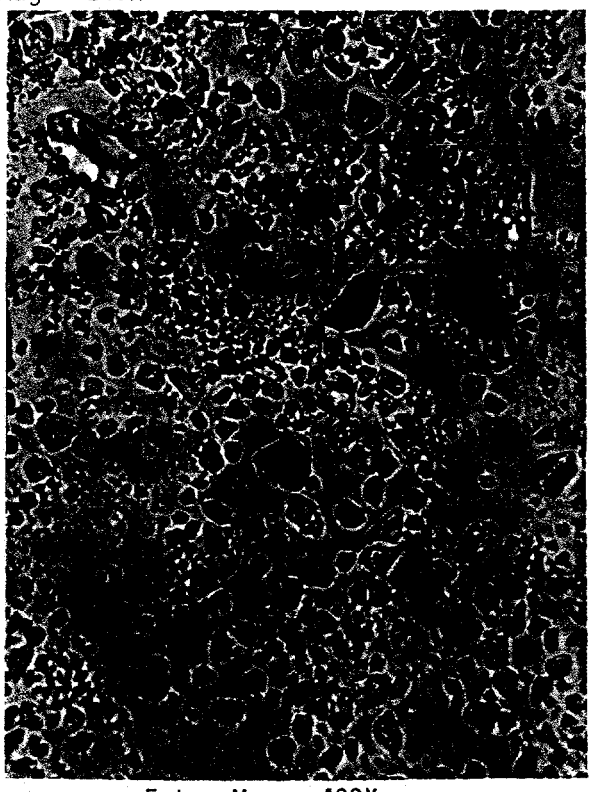
PETN 8148-304-01, 03, 05



Original Mag ∿ 140X



Extex Mag ∿ 400X

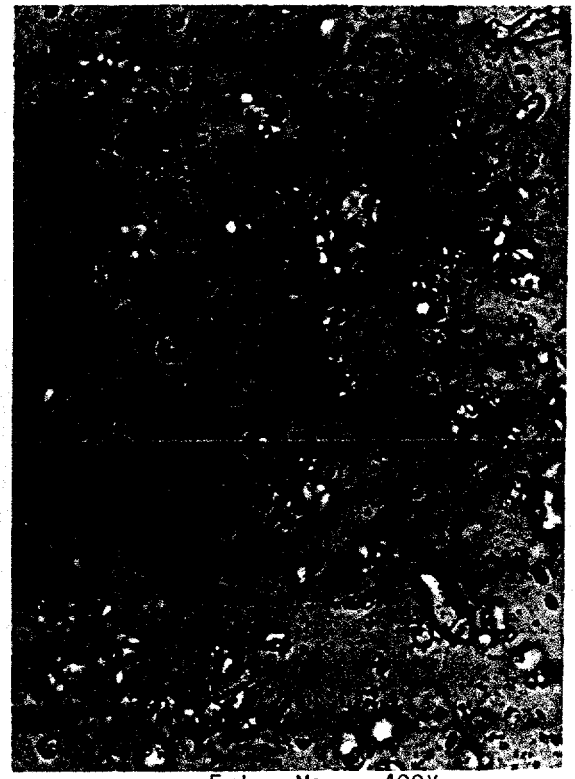


Extex Mag ∿ 400X

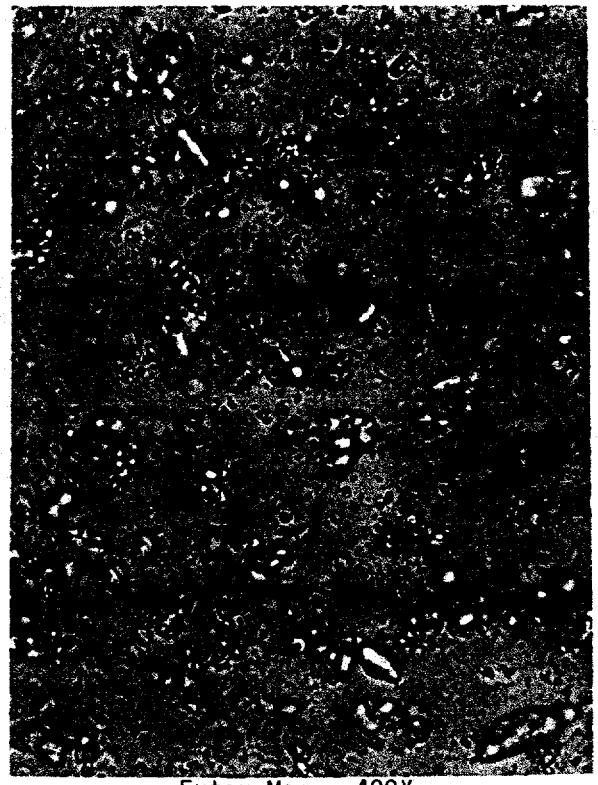
Fig. 6 Extex 2069-156-01

PETN 1069-154-01

Original Mag ∿ 564X



Extex Mag ∿ 400X



Extex Mag ∿ 400X

FUTURE WORK

Additional investigation shall be made on the amount of change that occurs during formulation. This shall be done by measuring particle distributions for various batches before and after formulation so that batch differences can be evaluated. The effects of mixing and milling shall also be evaluated.