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**The Feasibility of Utilizing a Fluid
Energy Mill in the Grinding of
Potassium Perchlorate**

L. Wayne Collins

September 28, 1977

Monsanto

MOUND LABORATORY

Miamisburg, Ohio
operated by

MONSANTO RESEARCH CORPORATION

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**UNITED STATES ENERGY RESEARCH
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Abstract

The feasibility of utilizing the fluid energy mill for grinding potassium perchlorate for use in pyrotechnic blends was investigated. It was found that the particle size of the material was significantly reduced by the action of the mill with no significant change in the level of contamination. The grinding parameters selected seemed to have no direct relationship with the physical properties of the final product. The action of the mill induced a significant electrostatic charge on the potassium perchlorate which affects the handling characteristics and possibly the chemical character. The feasibility but not the desirability of utilizing the fluid energy mill to grind potassium perchlorate was demonstrated.

Introduction

The fluid energy mill is a device which suspends particles of a material in a systemized air flow pattern which facilitates particle-particle collisions to create a grinding effect. Since the grinding action is produced through a particle-particle interaction rather than through forces applied through an agent such as metal balls or blades, contamination from the grinding agent should be eliminated or greatly reduced. This prospect of a decreased contaminant level suggests that the fluid energy mill might offer significant improvements over present grinding techniques used in the processing of materials for pyrotechnic blends. Therefore, the feasibility of using this device to obtain desired physical parameters without altering chemical composition through the introduction of contaminants was investigated using two different grades of potassium perchlorate.

Description of the fluid energy mill

The fluid energy mill is constructed from stainless steel and measures about 46 cm in height (with collector pan) by about 25 cm in diameter. Air was used as the grinding fluid in this study although almost any noncorrosive gas can be used. Air was supplied to the mill by an air compressor via a manifold regulator system. The air was circulated through a drying tank to remove moisture and through two Deltech Filters to remove particulate contaminants prior to entering the mill. The possibility of using bottled nitrogen or air to reduce the likelihood of contamination was investigated but it was found that the mill required an air flow rate of 710 liters/min which consumes a standard K cylinder of nitrogen in 9 min. The filtered system which was used was sampled for 96.5 hr at a flow rate of 1.5 ft³/hr and indicated a contaminant content of 9×10^{-5} mg/ft³.

as determined by weight gain. Infrared analysis indicated a contaminant level of 3×10^{-5} mg/ft³. However, contaminant content at this level is meaningless because of the likelihood of external sources of contamination and the inability to accurately control sampling variables to the high degree of precision required. The analysis does indicate that no gross contamination is present in the air system.

Some problems were also encountered in the operation of the mill. The construction of the mill does not allow for adequate sealing so that as much as 25% of the sample being ground was lost through leakage into the air. The major portion of this leakage was eliminated by fitting all sealing junctures with "O" rings. There was no apparent change in the operation of the mill with the addition of these seals, and the rings showed no indication of wear after extended periods of operation in the mill. It should be noted that none of the "O" ring seals were in the direct air flow pattern and seemed to be insulated from the working area by a layer of KClO₄ which remained packed into the crevice accommodating the ring. The powder continued to be lost through the air exit port but this amounted to less than 10% of the sample introduced into the mill. Attempts to eliminate this source of leakage were unsuccessful; consequently, all operations were performed in a hood.

The vibrating tube feed mechanism presented another problem. The flow properties of the potassium perchlorate powder were such that a smooth reproducible feed rate into the mill could not be obtained. For a

particular feed rate setting, the actual measured rate varied by as much as a factor of three for the same sample and by even more for different samples of potassium perchlorate. A somewhat smoother feed rate was obtained after fastening a #20 mesh sieve over the end of the feed tube to break the agglomeration of the potassium perchlorate particles although a consistent feed rate was never obtained. An auger type feed mechanism would probably give better control of the feed rate when powdered samples such as the potassium perchlorate are used.

During operation of the mill, there was some problem with condensation of water in the sample collector container. This water was brought in with outside air which was drawn in through the feed hopper of the mill by the vacuum produced to carry the sample to the grinding chamber. The rapid expansion of the air within the mill caused the moisture to precipitate into the sample container. This necessitated drying the sample after grinding and could lead to particle growth with prolonged exposure. For this reason, no more than 1 kg of material was passed through the mill before the mill was disassembled, cleaned, and dried. This prevented the accumulation of large quantities of water and minimized the opportunity for crystal growth.

Materials

Two types of potassium perchlorate were used in this study: military specification grade and high-purity grade. The military specification grade was designated QC1810 and was supplied by Hummel Chemical Company, South Plainfield, New Jersey. The high-purity

material was designated QC1808 and was supplied by G. Fredrick Smith Company, Columbus, Ohio. The analytical data for these materials are given in Tables 1 and 2.

Both of the potassium perchlorate samples were sieved through a #20 mesh sieve and dried at 65°C for 24 hr in vacuo prior to using. All samples were obtained by quartering the entire bulk of material to near the sample size required and then spin-riffling this portion to get the correct sample size. Also, all materials which were passed through the Fluid Energy Mill were redried for 24 hr at 65° in vacuo before the analytical samples were obtained.

It should be noted that a greenish color was observed in the high purity material when the package was opened. However, chemical analysis did not show any significant contamination of this material.

Operating conditions

There are two operating parameters which influence the performance of the Fluid Energy Mill: grinding pressure and feed rate. In this study, only the grinding pressure was controllable due to the flow characteristics of the potassium perchlorate as previously mentioned. Grinding pressures of 20, 30, and 42 psig were selected as variables in the study.

The working fluid enters the mill by two ports through the manifold which is the grinding area and through the feed hopper in which an air flow is used to carry the sample to the grinding area. The air pressure to the feed hopper must always be greater than the grinding pressure;

otherwise, the sample is blown back out of the feed hopper. Through trial and error it was found that with a total working pressure of 80 psig, no more than 42 psig could be distributed to the grinding chamber without producing occasional blowbacks into the feed hopper. Thus the upper limit for pressure in the study was established.

In order to prevent the feed rate from being totally unknown, the perchlorate was divided into 200-g samples with the total time required to pass through the feeder being recorded. An average of the time required for the 200-g samples to pass through the feeder was taken for each sample and used as the feed rate. Feed-rate settings were changed when visual observation indicated that the flow had slowed or increased. With these visual adjustments, the feed rate varied by a factor of two or less. A feed rate of about one-half kilogram per hour was maintained.

Sampling and identification

One kilogram of each of the potassium perchlorate samples was run through the mill for each operating condition (manifold pressure). The sample was then dried at 65° for 24 hr in vacuo and quartered, and the final quarter was spinruffled to obtain the analytical sample. It was noted that after grinding and drying, the potassium perchlorate acquired a large static charge. The fine powder was strongly attracted to the sides of containers and to metal spatulas. This could lead to some difficulty in preparing stoichiometric blends.

The samples which were ground in the mill were identified by their QC number followed

Table 1 - CHEMICAL ANALYSES OF HIGH-PURITY POTASSIUM PERCHLORATE
BEFORE AND AFTER GRINDING IN THE FLUID ENERGY MILL

QC number	Color	Cl ⁻ (ppm)	ClO ₃ ⁻ (ppm)	ClO ⁻	BrO ₃ ⁻	pH	KClO ₄ (wt %)	Grit (wt %)	Ca (ppm)	Mg (ppm)	Na (ppm)
1808	White	<0.35	<0.35	ND	ND	5.8	100.1	0.005	<10	<10	<10
1808-20 ^a	White	<0.35	<0.35	ND	ND	5.7	99.32	0.007	<10	<10	<10
1808-30	White	<0.35	<0.35	ND	ND	5.7	99.09	0.004	<10	<10	<10
1808-42	White	<0.35	<0.35	ND	ND	5.8	99.54	0.003	<10	<10	<10
1808-R ₁ ^b	White	<0.35	<0.35	ND	ND	5.7	99.24	0.010	<10	<10	<10
1808-R ₂	White	<0.35	<0.35	ND	ND	5.5	99.48	0.019	<10	<10	<10
1808-R ₃	White	<0.35	<0.35	ND	ND	5.5	99.01	0.014	<10	<10	<10

^aThe -XX numbers following the QC number refer to the manifold pressure of the Fluid Energy Mill at which the sample was ground.

^bThe -R_X designation refers to the repeat samples at a manifold pressure of 42 lb used to demonstrate the reproducibility of the grinding technique.

Table 2 - CHEMICAL ANALYSES OF MILITARY SPECIFICATION POTASSIUM
PERCHLORATE BEFORE AND AFTER GRINDING IN THE FLUID ENERGY MILL

QC number	Color	Cl ⁻ (ppm)	ClO ₃ ⁻ (ppm)	ClO ⁻	BrO ₃ ⁻	pH	KClO ₄ (wt %)	Grit (wt %)	Ca (ppm)	Mg (ppm)	Na (ppm)
1810	White	0.016	0.005	ND	ND	5.9	100.6	0.009	<10	<10	210
1810-20	White	0.030	0.030	ND	ND	5.8	99.57	0.015	<10	<10	195
1810-42	White	0.008	0.006	ND	ND	5.8	99.54	0.020	<10	<10	200
1810-R ₁	White	0.024		ND	ND	5.6	99.08	0.013	<10	<10	225
1810-R ₂	White	0.024		ND	ND	5.7	99.16	0.016	<10	<10	233
1810-R ₃	White	0.025		ND	ND	5.7	99.03	0.014	<10	<10	239

by a two digit number which indicates the grinding pressure in the mill. Three samples of each QC number were ground at the 42 psig grinding pressure to establish the reproducibility of results. These samples were labeled as the QC number followed by R₁, R₂, or R₃ to indicate repeat run #1, repeat run #2, etc.

Chemical analysis

Chemical analyses were performed on all samples according to SS308214.* The results of these analyses are presented in Tables 1 and 2.

The analyses of the potassium perchlorate samples indicate that no significant level of contamination was introduced by the action of the Fluid Energy Mill. Initially, six of the samples failed to pass the grit and water insoluble tests. However, there was some question as to the validity of the analytical procedure used in testing these samples. Repetition of these tests yielded lower values for grit and water insolubles which fall within the specification limits.

X-ray patterns for a sample of QC 1808 contained 100 lines whereas the patterns for QC 1810 showed 78 lines. All the lines were indexable for potassium perchlorate either by the experimental pattern for QC 1601 or by theoretical d-spacings calculated from lattice parameters (JCPDS pattern I 7-211) with the exception of three weak lines in each pattern (d-spacings of 4.9663, 4.2101, and 3.8839Å).

*Analytical Specification, Sandia Laboratories, Albuquerque

Physical characterization

The change in the surface area of potassium perchlorate after grinding in the Fluid Energy Mill as determined by the Brunauer-Emmett-Teller (BET) method is shown in Table 3. This table shows that a significant gain in surface area can be obtained with the mill although the precision of the measurements in this range of surface areas is so low as to preclude quantitative comparisons. The data show approximately the same final surface areas for the high purity material as for the military specification material although the starting products were visibly different. The increased surface area of both materials could be detected visually. It should also be noted that a substantial reduction in particle size is necessary to increase the surface area of

Table 3 - CHANGE IN THE SURFACE AREA (BET) OF POTASSIUM PERCHLORATE AFTER GRINDING IN THE FLUID ENERGY MILL

QC number	Surface Area (m ² /g)
1808	0.05
1808-20	1.05
1808-30	1.39
1808-42	1.02
1808-R ₁	1.61
1808-R ₂	1.51
1808-R ₃	1.58
1810	0.05
1810-20	0.78
1810-42	1.22
1810-R ₁	1.50
1810-R ₂	1.14
1810-R ₃	1.02

a relatively nonporous material such as the potassium perchlorate by the magnitude indicated by the BET measurements. The large increase in surface area is then an indicator of the effectiveness of the grinding properties of the Fluid Energy Mill.

The particle size data for the potassium perchlorate samples as determined by the Zeiss method is given in Tables 4 and 5. These data indicate that the particle size distribution based on number of particles shows a particle size reduction of about one-half. This is in agreement with the Coulter counter data presented later. The variation between the samples probably contains an element of error attributed to agglomeration of very small particles due to the relatively strong static attractive forces. If these were not adequately dispersed by the oil, then estimation of the number of particles agglomerated could tend to bias the mean particle size toward larger values. Repetition of these analyses produced order of magnitude differences in the results reported for some samples which indicates that this method of particle size determination is suspect for samples of this type.

Optical photographs taken at 400X are shown in Figures 1 to 4 and indicate a significant reduction in the particle size of both the military specification and the high purity potassium perchlorate after passage through the Fluid Energy Mill. There seems to be some slight variation in the apparent particle size for those samples tested for reproducibility although these differences are probably not significant. All samples which passed through the Fluid Energy Mill have substantially smaller particle sizes than the original material.

The scanning electron microscopy (SEM) photographs in Figures 5 to 17 show the

agglomeration of the very fine particles of samples after passage through the Fluid Energy Mill. No significant change in the particle morphology is discernible although this aspect is difficult to judge from the photographs. Again, a significant particle size reduction is indicated.

The Coulter Counter particle size data for the samples are presented in Figures 18 to 43. These data show both a smaller average particle size after grinding in the Fluid Energy Mill and a smaller range of particle sizes. For example, the QC 1808 potassium perchlorate data shown in Figures 18 and 19 exhibit a distribution in which 50% of the particles by weight have diameters of 90 μm or greater, or 50% by number have diameters of 6 μm or greater. After grinding at a mill pressure of 20 psig, the distribution had shifted to where the 50% by weight point occurred at about 6 μm (Figures 20 and 21). The grinding produced a fairly reproducible particle size range, much improved over the untreated samples, although the grinding pressure seemed to make little difference in the final product.

Conclusions and recommendations

The fluid energy mill significantly reduced the range of particle sizes and the mean particle size of both high purity and military specification potassium perchlorate without introducing significant levels of contamination. No definite relationship between manifold grinding pressures and the physical parameters of the product could be determined; grinding pressures between 20 and 42 psig generate about the same physical product. Also, the physical characteristics of the initial material

Table 4 - ZEISS PARTICLE SIZE MEASUREMENTS FOR HIGH-PURITY POTASSIUM
PERCHLORATE BEFORE AND AFTER GRINDING IN THE FLUID ENERGY MILL

QC number	Arith.	Std.	Geom.	Std.	Range	Mode	Percentiles				Surface Area	
	Mean	Dev.	Mean	Dev.			25%	50%	75%	100%	Arith.	Geom.
	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(m^2/g)	(m^2/g)
1808	7.74	5.21	6.04	1.88	25.39	4.25	4.11	5.91	9.63	27.16	0.31	0.39
1808-20	6.24	4.37	4.85	2.06	25.39	3.70	3.05	4.87	8.07	27.16	0.38	0.49
1808-30	3.52	3.85	2.40	1.61	25.39	2.04	1.77	2.38	3.37	27.16	0.68	0.99
1808-42												
1808-R ₁	4.96	3.36	4.06	1.52	24.84	3.14	3.14	4.02	5.53	26.60	0.48	0.59
1808-R ₂	5.85	3.89	4.71	1.82	25.39	4.80	3.22	4.73	7.20	27.16	0.41	0.51
1808-R ₃	6.23	4.37	4.96	2.06	25.39	3.70	3.09	5.05	8.20	27.16	0.38	0.48

Table 5 - ZEISS PARTICLE SIZE MEASUREMENTS FOR MILITARY SPECIFICATION GRADE
POTASSIUM PERCHLORATE BEFORE AND AFTER GRINDING IN THE FLUID ENERGY MILL

QC number	Arith.	Std.	Geom.	Std.	Range	Mode	Percentiles				Surface Area	
	Mean	Dev.	Mean	Dev.			25%	50%	75%	100%	Arith.	Geom.
	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(μm)	(m^2/g)	(m^2/g)
1810	11.04	7.34	9.08	2.29	25.94	11.42	5.23	9.49	15.96	27.71	0.22	0.26
1810-20	7.19	5.16	5.75	2.18	24.84	5.90	3.57	5.52	10.24	26.60	0.33	0.41
1810-42	4.31	2.92	3.52	1.61	24.84	3.14	2.60	3.51	4.96	26.60	0.55	0.68
1810-R ₁	4.94	4.12	-	-	25.39	3.70	3.43	0.42	4.11	27.16	0.48	-
1810-R ₂	5.98	5.25	-	-	25.39	2.59	2.56	3.84	7.30	27.16	0.40	-
1810-R ₃	6.36	5.56	-	-	25.39	3.70	2.57	3.97	8.27	27.16	0.37	-

seemed to have little effect on the final product; the high purity material initially had the consistency of sugar and the military specification material had the consistency of flour, yet the final products after grinding were almost identical. The feed rate into the mill also had little effect as long as the potassium perchlorate was not forced in at such a rate as to hinder proper operation. In addition to the reduction in particle size, the potassium perchlorate acquired a substantial static charge which significantly altered its handling characteristics and possibly its chemical character.

In summary, this study demonstrated the feasibility of using the fluid energy mill to grind potassium perchlorate without the introduction of contaminants but does not demonstrate the desirability of utilizing this process. To determine whether the high surface area is a desirable quality, the following studies should be performed to evaluate the applicability of high surface area potassium perchlorate to pyrotechnic formulations:

1. A study should be performed to determine the blending characteristics of the potassium perchlorate. The high surface area and significant static charge make handling much more difficult and could lead to problems in maintaining stoichiometric blends.
2. The compaction characteristics of blends formulated from the perchlorate should be determined. Experience has shown that the loading characteristics of the pyrotechnic blend can affect the performance of the device into which it is loaded.

Since the $KClO_4$ is much less dense after grinding (approximately one-half) the loading characteristics would be different. Also, the tenacity with which the charged particles cling to objects could introduce problems in using automatic loaders.

3. The performance of the blends should be evaluated. The more finely divided state could alter firing and performance characteristics through increased surface contact or through alterations in the chemical properties induced by the static charge. Effects of this type might be predicted by the Evans-Polyani effect. The solid-state chemistry of the charged material could also be studied which might give significant insight into the nature of pyrotechnic reactions.

4. Stability studies should be performed to ensure that the physical characteristics of the blend do not contribute to an increase in the low temperature reaction rate of the materials, i.e., to ensure the "compatibility" of the materials.

FIGURE 1 - Optical photographs of high-purity potassium perchlorate samples QC 1808, QC 1808-20, QC 1808-30, and QC 1808-42 at 320X.

FIGURE 2 - Optical photographs of high-purity potassium perchlorate samples QC 1808-R₁, QC 1808-R₂, and QC 1808-R₃ at 320X.

FIGURE 3 - Optical photographs of military specification potassium perchlorate samples QC 1810, QC 1810-20, and QC 1810-42 at 320X.

FIGURE 4 - Optical photographs of military specification potassium perchlorate samples QC 1810-R₁, QC 1810-R₂, and QC 1810-R₃ at 320X.

FIGURE 5 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample QC 1808 at 80X, 320X, 560X, and 1280X.

FIGURE 6 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample QC 1808-20 at 320X, 560X, 800X, and 1600X.

FIGURE 7 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample QC 1808-30 at 320X, 560X, 800X, and 1600X.

FIGURE 8 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample QC 1808-42 at 320X, 560X, 800X, and 1600X.

FIGURE 9 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample 1808-R₁ at 320X, 560X, 800X, and 1600X.

FIGURE 10 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample 1808-R₂ at 320X, 560X, 800X, and 1600X.

FIGURE 11 - Scanning Electron Microscopic photographs of high-purity potassium perchlorate sample 1808-R₃ at 160X, 320X, 560X, and 800X.

FIGURE 12 - Scanning Electron Microscopic photographs of military specification potassium perchlorate at 80X, 320X, 560X, and 800X.

FIGURE 13 - Scanning Electron Microscopic photographs of military specification potassium perchlorate at 320X, 560X, 800X, and 1600X.

FIGURE 14 - Scanning Electron Microscopic photographs of military specification potassium perchlorate QC 1810-42 at 320X, 560X, 800X, and 1600X.

FIGURE 15 - Scanning Electron Microscopic photographs of military specification potassium perchlorate QC 1810-R₁ at 320X, 560X, 800X, and 1600X.

FIGURE 16 - Scanning Electron Microscopic photographs of military specification potassium perchlorate QC 1810-R₂ at 320X, 560X, 800X, and 1600X.

FIGURE 17 - Scanning Electron Microscopic photographs of military specification potassium perchlorate QC 1810-R₃ at 320X, 560X, 800X, and 1600X.

FIGURE 18 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808.

FIGURE 19 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808.

FIGURE 20 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808-20.

FIGURE 21 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808-20.

FIGURE 22 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808-30.

FIGURE 23 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808-30.

FIGURE 24 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808-42.

FIGURE 25 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808-42.

FIGURE 26 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808-R₁.

FIGURE 27 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808-R₁.

FIGURE 28 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808-R₂.

FIGURE 29 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808-R₂.

FIGURE 30 - Coulter Counter weight distribution for high-purity potassium perchlorate sample QC 1808-R₃.

FIGURE 31 - Coulter Counter number distribution for high-purity potassium perchlorate, QC 1808-R₃.

FIGURE 32 - Coulter Counter weight distribution for military specification potassium perchlorate sample QC 1810.

FIGURE 33 - Coulter Counter number distribution for military specification potassium perchlorate sample QC 1810.

FIGURE 34 - Coulter Counter weight distribution for military specification potassium perchlorate sample QC 1810-20.

FIGURE 25 - Coulter Counter number distribution for military specification potassium perchlorate sample QC 1810-20.

FIGURE 36 - Coulter Counter weight distribution for military specification potassium perchlorate sample QC 1810-42.

FIGURE 37 - Coulter Counter number distribution for military specification potassium perchlorate sample QC 1810-42.

FIGURE 38 - Coulter Counter weight distribution for military specification potassium perchlorate sample QC 1810-R₁.

FIGURE 39 - Coulter Counter number distribution for military specification potassium perchlorate sample QC 1810-R₁.

FIGURE 40 - Coulter Counter weight distribution for military specification potassium perchlorate sample QC 1810-R₂.

FIGURE 41 - Coulter Counter number distribution for military specification potassium perchlorate sample QC 1810-R₂.

FIGURE 42- Coulter Counter weight distribution for military specification potassium perchlorate sample QC 1810-R₃.

FIGURE 43 - Coulter Counter number distribution for military specification potassium perchlorate sample QC 1810-R₃.

Distribution

External

TID - 4500, UC - 25 (238)
N. E. Brown, SLA
E. A. Kjeldgaard, SLA
T. Massis, SLA 2516 (3)
R. K. Quinn, SLA
R. K. Flitcraft, MRC

Internal

R. T. Braun
R. S. Carlson
R. J. DeSando
C. S. Friedman
L. D. Haws
C. W. Huntington
L. V. Jones
D. P. Kelly
G. E. Kettling
C. M. Love
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