Indian Head Division
Naval Surface Warfare Center
Indian Head, MD 20640-5035

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CL-20 SENSITIVITY ROUND ROBIN

Nancy C. Johnson

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FOREWORD

This work was sponsored by the Office of Naval Research Manufacturing Technology Program. This report summarizes testing conducted to determine whether the method used to prepare CL-20 has significant influence on its impact and friction sensitivity. This report is a public release version of IHTR 2339.

The author wishes to recognize the following people for their contributions: Mr. John Brough and Mrs. Karen Burrows (both of NSWC, Indian Head Division), who manage the Navy Manufacturing Technology Program and the CL-20 portion of that program, respectively; Dr. Dorothy Cichra (of Indian Head), who provided valuable assistance with sample distribution and data interpretation; and Mr. Scott Hamilton and colleagues at ATK Thiokol Propulsion Company, Dr. Peter Zarras and colleagues at NAWC/WD, China Lake, Dr. William Koppes and colleagues at Indian Head, and Mr. Mark Metzger at TACOM-ARDEC, who provided CL-20 samples for the round robin. CL-20 samples were also provided by Aerojet Corporation, Bofors, and SNPE. Special thanks go to Mr. Ted Fillman and Mr. Bill Beadle of Indian Head for sample preparation and distribution; to Dr. Stan Caulder of Indian Head for BET analyses and SEM photography; to the sensitivity test groups at Indian Head, China Lake, Thiokol, and TACOM-ARDEC; and to Mr. Devon McIntosh (Indian Head) for statistical analysis of the round robin results.

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ACRONYMS AND ABBREVIATIONS

ABL	Allegheny Ballistics Laboratory
BAM	Bundesantalt fur Material Prufung
BET	Brunauer, Emmett, and Teller (the inventors) method of surface area analysis
ERL	Explosive Research Laboratory
FTIR	Fourier transform infrared spectroscopy
GPC	Gel permeation chromatography
HPLC	High performance liquid chromatography
SEM	Scanning election microscopy
TACOM-ARDEC	U.S. Army Tank-Automotive and Armaments Command Armament Research, Development and Engineering Center, Picatinny Arsenal
TADA	Tetraacetyldiamino hexaazaisowurtzitane
TADF	Tetraacetyldiformyl hexaazaisowurtzitane
TAIW	Dibenzyltetraacetyl hexaazaisowurtzitane

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INTRODUCTION

CL-20 (hexanitrohexaazaisowurtzitane) is the highest energy conventional explosive ingredient currently available for military use. It was first prepared in 1987 by Dr. Arnold T. Nielsen at NWC (now NAWC/WD) China Lake.[1] CL-20-based compositions provide significantly greater energy and higher density than similar HMX-based compositions. CL-20-based formulations are being developed and evaluated in explosive, rocket, and gun propellant applications. More than 10,000 pounds of CL-20 have been prepared to date. The main obstacles that limit wide acceptance of CL-20 are (1) batch-to-batch variations in CL-20 crystal quality and particle size distribution at the production scale and (2) its high cost relative to HMX.

A number of studies have been conducted to determine the sensitivity of CL-20 relative to HMX and to identify the parameters that control CL-20 sensitivity.[2,3] There has been debate as to whether neat CL-20 is more like HMX or PETN in impact and friction sensitivity. Many investigators have suggested that the quality of the crystal directly affects CL-20 sensitivity. The term “crystal quality” includes factors such as single crystal versus polycrystalline habit, sharp versus rounded crystal edges, and the presence or absence of defects in the crystal. Another factor that may affect CL-20 sensitivity is the presence of impurities.

A Navy Manufacturing Technology (Mantech) Program for CL-20 began in 1997 to develop and demonstrate a reproducible manufacturing process for CL-20 on a production scale. The program’s objectives include—

- Ensuring product reproducibility and consistency from batch-to-batch
- Improving CL-20 crystal quality along with determining whether the sensitivity of CL-20 can be improved
- Lowering the manufacturing costs of CL-20.

Before CL-20 will gain wide acceptance for DoD use, the CL-20 manufacturing process must be optimized to provide affordable, reproducible, consistent material.

As part of the Navy CL-20 Mantech Program, an effort was undertaken to determine whether the sensitivity of neat CL-20 to impact and friction stimuli may be affected by changes in precursor type, crystallization method, and impurity level. CL-20 samples were prepared by precipitation, evaporative, or polymer gel crystallization techniques. Three precursor types (TAIW, TADF, and TADA) were used to prepare the CL-20 samples. A round robin series of drop-weight impact and friction sensitivity testing was conducted at four sensitivity test sites on the CL-20 samples along with two reference materials. The samples were labeled in a “blind” manner with the true identity of each individual sample coded to prevent identification by the testing facilities. Multiple test facilities were used to establish confidence in the results.

ROUND ROBIN METHODOLOGY

Table I lists the 14 CL-20 samples tested with information on the sample source, precursor type, and crystallization method. Table II lists the chemical and polymorph purity for each sample. RDX (class 1) and HMX (class 5) were used as reference standards and were obtained from production lots HOL 86D515-085 and HOL 86D200-033, respectively, manufactured by Holston Army Ammunition Plant. Round robin samples were sent to the following facilities for impact and friction testing:

- Naval Surface Warfare Center, Indian Head Division (hereafter referred to as Indian Head)
- Naval Air Warfare Center/Weapons Division, China Lake (China Lake)
- ATK Thiokol Propulsion Company (Thiokol)
- Tank Automotive Command—Armament Research and Development Center (TACOM-ARDEC), Picatinny Arsenal.

Table I. CL-20 Round Robin Samples

Sample ID	Source of sample	Precursor type[a]	CL-20 used in crystallization	Crystallization method
1696-47-1	Thiokol	TADF	PCL 74	Evaporative
1696-73	Thiokol	TADF	PCL 74	Evaporative
1697-42	Thiokol	TADF	PCL 74	Crash precipitation
1697-53	Thiokol	TADA	Information not provided	Crash precipitation
CL-1	China Lake	TADF	PCL 62	Trisolvent recrystallization
CL-2	China Lake	TADF	PCL 74	Thermoreversible gel recrystallization
CL-3	China Lake	TADF	PCL 74	Thermoreversible gel recrystallization
IHD174	Indian Head	TADF	PCL 74	Crash precipitation
IHD274	Indian Head	TADF	PCL 74	Crash precipitation
PCL 74	Thiokol	TADF	Information not provided	Crash precipitation
0174/2	Aerojet	TAIW	Information not provided	Proprietary process
X991027	TACOM-ARDEC	TADF	PCL 74	Proprietary process— from alpha hydrate
M4, 2000-01	Nexplo-Bofors	Information not provided	Information not provided	Proprietary process
99A1286	SNPE	Information not provided	Information not provided	Proprietary evaporative process

[a]Dibenzyltetraacetyl hexaazaisowurtzitane (TAIW), tetraacetyldiformyl hexaazaisowurtzitane (TADF), tetraacetyldiamino hexaazaisowurtzitane (TADA).

Table II. Purity of CL-20 Round Robin Samples

Sample ID	Chemical purity (% CL-20)[a]	Polymorph purity (% epsilon)[b]
1696-47-1	97	> 95
1696-73	97	> 95
1697-42	96	> 95
1697-53	99	> 95
CL-1	97.5	95
CL-2	95[c]	95
CL-3	96[c]	95
IHD174	97.6	> 95
IHD274	99.5	> 95
PCL 74	97	> 95
0174/2	> 99.9	> 90
X991027	> 97	> 97
M4, 2000-01	98.6	All
99A1286	99	> 95

[a]Determined by HPLC (method described in CL-20 STANAG 4566).
[b]Determined by FTIR (method described in CL-20 STANAG 4566).
[c]Based on combination of HPLC and GPC results.

Sample identities are provided in Table III. The CL-20 and reference standard samples were coded with a number and provided to each test facility to eliminate any bias during testing. Each facility's test group was instructed to handle the samples as though they were all CL-20 and was instructed to run drop-weight impact and friction sensitivity testing on the samples. The samples were sent out in three shipments because they were not all available at the same time. The CL-20 samples from Thiokol, China Lake, Indian Head, and Aerojet plus the RDX and HMX reference standards were the first set of samples sent for sensitivity testing. The second set of samples consisted of the CL-20 samples from TACOM-ARDEC and Bofors plus PCL 74 (labeled "PCL 74 R" in data tables). The third set of samples consisted of the SNPE CL-20 sample and PCL 74 (labeled "PCL 74 R2" in data tables). Sample PCL 74 was included in each set of samples distributed to gain multiple data points on the same sample.

Table III. Blind Sample Identity

Round robin sample	Blind sample No. to Indian Head for sensitivity testing	Blind sample No. to Indian Head for BET	Blind sample No. to Thiokol	Blind sample No. to China Lake	Blind sample No. to TACOM-ARDEC
1696-47-1	24	25	23	21	22
1696-73	29	30	28	26	27
1697-42	34	35	33	31	32
1697-53	59	60	58	56	57
CL-1	39	40	38	36	37
CL-2	44	45	43	41	42
CL-3	49	50	48	46	47
IHD174	64	65	63	61	62
IHD274	4	5	3	1	2
PCL 74	19	20	18	16	17
0174/2	54	55	53	51	52
RDX, Cl 1	9	10	8	6	7
HMX, Cl 5	14	15	13	11	12
PCL 74 R	79	Not retested	76	77	78
X991027	69	70	66	67	68
M4, 2000-01	74	75	71	72	73
99A1286	83	84	80	81	82
PCL 74 R2	88	Not tested	85	86	87

Samples

CL-20 samples for the round robin test series were prepared by investigators at Thiokol, China Lake, and Indian Head using different synthesis routes and crystallization techniques. The samples were prepared using the dibenzyltetraacetyl hexaazaisowurtzitane (TAIW), tetraacetyldiformyl hexaazaisowurtzitane (TADF), or tetra-acetyldiamino hexaazaisowurtzitane (TADA) precursor. Crystallization techniques used included precipitation, evaporative, or polymer gel crystallization. Many of the samples were crystallized from CL-20 lot number PCL 74 to keep impurities constant. The precursor type and crystallization method used are given in Table I. More detailed information is provided in the datasheets of Appendix A. Sample IHD274 was prepared as an ultrapure material in order to determine the effect of impurities (or the lack thereof) on sensitivity.

Additional CL-20 samples for round robin testing were supplied by Aerojet, TACOM-ARDEC, Bofors, and SNPE. The Aerojet sample was prepared approximately 12 years ago by the original CL-20 synthesis route using the TAIW precursor. That sample as well as those from TACOM-ARDEC, Bofors, and SNPE were crystallized by proprietary processes.

The supplier of each sample was asked to provide the following information for the CL-20 sample:

- Crystallization batch size
- Crystallization procedure
- Chemical and polymorph purity
- Particle size distribution
- Photomicrograph
- Impact and sliding friction sensitivity.

BET surface area was measured for each of the samples by Indian Head. Datasheets for each sample are provided in Appendix A.

Particle size and surface area data are given in Table IV for all of the CL-20 samples. For some samples, surface area and particle size do not appear to correlate. The reason for this lack of correlation is not clear.

Table IV. Particle Size Distribution and Surface Area of Samples

CL-20 ID	Particle size distribution (micron)[a]			BET surface area[b] (m^2/g)
	10%	50%	90%	
1696-47-1	70	141	217	0.0367
1696-73	71	141	221	0.0474
1697-42	85	166	232	0.0759
1697-53	62	159	222	0.0525
CL-1	15	35	73	0.2108
CL-2	51	175	400	0.1823
CL-3	100	258	467	0.1075
IHD174	30	141	317	0.1175
IHD274	23	185	311	0.0745
PCL 74	50	157	343	0.0844
0174/2	—	40	—	0.0764
X991027	—	12.5–25	—	0.1204
M4, 2000-01	93	180	307	0.1101
99A1286	12	27	50	0.2765

[a]Data provided by supplier of sample, using Microtrac or Malvern instrumentation in most cases.

[b]BET analysis performed by Indian Head.

Testing

Each test facility conducted drop-weight impact and friction sensitivity testing on the blindly labeled round robin samples. The drop-weight impact sensitivity test is performed by dropping a weight from various heights onto the test samples. Frequently, the height that is determined corresponds to a 50% probability of inducing reaction in the sample. Scatter in the data frequently occurs due to factors related to the test equipment and detection methods. The friction sensitivity test is conducted by rubbing the explosive sample between two surfaces. The results are observed as either a reaction (i.e., flash, smoke, and/or audible report) or no reaction.

Each facility provided a brief description of the tests that were run. The descriptions are provided below and summarized in Table V. The test equipment and procedures used to evaluate the sensitivity properties of the samples were not uniform from facility to facility and made the test results difficult to compare. RDX and HMX reference standards were tested along with the CL-20 samples to facilitate comparison of the results from the different test sites and to determine relative sensitivities between these commonly used nitramines and CL-20.

Table V. Sensitivity Tests Used for Round Robin

Activity	Drop-weight impact sensitivity			Friction sensitivity test		
	Test procedure[a]	Drop-weight mass (kg)	Data reported as[b]	Test procedure[a]	Sliding surfaces	Data reported as[b]
Indian Head	ERL impact	2.5	50% probability of reaction	ABL sliding friction	Steel plate, steel wheel	TIL at 8 or 3 ft/s
China Lake	ERL impact	2.5	50% probability of reaction and low fire point	ABL sliding friction	Steel plate, steel wheel	50% probability of reaction and low fire point at 8 ft/s
Thiokol	ABL impact	2	TIL and 50% probability of reaction	ABL sliding friction	Steel plate, steel wheel	TIL at specific ft/s
TACOM-ARDEC	ERL impact	2.5	50% probability of reaction	BAM[c] friction	Porcelain plate, porcelain pin	TIL

[a]ERL: Explosive Research Laboratory; ABL: Allegheny Ballistics Laboratory.

[b]TIL: Threshold initiation level.

[c]BAM: Bundesanstalt fur Material Prufung.

ERL Impact (Indian Head): The Explosive Research Laboratory (ERL) tester with type 12 tooling, garnet sandpaper, and a 2.5-kg drop-weight was used. The drop height corresponding to the 50% probability of initiation is measured for 25 samples. The sample size is 35 mg per drop. A noise meter is used to determine a positive or negative result.

ERL Impact (China Lake): The ERL tester with type 12 tooling, garnet sandpaper, and a 2.5-kg drop-weight was used. The drop height corresponding to the 50% probability of initiation is measured for 20 samples. A positive test is detected by a smudge on the sandpaper and observation of smoke, spark, odor, or sound. The low fire point (the lowest level at which a fire is obtained, approximating the level at which the sample will fire 10% of the time) can also be determined.

ABL Impact Test (Thiokol): The Allegheny Ballistics Laboratory (ABL) impact test with a 2-kg drop weight was used. The test is conducted by dropping the weight onto the sample. If a "go" response as indicated by smoke, sparks, or ignition is obtained before ten samples are tested at the 80-cm height, the drop height is decreased. If a "go" is not obtained within 10 consecutive tests, a greater than 80-cm threshold initiation level (TIL) is reported. Each time a "go" is obtained, the drop height is decreased until a level is reached where 20 consecutive tests can be conducted without a "go." Standard ABL impact drop heights are 80, 64, 51, 41, 33, 26, 21, 17, 13, 11, 6.9, 3.5, 1.8, 1.1, and 1.0 centimeters. In addition to reporting the TIL, a 50% ignition level can be calculated.

ERL Impact (TACOM-ARDEC): The ERL tester with type 12 tooling and a 2.5-kg drop-weight was used. The drop height corresponding to the 50% probability of initiation is measured using the procedure provided in MIL-STD-1751 (USAF), 20 August 82, "Impact Test (Laboratory Scale)," Method 2.

ABL Sliding Friction (Indian Head): The ABL sliding friction test consists of a steel wheel that slides over a steel plate. The sample being tested is placed at the interface between the wheel and plate, and a load is applied to compress the sample. The test is conducted by sliding the plate under the fixed wheel. Plate travel is 1.0 inch. Each time a test is conducted, the wheel is moved to a fresh wheel and plate surface. Both are ground perpendicular to the direction of travel to a surface finish of 125 microinches per inch to provide consistency. The wheel is nominally 2.0 inches in diameter and 0.125-inch thick steel and hardened to a Rockwell C 45-50 hardness. The plate is 2.25 inches wide by 6.5 inches long and hardened to a Rockwell C 58-62 hardness. The pressure between the wheel and plate is varied, using 13 levels between 980 and 30 psig, and the TIL (where 20 negatives are observed with at least one positive at the next higher level) is determined. The standard speed of the plate is 8 ft/s. Sensitive materials are often run at 3 ft/s. The sample size is 35 to 45 mg per trial. Results are reported as the gage pressure reading (pounds per square inch, gage or psig) at a particular feet per second. Since the surface area of the hydraulic piston is one square inch, the pressure on the wheel (in pounds) is the same as the gage pressure, and the units "psig" and "pounds" are interchangeable.

ABL Sliding Friction (China Lake): The test equipment is the same as described above. The speed of the plate is 8 ft/s. A positive test is detected by a smudge on the platen, observation of smoke or a spark, or odor of fire. The data represent the pounds of force at which the sample will ignite 50% of the time. A low fire value may also be determined.

ABL Sliding Friction (Thiokol): The test equipment is the same as described above. A TIL level is determined from this test using load on the sample and plate velocity as variables. The applied load is varied, using 12 levels between 800 and 25 pounds. Plate velocities start at 8 ft/s and decrease to 6, 4, 3, and 2 ft/s, if necessary. Tests continue at successively lower velocities and loads until 20 no-fires are obtained. The level at which 20 no-fires are obtained is reported as the TIL. Results are reported in pounds at a given plate velocity. A 50% ignition level can also be calculated.

BAM Friction (TACOM-ARDEC): This test uses a porcelain plate which slides against a porcelain pin. The force between the pin and the plate is varied to a maximum of 360 newtons. Approximately 30 mg of sample is placed on the porcelain plate. The porcelain pin is lowered onto the sample and a weight is placed on the arm to produce the desired load. The tester is activated and the porcelain plate is reciprocated once to and fro. The results are observed as either a reaction (i.e., flash, smoke, and/or audible report) or no reaction. If a reaction occurs in ten trials, the load is reduced until no reactions are observed in ten trials. The TIL is reported.

TEST RESULTS

Drop-weight impact sensitivity results for the round robin samples are given in Table VI. The 50% impact height was determined by Indian Head, China Lake, and TACOM-ARDEC. China Lake also determined the low fire result. Thiokol measured the threshold initiation level and calculated a 50% height. The 50% impact height results (relative to the impact height of the RDX reference material) from Indian Head, China Lake, TACOM-ARDEC, and Thiokol have been plotted in Figure 1.

The most impact-insensitive CL-20 samples according to the Indian Head test were CL-3, X991027, PCL 74 R2, IHD274, and CL-1 (with the last four samples having identical results). However, these samples were not appreciably less sensitive than the other CL-20 round robin samples since the range from least-to-most sensitive was only 8 to 13 cm. Due to the variability in the test method, those results indicated that no CL-20 round robin sample is significantly less impact sensitive than another. From the China Lake results, the impact sensitivities of CL-20 samples PCL 74 R2 and X991027 were comparable to those of the RDX and HMX reference materials. Sensitivity of the other CL-20 samples fell between 5 and 12 cm (which because of variability in the test method does not represent a significant difference in impact sensitivity). The TACOM-ARDEC results indicated that CL-20 sample 99A1286 was considerably less impact sensitive than the RDX and HMX reference materials. PCL 74 R2 and CL-1 were the least impact sensitive of the remaining round robin samples. The impact results from Thiokol showed little differentiation among the CL-20 samples and the HMX reference standard. Only the RDX sample showed considerably less sensitivity than the CL-20 samples.

Table VI. Drop-Weight Impact Sensitivity Test Results

Sample identification	Indian Head [ERL impact] 50% height (cm)	China Lake [ERL impact] 50% height (cm)	China Lake [ERL impact] low fire (cm)	TACOM-ARDEC [ERL impact] 50% height (cm)	Thiokol [ABL impact] TIL (cm)	Thiokol 50% height (cm)
1696-47-1	8	5	5	11.9	3.5	5.5
1696-73	10	9	8	14.5	3.5	4.5
1697-42	8	9	8	9.6	1.8	4.9
1697-53	10	10	10	13.6	3.5	5.3
CL-1	12	9	6	17.3	1.8	4.2
CL-2	9	11	10	10.4	3.5	7.3
CL-3	13	10	8	12.1	3.5	5.6
IHD174	9	10	8	11.9	3.5	5.8
IHD274	12	9	8	11.3	1.8	4.7
PCL 74	10	7	6	13.4	3.5	5.5
PCL 74 R	10	9	6	Not tested	3.5	4.6
PCL 74 R2	12	21	16	18.2	1.8	5.7
0174/2	9	8	6	12.5	3.5	5.0
X991027	12	15	13	13.8	1.8	4.0
M4, 2000-01	10	8	8	12.4	3.5	4.3
99A1286	10	12	10	39.0	1.8	5.0
RDX, Cl 1	21	14	16	23.8	17.0	51.0
HMX, Cl 5	20	21	20	20.9	3.5	13.2

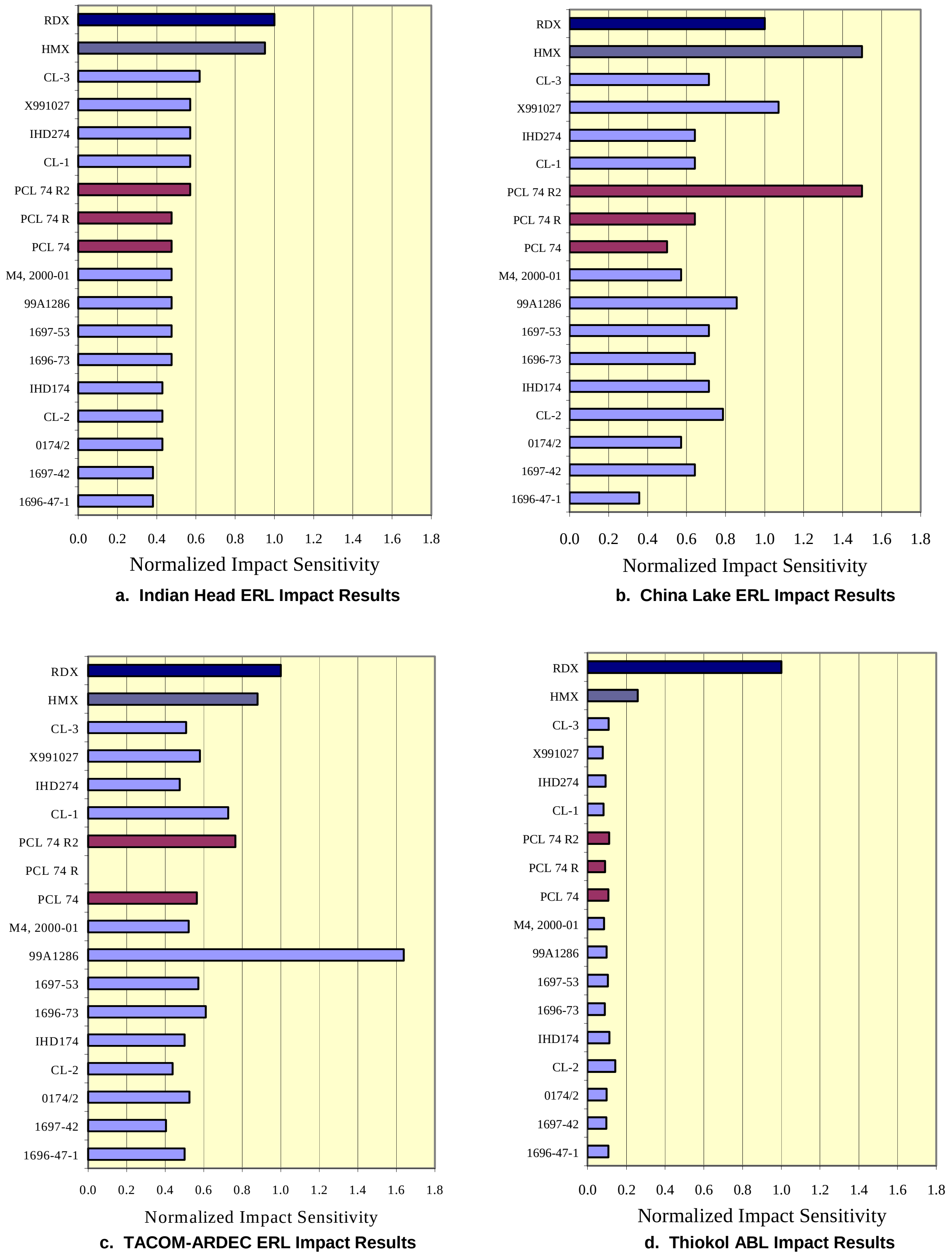


Figure 1. Normalized Impact Sensitivity (Sample/RDX) of Round Robin Samples

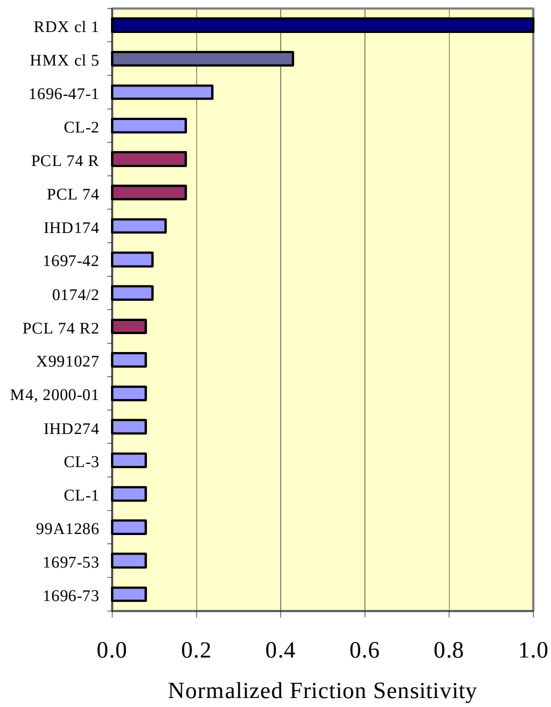
Impact sensitivity testing for two CL-20 samples, IHD274 and 1696-47-1, was re-run by Indian Head to look at the reproducibility of the test results. In the round robin testing, the impact sensitivity of samples IHD274 and 1696-47-1 was measured as 8 and 12 cm, respectively. On retesting the samples, results of 8 and 11 cm, respectively, were obtained.

Friction sensitivity results for the round robin samples are given in Table VII. The ABL sliding friction test was run by Indian Head, China Lake, and Thiokol. Indian Head and China Lake tested the samples at a plate velocity of 8 ft/s. Thiokol varied the rate. TACOM-ARDEC used the BAM friction test to determine a TIL. The friction results from Indian Head, China Lake, and TACOM-ARDEC have been plotted in Figure 2. The least friction-sensitive CL-20 sample according to the Indian Head ABL friction test results was 1696-47-1, followed by PCL 74 R, PCL 74, and CL-2 (with the last three having identical results). There was no appreciable differentiation among the remaining CL-20 round robin samples. The China Lake results indicated that samples X991027 and M4 2000-01 were the least friction sensitive. TACOM-ARDEC BAM friction results indicated that CL-3 was comparable to the RDX and HMX reference materials in friction sensitivity, followed by CL-1 and CL-2. Thiokol friction sensitivity results have been ranked from least-to-most friction sensitive in Table VIII. The Thiokol results indicated that samples 99A1286 and M4 2000-01 were the least friction-sensitive CL-20 round robin samples and comparable in sensitivity to the RDX and HMX reference standards.

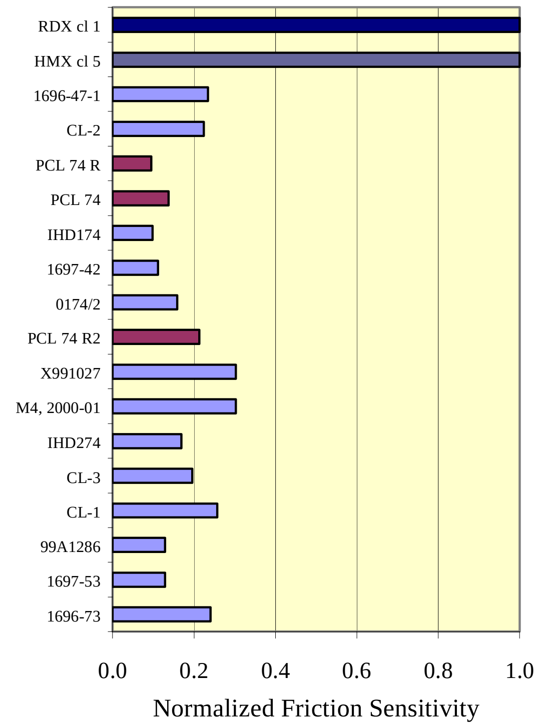
Table VII. Friction Sensitivity Test Results

Sample identification	Indian Head [ABL friction] TIL at 8 ft/s (lb)	China Lake [ABL friction] 50% point at 8 ft/s (lb)	China Lake [ABL friction] low fire at 8 ft/s (lb)	TACOM-ARDEC [BAM friction] TIL (kg)	Thiokol [ABL friction] TIL (lb)	Thiokol 50% point at 3 ft/s (lb)
1696-47-1	75	155	126	11.2	25 at 3 ft/s	371
1696-73	< 30	159	159	12.8	25 at 3 ft/s	165
1697-42	30	74	63	12.8	180 at 2 ft/s	188
1697-53	< 30	85	50	16	25 at 4 ft/s	205
CL-1	< 30	170	159	21.6	130 at 2 ft/s	195
CL-2	55	148	126	19.2	25 at 4 ft/s	260
CL-3	< 30	129	126	25.2	50 at 4 ft/s	185
IHD174	40	65	50	12.8	50 at 3 ft/s	170
IHD274	< 30	112	63	16.8	50 at 4 ft/s	210
PCL 74	55	91	63	14.4	50 at 2 ft/s	252
PCL 74 R	55	63	98	Not tested	50 at 6 ft/s	255
PCL 74 R2	< 30	141	126	6.4	50 at 4 ft/s	Not determined
0174/2	30	105	79	16	25 at 4 ft/s	163
X991027	< 30	200	159	12	25 at 6 ft/s	185
M4, 2000-01	< 30	200	200	12	25 at 8 ft/s	195
99A1286	< 30	85	50	9.6	50 at 8 ft/s	Not determined
RDX, Cl 1	315	661	631	28.8	25 at 8 ft/s	__[a]
HMX, Cl 5	135	661	631	24	25 at 8 ft/s	__[a]

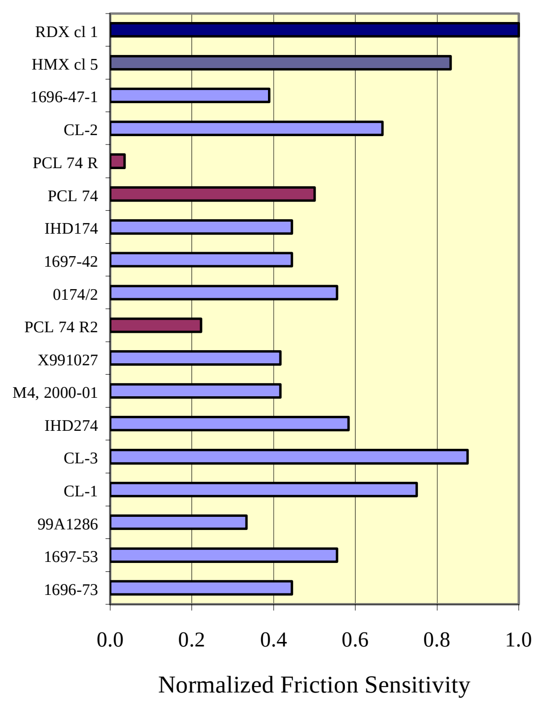
[a]RDX and HMX were not friction-sensitive enough at 3 ft/s to obtain a probability curve; therefore, a 50% point could not be calculated.



a. Indian Head ABL Friction Results (TIL)



b. China Lake ABL Friction Results (50% Point)



c. TACOM-ARDEC BAM Friction Results (TIL)

Figure 2. Normalized Friction Sensitivity (Sample/RDX) of Round Robin Samples

Table VIII. Thiokol Friction Sensitivity Results Ranked

Sample ID	Thiokol [ABL friction] TIL (lb)
99A1286	50 at 8 ft/s (least sensitive)
M4, 2000-01	25 at 8 ft/s
RDX cl 1	25 at 8 ft/s
HMX cl 5	25 at 8 ft/s
PCL 74 R	50 at 6 ft/s
X991027	25 at 6 ft/s
CL-3	50 at 4 ft/s
IHD274	50 at 4 ft/s
PCL 74 R2	50 at 4 ft/s
1697-53	25 at 4 ft/s
CL-2	25 at 4 ft/s
0174/2	25 at 4 ft/s
IHD174	50 at 3 ft/s
1696-47-1	25 at 3 ft/s
1696-73	25 at 3 ft/s
1697-42	180 at 2 ft/s
CL-1	130 at 2 ft/s
PCL 74	50 at 2 ft/s (most sensitive)

Friction sensitivity testing was re-run by Indian Head on the least sensitive (as determined by Indian Head testing) and one of the most sensitive samples, 1696-47-1 and IHD274, respectively, to look at the reproducibility of the test results. In the round robin testing, the friction sensitivity of samples 1696-47-1 and IHD274 was measured as 75 and less than 30 lb, respectively. On retesting, results of less than 30 lb were obtained for each sample.

Relative trends in sensitivity ranking from lab to lab were not consistent, making it difficult to make correlations and to draw conclusions.

As another comparison of sensitivity results, the impact and friction data for each CL-20 sample provided by the supplier of the material were compared with the round robin test results from the sample supplier when the sample was tested as a blind sample. Those results are included in Table IX. With the exception of the Thiokol friction results, the test results did not change significantly when the sample was tested blind or when it was tested with the identity known.

Table IX. Comparison of Source Versus Sensitivity Test Results

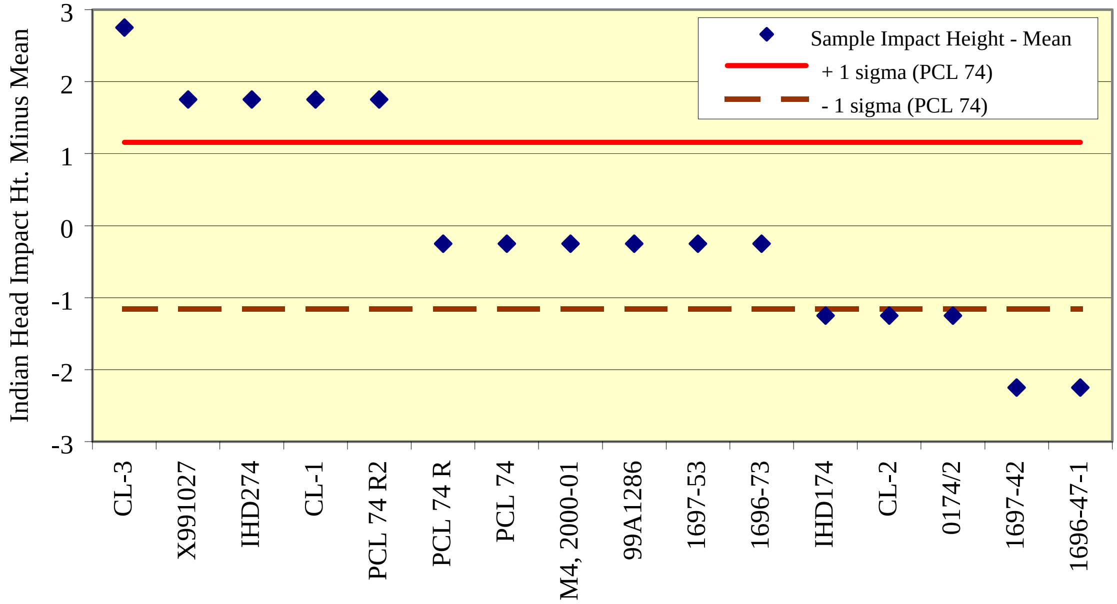
Sample identification	Source data		Test data	
	Impact (cm)	Friction	Impact (cm)	Friction
1696-47-1	1.8	50 lb at 8 ft/s	3.5	25 lb at 3 ft/s
1696-73	3.5	25 lb at 4 ft/s	3.5	25 lb at 3 ft/s
1697-42	3.5	50 lb at 4 ft/s	1.8	180 lb at 2 ft/s
1697-53	1.8	25 lb at 6 ft/s	3.5	25 lb at 4 ft/s
CL-1	10	107 lb	9	170 lb
CL-2	9	129 lb	11	148 lb
CL-3	7	174 lb	10	129 lb
IHD174	11	40 lb	9	40 lb
IHD274	11	< 30 lb	12	< 30 cm
PCL 74	1.8	50 lb at 8 ft/s	3.5	50 lb at 2 ft/s
X991027	16.6	16.8 kg	13.8	12 kg

A rough estimate of the standard deviation (sigma) of each test facility's measurement precision for the impact test was calculated from the results for sample PCL 74, which was tested three times during the course of the round robin. The standard deviation for all of the measurements at a given test facility for a particular test was assumed to be the same as that for the PCL 74 samples. Since measurements that are within one-sigma of each other are not distinctly different, plots were generated with a one-sigma band indicating which samples are distinctly more or less sensitive than the average. The plots for impact sensitivity, with the mean of the CL-20 results subtracted (i.e., adjusted to zero), are shown in Figures 3 and 4 for Indian Head and China Lake results and for TACOM-ARDEC and Thiokol results, respectively. The data points above the one-sigma band represent the less impact sensitive samples. Results from the Indian Head impact test indicated that CL-20 samples CL-3, X991027, IHD274, CL-1, and PCL 74 R2 were distinctly less impact sensitive than the average. The China Lake impact test results have a wide one-sigma band. Only sample PCL 74 R2 was distinctly less impact sensitive than the average. In the TACOM-ARDEC test, sample 99A1286 was clearly less impact sensitive than other CL-20 samples. Sample CL-2 was the less impact sensitive sample in the Thiokol test. The only CL-20 sample that was shown to be distinctly less impact sensitive than the average in more than one facility's test was PCL 74 R2. However, that same material tested as samples PCL 74 and PCL 74 R did not fall outside of the one-sigma band. Impact testing has not differentiated among these CL-20 samples prepared by different synthesis and crystallization methods.

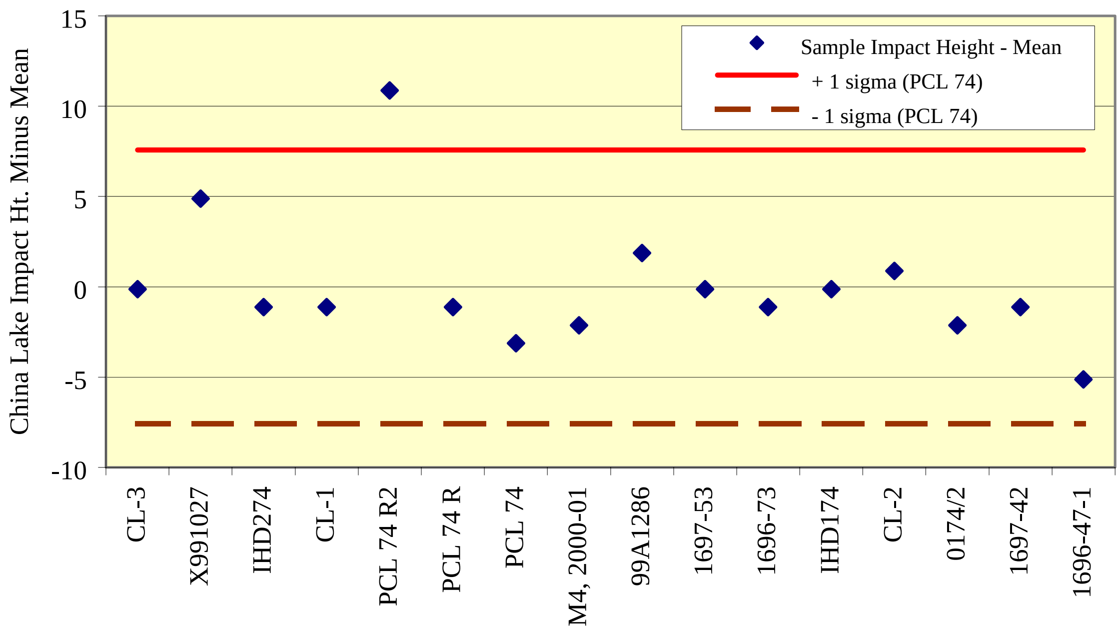
Similar to the data treatment used for the impact tests, a rough estimate of the standard deviation of each test facility's measurement precision for the friction test was calculated from the results for sample PCL 74. The standard deviation for all of the friction measurements at a given test facility was assumed to be the same as that for the PCL 74 samples. Plots were generated with a one-sigma band indicating which CL-20 samples are distinctly more or less sensitive than the average. The plots for friction sensitivity, with the mean of the CL-20 results subtracted (i.e., adjusted to zero), are shown in Figures 5 and 6 for Indian Head and China Lake results and for TACOM-ARDEC and Thiokol results, respectively. The data points above the one-sigma band represent the less friction sensitive samples. Since the Thiokol friction test results are reported at various plate velocities, power was calculated by multiplying the applied load by the plate velocity to compare results for the round robin samples.

Results from Indian Head indicated that samples 1696-74-1, CL-2, PCL 74 R, and PCL 74 were distinctly less friction sensitive than the average. Based on the China Lake results, samples X991027, M4, 2000-01, and CL-1 were distinctly less friction sensitive than the average. According to the Thiokol friction results, CL-20 samples 99A1286, 1697-42, and PCL 74 R were distinctly less friction sensitive. The BAM friction tester was used at TACOM-ARDEC. Based on the BAM results, samples CL-3 and CL-1 were distinctly less friction sensitive than the average. (Samples CL-2 and CL-3 contained residual gel from the crystallization process; the gel may have desensitized those CL-20 samples.) Only CL-20 samples PCL 74 R and CL-1 were shown to be distinctly less friction sensitive than the average in two facilities tests. In a third facility's test, PCL 74 R fell below the one-sigma band, indicating that it is distinctly more friction sensitive than the average. Friction testing has not clearly differentiated among the CL-20 samples.

Correlation plots were generated to determine the extent of measurement correlation between test facilities. Results obtained at one facility were plotted against results obtained for the same materials at another facility. A correlation plot of two perfectly correlated sets of data would yield points that fall on a straight line with a positive slope. Correlation plots for the Indian Head versus the other test facilities' data for impact and friction testing are shown in Figures 7 and 8, respectively. The limit lines that bound the best-fit trend line represent an approximation of the one-sigma measurement uncertainty. In general, the one-sigma band is so large in these plots that it is difficult to determine if any correlation exists.

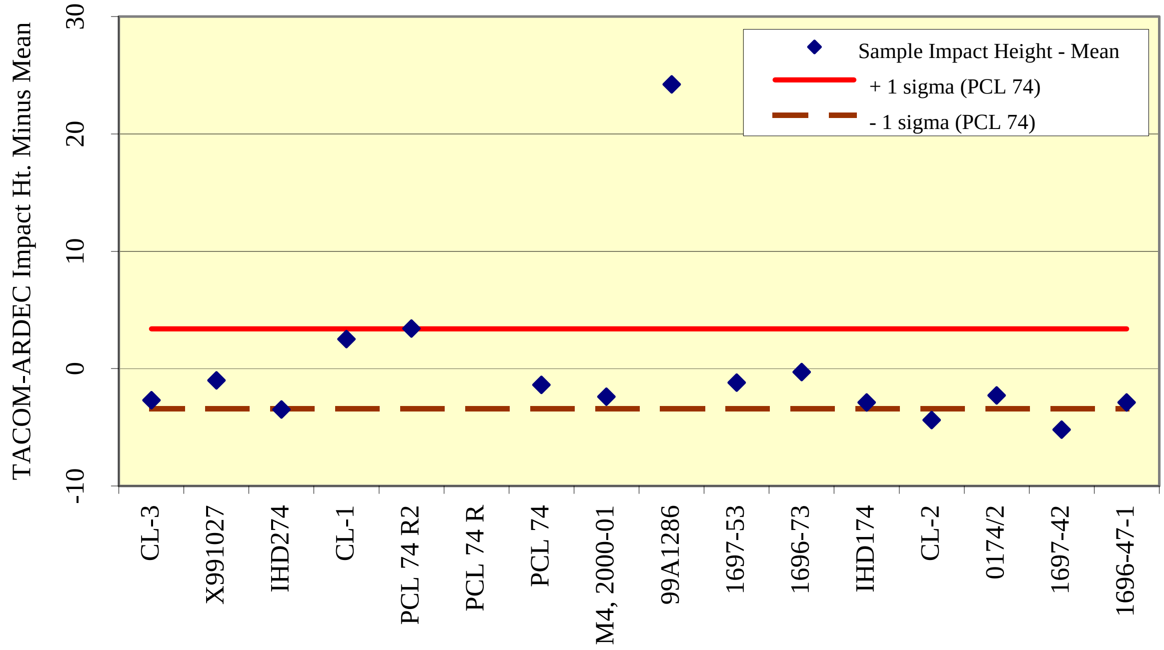


a. Indian Head Results

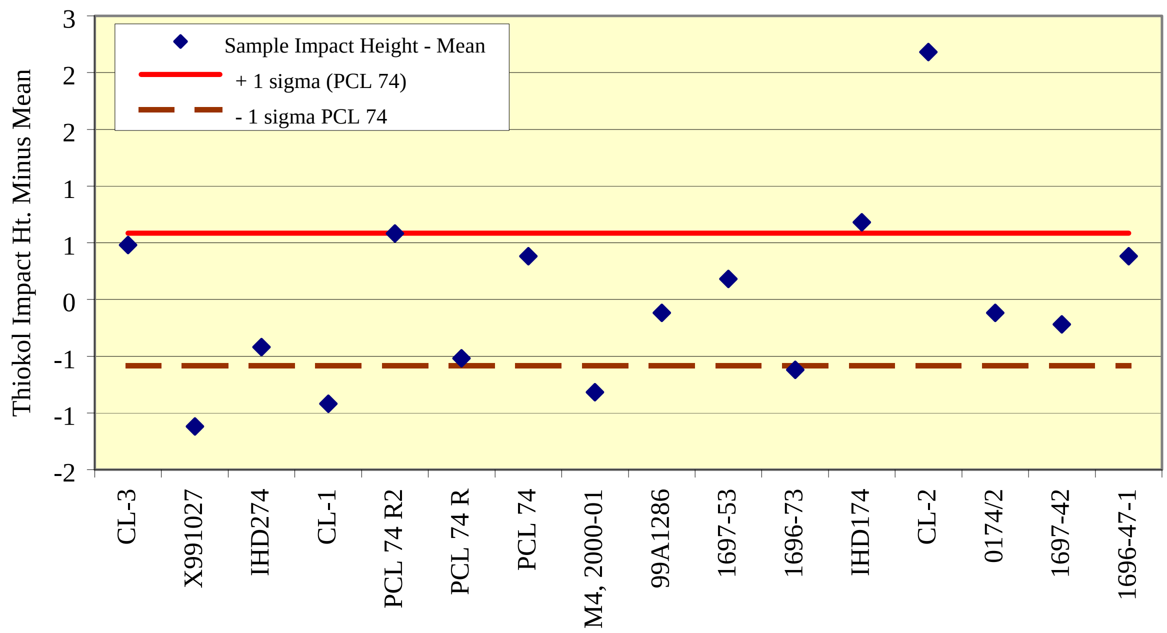


b. China Lake Results

Figure 3. Analyses of Indian Head and China Lake Impact Results for Round Robin Samples

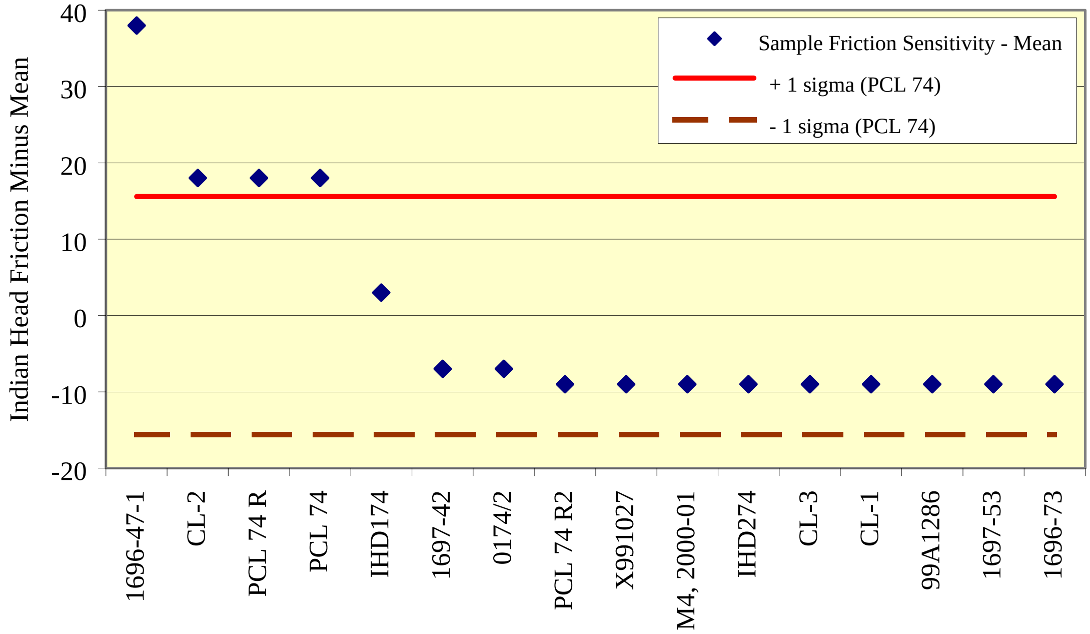


a. TACOM-ARDEC Results

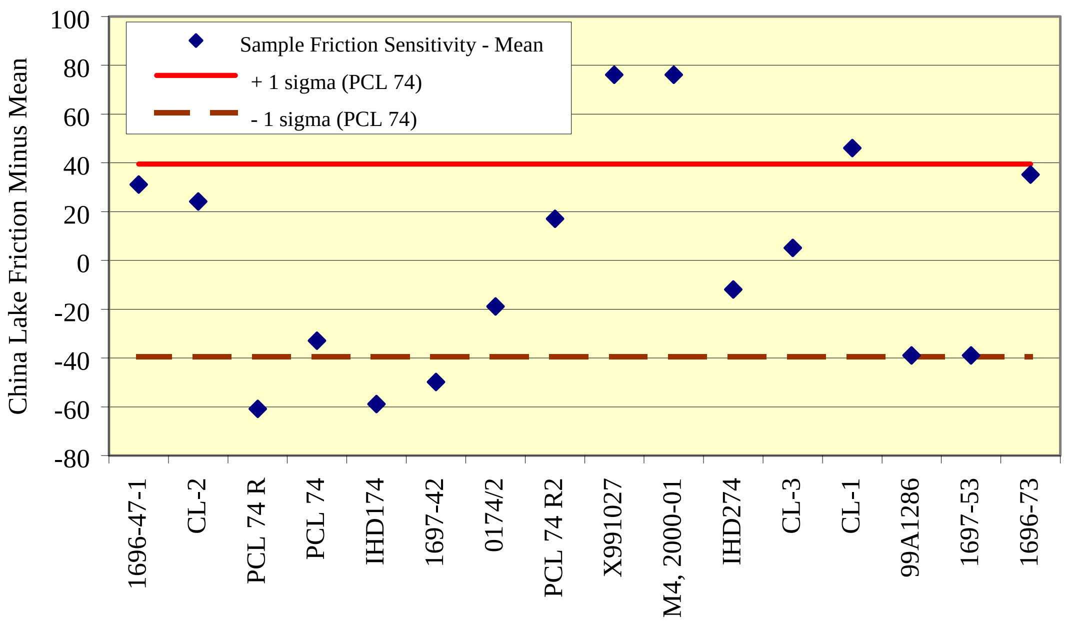


b. Thiokol Results

Figure 4. Analyses of TACOM-ARDEC and Thiokol Impact Results for Round Robin Samples

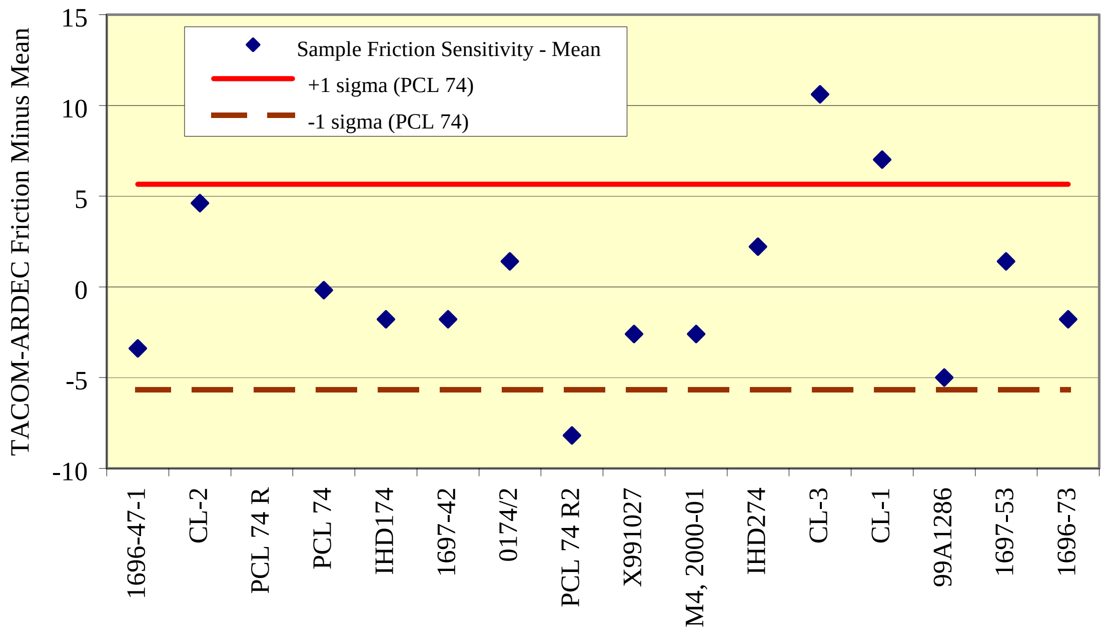


a. Indian Head Results

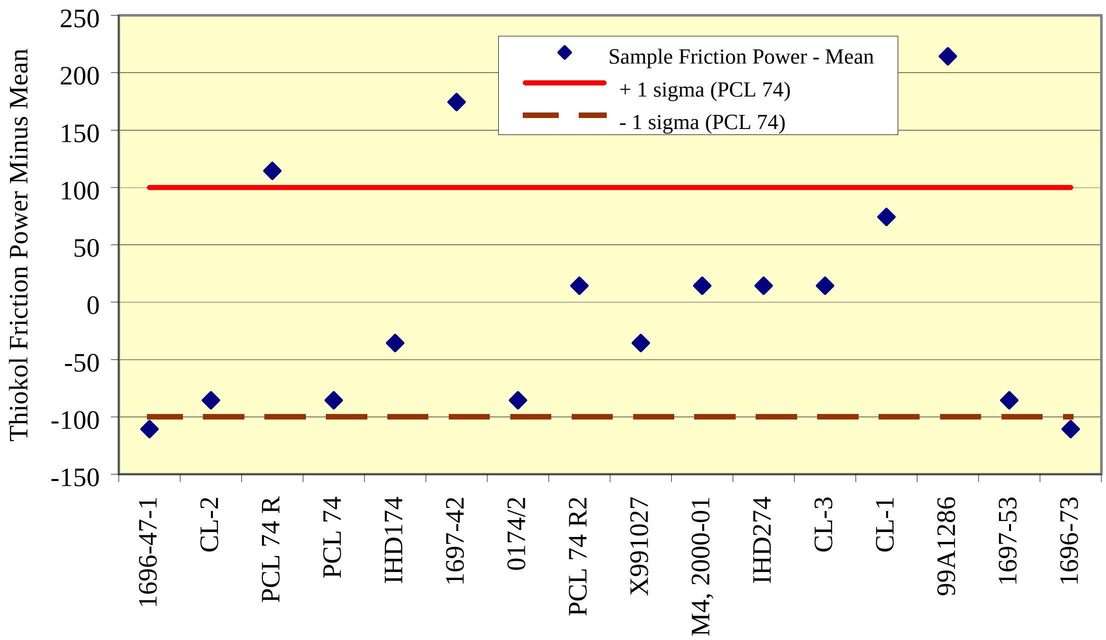


b. China Lake Results

Figure 5. Analyses of Indian Head and China Lake Friction Results for Round Robin Samples

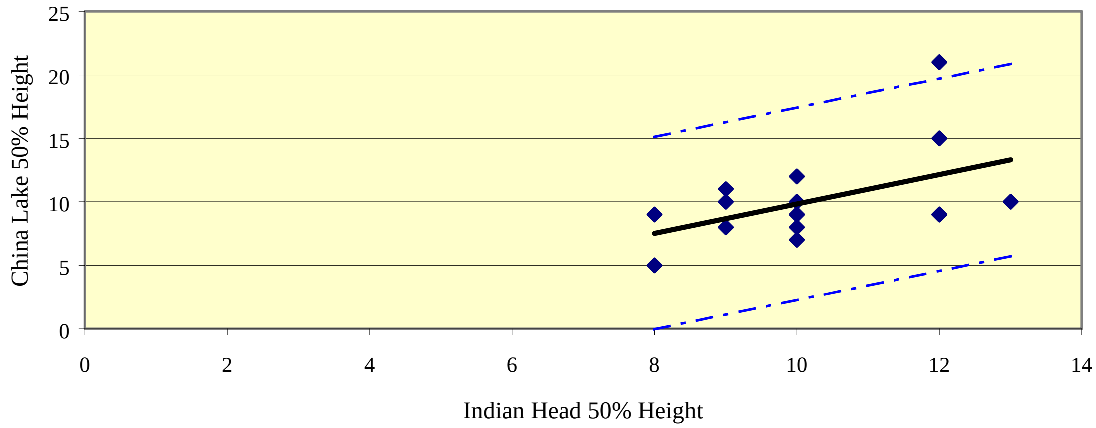


a. TACOM-ARDEC Results

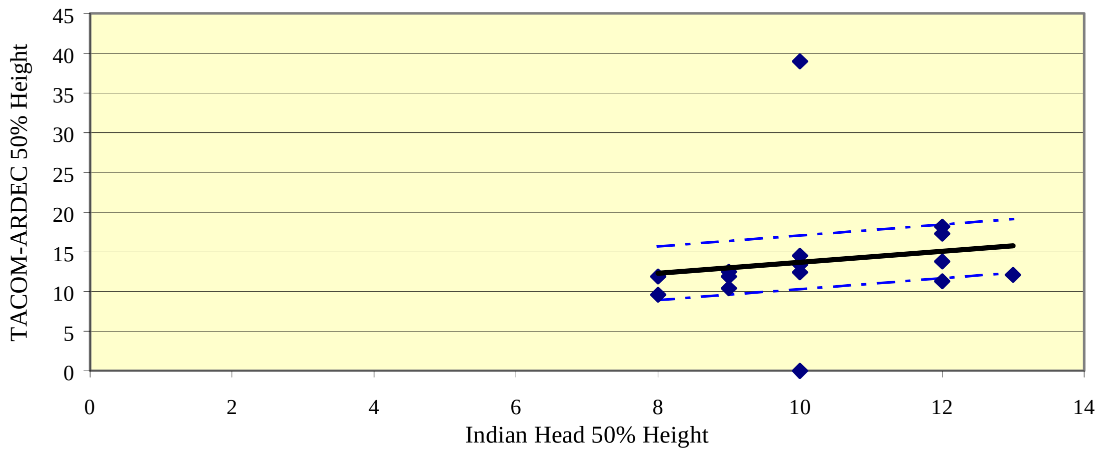


b. Thiokol Results

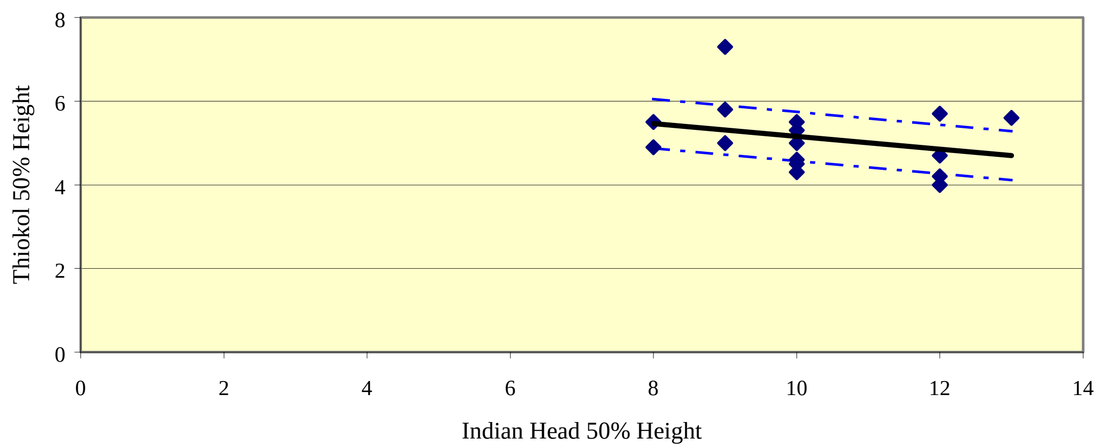
Figure 6. Analyses of TACOM-ARDEC and Thiokol Friction Results for Round Robin Samples



a. Indian Head Versus China Lake Results

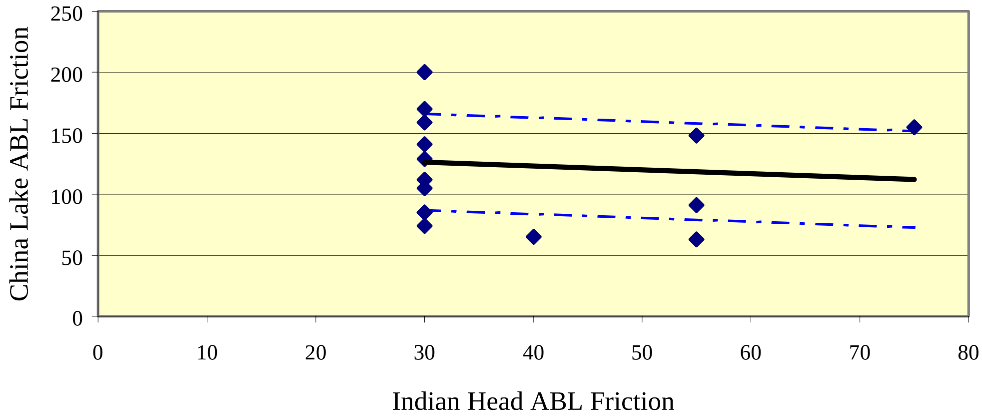


b. Indian Head Versus TACOM-ARDEC Results

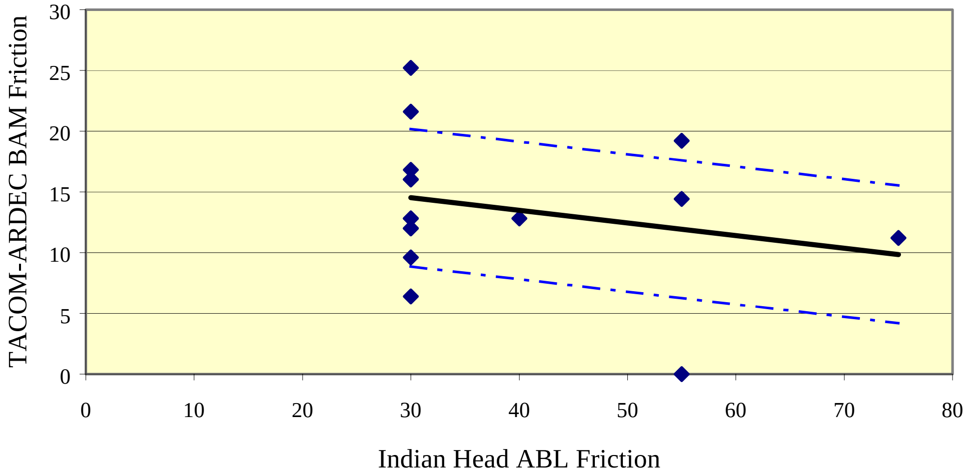


c. Indian Head Versus Thiokol Results

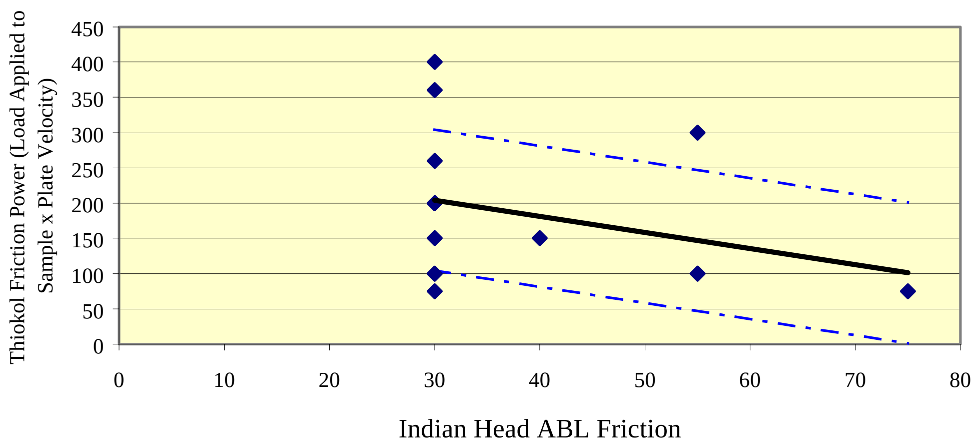
Figure 7. Correlation Plots of Impact Sensitivity with Trendline and 1-Sigma Measurement Error Tolerance Limits



a. Indian Head Versus China Lake Results



b. Indian Head Versus TACOM-ARDEC Results



c. Indian Head Versus Thiokol Results

Figure 8. Correlation Plots of Friction Sensitivity with Trendline and 1-Sigma Measurement Error Tolerance Limits

Friction sensitivity versus impact sensitivity is plotted in Figure 9 for the Indian Head, China Lake, and TACOM-ARDEC round robin data along with data for PETN, a sensitive explosive material. The CL-20 results are clustered near the PETN data; results for the RDX and HMX reference materials are clearly separated from the CL-20 data.

To determine whether CL-20 purity and particle size influenced the impact sensitivity of the round robin samples, these properties were plotted against the normalized impact sensitivity results from Indian Head and TACOM-ARDEC in Figure 10. Similar plots were made for friction sensitivity and are shown in Figure 11. Neither purity nor average particle size seemed to have an effect on the sensitivity of the round robin samples measured by Indian Head. Sample 99A1286, which was one of the higher purity samples and finer particle size materials, did test as significantly less impact sensitive than other CL-20 samples on the TACOM-ARDEC impact test.

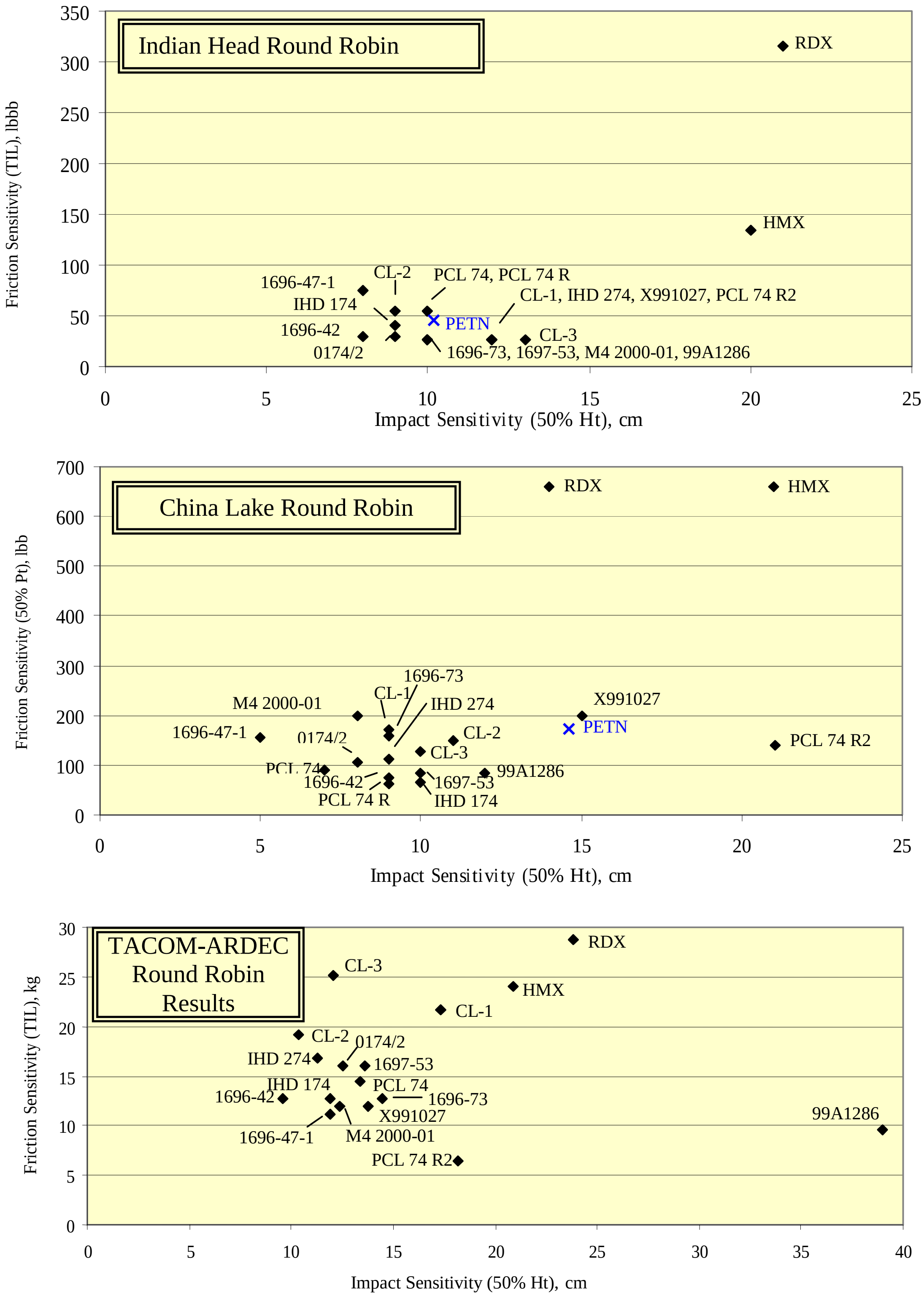
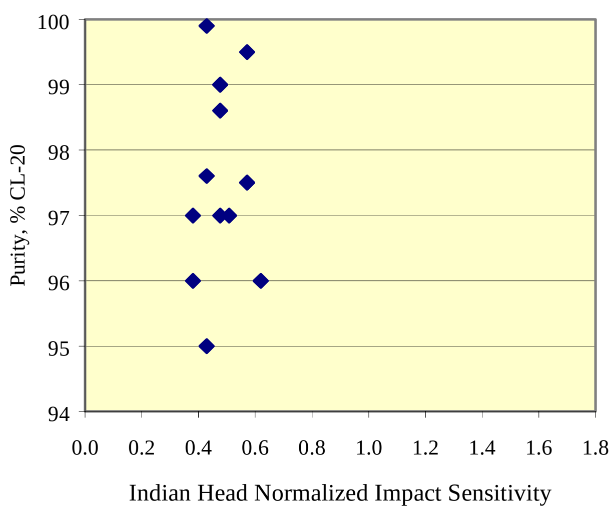
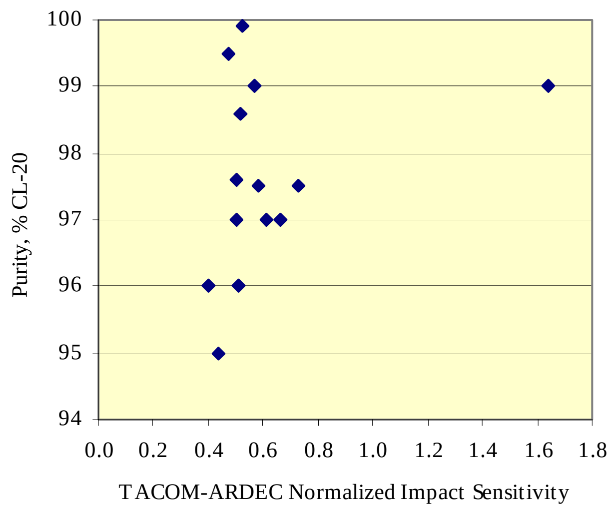


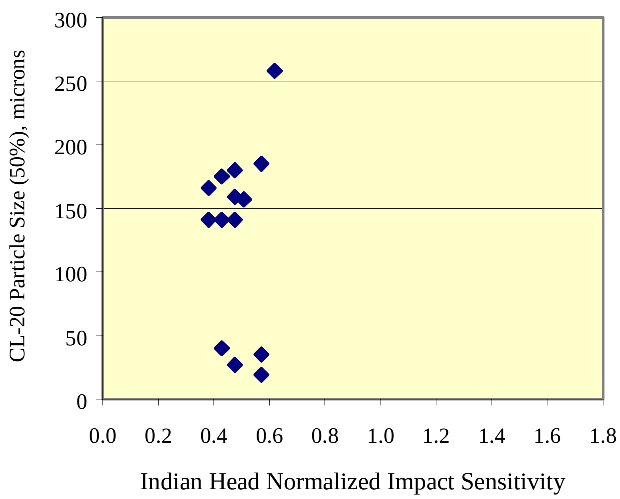
Figure 9. Friction Versus Impact Sensitivity



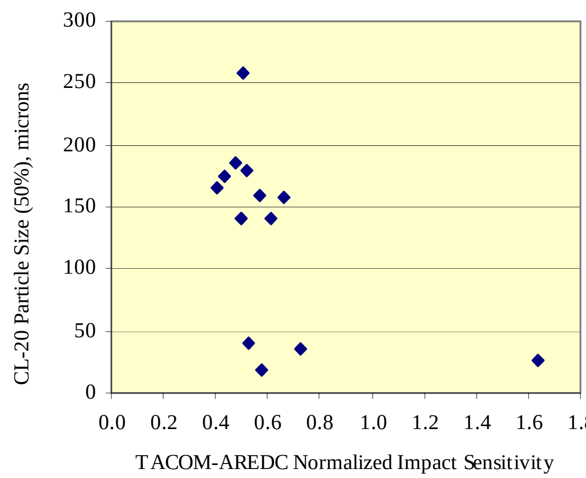
a. Purity-Indian Head



b. Purity-TACOM-ARDEC

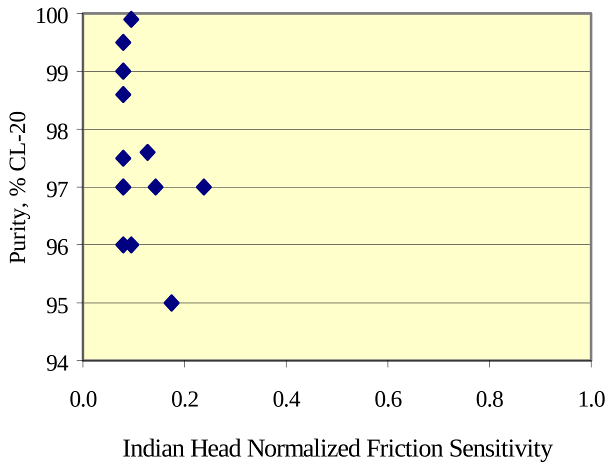


c. Particle Size-Indian Head

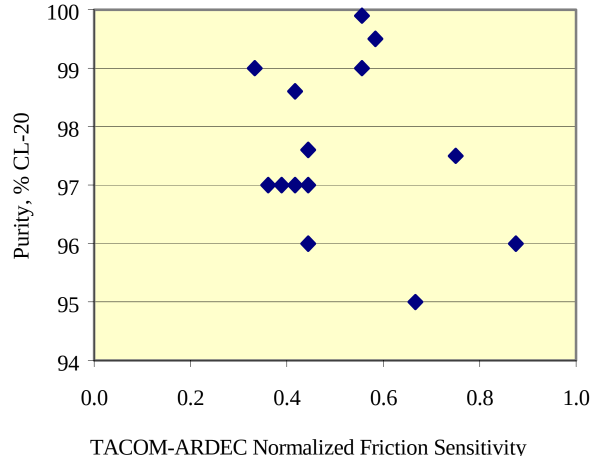


d. Particle Size-TACOM-ARDEC

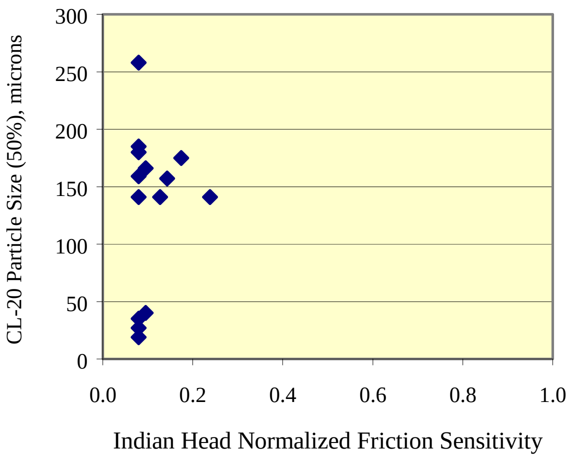
Figure 10. Effect of Purity and Particle Size on Impact Sensitivity of CL-20 Round Robin Samples



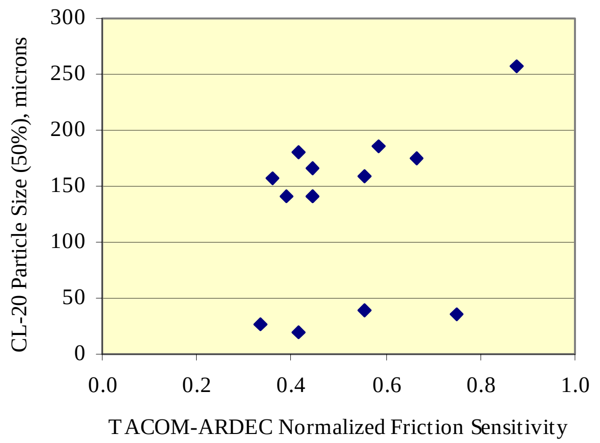
a. Purity-Indian Head



b. Purity-TACOM-ARDEC



c. Particle Size-Indian Head



d. Particle Size-TACOM-ARDEC

Figure 11. Effect of Purity and Particle Size on Friction Sensitivity of CL-20 Round Robin Samples

CONCLUSIONS

A round robin test series of impact and friction sensitivity was conducted on 14 samples of CL-20 prepared by different methods and having different crystal characteristics. The results indicate that the small-scale impact and friction sensitivity tests did not differentiate among these CL-20 samples prepared by different CL-20 synthesis routes or crystallization techniques and having different levels of impurities or single crystal versus polycrystalline habit. The round robin results indicate that the sensitivity of CL-20 is comparable to PETN and that CL-20 is more impact and friction sensitive than RDX and HMX.

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1. Nielsen, A. T., *Polynitropolyaza Caged Explosives – Part 7*, NWC TP 7020, November 89, Naval Weapons Center, China Lake, CA.

2. "Safety and Hazards of CL-20 and Propellants and Explosives Based on CL-20", CPIA Publication 668, November 97.

3. S. Nicolich, M. Mezger, D. Geiss, E. Heider, "CL-20 Round Robin Sensitivity Testing Interim Report," *1998 NDIA Insensitive Munitions & Energetic Materials Technology Symposium Proceedings*, November 98.

Appendix A

CL-20 SAMPLE AND REFERENCE MATERIAL DATASHEETS

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1. Lot/Sample ID Number: **1696-47-1**
   Source of Sample: Thiokol

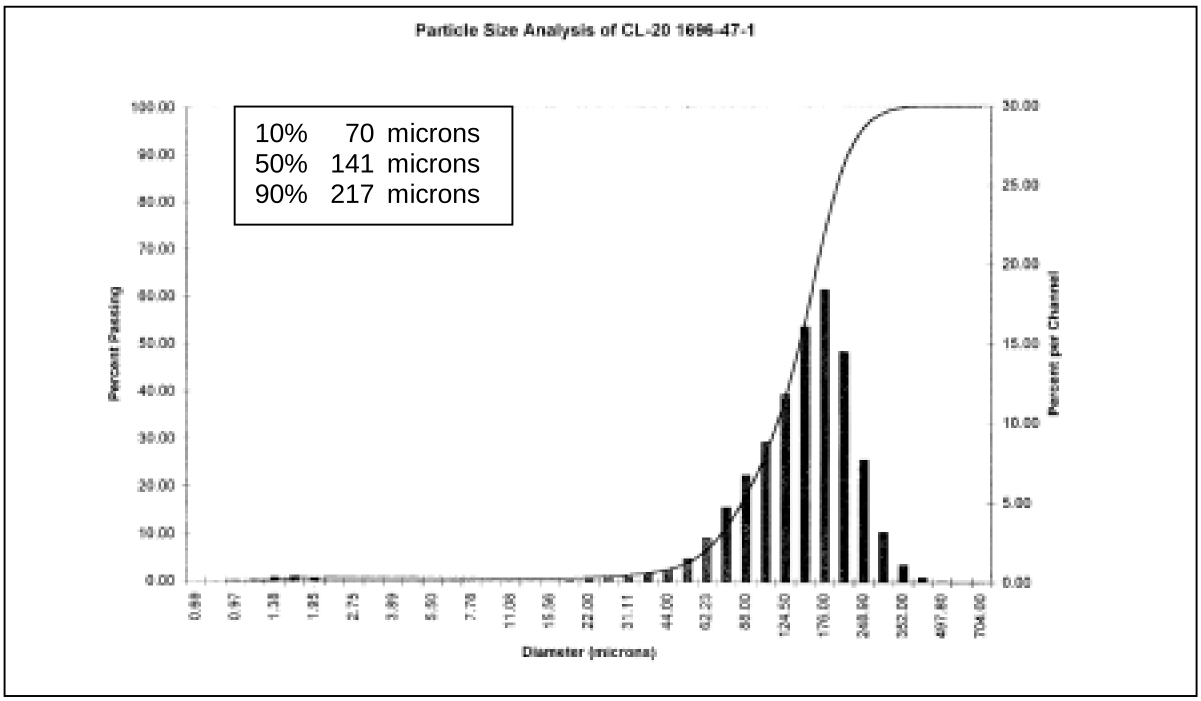
2 Batch Size: 120 grams

3. Number of Batches Used for Lot: one

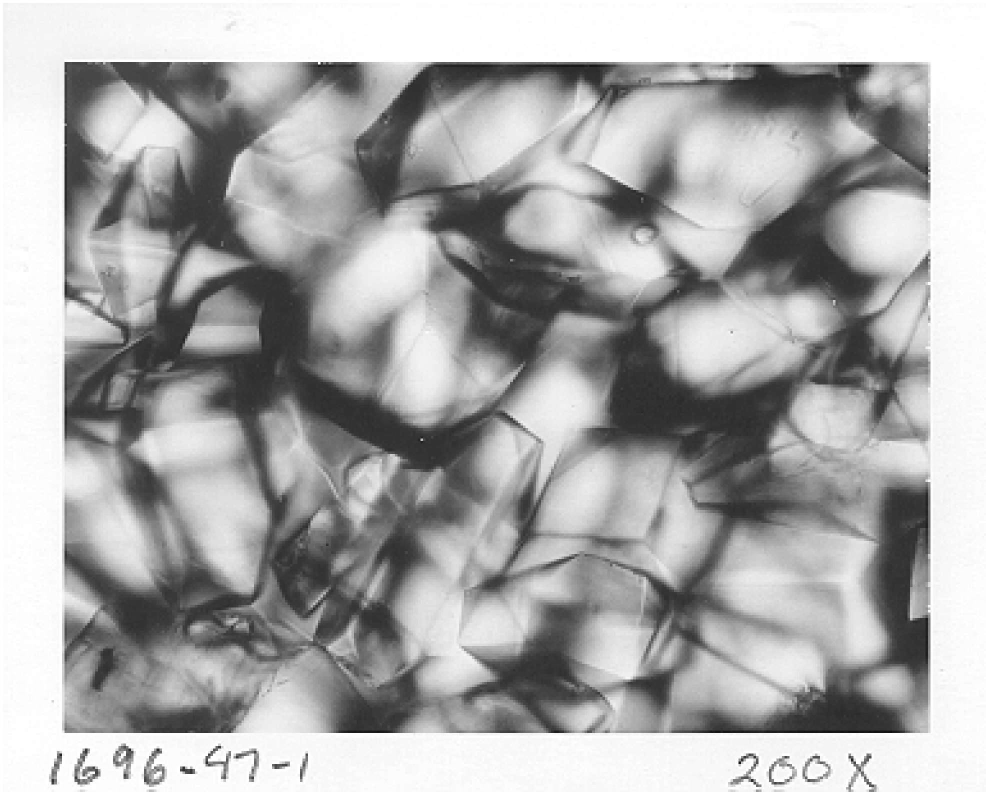
4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: 97% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   Mean Particle Size: 141 microns
   Equipment Used: Microtrac



5. Optical Photomicrograph:



6. Impact Sensitivity (ABL Impact, 2 kg weight): 1.8 cm (HMX reference: 3.5 or 6.9 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): 50 lb (HMX reference, 50-100 lb)

1. Lot/Sample ID Number: **1696-73**
   Source of Sample: Thiokol

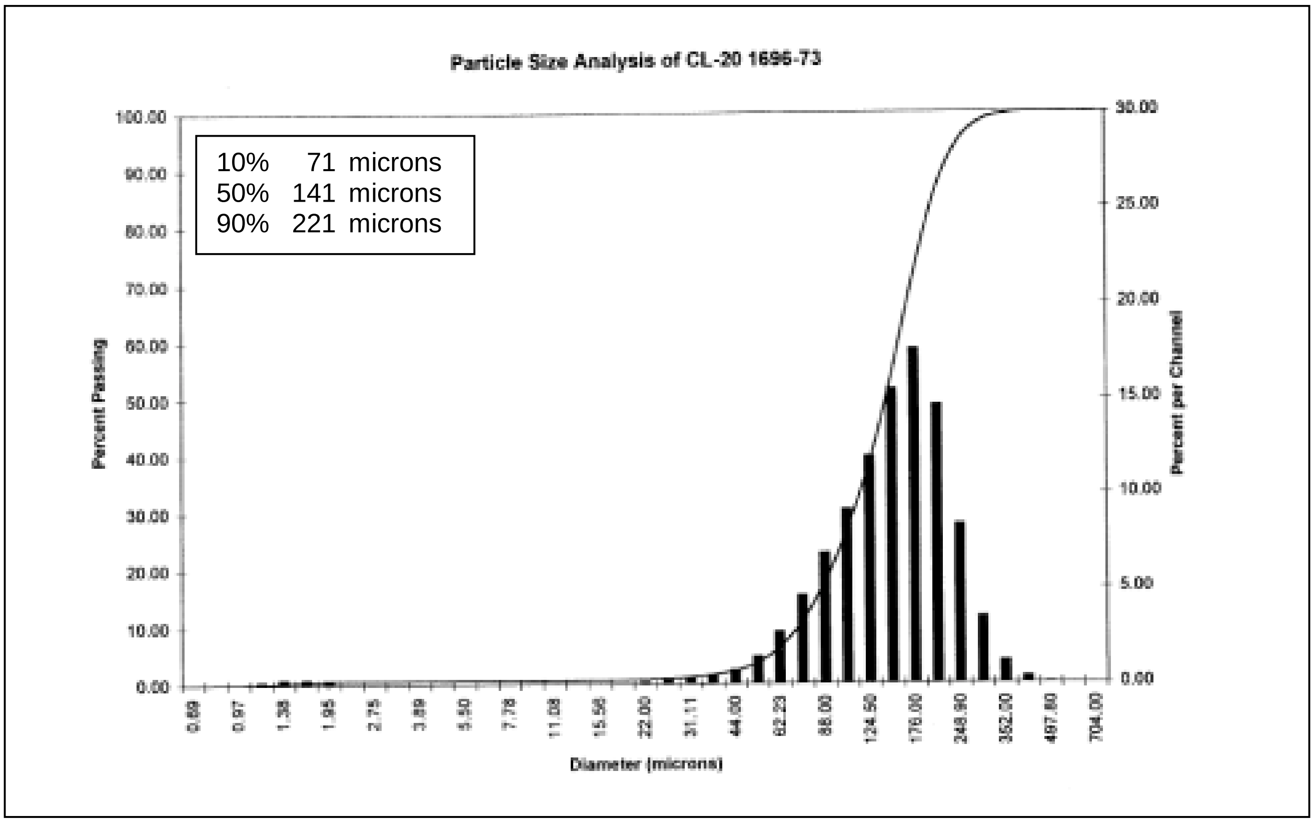
2 Batch Size: 100 grams

3. Number of Batches Used for Lot: one

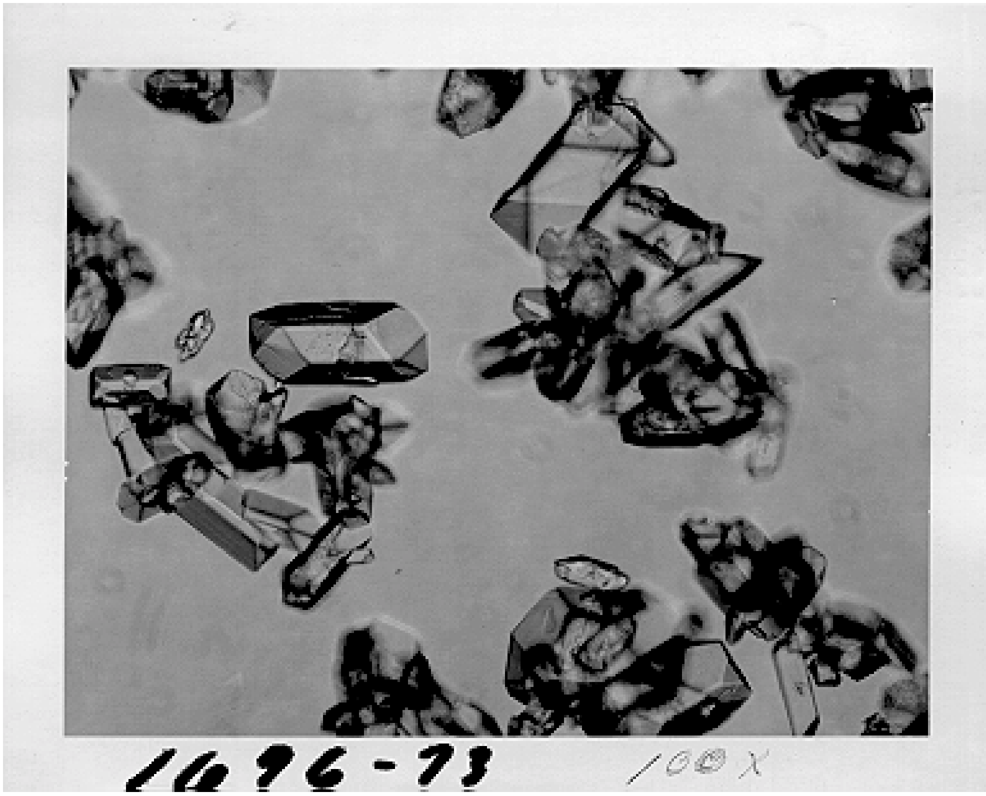
4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: 97% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   - Mean Particle Size: 141 microns
   - Equipment Used: Microtrac



5. Photomicrograph:



6. Impact Sensitivity (ABL Impact, 2 kg weight): 3.5 cm (HMX reference: 3.5 or 6.9 cm)

7. Friction Sensitivity (ABL Friction): 25 lb at 4 ft/sec (HMX reference, 50-100 lb at 8 ft/sec)

1. Lot/Sample ID Number: **1697-42**
   Source of Sample: Thiokol

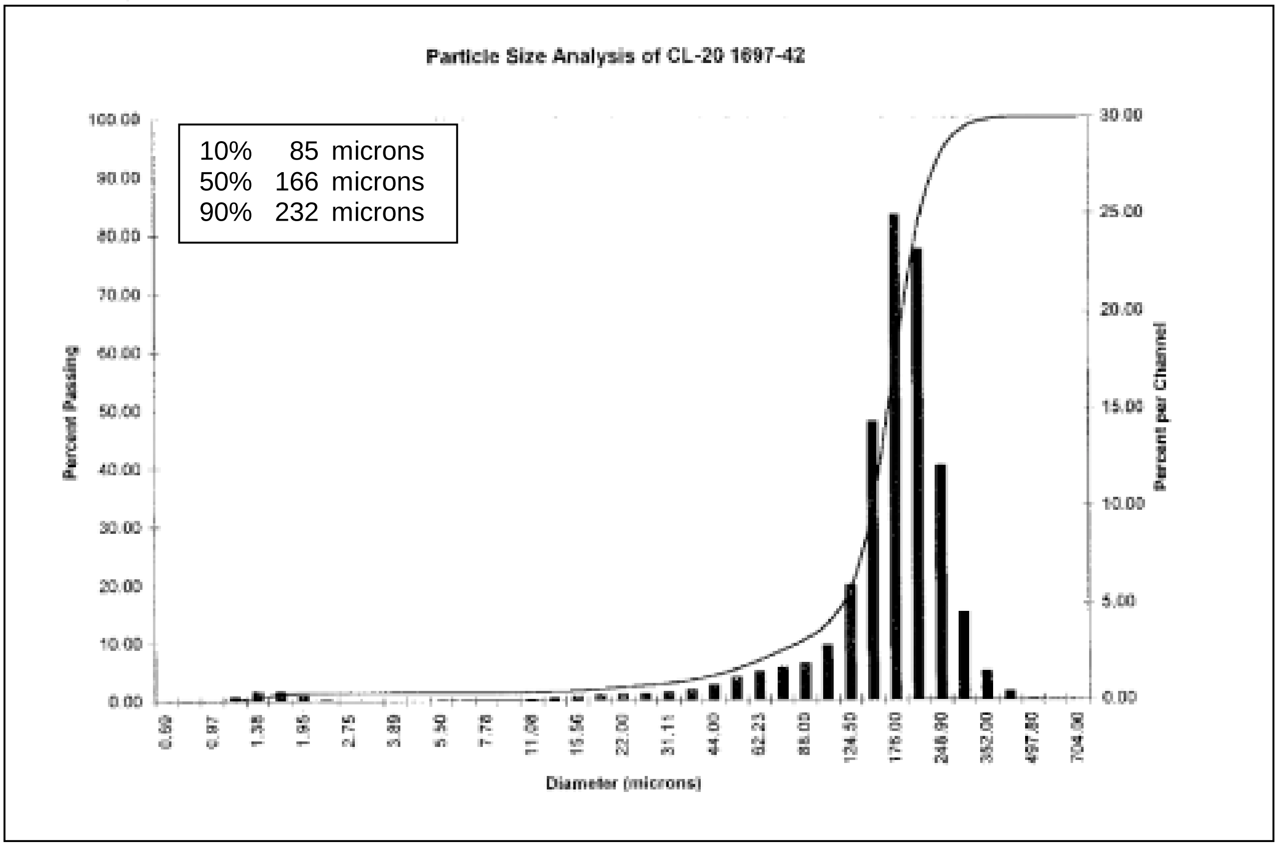
2 Batch Size: 100 grams

3. Number of Batches Used for Lot: one

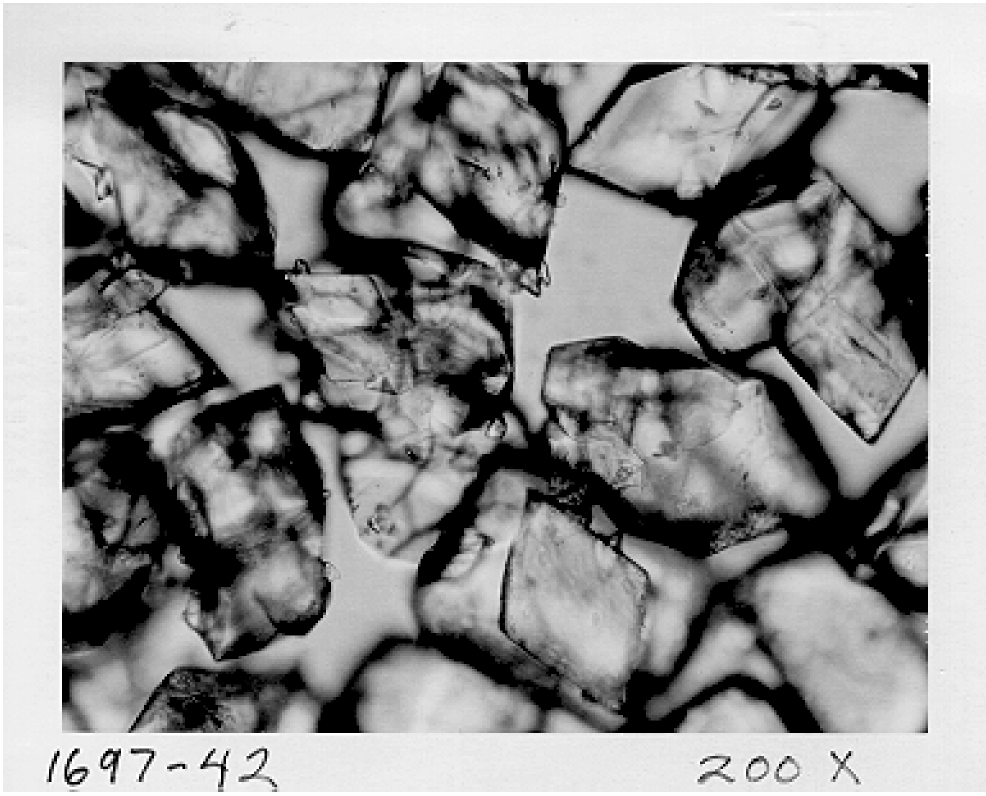
4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: 96% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   Mean Particle Size: 166 microns
   Equipment Used: Microtrac



5. Optical Photomicrograph:



6. Impact Sensitivity (ABL Impact, 2 kg weight): 3.5 cm (HMX reference: 3.5 or 6.9 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): 50 lb at 4 ft/sec (HMX, 50-100 lb)

1. Lot/Sample ID Number: **1697-53**
   Source of Sample: Thiokol

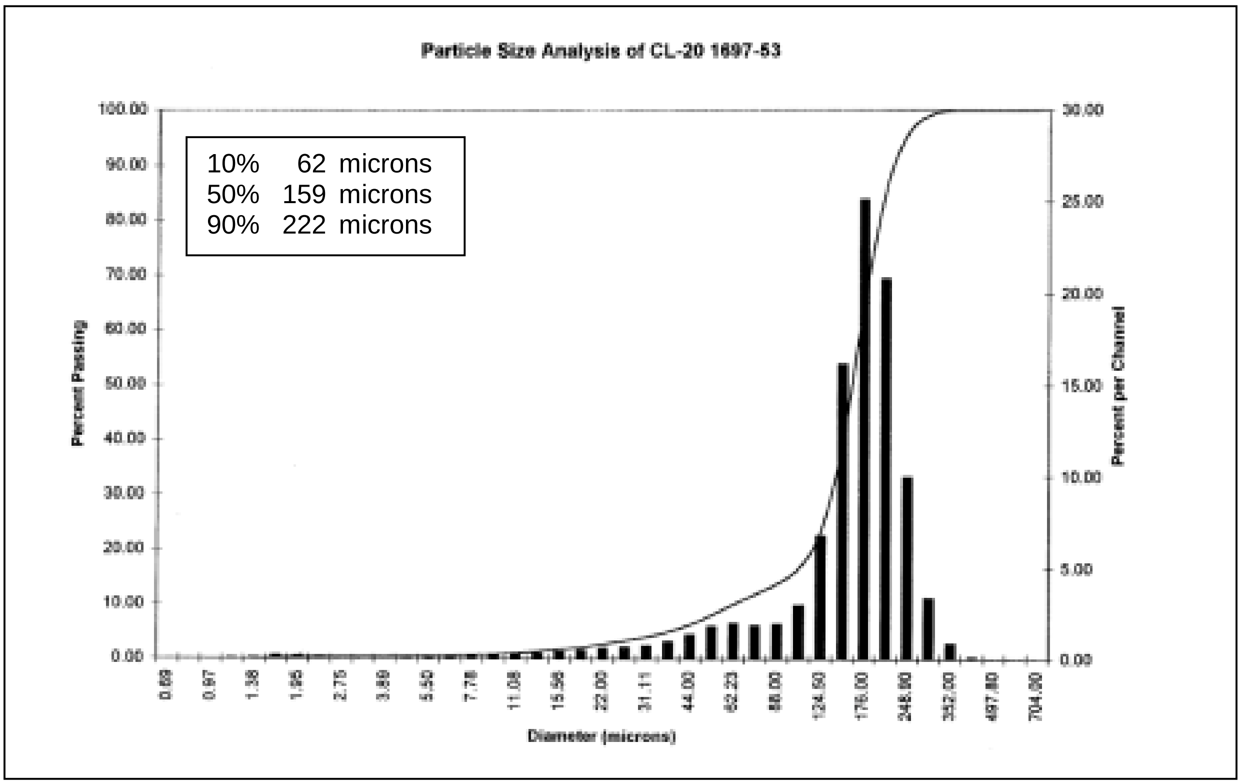
2 Batch Size: 75 grams

3. Number of Batches Used for Lot: one

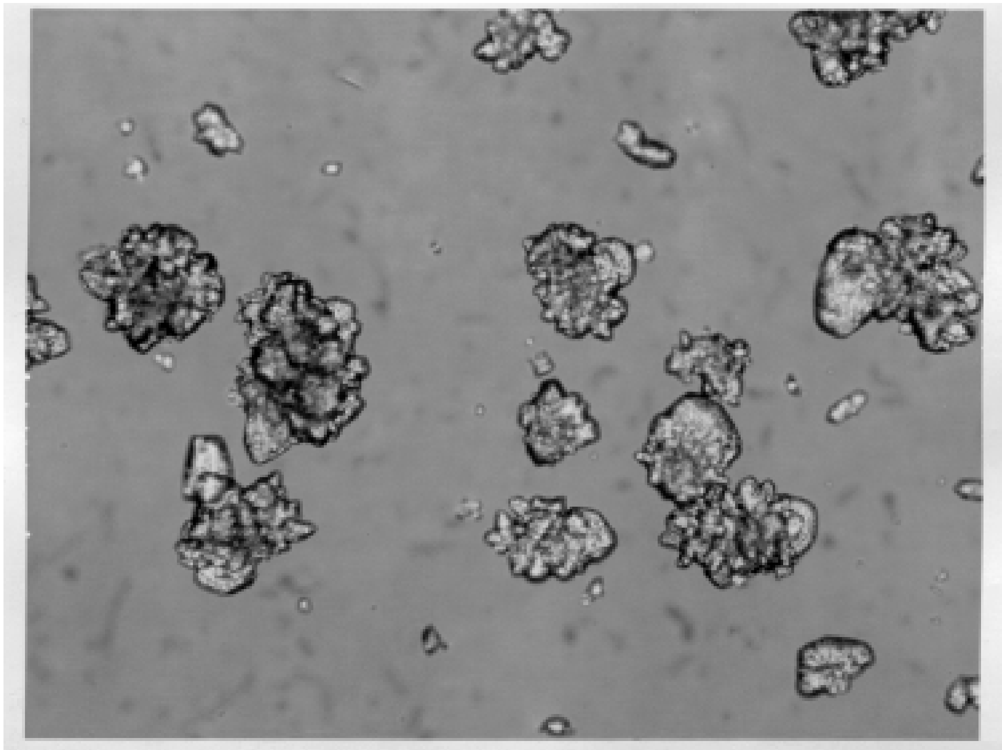
4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: 99% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   Mean Particle Size: 159 microns
   Equipment Used: Microtrac



5. Optical Photomicrograph:



6. Impact Sensitivity (ABL Impact, 2 kg weight): 1.8 cm (HMX reference: 3.5 or 6.9 cm)

7. Friction Sensitivity (ABL Friction): 25 lb at 6 ft/sec (HMX reference, 50-100 lb at 8 ft/sec)

1. Lot/Sample ID Number: **CL-1** (BZ63099)
   Source of Sample: China Lake

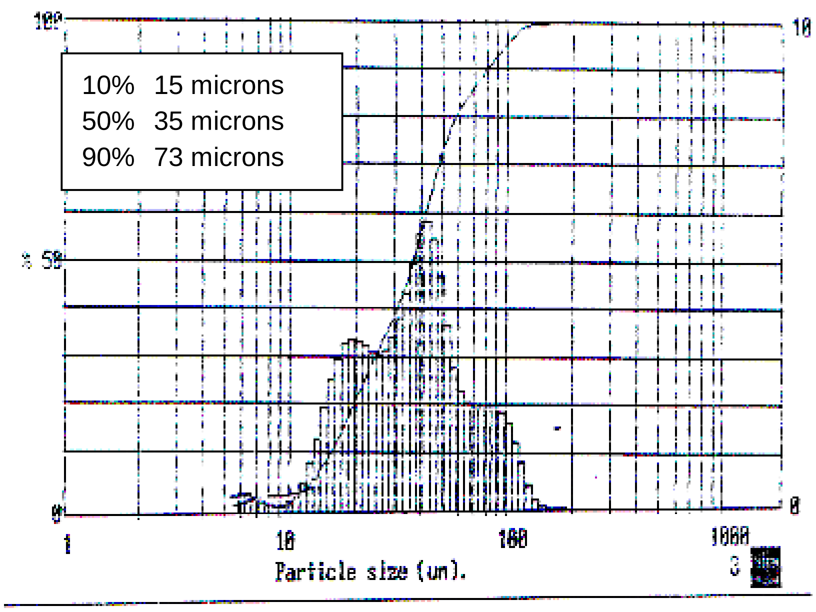
2. Batch Size: 400 grams

3. Number of Batches Used for Lot: one

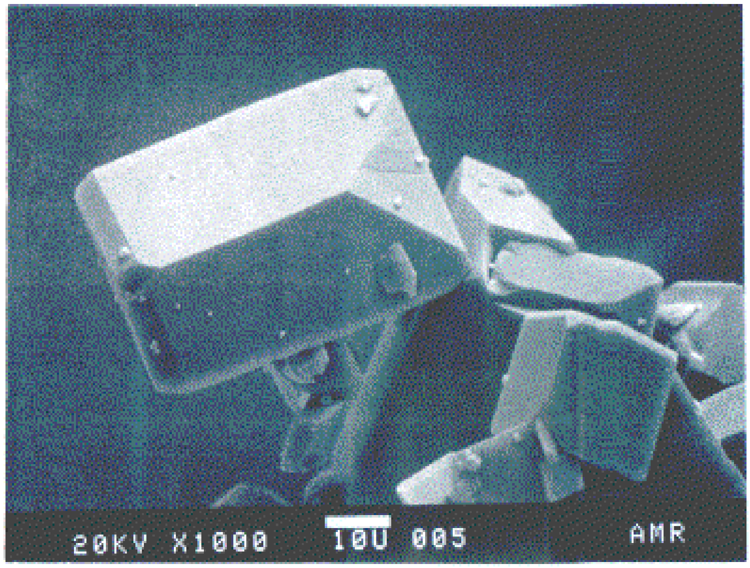
4. STANAG TESTING (test methods described in STANAG 4566):

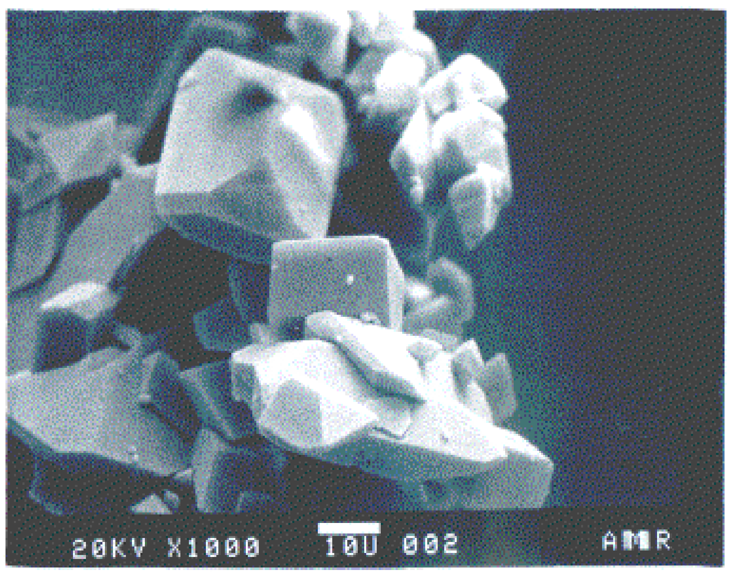
   Chemical Purity: 97.5% Polymorph Purity: 95% epsilon

   Particle Size Distribution:
   Mean particle size: 35 microns
   Equipment Used: Malvern



5. SEM Photomicrographs:





6. Impact Sensitivity (ERL Impact, 2.5 kg weight): 10 cm (RDX reference, 17 cm)

7. Friction Sensitivity (ABL Sliding Friction at 8 ft/sec): 107 lb (RDX reference, 447 lb)

1. Lot/Sample ID Number: **CL-2 (GXDC012500)**
   Source of Sample: China Lake

2 Batch Size: approximately 25 grams

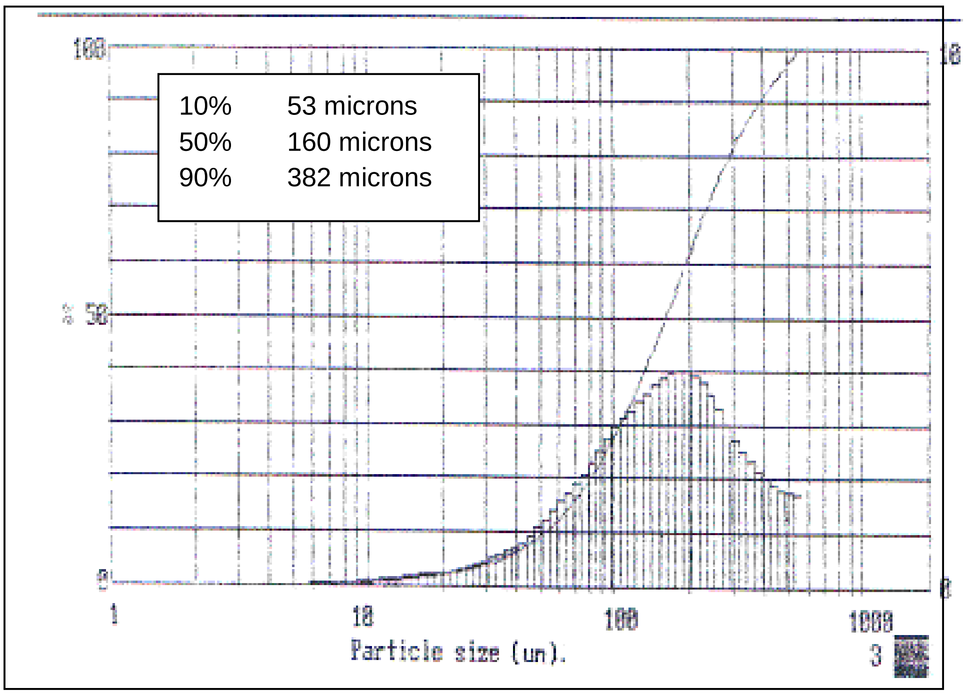
3. Number of Batches Used for Lot: four

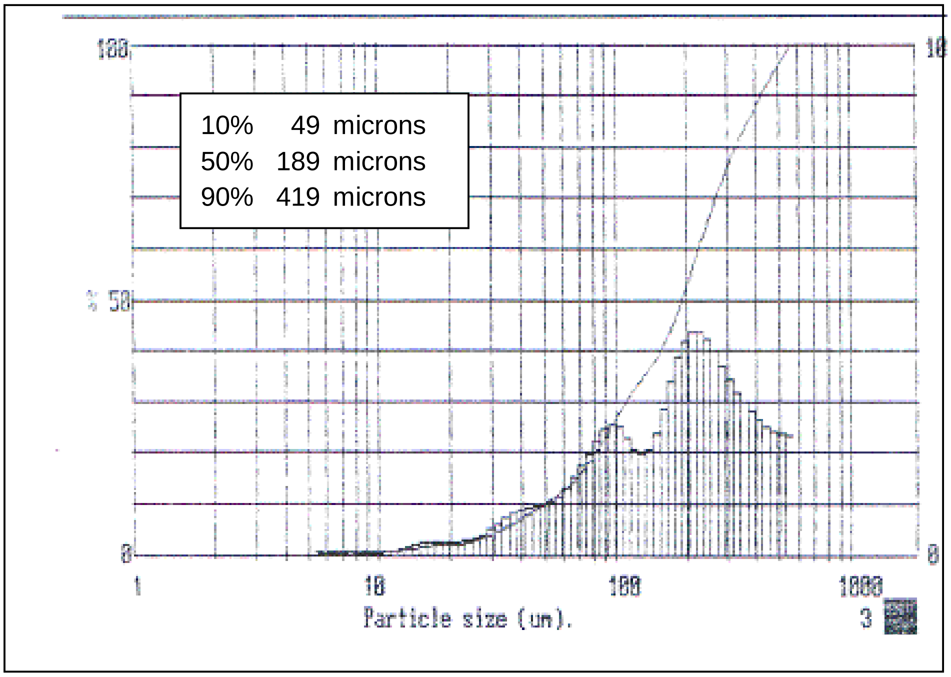
4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: 95% (using combination of HPLC and GPC results)

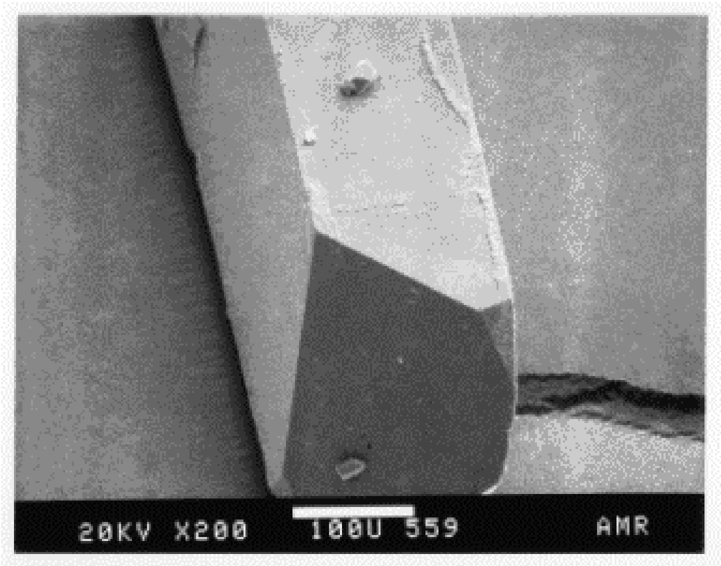
   Polymorph Purity: 95% epsilon

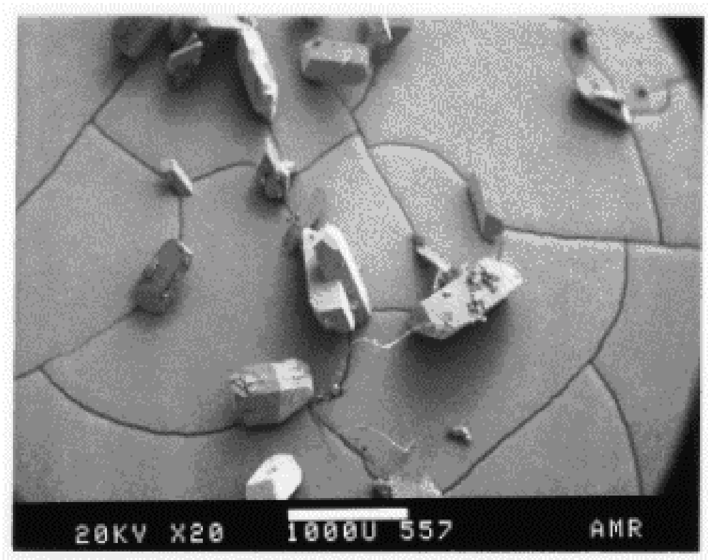
   Particle Size Distribution:
   Mean Particle Size: 175 microns
   Equipment Used: Malvern





5. SEM Photomicrographs:





6. Impact Sensitivity (ERL Impact, 2.5 kg weight): 9 cm (RDX reference, 17 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): 129 lb (RDX reference, 447 lb)

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1. Lot/Sample ID Number: **CL-3 (PZ1578-57)**
   Source of Sample: China Lake

2. Batch Size: approximately 30 grams

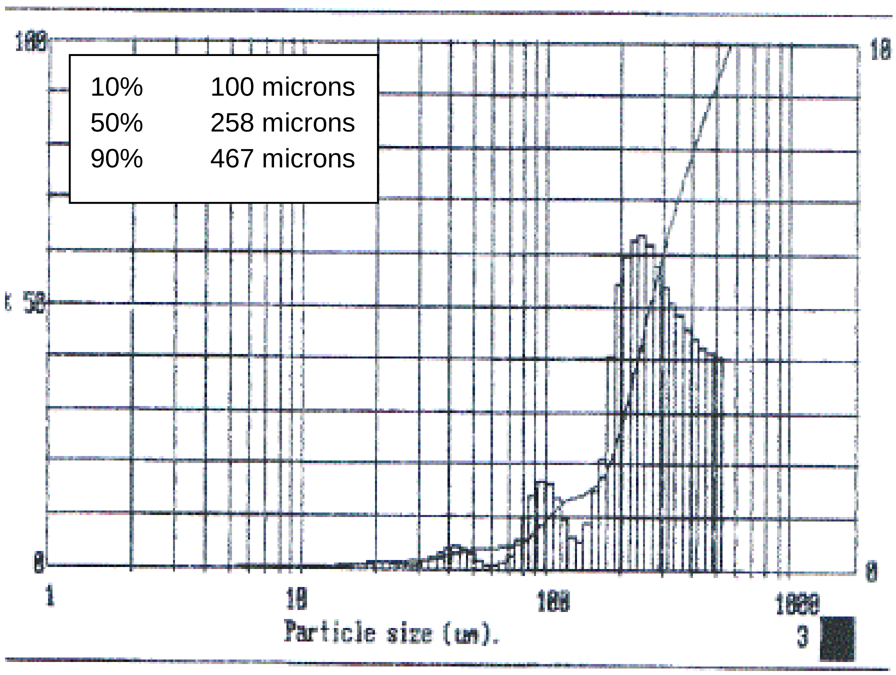
3. Number of Batches Used for Lot: three

4. STANAG TESTING (test methods described in STANAG 4566)

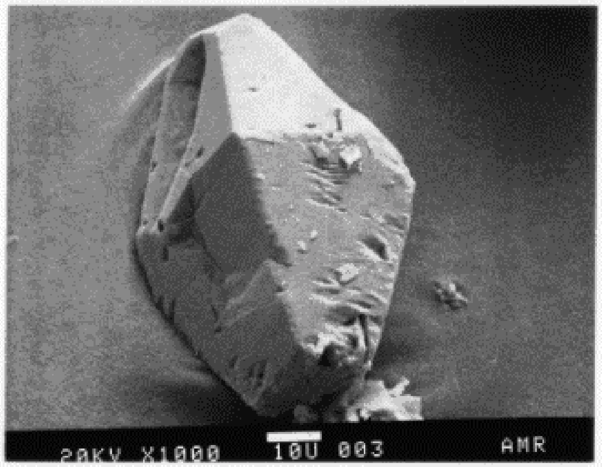
   Chemical Purity: 96% (using combination of HPLC and GPC results)

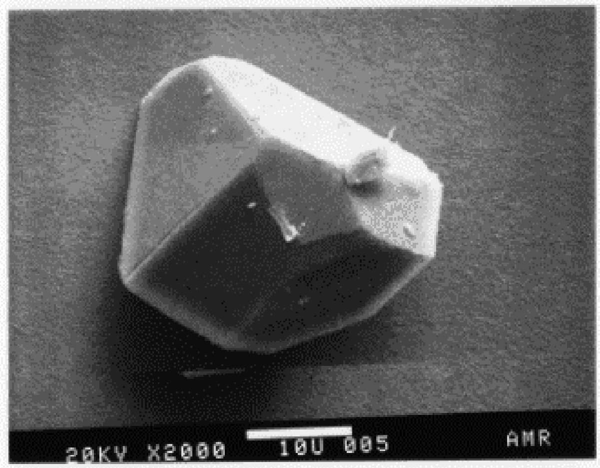
   Polymorph Purity: 95% epsilon

   Particle Size Distribution:
   Mean Particle Size: 258 microns
   Equipment Used: Malvern



5. SEM Photomicrographs:





6. Impact Sensitivity (ERL Impact, 2.5 kg weight): 7 cm (RDX reference, 17 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): 174 lb (RDX reference, 447 lb)

1. Lot/Sample ID Number: **PCL 74**
   Source of Sample: Thiokol

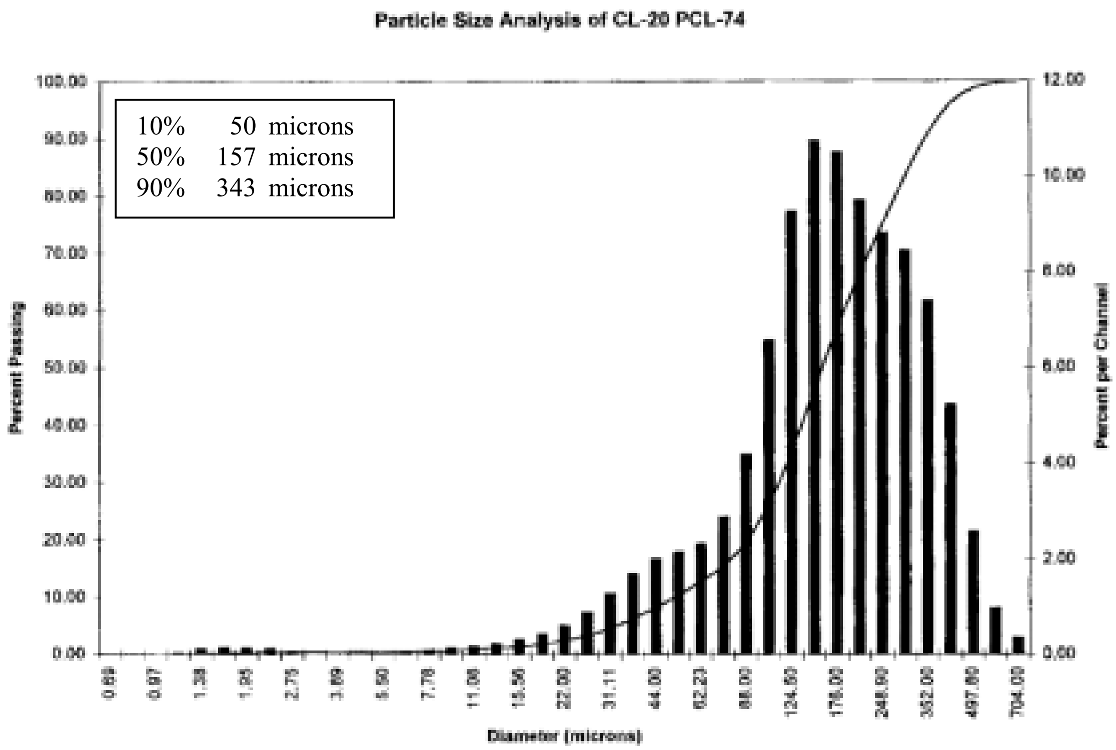
2 Batch Size: Production Lot

3. Number of Batches Used for Lot: one

4. STANAG TESTING (test methods described in STANAG 4566):

   Chemical Purity: 97% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   Mean particle size: 157 microns
   Method Used: Microtrac



5. Photomicrograph:

6. Impact Sensitivity (ABL Impact, 2 kg weight): 1.8 cm (HMX reference, 3.5 or 6.9 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): 50 lb (HMX reference, 50-100 lb)

1. Lot/Sample ID Number: **IHD 174**
   Source of Sample: Indian Head

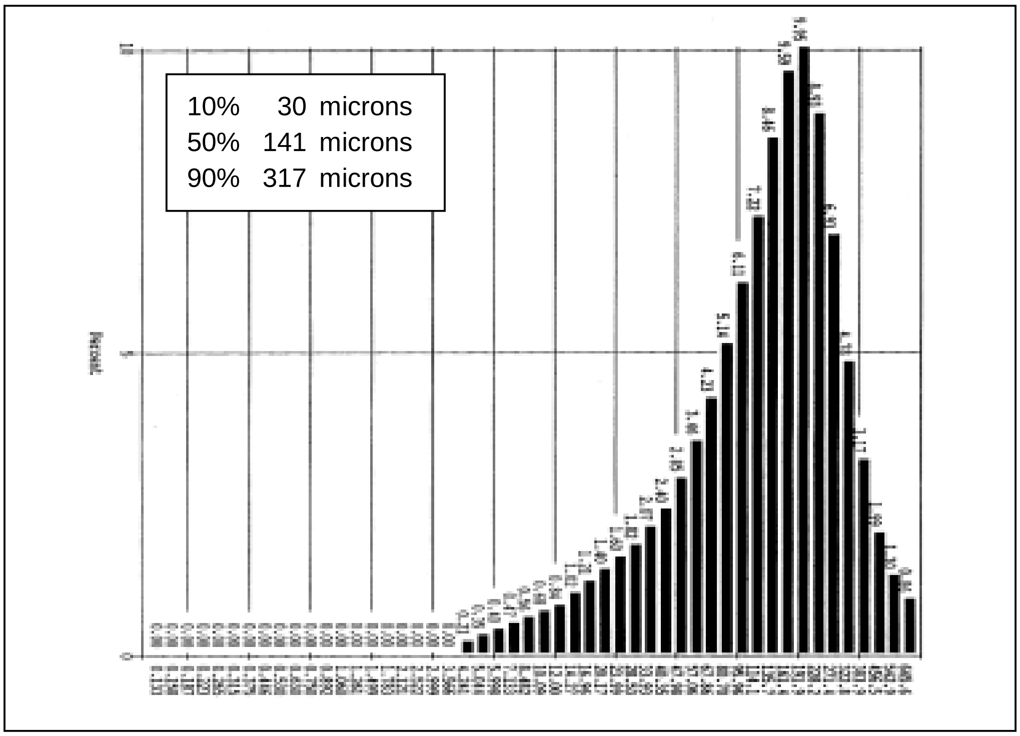
2 Batch Size: 70 grams

3. Number of Batches Used for Lot: one

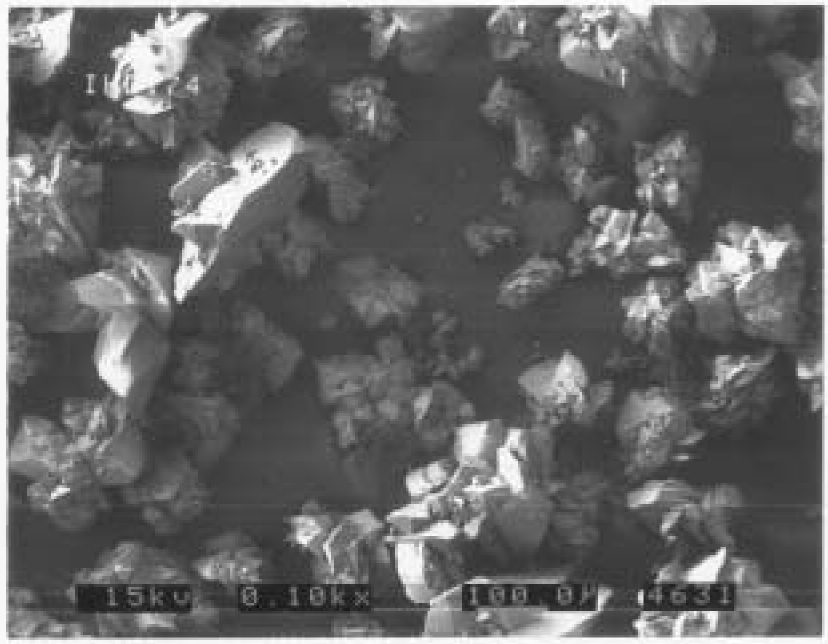
4. STANAG TESTING (test methods described in STANAG 4566)

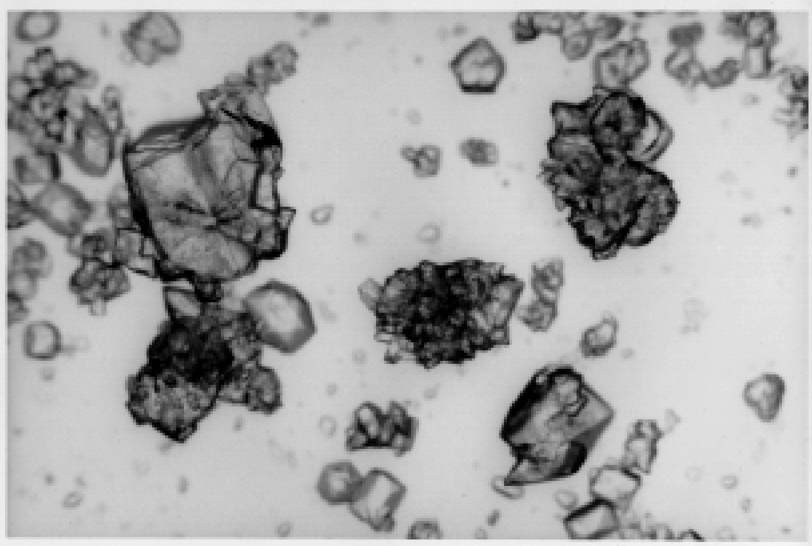
   Chemical Purity: 97.6% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   Mean Particle Size: 141 microns
   Equipment Used: Microtrac



5. SEM and Optical Photomicrographs:




CL-20 IHD 174 (FOCUS UP), 112X ⊢ 100µm
R.RENNER 1/19/00

6. Impact Sensitivity (ERL Impact, 2.5 kg weight): 11 cm (RDX reference, 17 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): 40 lb (RDX reference, 100 lb)

1. Lot/Sample ID Number: **IHD 274**
   Source of Sample: Indian Head

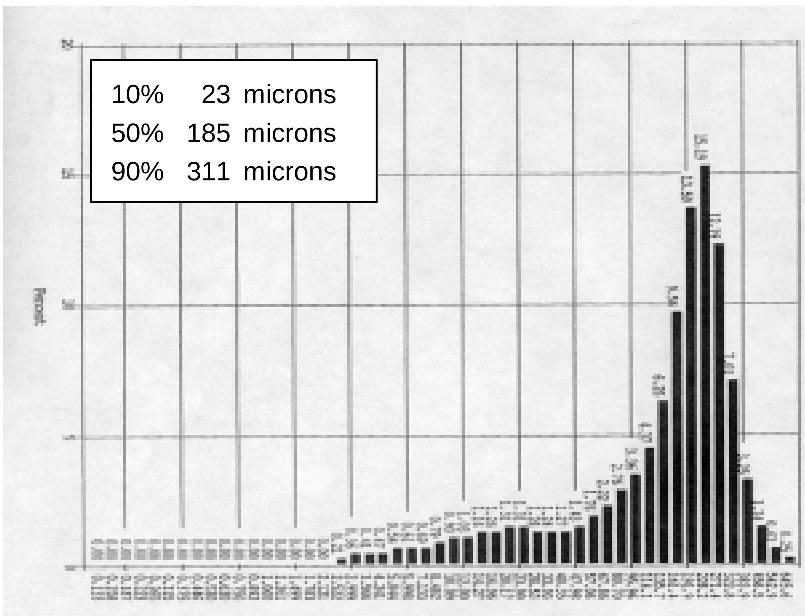
2 Batch Size: 70 grams

3. Number of Batches Used for Lot: one

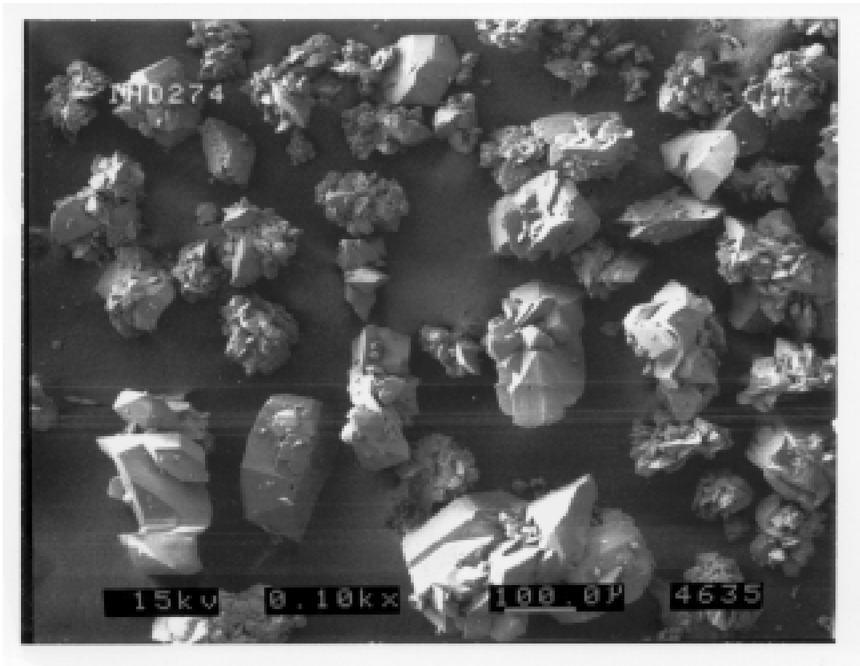
4. STANAG TESTING (test methods described in STANAG 4566)

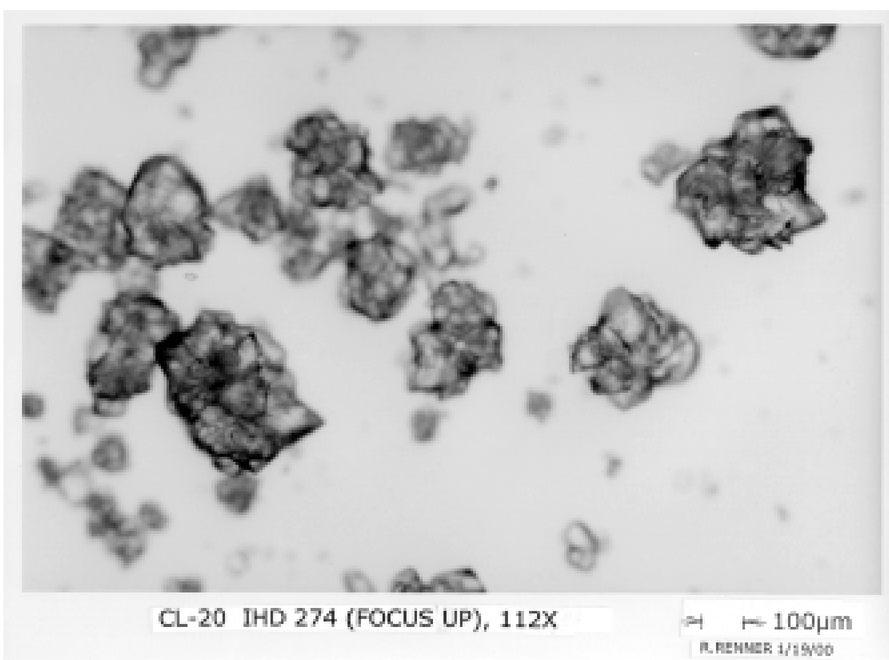
   Chemical Purity: 99.5% Polymorph Purity: > 95% epsilon

   Particle Size Distribution:
   Mean Particle Size: 185 microns
   Equipment Used: Microtrac



5. SEM and Optical Photomicrographs:





6. Impact Sensitivity (ERL Impact, 2.5 kg weight): 11 cm (RDX reference, 17 cm)

7. Friction Sensitivity (ABL Friction at 8 ft/sec): < 30 lb (RDX reference, 100 lb)

1. Lot/Sample ID Number: **0174/2**
   Source of Sample: Aerojet

2 Batch Size: 12.6 kilograms

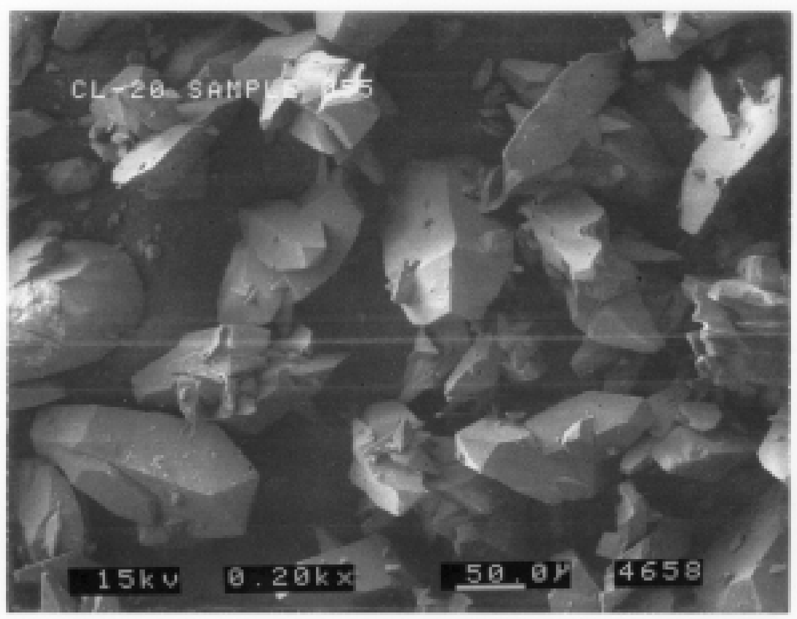
3. Number of Batches Used for Lot: one

4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: > 99.9% (Aerojet data) Polymorph Purity: > 90% epsilon
   99.2% (Indian Head measurement)

   Particle Size Distribution:
   Mean Particle Size: 40 microns
   Measurement Method: not reported

5. SEM Photomicrograph:



6. Impact Sensitivity (Aerojet test, 2 kg weight): 15 cm (HMX reference, 32 cm)

7. Friction Sensitivity (Aerojet roto friction test): > 4000 (result > 4000 is acceptable)

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1. Lot/Sample ID Number: **X991027**
   Source of Sample: TACOM-ARDEC

2 Batch Size: 450 gram

3. Number of Batches Used for Lot: one

4. STANAG TESTING (test methods in STANAG 4566):

   Chemical Purity: > 97% Polymorph Purity: > 97%

   Particle Size Distribution:

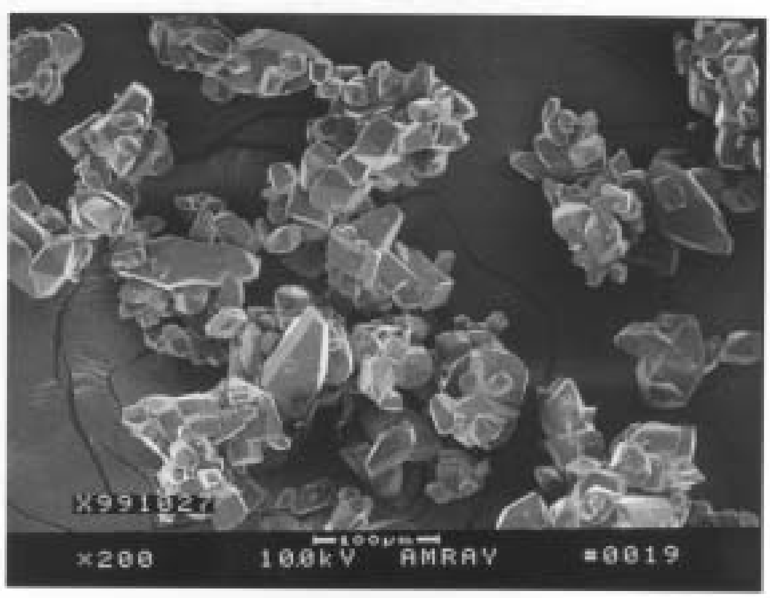
   Median Particle Size: 7 micron (4 – 20 micron size range)

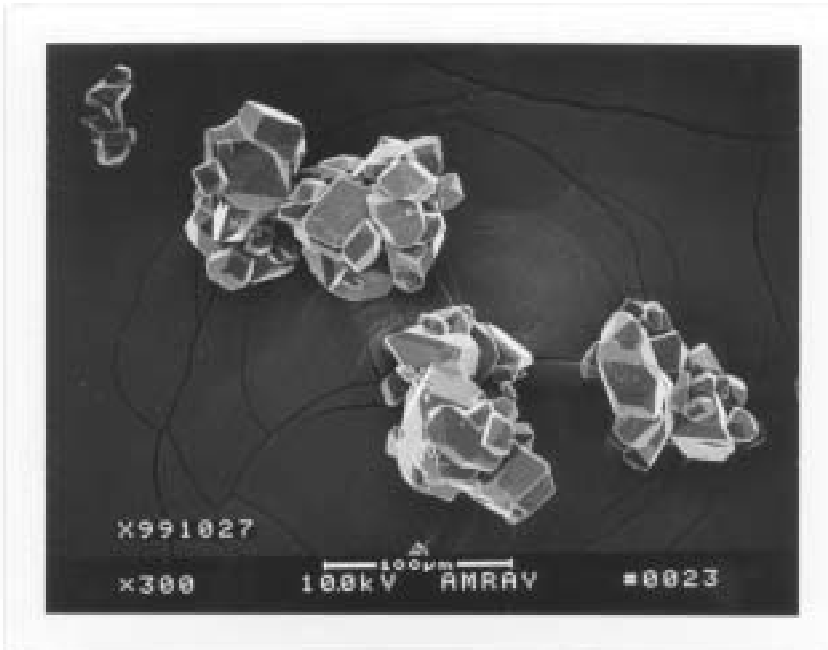
   Equipment Used: Brinkmann PSA 2010

   The particle analyzer cut off the sizing at 20 microns. There are larger particles present, so a size analysis was performed from SEM micrographs. The following particle size data was obtained by analysis of several SEMs.

Less than 12.5 microns =	21.5%
12.5 – 25 microns =	41%
25 – 37.5 microns =	15.5%
37.5 – 50 microns =	15%
50 – 62.5 microns =	2%
above 62.5 microns =	4%

5. SEM Photomicrographs:





6. Impact Sensitivity (50% point, 2.5 kg drop weight, ERL type 12 tooling): 16.6 cm (RDX reference, 25-26 cm)

7. Friction Sensitivity (BAM Friction): 16.8 kg

1. Lot/Sample ID Number: **M4, 2000-01**
   Source of Sample: Bofors

2 Batch Size: 20 grams

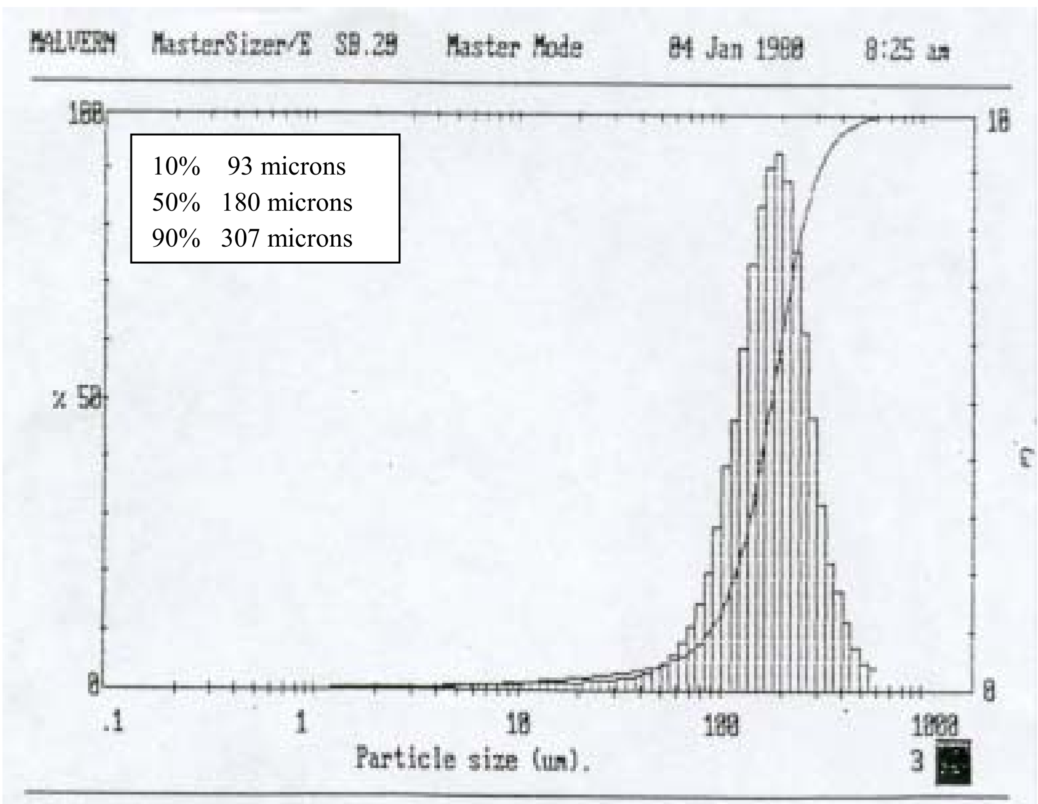
3. Number of Batches Used for Lot: two

4. STANAG TESTING (test methods described in STANAG 4566)

   Chemical Purity: 98.6%
   (99.2%, Indian Head measurement)

   Polymorph Purity: all epsilon
   (> 95% epsilon, Indian Head measurement)

   Particle Size Distribution:
   Mean Particle Size: 180 microns
   Equipment Used: Malvern



5. SEM Photomicrograph:

6. Impact Sensitivity (BAM Impact, 1 kg drop weight): 10 cm

7. Friction Sensitivity (BAM Friction): 94 N

1. Lot/Sample ID Number: **99A1286**
   Source of Sample: SNPE

2 Batch Size: 50 grams

3. Number of Batches Used for Lot: one

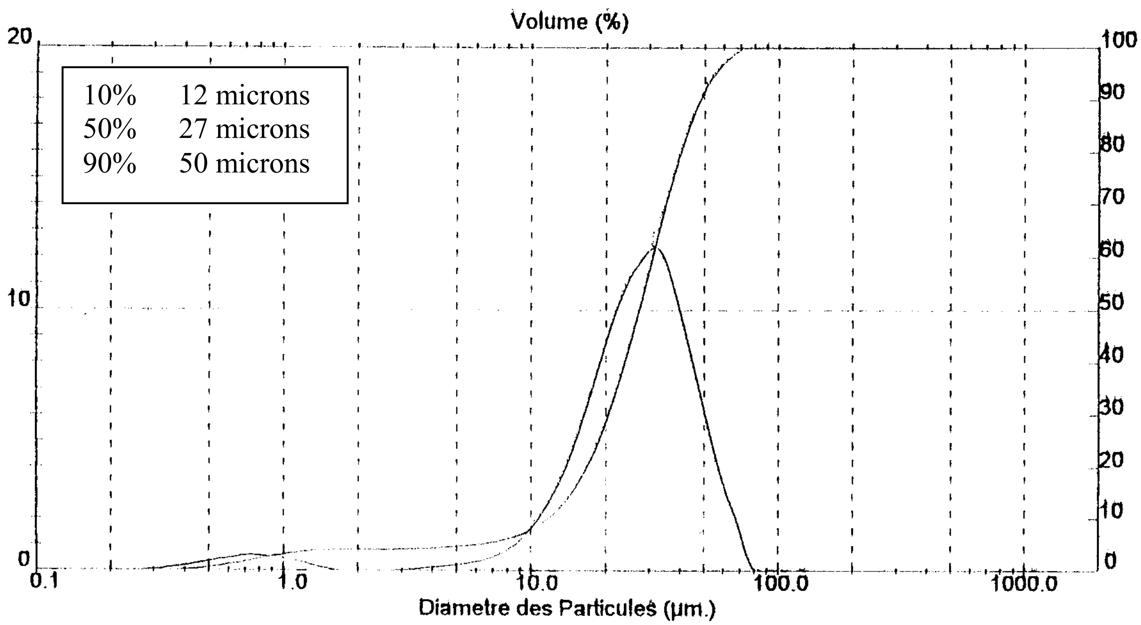
4. STANAG TESTING (Use test methods as described in STANAG 4566)

   Chemical Purity: 99% Polymorph Purity: ≥ 95%

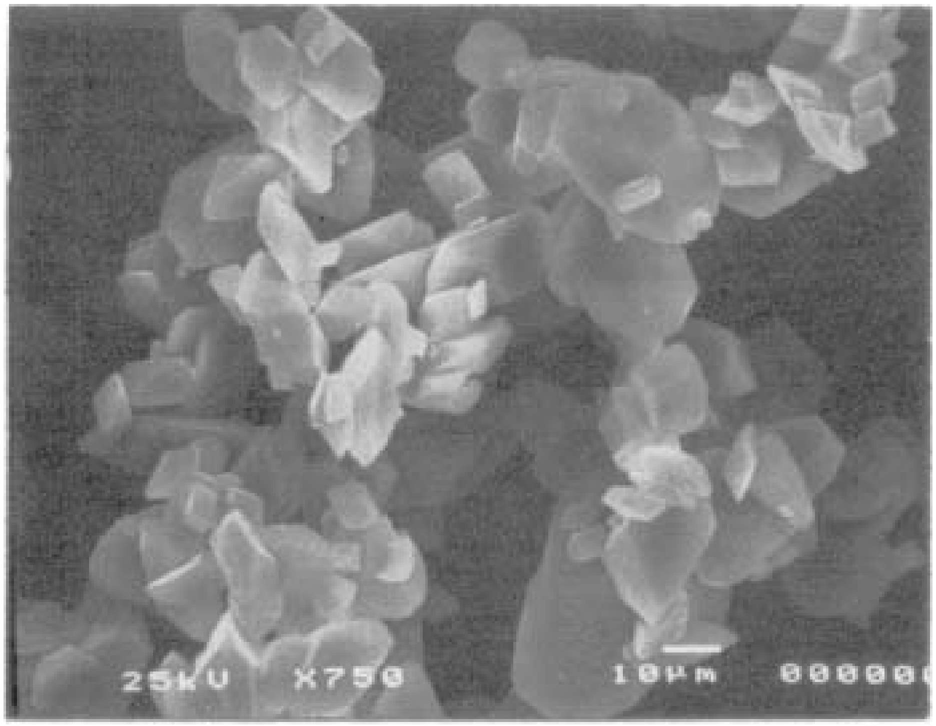
   Particle Size Distribution:

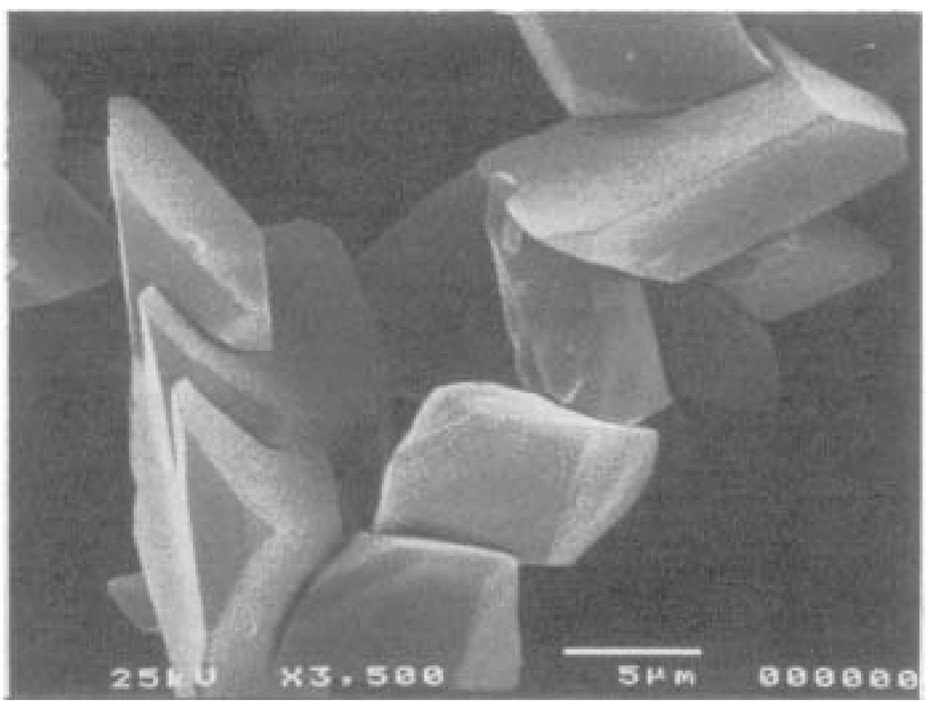
   Mean Particle Size: 27 microns

   Equipment Used: Malvern



5. SEM Photomicrographs:

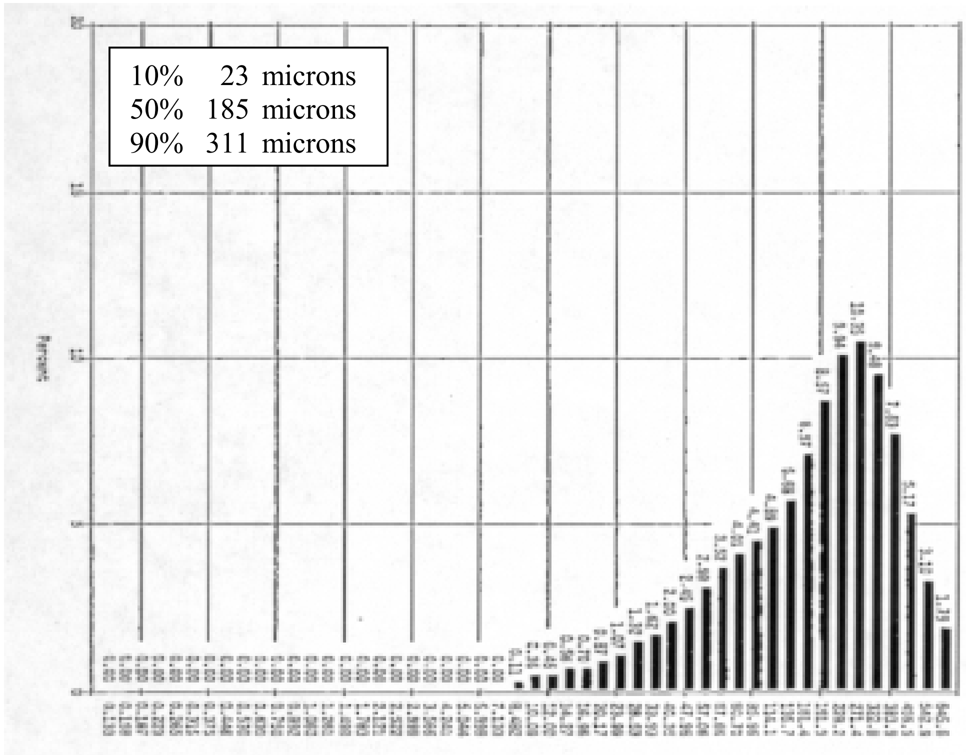




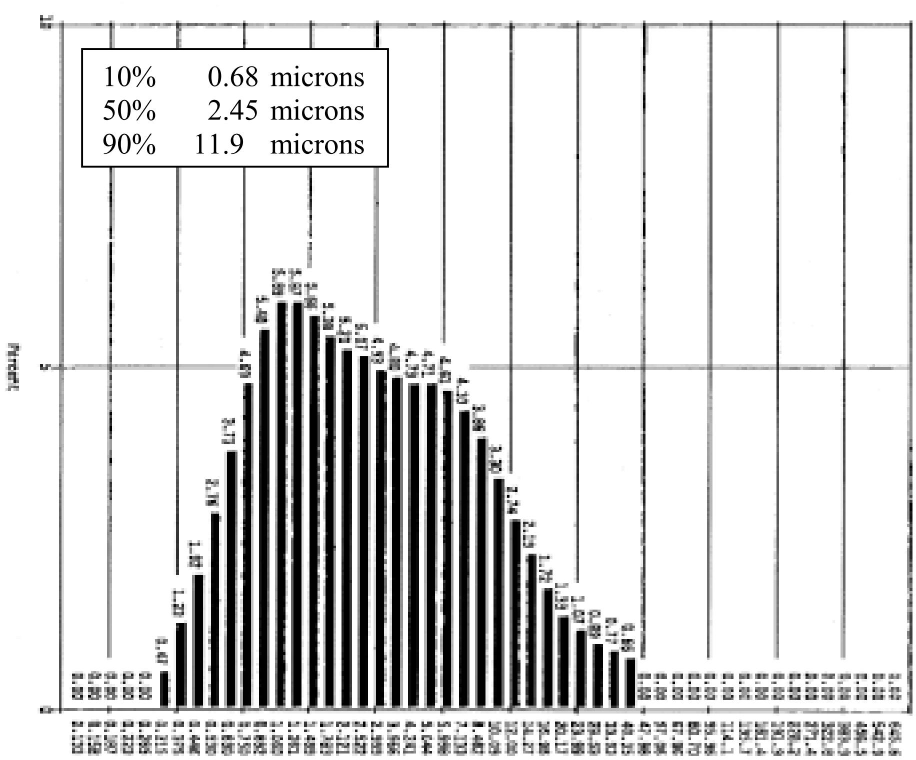
6. Impact Sensitivity (BAM Impact), 50% Point: 1.5 Joules (HMX reference [100-200 microns], 4J)

7. Friction Sensitivity (BAM friction), 50% Point: 73N (HMX reference [100-200 microns], 130N)

RDX Class 1 Reference Lot No. HOL 86D515-085



HMX Class 5 Reference Lot No. HOL 86D200-033



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