

HE PARTICLE CHARACTERIZATION

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QUALITY DIVISION

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Normal Process Development
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The purpose of this project is to transfer the particle characterization techniques obtained in the Development Division to the routine testing requirements of the Chemistry Laboratory of the Quality Division.

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Section W

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ABSTRACT

A comparison of data obtained in the Quality Division and the Development Division using the Fisher Sub-Sieve Sizer has been made. The results show no statistically significant difference between the two laboratories.

The method of obtaining suitable photomicrographs for Zeiss particle analysis has been adapted to the Chemistry Laboratory equipment.

DISCUSSION

This project was started in November of the reporting period, and is actually a modification of an earlier project called Surface Area Determination.

Initially we are looking at particle characterization procedures that are common to both the Chemistry Laboratory of the Quality Division and the Development Division. Fisher Sub-Sieve Sizer for surface area measurements and sieve analysis for particle size distribution are the two common procedures. In addition Zeiss analysis, currently done solely in Development, will be adopted to routine testing by Quality.

FISHER SUB-SIEVE SIZER

Thirty-three lots of PETN were analyzed in both laboratories at porosities of 0.60, 0.55, 0.50, and 0.468. The results of these measurements are listed in Table I. Statistical tests were performed on the sets of data, and the results are summarized in Table II. The conclusion can be made that there is no statistically significant difference between the data obtained in the two laboratories.

SIEVE ANALYSIS

This analysis of particle size distribution is normally performed on lots of HMX. The computer program for analyzing and plotting this data is currently undergoing revision by the Development Division. When this effort is completed, data from common lots of HMX will be compared.

ZEISS PARTICLE ANALYSIS

This is a method for determining particle sizes, relative numbers of each size and shape factors. In the past this work has been done solely in the Development Division. As part of this project, the Quality Division will start doing Zeiss analysis. The first step is taking the photomicrographs on which the particle counting is done. A procedure for doing this using the Chemistry Laboratory equipment has been worked out.

Table I. Fisher Sub-Sieve Size Analysis
Inter-Laboratory Comparisons

Surface Areas, sq. cm/g						
PETN LOT #	Porosity = 0.60			Porosity = 0.55		
	Chem.	Dev.	Diff.	Chem.	Dev.	Diff.
1070-144-01	4709	4741	- 32	5141	5136	5
154	4913	4827	+ 36	5205	5347	-142
164	5308	5415	-107	5559	5845	-286
174	4807	4725	+ 82	5497	5256	241
183	4867	4843	+ 24	5163	5247	- 84
193	5653	5770	-117	5968	6138	-167
203	5109	5117	- 8	5283	5467	-184
224	5512	5078	+434	5973	5424	549
231	5130	5059	+ 71	5590	5338	252
241	5020	4872	+143	5381	5276	-105
259	4571	4402	+169	4625	4758	-133
*259	4914	4402	+512	5216	4388	828
275	4460	4374	+486	5022	4669	353
280	4580	4741	-161	4858	5109	-251
284	4752	4758	- 6	5086	5078	8
294	4625	4843	-218	5197	5195	2
295	4753	4897	-144	5218	5195	23
304	4497	4860	-363	4996	5117	-121
319	4922	4808	+114	5347	5136	211
320	5119	4949	+170	5697	5276	421
323	5176	4913	+263	5650	5175	475
324	5105	4913	+192	5490	5215	275
338	4712	4843	-131	5177	5136	41
339	4274	4237	+ 37	4572	4565	7
344	4563	4402	+161	4824	4808	16
363	4220	4520	-300	4711	4843	-132
372	3942	4012	- 70	4291	4354	- 63
386	4560	4565	- 5	5060	4843	217
391	4423	4374	+ 49	4628	4692	- 64
411	4541	4612	- 71	4777	4949	-172
406	3131	3433	-302	3459	3568	-109
426	4709	4825	-116	5011	5215	-204
416	4741	4877	-136	5341	5235	106
Average	4737	4729	7.76	5122	5063	61.3

* Remun two weeks later.

Table I (Cont'd)

PETN Lot #	Porosity = 0.50			Porosity = 0.468		
	Chem.	Dev.	Diff.	Chem.	Dev.	Diff.
1070-144-01	5136	5529	-393	5540	6053	-513
154	5410	5814	-404	5848	6080	-232
164	5924	6254	-330	5943	6627	-684
174	5288	5673	-385	5539	6108	-569
183	5410	5650	-240	5746	6158	-412
193	6163	6627	-464	6438	6990	-552
203	5483	5819	-336	5934	5999	- 65
224	6055	5673	380	6583	6108	+475
231	5747	5650	97	6058	5870	+188
241	5603	5381	223	5933	5605	+283
259	4870	4985	-115	5203	5296	+ 93
259	5896	5021	875	6108	5276	+832
275	5271	5022	249	5635	5284	+351
280	5143	5446	-303	5165	5770	-605
284	5592	5359	233	6090	5650	+440
294	5560	5472	88	6277	5697	+580
295	5589	5490	99	6440	5947	+493
304	5312	5424	-112	5714	5697	+ 17
319	5745	5467	278	6126	5819	+301
320	5795	5603	192	5871	5895	- 24
323	5795	5512	183	5896	5795	+101
324	5673	5603	70	5820	5819	+ 1
338	5411	5459	- 48	5605	5819	-214
339	4764	4931	-167	4926	5195	-269
344	5094	5117	- 23	5298	5381	- 83
363	5162	5235	- 73	5879	5603	+276
372	4710	4702	8	5112	4913	+199
386	5497	5235	262	6018	5445	+573
391	5271	5156	115	5487	5459	- 28
411	4918	5402	-484	5062	5626	-564
406	3726	3766	- 40	4085	3976	+109
426	5442	5626	-184	6018	5795	+213
4116	5603	5580	23	6059	5795	+264
Average	5396	5418	- 22.0	5741	5715	25.9

Table II. Statistical Test on Differential Data
of Table I

Porosity =	0.468	0.50	0.55	0.60
\bar{X}	25.909	-22.000	61.303	7.758
6	388.87	286.35	250.99	189.79
T	0.231	1.382	0.435	0.377
N	33	33	33	33

There are $33-1=32$ degrees of Freedom. Using a t - distribution table at 95% confidence limit for 30 degrees of Freedom, $t = 2.042$. Since all calculated T values are less than 2.042 there is no reason to believe that the means differ significantly from zero. Hence it can be concluded that there are no statistical differences between the data from the two laboratories.

CONCLUSIONS

The future course of this work will see the adoption of the Zeiss Particle Size Analysis completely by the Chemistry Laboratory. As part of a continuing program, periodic checks of analysis that are performed in common by both laboratories will be made.