RJR
SECRET
No. 219 By

INTEROFFICE MEMORANDUM

TO: Mr. K. L. Rush

FROM: L. R. Hedric
K. A. Jackson
S. S. Meriwether

RE: Grace Alumina pH Qualification **DATE:** May 5, 1989

SUMMARY:

Excess CO_2 and H_2O were the only compounds found on the alumina lots tested that would cause a decrease in the pH thus resulting in an increase to the pH target when heated to 150°C for 2 hours. Of the 5 lots tested, no significant effect on the PREMIER cigarette attributes could be related to the difference in the pH of the lots.

INTRODUCTION:

At the end of October, 1988, RJR began to reject large quantities of Grace alumina because of low pH (pH range 9.3-9.7). When retested, after approximately one month, the pH of the alumina was in spec. Grace was informed of this occurrence and began immediate investigation to determine the source of the problem. Their investigation led to extensive analytical testing of the alumina from which they determined the following:

- Grace alumina product requires surface alkalinity beyond that provided by a pure a-A1203 surface in order to meet the pH specification.
- The "Free" surface Na results in an aging phenomenon where atmospheric CO_2 and H_2O are slowly absorbed resulting in a slow but very significant drop in the product pH over time.
- A small increase in the free surface Na is required to significantly increase the product pH.
- However, in the Grace process a small but uniform increase in surface Na requires more significant increases in bulk Na20.

In summary, Grace attributed the pH decrease to the alumina's ability to absorb atmospheric CO_2 and H_2O. A small increase of Na to the surface of the alumina would raise the pH back to target. They believe that the base pH of all of their alumina is

the same. Activation (Heating at 150°C for two hours), of the low pH alumina, would drive the CO_2 off thus resulting in a reading of the base pH around the specified target, 9.5. If however, RJR did not feel comfortable with these conclusions, an increase in Na could be made to the surface of the alumina to insure a pH of 9.3-9.5 upon receipt. RJR did not feel that an increase in Na would be advantageous to the alumina. A decision was made to run the necessary tests on the alumina to confirm Grace's finding.

TEST DESIGN:

The objectives of the test were defined as follows:

- To determine what was being absorbed by the alumina at Grace and being released at RJR.
- To determine differences in performance of alumina that has:
  - In spec pH vs. low pH due to acidic absorption.
  - In spec pH vs. in spec pH activated.
  - In spec pH vs. low pH activated.
- To determine if there were differences in pH of the alumina that could be associated between calciners.

An outline of the characteristics for the lots to be tested was given to Grace. Grace isolated lots that met these requirements and sent samples to RJR to confirm the pH. The lots were approved by RJR then those specified were activated at Grace. The description of the lots chosen and the steps of the testing done at RJR are as follows:

LEG I.D.	LOT	CHARACTERISTICS	REQUESTED pH
9038 A	AG-D0276	Lot pH was in-spec upon receipt. This lot was used as the CONTROL.	9.3-9.7
9038 B	AG-D0279	Lot pH was in spec as Leg A. This lot will be activated.	9.3-9.7
9038 C	AG-D0146	Lot pH was out of spec upon receipt.	Below 9.3
9038 D	AG-D0245	Lot pH was out of spec upon receipt. This lot was activated.	Below 9.3

9038 E	AG-B0141	Lot pH was out of spec upon receipt. This lot will be from a different calciner.	Below 9.3

TESTING:

STEP	ANALYSIS	REPLICATES	LOCATION	CONTACT
1	pH Analysis Before Activation	10-Replicate	QC	M. Jones
2	Alumina Acceptance Analyses QC-7 Standard Ana.	5-Replicate (pH Only)	QC	M. Jones
	R&D-3 Standard Ana.		R&D	A. Milhous
3	Sampling for Retains and R&D Additional Tests	Two jars for each lot	QC	M. Jones
			R&D	K. Jackson
4	R&D Additional Tests	TBD	R&D	
	*GC/MS	"		M.Borgerding
	*MS Probe	"		"
	*(SEM/EDX)	"		G. Spence
	*XRF	"		A. Milhous
5	Load Alumina	1000 lbs. of each lot	Star	D. Dillon
6	QC Analyses of substrate	5-Replicates (pH Only)	QC	M. Jones
7	Production Run Routine Test		631	D. Marsh
8	Analytical Smoking	Standard	R&D	
	*Total 50/30			B. Hamlin
	*P/P 50/30			A. Milhous
	*CO			
9	Informal Sensory Testing		R&D	K. Hege
10	Data Analysis & Report	Statistical	QA	L. Hedric
				S. Meriwether
			R&D	K. Jackson

TEST RESULTS:

Step 1 - pH ANALYSIS BEFORE ACTIVATION

Ten tests were run on each lot of material to confirm that the alumina pH was the same as specified in the test design. (Leg C was an exception - it only had three confirmation tests run.) Overall the material was as requested. For Leg B, six of the ten tests were below the lower spec limit for pH. (Figure 1; Table 1)

Step 2 - ALUMINA ACCEPTANCE ANALYSIS

Routine acceptance analyses were done on the five lots of alumina upon receipt at RJR. These included seven QC and three R&D analyses. All analyses were within specification with the exception of pH and particle size. The pH was as requested with the activated lots right on target. The particle size was corrected in the Star loading process. (Table 2)

Step 4 - R&D ADDITIONAL TESTS

Samples were submitted to various areas in R&D in hope of isolating foreign elements that may be absorbed on the alumina thus resulting in differences in the pH level.

*GC/MS - The results of this analysis were as follows:

1. Only excess CO_2 and H_2O were found that would result in a decrease of the alumina pH.

2. A linear increase in absorbed CO_2 as the alumina pH decreased. (Attachment I)

*SEM/EDX - No significant differences were found between the samples when scanned for varying levels of Na on the surface. Only Al, O and C were found. The latter resulting from carbon coating applied to the samples for analysis. The morphology of the samples revealed platy structures in all except sample B. Since only approximately four pellets were used for this analysis, it cannot be concluded that platy structures were not in sample B, they just were not found. The platy structure has been seen previously in our alumina and has been known to result in a more stable product over pearly structure. (Attachment II).

*XRF - Samples were scanned by X-Ray Fluorescence. The only elements detected were Ca, K and Ga. The levels of these elements were constant throughout the five samples (Attachment III).

Step 6 - SUBSTRATE ANALYSES

An analysis of variance was performed on the loaded substrate parameters. It showed that Leg A had a statistically higher pH value in comparison to the other four legs. There were no differences among the legs for the other loaded substrate parameters. Although Leg A had a higher pH, all five legs had pH values well within the spec range of 5.3-5.7. Leg A may have had a lower spray dry application which would have resulted in the lower nicotine value and also, a high pH value. The moisture was also high for Leg A which tends to make nicotine lower. (Tables 3, 4 & 5).

Step 7 - PRODUCTION RUN

Bosch Data - Legs C and E appeared to have higher weight values which may have been due to the high bulk density of the alumina for these legs. Leg C had higher draft values than the other legs but no assignable cause could be determined. (Figure 2 and 3).

FA Data - Legs C and E again appeared to have higher weight which is most probably the result of higher weight values for the loaded capsules from the Bosch. Leg D had considerably lower circumference values than the other legs. Legs C, D and E appeared to have higher draft values than Legs A and B. Leg C had higher dilution than the other legs of the test. The pressure drop values for all five legs were extremely consistent. (Figures 4-8).

Step 8 - ANALYTICAL SMOKING

Total 50/30 - Table 6 summarizes the ten trays smoked for each leg of the test. (Leg B had only nine trays tested) Tables 7 & 8 are summaries of the delivery data for each leg. An analysis of variance was performed on all delivery parameters to detect differences between the average level for each leg. Legs B and C had statistically lower nicotine and glycerin delivery values although they had higher substrate nicotine and glycerin values. The differences seen cannot be attributed to pH since Leg B was in spec for pH and Leg C was out of spec for pH. Although there were differences, the values obtained for all legs were within the specified ranges of 10-17 mg for glycerin, .7-.9 mg for nicotine. (Figures 9-12 are boxplots of the individual tray averages).

Puff/Puff 50/30 - Puff/Puff analysis was done on the samples to compare the profiles. No significant difference in the profile was noted. (Figures 13-15).

Step 9 - INFORMAL SENSORY

An informal sensory test was conducted on the five samples in R&D. There were no differences found between the samples.

CONCLUSION:

There were no significant differences found in the various analyses done for this test that could be attributed to the difference in pH level of the lots of alumina tested. The results of all R&D analyses done has led to a confirmation of Grace's theory that excess CO_2 was absorbed on the alumina thus resulting in a decrease in the pH. As a result of these findings, the alumina team has determined:

- That alumina received from Grace and has low pH due to absorption of CO_2, will not affect our process nor the PREMIER cigarette. Therefore, it can be accepted by RJR.

- However, the QC laboratory at RJR does not have the capability of determining the cause of the low pH, therefore, Grace will be required to certify that CO_2 is the cause.

The alumina team feels that the latter can be done either by activation of the alumina, elimination of the CO_2 absorption or by detailed analyses by Grace's R&D facility.

NEXT STEPS:

The forty-four lots of Grace alumina, on hold at Star, will be analyzed to determine if they follow the pattern of findings from this test. The status of these lots and the results of this test will be shared with Grace at the next technical meeting. Their response will be evaluated and considered.

LRH/KAJ/SSM/fp
Attachments

CC - Mr. L. L. Bass, III
Ms. L. E. Curtis
Mr. J. B. Dempster
Dr. R. A. Lloyd

FIGURE 1

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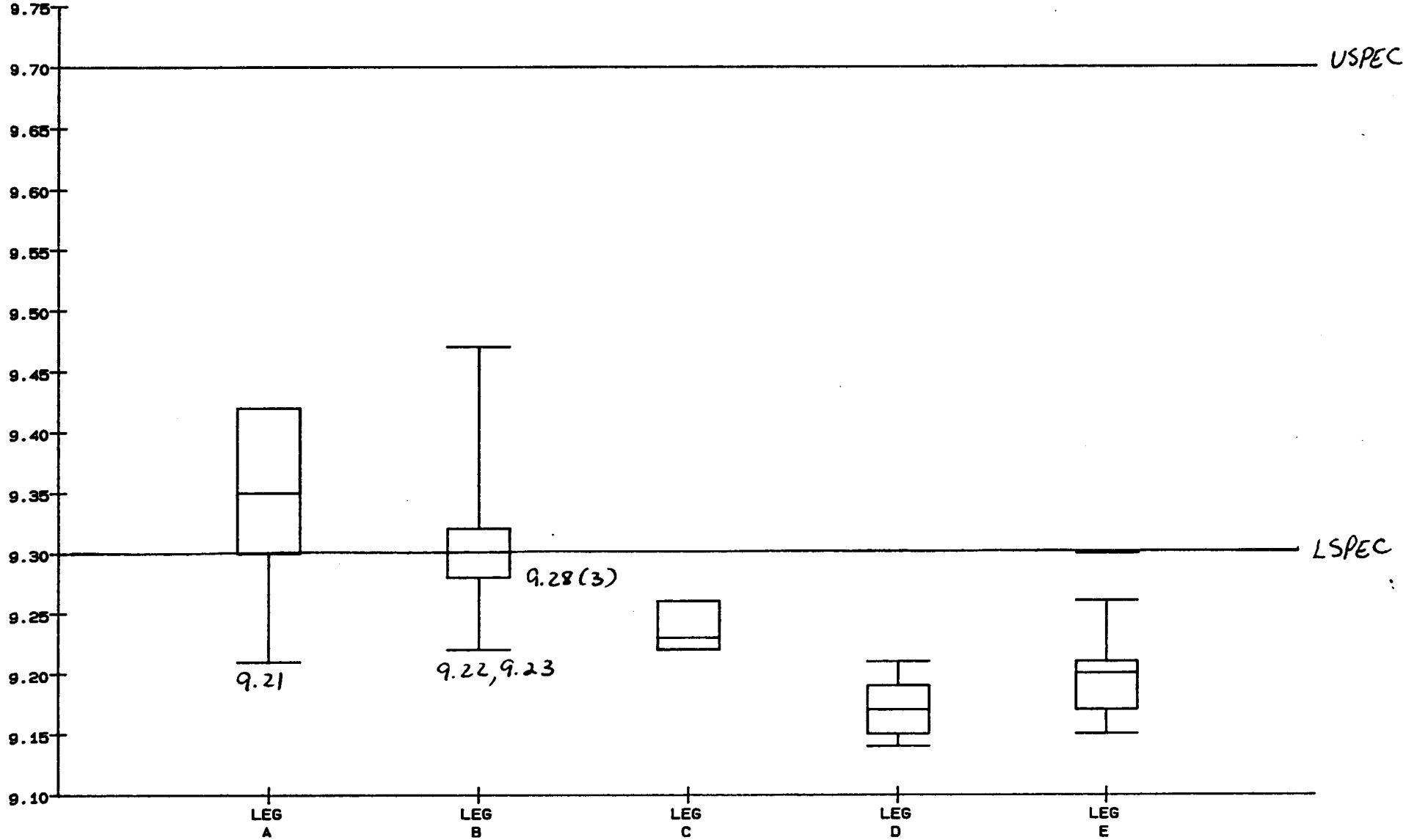
UNACTIVATED ALUMINA PH



LEG C HAS ONLY THREE TESTS. ALL OTHERS HAVE TEN TESTS.

TABLE 1

ALUMINA PH (UNACTIVATED)

CONFIRMATION RESULTS

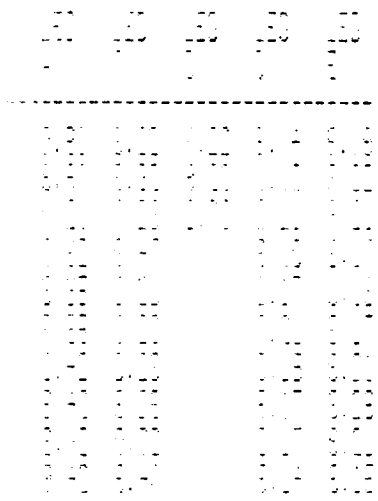


TABLE 2

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GRACE ALUMINA pH QUALIFICATION
ALUMINA ANALYSES

	A AG-D0276	B AG-D0279	C AG-D0146	D AG-D0245	E AG-B0141	SPEC
PARTICLE SIZE						
14	0.7	0.5	0.6	1.0	2.1	-
16	40.6	36.3	33.5	28.8	39.6	25% + 10
18	42.4	45.0	38.9	44.3	38.4	50% + 10
20	16.3	17.5	25.8	25.7	19.5	25% + 10
-20	0.9	1.1	1.6	1.4	2.5	3% Max.
%MOIST.	0.31	0.34	0.32	0.45	0.42	1.0% Max.
pH	9.39	9.49	9.22	9.52	9.22	9.3-9.7
pH 2nd TEST	9.38	9.50	9.26	9.59	9.22	"
%H2O RET.	61.75	62.93	62.02	63.38	61.10	58-64
FILTER FINES	.2	.3	.3	.4	.3	0.4 Max.
REAL DENSITY	3.95	3.95	3.95	3.95	3.95	3.85-4.04
INTRUSION VOLUME	0.62	0.63	0.62	0.63	0.60	.58-.65
MEDIAN PORE	1.00	1.00	0.89	0.90	0.89	.85-1.05
BULK DENSITY	1.12	1.12	1.15	1.13	1.14	1.10-1.15
BET SURFACE AREA	2.24	2.14	2.08	2.19	2.23	8.0 Max.