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A STATISTICAL ANALYSIS OF THE RESULTS FROM A  
CCRA SPONSORED ROUND-ROBIN TEST SERIES ON THE  
ASTM TUMBLER TEST FOR COKE

by

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METALS REDUCTION AND ENERGY CENTRE

- PROGRESS REPORT -

A STATISTICAL ANALYSIS OF THE RESULTS FROM A  
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ABSTRACT

A study of the round-robin results in the ASTM Tumbler Test for Coke has been completed between the Metals Reduction and Energy Centre (MREC) and the four integrated Canadian steel plants.

Excepting one anomalous result, the average of the company Stability and Hardness Factors (referred to as stability and hardness) agreed very well with that determined at MREC. Statistical calculations show that the probability that the anomalous result represents a significant difference between the values for stability determined by the company and by MREC is very high. Further tests including improved standardization of procedures are being considered to clarify this problem.

Except for one case, all organizations doing repetitive tests obtained reproducibilities that were better than the ASTM recommended standard deviations of 1.5 and 1.0 units for stabilities and hardnesses, respectively. In the one case, the larger range in the hardnesses of the supplied samples could be accounted for by the variation in the quality of the coke which had been sampled over a two-week period.

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1.

## INTRODUCTION

The standard methods of measuring the strength properties of coke are almost exclusively confined to the drum type of test. The coke is tumbled under specified conditions and the breakdown of the coke particle size expressed by various indices. The ASTM Tumbler Test for Coke (D294-64) is the standard method used on the North American continent and the Stability Factor of this test method is the prime parameter.

As the Stability Factor has been used as the main criteria of coke quality assessment, it is mandatory that the results obtained remain within the tolerances permitted by the ASTM Test Method. Over the years the Mines Branch has participated in round-robin studies sponsored by ASTM Committee D-5. Periodically comparative studies have also been carried out with various steel plants when the reliability of the results were under suspicion.

At the 41st CCRA Technical Committee meeting in June 1972, it was proposed that each of the four integrated Canadian steel plants would submit samples of their commercial coke to the Mines Branch for ASTM Tumbler Tests. The results obtained were then to be compared with those obtained by industry. This investigation is now complete and the results obtained are statistically analysed herein.

2.

RESULTS

A summary of the stabilities and hardnesses obtained are given in Table 1. The test procedure used was in accordance with the American Society for Testing and Materials, Tumbler Test for Coke which is designated D-294-64(1).

Each group of data was statistically treated by calculating means and standard deviations. The results of each company were compared with the MREC results by means of the t-test(2). The hypothesis being tested was that the mean stability and hardness obtained by MREC was the same as those obtained by the company. Where there were no duplicate stability results from Companies 1 and 4, the t-test was performed, assuming that the company value was the hypothesized correct value, and then testing the hypothesis that the mean MREC stability or hardness was equivalent to "correct" company value(2).

The t-test in Table 2 was valid because, when possible, the calculated F-ratios confirmed the hypothesis that the variances of the populations being compared could be considered equal(2). It was assumed in all cases that "samples" were obtained "independently" from populations having normal distributions. The Statistical Results appear in Tables 2 and 3.

TABLE 1

THE STABILITY AND HARDNESS RESULTS OBTAINED FROM THE  
ROUND-ROBIN OF ASTM TUMBLER TESTS - PROJECT NO. 03-3-0/17-7

Coke From	TEST RESULTS*			
	STABILITY		HARDNESS	
	M.R.E.C. Values	Company	M.R.E.C. Values	Company
Company 1	55.1	56.2	71.5	Not Submitted
	54.3		71.0	
	55.5		71.5	
	55.8		71.4	
	54.9		70.8	
	54.4		70.7	
	56.5		72.8	
	56.3		72.4	
Company 2	56.9	55.0	69.6	Not Submitted
	56.8	54.1	69.7	
	57.4	53.6	69.0	
	57.6	54.5	69.8	
	57.9	54.5	70.8	
	58.0	54.5	70.0	
	57.0	55.0	70.4	
	57.3	55.5	69.6	
Company 3	57.4	54.0	68.3	67.0
	55.6	56.0	66.7	68.5
	55.6	55.0	66.0	67.0
	55.6	55.0	66.2	67.5
	55.3	55.0	67.1	64.5
	55.0	54.0	66.2	66.0
	54.6	57.0	67.2	64.0
		56.0		67.5
Company 4	57.9	58.2	66.0	65.7
	58.3		67.5	
	58.0		67.5	
	58.5		67.0	

\*According to the ASTM D-294-64 Tumbler Test for Coke.

TABLE 2  
THE STATISTICAL RESULTS OF THE  
ROUND-ROBIN FINDINGS FOR STABILITIES

Coke From Company	1		2		3		4	
Laboratory Tested in:	MREC	CO.	MREC	CO.	MREC	CO.	MREC	CO.
Mean Stability <sup>a</sup>	55.4	56.2	57.4	54.6	55.6	55.2	58.2	58.2
N <sup>b</sup>	8	1	8	8	7	9	4	1
Range <sup>c</sup>	2.2	-	1.0	1.9	2.8	3.0	0.5	-
Standard Deviation (s) <sup>d</sup>	0.8	-	0.5	0.6	0.9	1.0	0.3	-
$\Delta$ <sup>e</sup>	-0.8		2.8		0.4		0	
t-test	0.98 <sup>f</sup>		10.6 <sup>f</sup>		0.77 <sup>f</sup>		0.00 <sup>f</sup>	
t-table	1.895		1.76		1.76		2.35	
Conclusion	Accept (Could not Reject)		Reject <sup>i</sup>		Accept <sup>i</sup> (Could not Reject)		Accept (Could not Reject)	

<sup>a</sup> Mean Stabilities =  $\Sigma X/N = \bar{X}$ , where X = the stability from one test.

<sup>b</sup> N = the number of tests carried out

<sup>c</sup> Range = X max. - X min. = largest observation - smallest observation

<sup>d</sup> Standard deviation =  $(\Sigma(X-\bar{X})^2/(N-1))^{1/2} = s$

(note:  $s^2$  = variance) ASTM specifies that s should be less than 1.5 for stabilities

<sup>e</sup>  $\Delta$  is the difference between the MREC mean stability and the company value.

<sup>f</sup> t-test =  $\Delta / (s / N^{1/2})$

<sup>i</sup> Decision

Reject if  $t > t_{table}$  (or  $t < -t_{table}$ )

<sup>f</sup> t-test; for the MREC mean stability being compared to the company mean stability. The null hypothesis is

$H_0$ : MREC mean stability = company mean stability.

The calculation is

$$t\text{-test} = (\text{MREC mean} - \text{company mean}) / s_{\bar{X}_1 - \bar{X}_2}$$

where

$$s_{\bar{X}_1 - \bar{X}_2}^2 = s_{*1}^2 / N_1 + s_{*2}^2 / N_2$$

$$s_{*}^2 = [(N_1 - 1)s_1^2 + (N_2 - 1)s_2^2] / (N_1 + N_2 - 2).$$

The "1" subscript refers to the MREC results and the "2" refers to the company results.

<sup>g</sup> t-table; this t value is looked up in a standard t-table for  $\alpha = 0.05$ .

In other words, we are risking a 5% chance that we will reject a hypothesis that is actually true. The value looked up is  $t_{(1-\alpha/2, N_1 + N_2 - 2)}$  = table value. (e.g.  $t_{(\alpha=0.05, 14)} = 1.76$  for the second results).

<sup>h</sup> The conclusion refers to whether the null hypothesis can or cannot be rejected. The hypothesis is rejected if  $|t\text{-test}| \geq |t\text{-table}|$ .

<sup>i</sup> F-tests; where the test is

F = larger variance/smaller variance, of the two populations being compared, indicated that the null hypothesis that the variances of the two populations being compared are equal, could not be rejected.

TABLE 3  
THE STATISTICAL CALCULATIONS FOR THE ROUND-ROBIN  
RESULTS OF HARDNESSES<sup>a</sup>

Coke From Company	1		2		3		4	
Laboratory Tested in:	MREC	CO.	MREC	CO.	MREC	CO.	MREC	CO.
Mean Hardness	71.5	-	69.7	-	66.8	66.6	67.0	65.7
N	8	-	8	-	7	9	4	1
Range	2.1	-	1.8	-	2.3	4.5	1.5	-
Standard Deviation (s)	0.75	-	0.55	-	0.80	1.50	0.71	-
$\Delta$	-		-		0.2		1.3	
t-test	-		-		0.32		1.64	
t-table	-		-		1.76		2.35	
Conclusion	-		-		Accept <sup>b</sup> (could not reject)		Accept (could not reject)	

<sup>a</sup> See Table 2 for details of the calculations. Note that an acceptable standard deviation for hardness is 1.0.

<sup>b</sup> The F-ratio indicated that an equality of the population variances could not be rejected.



3.

## DISCUSSION

### 3.1 Reproducibility on Tests

All the stabilities determined seemed to fall within an acceptable precision of 1.5 stability units, as set down in the ASTM Tumbler Test for Coke(1). The largest standard deviation reported was 1.0 stability units.

Unfortunately, only two hardness results were obtained from the companies and one of these was only a single number. Company 3 which did report the results of a number of tests had a rather large standard deviation on their hardnesses, whereas all the MREC results were within the acceptable limits. Closer inspection disclosed that this was the company with the largest standard deviation on the stabilities. The reason for this divergence for Company 3 seems to be that the samples were obtained daily, over a period of weeks, and consequently a range of coke quality was introduced into the tests. Because the method by which the remaining companies sampled their coke was unknown, comparisons could not be made. It would seem reasonable that a standard method of sampling be agreed upon in any future round-robins, so that errors or large deviations introduced by the method of sampling can be eliminated.

### 3.2 Comparisons

Except for Company 2, the mean value of the statistics obtained at MREC for each company's coke, agreed with the results obtained by the company. The t-tests indicated that Companies 1, 3, and 4 obtained the same mean stabilities and hardnesses as MREC. The results from Company 2 differed drastically from the MREC results.

Company 2 results indicate that the errors in obtaining their stabilities were small because the standard deviation on the mean coke stability was small. The same was found for the MREC results from this coke.

However, it is highly probable that the mean stabilities are different. Statistically there is less than a 0.01 per cent probability that the company's result does not represent a significant difference from the mean stability determined at MREC. One possible explanation is that Company 2 is conducting their stability tests differently than MREC or the other companies; this seems so because the MREC results agree very closely with the results from the other companies.

4.

#### CONCLUSIONS

4.1 Except for one hardness test done by Company 3, all the results were within the ASTM limits of precision.

4.2 The MREC tumbler tests for stability or hardness could not be statistically differentiated (at reasonable levels of probability; e.g. a 5% chance of being wrong) from the results obtained by three of the companies. Company 2 differed.

4.3 The mean stability obtained by Company 2 differed significantly from the MREC result. The chance that the difference is not significant is less than 0.01 per cent according to the t-test. Because MREC agreed with the other companies, it seems possible that this discrepancy could lie with the company's method of testing, although such errors as mixing of drums cannot be discounted.

4.4 If the correlation of the MREC coke ovens and those used in industry is to be meaningful or significant, the tumbler tests performed by MREC and the companies must agree.

4.5 A standard sampling method should be agreed upon for future round-robin tumbler tests, so that discrepancies due to different sampling procedures can be eliminated.

5.

REFERENCES

1. 1972 Annual Book of ASTM Standards (1972), Philadelphia, Pa., American Society for Testing and Materials, Tumbler Test for Coke, (ASTM designation: D-294-64), Vol.19, p. 41.
2. Ostle, B. (1969) Statistics in Research. Ames, Iowa, The Iowa State University Press.