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**ANALYSIS OF CRUSHED PELLETS PRODUCED  
BY ERIE MINING COMPANY FOR  
STANDARDIZATION PURPOSES**

by

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F. W. BRETHOUR & W. R. INMAN**

**MINERAL SCIENCES DIVISION**

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D.J. Charette<sup>\*</sup>, J.C. Hole<sup>\*\*</sup>, F.W. Brethour<sup>\*\*\*</sup>  
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INTRODUCTION

The Steel Company of Canada requested the Analytical Chemistry Subdivision to determine total iron, ferrous iron, silica, phosphorus, sulphur, manganese, alumina, lime and magnesia in a sample of crushed pellets. This request was made in connection with a project of the Research Division of the Erie Mining Company regarding the variation among analytical results from various laboratories on a standard sample. It was suggested that the Analytical Chemistry Subdivision act as referee analyst in this study. All participating laboratories are to send their results to the Erie Mining Co., who will tabulate the data, and each participating laboratory will receive copies of all results.

METHODS AND RESULTS

Total Iron

The sample was decomposed by treatment with hydrochloric acid, filtered, and the residue dissolved with hydrofluoric acid. Sulphuric acid was added and the solution brought to fumes. Both solutions were then combined and brought to fumes of sulphuric acid. The iron was

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reduced with hydrogen sulphide, the excess  $H_2S$  removed by boiling and the solution titrated with potassium dichromate.

#### Ferrous Iron

The sample was decomposed by hydrochloric and hydrofluoric acids. When dissolution was complete, the hot solution was poured into a solution of boric acid and titrated immediately with potassium dichromate.

#### Metallic Iron

It was discovered that the sample contained some metallic iron. The ferrous iron figure was corrected for this. The metallic iron was determined by dissolution of the metallic fraction with mercuric chloride and titration of the resultant ferrous iron with potassium dichromate.

#### Silica

The bulk of the iron was dissolved in hydrochloric acid. The insoluble residue was fused with sodium carbonate, the fusion cake dissolved by hydrochloric acid, and silica recovered by double dehydration with perchloric acid.

#### Manganese

The sample was treated with hydrochloric acid. The residue was dissolved by hydrofluoric and hydrochloric acids. Sulphuric acid was then added and the solution evaporated to fumes. This was taken up in water and added to the main solution. The combined solutions were evaporated to fumes of sulphuric acid and then made to a definite volume with water. Manganese was determined on an aliquot by the spectrophotometric periodate procedure.

#### Sulphur

Sulphur was determined by the combustion technique using the silver nitrate absorption solution and titrating with standard sodium hydroxide.

#### Phosphorus

The sample was dissolved in hydrochloric acid. Perchloric acid was added and the solution evaporated to fumes. After adjusting the acidity the iron was removed by electrolysis with the mercury cathode. Phosphorus was determined in the iron free solution by the spectrophotometric "molybdenum blue" procedure.

### Alumina, Lime and Magnesia

From conversations with members of Stelco's Research Division, it was understood that the analytical situation with respect to the above three constituents was not entirely satisfactory. Therefore, these were determined by two procedures, as follows:

#### a) Classical Procedure

The sample was decomposed by hydrochloric and hydrofluoric acids as detailed under the method for manganese. When the solution was adjusted to appropriate acidity, iron and other interfering elements were separated by extracting their cupferrates into chloroform. On the aqueous solution the classical determinations were performed, viz.,

Alumina, as  $\text{Al}_2\text{O}_3$ , following double precipitation with ammonium hydroxide.

Lime, on the filtrate from the above by double precipitation as the oxalate, followed by permanganate titration.

Magnesia, on the filtrate from the above, after removal of excess ammonium salts, by double precipitation as magnesium ammonium phosphate, and then weighing as magnesium pyrophosphate.

#### b) Spectrophotometric Alumina and Complexometric Titration of Lime and Magnesia

One gram samples were dissolved in hydrochloric and hydrofluoric acids and brought into sulphuric acid solution; iron and other interfering elements were removed with cupferron as above.

The aqueous solution was made to volume of 200 millilitres.

Alumina was determined on a three (3) millilitre aliquot using the spectrophotometric chromoxene cyanine RA reagent.

Lime and magnesia were determined together by titration of fifty (50) millilitre aliquots with ethylene-diamine-tetraacetic acid (EDTA). Eriochrome Black T was used as indicator, in the presence of triethanolamine, cyanide, and ferrocyanide as masking agents for possible inter-ferents. On other fifty (50) millilitre aliquots, the calcium was removed by double oxalate precipitation, organic matter in the filtrate destroyed by nitric and perchloric acids, and magnesium determined by EDTA titration. Thus CaO was determined by difference.

The alumina figures by both methods were in substantial agreement. The checks on the lime and magnesia cannot be considered satisfactory and further work is underway to attempt to resolve the differences. To assist the co-ordinator in assessing the reliability of the two methods, all replicates are given for  $\text{Al}_2\text{O}_3$ , CaO and MgO.

Results are presented in Table 1.

TABLE 1

Analysis of Crushed Pellets

	<u>Per cent</u>	
Total Fe	63.99 (63.33 = 90.55 Fe <sub>2</sub> O <sub>3</sub> )	
Ferrous Fe	0.45 (0.57 FeO)	
Metallic Fe	0.21	
SiO <sub>2</sub>	6.49	
MnO	0.17	
S	0.004	
P	0.003	
	<u>(Gravimetric)</u>	<u>(Spectrophotometric)</u>
Al <sub>2</sub> O <sub>3</sub>	0.29, 0.29	0.27, 0.25, 0.25
	<u>0.26, 0.28</u>	<u>0.26, 0.27</u>
	0.28	0.26
	<u>(KMnO<sub>4</sub> Titration)</u>	<u>(EDTA - Difference)</u>
CaO	0.82, 0.76	0.64, 0.64, 0.64
	<u>0.77, 0.76</u>	<u>0.66, 0.62</u>
	0.78	0.64
	<u>(Pyrophosphate)</u>	<u>(EDTA - Titration)</u>
MgO	0.53, 0.44	0.36, 0.36, 0.36
	<u>0.44, 0.49</u>	<u>0.36, 0.36</u>
	0.48	0.36
Totals	99.54	99.26

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