

DETERMINATION OF OLIVINE
LOSS ON IGNITION

by

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ABSTRACT

A problem has been identified concerning the reporting of olivine Loss on Ignition (LOI) analysis data. Various olivine producers are reporting test results to potential buyers without these olivine producers using the same Loss on Ignition procedures. Since LOI can be a critical factor to olivine buyers, these different LOI procedures cause the buyers to have an inaccurate assessment of the quality of olivine being presented to them for purchase.

This report identifies some of the critical differences between these various LOI procedures and offers possible solutions for the accurate comparison of different olivine samples.

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INTRODUCTION

Toward the end of 1987, there began to appear definite signs of problems in the reporting of olivine Loss on Ignition (LOI) data by various olivine producers. Olivine samples from various European and United States suppliers that had very similar elemental analyses, were being reported to potential olivine buyers as having quite different LOI values.

The mineral olivine is a general term for a mixture of Forsterite (Mg_2SiO_4) and Fayalite (Fe_2SiO_4) (1). The two most common uses of olivine are as (a) a slag conditioner for blast furnace use, and (b) as a foundry sand in molds for various metals. For both of these uses an olivine with a low LOI is best. Therefore, the analysis of the LOI on an olivine sample is of great importance to the user. So in general, if multiple sources of olivine are available to a buyer that meet the elemental specifications, the olivine with the lower Loss on Ignition will be preferred and in most cases be purchased.

The reason that an LOI analysis can be a problem is because of the Fayalite constituent of olivine. The iron in Fayalite will be oxidized when heated in an oxidizing atmosphere to the normal LOI temperature of approximately 950 degrees Centigrade. As the ferrous iron in the Fayalite oxidizes to form ferric iron

oxide (Fe_2O_3), weight is added to the dry sample in the form of oxygen (2). This causes the sample to not lose as much weight as it would normally in a non-oxidizing atmosphere. Therefore, the LOI result will be lower than expected.

To aid in the identification of the LOI analytical problem, an in-house olivine sample was prepared at the Minerals Research Laboratory using a high grade North Carolina olivine product. This sample was then sent to various European and United States laboratories for chemical analysis. The results are shown in Table I.

TABLE I
HIGH GRADE OLIVINE

	<u>Lab. #1</u>	<u>Lab. #2</u>	<u>Lab. #3</u>	<u>Lab. #4</u>	<u>Lab. #4</u>
MgO	48.3%	48.5%	48.6%	48.5%	-----
SiO ₂	41.5%	41.4%	41.5%	41.4%	41.7%
LOI	0.26%	0.40%	0.89%	1.01%	0.42%

Lab #1 = European Producer Laboratory, Norway

Lab #2 = European Producer Laboratory, Norway

Lab #3 = U. S. Analytical Laboratory

Lab #4 = Minerals Research Laboratory, Asheville, NC., U.S.A.

Lab #5 = European Analytical Laboratory, West Germany

As can be seen from Table I, the MgO and SiO₂ are in good agreement, while the LOI values are extremely varied. Inquiries were made as to how each laboratory analyzed for olivine LOI. Laboratories #1, #2 and #5 all ran their LOI tests in furnaces

that were open to the atmosphere, whereas Laboratories #3 and #4 ran their LOI tests in an inert (argon) atmosphere. It was felt by the Minerals Research Laboratory that Laboratories #1, #2 and #5 were having the olivine oxidize which would cause their LOI values to be much lower than Laboratories #3 and #4. This concern at the Minerals Research Laboratory was greatly increased when one of the Norway olivine producers stated that there was "no appreciable oxidation of olivine" at high temperatures with the oxygen available from the air.

After receiving these varied results, there was concern that North Carolina olivine producers might not be getting a fair evaluation of their product in relation to the European olivine that is being imported into the U.S.A.

With the help of a North Carolina olivine producer, testing began on a high grade olivine and a low grade olivine prepared at the Minerals Research Laboratory. The purpose of this testing was to show the effect of LOI tests done in oxidizing and non-oxidizing conditions. Recommendations would also be made as to the best way to perform LOI analyses with a minimum of variation between different analytical laboratories.

EFFECT OF OXIDIZING ATMOSPHERE ON LOI

Testing was started at the Minerals Research Laboratory using the before mentioned high grade olivine from North Carolina. A Burrell tube furnace with a tapered ceramic tube was used for the testing. The tapered tube allowed for easy addition to the tube of inert gas. The tests with a non-oxidizing atmosphere had argon gas flowing through the tube at a rate of 2 liters per minute per sample. Tests in an oxidizing atmosphere were done with both ends of the ceramic tube open to the air in the laboratory. A more detailed description of the procedure that was used at the Minerals Research Laboratory is given at the conclusion of this report.

The results from the testing of the high grade olivine is shown in Table II.

TABLE II
HIGH GRADE OLIVINE

<u>Test #</u>	<u>% LOI</u> <u>Non-oxidizing conditions</u>	<u>% LOI</u> <u>Oxidizing conditions</u>
1	1.01	0.51
2	1.03	0.49
3	1.03	0.49
4	1.02	0.51
5	1.02	0.53
6	1.00	0.51
7	1.02	0.49
8	1.02	0.53
9	1.02	0.54
10	<u>1.02</u>	<u>0.50</u>
	Average = 1.02	0.51

After testing of the high grade olivine, the low grade olivine was tested in the same way with the following results as shown in Table III.

TABLE III
LOW GRADE OLIVINE

<u>Test #</u>	<u>% LOI</u> <u>Non-oxidizing conditions</u>	<u>% LOI</u> <u>Oxidizing conditions</u>
1	5.00	4.47
2	5.01	4.71
3	4.97	4.53
4	4.96	4.47
5	5.02	4.52
6	5.02	4.53
7	4.98	4.53
8	4.95	4.54
9	5.02	4.55
10	<u>5.05</u>	<u>4.53</u>
	Average = 5.00	4.54

CONCLUSIONS

In Tables II and III, it can be seen that there is drastic change in LOI values when olivine is heated in an oxidizing atmosphere instead of a non-oxidizing (inert) atmosphere. These changes could definitely cause confusion in regards to buyer-seller negotiations for olivine.

In the case of a high grade olivine, it can be seen that there is a 50% reduction of the LOI value when the test is carried out in oxidizing conditions. Since low LOI results are preferred by olivine buyers, the olivine producer who runs his LOI tests under non-oxidizing conditions would definitely be at a disadvantage when competing with a producer that runs their LOI tests under oxidizing conditions.

The low grade olivine shows the same downward trend in LOI

value when the LOI is performed in oxidizing conditions. The data showed that there is approximately a 9.2% reduction in the LOI value from the non-oxidizing results. While this lower LOI shift is not as drastic as in the case of the high grade olivine, it is still not acceptable from an analytical chemistry point of view.

Loss on Ignition testing appears from this data to be very dependent on the type of atmosphere (oxidizing or non-oxidizing) used for the test. The procedure at the end of this paper warns of many other possible problems that can occur. Problem factors such as mesh size of the sample, size of sample analyzed, furnace boat size, purity of the inert gas used, furnace temperature, and time of sample in the hot zone of the furnace, are all critical for getting good reproducible results.

Loss on Ignition testing can be done so as to be very reproducible. It does appear that for different analytical laboratories to get the same results on a given sample, they must use very similar procedures. Olivine buyers need to be wary of how a Loss on Ignition is performed. Otherwise, confusion will continue in regards to who has the "best" olivine product.

REFERENCES

1. Teague, K. H., "Olivine," Industrial Minerals and Rocks, 5th Edition, Vol. 2, pp. 989-994, 1983.
2. Redeker, I. H., "Beneficiation of Olivine for Foundry Sand by Calcining," Report No. MRL-2, North Carolina State University Minerals Research Laboratory, Asheville, NC, 1972.

OLIVINE LOSS ON IGNITION PROCEDURE

For Olivine samples do the following:

1. Record the weight on the dry Porcelain boat. Note: the boat should be baked in the furnace for 30 minutes at 950 degrees Centigrade (C), then cooled in a desiccator to room temperature before use.
2. Place approximately 1 gram of the olivine in the boat and then place the boat with sample in an oven at 120 degrees C. for 1 hour. Cool the boat in a desiccator to room temperature and record the weight. The difference between the dry boat and the dry boat with sample gives the actual dry olivine weight for the Loss on Ignition test (LOI).
3. Pre-heat the tube furnace to 950 degrees C. and purge the tube with Argon at a flow rate of 2 liters per minute per boat. (Nitrogen may be used instead of Argon if the nitrogen is oxygen free.)
4. Place the boat with sample into the furnace at the pre-heat position for 2 minutes. Next, push the boat into the 950 degrees C. position for 10 minutes. Then pull the boat back to the pre-heat for 2 minutes. Put the boat in the desiccator and cool to room temperature. Weigh the boat with sample. The difference between the dry boat with sample before LOI and the boat with sample after LOI is the actual weight loss.
5. $(\text{LOI weight loss/dry sample weight}) \times 100 = \% \text{ LOI}$

NOTES

- a. To avoid multi-laboratory differences in LOI values, the labs involved must use exactly the same procedure.
- b. Sample preparation must be kept the same. You cannot have one lab running a LOI on ground material while another lab runs LOI on unground material.
- c. Beware of low quality porcelain boats. They have a tendency to break or chip when placed in the 950 degrees C. area of the tube furnace.
- d. Argon or Nitrogen flowrate through the tube must also be kept the same for all labs.