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ASTM C-114 Qualification Test Method to Improve Analytical Results Using the Fused Beads Technique

International Cement Microscopy Association 2001

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ABSTRACT

The 21st century offers a host of new challenges, especially in analytical chemistry, where quality standards are becoming increasingly demanding. To meet these challenges, the Cement industry was among the first to have understood the potential of XRF analysis using fused bead technique. This method is well established as a quick and reliable method, which allows the obtaining of highly accurate and precise results. The purpose of this paper is to show how to qualify for ASTM C-114 certification using fused bead technique for chemical analysis of hydraulic cement. Important considerations and the most common problems that may be encountered will be discussed. Particular attention will be given to several reproducibility issues, often overlooked and misunderstood.

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1. INTRODUCTION

ASTM C-114 is a quality standard that is now widely known in the chemical analysis of the hydraulic cement area. Its requirements are simple but passing the test itself is not always that easy since both precision and accuracy have to be maintained. For many years, causes affecting precision and accuracy when using the fused bead technique were not completely understood. Sometimes, unsatisfactory results were attributed to multi-position fusion instruments, even though the causes were related to less obvious factors. This situation can now be controlled better since recent experimental results have clarified most of these issues.

This paper shows several experimental results demonstrating the effects of different overlooked parameters having a major impact on precision and accuracy. Knowing them and understanding how to control them allow analysts to fully benefit from the precision offered by fusion. This also facilitates ASTM C-114 qualification. Among various factors, the following will be treated:

- The mold effect;
- The non-wetting agent (NWA);
- Temperature and flux.

2. EXPERIMENTAL

2.1. The mold effect

It is well known that mold surface has a certain effect on results. Invariably, the mold surfaces change after a certain amount of use. Even though this phenomenon is known, the importance of it is not clear. To what extent do the mold surfaces affect precision? Several experiments have been carried out in order to clarify and quantify this effect. The results obtained have been very surprising and because of its importance, the mold effect has been given much consideration in this paper.

Figures 1 and 2 show common mold imperfections. These imperfections are the result of hundreds of heating and cooling cycles cracked beads, crystallized beads, etc. These represent normal surface wear when the same platinum molds are used over a long period of time. One characteristic of platinum molds is that their prints are fully reproduced on the glass disks during cooling. These imperfections are printed on the surface of the glass disks, where the X-ray beam strikes during the analysis (Fig 3).

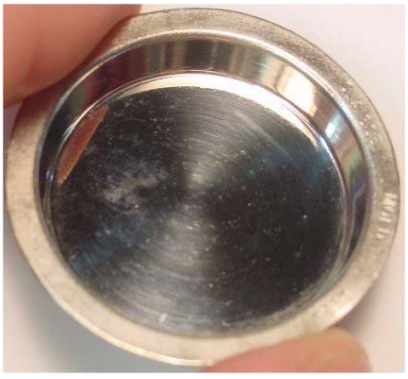


Fig 1. Example of mold imperfections on a recent mold

Fig 2. Example of mold imperfections on an old mold

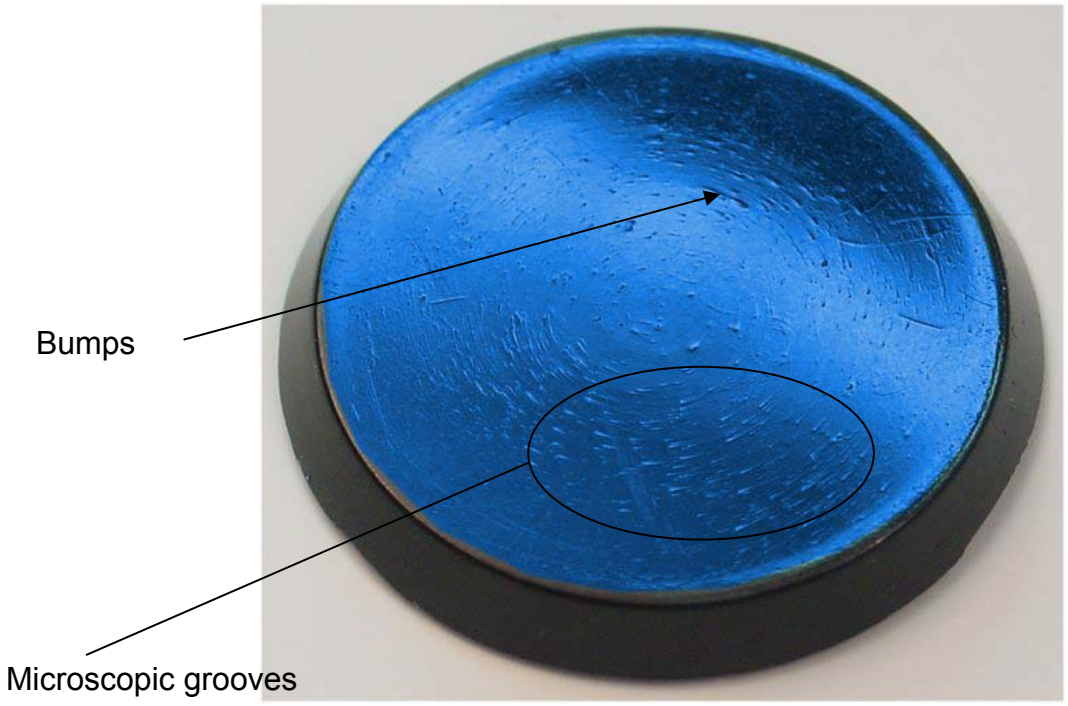


Fig 3. Mold prints on a 32-mm glass disk

An experience has been carried out to demonstrate the impact of the molds on the analytical results. Figures 4, 5 and 6 show the actual molds used in the experiment. One was new, the other one was moderately used and the last one was very old and scratched. A Claisse M4-fluxer was used to process the fusions and a PW 2600 XRF instrument was used to make the analysis. Nine fusions were performed. After each fusion, the molds were permuted from one position to another in order to ensure that the experiment would show the mold effect and not the burner effect, if any.

Fig 4. New mold

Fig 5. Moderately used mold

Fig 6. Extremely used mold

% CaO		% CaO		% CaO
64.07		63.62		63.22
64.22		63.72		63.05
64.18		63.72		63.05
63.96		63.58		63.15
64.07		63.50		63.42
64.18		63.54		63.16
64.11		63.74		63.17
64.16		63.43		63.14
64.04		63.59		63.19
64.11	**Average**	**63.60**	**Average**	**63.17**
0.08	**Std**	**0.11**	**Std**	**0.11**
0.13	**RSD%**	**0.17**	**RSD%**	**0.17**

There is no need to compare results from a new mold to those of an extremely used one to notice significant mold effects. These effects are significantly noticeable even when a new mold is compared to a moderately used one. Systematically, the flatter and smoother the surface is, the higher the intensities are. The new mold shows the highest intensities while the older mold shows the lowest. Still, the results measured from each mold are very repeatable, even from the old scratched one. When using the same mold, the repeatability is very good;

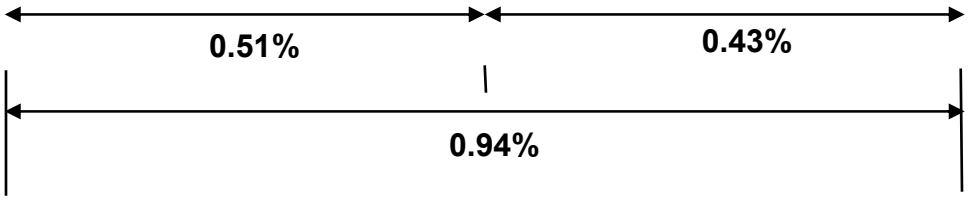
all of them show a RSD lower than 0.17 %. However, when comparing a mold with one that has different surface wear, the deviation can become quite significant.

Figures 7, 8 and 9 show the absolute %CaO difference between the molds. There is a 0.51% difference between the new and the moderately used mold and a 0.43% difference between the moderately used and the old mold. This means an absolute difference of 0.94% between the new and the old mold. That is obviously quite significant. This experiment demonstrates that the mold effect must never be underestimated. It is fortunately possible to limit this effect by taking good care of the platinumware and by avoiding mixing new and old platinum molds together. Once in a while, fusing an internal standard and checking the results on a control card can monitor the «mold drift». It is therefore possible to follow the effect and act upon it when the deviation between molds becomes too high. Obviously, XRF calibration should always be performed using perfect molds.

Fig 7. Mold never used

Fig 8. Mold moderately used

Fig 9. Mold extremely used



In short:

- The mold is often responsible for many analytical repeatability issues;
- Individually, each mold gives repeatable intensities;
- The older the mold, the lower the intensities;
- The more different the molds, the greater the differences between the results;
- Mold effect is often mistaken with position effect in multi-position fluxers;
- Mold effect can be monitored and limited to a minimum.

2.2. The non-wetting agent

Depending on their composition, liquid or solid borate glasses have a variable tendency to stick to the platinumware. In most cases though, the use of a non-wetting (releasing) agent is beneficial or even essential. Halogens are the only elements known to be efficient as non-wetting agents, but iodine and bromine are the only two that are practical. They can be added as alkali salts or solutions into the mixture before the fusion. They can also be directly integrated (prefused) to the flux. The most common non-wetting agents used are: LiI, KI, NaI, NH_4I, NaBr, KBr, NH_4Br and LiBr.

2.2.1. Volatility

Volatility of halogenides is high and increases rapidly with the atomic number. Iodides are the most volatile. Less than 5% of the original amount may remain in the melt after a normal fusion. Bromides are more stable but still very volatile.

2.2.2. Oxidability

Non-wetting agents are good reducers and can therefore easily be oxidized and decomposed. A typical oxidation reaction is shown below:

$$2\ LiBr + ½\ O_{2\ (gas)} \rightarrow Li_2O + Br_{2\ (gas)}$$

The oxygen contained in the atmosphere is sufficient to produce such a reaction. Obviously, any oxidizers used during fusion will also lead to the decomposition of the non-wetting agent.

2.2.3. Effect on XRF intensities

Non-wetting agents (NWA) are made of materials that absorb X-ray radiations and interfere during XRF analysis. In order to show the effect on precision, two series of 14 glass disks have been made using the same cement sample varying only the fusion recipe. One fusion did not use any oxidizer while the other used lithium nitrate as an oxidizing agent. In both cases, the same amount of non-wetting agent was accurately added. The results are shown in Table 1 (%CaO shown in ascending order for an easier comparison). It shows the measured %CaO and the corresponding Br Kα intensities from the added non-wetting agent.

Table 1. Results From Trials on 14 Replicates With 50 mg LiBr as NWA

	Fusion without oxidizer		Fusion with oxidizer	
	CaO %	Br Kcps	CaO %	Br Kcps
	64.88	84.96	64.42	74.15
	64.98	90.07	64.65	74.94
	64.99	89.31	64.72	74.47
	65.11	87.84	64.76	78.46
	65.13	89.67	64.80	66.02
	65.14	88.62	64.87	64.81
	65.14	87.76	64.95	61.75
	65.14	86.67	64.96	43.30
	65.16	85.96	65.17	46.30
	65.19	87.52	65.18	45.44
	65.26	90.04	65.26	55.59
	65.28	88.21	65.27	40.42
	65.32	86.36	65.34	48.52
	65.32	86.33	65.61	26.27
Average	**65.15**	**87.81**	**65.00**	**57.17**
STD	**0.13**	**1.61**	**0.32**	**15.87**
%RSD	**0.20**	**1.84**	**050.**	**27.76**

2.2.3.1. Fusion without an oxidizer

The results are very explicit. The fusion without an oxidizer shows good precision for %CaO (0.20 %RSD). The Br intensities are also relatively precise (1.84 %RSD) if we consider that bromides are very volatile.

2.2.3.2. Fusion with an oxidizer (NWA partly decomposed by the oxidizer)

The fusion that used an oxidizer shows a precision 2.5 times poorer on CaO (0.50 %RSD) and 15 times poorer on Br intensities (27.76 %RSD). In fact, because the oxidizer decomposes bromine during fusion and this decomposition is not quantitatively reproducible; the amount of bromide remaining in the glass disk is never the same.

The interesting phenomenon is the correlation between %CaO and Br intensities. The higher the Br intensities are, the lower the %CaO is. The lower the Br intensities are, the higher the %CaO is. The correlation is shown on Figure 10. The effect is almost perfectly linear.

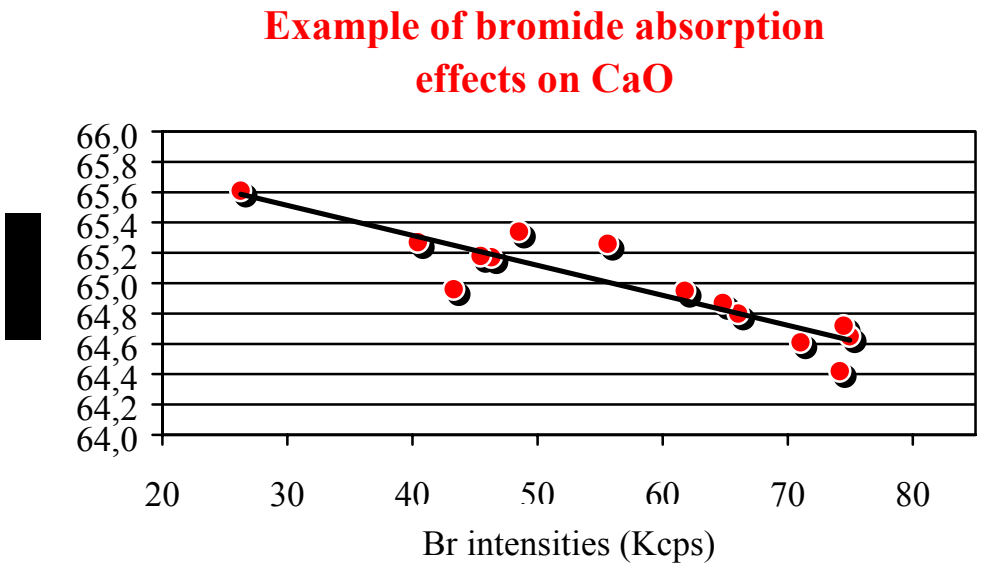


Fig 10. Relation Between %CaO and Br Intensities

It is clear that Br affects the %CaO, which is normal since Br is a good X-ray absorber. Thus, if Br is unstable during fusion, the concentration of CaO will also vary, yielding to lower precision. The same effect has been observed on other oxides and other non-wetting agents producing this same kind of effect. Consequently, the non-wetting agent plays a significant role regarding precision and accuracy. Thus, NWA must be stabilized as much as possible.

To conclude, the non-wetting agent is easily oxidized during fusion, extremely volatile and its losses are not reproducible, even when using the same fusion program, recipe and conditions. In addition, it absorbs X-ray radiation, and thus interferes with other elements. To avoid these problems, it must be added very

accurately. Quantities must be reduced to a minimum. We recommend customers to use fused fluxes with integrated NWA.

2.2. Temperature and flux

The fusion temperature and the flux composition are two important factors that play a role in precision and accuracy, as they will or will not stabilize the sample in the flux. These factors are now better known, and there are already a few papers[1] and books[2] that contain information on the subject. They will therefore be discussed briefly.

2.2.1. The temperature

The fusion temperature should always be kept as low as possible in order to avoid losses by volatilization of important elements from the sample or the flux. A temperature between 1000 and 1050ºC is sufficient to melt borate fluxes and dissolve oxides. In a fusion context, the flux must melt and then, the sample will dissolve into the molten flux. It is not necessary to reach a temperature that would melt the sample. As long as the flux itself is a liquid, the sample dissolves in it, just like sugar dissolves in coffee without having to melt the sugar. It is also important to note that some species, such as SO_3 and alkaline oxides, are somewhat volatile and tend to be lost by volatilization if the temperature is too high.

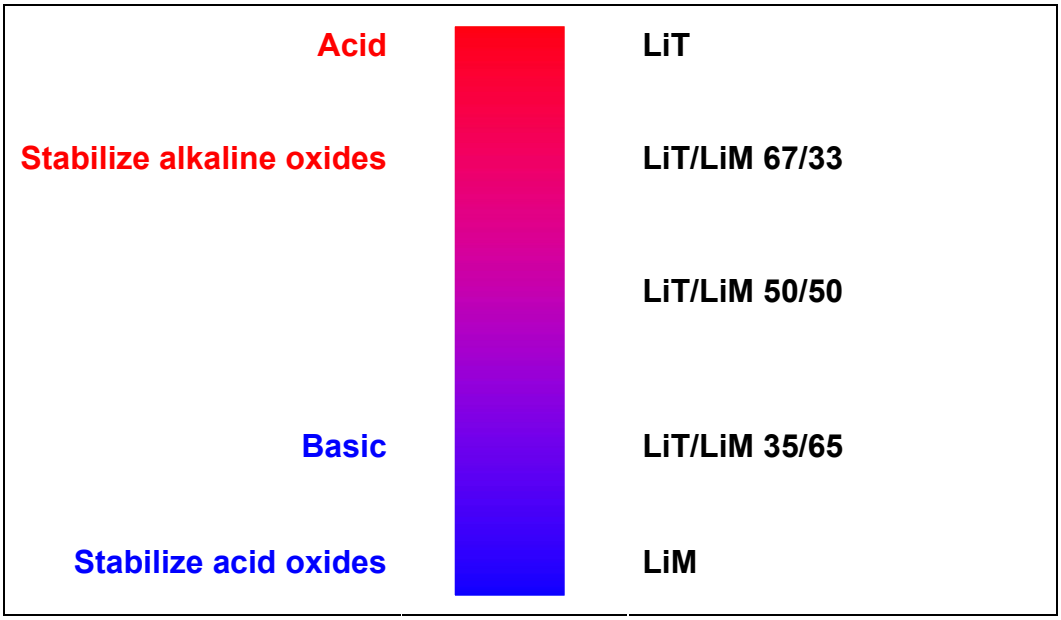
Therefore, short low temperatures fusions (< 1050ºC) are recommended because only the flux needs to melt, not the sample.

2.2.2. The flux

The flux composition plays a significant role in the sample dissolution. Volatility of certain species (such as SO_3) is also related to the acidity of the flux. In general, acid oxides like SiO_2 will dissolve better in a basic flux, while basic oxides like CaO will dissolve better in an acidic flux. Several researchers also demonstrated that acidic fluxes may lead to the reduction of SO_3 into gaseous SO_2.

In short:

- Flux selection plays an important stabilizing role;
- The solubility of oxides in the fluxes varies with the flux composition;
- The melting point of the sample/flux mixture varies with the flux composition.



3. CONCLUSION

Knowing and controlling the important factors described in this paper, ASTM C-114 qualification has been tried and succeeded using a PW 2600 spectrometer and a Claisse M4-fluxer. Three new molds were used. The qualification is summarized in Table 2 and the details of the results are shown in Tables 3-9. This experience, performed at Ciment Quebec Inc., demonstrates and confirms that the fusion technique using multi-position flame fluxer yields very high quality results, and thus, qualifies for ASTM C-114.

Table 2. ASTM C-114 Summary

Element	Allowed		SRM 1880		SRM 1881		SRM 1884		SRM 1886		SRM 1887		SRM 1888		SRM 1889		Final success
	Precision	Accuracy	Prec.	Acc.	Prec.	Acc.	Prec.	Acc.	Prec.	Acc.	Prec.	Acc.	Prec.	Acc.	Prec.	Acc.	
SiO_2	0,16	0,20	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
Al_2O_3	0,20	0,20	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
Fe_2O_3	0,10	0,10	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
CaO	0,20	0,30	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
MgO	0,16	0,20	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
SO_3	0,10	0,10	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
Na_2O	0,03	0,05	✔	✔	✔	x	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
K_2O	0,03	0,05	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
TiO_2	0,02	0,03	✔	✔	✔	✔	✔	✔	✔	x	✔	✔	✔	✔	✔	✔	Passed
P_2O_5	0,03	0,03	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
ZnO	0,03	0,03	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
Mn_2O_3	0,03	0,03	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed
Cl	0,02	--	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	Passed

Legend:

✔	Passed
×	Failed but remaining difference is lower than twice permissible value, so acceptable
Prec.	Precision
Acc.	Accuracy

Table 3. ASTM C-114 Results Obtained For SRM 1880

SRM 1880										
Element	Certified values	Experimental values			Precision			Accuracy		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	19,82	19,93	19,87	19,90	0,06	0,16	Passed	0,08	0,2	Passed
Al_2O_3	5,03	4,99	5,01	5,00	0,02	0,20	Passed	0,03	0,2	Passed
Fe_2O_3	2,91	2,96	2,95	2,96	0,01	0,10	Passed	0,04	0,10	Passed
CaO	63,14	63,12	63,07	63,10	0,05	0,20	Passed	0,05	0,3	Passed
MgO	2,69	2,65	2,65	2,65	0,00	0,16	Passed	0,04	0,2	Passed
SO_3	3,37	3,29	3,31	3,30	0,02	0,10	Passed	0,07	0,1	Passed
Na_2O	0,28	0,32	0,31	0,32	0,01	0,03	Passed	0,04	0,05	Passed
K_2O	0,91	0,92	0,92	0,92	0,00	0,03	Passed	0,01	0,05	Passed
TiO_2	0,23	0,26	0,26	0,26	0,00	0,02	Passed	0,03	0,03	Passed
P_2O_5	0,29	0,29	0,29	0,29	0,00	0,03	Passed	0,00	0,03	Passed
ZnO	0,01	0,01	0,01	0,01	0,00	0,03	Passed	0,00	0,03	Passed
Mn_2O_3	0,08	0,08	0,08	0,08	0,00	0,03	Passed	0,00	0,03	Passed
Cl	0,02	0,02	0,01	0,02	0,01	0,02	Passed	0,01	--	Passed

Table 4. ASTM C-114 Results Obtained For SRM 1881

SRM 1881										
Element	Certified values	Experimental values			Precision			Accuracy		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	22,25	22,29	22,22	22,26	0,07	0,16	Passed	0,00	0,2	Passed
Al_2O_3	4,16	4,22	4,24	4,23	0,02	0,20	Passed	0,07	0,2	Passed
Fe_2O_3	4,68	4,73	4,74	4,74	0,01	0,10	Passed	0,06	0,10	Passed
CaO	58,67	58,56	58,37	58,47	0,19	0,20	Passed	0,20	0,3	Passed
MgO	2,63	2,71	2,69	2,70	0,02	0,16	Passed	0,07	0,2	Passed
SO_3	3,65	3,62	3,62	3,62	0,00	0,10	Passed	0,03	0,1	Passed
Na_2O	0,04	0,11	0,14	0,13	0,03	0,03	Passed	0,09	0,05	Failed
K_2O	1,17	1,18	1,18	1,18	0,00	0,03	Passed	0,01	0,05	Passed
TiO_2	0,25	0,24	0,22	0,23	0,02	0,02	Passed	0,02	0,03	Passed
P_2O_5	0,09	0,11	0,12	0,12	0,01	0,03	Passed	0,03	0,03	Passed
ZnO	0,01	0,01	0,01	0,01	0,00	0,03	Passed	0,00	0,03	Passed
Mn_2O_3	0,26	0,25	0,25	0,25	0,00	0,03	Passed	0,01	0,03	Passed
Cl	0,01	0,00	0,00	0,00	0,00	0,02	Passed	0,01	--	Passed

Table 5. ASTM C-114 Results Obtained For SRM 1884

SRM 1884										
Element	Certified values	Experimental values			Precision			Accuracy		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	23,19	23,26	23,31	23,29	0,05	0,16	Passed	0,09	0,2	Passed
Al_2O_3	3,31	3,35	3,37	3,36	0,02	0,20	Passed	0,05	0,2	Passed
Fe_2O_3	3,30	3,34	3,34	3,34	0,00	0,10	Passed	0,04	0,10	Passed
CaO	64,01	64,00	63,96	63,98	0,04	0,20	Passed	0,03	0,3	Passed
MgO	2,32	2,39	2,39	2,39	0,00	0,16	Passed	0,07	0,2	Passed
SO_3	1,67	1,67	1,66	1,67	0,01	0,10	Passed	0,00	0,1	Passed
Na_2O	0,13	0,17	0,18	0,18	0,01	0,03	Passed	0,05	0,05	Passed
K_2O	0,51	0,52	0,52	0,52	0,00	0,03	Passed	0,01	0,05	Passed
TiO_2	0,16	0,18	0,18	0,18	0,00	0,02	Passed	0,02	0,03	Passed
P_2O_5	0,12	0,12	0,13	0,13	0,01	0,03	Passed	0,01	0,03	Passed
ZnO	0,02	0,01	0,01	0,01	0,00	0,03	Passed	0,01	0,03	Passed
Mn_2O_3	0,11	0,10	0,11	0,11	0,01	0,03	Passed	0,00	0,03	Passed
Cl	0,00	0,00	0,00	0,00	0,00	0,02	Passed	0,00	--	Passed

Table 6. ASTM C-114 Results Obtained For SRM 1886

SRM 1886										
Element	Certified values	Experimental values			Precision			Accuracy		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	22,53	22,56	22,53	22,55	0,03	0,16	Passed	0,02	0,2	Passed
Al_2O_3	3,99	4,06	4,07	4,07	0,01	0,20	Passed	0,07	0,2	Passed
Fe_2O_3	0,31	0,29	0,29	0,29	0,00	0,10	Passed	0,02	0,10	Passed
CaO	67,43	67,52	67,43	67,48	0,09	0,20	Passed	0,04	0,3	Passed
MgO	1,60	1,71	1,69	1,70	0,02	0,16	Passed	0,10	0,2	Passed
SO_3	2,04	1,95	1,96	1,96	0,01	0,10	Passed	0,09	0,1	Passed
Na_2O	0,02	0,07	0,07	0,07	0,00	0,03	Passed	0,05	0,05	Passed
K_2O	0,16	0,16	0,16	0,16	0,00	0,03	Passed	0,00	0,05	Passed
TiO_2	0,19	0,23	0,22	0,23	0,01	0,02	Passed	0,04	0,03	Failed
P_2O_5	0,03	0,03	0,03	0,03	0,00	0,03	Passed	0,01	0,03	Passed
ZnO	<0,01	0,01	0,01	0,01	0,00	0,03	Passed	0,00	0,03	Passed
Mn_2O_3	0,01	0,01	0,01	0,01	0,00	0,03	Passed	0,00	0,03	Passed
Cl	0,00	0,00	0,00	0,00	0,00	0,02	Passed	0,00	--	Passed

Table 7. ASTM C-114 Results Obtained For SRM 1887

SRM 1887										
Element	**Certified values**	**Experimental values**			**Precision**			**Accuracy**		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	19,98	20,09	20,11	20,10	0,02	0,16	**Passed**	0,12	0,2	**Passed**
Al_2O_3	5,59	5,65	5,64	5,65	0,01	0,20	**Passed**	0,05	0,2	**Passed**
Fe_2O_3	2,16	2,19	2,17	2,18	0,02	0,10	**Passed**	0,02	0,10	**Passed**
CaO	62,88	63,10	62,93	63,02	0,17	0,20	**Passed**	0,13	0,3	**Passed**
MgO	1,26	1,40	1,41	1,41	0,01	0,16	**Passed**	0,15	0,2	**Passed**
SO_3	4,61	4,52	4,52	4,52	0,00	0,10	**Passed**	0,09	0,1	**Passed**
Na_2O	0,10	0,15	0,15	0,15	0,00	0,03	**Passed**	0,05	0,05	**Passed**
K_2O	1,27	1,29	1,29	1,29	0,00	0,03	**Passed**	0,02	0,05	**Passed**
TiO_2	0,27	0,29	0,30	0,30	0,01	0,02	**Passed**	0,03	0,03	**Passed**
P_2O_5	0,08	0,06	0,06	0,06	0,00	0,03	**Passed**	0,02	0,03	**Passed**
ZnO	0,01	0,01	0,01	0,01	0,00	0,03	**Passed**	0,00	0,03	**Passed**
Mn_2O_3	0,07	0,07	0,06	0,07	0,01	0,03	**Passed**	0,01	0,03	**Passed**
Cl	0,01	0,01	0,01	0,01	0,00	0,02	**Passed**	0,00	--	**Passed**

Table 8. ASTM C-114 Results Obtained For SRM 1888

SRM 1888										
Element	**Certified values**	**Experimental values**			**Precision**			**Accuracy**		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	20,86	20,87	20,97	20,92	0,10	0,16	**Passed**	0,06	0,2	**Passed**
Al_2O_3	5,35	5,39	5,40	5,40	0,01	0,20	**Passed**	0,04	0,2	**Passed**
Fe_2O_3	3,18	3,22	3,26	3,24	0,04	0,10	**Passed**	0,06	0,10	**Passed**
CaO	63,78	63,90	64,08	63,99	0,18	0,20	**Passed**	0,21	0,3	**Passed**
MgO	0,71	0,86	0,88	0,87	0,02	0,16	**Passed**	0,16	0,2	**Passed**
SO_3	3,16	3,06	3,07	3,07	0,01	0,10	**Passed**	0,10	0,1	**Passed**
Na_2O	0,14	0,18	0,18	0,18	0,00	0,03	**Passed**	0,04	0,05	**Passed**
K_2O	0,56	0,57	0,57	0,57	0,00	0,03	**Passed**	0,01	0,05	**Passed**
TiO_2	0,29	0,32	0,32	0,32	0,00	0,02	**Passed**	0,03	0,03	**Passed**
P_2O_5	0,09	0,10	0,10	0,10	0,00	0,03	**Passed**	0,02	0,03	**Passed**
ZnO	0,01	0,01	0,01	0,01	0,00	0,03	**Passed**	0,00	0,03	**Passed**
Mn_2O_3	0,03	0,02	0,02	0,02	0,00	0,03	**Passed**	0,01	0,03	**Passed**
Cl	0,02	0,02	0,02	0,02	0,00	0,02	**Passed**	0,01	--	**Passed**

Table 9. ASTM C-114 Results Obtained For SRM 1889

SRM 1889										
Element	**Certified values**	**Experimental values**			**Precision**			**Accuracy**		
		1st result	2nd result	Average (1st and 2nd results)	Absolute difference (1st and 2nd results)	Allowed difference between duplicates (1st and 2nd results)	Success	Absolute difference between certified value and duplicates average (1st and 2nd results)	Allowed maximum difference between certified value and duplicates average (1st and 2nd results)	Success
SiO_2	20,44	20,40	20,47	20,44	0,07	0,16	**Passed**	0,01	0,2	**Passed**
Al_2O_3	5,61	5,64	5,66	5,65	0,02	0,20	**Passed**	0,04	0,2	**Passed**
Fe_2O_3	2,67	2,68	2,67	2,68	0,01	0,10	**Passed**	0,00	0,10	**Passed**
CaO	65,08	65,17	65,21	65,19	0,04	0,20	**Passed**	0,11	0,3	**Passed**
MgO	1,38	1,50	1,51	1,51	0,01	0,16	**Passed**	0,13	0,2	**Passed**
SO_3	2,68	2,64	2,62	2,63	0,02	0,10	**Passed**	0,05	0,1	**Passed**
Na_2O	0,11	0,16	0,16	0,16	0,00	0,03	**Passed**	0,05	0,05	**Passed**
K_2O	0,32	0,32	0,32	0,32	0,00	0,03	**Passed**	0,00	0,05	**Passed**
TiO_2	0,21	0,24	0,24	0,24	0,00	0,02	**Passed**	0,03	0,03	**Passed**
P_2O_5	0,15	0,18	0,18	0,18	0,00	0,03	**Passed**	0,03	0,03	**Passed**
ZnO	<0,01	0,01	0,01	0,01	0,00	0,03	**Passed**	0,00	0,03	**Passed**
Mn_2O_3	0,24	0,23	0,24	0,24	0,01	0,03	**Passed**	0,01	0,03	**Passed**
Cl	0,00	0,00	0,01	0,01	0,01	0,02	**Passed**	0,00	--	**Passed**

4. REFERENCES

1. Fernand Claisse (1995), *Glass Disks and Solutions by Fusion in Borates for Users of Claisse Fluxers*, 2ND Edition.

2. Graham Oliver and Harry Bennett (1992), *XRF Analysis of Ceramics, Minerals and Allied Materials.*