The Use of Quartz Crucibles in Place of Platinum Crucibles in the VITRIOX® Electrical Fusion Machine

Introduction

Sample fusion of non-metal solids is conducted routinely in daily analytical life. This report presents the use of quartz crucibles as an alternative to platinum crucibles for automatic fusion processes.

The fusion of samples that contain the so-called platinum poisons (sulfides, metals – especially aluminum – carbides, nitrides) frequently leads to considerable damage to the platinum/gold crucibles used.

For this reason, the wish for crucibles made of alternative materials is often expressed. The use of quartz crucibles as an alternative was examined together with the Hochschule Rhein-Waal in Kleve, Germany.

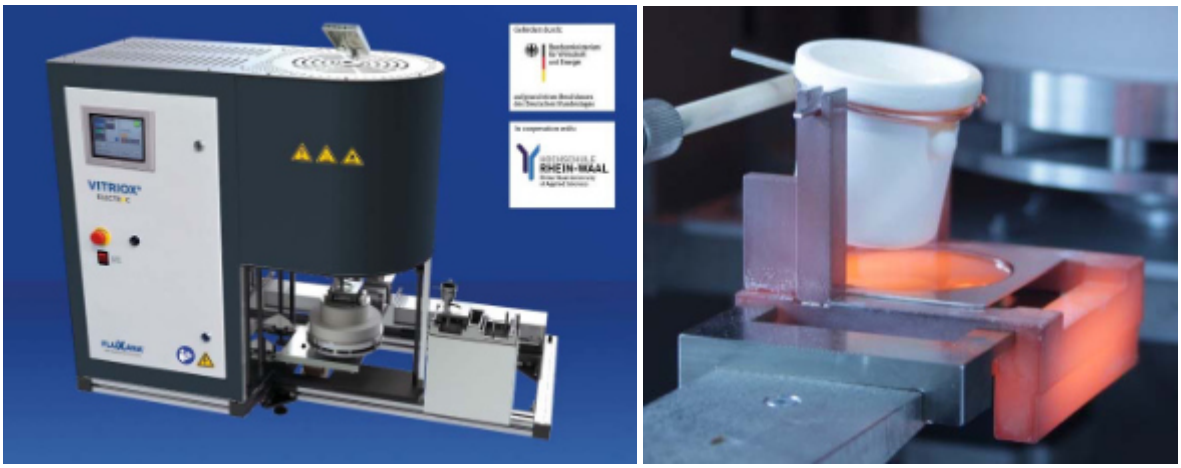

Figure 1: Electrical fusion machine with two stations; quartz crucible in use.

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For the practical application, a calibration based on the **application set "Slags and Dusts"** using quartz crucibles was conducted. This application is characterized by samples that contain reduced components such as sulfides, nitrides, carbides, carbon, metals or similar constituents that are oxidized before the actual fusion using a strong oxidizing agent so that the fusion can be successfully conducted.

Sample Preparation

Sample	0.4 g
Flux FX-X65*	9 g
Oxidizing agent FX-OXY7**	1 g

*66% Lithium tetraborate + 34% Lithium metaborate
**Strontium nitrate

	Slags and Dusts	
Component	Min %	Max %
Al_2O_3	0	78,0
CaO	0	98,0
Cr_2O_3	0	18,0
Cu	0	0,2
Fe	0	70,0
K_2O	0	2,5
MgO	0	22,0
Mn	0	10,0
Na_2O	0	3,0
Ni	0	0,04
P_2O_5	0	16,0
Pb	0	4,0
S	0	1,5
SiO_2	0	60,0
TiO_2	0	13,0
V_2O_5	0	1,0
Zn	0	20,0

Table 1: Calibration range for the application set Slags and Dusts.

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Procedure

The flux material is placed in the quartz crucible. The sample is mixed with the oxidizing agent and placed on top of the flux material. Then the sample is fused at 1200 °C in the VITRIOX fusion machine.

Calibration

Table 1 gives the calibration range covered by the standard samples for the application set. Figure 2 shows by way of example the calibration for the element calcium.

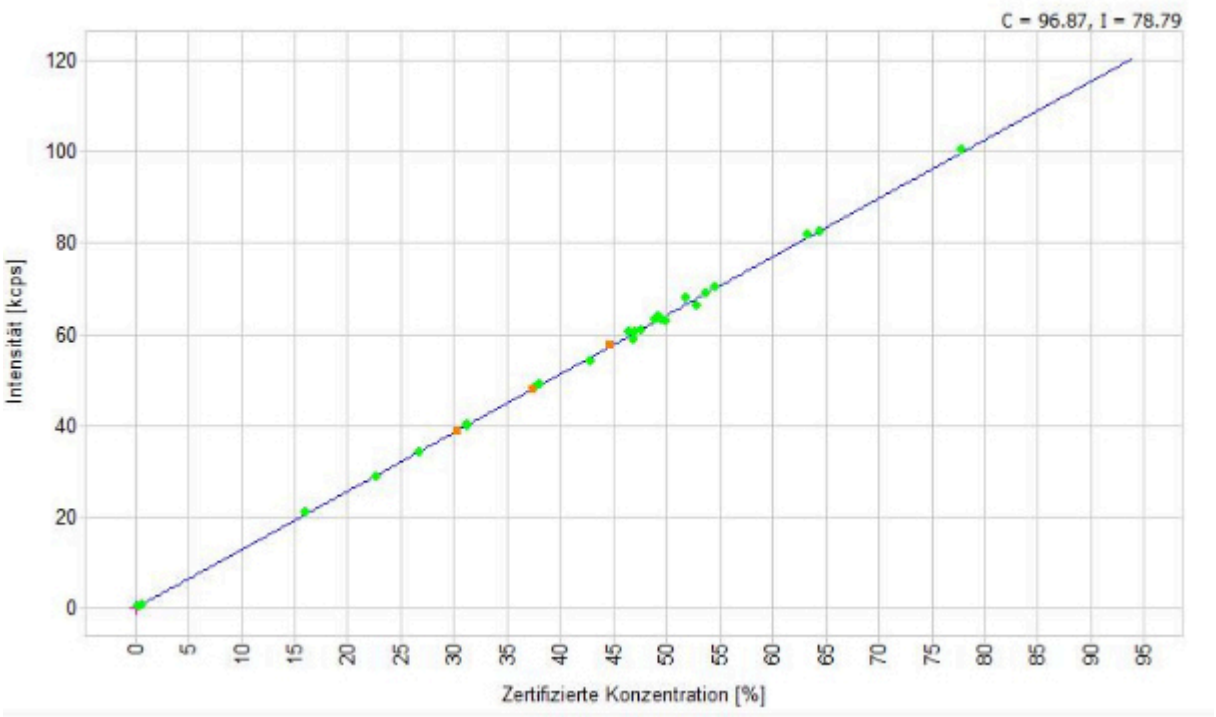

Figure 2: Calibration of CaO using CRM based on slag samples fused in quartz crucibles. Calibration error SEE = 0.5%. The Ca Ka line was normalized to the Sr La line.

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Results

Table 2: Analysis of 3 CRM control samples for validation of the "Slags and Dusts" fused in quartz crucibles calibration.

BS 101/5	Al2O3	CaO	Cr2O3	Fe	K2O	MgO	Mn	Na2O	P2O5	S	SiO2	TiO2
cert. %	0.57	46.00		19.20	0.01	5.50	4.42	0.04	0.71	0.12	14.90	1.10
Prep# 1	0.77	46.07	0.14	18.96	0.01	5.56	4.36	0.06	0.69	0.12	11.77	1.07
Prep# 2	0.71	46.19	0.14	19.03	0.00	5.49	4.38	0.14	0.69	0.12	11.67	1.01
Mean	**0.74**	**46.13**	**0.14**	**19.00**	**0.01**	**5.53**	**4.37**	**0.10**	**0.69**	**0.12**	**11.72**	**1.04**
Diff	0.17	0.13		-0.20	0.00	0.03	-0.04	0.06	-0.02	0.00	-3.18	-0.06

BS Slag 1	Al2O3	CaO	Cr2O3	Fe	K2O	MgO	Mn	Na2O	P2O5	S	SiO2	TiO2
cert. %	18.50	30.20		0.28	0.36	11.01	0.86	0.20		1.80	36.70	0.42
Prep# 1	18.29	29.94	<	0.31	0.37	10.96	0.89	0.24	0.04	1.17	36.87	0.41
Prep# 2	18.14	29.80	<	0.30	0.37	10.95	0.89	0.39	0.02	1.13	37.07	0.42
Mean	**18.22**	**29.87**		**0.31**	**0.37**	**10.96**	**0.89**	**0.32**	**0.03**	**1.15**	**36.97**	**0.42**
Diff	-0.29	-0.33		0.03	0.01	-0.05	0.03	0.12		-0.65	0.27	-0.01

FLX-CRM 100	Al2O3	CaO	Cr2O3	Fe	K2O	MgO	Mn	Na2O	P2O5	S	SiO2	TiO2
cert. %	5.54	64.51	0.01	1.75	0.82	1.47	0.05	0.23	0.17	1.19	20.89	0.28
Prep# 1	5.66	64.42	0.01	1.87	0.84	1.52	0.05	0.09	0.17	1.16	21.33	0.27
Prep# 2	5.52	63.97	0.01	1.85	0.84	1.51	0.05	0.18	0.14	1.14	21.97	0.25
Mean	**5.59**	**64.20**	**0.01**	**1.86**	**0.84**	**1.52**	**0.05**	**0.14**	**0.16**	**1.15**	**21.65**	**0.26**
Diff	0.05	-0.32	0.00	0.11	0.02	0.05	0.00	-0.10	-0.01	-0.04	0.76	-0.02

The results demonstrate excellent agreement between the certified and the analyzed concentrations for almost all elements. Because SiO2 cannot be directly determined due to contamination by the quartz crucible, the given SiO2 concentration is calculated from the difference to 100%.

Summary

The results presented here clearly demonstrate that the use of quartz crucibles is a serious alternative to the use of platinum crucibles. Samples that contain platinum poisons can be fused without causing damage to the crucible. The disadvantage that samples are contaminated with SiO2 can be compensated for by the addition of an internal standard.

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References

[1] Rainer Schramm, X-Ray Fluorescence Analysis: Practical and Easy - 2nd edition, FLUXANA (2017).

[2] www.fluxana.com