

# XRF Scientific Ltd

## FERRO-ALLOYS

### PRINCIPLES OF THE TECHNIQUE

Direct fusion of ferro-alloys in molten lithium tetraborate is not possible. The main reasons are that ferro-alloys will not readily dissolve and they attack and destroy the crucible metal. The present technique overcomes both problems to achieve the successful fusion of ferro-alloys.

All ferro-alloys to be fused using this technique must finely grind in order to obtain particle size, i.e. **less than 80 µm**. This fine size is achieved by crushing a portion of the ferro-alloy, of size 5 mm or less, in a laboratory swing/vibratory grinding mill. The grinding container and components are made from hardened alloy steel.

(Wolfram-carbide or Agate bowls)

#### Principles/theory

a) in a muffle furnace

A solid protective wall is prepared, from molten lithium tetraborate, inside the crucible. The finely ground ferroalloy perfectly mixed with the OXYDANT MIX is added carefully to the crucible, to form a thin layer on the solid wall. The crucible is placed in the furnace, set at 800°C, for a minimum of 3 h. The melting point of lithium tetraborate is 920 °C and therefore the wall remains solid at 800°C (note: the ferro-alloy is not in contact with the crucible metal). During this period, preliminary oxidation takes place. The degree of oxidation depends on the type of ferroalloy and particle size. This preliminary oxidation stage is important. Furnace design ensures a good airflow through the furnace to provide the necessary oxygen. If time permits, the samples are allowed to oxidise overnight. This maximises the analyst's time.

After the preliminary oxidation/dissolution process, the crucible is removed from the furnace and the crucible is transferred to a furnace, set at 1200°C, for 10 min. All the ferro-alloy metal has been oxidised and dissolved in the OXYDANT MIX and therefore a normal fusion can take place without damage to the crucible. After the fusion period, the melt is poured into a preheated casting dish.

### EXPERIMENTAL

Weigh 8.000 g of dried lithium tetraborate and transfer it into the crucible. Place the crucible in the furnace, set at 1200°C, for 3 min. After 3 min, using tongs, remove the crucible containing the molten lithium tetraborate. Holding the crucible firmly with the tongs, slowly rotate the crucible. Continue this crucible rotation to form a layer of lithium tetraborate half way up the inner crucible wall. As the lithium tetraborate cools and solidifies, a wall is formed of uniform thickness. When the wall has solidified, allow the crucible to cool.

Weigh 0.4000-g –0.4000 g of dried ferro-alloy. Mix well the ferro-alloy with 3.000 g of **OXYDANT MIX**. Add the mix of oxidant and ferroalloy to the cool crucible to form a layer in its base. Place the crucible in the furnace, set at 800°C, for a minimum of 3 h-4 h. Good airflow through the furnace is essential.

XRF Scientific Ltd  
1/12 Farrall Road  
Midvale, WA 6056

Tel: 61 8 9274 9100 Fax 61 8 9274 9144

[www.xrfscientific.com](http://www.xrfscientific.com) e-mail: [sales@xrfscientific.com](mailto:sales@xrfscientific.com)

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After this oxidation/dissolution period, the crucible is transferred to a **PHOENIX Fusion machine** set at 1200°C. The total fusion time is 10 min. At 4 and 8 min into the fusion process, the crucible is agitated to ensure that the melt becomes homogeneous. After 10 min the melt is poured into a preheated casting dish.

When the casting dish is cold, the bead is removed and transfers to the spectrometer using a suitable calibration.

**Ferro-chromium is the most difficult ferro-alloy to oxidise and therefore 0.1000 g of sample is used.**

**Other ferro-alloys: 0.4000 g- 0.2500 g of sample could be used**

## METHOD WITH THE PHOENIX FUSION MACHINE

Weight ca 0.250 g of ferro-alloy sample and mix it carefully with 3 g of OXYDANT MIX.

Weight 9.000 g of lithium tetraborate and place approximately 7 g in the crucible.

Melt the lithium tetraborate until mobile liquid and with the help of platinum tip tongs swirls the crucible to form a protective wall. Cool the crucible until the lithium tetraborate is solidifying.

Add the mixture ferro-alloy sample with Oxidant mix into the crucible and cover it with the remaining flux.

On the premelt program (flames temperature set at 700-800°C) heat for 15 minutes.

Then fuse on heating program without swirling for 8 minutes and. use the oxygen injector if available when the blend is totally fused and put the heating with swirling at 4 minutes.

Temperature of flames set at 1350°C.

**CAUTION:** it is necessary to test the method in order to check if there will be no damage to the platinum labware.

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