

XRF Scientific Ltd

ANALYSIS OF SULPHUR AND SODIUM

ON CEMENT~~ BY AN X~RAY

FLUORESCENCE FUSION METHOD

**METHOD OF SAMPLE
PREPARATION ON A
PHOENIX FUSION MACHINE
WITH XRFs LT66:MT34**

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Materials and equipment

Crucibles

The composition of the crucible is 95% platinum + 5% gold. The approximate dimensions are top diameter =36 mm, and depth =36 mm. Weigh =40 g, capacity =35 cc.

Casting dishes moulds

The composition of the dish is 95% platinum + 5% gold, Weigh = 60 g. These dishes will produce a bead of the following approximate dimensions: top diameter = 41 mm, bottom diameter = 39 mm and thickness =3 mm.

Chemicals

XRFS LT66:MT34. Molten homogeneous flux anhydrous containing 66% Lithium Tetraborate with addition of 34% Lithium Metaborate, produced by XRF Scientific Ltd.

Fusion machine

PHOENIX 6000/M: No of burners = 12, beads produced per cycle =6.

This type of machine is designed to allow the preparation of permanent and homogeneous fused beads under accurately reproducible conditions. The process involves dissolving samples and mixing them into a lithium borate flux at temperatures ranging from 800 to 1600°C. This is done over oxygen-enriched flames using specially designed burners. The sample is automatically poured into preheated moulds. The moulds are then retracted over separate cooling jets for precise control of cooling. This machine is manufactured by XRF Scientific Ltd and available in Europe from SOCACHIM Fine Chemicals.

PRINCIPLES OF THE TECHNIQUE

Its is possible to analyse the total sulphur content of the sample without prior oxidation. With the use of a low temperature flux such as XRFS LT66:MT34 (875°C). All cement, clinker, to be fused must finely grind in order to obtain particle size, i.e. less than 100 um. This fine size is achieved by grinding a portion of the cement, clinker in a laboratory swing/vibratory grinding mill. The finely ground cement, clinker perfectly mixed with the XRFS LT66:MT34 is added to the crucible.

The crucible is heated at 1000°C - 1050°C ,for -7 mm. After the fusion period, the melt is automatically poured into a preheated casting dish.

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Method

Weigh 0.8 g of
dried/grinded cement
8.8g of XRFs LT66:MT34
and mix them together

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Transfer the mixture
into the crucible

PROGRAM OF FUSION

1. PREMELT

850⁰C (flame temperature)
30 sec

2. FUSION (without swirling)

1200⁰G (flame temperature)
3 min.

3. FUSION (with swirling)

1200⁰C (flame temperature)
4 min.

4. MOULD CONTROL (mould pre41eated)

1100⁰C (flame temperature)
2 min.

5. COOLING (bi~level cooling)

3 min.

TOTAL FUSION TIME = 7 min. 30 sec. (fusion)
+ 3 min (cooling)
- 10min.30sec.