Determination of Fluoride Content in Powders

Application Note 7

Fluorides are used in many industrial processes, of which the production of hydrochloric acid and smelting are two examples. It is important to regulate these processes, so that the yield of product is maximised and the emission of toxic fluoride gases prevented. The **MQC+** is capable of accurately and rapidly measuring the fluoride content of solids in powder form.

Method

Conventional methods of measurement include titrometry, ion selective electrode, and ion chromatography. All of these methods are cumbersome, time consuming and require skilled chemists. Furthermore, sample preparation may often require the use of hazardous chemicals which require disposal.

The **MQC+** provides a rapid and effective alternative method for determining fluoride content, and offers a number of advantages over secondary methods:

- It can be calibrated to cover a concentration range from 0 to 100%.

- It can be calibrated using only 6 samples.

- NMR is very stable over the long term, so calibrations will rarely require adjustment.

- NMR is insensitive to air voids between grains of powder.

- The measurement time is short (typically 32 seconds).

- The NMR technique is non-destructive, so samples analysed may be re-used.

Samples are simply loaded into pre-tared glass vials, weighed, conditioned, then inserted into the instrument which detects the sample, automatically starting the NMR analysis. The instrument returns the fluoride content values in less than one minute.

Calibration and Results

Eleven artificial standards of Calcium Fluoride ranging from 1 to 100% CaF2 content were made up for analysis of fluoride content. Each sample was weighed into a tared 26mm glass tube and preconditioned for 20 minutes at 40°C, before being placed in the **MQC+** for analysis. The results of this analysis are seen in Figure 1. The quality of the calibration generated is good, especially as such a large range of fluoride is analysed.
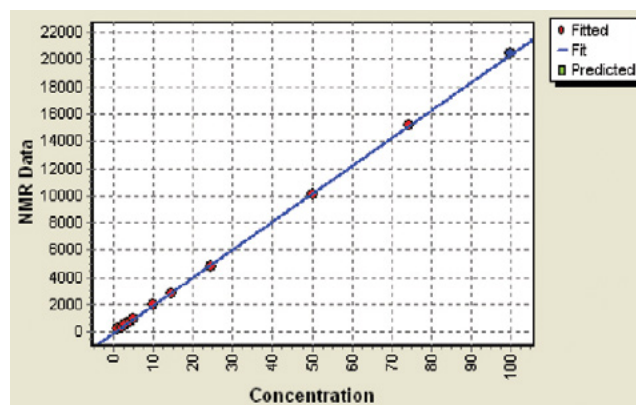
Figure 1: Calibration of NMR data for fluoride content of calcium fluoride samples. Correlation Coefficient: r = 1.00; SD = 0.41

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Table 1 shows that the repeatability (or precision) of the NMR measurement of the same sample is excellent.

Repeat number	Fluoride Content (%)
1	24.8
2	24.7
3	24.8
4	24.7
5	24.8
6	24.8
7	24.9
8	24.5
9	24.9
10	24.8
SD:	0.116

Table 1: *Repeatability of NMR Measurement of Calcium Fluoride samples.*

Recommended Instrument Configuration

The **MQC+** with a 0.55 Tesla (22 MHz fluorine resonance) magnet, fitted with a 26mm diameter (14ml sample) probe is a suitable instrument for this application. The Fluoride Content in Powder package comprises:

- **MQC+** with a built-in computer operating Microsoft® Windows® 10 (no separate PC is required).
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Fluoride in Powder' application.
- 26mm diameter glass tubes.
- User Manuals
- 'Fluoride in Powder' method sheet.

In addition to this package you will also require:

- A dry heater and aluminium block with 26mm holes for sample conditioning at 40ºC.
- A precision balance.

The instrument offers advantages over others on the market:

- High signal sensitivity.
- Small benchtop footprint.
- Low maintenance.
- The sample tubes are recyclable, lowering consumable costs.
- Minimal sample preparation.

visit **www.oxford-instruments.com/mqc** for more information or email: **magres@oxinst.com**

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