LGC Health Sciences

COMMERCIAL-IN-CONFIDENCE

Determination of Total Silicon in Milk Sample ex. MHRA

[redacted]

MHRA

September 2012

LGCNW15756

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Date: September 2012

Report Reference: LGCNW15756

1. Background

Determination of total Silicon (Si) content in sample of human breast milk from implant patient using ICP-OES.

2. Sample Details

Description	Customer Reference	LGC Reference	Date Received
Milk	[redacted]	15756-1	2nd July 2012

3. Glossary of Terms

ICP-OES Inductively Coupled Plasma Optical Emission Spectroscopy

4. General Procedure

In the absence of a suitable placebo sample the initial method development was carried out using control milk (see below) to optimise the sample loading and dissolution procedure and not to provide comparative data.

The milk samples were prepared by drying, charring and fusion with lithium tetraborate in platinum crucibles at 1000°C followed by dissolution in nitric acid. The fusion process converts all silicon containing species (e.g siloxanes) to silicon. The total silicon content is then determined by ICP-OES using matrix matched calibration standards.

4.1 Control Sample

The control milk was standard commercially available semi-skimmed cow milk (stored at 4°C).

4.2 Standard Preparation

0, 4, 20 and 40 ppb standards were prepared and matrix matched using 10% nitric acid and lithium tetraborate as per the milk samples. These were prepared from stock standard Romil 1000ppm Si E3SI4/F637391 expiry date June 2014

5. Results

Experimental data tables to show the following results for standards;

QC Data		
	Si 212.4nm (ppb)	**% Recovery**
20 ppb QC	21.09	105
4 ppb QC	3.734	93
40 ppb IQC	36.98	92
20 ppb spiked fusion mixture (no sample)	26.11	131*

* Possible contamination issue due to sample preparation procedure

Results from the initial experiment gave acceptable recovery but did indicate some contamination issues at low levels (46ppb for a 40ppb spike on a blank fusion flux).

Si Results (in sample – based on sample preparation in triplicate)		
Sample	**Si (in solution) 212.4nm (ppb)**	**Total Si (ppb)**
Prep 1	8.991	90
Prep 2	7.957	80
Prep 3	5.301	53
Mean =		**74**
sd =		19
% rsd =		26

However it should be noted that the fusion sample preparation method appears to be very prone to contamination at the low levels being analysed and is therefore potentially likely to produce erroneously high results.

<100ppb total silicon can be quoted with some confidence but the method should only be considered as an approximation at this stage until further data can be obtained to confirm the accuracy of the results.

6. Discussion - Experimental Design

The fusion technique is normally used for preparation of samples containing significantly higher levels of silicon than that detected in the milk sample.

Initial tests with different fusion mixtures using regular milk were made to assess the overall sensitivity of the method. The lithium tetraborate fusion mixture appears to more suitable than sodium carbonate which produces high blanks.

Relatively large samples (10ml milk made to 100ml after fusion) were used to maximize sensitivity. Initially the semi-skimmed milk control was used to establish if any silicon was detectable using the fusion method. This experiment established that the Si 212.4nm emission line was the preferred line. There was a measurable signal above the blank at approx 50ppb which equates to approx 500ppb in the milk. Results from this initial experiment gave acceptable recovery (46ppb for a 40ppb spike on a blank fusion flux) at this stage of method development but also indicated some possible contamination issues. However, there was also significant level of silicon detected in the blank therefore this is result can only be considered as a very rough approximation. This should not be considered as an accurate result based on this single sample. More data is necessary to improve confidence in this result.

Although the method is sensitive enough to measure low level silicon in the milk, it is operating close to the detection level (approximately 10ppb in solution). As a consequence there is an adverse affect on the precision / reproducibility of the measurements.

The current method appears to be very prone to contamination and likely to produce high results. A significant portion of the potential contamination arises from the sample preparation stage.

7. Conclusion

The initial results indicate the total silicon content of the supplied human breast milk sample is <100 ppb (see discussion).

In the absence of validated control data these results need to be compared against available literature values to assess significance.

8. Standard Operating Procedures Used

The following Standard Operating Procedures (SOPs) have been used in this analysis:

LGC/SOP/EA/NW/90 v3	- Operation of Thermo Iris Advantage ICP-OES
LGC/SOP/EA/NW/226 v1	- Fusion Procedures for the Dissolution of Solid Samples

8. Records

Lab note book and data folder	LGCNW/634