

ASOMA® PHOENIX II

Determination of Iron in Cocoa Products Using Direct Excitation

Summary

This report demonstrates the suitability of the PHOENIX II Benchtop XRF analyzer with direct excitation optics for use in the quality control of iron in cocoa powder, cocoa liquor or Massa. It will show that use of this instrument will deliver improvements in product quality by accurately and rapidly determining the iron in cocoa products.

It can also be seen that there is another advantage in easily and quickly measuring this element during the production process. Namely, by ensuring ideal iron content during production, significant reductions in operational costs can be achieved.

These benefits work together to maximize quality and reduce operational costs.

Introduction

The PHOENIX II is a powerful tool for monitoring iron in cocoa products. The analyzer uses a rugged, time-proven proportional counter as its detection system and a direct excitation X-ray tube. This combination of ruggedness, power and simplicity using a small benchtop analyzer enables fast and precise results.

The PHOENIX II uses an onboard PC computer with a simple touch screen interface. Thus, an external computer is not required. Data handling and results storage can be obtained on a thermal paper print out and are also stored in the hard drive of the PHOENIX II. The data can be readily transferred to a USB thumbdrive or a network Ethernet connection.

Calibrations are readily carried out using assayed standards. This ensures easy traceability of results for quality purposes. This initial calibration process is a “once only” procedure. Subsequently, the curve can be restandardized, if required, by the touch of a button on the main analysis screen.

The PHOENIX II offers power, versatility and performance all in a small, compact, easy-to-use design.

Experimental Portion

Equipment

All measurements were conducted using a PHOENIX II XRF analyzer using direct excitation optics. Performance is shown for a measurement time of 100 seconds.

Sample Preparation

A prepared sample cup is filled with the cocoa powder; pressed with 250 in•lb of torque using a manual sample press; and placed on the aperture for analysis.



Measurement Parameters

All measurement parameters are easily controlled through the touch screen on the display panel. Operators simply choose the correct method from the analysis screen (there may be more than one method stored, e.g. to deal with powder, Massa, etc.) and then press the green ANALYZE button.

The results can be reported using a variety of different options: results are reported on the display

screen; on a thermal paper printout; on an optional external printer; and in the database history within the analyzer.

Instrument Configuration

ASOMA® PHOENIX II

Excitation: Direct excitation 30 kV 9 W Air-cooled X-ray tube

Detection: Gas-filled Proportional Counter

Analytes Optimization: X-ray voltage, current and X-ray filters

Atmosphere: Air

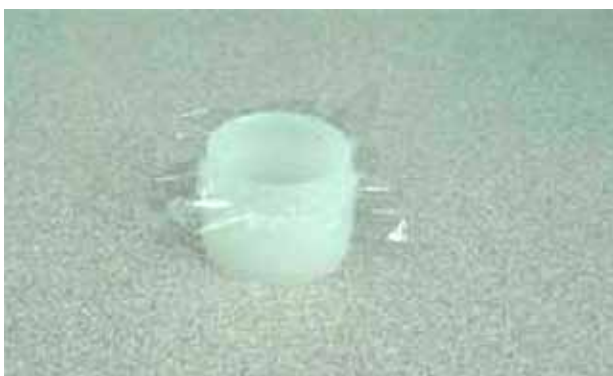
Options: Tube filter; Detector filter; Manual sample press; Polypropylene 6.0um film

Note: No consumable gases required.

XRF Sample Cup



Easy assembly with film window



The following results section show performance for typical iron in cocoa samples, submitted by a user. The PHOENIX II is equipped to measure many others. Please contact AMETEK for more information.

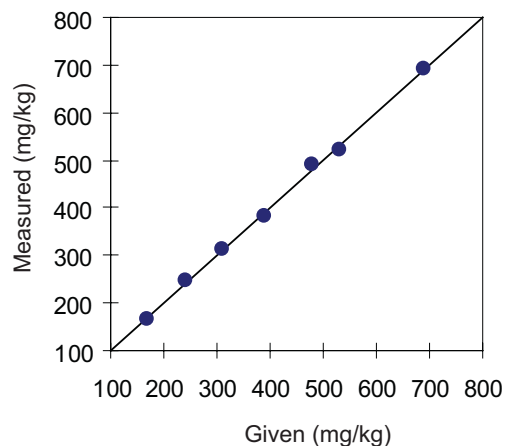
Results

Note: 1 mg/kg = 1 ppm

Calibration for Fe in Cocoa Powder

Element: Fe	Std. Error of Estimate: 8.79	
Units: mg/kg	R.M.S.: 2.51	
Sample	Given	Measured
1	310	313.2
2	530	520.1
3	480	489.1
4	170	165.4
5	240	247.1
6	390	383.3
7	690	691.9

Correlation Fit for Fe



Precision for Fe in Cocoa Powder
10 repeat analyses at 100 seconds per measurement

Element: Fe	Units: mg/kg			
Sample	Given	Mean	Std. Dev.	%Rel.
1	310	319.8	2.44	1.6
4	170	169.8	2.75	0.76
7	690	692.0	6.17	0.89

Minimum Detection Limit (MDL)**Fe in Cocoa Powder**

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank sample containing no Fe. The following MDL was derived using this empirical method and applies to this matrix and concentration range. Similar results would be expected with iron in cocoa liquor or Massa samples.

Element	Count Time	MDL
Fe	100 sec	5 mg/kg

Conclusion

As can be seen from the above data, the use of the PHOENIX II XRF system using direct excitation gives excellent performance when applied to the determination of iron in cocoa powder. Results are rapid, precise and analysis is easily carried out, even by non-laboratory personnel. Because no consumable chemicals are used, the relative “cost of ownership” is much lower than other analytical techniques.



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