Laboratory accreditation

Production of high quality goods starts with the selection of the raw materials on key parameters on cocoa bean quality. During production important parameters are measured continuously and when deviations occur the processing is adjusted accordingly. After production the final product is analyzed according to the criteria that are agreed between buyer and seller. The product is released and results are mentioned on a test report (also called certificate of analysis).

Operators carry out quality control during the processing of the beans. Cargill Cocoa Laboratory (CCL) is responsible for monitoring in line samples, analyzing the final product and generating the certificates of analysis.

Official methods for the analysis of many important parameters to judge the quality of the cocoa beans exist already for a long time. These so-called IOCCC (International Office of Cocoa, Chocolate and Confectionary) are often applied but over time, many laboratories have made smaller or bigger adjustments for numerous reasons i.e. to shorten analysis time and/or to simplify the method itself. In addition, during the last decade many new and advanced techniques became available – such as Near Infra Red for the determination of fat and moisture in food products.

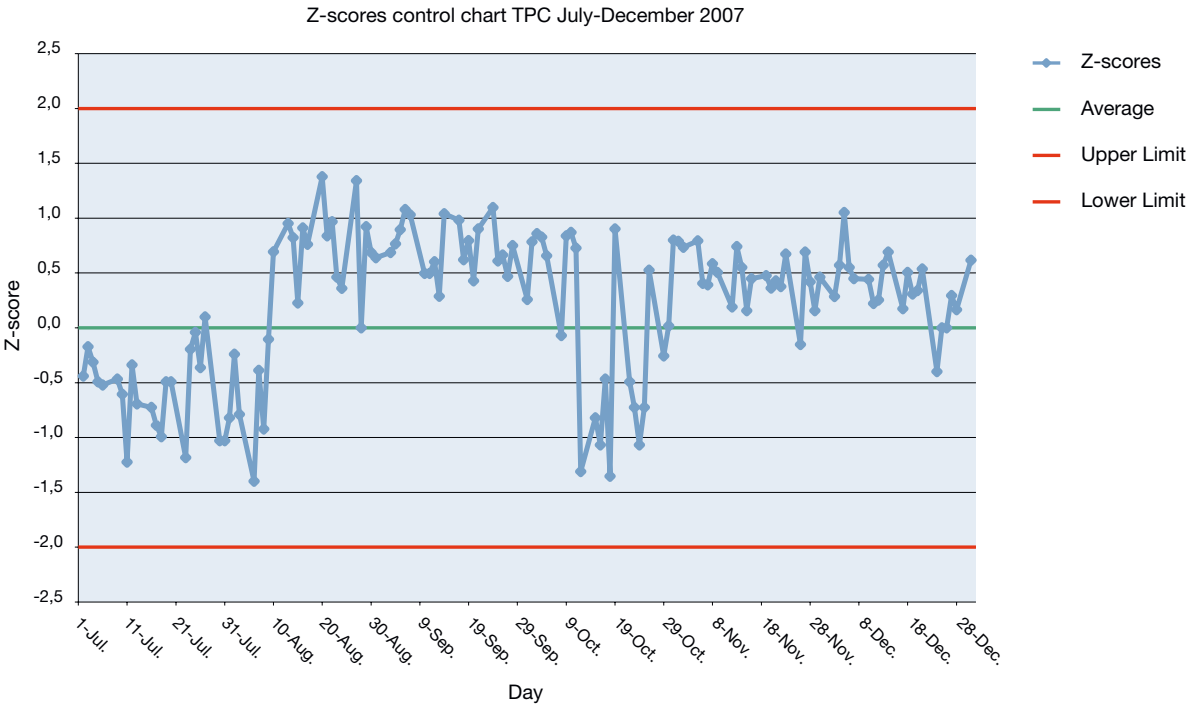
The performance of an analytical method depends on many factors. An ISO 9001 certified testing laboratory does not need to demonstrate competence to produce valid data and results. To ensure reliable test reports we, as Cargill Cocoa Laboratory (CCL), have decided to accredit the most important analytical methods we use to base our – test reports on. The laboratory has been accredited conform the ISO 17025:2005 standards by an independent organization called Dutch Accreditation Council (RVA). In this publication we explain the basics of an ISO 17025 accreditation.

Laboratory accreditation according to ISO 17025:2005 does not only audit the quality system, including procedures and methods. Also the competence of the laboratory technicians itself is audited. To clarify: when only methods and procedures are audited – it is called certification. When also competencies of laboratory technicians are audited it is called accreditation.

The main characteristics of CCL as an ISO 17025 accredited testing laboratory[1]):

1. CCL is an independent legal entity embedded in Cargill Cocoa.
2. The scope of the accreditation is the analysis of cocoa liquor and cocoa powder.
3. CCL has a management system suitable for the scope of its' activities.
4. CCL has a document control system, which includes approval by authorized personnel and document change review procedures.
5. CCL has a complaint management system including regular review procedures to evaluate complaints and where necessary to take corrective action.
6. Specific procedures are in place to ensure compliance with its own policies and procedures. If this is not the case, actions such as halting of work and withholding of test reports are defined. A detailed investigation will be carried out, such as cause analysis and selection and implementation of corrective actions. The latter has to be monitored to ensure effective implementation and also preventive action procedures should be established.
7. Improvement on effectiveness of the management system is embedded in the system using for example data analysis and corrective and preventive actions. Also internal audit results are used for this.

8. Records control procedures are in place. Where possible all data are initialized and therefore at all times we can identify who preformed a specific task.
9. Management reviews will be carried out to determine the suitability and continuing effectiveness and to introduce changes and/or improvements when applicable.
10. The correctness and reliability of tests are dependent on many factors among others personnel and the test method validation. Within CCL the competence of **all technicians** for **all** the individual tests they carry out is ensured. Only qualified technicians may generate results and non-qualified technicians i.e. under temporary contract may only work under supervision. Training and education needs are known and will be carried out on individuals and/or groups.
11. All methods used within CCL are conform or equivalent to the official IOCCC and/or AOAC (Association of Analytical Communities) and/or ISO methods or are fully validated by ourselves. The uncertainty of measurement of **all** methods is estimated.
12. All measuring (related) equipment is calibrated. For example temperature profiles in incubators for microbiological tests have been tested and must stay within the given limits as guaranteed by the manufacturer.
    These incubators are equipped with continuous temperature measurement devices and supervisors will be automatically informed, 24 hours a day, in case the given limits are exceeded. This ensures that corrective actions can be taken immediately.
13. Sampling plans and procedures are available to assure testing of only representative sample(s) of a batch.
14. To assure the quality of the test methods;
    1. We use so-called first line controls (see Graph 1). For example we have co-developed the cocoa powder samples for the microbiological methods. Each of these samples has a defined number of colony forming units per gram (cfu/g), the so-called assigned value. For the determination of the total plate count [2]) we analyse each day such a sample and the results should be within given limits assuring the quality of the test.



Graph 1: First line control results for Total Plate Count determination

The z-score is defined as:

z = (X – Xa)/σ

X = value found by CCL

Xa = assigned value

σ = standard error

We co-developed (on lab scale) cocoa powder samples with a very low number of *Salmonellae* (so-called positive control). Every day we analyse one of these samples and *Salmonellae* [2]) should be detected in the positive control. If not all the test results of that day are not valid and should be re-analysed. Also negative controls are analysed every day. The positive control sample is always tested together in 100 gram cocoa powder and our method has been proven to detect around 10-20 colony-forming units *Salmonellae* per 100 gram with a confidence interval of 95%. It is important to mention that no test reports can be generated when control samples give wrong results.

2. We perform inter-laboratory comparison studies within Cargill Cocoa and Chocolate.
3. We participate in external proficiency testing programs.

15. We have determined the accuracy of all methods and consequently the results are reported accordingly. This may lead to changes in reporting i.e. for fineness results instead of two digits we now report only in one digit.

Table 1: The methods for which Cargill Cocoa Laboratory has been accredited:

Method	Description	Method
1.	Determination of fat content of cocoa mass by soxhlett extraction; gravimetric.	Equivalent to IOCCC-14
2.	Determination of sieve residue of cocoa mass; wet sieving; gravimetric.	Internal method
3.	Determination of pH of cocoa mass; electrochemical.	Equivalent to IOCCC-15
4.	Determination of the color; spectrophotometric.	Internal method
5.	Determination of moisture content and fat content of cocoa mass by NIR.	Equivalent to IOCCC-14 and IOCCC-1
6.	Determination of moisture content of cocoa mass by oven drying method; gravimetric.	Conform IOCCC-1
7.	Determination of sieve residue of cocoa powder; wet sieving; gravimetric.	Internal method
8.	Determination of fat content of cocoa powder by Soxhlett extraction; gravimetric.	Equivalent to IOCCC-37
9.	Determination of pH of cocoa powder; electrochemical.	Equivalent to IOCCC-15
10.	Determination of color in water of cocoa powder; spectrophotometric.	Internal method
11.	Determination of fat content of cocoa powder; refractometer.	Internal method
12.	Determination of moisture content and fat content of cocoa powder by NIR.	Equivalent to IOCCC-37 and IOCCC-1
13.	Determination of moisture content of cocoa powder by oven drying method; gravimetric.	Conform IOCCC-1
14.	Determination of TPC; plating.	Conform IOCCC-39 number 1 and 2
15.	Detection of *Enterobacteriaceae*; presence/absence reaction.	Internal method
16.	Enumeration of *Enterobacteriaceae* (without confirmation); plating.	Internal method
17.	Detection of *Salmonella*; presence/absence reaction.	Equivalent to IOCCC-39

All these aspects will be audited on yearly basis by the RvA (see Table 1). When we do not work conform the ISO 17025 requirements actions will be taken by the RvA, for example they may force us to take corrective action within 24 hrs or in worse case to withdraw the accreditation, which will be published on the RvA website (www.rva.nl).

Summary

Within an ISO 17025 accredited laboratory samples are analyzed by **proven** competent technicians and the quality of the tests are checked **on daily basis** with the so-called first line control giving **reliable results** which can be used by our customers as incoming ingredients control minimizing the time for acceptance and usage.

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Literature

[1)] ISO/IEC 17025:2005 (E) General requirements for the competence of testing and calibration laboratories.
[2)] Microbiological examination of chocolate and other cocoa products, IOCCC 39, 1990.