



#### Application Note AN-I-033

# Determination of ammonia in cacao

## Reliable cost- and timesaving determination of ammonia by standard addition

Ammonia is one of the most widely manufactured chemicals. It is also produced naturally in our bodies, by fermentation processes, and can be found in different products. Additionally, ammonia is formed by the bacterial decomposition of animal and plant matter in soil.

In the case of cacao, ammonia is naturally formed by fermentation of the cacao beans. The addition of ammonia during the alkalization process is a common

practice to give an intense black color to the cacao nibs and to modify their flavor.

Ammonia is usually determined by ion chromatography by converting it into the ammonium form. This Application Note offers an easy way to determine the ammonia content in cacao nibs by using ion measurement, applying the standard addition technique in a reliable cost- and timesaving manner.

SAMPLE AND SAMPLE PREPARATION

The sample is homogenized in 400 mL diluted hydrochloric acid (HCl) for 30 seconds. Afterwards, it is filtered using gravity through a folded filter paper into

a volumetric flask. The resulting filtered solution is filled up to the mark on the flask with deionized water.

EXPERIMENTAL

The filtered sample solution is pipetted into a beaker and filled up to 50 mL with deionized water. Highly concentrated sodium hydroxide (NaOH) solution is added, and the standard addition is performed.



Figure 1. 867 pH Module controlled by tiamo software for performing standard addition.

RESULTS

The standard addition is performed automatically within 10 minutes using the setup in Figure 1. The additions of the reagent increments as well as the calculation of the ammonia content in cacao are performed automatically by tiamo™.

Table 1. Ammonia content of Sample B and Sample X (cacao). Both samples were obtained in their natural and their alkalized form. Sample B was alkalized by using ammonia, while sample X was alkalized without using ammonia.

|                     | Mean / mg/kg | SD /mg/kg | RSD/% |
|---------------------|--------------|-----------|-------|
| Sample B            | 151.6        | 2.6       | 1.71  |
| Sample B, alkalized | 499.0        | 6.9       | 1.39  |
| Sample X            | 136.5        | 1.8       | 1.35  |
| Sample X, alkalized | 189.7        | 0.8       | 0.43  |

## CONCLUSION

This Application Note shows how ammonia in cacao samples can be determined in a straightforward manner by using the standard addition method. The standard addition method is easy to apply and does not require much system maintenance. Additionally,

this method is matrix-independent and does not need an external calibration.

The obtained results exhibit good reproducibility with a relative standard deviation <2 % which is very satisfactory for ion measurement.

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## CONFIGURATION



### 867 pH Module with *tiamo*™ light

High-end pH/ion meter based on the 867 pH Module, including *tiamo*™ light, 854 iConnect and intelligent "iUnitrode" pH glass electrode.

In addition to measurements of pH, temperature, mV, I<sub>pol</sub>, U<sub>pol</sub> and concentration, the pH Module can perform standard additions (manual, dos, autodos) and Liquid Handling (add, prep, empty). It enables the use of both conventional and intelligent sensors for measuring. Also integrated in the software is an automatic GLP-compliant pH electrode test.

The pH Module has two USB interfaces for connecting printers, barcode readers or sample changers and four MSB interfaces for stirrers or Dosinos (for the addition of auxiliary solutions or for standard addition).

Integrated in *tiamo*™ (from 2.0), it is in compliance with GLP and FDA 21 CFR part 11 requirements.