# International Standard 4052

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# Coffee — Determination of caffeine content (Reference method)

*Cafés — Détermination de la teneur en caféine — Méthode de référence*

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## Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4052 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in May 1982.

It has been approved by the member bodies of the following countries :

| | | |
|---|---|---|
| Austria | Israel | Tanzania |
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The member body of the following country expressed disapproval of the document on technical grounds :

Malaysia





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## 0 Introduction

The method described in this International Standard has been chosen from amongst several methods, a comparative study of which was carried out, because of its general applicability, its reproducibility, its specificity, its ease of application and its rapidity.

However, the method is particularly sensitive to variations in its application and it is therefore essential to follow the instructions in every detail.

## 1 Scope and field of application

This International Standard specifies the reference method for the determination of the caffeine content of coffee.

The method is applicable to green coffee, decaffeinated green coffee, roasted coffee, decaffeinated roasted coffee, extracts of coffee, both dried and liquid, and decaffeinated extracts, both dried and liquid.

The lower limit of detection is 0,02 % caffeine on the dry basis.

## 2 References

ISO 1447, *Green coffee — Determination of moisture content (Routine method).*

ISO 3726, *Instant coffee — Determination of loss in mass at 70 °C under reduced pressure.*

ISO 4072, *Green coffee in bags — Sampling.*

ISO 6670, *Instant coffee in cases with liners — Sampling.*[1)]

ISO 6673, *Green coffee — Determination of loss in mass at 105 °C.*[1)]

## 3 Principle

Extraction of the caffeine from a test portion, in an ammoniacal medium. Successive purification, with diethyl ether, on two chromatographic columns, the first in an alkaline medium, the second in an acid medium, followed by elution of the caffeine by chloroform.

Spectrometric measurement of the eluate at the wavelength of maximum absorbance (in the ultraviolet region).

## 4 Reagents

All reagents shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

**4.1 Sulphuric acid,** 200 g/l solution [$c(H_2SO_4) \approx 2$ mol/l].

**4.2 Sodium hydroxide,** 80 g/l solution [$c(NaOH) \approx 2$ mol/l].

**4.3 Diatomaceous earth**

The product used shall ensure at least 98 % recovery of caffeine from the test portion.

NOTE — Celite 545 has been found to be suitable.

**4.4 Ammonia,** 70 g/l solution (1 volume of concentrated ammonia solution, $\varrho_{20} \approx 0{,}9$ g/ml, + 2 volumes of water).

**4.5 Diethyl ether,** pure, or repurified (see 7.5) by chromatography as follows, and saturated with water.

Pass 800 ml of diethyl ether through a column containing 100 g of basic aluminium oxide of activity grade 1. The diethyl ether, thus repurified, shall be kept in dark bottles until used.

(Alternatively, diethyl ether, recently distilled and free of peroxides, can be used instead of diethyl ether repurified by chromatography.)

**4.6 Caffeine** [1,3,7-trimethyl-2,6-dioxopurine ($C_8H_{10}N_4O_2$)], pure, anhydrous.

**4.7 Chloroform,** pure, or repurified (see 7.5) by chromatography as described in 4.5, and saturated with water.

1) At present at the stage of draft.