

Australian Standard™

Coal and coke—Analysis and testing

Part 10.1: Determination of trace elements—Coal, coke and fly-ash— Determination of eleven trace elements—Flame atomic absorption spectrometric method



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This Australian Standard was prepared by Committee MN-001, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 28 February 2003 and published on 5 March 2003.

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STANDARDS AUSTRALIA

RECONFIRMATION

OF

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Coal and coke—Analysis and testing

Part 10.1: Determination of trace elements—Coal-coke and fly-ash—Determination of eleven trace elements—Flame atomic absorption spectrometric method

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NOTES

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PREFACE

This Standard was prepared by the Standards Australia Committee MN-001, Coal and Coke, to supersede AS 1038.10.1—1986, *Coal and coke—Analysis and testing, Part 10.1: Determination of trace elements—Coal, coke and fly-ash—Determination of eleven trace elements—Flame atomic absorption spectrometric method*.

This revision confirms the flame atomic absorption spectrometric method for the determination of eleven trace elements in coal, coke and fly-ash. Editorial changes have been made to bring the Standard into line with current style.

The objective of this Standard is to provide those responsible for testing coal, coke and fly-ash with a standardized method for measuring eleven trace elements by flame atomic absorption spectrometry and to provide an alternative flame atomic emission spectrometric method for measuring barium in an ashed sample.

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is only for information and guidance.

CONTENTS

	<i>Page</i>
1 SCOPE	4
2 REFERENCED DOCUMENTS	4
3 DEFINITIONS	4
4 PRINCIPLE.....	5
5 SAFETY	5
6 REAGENTS	5
7 APPARATUS.....	10
8 SAMPLE	11
9 PROCEDURE	13
10 CALCULATION.....	15
11 REPORTING OF RESULTS.....	16
12 ACCEPTANCE OF RESULTS	16
13 PRECISION	16
14 TEST REPORT	17
APPENDIX A PRECISION DATA FOR DETERMINATION OF ELEVEN TRACE ELEMENTS IN COAL, COKE AND FLY-ASH	18

STANDARDS AUSTRALIA

Australian Standard

Coal and coke—Analysis and testing

Part 10.1: Determination of trace elements—Coal, coke and fly-ash—
Determination of eleven trace elements—Flame atomic absorption
spectrometric method**1 SCOPE**

This Standard sets out a method for the preparation of the ash and a flame atomic absorption spectrometric method for the determination of barium, beryllium, chromium, copper, lead, lithium, manganese, nickel, strontium, vanadium and zinc in ash prepared in the laboratory from higher rank coal, coke and fly-ash.

The Standard also provides an optional flame atomic emission spectrometric method for the determination of barium, as it gives improved sensitivity over flame atomic absorption spectrometry and it is not possible to determine barium by the latter technique with some instruments.

NOTE: Two approaches, gravimetric and volumetric, have been specified for solution preparation, both give the same results provided that, in the volumetric approach, plastics volumetric ware is used.

2 REFERENCED DOCUMENTS

The following standards are referred to in this standard:

AS

1038	Coal and coke—Analysis and testing
1038.3	Part 3: Proximate analysis of higher rank coal
1038.4	Part 4: Coke—Proximate analysis
1038.16	Part 16: Assessment and reporting of results
2134	Recommended practice for chemical analysis by atomic absorption spectrometry
2134.1	Part 1: Flame atomic absorption spectrometry
2243	Safety in laboratories (series)
2418	Coal and coke—Glossary of terms
2508	Safe storage and handling information card (series)
2850	Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct
4264	Coal and coke—Sampling
4264.1	Part 1: Higher rank coal—Sampling procedures
4264.2	Part 2: Coke—Sampling procedures

3 DEFINITIONS

For the purpose of this Standard the definitions in AS 2418 apply.

4 PRINCIPLE

Coal, coke or fly-ash is ashed under controlled laboratory conditions. The resulting ash is dissolved by mineral acids and the individual elements are determined by flame atomic absorption or emission spectrometry.

5 SAFETY

For information on laboratory safety, reference should be made to the relevant parts of AS 2243 and AS 2508.

6 REAGENTS

6.1 General

Unless otherwise specified, all reagents shall be of analytical reagent grade, and only distilled water, or water of equivalent purity, shall be used. All single-element standard solutions shall be stored in polyethylene bottles with leakproof screw caps and labelled with the date of preparation. They should be discarded after a period of not longer than 12 months.

6.2 Solutions

6.2.1 Nitric acid

(ρ_{20} 1.42 g/mL).

6.2.2 Nitric acid

(ρ_{20} 1.42 g/mL), diluted 1 + 9.

6.2.3 Nitric acid

(ρ_{20} 1.42 g/mL), diluted 1 + 19.

6.2.4 Hydrochloric acid

(ρ_{20} 1.16 g/mL).

6.2.5 Aqua regia solution

Add carefully, with stirring, 200 mL of nitric acid (6.2.1) to 200 mL of water. Add 600 mL of hydrochloric acid (6.2.4), mix and store in a polyethylene bottle.

SAFETY NOTE: Polypropylene bottles are unsuitable for storage of this aqua regia solution because they have been found to become embrittled by it after a few months. Polyethylene bottles, on the other hand, have been found to take four years to become embrittled by this solution. Hence it is recommended that the polyethylene bottle used for storage of this aqua regia solution be replaced every two years.

6.2.6 Hydrofluoric acid

(ρ_{20} 1.15 g/mL, i.e. 40 percent m/m).

SAFETY NOTE: All acids should be handled with care but extra precautions are required with hydrofluoric acid. Even when diluted, this is an extremely dangerous acid, harmful to the eyes and skin; rubber gloves and goggles should be worn when using this acid. Any acid that touches the skin should be washed off immediately with copious quantities of water. A physician should be consulted immediately if any acid is sprayed into the eyes or if prolonged contact with the skin occurs.

Solutions for atomic absorption measurement contain some hydrofluoric acid, formed by hydrolysis of fluoroboric acid, and so must be handled with care.

6.2.7 Boric acid solution

(40 g/L). Dissolve 80.0 ± 0.1 g of boric acid powder in 1900 mL of water, dilute to 2000 mL with water and mix.