

ICS 83.140.30

CCS G 33



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 13021-2023

Replacing GB/T 13021-1991

**Polyolefin pipes and fittings - Determination of carbon black
content - Test method of calcination and pyrolysis**

Issued on: August 6, 2023

Implemented on: March 1, 2024

**Issued by: State Administration for Market Regulation, China National
Standardization Administration**

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1. Structure and Drafting Rules of Standardization Documents"

Drafting.

This document replaces GB/T 13021-1991 "Determination of carbon black content in polyethylene pipes and fittings (thermal weight loss method)" and is consistent with

Compared with GB/T 13021-1991, in addition to structural adjustments and editorial changes, the main technical changes are as follows.

---Changed the scope of application (see Chapter 1, Chapter 1 of the 1991 edition);

---Added method B and method C (see Chapter 6 and Chapter 7);

- Added requirements for balance and timer accuracy (see 5.2, 6.1);
- Added the requirements for the length of tubular electric furnaces and quartz tubes (see 5.2);
- Added state adjustment of the specimen (see 5.3.3);
- Changed the calcination temperature in the tubular electric furnace method (see 5.3.6, 5.3.5 of the.1991 edition).

This document is equivalent to ISO 6964.2019 "Test Method for Determination of Carbon Black Content by Calcination and Pyrolysis of Polyolefin Pipes and Fittings".

A "Terms and Definitions" chapter has been added to this document.

This document has made the following minimal editorial changes.

- Change the name of the standard to "Determination of carbon black content in polyolefin pipes and fittings, calcining and pyrolysis method".

Please note that some content in this document may be subject to patents. The publisher of this document assumes no responsibility for identifying patents.

1 Scope

This document specifies the test methods for the determination of carbon black content in polyolefin pipes, pipe fittings and their raw materials, and provides a method suitable for polyethylene pipes.

and pipe fittings basic methods.

This document is applicable to the determination of carbon black content in polyolefin pipes, pipe fittings and their raw materials.

2 Normative reference documents

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, the dated quotations

For undated referenced documents, only the version corresponding to that date applies to this document; for undated referenced documents, the latest version (including all amendments) applies to

this document.

polymers-Part 1.Generalprinciples]

Note. GB/T 33047.1-2016 Thermogravimetric method for plastic polymers (TG) Part 1.General principles (ISO 11358-1.2014, MOD)

3 Terms and definitions

There are no terms or definitions to be defined in this document.

4 Method overview

The carbon black content of the polyolefin sample was measured by the following method.

---Method A. Place the sample in a tubular electric furnace, pyrolyze with nitrogen flow at 550 degrees C $\pm$ 50 degrees C for 45min, and then pyrolyze at 900 degrees C $\pm$ 25 degrees C

Calcination.

---Method B. Put the sample into a covered quartz crucible, place it in a box-type resistance furnace (commonly known as "muffle furnace") or microwave muffle furnace for calcination and

Pyrolysis. The testing process is divided into two types based on the type of instrument used.

* Box-type resistance furnace method (Method B1). Heating from 325 degrees C $\pm$ 25 degrees C to 550 degrees C $\pm$ 25 degrees C at a certain heating rate, and

Maintain 550 degrees C $\pm$ 25 degrees C for 10min $\pm$ 0.5min for pyrolysis. After pyrolysis, it is calcined at 900 degrees C $\pm$ 25 degrees C.

*Microwave muffle furnace method (Method B2). After pyrolysis at 520 degrees C $\pm$ 25 degrees C for 10min $\pm$ 0.5min, pyrolyze at 900 degrees C $\pm$ 25 degrees C

Calcination.

---Method C. Put the sample into a thermogravimetric analyzer (TGA), pass in inert gas, heat it up to 800°C at a certain rate for pyrolysis, and then

Finally, the inert gas is switched to oxygen and calcined at 900°C.

Note 1. Carbon black begins to oxidize in air or oxygen at 500°C. The quality loss of carbon black will occur between 500 degrees C~700 degrees C in air or oxygen.

Note 2. If the sample contains additives other than carbon black that can also decompose at 900°C, such as calcium carbonate, the measured carbon black content may exceed the actual value.

actual value. If the ash content is greater than 1%, further verification is required.

The carbon black content was calculated based on the mass difference before and after calcination and pyrolysis.