International Standard 6792

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Butadiene for industrial use — Determination of oxygen and argon in the gaseous phase above liquid butadiene — Gas chromatographic method

Butadiène à usage industriel — Dosage de l'oxygène et de l'argon dans la phase gazeuse au-dessus du butadiène liquide — Méthode par chromatographie en phase gazeuse

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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 6792 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in November 1980.

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

Australia	Germany, F. R.	Poland
Austria	Hungary	Portugal
Belgium	India	Romania
Brazil	Ireland	South Africa, Rep. of
China	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
Egypt, Arab Rep. of	Mexico	USSR
France	Philippines	

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Butadiene for industrial use — Determination of oxygen and argon in the gaseous phase above liquid butadiene — Gas chromatographic method

1 Scope and field of application

This International Standard specifies a gas chromatographic method for the determination of oxygen and argon in the gaseous phase above liquid butadiene for industrial use.

The method is applicable to butadiene of which the gaseous phase above the liquid contains more than 100 ml of oxygen and argon per cubic metre.

It can be operated at two temperatures, either at − 78 °C or at ambient temperature. At ambient temperature, oxygen and argon are not separate and the method thus only allows the total content of these two gases to be determined. When the operations are carried out at − 78 °C, these two gases are separated.

2 Reference

ISO 6377, *Light olefins for industrial use — Determination of impurities by gas chromatography — General considerations.*

3 Principle

Analysis by gas chromatography using a thermal conductivity detector.

4 Materials

4.1. Carrier gas : Helium or hydrogen of purity ⩾ 99,9 %, and containing no organic impurities, water or carbon dioxide.

4.2 Standards

Prepare or obtain standard mixtures containing variable and known quantities of oxygen (and if necessary argon) in pure or purified nitrogen with appropriate absorber. The range of concentration of standards will be 50 to 5 000 ml/m^3.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Chromatograph

Use a gas chromatography apparatus with an injection device and a thermal conductivity detector.

If it is desired to separate the oxygen and the argon, it is necessary to maintain the column at the temperature of dry ice (− 78 °C). In order to do this, place the column in a Dewar flask filled with dry ice and acetone and connect it to the chromatograph by means of two stainless steel capillary tubes; in addition, provide a pre-column filled with activated carbon for the retention of hydrocarbons. It shall be possible to back-flush this pre-column.

5.1.1 Injection device

See ISO 6377.

5.1.2 Pre-column

5.1.2.1 Filling : activated carbon of particle size 0,17 to 0,25 mm, for gas chromatography.

5.1.2.2 Tube :

- stainless steel
- length : 80 cm
- internal diameter : 4 mm

5.1.2.3 Temperature : between 15 to 25 °C maintained constant to ± 1 °C.

5.1.3 Separating column

5.1.3.1 Filling : molecular sieve 5 A (calcium aluminosilicate, 0,17 to 0,25 mm).

5.1.3.2 Tube :

- stainless steel
- length : 200 cm
- internal diameter : 4 mm

5.1.3.3 Temperature : temperature between 15 to 25 °C maintained constant at ± 1 °C or − 78 °C (dry ice).

5.1.4 Detector : thermal conductivity type

5.2 Recorder

6 Sampling

It is advisable to take the necessary precautions to prevent the introduction of air during sampling or during storage either by infiltration at joints which are not air-tight or by diffusion across the walls of a tube. Use sample chambers of the type shown in figure 1 of ISO 6377.

The connection tubing shall be as short as possible and shall preferably be made of glass or metal. Flush the sample chamber by passing through it a volume of olefin to be sampled equal to approximately 100 times the volume of the tube; this flushing stream shall be as strong as possible. After flushing, close the valves so as to keep a quantity of olefin in the sample chamber such that there is still a slight overpressure which eliminates the risk of air penetrating into the sample chamber.

7 Procedure

7.1 Preparation of the apparatus

After filling with molecular sieve, bring the separating column to 310 to 320 °C in a stream of hydrogen (approximately 100 ml/min) to eliminate the water, carbon dioxide and traces of organic products. This may be done in the chromatograph oven if it will reach this temperature. The time necessary for this reconditioning is between 1 and 4 h depending on the humidity. During cooling, the hydrogen stream shall be maintained. If it is not possible to heat the column in the chromatograph, use another suitable oven. The connections shall be changed as quickly as possible.

7.2 Injection of the test portion

Inject the test portion in the form of a gas. Follow the indications of clause 4 of ISO 6377.

7.3 Calibration

Successively inject standard mixtures (4.2) so as to have three peaks corresponding to three different concentrations.

7.4 Determination

After flushing the injecting device with the sample gas, inject a test portion of 1 ml for concentrations greater than 2 000 ml/m^3. For concentrations less than 2 000 ml/m^3, inject 5 ml. Record the chromatogram.

NOTE — When the temperature of the column is − 78 °C, after 10 injections, the separating column should be brought to ambient temperature for approximately 20 min in order to eliminate the components retained (nitrogen, methane, carbon monoxide). If this operation is omitted, the base line of subsequent chromatograms becomes irregular.

7.5 Calculation

See clause 6 of ISO 6377.

8 Expression of results

Express the oxygen-argon contents, or the oxygen content and the argon content, in millilitres per cubic metre.

9 Test report

The test report shall include the following information :

a) all information necessary for the complete identification of the sample (lot, date, time and duration of each sampling, etc.);

b) a reference to this International Standard;

c) the concentration of each impurity as required;

d) the contents of oxygen and of argon of the gaseous standard mixture;

e) a statement of any experimental conditions which are regarded as optional :

— a description of the column or combination of columns used, or reference to the column or combination of columns specified in the International Standard,

— the nature of the carrier gas,

— the pressure, in megapascals or in bars,[1] of the carrier gas at the entrance to the column or in the first part of the column,

— the flow rate of the carrier gas, in litres per hour, measured at standard atmospheric pressure,

— the volume, in millilitres measured at standard atmospheric pressure, of gas injected for each test,

— the duration of recording;

f) details of any unusual features noted during the determination;

g) details of any operations not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.

1) 1 bar = 10^5 Pa