
International Standard



7381

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Butadiene for industrial use — Determination of oligomers — Gas chromatographic method

Butadiène à usage industriel — Dosage des oligomères — Méthode par chromatographie en phase gazeuse

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Foreword

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International Standard ISO 7381 was prepared by Technical Committee ISO/TC 47, *Chemistry*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Butadiene for industrial use — Determination of oligomers — Gas chromatographic method

1 Scope and field of application

This International Standard specifies a gas chromatographic method for the determination of oligomers in butadiene for industrial use.

The method is applicable to butadiene having oligomer contents greater than 10 mg/kg.

The oligomers which may be present¹⁾ are

- 4-vinyl cyclohexene;
- 1,3-cyclooctadiene;
- 1,5-cyclooctadiene.

2 References

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

ISO 8563, *Propylene and butadiene for industrial use — Sampling in liquid phase*.²⁾

3 Principle

After obtaining a liquid sample at low temperature, injection of a liquid or gaseous test portion into a gas chromatography column.

Passage of the test portion through this column, allowing the separation of the oligomers, detection by flame ionization and comparison of the peaks obtained with those derived from an external standard.

4 Materials

4.1 Carrier gas

Nitrogen, helium or argon of purity > 99,9 %, containing no organic impurities or water.

4.2 Standard mixture

For each determination, prepare each time a standard mixture by dissolving an appropriate quantity, weighed to the nearest 0,1 mg, of 4-vinyl cyclohexene in a mixture of saturated hydrocarbons ranging from butane to heptane, so that the concentration of 4-vinyl cyclohexene corresponds to its expected concentration in the sample.

Transfer the mixture obtained to a vessel similar to that used for the sample and pressurize with an appropriate gas.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Dewar flask, transparent, permitting the butadiene to be maintained in the liquid state (see figure 1).

5.2 Test tube, for holding the liquid sample.

5.3 Chromatograph.

Use a gas chromatograph complying with the requirements specified below and which will yield a peak height of at least five times the background noise level for each oligomer at a concentration of 10 mg/kg.

5.3.1 Injection device.

a) Liquid sampling valve, allowing the successive introduction of liquid butadiene at its vapour pressure or at a greater pressure, and in adequate supply to satisfy the requirements of sensitivity and resolution according to the nature of the column (5.3.2) and of the detector (5.3.3).

b) Syringe, graduated in microlitres.

c) Stainless steel gas sampling valve with sample loop (if low oligomer contents presumed, see 3.2 in ISO 6377).

1) With an appropriate choice of chromatographic conditions, other impurities such as acetonitrile, ethylbenzene or styrene (vinylbenzene) can also be determined by this method in recycled samples, which is illustrated in annex B.

2) At present at the stage of draft.