International Standard 6191

W-73-30

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Light olefins for industrial use — Determination of traces of water — Karl Fischer method

Oléfines légères à usage industriel — Dosage des traces d'eau — Méthode de Karl Fischer

First edition — 1981-08-15

UDC 661.715.3 : 543.812 **Ref. No. ISO 6191-1981 (E)**

Descriptors : olefinic hydrocarbons, chemical analysis, determination of content, water, Karl Fischer method, apparatus, sampling, inspection, test results.

Price based on 8 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 6191 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in March 1979.

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

Australia	Hungary	Romania
Austria	Israel	South Africa, Rep. of
Belgium	Italy	Spain
China	Libyan Arab Jamahiriya	Switzerland
Czechoslovakia	Mexico	Turkey
Egypt, Arab Rep. of	Netherlands	USSR
France	Philippines	
Germany, F. R.	Poland	

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

United Kingdom

Printed in Switzerland

6191-81 4851903 0049222 1

Light olefins for industrial use — Determination of traces of water — Karl Fischer method

WARNING — Carry out the operations specified in this International Standard in a well-ventilated room (handling of inflammable gas and pyridine).

1 Scope and field of application

This International Standard specifies the manner of application of the Karl Fischer method for the determination of traces of water in light olefins from C_2 to C_4 for industrial use.

The method is applicable to products having water contents between 10 and 100 mg/kg.

For higher contents use ISO 760 as sampling is not valid when there is a risk that condensation of water may occur.

2 References

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling.*

3 Principle

Absorption of the traces of water in a test portion in a solvent to which a sufficient quantity of Karl Fischer reagent has been added so that there is neither an excess of water nor an excess of the Karl Fischer reagent, when the solvent is said to be at the equivalence point.

The principle and the chemical reaction of the Karl Fischer method are given in ISO 760.

4 Reagents

Karl Fischer reagent and the solvents are specified in ISO 760.

4.1 Use the Karl Fischer reagent in a more diluted form than that indicated in ISO 760; after having previously determined the water-equivalent of the available reagent, dilute the latter with the solvent so that its water-equivalent is between 0,8 and 1,2 mgH_2O/ml.

4.2 Use methanol (containing less than 0,01 g of water per 100 ml) as the solvent for the lightest olefins (ethylene and propylene) and ethylene glycol (containing less than 0,01 g of water per 100 ml) for the heavier olefins such as butadiene which are more soluble in this solvent.

5 Apparatus

5.1 Composition

5.1.1 Three stainless and low-temperature resistant steel cylinders, suitable for the sampling of light olefins. Each shall have an available capacity of about 150 ml, account being taken of an ullage of about 50 ml, and shall be fitted, *inter alia*, with two valves which permit the total available capacity (for the liquid and the gas) to be swept by a current of gas after the contents have been discharged.

NOTE — The sampling cylinders must satisfy the tests indicated in the actual legislation in each country and also for marking[1]. It is recommended that the internal surfaces of the cylinders be highly polished.

5.1.2 Vacuum oven, capable of being controlled at any desired temperature between 60 to 100 °C.

5.1.3 Cylinder containing compressed nitrogen, fitted with a perfectly clean pressure-reducing valve.

5.1.4 Drying device, capable of treating 60 litres of nitrogen per hour and of bringing it to a water content compatible with the requirements of 5.3.

NOTE — The drying agents recommended are phosphorus(V) oxide or "5A" molecular sieve of recognized analytical grade.

5.1.5 Karl Fischer apparatus, complying with the requirements of ISO 760, except with regard to the construction of the reaction vessel (see 5.1.6), and fitted with burettes of capacity 5 ml, graduated in 0,01 ml, or, alternatively, burettes of the same capacity, with capillary end of 0,1 mm, supplied under pressure.

1) The marking of sampling cylinders will be specified in ISO 7382.