UDC 661.7 : 620.1

Ref. No. : ISO/R 761-1968 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 761

METHOD FOR THE DETERMINATION OF BROMINE INDEX

1st EDITION
June 1968

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 761, *Method for the determination of bromine index*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1956 and led, in 1962, to the adoption of a Draft ISO Recommendation.

In November 1963, this Draft ISO Recommendation (No. 661) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Portugal
Austria	India	Romania
Belgium	Israel	Spain
Chile	Italy	U.A.R.
Colombia	Japan	United Kingdom
Czechoslovakia	Korea, Rep. of	U.S.S.R.
France	Netherlands	Yugoslavia
Germany	Poland	

One Member Body opposed the approval of the Draft :

New Zealand

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in June 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 761 June 1968

METHOD FOR THE DETERMINATION OF BROMINE INDEX

1. SCOPE

This ISO Recommendation describes a method for the determination of bromine index, in particular of acetic anhydride and of *n*-butanol.

2. DEFINITION

The bromine index is defined as the number of grammes of bromine consumed by 100 g of the sample under the conditions of test.

3. PRINCIPLE

Treatment of the sample, in acid solution, with excess potassium bromide-bromate solution and, after addition of potassium iodide solution, titration of the liberated iodine with a standard volumetric solution of sodium thiosulphate.

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Acetic acid,* glacial.

4.2 *Hydrochloric acid, d* = 1.18.

4.3 *Carbon tetrachloride.*

4.4 *Potassium iodide,* 150 g/l solution.

4.5 *Starch,* 5 g/l solution, freshly prepared.

4.6 *Sodium thiosulphate,* 0.10 M standard volumetric solution.

4.7 *Potassium bromide-potassium bromate* standard volumetric solution, approximately 0.1 N, accurately standardized. Dissolve 10.2 g of potassium bromide and 2.8 g of potassium bromate in water, dilute to 1000 ml. To determine the strength (N_B) of this solution accurately, take 25 ml, add 5 ml of potassium iodide solution (4.4) and 1 ml of hydrochloric acid solution (4.2), then titrate with the 0.10 M sodium thiosulphate standard volumetric solution (4.6).

NOTE. – 1 ml of 0.10 M sodium thiosulphate solution corresponds to 0.00799 g Br_2.

5. APPARATUS

Ordinary laboratory apparatus, and

5.1 *Three iodine flasks,* capacity 500 ml, glass-stoppered.

6. PROCEDURE

6.1 Blank test

At the same time as the determination on the sample, carry out a blank test in the same manner, but omitting the test portion.

6.2 Determination

6.2.1 Weigh accurately 3 to 5 g of the test sample, or pipette an equivalent quantity into a 50 ml one-mark volumetric flask containing 25 ml of carbon tetrachloride (4.3) as a solvent. Dilute to the mark with carbon tetrachloride and mix well.

6.2.2 Pipette immediately 10 ml of this solution into one of the iodine flasks (5.1) containing 50 ml of glacial acetic acid (4.1). Add 1 ml of hydrochloric acid solution (4.2). Shield the flask and contents from direct sunlight and keep it at a temperature of 20 ± 5 °C. With constant swirling of the contents of the flask, titrate with bromide-bromate solution (4.7) from a burette at the rate of 1 to 2 drops per second until the contents of the flask have assumed a yellow colour that persists for at least 5 seconds.

The yellow colour should match that obtained in a second iodine flask (5.1) by adding 2.5 ml of the bromide-bromate solution (4.7) to 50 ml of glacial acetic acid (4.1), 10 ml of carbon tetrachloride (4.3) and 1 ml of hydrochloric acid solution (4.2).

6.2.3 Add from a burette an additional 5 ml of bromide-bromate solution (4.7) as quickly as possible, stopper the flask, and immediately continue swirling for 40 ± 5 seconds.

Then add 5 ml of potassium iodide solution (4.4) by placing it in the cup of the flask and lifting the stopper slightly, thus preventing any possible loss of bromine vapour. Replace the stopper, shake vigorously, add 100 ml of water, again shake the solution vigorously for one minute and titrate it immediately with sodium thiosulphate solution (4.6) from another burette adding 1 ml of starch solution (4.5) towards the end of the titration. This back titration should normally use 5 to 10 ml of the sodium thiosulphate solution. If the amount used differs widely from this figure, repeat the determination with an adjusted mass of sample.

7. EXPRESSION OF RESULTS

$$\text{Bromine index} = 40 \cdot \frac{(V_B - V_B')\,N_B - (V_T - V_T')\,0.10}{M}$$

where

V_B is the volume, in millilitres, of bromide-bromate solution (4.7) used for the test portion;

V_T is the volume, in millilitres, of thiosulphate solution (4.6) used for the test portion;

V_B' is the volume, in millilitres, of bromide-bromate solution (4.7) used for blank;

V_T' is the volume in millilitres, of thiosulphate solution (4.6) used for blank;

N_B is the normality of bromide-bromate solution;

M is the mass, in grammes, of test sample taken.

Report the result to the nearest 0.1.