

INTERNATIONAL STANDARD

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Rubber — Polychloroprene latex — Determination of alkalinity

Caoutchouc — Latex de polychloroprène — Détermination de l'alcalinité



Reference number
ISO 13773:1997(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13773 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

Annexes A and B of this International Standard are for information only.

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Rubber – Polychloroprene latex – Determination of alkalinity

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a method for the determination of the alkalinity of polychloroprene latex prepared by the polymerization of chloroprene in an alkaline emulsion. It is not suitable for polychloroprene latex prepared in a non-ionic emulsion, for natural rubber latex concentrate nor necessarily for synthetic rubber latices other than polychloroprene.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling*.

ISO 976:1996, *Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

3 Principle

A test portion of the latex is titrated electrometrically against hydrochloric acid to the second point of inflexion of the titration curve (pH in the range 4 to 5). The alkalinity is calculated from the amount of acid required.

The alkalinity is commonly expressed as its two component parts in practice, i.e. the amount of acid required to the first point of inflexion (pH in the range 10 to 11) which measures the amount of free alkali (the alkaline reserve) and the amount of additional acid required to the second point of inflexion which is a measure of the amount of saponified anionic organic surfactants in the latex, usually referred to as the "delta titre".

NOTE — When using a pH-meter to control the titration, it was formerly the practice to define the points of inflexion as pH 10,5 and 4,5. However since these do not always correspond to the points of inflexion the results obtained would be approximate.

The results are expressed in millimoles of hydrochloric acid per 100 g of latex.

4 Reagents

For the analysis, use only reagents of recognized analytical grade and only distilled water free of dissolved carbon dioxide, or water of equivalent purity (grade 3 as defined in ISO 3696).

4.1 Stabilizer solution: a 10 % (m/m) solution of a non-ionic stabilizer of the alkyl phenol polyethylene oxide condensate type.

NOTE — The precise nature of the stabilizer is not critical provided that it imparts acid stability to the latex and does not interfere with the titration.

4.2 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0,1 \text{ mol/dm}^3$.

5 Apparatus

Standard laboratory apparatus, plus the following:

5.1 Automatic potentiometric titrator, fitted with an automatic burette and capable of delivering a volume of up to 50 cm^3 .

NOTE — It is permissible to use a manually operated 50 cm^3 burette. In this case, it is desirable to make a preliminary assessment of the approximate end-points.

5.2 Combined pH-electrode, with built-in glass electrode, suitable for use with solutions up to pH 14,0.

5.3 Magnetic or mechanical stirrer, with variable speed and non-metallic paddle or magnetic bar. The motor shall be properly grounded to avoid interference.

6 Sampling

Carry out sampling in accordance with one of the methods specified in ISO 123.

NOTE — Unsealed samples of alkaline polychloroprene latex rapidly absorb carbon dioxide from the air.

7 Procedure

Adjust the temperature of the reagents and test sample to $23 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ and calibrate the titrator in accordance with the manufacturer's instructions or as described for pH-meters in ISO 976.

Weigh, to the nearest 0,01 g, an approximately 35 g test portion of the latex into a clean glass or plastic beaker of about 150 cm^3 capacity. Insert and start the stirrer (5.3). Adjust the speed of the stirrer to obtain a small vortex at the surface of the liquid and slowly add 10 cm^3 of the stabilizer solution (4.1) from a pipette. Insert the electrode (5.2) and the reagent discharge tip of the titrator.

If there are signs of shock or coagulation on adding the stabilizer, dilute it with an equal volume of water before addition.

Start the titration for an automatic controlled addition of up to 50 cm^3 of hydrochloric acid (4.2) where the volume increment and addition speed are based on the pH change. Record the volumes of acid V_1 and V_2 measured at the first and second inflexion points of the titration curve (approximately pH 10,5 and 4,5, respectively). Record the precise pH at the points of inflexion.

If a manually operated burette is used (see note to 5.1), it is desirable to carry out a preliminary titration to give an approximate indication of the end-points. In subsequent titrations, add the hydrochloric acid in small increments through the range of the point of inflection.

If there are indications of coagulation during the determination, repeat the procedure but using 20 cm^3 of stabilizer solution.

Repeat the determination using a fresh test portion of latex. If the calculated figures for the alkalinity differ by more than 0,02 mmol, carry out the determination again using two fresh test portions.

8 Expression of results

8.1 Set the calculation parameters of the automatic titrator so as to obtain the alkaline reserve AR of the latex, expressed in millimoles of hydrochloric acid per 100 g of latex, using the equation:

$$AR = \frac{cV_1}{m} \times 100$$

where

c is the actual concentration of the hydrochloric acid, expressed in moles of HCl per cubic decimetre;

V_1 is the volume, in cubic centimetres, of hydrochloric acid required to reach the first end-point;

m is the mass, in grams, of the test portion.

8.2 Calculate the delta titre ΔT of the latex, expressed in millimoles of hydrochloric acid per 100 g of latex, as follows:

$$\Delta T = \frac{c(V_2 - V_1)}{m} \times 100$$

where

V_2 is the volume, in cubic centimetres, of hydrochloric acid required to reach the second end-point;

c , V_1 and m are as defined in 8.1.

8.3 Calculate the alkalinity A , of the latex, expressed in millimoles of hydrochloric acid per 100 g of latex, as follows:

$$A = AR + \Delta T$$

where AR and ΔT are as defined in 8.1 and 8.2.

9 Test report

The test report shall include the following particulars:

- a reference to this International Standard;
- all details necessary for identification of the sample;
- the alkalinity of the latex, expressed to the nearest 0,1 mmol;
- the alkaline reserve and the delta titre of the latex, expressed to the nearest 0,1 mmol of hydrochloric acid per 100 g of latex;
- the pH recorded at the first and second points of inflexion;
- the amount of stabilizer solution used;

- g) details of any unusual features noted during the determination;
- h) details of any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;
- i) the date and place of the test.

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Annex A

(informative)

Precision of the test method

With accurate operation and control, it is possible to attain the following precision:

A.1 Repeatability

Within the range $\pm 0,02$ mmol.

A.2 Reproducibility

Not established.

NOTE — The work carried out to generate the precision data was initiated before the publication of ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*. Consequently, the data are not expressed in the format recommended by this Technical Report.

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Annex B

(informative)

Example of the variation in pH during a titration

Volume cm ³	Increment cm ³	Signal pH	Signal change Δ pH	Difference Δ pH/cm ³
0,00	0,00	12,603	0,000	0,000
1,71	1,71	12,511	0,092	0,054
2,57	0,86	12,458	0,053	0,062
3,00	0,43	12,429	0,029	0,067
3,86	0,86	12,369	0,060	0,070
4,86	1,00	12,293	0,076	0,076
5,86	1,00	12,208	0,085	0,085
6,86	1,00	12,108	0,100	0,100
7,86	1,00	11,988	0,120	0,120
8,83	0,97	11,842	0,146	0,151
9,54	0,71	11,703	0,139	0,196
10,09	0,55	11,564	0,139	0,253
10,53	0,44	11,421	0,143	0,325
10,86	0,33	11,286	0,135	0,409
11,14	0,28	11,148	0,138	0,493
11,37	0,23	11,018	0,130	0,565
11,58	0,21	10,891	0,127	0,605
11,79	0,21	10,766	0,125	0,595
12,02	0,23	10,634	0,132	0,574
12,27	0,25	10,507	0,127	0,508
12,58	0,31	10,372	0,135	0,435
12,95	0,37	10,236	0,136	0,368
13,40	0,45	10,099	0,137	0,304
13,97	0,57	9,960	0,139	0,244
14,69	0,72	9,819	0,141	0,196
15,59	0,90	9,681	0,138	0,153
16,59	1,00	9,558	0,123	0,123
17,59	1,00	9,456	0,102	0,102
18,59	1,00	9,368	0,088	0,088
19,59	1,00	9,290	0,078	0,078
20,59	1,00	9,217	0,073	0,073
21,59	1,00	9,150	0,067	0,067
22,59	1,00	9,087	0,063	0,063
23,59	1,00	9,023	0,064	0,064
24,59	1,00	8,961	0,062	0,062
25,59	1,00	8,899	0,062	0,062
26,59	1,00	8,836	0,063	0,063
27,59	1,00	8,770	0,066	0,066
28,29	0,70	8,702	0,068	0,097
29,59	1,30	8,630	0,072	0,055
30,59	1,00	8,556	0,074	0,074
31,59	1,00	8,473	0,083	0,083
32,59	1,00	8,381	0,092	0,092
33,59	1,00	8,281	0,100	0,100
34,59	1,00	8,168	0,113	0,113
35,59	1,00	8,035	0,133	0,133
36,49	0,90	7,899	0,136	0,151
37,29	0,80	7,758	0,141	0,176
37,94	0,65	7,626	0,132	0,203
38,55	0,61	7,492	0,134	0,220

39,10	0,55	7,351	0,141	0,256
39,57	0,47	7,219	0,132	0,281
40,01	0,44	7,073	0,146	0,332
40,41	0,40	6,956	0,117	0,293
40,96	0,55	6,775	0,181	0,329
41,37	0,41	6,626	0,149	0,363
41,78	0,41	6,469	0,157	0,383
42,20	0,42	6,303	0,166	0,395
42,62	0,42	6,130	0,173	0,412
43,05	0,43	5,934	0,196	0,456
43,48	0,43	5,700	0,234	0,544
43,91	0,43	5,402	0,298	0,693
44,35	0,44	4,987	0,415	0,943
44,79	0,44	4,504	0,483	1,098
45,24	0,45	4,076	0,428	0,951
45,69	0,45	3,739	0,337	0,749
46,15	0,46	3,454	0,285	0,620
46,61	0,46	3,216	0,238	0,517
47,08	0,47	3,017	0,199	0,423
47,55	0,47	2,860	0,157	0,334
48,03	0,48	2,735	0,125	0,260
48,75	0,72	2,591	0,144	0,200
49,68	0,93	2,450	0,141	0,152
50,00	0,32	2,411	0,039	0,122

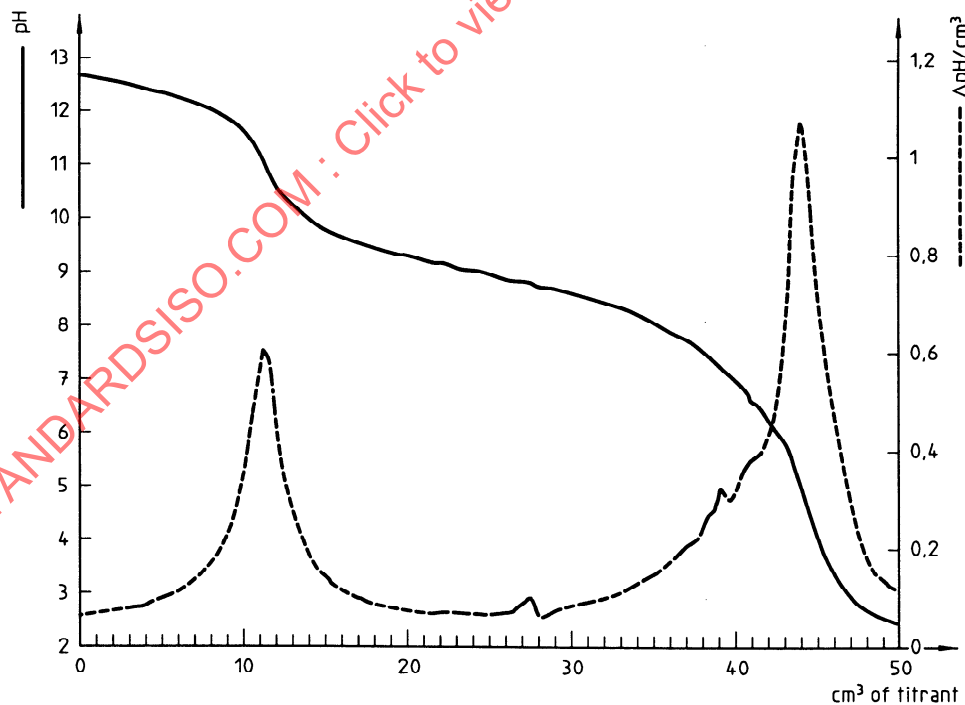


Figure B.1 – Example of titration and derivative curves