



DRAFT TANZANIA STANDARD

Methods of sampling and test for crude and refined glycerine
Part 3: Determination of glycerol content

TANZANIA BUREAU OF STANDARDS

Foreword

This Draft Tanzania Standard was prepared under the direction of Cosmetics and cremeries Technical Committee, which is under the supervision of Chemicals Divisional Standards Committee.

For the purpose of deciding whether a particular requirement of this Tanzania Standard is complied with, the final value, observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with TZS 4 (see clause 2).

This second edition cancels and replaces the first edition (TZS 1199-3:2015), which has been technically revised.

Draft Standard for Comment Only

Methods of sampling and test for crude and refined glycerine

Part 3: Determination of glycerol content

1 Scope

This Draft Tanzania Standard describes a method for the determination of the glycerol content of crude, technical, dynamite grade double-distilled and chemically pure glycerols. Other polyhydric alcohols with three or more adjacent hydroxyl groups also produce formic acid and so interfere with glycerol determination. The method cannot, therefore, be used directly on samples containing sugars.

1.1 Principle of the method

Glycerol reacts with sodium meta periodate (NaIO_4) in acid aqueous solution to produce formaldehyde and formic acid, and the amount of the latter is used as a measure of the glycerol content.

2 Normative references

The following referenced documents are indispensable for the application of this Tanzania Standard:

TZS 1199–2 *Crude and refined glycerine — Methods of sampling and test Part 2: Methods of sampling*

TZS 59/ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Definitions

For the purpose of this Tanzania Standard the following definitions shall apply:-

3.1 Standard glycerol

an aqueous solution containing about 90 percent of chemically pure glycerine complying with 3.2 and the concentration of which is known from density at 30 °C (see table 2) and moisture

3.2 Analytical reagent (AR) glycerine

the material shall be a clear, colourless, odourless, syrupy liquid, consisting essentially of glycerol. It shall have a sweet taste followed by a sensation of warmth. It shall be miscible in all proportions with water and with ethyl alcohol (90 percent (v/v), and it shall not be soluble in diethyl ether, chloroform or fixed oils. It shall be free from visible foreign matter.

4 Apparatus

4.1 **Burette** – 50 mL capacity, with 0.05 mL graduations.

4.2 **Pipette** – 50 mL capacity.

4.3 **Conical flasks** – 500 mL capacity, with glass stopper.

4.4 **pH Meter** – with glass electrode.

4.5 **Weighing pipette or bottle**

5 Reagents

5.1 Standard Sodium Hydroxide Solution – 0.1 N, accurately standardized. The solution shall be carbonate-free and should be crosschecked periodically against standard glycerol (see 3.1) by the method described under clause 6.

5.2 Sodium Hydroxide Solution – 0.05 N.

5.3 Phenol Red Indicator Solution – 0.05 percent (m/v) solution. Dissolve 0.05 g of phenol red in 10 mL of ethyl alcohol and make up to 100 mL with water.

5.4 Dilute Sulphuric Acid – 0.1 N.

5.5 Sodium Metaperiodate Solution – Dissolve 70 g of sodium metaperiodate in one litre of water containing 10 mL of approximately 0.5 N sulphuric acid, and store the solution in an amber coloured bottle in the dark.

NOTE – Sodium metaperiodate shall be a white crystalline powder containing not less than 98 percent sodium metaperiodate. If metaperiodate does not dissolve, it is not of reagent quality and a new supply should be obtained.

5.6 Ethylene Glycol – neutral and free from glycerol.

5.7 Standard Glycerol – 90%

NOTE – If necessary, density of glycerine may be calculated by multiplying its relative density (see CDC3 (4266) P1) by the density of water at the desired temperature.

6 Sampling

Prepare the laboratory samples as described in TZS 1199–2 .

7 Procedure

Using a weighing pipette or bottle, transfer into a conical flask a well-mixed and accurately weighed sample (see Note 1).

Add 100 mL of carbon dioxide-free water and 3 drops of phenol red indicator solution, and acidify with sulphuric acid solution to a definite yellow colour. Heat to boiling and cool to room temperature.

Adjust the pH value to about 8 ± 0.1 by adding 0.05 N sodium hydroxide solution drop by drop until the colour changes from yellow to just pink. If the colour of the original solution interferes with detection of the colour change of the indicator, use a pH meter.

Pipette accurately 50 ml of sodium metaperiodate solution, replace the glass stopper, mix by swirling gently and allow the flask to stand in the dark for 30 min.

Wash down the sides of the flask with distilled water, add 5 mL of ethylene glycol, replace the stopper, swirl gently, and allow the flask to stand in the dark for further 20 min. Titrate the liberated formic acid with 0.1 N standard sodium hydroxide solution (see 5.1) from yellow to pink end point.

Carry out a blank test simultaneously under similar test conditions (see Note 2).

NOTE 1 – The mass in grams of the sample should be within the limits given by the formula $(41 \pm 9)/P$, where P is the expected percentage of glycerol in the sample. When the glycerol content is not known, weigh the amount specified for 100 % glycerol, and from the results of this test, select the proper size sample taking into account that the results will tend to be high if the sample weighed is smaller than that required for the most accurate results.

NOTE 2 – If a pH meter is used, the end point for the blank should be $\text{pH } 6.5 \pm 0.1$ and that for the sample 8.1 ± 0.1 .

Table 1 – Glycerol content and density of aqueous solutions of refined glycerine

Density of Glycerine-Water Solutions											
Glycerine (%)	Density (g/cm ³)					Glycerine (%)	Density (g/cm ³)				
	15°C	15.5°C	20°C	25°C	30°C		15°C	15.5°C	20°C	25°C	30°C
100	1.26415	1.26381	1.26108	1.25802	1.25495	50	1.12870	1.12845	1.12630	1.12375	1.12110
99	1.26160	1.26125	1.25850	1.25545	1.25235	49	1.12600	1.12575	1.12360	1.12110	1.11845
98	1.25900	1.25865	1.25590	1.25290	1.24975	48	1.12325	1.12305	1.12090	1.11840	1.11580
97	1.25645	1.25610	1.25335	1.25030	1.24710	47	1.12055	1.12030	1.11820	1.11575	1.11320
96	1.25385	1.25350	1.25080	1.24770	1.24450	46	1.11780	1.11760	1.11550	1.11310	1.11055
95	1.25130	1.25095	1.24825	1.24515	1.24190	45	1.11510	1.11490	1.11280	1.11040	1.10795
94	1.24865	1.24830	1.24560	1.24250	1.23930	44	1.11235	1.11215	1.11010	1.10775	1.10530
93	1.24600	1.24565	1.24300	1.23985	1.23670	43	1.10960	1.10945	1.10740	1.10510	1.10265
92	1.24340	1.24305	1.24035	1.23725	1.23410	42	1.10690	1.10670	1.10470	1.10240	1.10005
91	1.24075	1.24040	1.23770	1.23460	1.23150	41	1.10415	1.10400	1.10200	1.09975	1.09740
90	1.23810	1.23775	1.23510	1.23200	1.22890	40	1.10145	1.10130	1.09930	1.09710	1.09475
89	1.23545	1.23510	1.23245	1.22935	1.22625	39	1.09875	1.09860	1.09665	1.09445	1.09215
88	1.23280	1.23245	1.22975	1.22665	1.22360	38	1.09605	1.09590	1.09400	1.09180	1.08955
87	1.23015	1.22980	1.22710	1.22400	1.22095	37	1.09340	1.09320	1.09135	1.08915	1.08690
86	1.22750	1.22710	1.22445	1.22135	1.21830	36	1.09070	1.09050	1.08865	1.08655	1.08430
85	1.22485	1.22445	1.22180	1.21870	1.21565	35	1.08800	1.08780	1.08600	1.08390	1.08165
84	1.22220	1.22180	1.21915	1.21605	1.21300	34	1.08530	1.08515	1.08335	1.08125	1.07905
83	1.21955	1.21915	1.21650	1.21340	1.21035	33	1.08265	1.08245	1.08070	1.07860	1.07645
82	1.21690	1.21650	1.21380	1.21075	1.20770	32	1.07995	1.07975	1.07800	1.07600	1.07380
81	1.21425	1.21385	1.21115	1.20810	1.20505	31	1.07725	1.07705	1.07535	1.07335	1.07120
80	1.21160	1.21120	1.20850	1.20545	1.20240	30	1.07455	1.07435	1.07270	1.07070	1.06855
79	1.20885	1.20845	1.20575	1.20275	1.19970	29	1.07195	1.07175	1.07010	1.06815	1.06605
78	1.20610	1.20570	1.20305	1.20005	1.19705	28	1.06935	1.06915	1.06755	1.06560	1.06355
77	1.20335	1.20300	1.20030	1.19735	1.19435	27	1.06670	1.06655	1.06495	1.06305	1.06105
76	1.20060	1.20025	1.19760	1.19465	1.19170	26	1.06410	1.06390	1.06240	1.06055	1.05855
75	1.19785	1.19750	1.19485	1.19195	1.18900	25	1.06150	1.06130	1.05980	1.05800	1.05605
74	1.19510	1.19480	1.19215	1.18925	1.18635	24	1.05885	1.05870	1.05720	1.05545	1.05350
73	1.19235	1.19205	1.18940	1.18650	1.18365	23	1.05625	1.05610	1.05465	1.05290	1.05100
72	1.18965	1.18930	1.18670	1.18380	1.18100	22	1.05365	1.05350	1.05205	1.05035	1.04850
71	1.18690	1.18655	1.18395	1.18110	1.17830	21	1.05100	1.05090	1.04950	1.04780	1.04600
70	1.18415	1.18385	1.18125	1.17840	1.17565	20	1.04840	1.04825	1.04690	1.04525	1.04350
69	1.18135	1.18105	1.17850	1.17565	1.17290	19	1.04590	1.04575	1.04440	1.04280	1.04105
68	1.17860	1.17830	1.17575	1.17295	1.17020	18	1.04335	1.04325	1.04195	1.04035	1.03860
67	1.17585	1.17555	1.17300	1.17020	1.16745	17	1.04085	1.04075	1.03945	1.03790	1.03615
66	1.17305	1.17275	1.17025	1.16745	1.16470	16	1.03835	1.03825	1.03695	1.03545	1.03370
65	1.17030	1.17000	1.16750	1.16475	1.16195	15	1.03580	1.03570	1.03450	1.03300	1.03130
64	1.16755	1.16725	1.16475	1.16200	1.15925	14	1.03330	1.03320	1.03200	1.03055	1.02885
63	1.16480	1.16445	1.16205	1.15925	1.15650	13	1.03080	1.03070	1.02955	1.02805	1.02640
62	1.16200	1.16170	1.15930	1.15655	1.15375	12	1.02830	1.02820	1.02705	1.02560	1.02395
61	1.15925	1.15895	1.15655	1.15380	1.15100	11	1.02575	1.02565	1.02455	1.02315	1.02150
60	1.15650	1.15615	1.15380	1.15105	1.14830	10	1.02325	1.02315	1.02210	1.02070	1.01905
59	1.15370	1.15340	1.15105	1.14835	1.14555	9	1.02080	1.02075	1.01970	1.01835	1.01670
58	1.15095	1.15065	1.14830	1.14560	1.14285	8	1.01840	1.01835	1.01730	1.01600	1.01440
57	1.14815	1.14785	1.14555	1.14285	1.14010	7	1.01600	1.01590	1.01495	1.01360	1.01205
56	1.14535	1.14510	1.14280	1.14015	1.13740	6	1.01360	1.01350	1.01255	1.01125	1.00970
55	1.14260	1.14230	1.14005	1.13740	1.13470	5	1.01120	1.01110	1.01015	1.00890	1.00735
54	1.13980	1.13955	1.13730	1.13465	1.13195	4	1.00875	1.00870	1.00780	1.00655	1.00505
53	1.13705	1.13680	1.13455	1.13195	1.12925	3	1.00635	1.00630	1.00540	1.00415	1.00270
52	1.13425	1.13400	1.13180	1.12920	1.12650	2	1.00395	1.00385	1.00300	1.00180	1.00035
51	1.13150	1.13125	1.12905	1.12650	1.12380	1	1.00155	1.00145	1.00060	0.99945	0.99800
						0	0.99913	0.99905	0.99823	0.99708	0.99568

8 Calculation

Glycerol content, percent by mass = $\frac{9.209 (S - B) N}{m}$

where

S = volume in mL of standard sodium hydroxide solution required for the sample,
 B = volume in mL of standard sodium hydroxide solution required for the blank,
 N = Normality of standard sodium hydroxide solution, and
 m = mass in g of the material taken for the test.

NOTE – When the titration is carried out with phenol red solution as the indicator, add 0.3 % (expressed on 100 % glycerol) to the observed glycerol result. No correction is necessary if a pH meter is used.

9 Test report

The test report shall include the following particulars:

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this Tanzania Standard, or regarded as optional.

Bibliography

TZS 1199-3:2015 *Methods of sampling and test for crude and refined glycerine Part 3: Determination of glycerol content*