1.11 Colour of liquid

2017-01

[Note from the Secretariat. In order to replace chromium (VI) salts in The International Pharmacopoeia the procedure previously used to determine the colour of liquids will be replaced gradually with the corresponding procedure taken over from the European Pharmacopoeia.

For the period of transition, both procedures are kept: the previous procedure under section 1.11.1 and the new procedure under section 1.11.2.

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1.11.1 Colour of liquids

The test for colour of liquids is carried out by comparing the test solution prepared as specified in the monograph with a standard colour solution indicated in the monograph. The composition of the standard colour solution is selected depending on the hue and intensity of the colour of the test solution corresponding to the limits permitted in the specifications.

Recommended procedure

Unless otherwise specified in the monograph carry out the comparison in flat-bottomed tubes of transparent glass that are matched as closely as possible in internal diameter and in all other respects (tubes of about 16 mm internal diameter are suitable). Use 10 mL of the test solution and 10 mL of the standard colour solution; the depth of liquid should be about 50 mm. The colour of the test solution is not more intense than the standard colour when viewed down the vertical axis of the tubes in diffused light against a white background.

Stock colour standard solutions

Yellow stock standard TS

To 9.5 mL of cobalt colour TS, add 1.9 mL of copper colour TS, 10.7 mL of dichromate colour TS, 4.0 mL of iron colour TS, dilute to 100.0 mL with sulfuric acid (~10 g/L) TS and mix.

Red stock standard TS

To 40.5 mL of cobalt colour TS, add 6.1 mL of copper colour TS, 6.3 mL of dichromate colour TS, 12.0 mL of iron colour TS, dilute to 100.0 mL with sulfuric acid (~10 g/L) TS and mix.

Green stock standard TS

To 3.5 mL of cobalt colour TS, add 20.1 mL of copper colour TS, 10.4 mL of dichromate colour TS, 4.0 mL of iron colour TS, dilute to 100.0 mL with sulfuric acid (~10 g/L) TS and mix.

Brown stock standard TS

To 35.0 mL of cobalt colour TS, add 17.0 mL of copper colour TS, 8.0 mL of dichromate colour TS, dilute to 100.0 mL with iron colour TS and mix.

Standard colour solutions

The standard colour solution is prepared by suitably diluting the stock standard solutions (yellow, red, green and brown stock standard TS) with sulfuric acid (~10 g/L) TS. The designation of the standard colour solution is composed of two letters indicating the stock standard solution (Yw for yellow, Rd for red, Gn for green and Bn for brown) and of a number describing the dilution as given below:

	Other stand and		
Dilution number	Slock standard	Sullunc aciu	
for standard	solution	(~10g/L) TS	
colour solutions	(mL)	(mL)	
0	0.78	99.22	
	4 50	00.44	
1	1.50	90.44	
2			
2	5.12	50.00	
3	6.25	93.75	
4			
4	12.50	87.50	
5	25.00	75.00	
°	20.00	10.00	
6	50.00	50.00	
7	100.00		
1	100.00	-	

Standard colour solution numbers 4-7 may be stored in sealed glass containers, protected from sunlight but the more dilute

standard colour solutions should be prepared as required.

Definition of "colourless"

A solution is considered colourless if it is not more intensely coloured than any of the standard colour solutions Bn0, Yw0, Gn0 or Rd0.

1.11.2 Degree of coloration of liquids

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The examination of the degree of coloration of liquids in the range brown-yellow-red is carried out by one of the two methods below as prescribed in the monograph.

A solution is colourless if it has the appearance of water R or the solvent or is not more intensely coloured than reference solution B₉.

METHOD I

Using identical tubes of colourless, transparent, neutral glass of 12 mm external diameter, compare 2.0 mL of the liquid to be examined with 2.0 mL of water R or of the solvent or of the reference solution (see tables of reference solutions) prescribed in the monograph. Compare the colours in diffused daylight, viewing horizontally against a white background.

METHOD II

Using identical tubes of colourless, transparent, neutral glass with a flat base and an internal diameter of 15 mm to 25 mm, compare the liquid to be examined with water R or the solvent or the reference solution (see tables of reference solutions) prescribed in the monograph, the depth of the layer being 40 mm. Compare the colours in diffused daylight, viewing vertically against a white background.

REAGENTS

Primary solutions

Yellow solution. Dissolve 46 g of ferric chloride R in about 900 mL of a mixture of 25 mL of hydrochloric acid (~330 g/L) TS and 975 mL of water R and dilute to 1000.0 mL with the same mixture. Titrate and adjust the solution to contain 45.0 mg of $\text{FeCl}_{3,6H}_2$ O per mL by adding the appropriate volume of the same acidic mixture. Protect the solution from light.

Titration. Place in a 250 mL conical flask fitted with a ground-glass stopper, 10.0 mL of the solution, 15 mL of water R, 5 mL of hydrochloric acid (~330 g/L) TS and 4 g of potassium iodide R, close the flask, allow to stand in the dark for 15 minutes and add 100 mL of water R. Titrate the liberated iodine with sodium thiosulfate (0.1 mol/L) VS, using 0.5 mL of starch solution TS, added towards the end of the titration, as indicator.

1 mL of sodium thiosulfate (0.1 mol/L) VS is equivalent to 27.03 mg of $\text{FeCl}_3, 6\text{H}_2\text{O}$.

Red solution. Dissolve 60 g of cobalt (II) chloride R in about 900 mL of a mixture of 25 mL of hydrochloric acid (~330 g/L) TS and 975 mL of water R and dilute to 1000.0 mL with the same mixture. Titrate and adjust the solution to contain 59.5 mg of $CoCl_2, 6H_2$ O per mL by adding the appropriate volume of the same acidic mixture.

Titration. Place in a 250 mL conical flask fitted with a ground-glass stopper, 5.0 mL of the solution, 5 mL of hydrogen peroxide (~30 g/L) TS and 10 mL of sodium hydroxide (~300 g/L) TS. Boil gently for 10 minutes, allow to cool and add 60 mL of sulfuric acid (~100 g/L) TS and 2 g of potassium iodide R. Close the flask and dissolve the precipitate by shaking gently. Titrate the liberated iodine with sodium thiosulfate (0.1 mol/L) VS, using 0.5 mL of starch solution TS, added towards the end of the titration, as indicator. The end-point is reached when the solution turns pink.

1 mL of sodium thiosulfate (0.1 mol/L) VS is equivalent to 23.79 mg of CoCl₂,6H₂O.

Blue solution. Dissolve 63 g of copper (II) sulfate R in about 900 mL of a mixture of 25 mL of hydrochloric acid (~330 g/L) TS and 975 mL of water R and dilute to 1000.0 mL with the same mixture. Titrate and adjust the solution to contain 62.4 mg of $CuSO_4$,5 H₂O per mL by adding the appropriate volume of the same acidic mixture.

Titration. Place in a 250 mL conical flask fitted with a ground-glass stopper, 10.0 mL of the solution, 50 mL of water R, 12 mL of acetic acid (~120 g/L) TS and 3 g of potassium iodide R. Titrate the liberated iodine with sodium thiosulfate (0.1 mol/L) VS, using 0.5 mL of starch solution TS, added towards the end of the titration, as indicator. The end-point is reached when the solution shows a slight pale brown colour.

1 mL of sodium thiosulfate (0.1 mol/L) VS is equivalent to 24.97 mg of $CuSO_4$,5H₂O.

Standard solutions

Using the three primary solutions, prepare the five standard solutions as follows (Table 1).

Table 1

	Volume in mL			
Standard solution	Yellow solution	Red solution	Blue solution	Hydrochloric acid (~10 g/L) TS
B (brown)	3.0	3.0	2.4	1.6
BY (brownish-vellow)	2.4	1.0	0.4	6.2
				7.0
	2.4	0.0	0.0	7.0
GY (greenish-yellow)	9.6	0.2	0.2	0.0
R (red)	1.0	2.0	0.0	7.0

Reference solutions for Methods I and II

Using the five standard solutions prepare the following reference solutions.

Table 2. Reference solutions B

	Volumes in mL	Volumes in mL		
Reference	Standard solution B	Hydrochloric acid (~10 g/L) TS		
solution				
B ₁	75.0	25.0		
B ₂	50.0	50.0		
B ₃	37.5	62.5		
B ₄	25.0	75.0		
B ₅	12.5	67.5		
B ₆	5.0	95.0		
B ₇	2.5	97.5		
B ₈	1.5	98.5		
B ₉	1.0	99.0		

Table 3. Reference solutions BY

Reference	Volumes in mL	Volumes in mL		
	Standard solution BY	Hydrochloric acid (~10 g/L) TS		
solution				
BY ₁	100.0	0.0		
BY ₂	75.0	25.0		
BY ₃	50.0	50.0		
BY ₄	25.0	75.0		
BY ₅	12.5	67.5		
BY ₆	5.0	95.0		
BY_	2 5	97.5		
5.7	2.0	01.0		

Table 4. Reference solutions Y

	Molunge in ml		
Deference	Otondowl colution V	- Underselatoria acid (_40 m/l \ TO	
L'elelelle	Stanuaru Solution T	Hydrochione acid (~ To g/L) To	
adution			
Solution			
N/	100.0		
1 1	100.0	0.0	
¥	75.0	25.0	
2	10.0	20.0	
		50.0	
Ϋ́ ₃	50.0	50.0	
5			
V		75.0	
¹ 4	23.0	75.0	

<u>M</u>		07 F
15	12.5	07.3
5		
V		
	0.0	90.0
0		
N .		
Ϊ ₇	Z.3	97.0
1		

Table 5. Reference solutions GY

	Volumes in mL			
	Oten dend celution OV			
Reference	Standard Solution GT			
solution				
GY ₁	25.0	75.0		
	45.0			
G1 ₂	15.0	05.0		
ev		01 5		
G1 ₃	0.5	91.5		
GY		as a		
0 ¹ 4	5.0	55.6		
GV		07 ∩		
0 ¹ 5	0.0	51.6		
QV	4 5	98.5		
G1 ₆	1.5	90.0		
0 /	0.75	00.05		
G17	0.75	99.20		

Table 6. Reference solutions R

Reference	Volumes in mL		
	Standard solution R	Hydrochloric acid (~10 g/L) TS	
solution			
R ₁	100.0	0.0	
R ₂	75.0	25.0	
R ₃	50.0	50.0	
R ₄	37.5	62.5	
+ R-	25.0	75.0	
5 R	12.5	87.5	
¹ ⁶	T2.5		
^г 7	5.0	95.0	

Storage

For Method I the reference solutions may be stored in sealed tubes of colourless, transparent, neutral glass of 12 mm external diameter, protected from light.

For Method II prepare the reference solutions immediately before use from the standard solutions.