Preparation of standard solutions

Solutions of accurately known strength are called standard solutions. A standard solution contains a known weight of reagent in a definite volume of solution. Molecular weight and atomic weight of commonly used chemicals has been shown in Table 6.1.

Molar solution

Molar solution is one, which contains one molecular weight of the reagent in one litre of the solution. Molarity is expressed as M.

Normal solution

Normal solution is one, which contains one equivalent weight of the reagent in one litre of the solution. Normality is expressed as N.

Equivalent weight of acid = Molecular weight/ No. of replaceable H ions

Table 6.1 Name, formula, molecular weight and equivalent weight of commonly used chemicals

Name	Formula	Mol. wt. (g/mol)	Eq. wt. (g/mol)
Hydrochloric acid	HCl	36.5	36.5
Nitric acid	HNO_3	63	63
Potassium hydroxide	KOH	56	56
Calcium hydroxide	$Ca(OH)_2$	74	37
Potassium dichromate	$K_2Cr_2O_7$	294	49
Sodium thiosulphate	$Na_2S_2O_3.5H_2O$	248	248
Sodium chloride	NaCl	58.5	58.5
Potassium chloride	KCl	74.5	74.5
Iodine	I_2	254	127

Most of the synthetic dyes in generally used as indicators are organic substances of complex structure. Among the most reliable of these indicators are methyl red and phenolphthalein (Table 6.2).

Table 6.2 Common indicators used in animal nutrition laboratory

Indicator	pH range	End point	Preparation
Methyl red	4.4 to 6.3	Pink in acidic medium and colourless in basic medium	For preparing a stock solution, 0.2 g of dye is dissolved in 100 ml of alcohol and filtered
Methyl orange	2.9 to 4.0	Orange in acidic medium and colourless in basic medium	For preparing a stock solution, 0.1 g of dye is dissolved in 100 ml of distilled water, filtered and used
Phenolphthalein	8.3 to 10.0	Pink in basic medium and colourless in acidic medium	For preparing a stock solution, 0.2 g of phenolphthalein is dissolved in 110 ml of alcohol and 90 ml of distilled water

There are a few standard solutions which are used for analysis of feed stuffs:

1. N/10 H_2SO_4
2. N/10 NaOH
3. N/10 $KMnO_4$

4. 0.256 N (1.25% (w/v)) H_2SO_4
5. 0.313 N (1.25% w/v) NaOH
6. 40 per cent NaCl (w/v)
7. 3 per cent KNO_3 (w/v)
8. 20 per cent ammonium molybdate (w/v)
9. 50 per cent HCl (w/v)

Certain primary standard solutions are also required for standardization of the above solutions. These are:

1. N/10 Na_2CO_3
2. N/10 $(COOH)_2 . 2H_2O$

Preparation of N/10 H_2SO_4

Equivalent weight of H_2SO_4 = 49 g

Specific gravity = 1.84 g/ml

So, volume of 49 g H_2SO_4 = 26.6 ml

Concentrated H_2SO_4 (reagent grade) is about 97 per cent pure.

Therefore, actual amount of concentrated H_2SO_4 required for 1.0 litre of N/10 H_2SO_4 solution =

$$\frac{100}{97} \times 26.6 = 27.42 \text{ ml}$$

Thus, for 1.0 litre of N/10 H_2SO_4 solution, 2.74 ml of concentrated H_2SO_4 is required.

Procedure

Take 2.74 ml sulphuric acid in a beaker half-filled with distilled water. Transfer the contents and washings to a volumetric flask (1 litre) and make volume up to the mark. Shake well and titrate this solution with 10 ml of 0.1 N Na_2CO_3 using mixed / methyl orange as an indicator. Repeat the titration to get at least three concordant readings.

Standardization

Suppose 10 ml of 0.1 N Na_2CO_3 = 9.5 ml of H_2SO_4

$$V_1N_1 = V_2N_2$$

$$10 \times 0.1 \text{ N} = 9.5 \times N_2$$

$$N_2 = 0.10526$$

To prepare 1 litre N/10 H_2SO_4, the volume of 0.10526 N acid required is 1000 x 0.1/0.10526 = 950 ml. Take 950 ml of 0.10526 N acid and dilute it to one litre. Check it again with N/10 Na_2CO_3 for three times. It must neutralize equal volume of N/10 Na_2CO_3 solution. Label it as 0.1 N H_2SO_4.

Precautions

Add H_2SO_4 with the help of a burette.

Never add water to an acid.

Preparation of N/10 NaOH solution

Molecular weight of NaOH = 40
Acidity (number of replaceable OH group) = 1
Equivalent weight of NaOH = 40

Therefore, 4 g of NaOH dissolved in one litre of solution will give N/10 NaOH solution.

Procedure

Weigh quickly 4 g NaOH in a beaker (as it is hygroscopic) and dissolve it in distilled water (preferably CO_2-free). Transfer the contents and the washings to a volumetric flask (1 litre). Cool and then make volume up to the mark. Shake well and standardize this solution against N/10 oxalic acid using phenolphthalein as an indicator. Label it as 0.1 N NaOH solution.

Preparation of N/10 $KMnO_4$ solution

Dissolve 3.2 g $KMnO_4$ in one litre of distilled water. Boil it for 10-15 minutes and then allow to stand for few days and then filter it through glass wool.

Take 10 ml of N/10 oxalic acid in a beaker. Add 5 ml dilute sulphuric acid, warm it to 60-70ºC and titrate against $KMnO_4$ from the burette till a light pinkish colour appears. Take three concordant readings.

Suppose 10 ml 0.1 N oxalic acid = 9.75 ml of $KMnO_4$

$$V_1N_1 = V_2N_2$$

$$10 \times 0.1\ N = 9.75 \times N_2$$

$$N_2 = \frac{10 \times 0.1\ N}{9.75} = 0.10256$$

To prepare 1000 ml 0.1 N $KMnO_4$ solution the volume of $KMnO_4$ will be taken.

$$\frac{100 \times 9.75 \times 0.1}{10 \times 0.1}$$

Now take 975 ml of prepared $KMnO_4$ solution and make it 1000 ml by adding distilled water.

Note:

Ordinary or even pure distilled water contains traces of organic matter which reduces the $KMnO_4$ solutions. That is why the solution is boiled and kept for some time before standardization.

In the absence of sufficient amount of dilute H_2SO_4 or due to the rapid addition of $KMnO_4$ in titration flask, brown turbidity (manganous oxide) may appear.

Preparation of N/10 Na_2CO_3 solution

Molecular weight of Na_2CO_3 = 106

$Na_2CO_3 = 2\ HCl = 2\ NaCl + H_2O + CO_2$

So, acidity of Na_2CO_3 = 2

Equivalent weight of Na_2CO_3 = 53

Therefore, 5.3 g Na_2CO_3 is required for each litre of solution to make N/10 Na_2CO_3. Na_2CO_3 is hygroscopic, therefore, it must be made perfectly anhydrous before it is weighed out. Quantity of acid/ alkali required for preparation of different molar/normal solutions has been shown in Table 6.3.

Table 6.3 Acid/ alkali required for preparation of different normal solutions

Normality	95% NaOH (g/l), M=N	36% HCl (ml/l), M=N	98% H_2SO_4 (ml/l), M=2N	<68% HNO_3 (ml/l), M=N
0.1 N	4.2	8.5	2.7	6.5
0.5 N	21.0	42.6	13.7	32.8
1.0 N	42.1	85.3	27.4	65.7
1.5 N	63.1	127.9	41.1	98.5
2.0 N	84.2	170.6	54.8	131.4
2.5 N	105.2	213.2	68.5	164.2
5.0 N	210.5	426.5	137	328.5
10.0 N	421	853	274	657

M = Molar; N = Normal

Procedure

Take 6-7 g of Na_2CO_3 (A.R.) in a nickel crucible and heat it in a hot air oven at about 100ºC for few hours so as to drive out any moisture and to convert any moisture and to convert any preformed $NaHCO_3$ to Na_2CO_3. Cool in a desiccator and weigh exactly 5.3 g dried salt and dissolve it in a little quantity of freshly boiled distilled water. Transfer it to one litre measuring flask and make volume up to the mark. Shake well and label it as 0.1 N Na_2CO_3 solution.

Preparation of N/10 oxalic acid

Oxalic acid $(COOH)_2.2H_2O$ is to be dissolved in one litre of distilled water to get N/10 oxalic acid solution.

Procedure

Weigh accurately 6.3 g $(COOH)_2 2H_2O$ and transfer it to a volumetric flask (1 litre), half-filled with distilled water. Shake well and make the volume up to the mark. Label it as N/10 oxalic acid solution.

Note: If anhydrous oxalic acid $(COOH)_2$ is available then dissolve 4.5 g of the acid in one litre of distilled water to get 0.1 N oxalic acid solution.

Preparation of standardized 0.313 N (1.25%) NaOH solution

Add 13.16 g of NaOH (95% NaOH) in one litre distilled water and shake well. Standardize this solution against known concentration of oxalic acid solution using phenolphthalein as an indicator.

Preparation of standardized 0.256 N (1.25per cent (w/v) H_2SO_4 solution

To prepare 1.25 per cent (w/v) H_2SO_4 solution, 12.5 g of H_2SO_4 (100 per cent) is to be added to distilled water to make the volume 1000 ml.

$$\text{Volume of } H_2SO_4 \text{ be taken} = \frac{12.5 \times 100}{1.84 \times 97} = 7 \text{ ml}$$

Procedure

Add 7.0 ml concentrated H_2SO_4 (specific gravity 1.84 g/ml and 97per cent concentration) in a 1000 cc volumetric flask half-filled with distilled water. Shake well and add distilled water to make volume up to the mark. Standardize this solution against known concentration of Na_2CO_3 using mixed/methyl orange indicator.

Precaution and preservation of standard solutions

The bottle must be kept tightly stoppered to prevent evaporation of solvent. Some solutions must be protected from atmospheric gases. For example, sodium hydroxide solution is affected by atmospheric CO_2.

$$2NaOH + CO_2 = Na_2CO_3 + H_2O$$

But never put a glass stopper on NaOH solution container because NaOH will react with air and glass between stopper and neck of volumetric flask. It will fix permanently and you cannot remove the glass stopper from volumetric flask. $KMnO_4$ solution should be preserved

in colour (amber) bottles. The container should be shaken well before the withdrawal of a portion of solution to ensure uniform composition of the solution.