SIDE-BENCH REAGENTS PREPARATIONS

The following is a list of reagents required to carry out chemical analysis and which should be readily available in reagent bottles on the side-bench in the laboratory. The directions for preparing the solutions are given below.

NOTE: When diluting any concentrated acid, always add acid to water and never add water to acid.

- **Dilute HCl, 2 molar:** Add 200cm^3 of concentrated acid to 800 cm^3 of water.
- **Dilute HNO_3, 2 molar:** Add 125cm^3 of concentrated acid to 875cm^3 of water.
- **DiluteH_2SO_4, 1 molar:** Add 55cm^3 of the concentrated acid to 500cm^3 of water, make up to 1dm^3 with distilled water.
- **Dilute ethanoic(acetic) acid, 2 molar:** Dilute 114cm^3 glacial acid with water to 1dm^3
- **Ammonia solution, 2 molar:** 153cm^3 commercial product per dm^3.
- **Sodiumhydroxide, 2molar :** Dissolve 80g of pellets in distilled water and make up to 1dm^3 with distilled water. Store in a reagent bottle with plastic stopper.
- **Ammonium trioxocarbonate(IV) [NH_4CO_3], 2 molar:** Dissolve 160g of commercial solid in a mixture of 200cm^3 of concentrated ammonia solution and 800cm^3 of water.
- **Barium chloride, 0.5 molar:** Dissolve122g of solid in 1dm^3 of water.
- **Bromine water:** Add 5cm^3 of bromine to every 100cm^3 of water shake well and store in amber coloured bottle.
- **Bromine in tetrachloromethane(carbon tetrachloride,CCl_4):** Add 5cm^3 of bromine to every 1000cm^3 of CCl_4 solution. Shake weel to dissolve, store in amber coloured(dark) bottle.
- **Calcium chloride, 0.5 molar:** Dissolve 55g of solid in water and make up to dm^3.
- **2,4-dinitrophenylhydrazine:** Dissolve 2.0g of the solid in 10cm^3 of concentrated H_2SO_4. Add this solution to 200cm^3 of absolute ethanol, dilute to 500cm^3 with water. Thoroughly, allow to stand, then filter and use the filtrate. OR Dissolve 40g of 2,4-dinitrophenylhydrazine in 80ml concentrated sulphuric acid. Cool and add 900ml methanol and 100ml water.

- **Fehling's solution A:** Dissolve 35g of hydrated $CuSO_4$ in water, add few drops of concentrated H_2SO_4 and dilute to 500cm^3 with water.
- **Fehling's solution B:** Dissolve 60g of pure NaOH and 173g of Rochette salt (sodium potassium tartarate) in 500cm^3 of water, filter if necessary. Mix equal volumes of Fehling's solutions A and B just before use.
- **Hydrogen peroxide:** 20 volume commercial product; or dilute 200cm^3 of 100 volume commercial product with water and make up to 1dm^3. Store in a dark bottle.
- **Lead ethanoate (acetate), 0.5 molar:** Dissolve 95g of solid in 500cm^3 of water. Shake with about 5g of calcium hydroxide in 1 dm^3 water. Allow to stand for a few hours. Filter and use the filtrate.
- **Iron(III) chloride;** Dissolve 67g of the crystals in 200cm^3 of water.
- **Mercury(II) chloride, 0.25 molar:** Dissolve 7.00g of solid in 500cm^3 of water.
- **Potassium heptaoxodichromate(VI),$K_2Cr_2O_7$:** Dissolve 1g of in a mixture of 200cm^3 of water and 40cm^3 of 1M H_2SO_4.
- **Potassium tetraoxomanganate(VII), $KMnO_4$:** Dissolve 1.5g of $KMnO_4$ in a mixture of 400cm^3 of water and 100cm^3 of 1M H_2SO_4. Store in amber coloured bottle.
- **Potassium hexacyanoferrate (II), 0.25 molar:** Dissolve 105gof solid in 1dm^3 of water.
- **Potassium iodide, 0.5 molar:** Dissolve 83g of solid in 1dm^3 of water.
- **Silvertrioxonitrate(V), 0.1 molar**: Dissolve 17g of solid in 1dm^3 of water. Store in amber coloured(dark) bottle.
- **Sodiumtrioxocarbonate(IV), 1.0 molar:** Dissolve 53g of solid in 500cm^3 of water.
- **Sodium tetraoxophosphate(V), 1.0molar**: Dissolve 60g in 500cm^3 of water.
- **Starch solution:** make a paste with 1g of soluble starch in a small amount of water and add about 100cm^3 of boiling water. Boil the mixture for a while to obtain a clear solution. Prepare when ready to use.
- **Tin(II)chloride, 1.0 molar:** Dissolve 56g of solid crystals in 500cm^3 of 1.0 molar HCl solution. To prevent oxidation, add a few pieces of tin metal.
- **Barium trioxonitrate(V), 0.5 molar**: Dissolve 130g of solid in 1000cm^3 of water.
- **Iodine solution**: Dissolve 120g of solid iodine in 1000cm^3 of distilled water to which 3.5g of KI have been dissolved before. OR Dissolve 20g KI and 10g iodine crystals in 100ml

water.

- **Disodium trioxosulphate(II);** $Na_2S_2O_3.5H_2O$, 1molar: dissolve 248g of solid in $1dm^3$of distilled water.
- **Benedict's solution(qualitative):** Dissolve 86.5g of sodiumcitrate and 50g of anhydrous sodium carbonate in about 350ml of water. Filter if necessary. Add a solution of 8.6g of $CuSO_4$ in 50 ml of water with constant stirring. Dilute to 500ml. The resultaing solution should be perfectly clear; if not, filter through a fluted filter paper.
- **Benedict's solution(quantitative):** Dissolve 200g of sodiumcitrate and 75g of anhydrous sodium carbonate and 125g potassium thiocynate in about 600ml of water. Dissolve separately ,18g of $CuSO_4$ in 100 ml of water. When the solutions have cooled, mix them together with stirring. Now add 5ml of a 5% potassium ferrocyanide to the solution, and make up to 1 litre.
- **Biuret reaget (quantitative):** Dissolve in order, 3g $CuSO_4$, 5g KI, 9g potassium sodium tartrate, and 8g NaOH in about 600ml water. Make up the dissolved solids to 1 litre solution.
- **Dimethyl glyoxime**: Dissolve 1g of the solid in 100ml of 95% ethylalcohol.
- **Ferric thiocyanate reagent**: dissolve 1.5g of ferric chloride and 2.0g of potassium thiocyanate in 100ml water.
- **Formaldehyde:** Dilute the commercial 40% solution (1 part) with water(7 parts).
- **Magnesium nitrate reagent**: Dissolve 130g of magnesium nitrate and 200g of ammonium nitrate in water, add 15-20ml of concentrated ammonia solution and dilute to 1 litre.
- **Picric acid:** Dissolve the equivalent of 1g of anhydroius trinitrophenol in 100ml of hot water. Cool and filter if necessary.
- **Potassiumcynaide:** Dissolve 10g of KCN in sufficient water to make 20ml. Dilute to 100ml . shake with dithizone solution to remove lead.
- **Schiff's reagent:**

  Method 1: Dissolve 0.2g of pure prosaniline hydrochloride in 20ml of a cold, freshly prepared, saturated aqueous solution of sulphur dioxide; allow the solution to stand for a few hours until it becomes colourless or pale yellow. Dilute the solution to 200ml and keep it in a tightly stoppered bottle. The solution keeps well, and should not be exposed to light or air. Store in dark.

Method 2: Add 2g of sodium bisulphate to a solution of 0.2g of prosaniline hydrochloride and 2mlof concentrated hydrochloric acid in 200ml of water.

- **Silver ammonium nitrate**: Dissolve1g of silvernitrate in 20ml of water. Add ammonia dropwise, with constant stirring, until the precipitate, is almost but not entirely dissolved. Filter and store in dark container.
- **Tollen's reagent**: Add sodiumhydroxide solution to silver nitrate solution to form a precipitate, and then add dilute ammonia solution until the precipitate dissolves.

HCl=11.6

HNO_3=14.8

H_2SO_4=18.4

NOTE:

- **Stock solution**: a concentrated chemical solution [a concentrate] that is diluted before use.

Preparation of solutions

- **Concentration expressions.** The most common concentration expressions are as follows.
  - ✔ Weight per volume
  - ✔ Molarity
  - ✔ Percents[three kinds]
    - (i) Weight per volume (w/v%)
    - (ii) Volume per volume (v/v%)
    - (iii) Weight per weight (w/w%)

Using percentage by weight(w/v)

The formula for weight percent(w/v) is: $\frac{\text{Mass of solute(g)}}{\text{Volume of solution(ml)}}$X100 [normal]

Caution: do not simply measure the required volume of water and add the measured mass of solute because this will introduce error since adding the solid will change the final volume of the solution and throw off the final percentage. Thus add the measured mass of solute to a small volume of water and then make up to the mark. It applies for the solid solutes. E.g.

Using percentage by volume (v/v)

When the solute is a liquid, it is sometimes convinient to express the solution concentration as a volume percent.

The formula for volume percent (v/v) is: $\frac{\text{Volume of solute(ml)}}{\text{Volume of solution(ml)}}$X100

E.g. make 1000ml of a 5% by volume solution of ethyleneglycol in water.

First, express the percent of solute as a decimal : 5% = 0.05

Multiply this decimal by the total volume: 0.05x1000ml=50ml(ethyleneglycol needed)

Substract the volume of solute(ethlyne glycol) from the total solution volume.

1000ml(total solution volume)-50ml(ethylene glycol volume)=950ml(water needed)

Then dissolve 50ml ethlyneglycol in a little less than 950ml of water. Now bring final volume of solution up to 1000ml with the addition of more water. (this eliminates any error because the final volume of the solution may not equal the calculated sum of the individual components).

So; $\frac{\text{50ml ethylene glycol}}{\text{1000ml solution}}$ = 5%(v/v) ethylene glycol solution

Making solutions from stock reagents

When buying a stock reagent it is always advisable to check that it has a label that clearly indicates its specific gravity(density). Percentage purity, relative formula mass. From this information diluted solutions can be made . the analaR grade chemicals have labels containing all the information you may require for making solution of specific concentrations as shown in the table below.

Compound	Formula	Mmolecular weight/formula mass	Specific Gravity/wt per ml at 20℃	% by weight/% purity/minimum assay	Conc.Reagent Molarity
Acetic acid/ethanoic acid	CH_3COOH	60.0	1.05	99.5	17.4
Formic acid	HCOOH	46.0	1.20	90	23.4
Hydrochloric acid	HCl	36.5	1.18	36	11.6
Nitric acid	HNO_3	63.0	1.42	71	16.0
Phosphoric acid	H_3PO_4	98.0	1.70	85	14.7
Sulphuric acid	H_2SO_4	98.1	1.84	96	18.0
Ammoniumhydroxide	NH_4OH/ NH_3	17.0	0.90	28	14.8
Potassium	KOH	56.1	1.52	50	13.5

hydroxide					
Sodiumhydroxide	NaOH	40.0	1.53	50	19.1

Examples

1. A reagent bottle containing concentrated HCl has specific gravity of 1.18, molecular mass of 36.5 and is 36% pure(Assay)

(a) Calculate the molarity of the conc.acid.

(b) What volume of the acid will be required to make a 1M dilute acid?

Solution

a) Specific gravity is same as density i.e. 1.18g/cm^3

$\Rightarrow$ 1cm^3weighs 1.18g

$\Rightarrow$ The mass of HCl(Assay 36%)in the 1cm^3 acid = $1.18x\frac{36}{100}$ = 0.425g

$\Rightarrow$ 1cm^3 of acid solution contains $\frac{0.425}{36.5}$ = 0.012 moles

$\Rightarrow$ 1cm^3 of acid solution contains 0.012 moles

$\Rightarrow$ 1000cm^3 of acid solution contains 1000x0.012 moles

= 12M[Molarity of concentrated acid]

b) $M_1V_1 = M_2V_2$

$M_1 = 12, V_1 = ? M_2 = 1 \quad V_2 = 1000$

$$V_1 = \frac{1X1000}{12}$$

$$= 83.3cm^3$$

2. A reagent bottle containing concentrated sulphuric acid has specific gravity of 1.84, molecular mass of 98.08 and is 98% pure(Assay).

Solution

(a) Specific gravity is same as density i.e. 1.84g/cm^3

$\Rightarrow$ 1cm^{3}weighs 1.84g

$\Rightarrow$ The mass of H_2SO_4(Assay 98.08%)in the 1cm^3 acid = $1.84x\frac{98}{100}$ = 1.8034g

$\Rightarrow$ 1cm^3 of acid solution contains $\frac{1.8034}{98.08}$ = 0.0184 moles

$\Rightarrow$ 1cm^3 of acid solution contains 0.0184 moles

$\Rightarrow$ 1000cm^3 of acid solution contains 1000x0.0184 moles

= 18.4M[Molarity of concentrated acid]

c) $M_1V_1 = M_2V_2$

M_1 = 18.4, V_1 = ? M_2 = 1 V_2 = 1000

$$V_1 = \frac{1X1000}{18.4}$$

$$= 54.35cm^3$$

NB:

- **Assay** is the active ingredient in a solution.
- When making solutions of **hydrated compound** it's better always to use hydrated formula mass.
- **Concentrated ammonia solution** is extremely damaging to eyes. Even contact with dilute ammonia solution can lead to serious eye damage. Toxic if swallowed; harmful if inhaled and in contact with skin. Corrosive; causes burns thus use eye goggles when dealing with concentrated ammonia.
- **Hydrogenperoxide** is a strong oxidizer that can cause severe burns. Wear goggles, plastic gloves and a laboratory coat while working with this chemical.

Preparation of indicators

(a) Litmus

Dissolve 25g of litmus powder with three successive 100ml portions of boiling alcohol, continuing each extraction for about 1 hour. Filter, wash with alcohol and discard the alcohol filtrate. Macerate the residue with about 25ml of cold water for 4 hours, filter and discard the filtrate. Dissolve the residue with 25ml of boiling water for 1 hour. Cool and filter.

(b) Methylorange

Dissolve 1g of methyl orange in 1 litre of water. Filter if necessary.

(c) Methyl red

Dissolve 100mg of methyl red in 100ml of 95% ethanol. Filter if necessary.

(d) Phenolphthalein

Dissolve 1g of phenolphthalein in 100ml of 95% ethanol. Filter if necessary.

(e) Universal indicator

Add 0.18grams of methyl red and 0.36 grams of phenolphthalein to 550ml of 95%ethylalcohol [ethanol] stir to dissolve. In a separate container, add 0.43grams of bromothymol blue to 200ml of distilled water, stir to dissolve. Mix together the two solutions; dilute to 1 liter with distilled water. Add 1M sodium hydroxide solution drop wise until the solution's colour is dark green and stir.[Use: P^H indicator chart, P^H4 = red, P^H5 = orange; P^H6 = yellow, P^H7 = light green,P^H8 = green-blue,P^H9 = darkblue-green, P^H10 = purple]

(f) Limewater

Add 25g of calcium hydroxide to 1 liter of distilled water; shake; allow the solid to settle before use. Keep container tightly closed [for detecting carbon dioxide gas]

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