

Standards can be divided into two types:

- Primary standard
- Secondary standard

Primary standard

A primary standard is a chemical or reagent which has certain properties such as-

(a) It is extremely pure – A primary standard material should be extremely pure which means that it should be a chemical of high grade of purity, preferably 99.98%. In a pharmaceutical analysis laboratory we come across chemicals of different grade of purity. If we check the label we will notice a number with percentage termed as purity. So when a chemical has purity of 99.98% or more it is a suitable material to be used as primary standard.

(b) It is highly stable – It should be highly stable which means it usually does not react easily when kept in its pure form or it should have very low reactivity. This is important because if a reagent reacts easily with atmospheric oxygen or water or changes its property over time then it is unreliable and such an unstable and unreliable chemical can never be used as standard.

(c) It is anhydrous- It should be anhydrous which means that it does not contain any water molecule in its molecular structure. For example, in a pharmaceutical analysis laboratory we come across same chemical with different number of water molecules attached with it e.g. magnesium sulphate (MgSO_4), which is also called Epsom salt. The Epsom salt which is found in drug store is a chemical with formula $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Therefore if we want to prepare a primary standard of magnesium sulphate we should purchase an anhydrous MgSO_4 preferably an analytical reagent grade chemical and with purity greater than 99.98%.

(d) It is less hygroscopic in nature- The chemical preferably should be less hygroscopic i.e. on opening the container it should not absorb water molecules from atmosphere.

(e) It has very high molecular weight- It has very high molecular weight compared to its other similar forms. For example Epsom salt. Take 1 gram of MgSO_4 for making a primary standard and name it as salt A. Now take 1 gram of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ for common uses and name it as salt B.

Now if we compare the actual weight (Molecular weight) of magnesium sulphate to make a standard solution for both chemicals then it is found that-

CHEMICALS	MOLECULAR WEIGHT
MgSO ₄	108
MgSO ₄ .7H ₂ O	234

In first case, molecule of salt A the weight of actual MgSO₄ will be 108 atomic mass unit. But in second case, molecule of salt B the weight of actual MgSO₄ will be 108 out of its total weight of 234 atomic mass unit.

108 gram salt A (MgSO₄) will give 108 gram of MgSO₄

So, 1 gram MgSO₄ salt will give = $108/108 = 1$ gram of MgSO₄

But 234 gram salt B (MgSO₄.7H₂O) will give 108 gram of MgSO₄

So, 1 gram MgSO₄.7H₂O salt will give = $108/234 = 0.461$ gram of MgSO₄

Therefore if by mistake we make a standard out of salt B, actually we are taking 0.461 gram of MgSO₄ and calculating it as 1 gram. So with this faulty standard estimation of MgSO₄ in other unknown solution will give less result than the actual concentration. Hence it is important that primary standards must be anhydrous and of high molecular weight.

(f) It can be weighed easily – It can be weighed easily because it is so pure that its weight is in fact a true representative of number of moles present in its actual weight.

(g) It should be ready to use and available

(h) It should be preferably non toxic

(i) It should not be expensive

Uses – Primary standard is used to standardize a volumetric solution i.e. they are used for standardization of titration of solutions. It can be used for titration of acids as well as bases. In a pharmaceutical analysis laboratory, for acid titration the most common basic chemical standard is sodium carbonate (Na_2CO_3), (TRIS) Trisaminomethane $[(\text{CH}_2\text{OH})_3\text{CNH}_2]$ etc. For base titration, potassium hydrogen phthalate [(KHP): $\text{KHC}_8\text{H}_4\text{O}_4$] etc. For redox titration, potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) & Sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) are very often used as primary standard.

– The primary standard is used for calibration of secondary standard or for method validation using a specific method.

Secondary standard

A secondary standard is involved in preparation of reagents and kits or laboratories responsible for producing quality control material for other laboratories. They use primary standard as the primary calibrator or primary reference material. Secondary standard is used for the purpose of calibration of control material in laboratory for analysis of unknown concentration of a substance. So basically, secondary standard serves the purpose of external quality control for laboratories. So it is essential that the secondary standard must first be standardized against the primary standard.

For preparation of secondary standard solution, aqueous solution must be of high grade purity. It must be deionized, if water is used as aqueous solvent.

A secondary standard is a chemical or reagent which has certain properties such as

- (a) The purity of secondary standard is less than primary standard
- (b) Secondary standard is less stable and more reactive than primary standard
- (c) The secondary standard solution remains stable for a long time
- (d) Secondary standard is titrated against primary standard

Example – 1: Anhydrous sodium hydroxide (NaOH). It is extremely hygroscopic. As soon as the bottle is opened, NaOH absorbs moisture from atmosphere and it becomes moist. Lets do

it practically, take an analytical balance and place a Petridish and make its weight as zero (Tare). Now open the NaOH bottle and place little NaOH crystal on the petridish and note the weight. Now keep the glass windows of the analytical balance open for few minutes and notice the gradual increase in its weight. This is because the NaOH crystals absorb water molecule from air.

Example – 2: Potassium permanganate (KMnO_4) very often used as secondary standard. It is a good oxidizing agent, that's why reactive and hence less stable. Its own oxidized product manganese oxide (MnO_2) contaminates the content. Hence it is unsuitable for being a primary standard.

Secondary standard is used as a calibrator by smaller laboratories involved in actual analysis of unknown samples.

Calibration

Calibration means to check whether an instrument is working properly or not i.e. the instrument is giving correct measurement or not. Calibration and standardization are synonyms of each other but in case of solution we use the word standardization and in case of instruments we use the calibration . This is the process by which we compare the measurements by a standard or an instrument (primary) with another standard or an instrument (secondary). By doing so, we try to eliminate any variation or difference in measurement by the secondary standard or an instrument.