







QUALITY ASSESSMENT OF

FRAGRANCE MATERIALS









QUALITY OF FRAGRANCE MATERIALS

- •The totality of properties/characteristics
- fulfilling the specified requirements of a fragrance material.
- •Possessing right configuration suitable for the right application.
- A measure of excellence or state of being free from added adulterants.
- •Reducing variations to a minimum limit.

- •Fragrance materials mainly include essential oils and aromatic chemicals etc.
- •Essential oils are produced all over the world and quality of these oils vary widely.



•These are generally procured either directly from farmers or wholesale traders.



- •Oils of same plant species vary due to variation in origin, cultivation practices of plant materials, distillation techniques, climatic conditions & soil types.
- •Farmers are unaware of the proper cultivation practices and specific techniques for distillation, owing to which oils distilled, vary in configuration and properties to a great extent.



- •Sometimes bad trade practices also play an important role in deteriorating the quality of a particular essential oil or aromatic material by mixing cheap grade materials as adulterants for profit making.
- For a user ,it is very important to know the quality of the material procured ----
- i)to use the right material having specific configuration for its right application.
- ii)also to get value for the money spent
- •This applies to every material procured either directly from farmer /cultivator or from a wholesale dealer.



How to check Quality of a

fragrance material

This involves planning for conducting quality checks and will include:

- •Preparing a list of materials to be tested.
- •Arranging available standards for these listed materials.
- •Setting up a laboratory set up for required analysis keeping in view the parameters to be checked as per standards available.
- •Arrangement of qualified manpower to carry out these analysis work.



Label Details and Physical State

Label details need to be checked for every material received for testing to know the name /quantity of the material and other related details.

•Based on label details , reference standards can be arranged and then accordingly list of parameters can be made for testing purpose.

 In case of both synthesized aromatic materials and natural materials including essential oils, it is very important to check label details related to safety/



handling/date of mfg/shelf life/storing details to prevent any kind of risks involved or deterioration in quality if stored in a wrong manner.

- •Physical state of a fragrance material means whether a material to be tested is a liquid, solid or semi solid type.
- •Depending on the physical state of material ---
- i. number of parameters to be analyzed are listed accordingly.
- ii. sample preparation is carried out
 accordingly e.g incase of solid samples,
 proper dissolution in specific solvent etc
 is carried out ,generally liquid Samples







need no sample preparation except for some dark/viscous samples where proper dilutions are made and for semi solid materials like waxy, fatty and oleoresins etc--samples are prepared accordingly i.e either by taking out volatile Oil contents or making a particular derivative as per requirement depending on particular application.

iii)Any modifications required in set Test methods for any particular parameter are also decided on the basis of physical state. •Every sample to be analyzed for various parameters need to be checked for presence of dirt or haziness etc and depending on the status it may be pre-processed before subjecting to further analysis.

•These preprocessing techniques may include sedimentation, decantation, filtration, evaporation or using sodium sulfate etc to remove moisture till a material to be tested is ready in desired form for further analysis(clear and free from moisture especially if to be checked in GC/GCMS.)



Different Parameters to be tested

Optical Rotation:

•This is an important property to check whether a material

is having dextro rotation or leavo rotation.

•Generally most of the known essential oils and aromatic materials are having optical rotation and a range is set for

individual materials as per nature of components present and standards are available.

This gives an idea about the nature of a particular material. •Generally samples not falling in set range of specified limits are doubted for presence of adulterants/mixing of cheap natural materials, or natural variations in composition of a material.



•For example in case of Indian sandal wood oil which has generally specified optical range from-13 to -20, sometimes it is observed that the range exceeds this set limit and it may be doubted for mixing of African variety of sandalwood oil which is highly leavo rotatory in nature. Such doubts need to be confirmed by other tools like GC &Olfaction etc.

•However samples falling in set range are not always genuine and need to be checked for other parameters also

•Optical rotation is carried out in a polarimeter (Manual and Digital)using polarimeter tubes .

•For Viscous/dark and solid materials specific rotation is carried out by making a dilute solution of known concentration in alcohol.

POLARIMETERS





Specific Gravity & Refractive Index:

•These are also important parameters to check quality of a fragrance material .

•SG is the ratio of density of a particular material w.r.t to the density of water at particular temperature.

•R.I is the ratio of sine of angle of incidence to sine of angle of refraction at particular temperature.

•A range has been set for most of the essential oils for these two parameters in the reference standards and any deviation is an indication for presence of adulterants, cheap materials or natural variations.

- •Along with these parameters ,other parameters are also checked for complete assessment.
- •SG and RI are checked by using pycnometer/ or a density meter and refractometer (manual or Digital) respectively.
- •Materials for which a range is not already specified , a range can be set after testing a number of samples of a single variant on an individual basis.

REFRACTOMETERS



Solubility

- •Solubility is an important parameter for checking the quality of an essential oil.
- •Generally solubility is checked in dilute solutions of absolute alcohol
- •Solubility test gives an idea about the presence of nature of components present in a material.
- However solubility test in alcohol is an important parameter to check presence of any added heavy material in an essential oil which may sometimes get undetected by analyzing other parameters including GC.

•In case of minute additions of some heavy materials, an oil may pass the solubility test upto the generally specified amount of alcohol volume, but on extending the addition of alcohol portion, it no more remains clear

- and shows haziness or even settling
- down of these added heavier
- fractions as tiny droplets.
- •This extended addition of alcohol portions beyond specified amount is helpful in checking addition of few natural materials to same type



Of materials which generally go undetected in GC etc due to similar peak pattern especially if added in small amounts.

•Apart from olfactory assessment no other parameter sometimes detects such additions.

•Solubility in water is also important in case of some oils especially sandalwood etc.to check presence of water soluble materials present in an oil.

Chemical Properties of Essential oils

Acidic constituents :

Since Most of the essential oils contain only small amounts of Free acids, hence acid contents are generally expressed as acid Numbers instead of acid percentages.

Acid Number is the number of mg of KOH used to neutralize the free acids.

The acid number usually increases on aging, especially if stored improperly and also due to oxidation of aldehydes and hydrolysis of esters.

Hence it is very important to store oils in airtight and dark conditions .

Esters :

•The determination of the ester percentage is of great importance in the evaluation of many essential oils and involves following formula:

No.of cc of 0.5NaOH used for sapon. X Mol.wt of the ester

20xwt. Of sample taken

•Ester numbers are used for oils containing small amount of esters. A high ester number in such cases is indicative of adulterations.

Alcoholic constituents in essentila oils

- •The alcoholic constituents of an essential oil are determined by acetylation .
- •The oil is acetylized with acetic anhydride and the ester content of the resulting oil is determined: from this value the percentage of alcohol in the original oil may be calculated.
- •For the evaluation of essential oils, it is often desirable to know the percentage of total alcohol; i.e., the percentage of free alcohol plus the percentage of alcohol combined as ester present in the original unacetylized oil.

Some other chemical parameters determined in essential oils include:

•<u>Stearoptene contents</u>:Generally checked in rose oils to know the solid paraffinic hydocarbons present naturally in rose oil.

•<u>Congealing Point</u>:Checked in Rose oil to determine the temperature at which rose oil congeals. Higher Congealing point is an indication of good quality rose oil.

•<u>Phenol Contents</u>: This is generally checked for clove oil to know the Eugenol content.

Gas Chromatograph Analysis:

•Gas Chromatogram is very important instrument to check composition of an essential oil/aromatic materials.

•This technique is helpful in knowing the composition of a complex material and purity of active components required as per application.

•Almost all the fragrance materials are checked by GC analysis by injecting direct samples or in diluted form as per the nature of material.Sample preparation is done accordingly. •This technique helps to detect presence of adulterants which if undetected may have a great impact on the quality/cost of fragrance material.

•Detector used for GC analysis is FID i.e Flame Ionization Detector. •For better confirmation regarding presence of adulterants and separation of components etc, it is always advisable to run a particular material on both Non Polar and Polar column.

•However as a rule every unknown material shall be first run on a Non Polar column to check for the presence of any high volatile component which can be eluted in this column as temperature limit is higher in case of Non Polar and only then after checking the configuration, the same sample may be run

Gas Chromatograph , Oven & Capillary column.







on a polar column to check separation of peaks in a better manner.

•Any gas chromatograph taken out needs to be checked against a reference standard i.e a reference standard sample needs to be run to mark key marker components.

•There may be more than one number of standard graph set for a particular oil depending on the application

•For example an oil used for flavoring purpose having a typical composition may not be that beneficial

for aromatherapy purpose due to variation in active

components responsible for aromatherapy benefits. Hence for one particular oil, two different reference standards may be set and later incoming lots can be matched against the concerned standard. For example cardamom oil to be used for flavoring purpose has to be a complete oil containing all minor components, whereas cardamom oil rich in mainly cineole content may be used for some other application.

These reference standard materials may be arranged from some authentic sources or may be distilled in house from plant materials.





<u>TLC</u>

TLC i.e Thin Layer Chromatography is again a very important technique to check the quality of an oil.

An oil which is passing all set parameters like OR,SG,RI,Solubility or even GC standards, may contain some added material which could not be detected in these parameters ,but TLC is a technique which may prove beneficial to detect such undetected materials as this process involves separation of various components in a sample due to capillary action /adsorption.

This technique involves use of TLC

plates available readily in the market

or made in house.





Olfactory Assessment •Olfactory assessment of an oil is a very important Parameter to be checked simultaneouslyby experts having sharp sense of smelling andregistering odor notes.

•This exercise is generally done parallel to other parameters being carried out by team .



 It is been observed that sometimes an oil/aromatic material passing all the suggested parameters does not pass olfactory assessment test and is rejected.



- •All above parameters are related to checking presence/absence,addition or reduction of various components in a fragrance material.
- •But for the purpose of identification of particular components present in a fragrance material irrespective of being a single peak material or multi peak material ,it is important to run the specific sample on **GC connected to MS detector.**
- •This instrument is more sophisticated than GC-FID.

GCMS



CONCLUSION

Quality check is an important and integral part of each and every raw material used in perfumery and fragrance materials---

- **1.To provide a consistent quality product to consumer.**
- 2.To get value for money spent.
- 3.To use a right kind of material for right application imparting a better odor profile.

4.To avoid any legal or regulatory hassles by checking raw materials for presence of any undesired component etc which may go into final product.

Quality checking in case of fragrance materials is a continuous process and needs regular upgrading to find out increasing variations and adulterations .

THANK YOU