SPECIFICATIONS IN THE FLAVOUR & FRAGRANCE INDUSTRY

Sitaram Dixit
Corporate Manager – Fragrances
S. H. Kelkar & Company P. Ltd.

We are aware that a product that confirms to the set specification generally means a job well done. Specifications protect the interests of both the supplier and the client and provide a common ground for resolving problems that can occur during manufacture. A quality specification although may not guarantee complete elimination of quality issues it does provide a framework for decision-making and help resolve quality problems in case of disputes.

In this paper we discuss quality assurance and quality control as is relevant to the flavours and fragrance industry so that we understand the various parameters that ensure creation of a flavour or fragrance suitable for the purpose it is procured for.

QUALITY ASSURANCE

The term quality assurance can be broadly defined as the total activity for delivering constant quality. It covers all monitoring and testing methods and procedures that are undertaken by a manufacturer to ensure that the product made meets requirements as per the set specifications range with regard to product composition and its performance during use. Quality assurance includes all activities on the development of a new fragrance right from customer enquiry for a fragrance to delivering a consistent performing final fragrance, in addition to collecting customer orders and prompt delivery every time. Quality assurance also encompasses activities like product safety, regulatory affairs, formulation control and audit or checks and suppliers to ensure consistent delivery of raw materials in time and in required quality and packs. Producing a quality manual and a procedure to conduct quality audit is also a key responsibility of quality assurance. Generally these systems are in line with quality standards namely ISO 9000, BS 5750, etc., that are used by the industry to maintain quality.

Quality control

Quality control means controlling the quality of the raw material that are used to make the finished product and includes controlling the processes used to manufacturer, the storage requirements and its subsequent transport to the customer for use. In light of the above quality control is just one activity within the ambit of quality assurance.

Quality control and assurance are introduced by first setting up the specifications for individual materials all comprising of a set of standards within reasonable values against which the test sample is compared with for acceptance. It is more appropriate if a reasonable range of specified values were permitted for quality acceptance.
Typically some standards can be as follows:

1. A minimum value,
2. A maximum value,
3. A minimum and maximum limiting value, namely a value range within which the product value should fall so as to qualify for acceptance with respect to that parameter, e.g., specific gravity and refractive index.
4. The composition of the raw material. This is very important in case of essential oils and finished fragrance that are a mixture of various aroma chemicals, e.g., GLC graphs, and
5. Last but not the least a suitable odour profile as per the control sample or reference standard.

In most cases failure to meet specifications leads to rejection of the material, however in case of aromatic material it may not necessarily lead to automatic rejection. In case of natural aromatic products than synthetic ones for e.g., if an undesirable note is observed in an essential oil it can be removed by airing the oil or if the oil is darker in colour then desired a suitable decolourising procedure can be employed to bring it in line with the requirements. In short it is quite possible that use of a simple inexpensive treatment on the product is sufficient to bring it within prescribed limit. It is also quite possible that in case of aromatic ingredient namely essential oil resinoids a consignment with a higher specification might be accepted on lower specification after renegotiation of the price with the supplier for use in the manufacture of lower quality fragrances. This type of situation generally occur in times of shortage as natural products are still depends on the vagaries of nature.

However if the material supplies is not in conformance with one or more parameters with no chance of rectification it is rejected. Without doubt it is vital that he quality tests are carried out to decide product acceptance or rejection is carried out correctly as per standard analytical methods that are not only accurate but also reproducible.

FRAGRANCE QUALITY

Finished fragrance quality is maintained by controlling the quality of the raw material used in compounding the fragrance. It is very important that correct systematic methods are followed to test the raw material used as any error in use of the material not meeting the specified standard can inflict irreparable damage to the fragrance compounded. It is also essential that tests to rule out toxicity, irritation and sensitivity to skin, shelf life and in usage tests be conducted to ensure mutual compatibility of the fragrance and the end product application for satisfactory fragrance performance.

Quality of natural ingredient:
Quality variations in natural aromatic materials are very common. Genetic makeup or genotype of the plant, soil and climatic influence of temperature, light conditions, rainfall, water supply, altitude above sea level, are all found to have a profound influence and effect the quality and quantity of essential oils produced. The success and failure of crops also depends on crop cultivation conditions, the skills and expertise used to nurse the crop right from germination to harvesting and have a large influence on the quality and quantity of yields. Processing of the aromatic plant pre and post harvest conditions of storage are other important influencing factors. In addition to all the above natural factors influencing quality, the bane of adulteration of valuable materials with poorer inexpensive substances sometimes very cleverly to avoid detection should also not be ignored.

Quality of synthetic ingredient:
Aroma chemicals produced are normally in the purest form as they are manufactures under controlled plant conditions and can be produces with minimum variation with respect to odour profiles and quality specifications. Although quality variations do exist in aroma chemicals, it can be easily analysed for any impurities and rectified by relevant purification processes. All raw material used for compounding fragrances are tested to confirm the specifications.

The tests used can be grouped with three distinct categories.

1. Chemical analysis
2. Organoleptic analysis and
3. Instrumental methods of analysis.

CHEMICAL ANALYSIS

Chemical analysis of perfumery raw materials are quite lengthy, time consuming, with possibility of human error and so expensive. Some typical tests include acid value, ester value, ester content, aldehydes content, ketone content, total alcohols, etc.

ORGANOLEPTIC ANALYSIS

Organoleptic analysis makes use of our sense organs namely eyes, nose tongue to analyse and come to a conclusion. Visual inspection and comparison of the ingredient appearance with that of the standard sample in terms of colour, viscosity and presence of visible impurities are noted. Normally a smelling strip made of absorbent paper is dipped to a depth of about 1 cm. in the sample under test. A similar strip is dipped in the standard reference sample at the same time. The strips are smelled for 4 or 5 minutes to compare ***"top notes"***. The strips are smelled again after 15 minutes to compare ***"middle notes"*** and then after about 24 hours to compare ***"back or base notes"***. At each stage the aired sample is compared for odour with a fresh dip.

Smelling strips are held about 2 cm. away from the nose taking care that it does not touch the nose. One should not take a nose full of odour. The strips are smelt alternatively, for the least possible time, but sufficiently enough to perceive the odour clearly. The time interval between two subsequent sniffs is adjusted to give a little rest to our nose and clear it of residual odour of the earlier sniff so that the differences between the two samples are easy to observe and record.

Top Notes
The odour impression of the material on immediate opening of the bottle or on smelling a freshly dipped smelling strip is termed as the top note in perfumery language. The first impression derived and that lasts for a short duration of time is the top notes. Freshly distilled or dull drum note, unwanted sharp, crude, acidic or other undesirable side – odour are all observed in the top note.

When comparing odour materials it is important that the standard and the test sample should be at comparable room temperature. The samples should not be smelled on immediately after removing from the refrigerator or on immediate recipient from transportation in hot sun. The samples should be allowed to come to room temperature before smelling otherwise they are most likely to be assessed wrongly.

Middle notes
The top note of an ingredient may or may not be the main odour of the aroma ingredient. The typical odour of the ingredient is noticed when the top note ends. The main or principal odour of an aroma ingredient is termed as the middle note. The odour nature, its strength and depth, the roundness of fullness are all studied during the evaluation of the middle note to decide on the quality of the material. The quality of middle note gives a body to the aroma ingredient and so the middle note is also called as the body note.

Normally the samples on smelling strips are smelled alternatively, with the reference sample followed by the test sample and then in the reverse order and evaluated. The two strips are also smelled blindly without knowing which is the reference standard and which one is the test sample to ascertain odour strength. It is advisable to repeat the tests a couple of times to confirm the results and avoid mistakes.

Base note
The base note starts when the middle note gets exhausted. When the characteristic odour of the material is not clearly noticeable the middle note is said to be over. The lingering or retentive nature of the material is studied during the evaluation of the base note. Undesirable odours on spoilage due to long and improper storage, polymerisation or high temperature heating during distillation can be easily detected in a dry out note. Similarly in case of isolates the unwanted odour of a high boiling fraction if any can be readily noticed in the base note.

Odour evaluations
Smelling test between test sample and standard if conducted conscientiously can reveal a lot. However the evaluator should have keen interest, have a good nose backed by sufficient experience to relate the odour differences to its likely causes to draw suitable conclusions is necessary. It is also quite likely that sometimes aroma chemicals whether natural or synthetic can present strange and unfamiliar notes that we may have to reject even though it may fulfil all other specifications and required parameters. These unfamiliar strange off odours could be due to the presence of extremely powerful chemical impurities in very small amounts viz., in ppm and ppb levels that are easily recognised by our nose but unlikely to be identified by sensitive analytical instruments available on date. Thus the smelling test is a very important one that is necessarily required to be done in case of all incoming raw materials in the F&F industry.

Flavour evaluations
The olfactory evaluation of flavour ingredients is also similar. In addition to smell, taste or flavour of the material is also assessed either directly or its solution in 10% sugar solution or alcohol.

INSTRUMENTAL METHODS OF ANALYSIS

Instrumental analysis is carried out to check both physical and phyisco – chemical properties of aroma ingredients and its finished produce. Typical instrumental methods used in the F&F industry are as follows.

1. ***Refractive Index:*** Refractive index is the measurement of refraction of light rays as these passes through the material. Refractometric measurements are used for qualitative, quantitative analysis as well as for structural investigation. However its use in the F&F industry is as a qualitative test of purity of aroma ingredients and finished fragrance oil.

2. ***Optical Rotation****:* Optical rotation is the power of the substance to rotate the plane of polarised light. Certain organic liquids when placed in the path of plane polarised light; has the ability to rotate the plane of polarisation. The property by virtue of which this rotation occurs is called as optical activity and substances possessing this property are said to be optically active. Substances that rotate the plane of polarised light toward the right (clockwise) are called dextro rotatory (+) while those that rotate towards the left (anticlockwise) are called leaveo rotatory (-). A mixture of these two varieties in equal proportion will be optically inactive. It is called the racemic form. Optical rotation of a pure optically active chemical substance is always the same when measured under standardised conditions. Thus optical rotation measurements can be used as a check on the purity of the substance. If an optically active impurity or an impurity of opposite rotatory power is present in the test material it will alter the value of the standard optical rotation of the substance and the presence of the impurity can be easily identified. Similarly if optical activity is found in substances traditionally known to be optically inactive, then the presence of impurity is known.

3. ***Specific gravity:*** Specific gravity of a substance is its density in relation to that of water. Specific gravity is therefore also known as relative density. It is defined as the ration of the mass of a given volume of the substance to the mass of an equal volume of water at a specified temperature. Specific gravity measurements are time-consuming practical techniques if one needs to obtain reproducible results. This test is very important as it can detect adulteration that even our human nose is unable to detect. For example Brazilian Boise de rose oil has a specific gravity of 0.8680 to 0.8910. Pure synthetic linalool has a specific gravity of 0.8580 to 0.8620. We know that the main constituent of Boise de rose oil is linalool. If in case genuine oil is adulterated with pure synthetic linalool, it might be very difficult to identify by smell, however specific gravity is very likely to drop to less that 0.8680 and so any adulteration easily identified.

4. ***Melting and congealing point:*** Substances that are in a solid state at normal room temperature are tested for melting point and substances that are liquid at room temperature are tested for congealing point. The melting point temperature at which a crystalline solid melts is constant for all pure chemically identical substances. Melting point and congealing points are generally precise and do not spread over wide range of values. Crystalline substances in fact have a very sharp melting point. Molecules are uniformly arranged in a crystal lattice. Impurities present in a substance cause irregularities in the crystal lattice and thereby weaken the molecular structure collapsing the crystal into a liquid state at a lower temperature than normal or the actual melting point of the purer stuff.

5. ***Pour point:*** The temperature at which a viscous liquid becomes pour able is called the pour point. If diluents are present in the supplied viscous liquid then the pour point is reduced. Pour points are generally not very accurate as they vary with every consignment noticeable in resinoids.

6. ***Gas liquid chromatography:*** Chromatography is an analytical separation method in which substances are separated on the basis of their differential migration rates in a system consisting of a stationary phase and a mobile phase. The difference in migration rates is dependant on the differential adsorption, partition coefficients, ion – exchange, molecular sieving effect, etc. Physical forces like electro static interaction between dipoles, Vander Waals forces in addition to the inherent chemical forces all affect the separation of the substance in this technique. GLC analysis is useful in determining both qualitative and quantitative purity of the liquid substance. It is very useful to determine the percentage composition of a mixture of liquids, essential oils, flavours and fragrances. A GLC instrument can carry out the analysis with very small quantities of the sample within an hour's time. GLC is nowadays used routinely in F& F industry for checking the composition of aromatic materials and fragrance compounds against chromatograph charts or graphs of standard reference samples prepared under identical conditions.

7. ***Infra Red spectroscopy:*** IR spectroscopy is based on a simple fact that chemical substances show a marked selective absorption in the infrared region. After absorption of IR radiations the molecules of the chemical substance vibrate at many rates of vibration giving rise to close

packed absorption band called IR absorption spectrum that corresponds to characteristic functional groups and bonds present in the chemical substance. IR spectra of a chemical substance thus are a fingerprint for its identification. IR spectroscopy is one of the most powerful analytical techniques, which offer the possibility of chemical identification. This technique is often coupled with other measurements for quantitative analysis. In the F&F industry IR spectroscopy's main use in QC is in combination to GLC for checking the quality of the test material of aromatic ingredients with respect to the reference standard. Gas chromatography gives qualitative data on the relative proportion of the various constituents of a mixture but does not identify the various components. An IR spectrum on the other hand supplies details on the chemical compositions of the material to which the spectrometer is responsive. It however does not separate or detect the individual components of a mixture.

8. ***Mass spectroscopy:*** Mass spectroscopy is another technique used in the F&F industry for research and for elucidating the molecular structure of a substance. It can be a newly discovered aroma ingredients or a recent isolated constituent of essential oil. However this technique is not used in routine quality assurance or control purposes.

9. ***Dermal tolerance test:*** Dermal tolerance test on perfume compounds is another important test that is also carried out. It is necessary that any fragrance compound should not affect skin when it comes in contact through its use in a consumer product. IFRA monographs give valuable inferences in safety of aromatic material for use in compound fragrance. However it is important to note that even if all ingredients used in compounding a fragrance are non – irritant or non – sensitising it does not guarantee that the compounded fragrance is also non irritant and non – sensitising in nature.

All incoming raw materials in a typical quality control system are checked for odour, refractive index, and specific gravity against reference standards. In some cases GLC analysis are also carried out. The criteria for selection and replacement of stocks however remain the price in relation to previous purchases, quality comparable to existing stocks available and the absence of adulterants. The quality control in compounding is also very important. Incorporation of each ingredient is doubly confirmed on the formula sheet so that any uncertainty of addition or omission is completely ruled out. In addition to normal odour system of checks and balances, chromatograph also plays an important role to confirm corrections in compounding. Finished fragrances are assured of quality by evaluation of the odour profile by trained panellists in addition to analytical tests of appearance, refractive index and specific gravity with respect to standard reference samples. GLC chromatograms again are a check to confirm the quality of the compounded fragrance oil.

Today the management of quality is a source of competitive advantage. Quality is no longer the responsibility of QC or QA functions alone but each and every member of the organisation. Organisations implement TQM programs, i.e., Total Quality Management programs that are not only about sampling, process testing, inspection and measurements, but involve a complete coordination of all staff activities carried out in the organisation as it is now recognised that **QUALITY AND CUSTOMER SATISFACTION ARE INSEPARABLES.**