Solutions: Solutions or Problems?

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It would seem that perfumers love to use solutions; dilutions of materials in low-odor or odorless solvents. They treat them as different materials with unique characteristics; sometimes, it would seem, almost mystical characteristics.

The compounding department, by contrast, hates solutions and quite justifiably so. When making a 1,000 kg batch of a fragrance, using 50 kg of a 10% solution would seem redundant. Surely it would be better to extend the original formula by adding 5 kg of the 100% material and 45 kg of the solvent. This saves on weighing time, a separate worksheet, batch records, quality control of the solution, tying up stock (in different solvent combinations), logistics and storage space.

Are solutions really necessary? Are they a perfumer's white elephant, given to compounders to maintain the authority of the perfumer and to preserve the mysticism of perfumery? Are there guidelines that can be established to rout out solutions that are unnecessary without risking quality assurance?

It attempting to address these questions it would be useful to examine why a perfumer might use solutions. Reasons may include:

- controlling the addition of high-impact raw materials
- addition of very small quantities of materials
- compound cost control of expensive materials
- handling of very viscous materials (e.g. benzoin resinoid in benzyl benzoate)
- stabilizing relatively unstable materials (e.g. lauric aldehyde in DPG)
- quenching hazardous materials (e.g. phenyl acetaldehyde in phenyl ethyl alcohol)
- the maturation effect produced (e.g. animal extract as tinctures in alcohol)

In the case of cost control, there seems to be no justification for using solutions unless one of the other factors comes into play. However, the handling of viscous materials, the stabilizing of unstable materials, quenching hazardous materials and those made for the maturation effect, I suggest, are not solutions per se. These are specific, special-purpose sub-compounds for which extending them into their component, individual materials might lead to other problems.

In considering the remaining cases of controlling the addition of high-impact materials and adding very small quantities of materials, the following two factors become relevant to the discussion. We should first consider the comparative accuracy of weighing in the perfumer's laboratory to that in the compounding department.

Typically, the presumption is that weighing materials in large amounts will be more accurate than weighing a small amount. One gram can surely be weighed more accurately than 0.1 g, and 1 tonne more accurately than 1 kg. Is this really the case? I thought so until one day recently, when this assumption was greatly undermined. My attention was called to a discrepancy in the balances in the compounding area and those in the perfumer's lab. The balances were all newly installed, calibrated and government sealed, because it was a new facility. The lab machines were from a famous German manufacturer, and those in the production area from a reliable Japanese maker. Investigation found that there was, even between the same make, a discrepancy of up to +/- 0.75 % between the balances. The large discrepancy was not explainable by experimental error and was reproducible. If one weighed accurately (to 2 decimal places in grams) 1 kg on the lab scale in 100 g lots and then checked these on the 3kg sample-capacity machine, there was up to a 7.5 g discrepancy. If 100 kg were weighed in 2 kg lots on the sample machine and transferred to the production scales, up to a 750 g discrepancy was produced. Errors followed a curve and generally, the area near the mid range point of the balances proved to be more congruent with scales from the same manufacturer. This was an eye-opener, because until this occurred, I had regarded weighing equipment as accurate and absolute.

The practical electronic balances used in perfumery laboratories or compounding areas of perfumery companies typically have a of readability of about 0.01% of their capacity. For example, a 100-kg capacity balance typically has a 10 g readability, a 10 kg capacity balance has a 1 g readability and a 100-200 g capacity balance has 0.01g divisions.

This leads to the conclusion that weighing 5 kg on the compounding floor cannot be presumed to be more accu-

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rate than weighing 5 g at the perfumer's bench because both have the same relative readability. More accurate balances are available, but these are usually only suitable in low-traffic areas because of sensitivity to draughts and vibration, and are generally reserved for analytical departments rather than compounding areas.

The second point of consideration is the degree of accuracy needed. How accurate does our weighing of individual components in a compound really need to be? I polled several friends and colleagues in the business and asked if they were aware of the tolerance their company allowed when weighing individual materials during the compounding of fragrances. All but one stated that they did not know or were not aware of this having been quantified and I had no reason to believe they were being evasive on this issue. The one that stated the policy of his company told me, "There is no allowable tolerance…we weigh accurately every time." These comments launched a series of experiments to determine how big an error is really necessary to produce significant differences in the odor of samples.

The experiments that followed showed that if accuracy to the target weight can be maintained in the range of +/- 10%, then the resultant difference in the compound's odor, as evaluated by a panel of testers, was found to be slight. In the case of more than one such error, particularly if the materials had substantially different odor characters from the finished compound, a detectable odor difference could occur.

When the accuracy approached the range of +/- 1% of the target weight, the resultant difference in the compound's odor was not detectable by the panel even in multiple under- or over-doses within this range. This is easily corroborated by accurately weighing a compound and deliberately making a second sample with the strongest or even most abundant material in the formula at +1% of target weight and a third sample at –1% of target weight. These results were irrespective of the material's abundance, strength, volatility or dilution. If the formula calls for 0.1% then the low figure is 0.099% and the high 0.101%. If the formula calls for 50% then the low is 49.5% and the high 50.5%.

If we accept that 1% is an acceptable error (if we require even greater accuracy the argument becomes even more compelling), we should only weigh a material with weighing equipment that is accurate to 1% of the target weight. Given that the typical readability found on balances typically used in perfumery houses is 0.01% of capacity, using the 1% rule means that the minimum quantity to weigh be limited to 100 x 0.01% or 1% of the capacity of the balance. However, it is not as easy as using a smaller balance to weigh smaller-quantity items than this because the variance between different capacity balances may well introduce a second error that can be greater than the acceptable 1% target range in any case. This accuracy limitation therefore would restrict the perfumer to weighing a minimum of 1 g on his or her two-decimal-place balance, and the compounder to 1 kg on their 100 kg capacity balance. Older-style lever balances with analogue readouts can be more accommodating, because a fraction of a division can be seen, even if not accurately noted. However, without RS232 outputs, they have lost popularity in GMP procedures.

General Principles

In consideration of these factors, I recommend the following guidelines to students when making samples or planning for production batches of their formulations:

- There would seem to be a case for using solutions to ensure accuracy when weighing amounts less than 1% of a formulation's total. Of course using a range of balances for different weights does help to overcome this. If balances are cross-calibrated and the error characteristics are determined, accuracy can be maintained. Using the mid range of a balance rather than the limits of its capacity generally gives better results. Specifying which balances are to be used for specific weight ranges also helps. The rule of two decimal places below (1% of target weight) is a good guide.
- Equipment chosen for weighing should be able to weigh to a readability of a decimal point that represents 1% of the target weight of that material. To weigh 0.1 g, our balance ideally should have readability to 0.001 g. To weigh 37.23 g, the balance ideally should have readability to at least 0.1 g. The small difference resulting from not being able to weigh the last 0.03 g in these guidelines my be regarded as a non-significant quantity. Weighing 37.1 g on a 0.1 readability machine and then 0.03 g on a 0.001 g readability machine is not a guarantee that total accuracy will improve because the inherent inaccuracy in the larger machine may have already added an error greater than this in any case.
- In view of the typical balance range found at the perfumer's organ (200 g capacity with a 0.01 g readability), the optimum minimum weighed in a 100 g sample is 1 g. Therefore, this restricts formulas to use not less than 1% of any single material. However, for practicality with initial experimental samples, I suggest that this could be extended to 0.1% (0.1 g in 100 g), less than the ideal and certainly not accurate enough for trail or sampling batches. If less than 1.0/0.1% is used, then that material is best used as a solution.
- Materials with an odor impact of 100 or less (where linalool synthetic is the control with an odor impact of 100) do not generally require dilution, unless they are less than 0.1% of the formula. Stocking dilutions can take the odor impact down to about 100, the same strength as linalool (e.g. Cis-3-hexenol has an impact of 700 [seven times the impact of linalool]). A 10% solution is generally acceptable for most compounds. For convention and simplicity's sake these are best kept as multiples of 10.

Table 1. Worksheet example

Raw material	Worksheet amount	Actual amount weighed
Aldehyde C11 Undecylenic	100 g	*101.2 g*
DPG	900 g	*918.8 g*
	Original total 1000 g	*New total 1,012.0 g*

- Solutions made only as 10% give better uniformity of results, subject to their solubility characteristics (some materials' solubility may be too low to allow 10%, such as maltol, soluble at a maximum of around 5% in DPG). While making 100 g of a 1% solution on a 200 g/0.01 readability balance, the 1.0 g indicated on the balance readout could actually be in the range of 0.995 and 1.015, because the third decimal place is unknown. Using the 10% rule, an extra decimal place of accuracy is added. A 1% solution is made from 10% of a 10% solution. A 0.1% solution is made from 10% of a 1% solution.
- Formulas or compounding worksheets for solutions formatted in the, "weigh accurately, approximately…" manner can give greater accuracy than fixed-quantity formulas. This means that when making a solution for use in production, the compounder should record the exact weight of the target material they have added to be diluted on the formula/worksheet. For example, while making 100 g of a 10% solution, if the compounder finds they have actually weighed out 10.15 g of the concentrated material, they adjust the new total from 100 to 101.50 g, indicating that the amount of solvent is to be increased from 90 g to 91.35 g. A further example is provided in Table 1.
- Solutions should pass QC the same as any other compound. Strength is best determined in QC by GC or smelling the solution at 1% in ethanol and dropping the same number of drops onto smelling strips.
- Laboratory use of solutions filled from production-area stock bottles that have passed QC ensures reproducibility.
- Solutions in ethanol or other materials that are not inert and undergo a maturation process or are dissolved in an odorous-materials process are best treated as distinctly separate sub-compounds and standardized in age and storage conditions before inclusion in their parent compounds.
- A formula follows these guidelines irrespective of batch size. If solution parameters fall out of these guidelines then they are best formulated as sub-compounds and labeled accordingly.

Conclusion

Weighing accurately with no tolerance is an unobtainable goal. The equipment, at its best, is perhaps only accurate to around 1% of target weight without incorporating the human element, which may increase this error. Because compounds frequently have materials at concentrations of less than 1% of the total formula, the use of solutions appears to be justified. If we can maintain an accuracy of 1% of the target weight of both the solute and solvent there should be no detrimental effect on the odor of the finished compound.

These guidelines can help students to plan formulations using solutions to achieve consistent perfume quality without the unnecessary inclusion of expendable dilutions (e.g. 5% of phenyl ethyl alcohol, a low-impact material 10% in DPG, a relatively odorless and inert solvent) and ease the compounder's burden slightly.

References

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