Regulatory Principles to Enhance Coherence and to Facilitate Trade in Wine

1. Executive Summary

This paper presents several principles surrounding the establishment of regulatory limits for wine and verifying compliance with those limits by analytical methods. In particular, the following principles are presented:

- Avoid establish limits that stimulate costly and unnecessary analyses.
- Harmonize limits where there is no scientific justification for national or regional differences.
- Give due regard to intergovernmental agreements and work done by other authorities when establishing new regulatory limits.
- Adopt a common system of scientific units for expressing regulatory limits.
- Express regulatory limits on a “per unit volume of wine” basis rather than “per unit volume of alcohol” in the wine.
- Adopt a common way of expressing results where this is done in relation to a single wine constituent (e.g. for Total Acidity expressed in terms of one specific acid).
- Consider the establishment of analytical “de minimis” values for substances or classes of substances in wine - values below which they will be deemed to all intents and purposes not to be present in the wine.
- Allow suitable transition arrangements when limits are tightened, provided public health considerations so permit, and exempt wine from the requirement to be labelled with an expiration date.
- Analyses of wine for compliance purposes should be undertaken by suitably accredited laboratories (or overseen by certified analysts) that perform acceptably for the specific test methods used.
- Analytical methods used for wine compliance purposes should be validated and/or have a demonstrably appropriate level of performance for wine.
- For wine authenticity analyses, the database of authentic samples with which the test samples will be compared must be sufficiently comprehensive to avoid the mis-categorization of legitimate samples as fraudulent.
- Laboratories testing for compliance purposes should supply measurement uncertainty information with their analytical results and the competent authorities should take this into account in interpreting analytical data.

It is proposed that these principles might form the basis for an understanding among the appropriate countries, that would have a significantly trade-facilitating effect.

2. Introduction

The facilitation of trade in wine and avoidance of obstacles to trade according to the rights and obligations of international accords have always been central tenets by which the work of key international bodies dealing with wine has been conducted:

It is the purpose of this paper to propose an area of activity by such bodies to facilitate trade in wine and to avoid the creation of unnecessary obstacles to that trade. This is the area of the establishment of regulatory limits for wine, and the application of laboratory testing to verify compliance with those limits.

It is clear that regulatory limits are necessary in certain cases to be able to maintain product safety and composition standards, together with a level playing field for wines to compete in the global market. Limits and methods are good in their proper place and achieve useful purposes in international trade. Nevertheless, there have been times historically when a less positive and constructive role has been assumed by limits and by laboratory testing. In addition, many national regulatory limits were established in days before the wine market became the global entity that it is today. For these reasons, there are steps that can and should be taken to facilitate trade in wine in the 21st century that will have no impact whatsoever on the protections afforded to consumers on the one hand and honest traders on the other.

This paper outlines several principles that, if implemented by the appropriate governments around the world, would have an enormous positive impact on trade facilitation. They would work to reduce confusion and unnecessary costs, as well as to enhance the confidence that all sectors could enjoy in the robustness of laboratory testing results.

For each principle, explanatory background information is supplied. The principles are presented first for the establishment of regulatory limits and then for the laboratory testing that is done to determine compliance with those limits.

While the thrust of this current initiative and the principles described below focus on “wine,” the principles could equally be extended to cover “wine based products.” This extension would take into account in a similar manner the regulatory disparity that exists among different markets in the classification of this category of products.

3. Establishment of regulatory limits

3.1. Establish only necessary regulatory limits

The establishment of limits for constituents and potential contaminants in wine is certainly necessary in many cases to provide governments with a degree of comfort surrounding the quality and safety of wines in international trade. However, it is also true that once a regulatory limit has been established, it is often included either in certificates of analysis requirements of importing countries, or in the private standard documentation that may be necessary to sell product in a given market.

Every analytical test performed on a wine has cost implications for the producer and the multiplication of unnecessary tests can serve to increase the cost of doing business in a given market, eroding margins to a point where it is no longer feasible to trade. Examples of limits that might generally be considered unnecessary include:

- Establishment of pesticide MRLs for wine when they are already established and effectively controlled at the level of the raw material, grapes.
- Setting specifications for pathogenic microorganisms for wine, which does not support the growth of pathogenic microorganisms. We know from the text books that the results of a determination for salmonella in wine will always be that it is not present. To perform a test to demonstrate what is already known is costly and wasteful.
- Setting a limit at a level much higher than quantities ever seen in wines around the world. The establishment of the limit means that costly confirmatory analyses will be performed, but no products will actually fail to comply.
- The establishment of limits for wine that do not give sufficient regard to the low food safety risk status of wine, and may be more demanding than similar, pre-existing limits for foods with a similar risk profile to that of wine

Principle: Governments are respectfully recommended to avoid setting limits that stimulate costly and unnecessary analyses.

3.2. Harmonize regulatory limits where possible

The limits established for many components in wine relate to elements of composition and have no public health significance. In a global market, trading in wine can become very difficult when each market has different limits for the same component. Frequently, there is no scientific justification for the differences in limits observed between markets. As an example, see Appendix I, which (only by way of illustration) presents the limits around the world for methanol in wine. These levels are comparable to what one would find in freshly squeezed fruit juices, and are set for technological rather than toxicological reasons. It is instructive to see how many numerically different limits there are for just this one component.

Wine trade would be greatly facilitated if such limits, where they are necessary, could all be set at the same level in the different markets around the world.

Principle: Governments are respectfully requested to harmonize limits where there is no scientific justification for national or regional differences

3.3. Take due account of intergovernmental agreements and work done by other governments when establishing a new regulatory limit

Where authorities decide that a new regulatory limit needs to be established for wine, this should be done with reference to limits already introduced by other authorities or recommended by appropriate intergovernmental organizations. If those limits exist, are suitably justified by the available science and are applicable taking account of any national or regional distinctives in the country proposing to

introduce the new limit, it would foster harmonization if the limit established was consistent with that imposed or recommended elsewhere. Care should be exercised to ensure that any adjustments to limits also take manufacturing realities fully into account.

Principle: Governments are respectfully requested to give due regard to intergovernmental agreements and work done by other authorities when establishing new regulatory limits.

3.4. Use common scientific units for the expression of regulatory limits

It is frequently the case when limits are set by governments, that they are expressed using different scientific units than used in other markets. This causes enormous confusion in international wine trade. Look again at Appendix I, and note that there are no less than 5 different ways that nations have chosen to express limits for methanol in wine. Note, also, that even where authorities set a limit that is identical quantitatively, they sometimes choose to express it differently in terms of units. For example, Ontario and Quebec apparently have the same limit for methanol but express it in different units. Methanol is only an illustrative example here of the confusion that also exists for all other regulatory limits established for wine around the world.

The United States industry considered this problem from a purely industry standpoint (where use of different units to express the same quantities can be problematic). It recommended to the US Government the adoption of a common set of units for expressing wine related values (see Appendix II). It would be greatly beneficial to trade in wine around the world if governments could reach agreement about the scientific units to be used in the expression of regulatory limits.

Principle: Governments are respectfully urged to adopt a common system of scientific units for expressing regulatory limits

3.5. Use a common basis for the expression of regulatory limits

3.5.1. Regulatory limits for wine expressed on a per unit of alcohol basis

Appendix I shows that some countries establish limits for methanol on the basis of the volume of alcohol in the wine and not on the volume of the wine itself. This means that as the alcohol content of a wine decreases, the relative content of a given substance expressed per unit volume of alcohol will increase:

Alcohol by Volume in Wine (%)	Methanol (mg) in 1L wine	Methanol (mg) per liter of alcohol in the same wine
15	100	670
14	100	710
13	100	770
12	100	830
11	100	910

This form of expressing regulatory limits is an increasing trend in regulatory proposals from various parts of the world over the last 2 years and has several negative consequences:

- It introduces the confusion of expressing a limit in a different way than other countries do (see 3.3 above)
- It can result in wines with lower alcohol contents approaching or exceeding regulatory limits, and therefore may effectively discourage producers from reducing the alcohol in their products to meet consumer demand.
- Where some governments who are less well-versed in wine have proposed regulatory limits on a per unit alcohol basis, the limits chosen would effectively have prevented any wine from entering the country because none is made that could comply with the proposed limit.

Note that the consumer is not exposed to any more or less of the limited substance by drinking a glass of any of the wines shown in the table above, because the content of methanol (in this case) per unit volume of the wine is the same in each case.

From the above it may be seen that limits set on the basis of the volume of alcohol in the wine seem not to be in the best interests of consumers or producers.

Principle: Governments are respectfully recommended to express regulatory limits on a "per unit volume of wine" basis rather than "per unit volume of alcohol" in the wine.

3.5.2. Different acids used as the basis for Titratable Acidity results expression

Many wine producing countries choose to establish limits for the amount of titratable acidity (TA) that a wine may contain. However, some choose to calculate the acidity measurement as though all the acid measured had been tartaric acid, and to express the limit in that way. Other countries choose to calculate as though all the acid had been sulfuric acid and express the limit that way. Although two limits may be identical, basing the expression of the limit in terms of different acids makes them look different and introduces the possibility for confusion in trade.

If there are other limits that are expressed in terms of a single wine constituent, but where different countries have chosen different substances for this purpose, this should also be reviewed.

Principle: Governments are respectfully recommended to agree on a common way of expressing results where this is done in relation to a single wine constituent.

3.6. Establish analytical "de minimis" provisions, or "action levels"

Over the last 10-20 years, great strides have been made in analytical sensitivity - both in terms of the ability to quantify substances that before were very difficult to measure, but also in terms of the sensitivity of analysis. In other words, it has become possible to measure smaller and smaller quantities of substances in wine, whether grape components or other chemicals that find their way into the product in the course of normal winegrowing operations. When regulations are framed in such a way that the presence of a substance is illegal, or else triggers some labeling requirement, this clearly has the potential to cause problems when combined with increasing analytical method sensitivity. Taken to the logical extreme, one molecule of a substance in a bottle of wine (if it could be measured) would be "presence" and could determine the legality of the product and/or that of its label. The scope for the inadvertent presence of such compounds in wine grows larger and larger with the ability to detect

smaller and smaller amounts. It includes, as examples, spray-drift of pesticides from a crop on which they are permitted to an adjacent vineyard on which they are not permitted, or residues of components used in tank sanitizing agents that cross-contaminate wine. The levels in question could never have been detected 10 years ago and are so low as to pose no known health risk to consumers, yet they can and do increasingly result in market access problems for wines subjected to the more advanced analytical methods.

In this situation, it would be helpful to establish for certain compounds or classes of compounds "de minimis" values - below which the substance(s) would not be counted as "present" for regulatory purposes. This would be set at a level to preserve consumer protections but to mitigate the potentially increasing trade issues that may arise in the next few years.

Principle: Where appropriate, Governments are respectfully requested to consider the establishment of analytical "de minimis" values for substances or classes of substances in wine - values below which they will be deemed to all intents and purposes not to be present in the wine.

3.7. Transition arrangements

From time to time, governments revise the limits in their regulations and lower them for various reasons. Wherever possible, this should be an effort harmonized with other countries (see 2.2 above). There have been cases in some markets around the world where limits have been lowered and enforcement action has begun overnight, even for issues where there was no pressing public health concern. Action has been commenced against wine that was produced perhaps 18 months to 2 years before the regulatory change was made and that was in complete compliance with the rules then in existence. Product has even been shipped from the producing country in perfect compliance with the rules then in force, only to arrive at the destination market as a non-compliant product because the government of that country chose to allow no transition arrangements and implemented new rules with immediate effect. Because of the nature of wine as a product with a potentially long shelf life, it is also important that consideration be given to grandfathering stocks of product that are already produced and in market but have significant shelf life remaining, so that they are not inadvertently and unnecessarily disadvantaged by changes in regulatory provisions.

In this situation, it would be very helpful for an agreement to be reached around transition timetables and grandfathering arrangements for various kinds of proposed regulatory changes (e.g. for labeling, composition etc.) in cases where there is no pressing need to act for public health reasons.

Principle: Governments are respectfully requested to agree upon and to allow suitable transition arrangements when limits are tightened, provided public health considerations so permit.

4. Laboratory Testing

4.1. Accredited/Certified laboratories and analysts

Laboratory accreditation systems and certification of laboratory analysts are means to ensure that Good Laboratory Practice is being followed within an analytical laboratory for certain analyses that it performs. This gives a level of confidence in the results that a laboratory produces.

Proficiency testing programs provide a means to determine how a laboratory performs for a given analysis in comparison with other laboratories, and can highlight systematic problems and lead to corrective actions.

The authorities in some jurisdictions offer certification programs for commercial and/or producer laboratories (or analysts), indicating that those labs or individuals have demonstrated proficiency with analyses required in international trade. The results produced by such labs with those methods can then be used on certificates of analysis or in other international trade scenarios, and the certified analyst can officially sign the documentation.

As far as possible, it would be helpful if those laboratories that perform analyses related to the international trade in wine were accredited/ had certified analysts for the methods they use for international trade purposes and could demonstrate an adequate level of performance for each of those methods.

Principle: Analyses of wine for compliance purposes should be undertaken by suitably accredited laboratories (or overseen by certified analysts) that perform acceptably for the specific test methods used.

4.2. Validated methods

Many laboratories that test wines for compliance with appropriate regulatory standards do so with methods that are validated in some way. Often this will mean that the method has been tested by 8-12 laboratories for the same sample(s) of the product for which the method is intended to be used and has demonstrated adequate performance for accuracy, repeatability etc.

It is clearly important for the international wine trade that the methods used to analyze the wines for compliance purposes are validated or have been shown by robust means to have performance characteristics that are acceptable when applied to wine.

Principle: Analytical methods used for wine compliance purposes should be validated and/or have a demonstrably appropriate level of performance for wine.

4.3. Authenticity methods

In almost all methods for determining wine authenticity, samples are tested for a certain characteristic and the results are then compared with those from known authentic wines. Problems have arisen historically when a method has been applied to wines whose origin or method of cultivation/production was not represented in the database of authentic samples with which its results were compared. Accordingly, the authenticity of perfectly legitimate wines has from time to time been called into question and this has caused problems in international trade.

It should therefore be a recognized principle in the authenticity testing of wine, that all the factors that might potentially influence the result of an analytical procedure are identified and taken into account in the construction of the database of authentic wines. Such factors may include (*inter alia*) production region, soil, rootstock, clone, variety, irrigation, trellising and pruning systems, leaf thinning, crop

thinning, climate, microclimate, season and all the possible permutations of winemaking practices that could legitimately have been used in the production of the wine, together with the age of the sample and the conditions under which it has been stored.

Principle: For wine authenticity analyses, the database of authentic samples with which the test samples will be compared must be sufficiently comprehensive to avoid the mis-categorization of legitimate samples as fraudulent.

4.4. Measurement uncertainty

The Methods of Analysis and Sampling Committee of the Codex Alimentarius Commission (CCMAS) has elaborated a revised explanatory note to an existing set of guidelines concerning measurement uncertainty (see Appendix III). The note makes it clear that it is anticipated that laboratories should assess the analytical uncertainty surrounding the determinations they make, and that they should make this available on request. In international trade situations, the note anticipates that the request for this information would be made.

It would clearly be a step towards objective and consistent enforcement for wines in international trade if the measurement uncertainty surrounding the various analyses was provided by the laboratories conducting the analysis, and if that information was taken into account in any decision to pursue enforcement action.

Principle: Laboratories testing for compliance purposes should supply measurement uncertainty information with their analytical results and the competent authorities should take this into account in interpreting analytical data

5. Conclusion

This document has proposed and explained several principles surrounding the establishment of regulatory limits on the one hand and the laboratory testing that is performed to establish compliance with those limits. Many of these principles are derived from real-world experiences in which unnecessary and costly problems have arisen. If all the principles mentioned above were uniformly adopted and applied by countries that trade in wine, the implications for market access and free movement of goods would be huge, but there would be no decrease in the level of protection that was afforded to consumers. Accordingly, a study of these principles by relevant governments is warmly commended, to see whether they might form the basis for an understanding among countries on these matters.

Appendix 1 - International Methanol Limits for Wine

Methanol Limits in Wine 1. Limits expressed on a volume methanol/volume wine basis

Argentina	0.35 ml/L of wine	*about 280 mg/L*

Methanol Limits in Wine 2. Limits expressed on a weight methanol /volume wine basis

OIV	400 mg/L of wine (Red) 250 mg/L of wine (White and Rosé)	
Canada (Ontario)	400 mg/L of wine	
Canada (Quebec)	0.4g/L of wine	*400 mg/L*
China	300 mg/L of wine (Red) 250 mg/L of wine (White & Rosé)	
Japan	1 mg/cubic cm of wine	*1000 mg/L*
South Africa	300 mg/L of wine	
Switzerland	300 mg/L of wine (Red & White) 150 mg/L of wine (Rosé)	

Methanol Limits in Wine 3. Limits expressed on a weight methanol /weight wine basis

Turkey	10 mg/kg of wine	*about 10 mg/L*

Methanol Limits in Wine 4. Limits expressed on a weight methanol/ volume of alcohol in wine basis

Australia	Domestic Production: White and Sparkling Wines – 2 g/L ethanol Other Products - 3 g/L ethanol Imported Wines: 3 g/L of ethanol
New Zealand	3 g/L of ethanol
Vietnam	3 g/L of ethanol
Taiwan	2 g/L of ethanol
Korea	2 g/L of ethanol
India	2 g/L of ethanol

Methanol Limits in Wine 5. Limits expressed on a percent basis (not clear if basis is weight or volume)

Russia	0.1% of the ethanol content

Appendix II

Technical Brief

Adoption of International Units of Measurement in United States Wine Analysis

Gordon Burns and Arthur Caputi, Jr.

The United States wine industry is competing in an increasingly global environment where attention to reduction of technical barriers to trade is an important consideration. One common existing technical barrier is the lack of a common language for expression of wine analytical results. The Wine Institute Technical Committee has adopted the consensus units described in this brief and recommends their adoption by the U.S. industry.

Key words: Wine analysis, international units, units of measurement, harmonization

Laboratories performing various wine and grape must and juice analyses report results in several different units of concentration. Frequently there are inconsistencies in reporting units, even within a given laboratory. It is common to find the concentration of analytes recorded in percent, g/L, g/100 mL, mg/100 mL, ppm, and mg/L on the same analytical report. This can easily cause confusion among organizations when attempting to reconcile results and can particularly become an issue in international trade.

In recent years, there has been significant progress toward acceptance of international standards, as evidenced by adoption of SI units for expression of many scientific terms and adoption of metric units for package sizes. The Wine Institute Technical Committee decided to investigate common wine analysis reporting procedures in the United States to determine how these compared to those used by the international wine community. The committee designed a survey to determine the units of concentration used by a number of U.S. wine industry laboratories for reporting of analytical results.

Eight laboratories were surveyed, representing the largest wine producers in the United States and an independent wine analysis laboratory. The Technical Committee determined that the resulting data would be representative of current U.S. wine analyses reporting practices. The results were then compiled and compared to the units used by the OIV (Office International de la Vigne et du Vin) in their Official Methods of Analysis [1]. Several differences in expression of results were observed, but after extensive discussion the Technical Committee reached a consensus as to the units it considered most appropriate. Results of the survey, current OIV units, and units agreed to by consensus are presented in Table 1.

Two issues prevent complete accord with current OIV units of expression, as can be noted in Table 1. The first is the choice of acid in which titratable acidity is expressed. The OIV standard, and that of the European Union, is the use of sulfuric acid as the reference acid, while the committee consensus and U.S. standard is tartaric acid. The Technical Committee agreed that the U.S. reference acid should be retained for two reasons. Not only is the use of tartaric acid as the reference compound a consistent and long-standing one in the United States, but also it seems the more logical choice, since tartaric is the primary acid found in grapes.

The second issue is choice of temperature at which percentage of alcohol by volume is expressed. The international standard temperature adopted everywhere except the United States is 20 degrees Celsius (20ºC). The United States still relies upon the temperature of 60 degrees Fahrenheit (60ºF, or 15.56 degrees Celsius). It is the hope of the Technical Committee that the U.S. Bureau of Alcohol, Tobacco and Firearms, in the interest of international harmonization of units, will take steps to address this discrepancy.

Members of the Technical Committee and the surveyed laboratories agreed to adopt the consensus units as presented in Table 1 for purposes of international trade and to move toward adoption of the consensus units for routine internal analyses. The Technical Committee additionally recommends that the consensus units be adopted by the U.S. industry in the interests of international harmonization.

Literature Cited

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Acknowledgments: The authors express their thanks to the other members of the Wine Institute Technical Committee: Terry Lee (Committee Chair), Greg Hodson, Wendell Lee, Anil Shrikhande, Jim Walls, John Franzia, Tom Smith, James Huntsinger, Kathy Meechan, Richard Peterson, Michael Martini, Glenn Proctor, David Stevens, and Elie Skofis.

Manuscript submitted April 2002; revised May 2002

Table 1 Units of measurement used in surveyed laboratories and by the OIV, and units agreed to by consensus of the Wine Institute Technical Committee.

	Current units: laboratories								OIV	Proposed
	IL[a]	A	B	C	D	E	F	G		
Total SO_2	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	**mg/L**
Free SO_2	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	**mg/L**
Ethanol	% v/v at 60°F	% v/v at 60°F	% v/v at 60°F	% v/v at 60°F	% v/v at 60°F	% v/v at 60°F	% v/v at 60°F	% v/v at 60°F	% v/v at 20°C	**% v/v at 20°C**
Total dry extract	g/L	g/L	g/L	g/L	g/L	g/L	g/L	g/L	g/L	**g/L**
Reducing sugar	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/10 mL	g/100 mL	g/L	**g/L**
Volume	mL	mL	mL	mL	mL	mL	mL	mL		**mL**
Sorbic acid	mg/L	mg/L	mg/L	mg/L		mg/L	mg/L	mg/L	mg/L	**mg/L**
Titratable acidity	g/100 mL as tartaric	g/100 mL as tartaric	g/100 mL as tartaric	g/100 mL as tartaric	g/100 mL as tartaric	g/100 mL as tartaric	g/100 mL as tartaric	g/100 mL as tartaric	g/L as sulfuric	**g/L as tartaric**
Volatile acidity	g/100 mL as acetic	g/100 mL as acetic	g/100 mL as acetic	g/100 mL as acetic	g/100 mL as acetic	g/100 mL as acetic	g/100 mL as acetic	g/100 mL as acetic	g/L	**g/L as acetic**
Citric acid	g/L	mg/L	mg/L	g/L		g/L	g/L	mg/L	g/L	**g/L**
Glucose + fructose	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/100 mL	g/L	**g/L**
Malic acid	g/L	mg/L	mg/L	mg/L		mg/100mL	mg/L	mg/L	g/L	**g/L**
Tartaric acid	g/L	mg/L	mg/L	g/100 mL		g/100 mL	mg/L	g/L	g/L	**g/L**
Lactic acid	g/L	mg/L	mg/L	mg/L		g/L	mg/L	mg/L	g/L	**g/L**
Ammonia	mg/L	mg/L	mg/L	mg/L		mg/L	mg/L	mg/L	mg/L	**mg/L**
Potassium	mg/L	mg/L	mg/L	mg/L			mg/L	mg/L	mg/L	**mg/L**
Calcium	mg/L	mg/L	mg/L	mg/L			mg/L		mg/L	**mg/L**
Benzoic acid	mg/L	mg/L	mg/L	mg/L			mg/L		mg/L	**mg/L**
4-Ethylphenol	ng/mL	ng/g					µg/L			**ug/L**
Copper	mg/L	mg/L	mg/L	mg/L	mg/L		mg/L	mg/L	mg/L	**mg/L**
Iron	mg/L	mg/L	mg/L	mg/L	mg/L		mg/L	mg/L	mg/L	**mg/L**
Fluoride	mg/L	mg/L	mg/L	mg/L			mg/L	mg/L	mg/L	**mg/L**
Magnesium	mg/L	mg/L	mg/L	mg/L			mg/L		mg/L	**mg/L**
Lead	mg/L	µg/L	µg/L				µg/L		µg/L	**ug/L**
Arsenic	mg/L	µg/L	mg/L				µg/L		mg/L	**mg/L**
Zinc	mg/L	µg/L	mg/L				mg/L		mg/L	**mg/L**
Acetaldehyde	mg/L	mg/L	mg/L				mg/L		mg/L	**mg/L**
Ethyl acetate	mg/L	mg/L	mg/L				mg/L		mg/L	**mg/L**
Pesticides	mg/L	mg/L					µg/L		mg/L	
Ethyl carbamate	ng/g	ng/g or µg/Kg	µg/Kg				µg/L		µg/L	**µg/L**
Total phenolics	mg/L	mg/L	mg/L	mg/L		mg/L	mg/L	mg/L	mg/L	
Methanol	mg/L	mg/L	mg/L				mg/L		mg/L	**mg/L**
Carbon dioxide	mg/100 mL	mg/100 mL	mg/100 mL	mg/100 mL	mg/100 mL	mg/L	mg/100 mL	mg/L	g/L	**g/L**

[a]IL: Independent wine analysis laboratory.

REP11/MAS

APPENDIX II

DRAFT REVISED GUIDELINES ON MEASUREMENT UNCERTAINTY

EXPLANATORY NOTES TO THE CODEX GUIDELINES ON MEASUREMENT UNCERTAINTY

(To be included as an Annex to the Guidelines on Measurement Uncertainty (CAC/GL 54-2004))

(At Step 8 of the Procedure)

1 What is Measurement Uncertainty?

It is not always appreciated that analytical results are variable, and just how large that variability may be, particularly when low concentrations of a measurand (i.e. ppb levels) are being determined. As stated in the Guidelines, "most quantitative analytical results take the form of "a ± 2u" or "a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "u" is the standard uncertainty to 68% level of confidence and "U" (equal to 2u) is the expanded uncertainty to 95% level of confidence.. The range "a ± 2u" represents a 95% level of confidence in which the true value would be found. The value of "U" or "2u" is the value which is normally used and reported by analysts, normally referred to as "measurement uncertainty" and may be estimated in a number of different ways. "

In food analysis it is the (approximately) 95% probability (i.e. 2u) which is used to calculate the expanded uncertainty. Other sectors may specify a different probability.

Thus measurement uncertainty can be regarded as the variability around the reported results which is quantified as the value "U" when considering the expanded uncertainty and within which the "true" result may be expected to lie.

2 Does the Measurement Uncertainty have to be Estimated in Codex?

Yes, one of the requirements of the ISO/IEC 17025:2005 Standard that Codex has adopted by reference is that the measurement uncertainty of a result shall be estimated and then made available if requested. The Codex Alimentarius Commission has developed Guidelines CAC/GL 27-1997 that require laboratories involved in the import/export of foods to comply with general criteria in ISO/IEC 17025. As Codex is concerned with goods moving in international trade it would be anticipated that the request for measurement uncertainty estimates will be made.

3 Does Measurement Uncertainty Arise From both Sampling and Analysis?

Measurement uncertainty applies to the whole measurement process. However, this guidance only considers analytical measurement uncertainty.

In many cases uncertainty of sampling is as large as or larger than analytical measurement uncertainty. Uncertainty of sampling is often the overriding factor in conformity assessment procedures. Sampling procedures in the *General Guidelines on Sampling* are designed to take account of uncertainty of sampling.

4 What is the Relationship between Measurement Uncertainty, the Analytical Result and the Method Used to Obtain the Result?

The uncertainty of test results is not associated with the method of analysis. However, the estimates of analytical performance characteristics that are obtained in the validation and/or in quality control of a method may be used to estimate the uncertainty of a result in some situations. The differentiation between measurement uncertainty associated with the result and precision obtained during the validation of the method is frequently not appreciated. As a consequence precision demonstrated for a validated method (the repeatability or reproducibility standard deviation) cannot be used as the sole estimate of the measurement uncertainty without qualification. In particular additional factors such as uncertainty associated with bias, matrix effect, and competence of laboratory must be considered.

5 Procedures for Estimating Measurement Uncertainty

There are many procedures available for estimating the measurement uncertainty of a result. The Codex guidelines do not recommend any particular approach, but it is important that whatever approach is used, the procedure is scientifically credible. No one approach may be said to be better than any other provided the procedure used is appropriate and credible - i.e. there is no "hierarchy" of the procedures.

In general, procedures are based on a component-by-component ("bottom-up") approach or on a "top-down" approach using data from collaborative trials, proficiency studies, validation studies or intra-laboratory quality control samples, or a combination of such data.

In the *Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and Export Control of Foods* (CAC/GL 27-1997) there is a requirement to use validated methods and so it is usually more cost-efficient to use data from the method validation studies rather than using another approach (i.e. the component-by-component approach).

Users of validation data should note that sources of uncertainty that are not or only partly covered by validation studies include[1]:

- Sampling
- Pre-treatment
- Method bias
- Variation in conditions
- Changes in sample matrix
- Imprecision in estimating method or laboratory bias

For methods operating within their defined scopes, when the reconciliation stage shows that all the identified sources have been included in the validation study or when the contributions from any remaining sources have been shown to be negligible, then the reproducibility standard deviation s_R, adjusted for concentration if necessary, may be used as the combined standard uncertainty.

It is recognised that further procedures for the estimation of measurement uncertainty are being developed, and that, in this evolving situation, further recommendations will be made as to acceptable procedures. It is anticipated that procedures based on results obtained from participation in proficiency testing programmes, as an example, will be developed.

6 Considerations when Estimating Measurement Uncertainty within the Context of Codex

It is important that the requirement to estimate measurement uncertainty does not impose any unnecessary additional workloads on laboratories.

When deciding on which procedure is to be used when estimating measurement uncertainty within the Codex context it is important to recognise that Codex has adopted a number of formal quality assurance measures that have to be implemented by control laboratories. In particular, such laboratories should:

- be in compliance with an internationally recognised standard (now with ISO/IEC 17025:2005 Standard); such compliance is aided by the use of internal quality control procedures,
- participate in proficiency testing programmes, and
- use validated methods.

[1] EURACHEM/CITAC Guide on the Use of uncertainty information in compliance assessment EURACHEM Secretariat, BAM, Berlin, 2007. This is available as a free download from http://www.eurachem.org/

It is essential that the information provided as a result of these requirements being implemented is used by laboratories when estimating their measurement uncertainties in order to avoid unnecessary work being carried out by laboratories. In Codex, where there is a high emphasis being placed on the use of "validated" methods of analysis, i.e. methods which have been validated through collaborative trials, information obtained from such trials can be used in many situations.

In addition, information derived from internal quality control procedures may also be used to estimate uncertainties in some situations.

This section re-emphasises that for the analyst it is important that no unnecessary duplication of existing work is undertaken.

7 Values of Measurement Uncertainty Estimates ~~Estimations~~

Stipulating information on the anticipated values of measurement uncertainty estimates is frequently not supported by analysts. The users of analytical data and the customers of the laboratories producing such data frequently ask for such information regarding the level of uncertainty that may be expected for test results. They have concerns that some laboratories underestimate the size of their uncertainties and so report unrealistically small uncertainties to their customers.

For chemical analyses, using the values of s_R from collaborative trials, it would be reasonable to anticipate that the (expanded) uncertainties reported by laboratories would be approximately the following:

Nominal Concentration	Typical Expanded Uncertainty	Expected Range of Results*
100g/100g	4%	96 to 104g/100g
10g/100g	5%	9.5 to 10.5g/100g
1g/100g	8%	0.92 to 1.08g/100g
1g/kg	11%	0.89 to 1.11g/kg
100mg/kg	16%	84 to 116mg/kg
10mg/kg	22%	7.8 to 12.2mg/kg
1mg/kg	32%	0.68 to 1.32mg/kg
< 100µg/kg	44%	0.56 x concentration to 1.44 x concentration µg/kg

* this effectively means that values falling within these ranges may be regarded as being of the same analytical population.

It would be expected that the reported measurement uncertainties by any laboratory would not significantly exceed the value estimated from the s_R at the concentration of interest if the laboratory is in "analytical control". Very experienced laboratories carrying out any particular analysis on a regular basis would be expected to obtain uncertainty values less than the values given above.

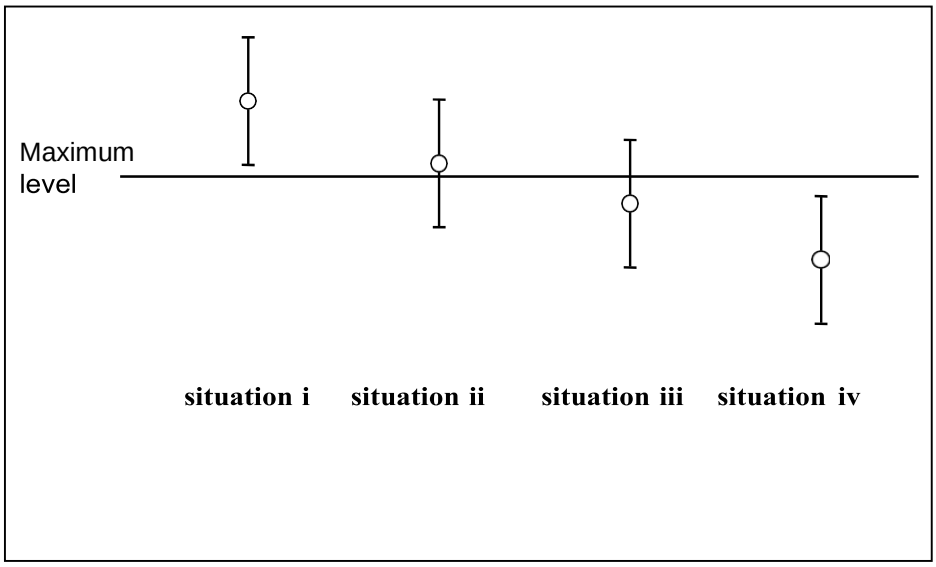
.8. Relationship between analytical results, measurement uncertainty and recovery factors

This section attempts to explain the significance of analytical results and their associated measurement uncertainties and recoveries.

8.1 Measurement Uncertainty

It is important that measurement uncertainty is considered when deciding whether or not a sample meets the specification. This consideration may not apply when a direct health hazard is concerned. The significance of this can be illustrated by an example shown in the diagram below, which shows the simplest case when decisions are made based on a single test sample.

The example shown here is one where the test result is compared against the specification consisting of a maximum level. It illustrates how the concept of measurement uncertainty could be taken into account when interpreting analytical results on a tested sample.



This diagram demonstrates the importance of defining clear guidelines to allow unambiguous interpretation of analytical results with respect to their measurement uncertainties.

Situation i

The analytical result minus the measurement expanded uncertainty exceeds the maximum level. The result indicates that the measured analyte in the test sample is above the specification.

Situation ii

The analytical result exceeds the maximum level by less than the expanded measurement uncertainty.

Situation iii

The analytical result is less than the maximum level by less than the expanded measurement uncertainty.

Situation iv

The analytical result is less than the maximum level by more than the expanded measurement uncertainty.

8.2 Recovery

The Codex Alimentarius Commission has adopted the IUPAC Guidelines on the use of recovery information by reference (see CAC/GL 37-2001).

Analytical results should be expressed on a recovery-corrected basis where appropriate and relevant, and when corrected they have to be stated as such.

If a result has been corrected for recovery, the method by which the recovery was taken into account should also be stated. The recovery rate is to be quoted wherever possible. The uncertainty of measurement should include the uncertainty associated with the recovery correction or be quoted in conjunction with the stated recovery.

When laying down provisions for standards, it will be necessary to state whether the result obtained by a method used for analysis within conformity checks is expressed on a recovery-corrected basis or not.

9 Useful References

These references are provided for information purposes only.

Guides for the Estimation of Measurement Uncertainty

Guide 98, Guide to the Expression of Uncertainty in Measurement (GUM) ISO, Geneva (1995)

EURACHEM/CITAC Guide Quantifying Uncertainty in Analytical Measurement (Second Edition), EURACHEM 2000. This is available as a free download from http://www.eurachem.org/

Analytical Methods Committee of the Royal Society of Chemistry "Uncertainty of Measurement - Implications of its use in *Analytical Science", Analyst, 1995,* ***120 (9)****, 2303-2308*

ISO 21748:2010 Guidance for the Use of Repeatability, Reproducibility and Trueness estimates in Measurement Uncertainty Estimation, ISO, Geneva (2010)

NIST Technical note 1297 (1994 Edition): "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results"

NMKL Procedure No. 5, 2nd edition (2003): "Estimation and Expression of Measurement Uncertainty in Chemical Analysis"

UKAS (United Kingdom Accreditation Service) 2000 The Expression of Uncertainty in Testing Edition 1, UKAS Publication ref: LAB 12

Eurolab technical Report No. 1/2007. Measurement Uncertainty Revisited: Alternative Approaches to Uncertainty Evaluation. Available as a free download from www.eurolab.org

Nordtest report TR 537. Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories. Available as free downloads from www.nordtest.org (although this handbook is directed towards environmental analyses, the approaches and examples described are applicable to the results from tests on foods and feeds)

Procedures for the Validation of Analytical Methods and Method Performance

"Precision of Test Methods", Geneva, 1994, ISO 5725, Previous editions were issued in 1981 and 1986. (not adopted by Codex)

"Protocol for the Design, Conduct and Interpretation of Method Performance Studies", ed. W. Horwitz, *Pure Appl. Chem.*, 1995, 67, 33 1-343 (adopted by Codex)

European Commission Decision 2002/657/EC implementing directive 96/23/EC Concerning the Performance of Analytical Methods and the Interpretation of Results, Off J Eur Comm, L22 1 (2002) 8-36

Validation of Chemical Analytical Methods. NMKL Procedure No 4, 4th Version, 2010

Accreditation etc

ISO/IEC 17025:2005, General Requirements for the Competence of Testing and Calibration Laboratories, ISO, Geneva (2005)

EURACHEM Guidance Document No. 1/WELAC Guidance No. WGD 2: "Accreditation for Chemical Laboratories: Guidance on the Interpretation of the EN 45000 series of Standards and ISO/IEC Guide 25"

Z., Ben-David, H., Mates, A. 2001 Proficiency testing as tool for ISO 17025 implementation in National Public Health Laboratory: a mean for improving efficiency. Accreditation & Quality Assurance, **6**: 190-194

NMKL Procedure No. 3 (1996) "Control charts and control samples in the internal quality control in chemical food laboratories" Örnemark, U., Boley, N., Saeed, K., van Berkel, P.M., Schmidt, R., Noble, M., Mäkinen, I., Keinänen, M., Uldall, A., Steensland, H., Van der Veen, A., Tholen, D. W., Golze, M., Christensen, J.M., De Bièvre, P., De Leer, W. B (ed). 2001

Proficiency testing in analytical chemistry, microbiology, and laboratory medicine – working group discussions on current status, problems, and future directions. Accreditation & Quality Assurance, **6**: 140-146

Compliance

EURACHEM/CITAC Guide on the Use of uncertainty information in compliance assessment EURACHEM, 2007. This is available as a free download from http://www.eurachem.org/

Terminology

ISO (2nd ed., 1993) VIM "International Vocabulary of Basic and General Terms in Metrology". Geneva

ISO Guide 99, International Vocabulary of Basic and General Terms in Metrology, 3rd Ed., VIM3, ISO, Geneva (2008)