codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 5 **CX/MAS 01/6-Add.1**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Twenty-Third Session
Budapest, Hungary, 26 February - 2 March 2001

CONSIDERATION OF HARMONIZED GUIDELINES FOR THE USE OF RECOVERY INFORMATION ON ANALYTICAL MEASUREMENT GOVERNMENT COMMENTS

FINLAND

The use of recovery factors to correct analytical results is a very complicated matter. One of many problems is that most food laboratories analyse such a wide range of matrices. It is only rarely possible to determine, to a sufficient degree the recovery from different foods.

The form of analyte to be added when determining recovery is crucial. It is a well known fact that an added analyte may be completely recovered while the naturally occurring analyte, which is chemically bound to various components of the food is not.

Most laboratories in Finland do not correct analytical results for recovery, especially not analytical results needed by the official food control and results relating to food moving in international trade. However, some laboratories use recovery information to correct surveillance results. In such cases the use of a recovery factor and its value is stated with the result.

Finland do not support adoption of the IUPAC harmonized guidelines. It is suggested that at the forthcoming session of the CCMAS it should be discussed whether it would be a good idea that whenever the recovery of a measurement has been determined, the recovery is stated together with the uncorrected result.

UNITED STATES

The United States endorses the scientific principles of the IUPAC “Harmonized Guidelines for the Use of Recovery Information in Analytical Measurement” but does not agree that analytical results should be corrected for recovery as a general policy. The purpose of establishing methods of analysis in this Codex Committee is to enforce Codex specifications, not necessarily for IUPAC’s aim “to achieve the best estimate of the true result”. This IUPAC objective is incorrect for may enforcement programs of regulatory agencies in the US and other countries, as well as for contracts that may specify not to correct or are silent in this regard. We fully agree with IUPAC’s and CCMAS’s desire to avoid confusion in reporting analytical results, but think that this can be accomplished by clearly identifying as to whether or not a recovery correction has been applied. Our position remains identical to that presented in CRD 8 at the last CCMAS meeting which is essentially reproduced below to provide the details supporting the main points that were captured in document CX/MAS 01/6

We recommend that this Committee thank IUPAC for this document and that the final publication be referenced in Codex Procedural Manual. We further suggest that CCMAS adopt a statement with respect to recovery somewhat as follows:

"With respect to specific chemical entities, results may be reported corrected or non corrected as stated or implied in the Codex standard. The report of analysis should give information as to use of a correction factor. If a correction is required or permitted, the calculation should be part of the method of analysis. If recovery is calculated and the directions are not present in the protocol, the report should state how the correction was derived."

DISCUSSION

The document continues to reiterate the incorrect premise that food laboratories wish to achieve "the best estimate of the true result". This is not correct because food standards and specifications in many cases are not set up in terms of "the true result", defined as a specific chemical entity or group of distinct entities. Food and food components have historically been defined in terms of practical specifications, which are often labelled as the chemical entity they are meant to simulate.

To take a simple example, many foods have a maximum limit for the entity "water", colloquially called "moisture", to control the addition of this adulterant. Water is the chemical entity dihydrogen oxide, for which a true value certainly exists, but which is practically unattainable because unmeasurable molecular forces cause adsorptive binding to container and food surfaces and because of variable hydrogen binding of water molecules to polar food matrix components such as proteins and carbohydrates. Although a method does exist for determining the chemical entity water in foods [the titrimetry Karl Fisher Method], it is rarely used because the legal standards for the control of moisture are usually specified in terms of drying the food for a certain time at a certain temperature. Food chemists have never considered the possibility of expressing the results in terms of the "true value" for dihydrogen oxide. If the value for "moisture" were to be corrected to the "best estimate of the true result" as suggested in this document, it would no longer correspond to the value required in the standard as "moisture".

A more complex example is exhibited by vitamins. Vitamin activity is often exhibited by a number of related compounds, each with different biological activity. Although the different forms can be separated by high performance liquid chromatography, this is not done; rather an all-inclusive generic method is usually applied to obtain, for example, "vitamin E activity". The B vitamins can be measured chemically to obtain "true values" or microbiologically to obtain "biological activity". Where discrepancies exist between the chemical "true value" and the biological activity, the biological value is usually accepted. When a dispute arose a few years ago with respect to the activities of synthetic vitamin D congeners, the rarely used rat bioassay was invoked to determine the "true activity" not the "true value". In some cases, the analyte cannot even be defined as a chemical entity, e.g peroxide value. In other cases, the analyte changes in accordance with viewpoint, e.g. crude fiber, nutritional fiber, structural fiber, soluble fiber, etc.

ANNEX II - IUPAC REPORT

FOREWORD – the opening sentence is unnecessarily "contentious". There are good reasons for current practices: legislative, scientific, and historical. We do not recall ever seeing the quoted phrase as an aim of IUPAC in any of its documentation. Although the document provides for some fields to exempt themselves from the provisions, it clearly is slanted in the direction of requiring correction to obtain the "best estimate of the true result", whether it is attainable or not.

INTRODUTION– The document does not recognize that the practice of correction is relatively recent. Food composition analysis is almost a century and a half old and the control of adulteration and misbranding is based almost entirely on empirical, uncorrected analytical values.

Specifications and limits involving food safety, such as pesticide and veterinary drug residues, are established as a result of animal feeding studies of the pure chemical. The amount of chemical residue left in the edible tissues, often extrapolated after application of safety factors as large as 1000x, at the point of no apparent harmful effect on the test animals, as determined by the method of analysis supplied by the sponsor of the chemical, is listed as the required analytical limit. Because of the large uncertainties involved in establishing this

limit, there was little point to attempting to attain a “true result” through the application of analytical correction factors. Losses entailed in establishing the analytical limit were inherently included in the limit. To now apply correction factors in such situations invokes the insidious “double counting” of correction factors so common in establishing the error budget method of calculating measurement uncertainties. The authors of the document are obviously unaware or have chosen to ignore this historical perspective.

In addition to ignoring the potential for double counting of correction factors, the effort involved in obtaining the factor is not worth an improvement in the “true result”, if any. Mc Kone and Bogen in their exhaustive report “Uncertainties in Health-Risk Assessment: an Integrated Case Study Based on Tetrachlorethylene in California Ground Water” (Regulatory Toxicology and Pharmacology (1992) 15, 86-103) estimated that concentration variance of this compound contributed only 20% of the total estimated variance of the total risk of this compound , per se. Thus a recovery correction to analytical values typically provides a second or third order correction factor to food safety, and is negligible.

The document fails to handle the problem in a simple, straight-forward manner for Codex purposes. All that is needed is a guideline statement that results should be reported in the same manner as the specification, limit or tolerance. If no statement is made it may be assumed to be established as is customary in the country whose legislation has jurisdiction. (In the United States, residue values for enforcement purposes are reported uncorrected). If two countries with different requirements are involved, all that is needed is a report in either system together with the recovery factor used in the calculation.

Further comments will be found in the essay “Correction Factors” that appeared in AOACI publication “Inside Laboratory management”, July 1997, pp.4-5.