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DETERMINATION OF ORGANOCHLORINE PESTICIDES IN TOMATO AND EVALUATION OF PROFICIENCY TESTING RESULTS

Burcu Binici Gökçen [1], *Fatma Akçadağ* [2]

[1] TUBITAK National Metrology Institute (UME), Kocaeli, Turkey, burcu.binici@ume.tubitak.gov.tr
[2] TUBITAK National Metrology Institute (UME), Kocaeli, Turkey, fatma.sogut@ume.tubitak.gov.tr

Abstract – In this study proficiency testing (PT) scheme on determination of organochlorine pesticides in tomato which is organized by TUBITAK National Metrology Institute (UME), is evaluated. Participating laboratories are supposed to analyse Endosulfan I, Endosulfan Sulfate, Alpha-BHC, Heptachlor, Aldrin and 4,4 DDD pesticides in tomato. The aim of proficiency testing scheme organization is to evaluate of performance of laboratories which analyse the pesticide residue in tomato.

Keywords: Organochlorine pesticide, proficiency testing

1. INTRODUCTION

Pesticide residues, both natural and synthetic, can be found in most of the things we eat, for example, fruits, vegetables, bread, meat, poultry, fish, and the processed foods made from them. Some of this pesticide contamination is legal, but does this mean it is safe? Much of it is illegal, with residues found in excess of regulatory safe levels. Identifying and determining the level of trace contaminants in our food and environment is critical in protecting and improving human health and the environment. In this concern, monitoring residues of pesticides in vegetables is nowadays a priority objective in pesticide research in order to get an extensive evaluation of vegetable quality to avoid possible risks to human health.[1]

The accurate measurement of residues helps to better protect our community and develop superior production practices. In this aspect, in order to increase the quality of chemical analysis of pesticides residues in tomato and awareness of metrology in Turkey, TUBITAK UME Chemistry Group Laboratories has been organizing Proficiency Testing Schemes in the field of organochlorinated pesticides in tomato since 2004. [2-5]

As a result of the PT schemes organized by TUBITAK UME, the accuracy and reliability of the results obtained by participating laboratories are increased and improved.

2. THE METHOD

2.1. Experimental

To prepare testing samples, 2 kg of tomato is minced and then standard reference material which is consist of 18 organochlorine pesticide is injected in it. Then this mixture is mixed for 1 hour to obtain a homogenous mixture. Then this mixture is separated as equal to 100 mL glass jar. 100 g testing sample is send to participating laboratories. The reference values of pesticides in tomato are determined by TUBITAK UME Chemistry Group Laboratories.

To analyse the pesticide residues in tomato, dichloromethane extraction method and clean-up with fluorosil are used. Analyses are carried out by GC-ECD.

2.2. Proficiency Testing Scheme Procedure

Different numbers are given to participating laboratories to follow up the study results. In this proficiency testing scheme, privacy of results and information of laboratories is considered.

Participating laboratories send their results to web site until given date. Participating laboratories are advised to treat PT samples in the same manner as routine samples, in order that the quality of routine measurements can more easily be assessed by the laboratory

The results of participating laboratories are evaluated as statistically and Z scores are calculated for each laboratory. At the end of the evaluation, the report is prepared which is consist of results and Z score values. Also, this report is issued on web site. Attendance Certificate is send to participants. [6-10]

2.3. Evaluation of the Results

In this study, preparation of samples and calculation of reference value and target standard deviation is executed by chemistry group laboratories. Z score is calculated to evaluate the results. Z score is independent of unit of result. Because of this, it can be used to compare different methods and results of analyses. It is easy to understand and to interpret the Z score. Z score is calculated by using equation (1)

$$z = \frac{x - X}{s} \tag{1}$$

The where X is reference value, x is an individual reported value found by the laboratory and s is target standard deviation. [11]

A common classification based on z scores can be made:

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$z \leq 2$ Satisfactory

$2 < z < 3$ Questionable

$z \geq 3$ Unsatisfactory

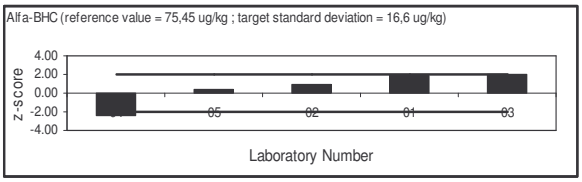

Figure 1: An illustration for the presentation of z score results in the report of proficiency testing

2.4. Determination of Target Standard Deviation, s

The target standard deviation of reproducibility found in collaborative trials is generally considered an appropriate indicator of the best agreement that can be obtained between laboratories. However, not all analyses have been characterised in this manner. In such cases, the predictive models of the appropriate form of the Horwitz Equation are valuable indicators of best practise.

This equation predicts a target standard deviation from a given concentration, *X*, and requires *X* to be expressed as a dimensionless target standard deviation predicted by the equation in the original concentration units it must be divided by the relevant mass ratio, e.g. 1 ppm$\equiv 10^{-6}$ or % $\equiv 10^{-2}$. It follows therefore that to express the dimensionless target standard deviation predicted by the equation in the original concentration units it must be divided by the relevant mass ratio:

i) for analyte concentrations < 120 ppb

$$s = 0.22C$$

ii) for analyte concentrations ≥ 120 ppb and 13.8 %

$$s = 0.02C^{0.8495}$$

iii) for analyte concentrations > 13.8 %

$$s = 0.01C^{0.5}$$

C is the reference concentration value and is obtained by using validated method.

In this study, target standard deviation, *s*, is calculated by using Horwitz equation and reference values is determined by using validated method.

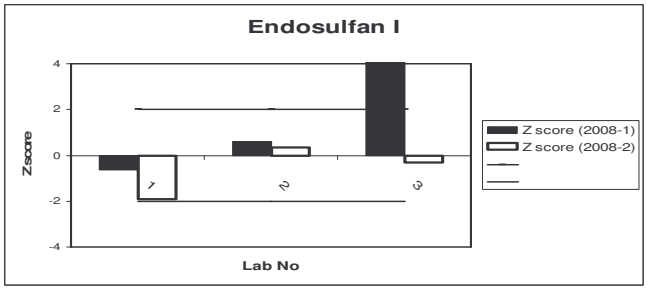

Figure 2: Distribution of results of organochlorine pesticide, endosulfan I, in tomato 2008

Figure 3: Distribution of results of organochlorine pesticide, aldrin, in tomato in 2008

The distribution of results of endosulfan I and aldrin in tomato PT scheme which is organized in 2008 is given at Figure 1 and 2.

The idea of the PT scheme is to enable participating laboratories to see their stability and improvement on analysing of the compounds in any matrix. In Figure 2 and 3, the results of the 1st and 2nd laboratory stay in limit values and there is an improvement on results of 3rd laboratory.

4. CONCLUSIONS

Proficiency testing schemes provide an excellent mechanism to collect, analyse and disseminate data on the relative performance of the different methods of analysis which can be used for any given determination. By driving this mechanism, the performance of the laboratories which analyse the pesticide residue in tomato will be kept high.

REFERENCES

[1]. D. K. Essumang, D. K. Dodoo, 1 C. K. Adokoh, and E. A. Fumador, "Analysis of Some Pesticide Residues in Tomatoes in Ghana" *Human and Ecological Risk Assessment,* vol. 14, pp. 796-806, 2008.

[2]. ISO GUIDE 43-1 Proficiency Testing by Interlaboratory Comparison Part 1-Development and Operation of Proficiency Testing Schemes, 1996.

[3]. ISO GUIDE 43-2 Proficiency Testing by Interlaboratory Comparison Part-2 Selection and Use of Proficiency Testing Schemes by Laboratory Accreditation Bodies, 1996

[4]. ILAC-G13:08/2007, Guidelines for the Requirements for the Competence of the Providers of Proficiency Testing Schemes,

[5]. ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories, 2005
[6]. T. Farrant, "Practical Statistics for the Analytical Scientist", *Royal Society Chemistry*, 1997.
[7]. R.E. Lawn, M. Thompson, and F. R. Walker, "Proficiency Testing in Analytical Chemistry", *Royal Society Chemistry*, 1997
[8]. K.W. Boyer, W. Horwitz, and R. Albert, *Analytical Chemistry*, vol. 57, pp. 454-459, 1985.
[9]. Domateste Pestisit Tayini Yeterlik Testi Çalışması Protokolü, TÜBİTAK UME, 2004-2008
[10]. Domateste Pestisit Tayini Yeterlik Testi Raporu, TÜBİTAK UME, 2004-2008
[11]. Proficiency Testing as a Tool for Accreditation Bodies and Customers of Laboratories, *A report on the feasibility of using PT as a tool to provide third parties with information on laboratory competence,* pp. 20*,* September,1999.