PROFICIENCY TEST FOR THE DETERMINATION OF PESTICIDES IN PAPAYA

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1 – Introduction

Since 2002, the National Institute for Quality Control on Health of the Fiocruz Foundation of Brazil (INCQS/FIOCRUZ) has organized Proficiency Tests (PT) on pesticide residues in fruits and vegetables (Table 1).

Year	Proficiency Test	Pesticides	Compounds	Matrix	Participants (Labs)
2002	INCQS.1.AG.1	Organophosphates	8	Tomato	4
2002	INCQS.1.AG.2	Organophosphates Organocholines Benzimidazols Dicarboximides	10	Tomato	17
2003	INCQS.1.AG3	Dithiocarbamates	1	Banana	14
2004	EP/INCQS AG4	Pyrethroids Organophosphates	4	Tomato	6

The results of a proficiency test (PT) organized jointly by The National Institute of Metrology (Inmetro/Dimci) and by the Oswaldo Cruz Foundation (Fiocruz/INCQS) of Brazil is presented. The aim of the study was the determination of pesticide residues in papaya (*Carica papaya*).

2 – Objectives

The objective of this PT was to provide information to the participating laboratories to help them to identify problems in the analytical procedures being used or/and to increase the confidence in the analytical results produced

3 – Methodology

Sample preparation - Known amounts of diazinon (0.1994 mg.kg^{-1}), parathion-methyl (0.0994 mg.kg^{-1}), ethion (0.3206 mg.kg^{-1}) and carbaryl (0.0281 mg.kg^{-1}), as methanol solutions, were added to 2 kg of a puree prepared from papaya samples bought in the city of Rio de Janeiro. Once fully homogeneized, aliquots of the sample material was transferred to glass jars and placed in the freezer until they were sent (frozen) to the participating laboratories. Only Dr. Ehrenstorfer (FRG) certified standards were used. Eleven laboratories participated in this study.

Homogeneity and stability tests – The homogeneity (five samples) and the stability of the samples (seven aliquots, seven weeks) were tested according the ISO GUIDE 35. To the homogeneity test analysis of variance (ANOVA) was used and for the stability the ANOVA was used in conjunction with residue analysis.

4- Reference values

Reference values for the concentrations of pesticides in the sample were assigned after analyzing statistically the analytical results supplied by the pesticide residues laboratories of the Food and Consumer Product Safety Authority (VWA/KvW, NE) and of the Fiocruz/INCQS for the reference material . The medium of these results were used as the reference values (Table 2).

Pesticide	Reference Value (mg/kg)
Carbaryl	0.029
Ethion	0.090
Diazinon	0.188
Parathion-methyl	0.103

5 - Laboratory performance criteria

The laboratory performance criteria was evaluated according to the ABNT ISO/IEC Guia 43-1. The parameter for evaluation of the performance of an individual laboratory was the z-score

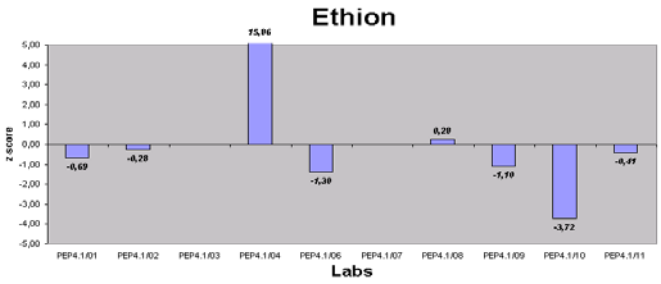
Equation 1:

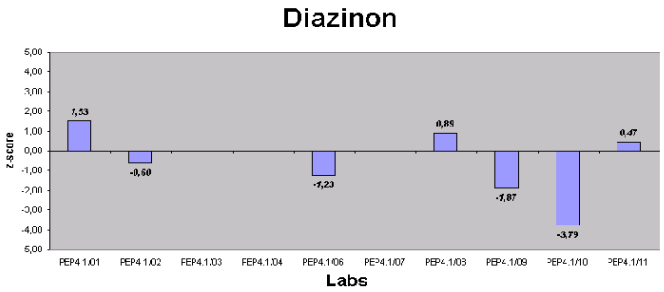
$$z_i = \frac{y_i - y_{ref}}{y_{ref}\,CV} \quad (1)$$

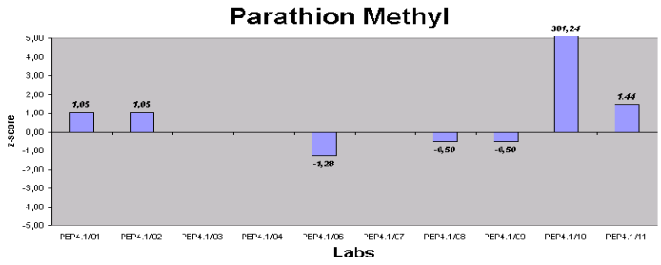
z_i: z-score
Y_i: result of the laboratory i
Y_{ref}: reference value
CV: coefficient of variation = 25%

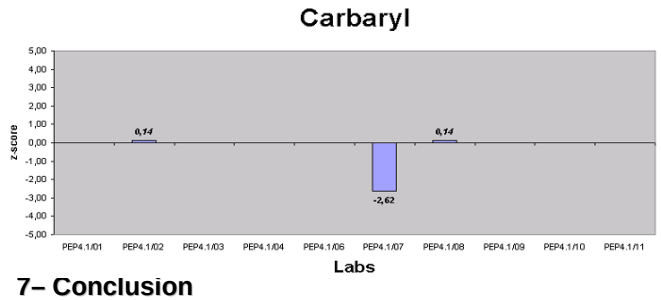
Criterion for acceptability of performance:
$|Z| \leq 2 \rightarrow$ acceptable
$2 < |Z| < 3 \rightarrow$ questionable
$|Z| \geq 3 \rightarrow$ unacceptable

6 - Results









7– Conclusion

The results of this proficiency study indicated that efforts are needed to increase the quality of the measurements of pesticide residues, in papaya, performed by some of the participating laboratories. Problems were found particularly in which it concerns the qualitative identification of the spiked pesticides, as 9.9% of the reported results were false positive and 21.8% false negatives.

8 – Acknowledgments

FINEP FINANCIADORA DE ESTUDOS E PROJETOS MINISTÉRIO DA CIÊNCIA E TECNOLOGIA
voedsel en waren autoriteit
FAPERJ
United Nations Educational, Scientific and Cultural Organization