SAG
Ministerio de Agricultura

Gobierno de Chile

THIRD REGIONAL INTERLABORATORY STUDY FOR DETERMINATION RESIDUES OF PESTICIDES IN APPLE, RESULT AND CONCLUSIONS

35 ARCAL

Aplicaciones nucleares para una REGIÓN sustentable

RALACA

INTRODUCTION

Interlaboratory tests are an important tool to evaluate the competence of analysis laboratories. In this intercomparison assay, 17 laboratories participated: ten from Chile and seven of other countries of Latin America.

The Test material was prepared from Apple Puree, which was processed in two batch: one for the spike sample and the other for the white sample.

The pesticides contained in the test material were added to the spike sample, and the subsamples were stored at -20 ° C until dispatched. From a list of 64 pesticides, the test material contained only 10 pesticides, delivering to each participant material sample homogenized Apple Spike (amount: 50 grams) and sample material homogenized of White Apple (amount: 50 grams).

Test of homogeneity and stability were found conform, according to the requirements of the test.

PROCEDURE

The Test material was prepared from apples obtained from an organic garden under official registration sample. The pesticides used in the test correspond to Sigma-Aldrich certified standards.

From a list of 64 pesticides, the test material contained: azoxystrobin, bifenthrin, bromacil, bromopropylate, chlorfenvinphos, chlorpyrifos ethyl, fenchlorphos, parathion methyl, pendimethanil and trifloxystrobin.

The test material was tested for homogeneity and stability.

STATISTICAL EVALUATION

Interlaboratory study was evaluated according to: ISO 13528:2005 Statistical methods for use in proficiency testing by interlaboratory comparisons and references.

The results reported by the participating laboratories were statistically analyzed in order to obtain the assigned value (Xa) of each pesticide. The assigned value was used in combination with the standard deviation of the Homogeneity test (σ_p), for the calculation of the Z-score of each reported result.

The assigned Xa value for each analyte was calculated from the consensus of the results reported by the participating laboratories.

STANDARD DEVIATION OF THE ASSAY (σ_p)

The value considered for the standard deviation of the assay (σ_p) for each analyte was obtained from the homogeneity test. The σ_P was derived from the Horwitz/Thompson equation.

The z-score of the participants for each pesticide was calculated according to:

Z-score= $(X – Xa)/\sigma$ (X = Result reported by the participants (ug/Kg), Xa = Assigned Value (ug/Kg)

σ = Standard Deviation of Homogeneity)

Calculation of uncertainty U

Uncertainty was calculated for each pesticide from the CV% variation coefficient of the Homogeneity test accuracy and the coverage factor for a 95% confidence interval.

RESULTS

The concentration of pesticide residues present in the test material was determined in ug/Kg, without correction for recovery, together with the information of % recovery, limit of quantification (LoQ), in addition to the information of pesticides not analyzed and/or not detected and the additional pesticides reported to the test sample (false positive).

TEST OF HOMOGENEITY AND DETERMINATION OF UNCERTAINTY

For the homogeneity test, the result are conforms, except for Bifentrin it did not show enough homogeneity, for which the results will be only referential.

Analyte	Nº Datos, n	Assigned value xa ug/kg	uncertainty U	Standard Deviation of Homogeneity
Azoxistrobina	18	42,7	15,8	Horwitz 8,49
Bromacilo	19	39,3	13	Horwitz 7,3
Bromopropilato	19	30,4	20,4	Horwitz 5,91
Clorfenvifos	19	33,6	17,2	Horwitz 6,53
Clorpirifos Etil	19	20,2	18	Horwitz 3,02
Fenclorfos	19	32,6	19,8	Horwitz 7,05
Paration Metil	19	22,7	21,8	Horwitz 4,91
Pendimetanil	19	26,6	21,8	Horwitz 4,39
Trifloxistrobina	19	34,8	19,2	Horwitz 7,10
Bifentrin	17		23,6	Horwitz 7,64

RESULT FOR PESTICIDE:
Figures: z score of the participating laboratories for analyte
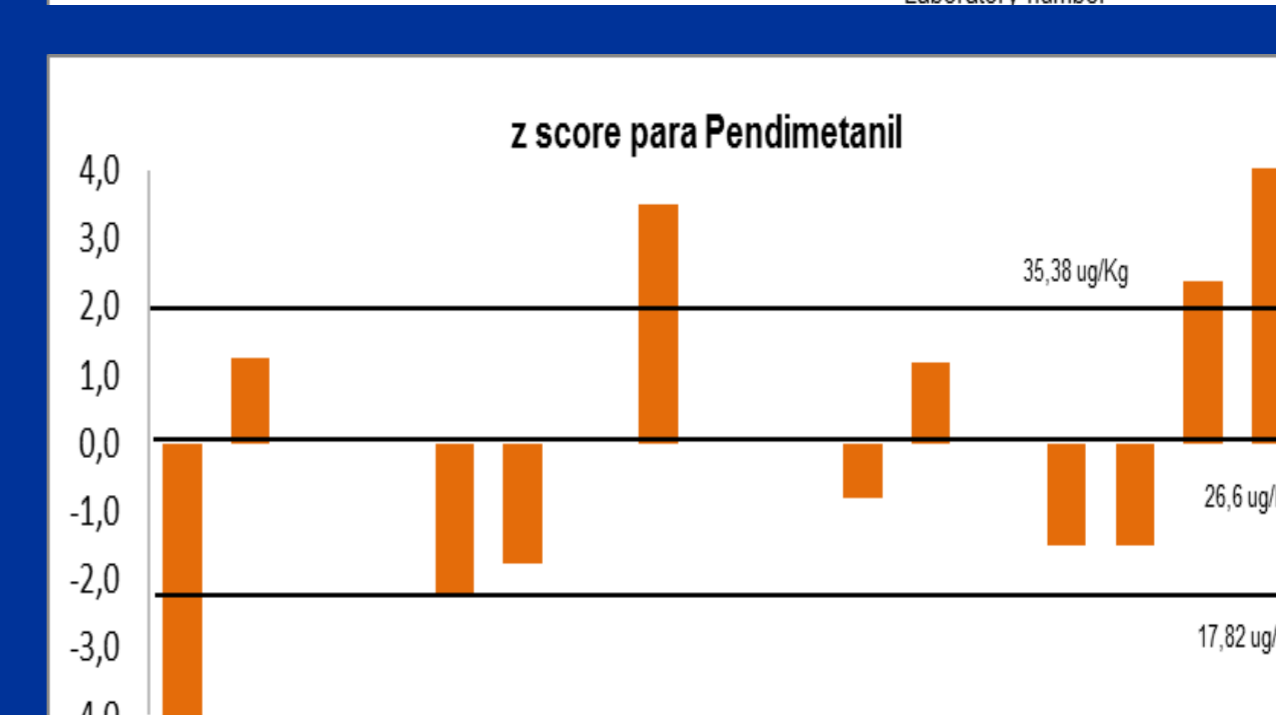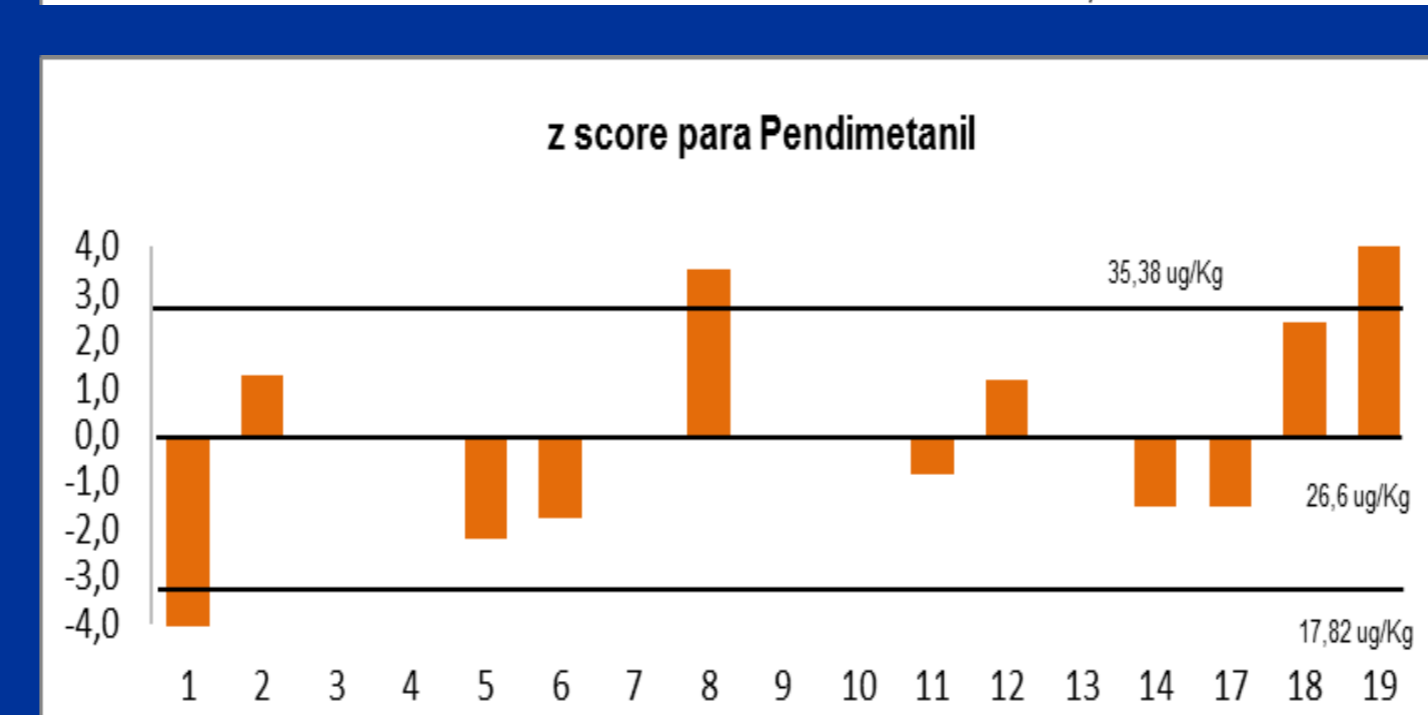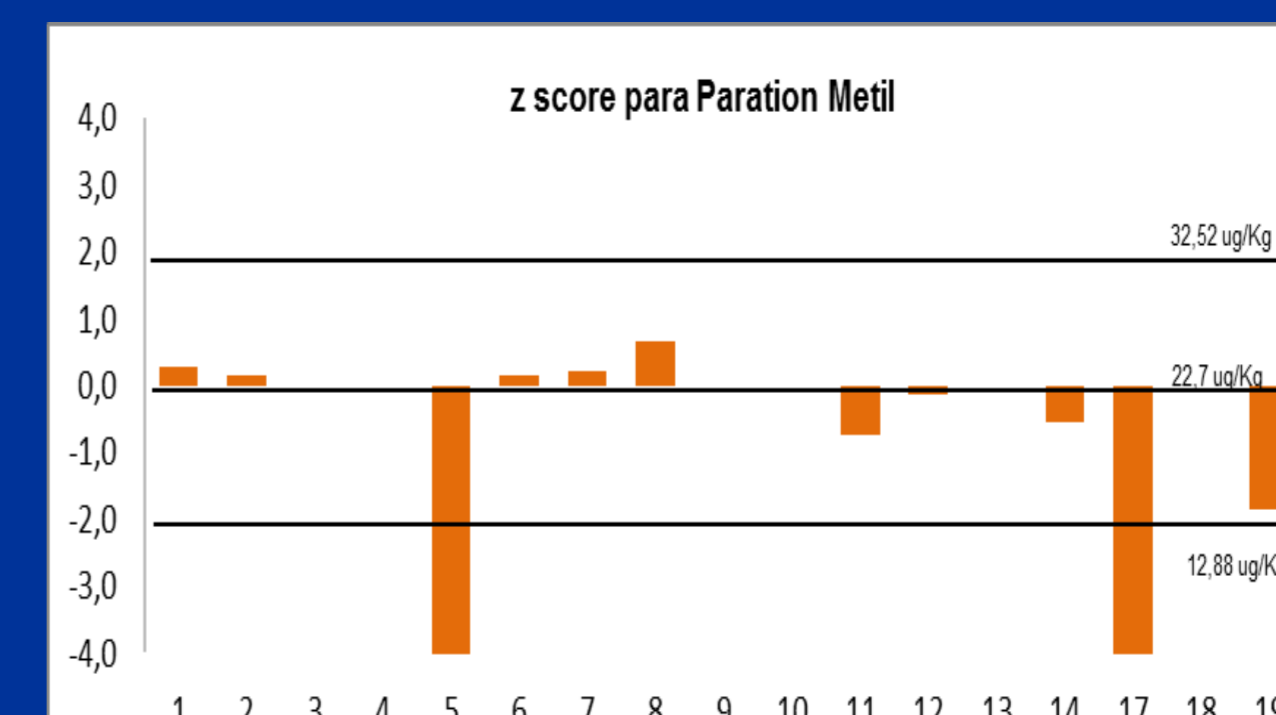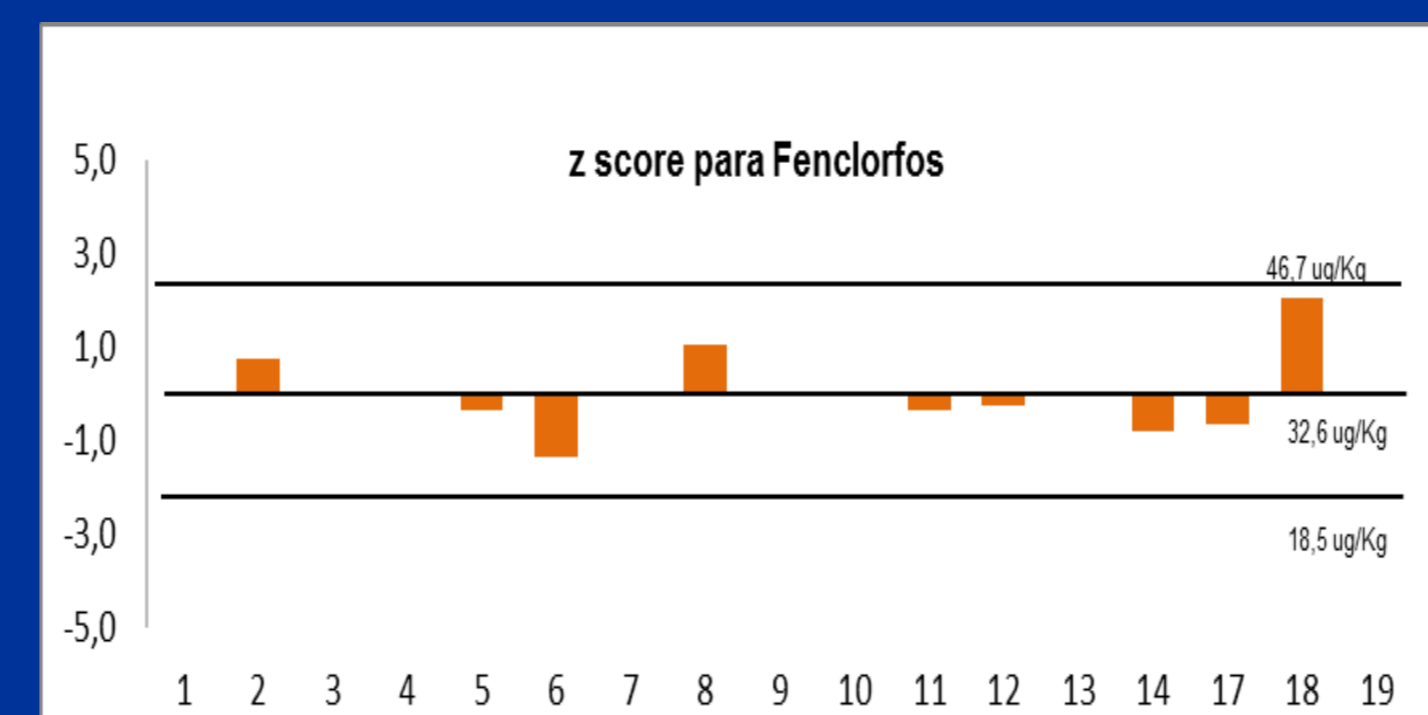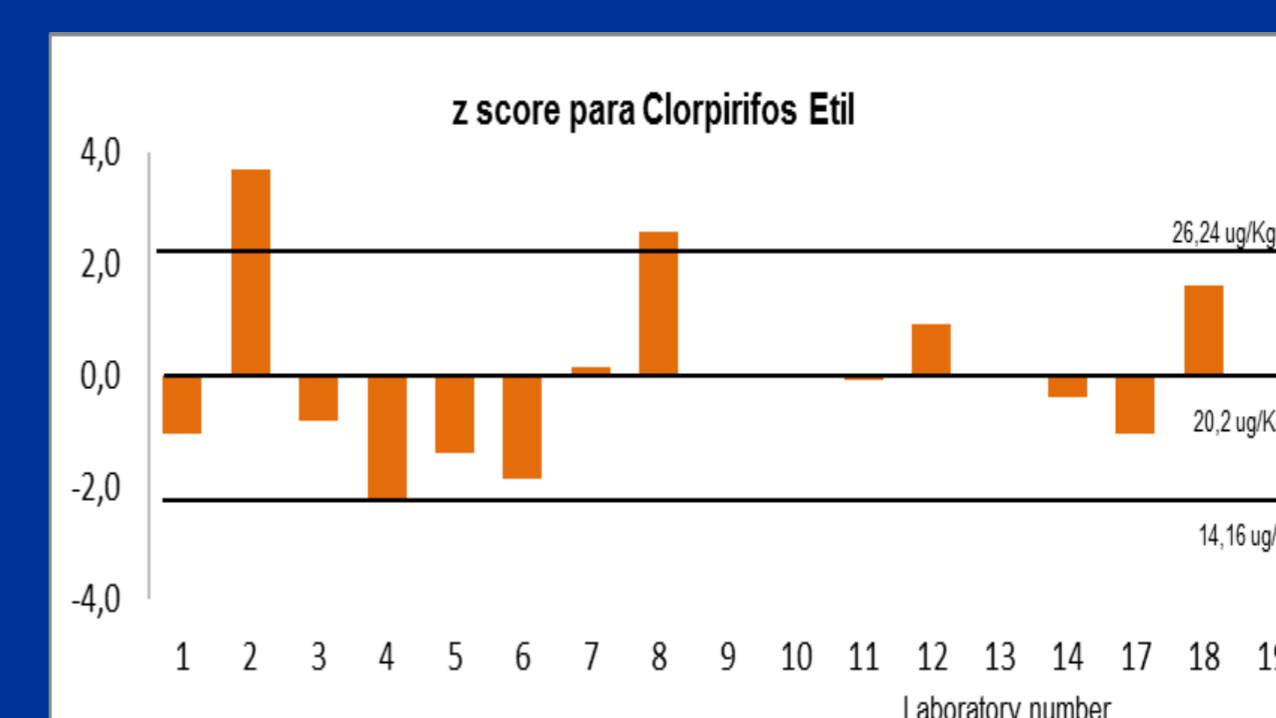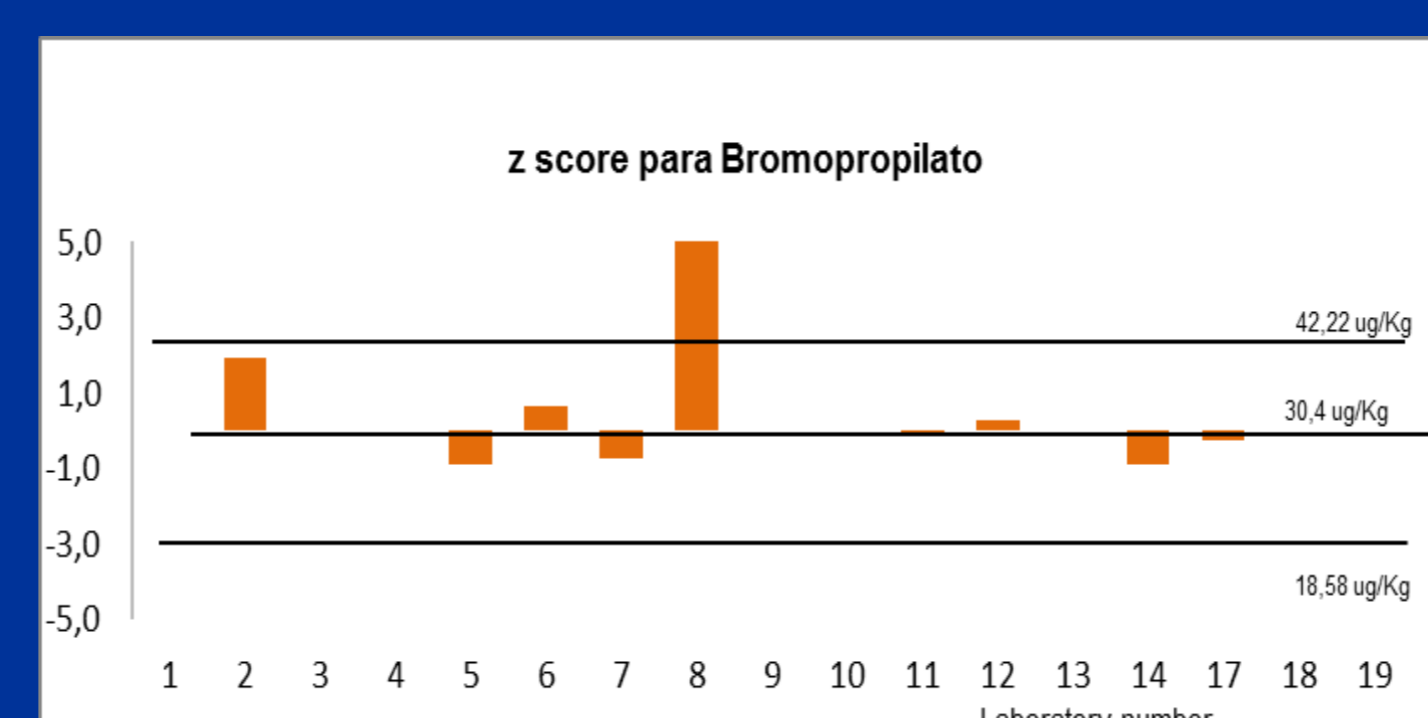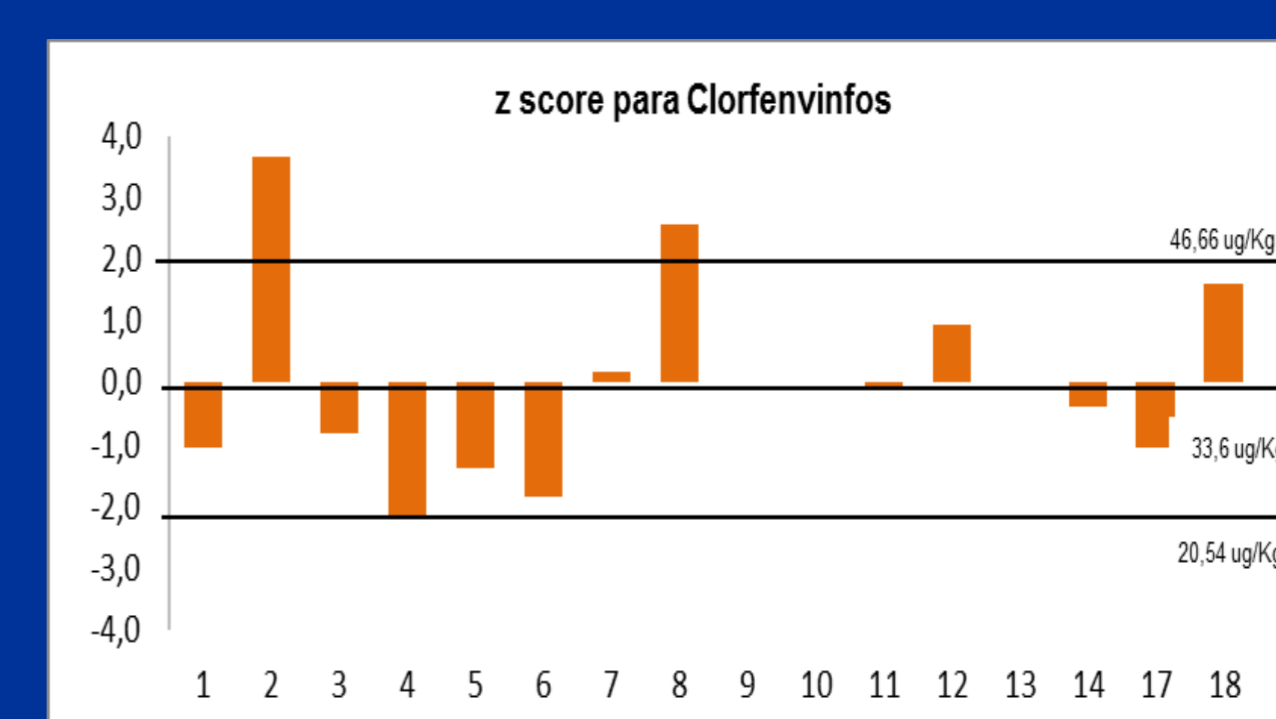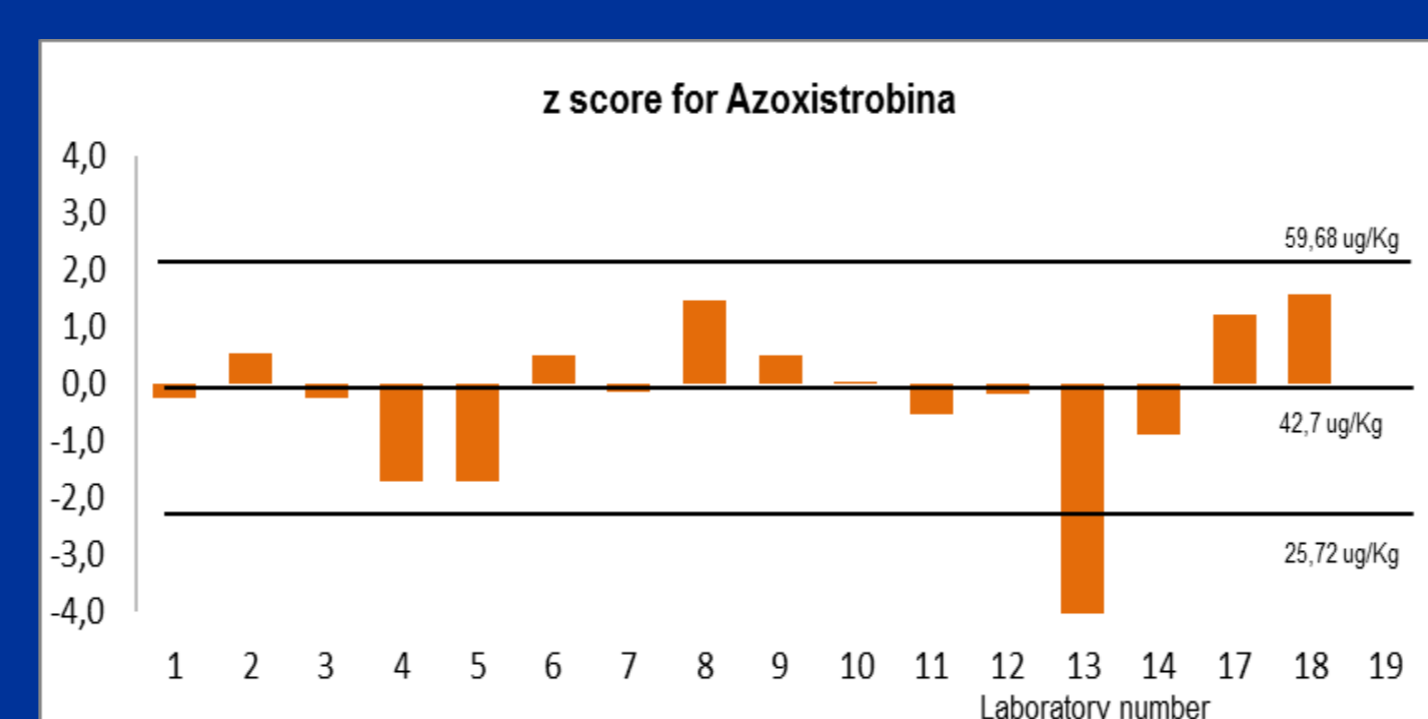
CONCLUSION:

- Of the 136 determinations, the laboratories analyzed 70%, an adequate number for this type of MR analysis.
- Most of the laboratories had results conforming to the defined z score, of the 95 reported results, 14 are on the Z score greater than I2I, which represents 15%.
- Laboratory No. 8 concentrates 38% (5/14) of nonconforming results, z score> I2I, the remaining 9 are divided among the other laboratories.
- Excellent response from the laboratories invited to participate in the trial, with a 100% response, , which demonstrates the interest of laboratories in demonstrating their competence through this type of tests.

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- CEIMIC Chile S.A.
- Laboratorio de Servicios Avanzados, Labser Chile.
- Agriquem América Chile S.A.
- Comercial Analab Chile S.A.
- Eurofins GCL Chile.
- Corthorn Quality Chile S.A.
- Bureau Veritas Chile S.A.
- Laboratorio de Residuos de Plaguicidas y Medio Ambiente, Instituto de Investigaciones.
- Agropecuarias (INIA), La Platina, Chile.
- Laboratorio Nacional de Salud, Ministerio de Salud Pública y Asistencia Social, Guatemala.
- Laboratorio de Análisis de Plaguicidas, Centro de Investigación en Contaminación Ambiental (CICA), San José, Costa Rica.
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