PTR 018

INTERLABORATORY CONTROL AMONG INCO – DEV MYCOTOX PROJEC LABORATORIES

Vargas, E.A.; Castro, L.; dos Santos, E. A.; Franca, R.C.A.; Cea, J.M.; Moriyama, C.; Vega, M.H.; Freitas-Silva, O and Souza, M.L.M.

Ministerio da Agricultura, Pecuária e Abastecimento – Laboratorio Nacional AGropecuário em Minas Gerais- MG, Brasil. E –mail: gena@cdlnet.com.br

The Work Package 1 " Development and standardization of effective analytical tools for mycotoxin (aflatoxins B_1, B_2 G_1, G_2 ochratoxin A, zearalenone, fumonisin B_1, B_2 and tricothecenes) determination in wheat and maize" aim to implement the interlaboratory control between the partners laboratories from Brazil, Uruguay, Chile and Argentina as part of the objectives of INCO-DEV MYCOTOX PROJECT 2003-2005 "The Development of a Food Quality Management System for the Control of Mycotoxins in cereal Production and Processing Chains in Latin America South Cone Countries". The ojectives of the interlaboratory control were: evaluate the performance of the laboratories and the main difficulties encountered in performing the analytical procedure for mycotoxins determination in maize and wheat; contribute to the harmonization of analytical procedures of the partners laboratories and contribute to the laboratory's proficiency in mycotoxin analysis. Maize reference materials for aflatoxins and zearealenone were prepared and used to the implementation of the interlaboratory control. In summary, the preparation of these samples involved: milling (<20 mesh), homogeneization, analysis to verify the homogeneity of the bulk material and packing (labelled vacuum "sachets" or plastic bottles) and mycotoxin analysis. The homogeneity of the material was investigated by the analysis of variance – ANOVA- according to International Harmonized Protocol for the Proficiency testing of (Chemical)Analytical Laboratories as established by ISO 43-1 – Annex at 95% of confidence level by calculating an F-statistic ans S_s/σ (σ =15%). All batches of test material were stored under – 18ºC and protected from light prior to and after packaging. Aflatoxins in the test materials were determinated by immunoaffinity with liquid chromatography (LC) with pos-column derivatization and thin layer chromatography (TLC). Zearalenone in the test materials were determined by solid phase column (Romer 224^{TM}) with LC. Four homogeneus maize materials were prepared: blank for zearalenone, blank for aflatoxins and two naturally contaminated for aflatoxins. These samples were used to validated analytical methods and as reference samples for proficiency test. The participating laboratories received refrigerated parcel containing: coded maize samples and blank for spiking purpose, test material receipt form, additional instructions, results reporting sheets and analytical work questionnaire in 3 rounds. The results were evaluated by using z-score function being calculated considering the best value representing the true measure of mycotoxin in the sample (as per evaluation in the homogeneity tests). Additional FAPAS test material for mycotoxins were purchased in order to assess the laboratory performance and to validated the reference materials. In case of "questionable" or "unsatisfactory" results, the Laboratory were advised to treate them as non conforming work, make the necessary modifications and adjustments in the methods, taking into account the method

performance criteria (CEN). The laboratories were strongly recommended to write a report containing the analysis of the causes and correctives actions proposed, giving special attention to: correct use of calibrated pipettes; chromatographic condition including the calibration curve and injected volume of extract and standard.

Authors would like to thanks European Commission to financial support to MICOTOX Project (contract ICA4-CT-2002-10043, INCO-DEV program)