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Report: Analysis of fosetyl-Aluminium residues in Peanut grain samples

Made by:

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For the realization of this report, the international peanuts trade situation was analyzed, in relation to the presence of residues of fosetyl-Al fungicide.

After consulting the regulations that is nowadays current in Argentina, SENASA (National Health Service and Food Quality) Res.934/2010, Imr_por_activo_y_cultivo_may_2015.xls, which shows that the use of fosetyl-AI is forbidden in peanut cultivation in Argentina and after studying the national and international literature about the presence of this pesticide residue, the following tasks were carried out:

- To determine fosetyl aluminum residues in 6 peanut grain samples detailed in Table 1.
- To settle down field a experiment using the different phosphites commercial products from Argentina and listed in Table 2.
- To determine if phosphites residues are confused with fosetyl residues in the Chromatographic analysis, in peanut grain samples.

Table 1: Samples of peanut kernels (if problem) - General Deheza Cordoba, analyzed to determine the presence of fosetyl-at Laboratory of INTA Castelar ITA chemical contaminants:

Reception Date: February 15, 2016

| INTA Laboratory ID. | | |
|---------------------|---|-----------|
| R004 | Peanut blanched-Lote BL 21056-caliber 40/50 | 11/8/2015 |
| R005 | Peanut blanched-Lote BL 21057-caliber 40/50 | 2/9/2015 |
| R006 | Peanut grain-Lote HPS 26710-caliber 40/50 | 24/6/2015 |
| R007 | Peanut grain -Lote HPS 26724-caliber 40/50 | 30/7/2015 |
| R008 | Peanut grain -Lote HPS 26712-caliber 40/50 | 28/7/2015 |
| R009 | Peanut grain -Lote HPS 41171-caliber 60/70 | 26/8/2015 |

Table 2: Samples of peanut kernels from the field experiment done with phosphites comertial productsused in peanut crop in Argentina.Reception Date: May 10, 2016

| INTA Laboratory ID. | Commercial products name | |
|---------------------|--|--|
| R111 | FULLTEC FLECHA (Ca, B phosphite) | |
| R112 | FULLTEC (K phosphite) | |
| R113 | FULLTEC TOP (Na phosphite) | |
| R114 | FULLTEC ULTRA (Mg phosphite) | |
| R115 | EUROFIT MAX (Fe, Mn, Zn phosphites) | |
| R116 | FULLTEC COBO (B, Cu phosphites) | |
| R117 | FULLTEC MAIS (N, P, B, Co, Mo, Zn phosphites) | |
| R287 | MEZCLA DE : R111- R112- R113- R114- R115- R116- R117 | |





The following methodology was used In the "Chemical contaminants Laboratory" at the Food technology Institute of INTA Castelar

- Equipment used AQUITY SQD UPLC / MS (Waters). Working with positive and negative electrospray ionization. Data acquisition was performed in SCAN.- mode
- Mobile phase: water gradient 0.01% formic acid and methanol 0.01% acid fórmico.-
- Column: Hypercarb 2.1 x 100 mm; CU5 (Thermo Scientific) .-
- Injection volume 10uL.-
- Fosetyl-Al standard solution in methanol / acetonitrile analyzed. The standard used was: ChemServiceN-12019-100MG.
- Aqueous solutions of the following commercial products were analyzed: EUROFIT MAX; FULLTEC Arrow; FULLTEC Cube; FULLTEC; Ultra FULLTEC; Top FULLTEC; FULLTEC Mais.-

Results: Fosetyl aluminum or/and phosphites residues cannot be identified in the materials tested.

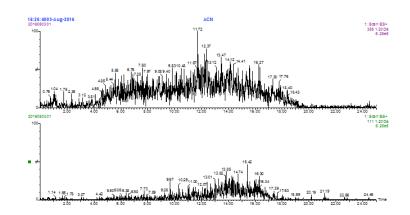
Following are the Chromatograms obtained from *Chemical contaminants Laboratory*" at the Food technology Institute of INTA Castelar:

Figures (F1 to F3) correspond to different injections performed varying the detector electronics to evidence fosetyl aluminum (MW: 354.1). The solution used was 10 ppm in acetonitrile.

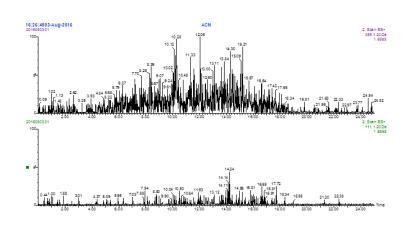
The same conditions were repeated electronic with acetonitrile for analysis by comparison seeking to find a corresponding signal to fosetyl-aluminum.

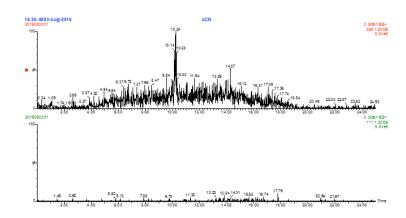
Modo ESI+

ACN

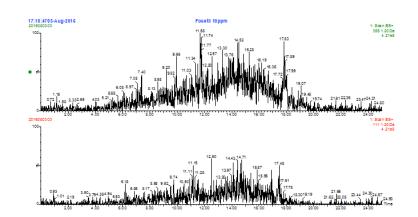






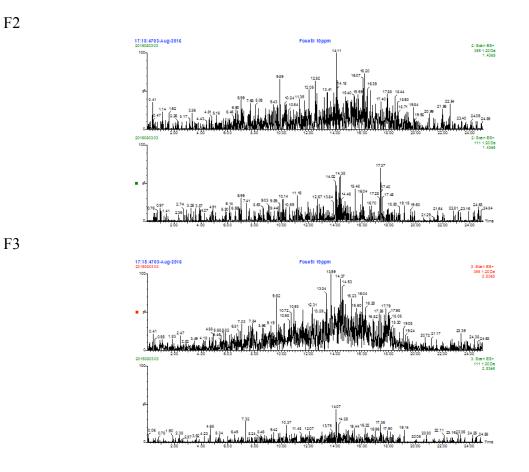






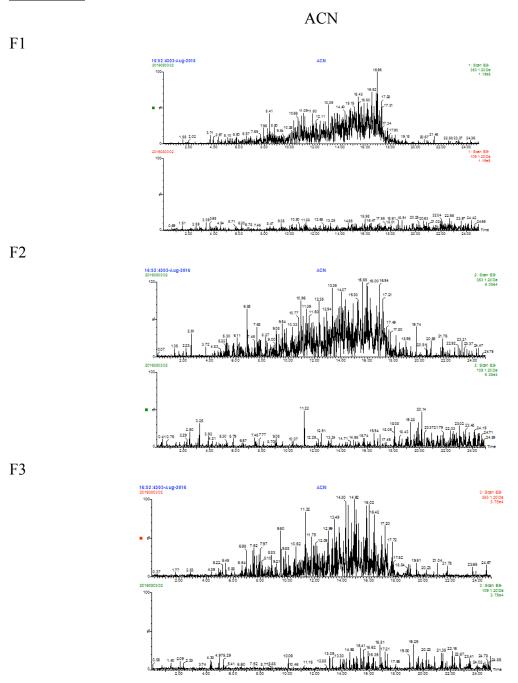






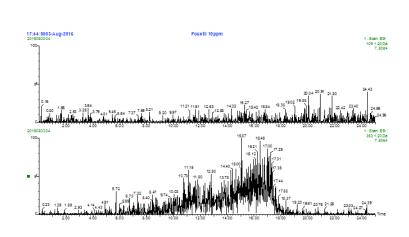


Modo ESI -



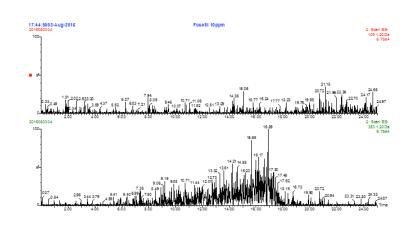


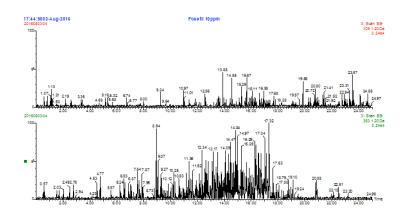
Fosetyl-Al



F2

F1









After these results, the samples R004, R005, R006, R007, R008, R009, R116, R287 were sent to JLA

Laboratory in Córdoba Argentina, accredited under ISO 17025 standardization method and

authorized by SENASA (National Health Service and Food Quality).

Fosetyl aluminum was NOT detected in any of the samples following the method developed and validated by this laboratory (JLA) and the Detection Limit used was of 0.1 mg / kg sample. Reports of the results obtained by JLA Laboratory, are attached.

The laboratory report from Germany, Europe is also attached. It would be important to find out what kind of certification and international accreditation has this laboratory and which technique has being used to determine fosetyl aluminum. Note that in the results from a laboratory cited in Germany, there has not been specified which technique they used for determination of fosetyl aluminum. Beyond the international laboratory's accreditation, it also has to be certified the fosetyl-Al technique for determining it in peanuts.

Conclusions:

In all samples analyzed in both laboratories being the INTA Castelar and JLA cited in Argentina, fosetyl-Al residues were NOT detected.