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Letter to the editor

Dear Dr. Birch,

I am writing with reference to the article "Analysis of some breakfast cereals on the French market for their contents of ochratoxin A, citrinin and fumonisin B1: development of a method for simultaneous extraction of ochratoxin A and citrinin by A" by Molinié et al., to be published in "Food Chemistry" volume 92, Issue 3, September 2005, pages 391–400, which is available on the website of sciencedirect.com as article in press.

In the manuscript, the authors analyze the content of ochratoxin A (OTA) and other mycotoxins in coreals on the market in France. The concentrations of OTA determined by HPLC-FLD are concluded by the authors to be confirmed by LC–MS/MS results presented in Table 3. Apparently, these analyses were performed in our laboratory at the Institute of Toxicology of the University of Würzburg, Germany. My student Herbert Zepnik, given as affiliated with the University of Würzburg (but not with the Institut für Pharmakologie und Toxikologie, where he performed the work for his PhD thesis), is acknowledged for his assistance with LC–MS/MS in the manuscript. The method (except for some minor, but important mistakes made in the description of aquisition parameters) and the instrument described is identical to the LC–MS/MS available in this department (the only instrument of this type at the University of Würzburg). V. Faucet, one of the authors, spent time in our laboratory, at the end of 2002, to analyze samples from incubations of OTA with pig kidney microsomes to study OTA-biotransformation. During the visit, V. Faucet has asked us if she might analyze somes samples of cereal to confirm the presence of OTA. This was permitted since we had a well-evaluated method for analyzing OTA in biological samples; however, we were never informed that the data recorded were intended to quantify OTA in these samples for the purpose of publication and we have not been asked to comment on quality and possible problems with the data.

After reading the manuscript, we compared the concentrations given in Table 3 of the manuscript with the raw data in our system on the basis of sample names identical to those used in the publication (cereal 12 etc.) with a recording date of December 12, 2002 and another dataset recorded on November 27, 2002.

In our opinion, the manuscript by Molinié et al, which now describes these data, shows a number of major problems:

- The method description is (except for some very minor, but important omissions of the second mass transition monitored) taken, word by word, from one of our publications (Zepnik et al. (2003). *Toxicology and Applied Pharmacology, 192*, 36–44) without citation of this publication. By the accepted ethical standards in scientific publising, the authors were obliged to cite the original publication and make a clear reference to the use of the published method and the location where in the samples were analyzed.
- The results of the LC–MS/MS analysis were included in the publication without requesting permission from us, despite the use of our instrument, our methodology, and our facilities. We were also not asked to comment on the quality of the data or the conclusions. This is again not acceptable by the standards of scientific publishing since we reserve the right to check all data generated and released from our laboratory for accuracy and correctness. Herbert Zepnik, whose assistance is acknowledged in the manuscript, does not recall having made quantitations based on the raw data and was also not informed of an intended publication.
- Based on the raw data available to us (the instrument is operated in line with requirements of, "Good Laboratory Practice") and the description of sample workup in the manuscript, we cannot confirm the OTA-concentrations given in Table 3 of the publication. Based on the chromatograms of the two transitions monitored during analysis for ochratoxin A, the concentrations of ochratoxin A can only be determined by integration in two of the 10 samples with the confidence required for a quantitation. We are unable to evaluate the other sample analyzed, due to unresolved peaks, incorrect retention times, or absence of coelution of the qualifier MRM (m/z 402/358) recorded with the quantifier MRM (m/z 402/167) for OTA (for an example chromatogram, see Fig. 1). The instrument was working according to specifications, as indicated by the performance with calibration samples included in the series of analysis between samples (for an example, see

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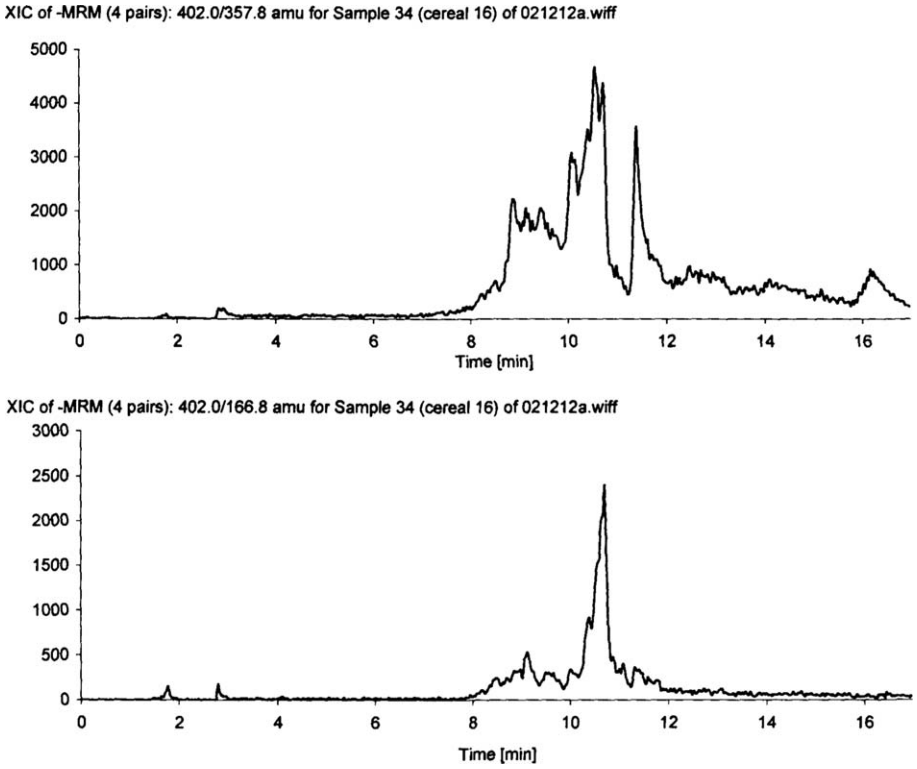

Fig. 1. Example of chromatogram cereal #16 given in Table 3 of Molinie et al. using two characteristic fragments. The transition from m/z 402 to m/z 358 was intended to be used by the authors as quantifier and transition m/z 402 to m/z 167 served as additional qualifier.

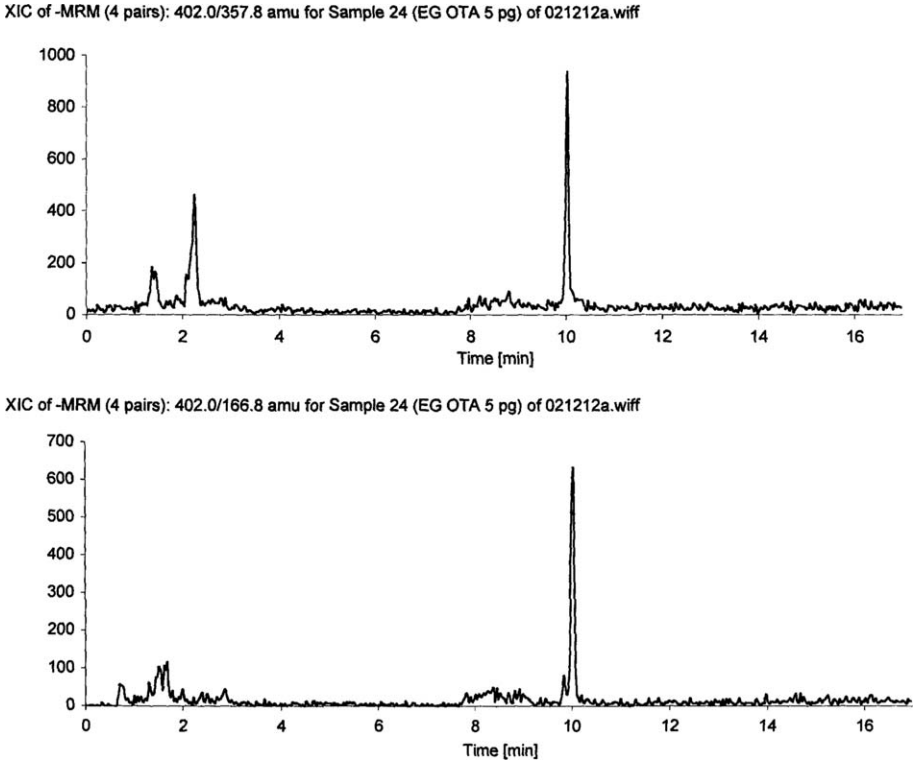

Fig. 2. Example of a separation of OTA standards included within the samples of cereals analyzed by Molinie et al. Total content of OTA was 5 pg on column; retention time varied less than 3 s within repetitive analyses of calibration standards.

Fig. 2). The calibration samples and all the biological samples analyzed by us from our experiments show a clear resolution of the OTA-peak and excellent peak shapes. We assume that the poor chromatography of the foreign samples, with multiple unresolved peak, is the consequence of sample preparation-using an antibody, and dissolving OTA in water for final LC-analysis. We cannot understand how ochratoxin A-content could be calculated from unresolved peaks with incorrect retention times or how absence of coelution of the qualifier MRM with the quantifier MRM can be translated into quantitative results as reported in Table 3 of the manuscript.

As already communicated by e-mail in October and November of 2004, we herewith formally inform you about: (i) the unauthorized use of data generated in our laboratory by the authors Molinié et al., (ii) the use of identical text passages, as in Zepnik et al., by Molinié et al. for description of the LC/MS–MS analysis without citation of the publication, and (iii) the apparent discrepancy between published data and raw data.

We clearly do not confirm the numbers given in Table 3 of the publication by Molinié et al. and we dissociate ourselves from any conclusion based on these numbers.

Sincerely,

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