Ochratoxin A in Coffee, Tea and Beer

H. BRESCH*, Marianne URBANEK and Karla HELL

Federal Research Centre for Nutrition, Institute of Hygiene and Toxicology, Karlsruhe, Germany

Introduction

Several investigations of the last ten years have shown that ochratoxin A (OTA) is frequently present in coffee and beer. Regularly consumed by many, these beverages can be assumed to contribute considerably to the total individual toxin load. In a research project evaluating the ochratoxin status of the German population, commercial coffee (357), tea (161) and beer samples (318) were analyzed for the toxin in the past 3 years, besides other food investigated by colleagues also participating in the collaborative study.

Materials and methods

Coffee, tea and beer samples purchased in retail shops in different regions of the country at different times of the year were stored at 10 °C until analysis. Of one type of produce, 15 samples at least were analyzed, provided no toxin was detected; in case of one positive result, 30 samples at least were analyzed. It was made sure that samples from one and the same company differed in batch numbers.

Chemicals of analytical grade or HPLC quality needed for analysis were obtained from Merck (Darmstadt, Germany), Ochratoxin A from Sigma (St.Louis, MO, USA), phosphate buffered saline tablets, pH 7.3, from Biocode (Coring, Gernsheim, Germany), filters FP 030/2 from Schleicher & Schuell (Dassel, Germany), immunoaffinity columns from Coring and Vicam (Watertown, MASS, USA). For HPLC analysis, a 250 x 4 mm column of Macherey-Nagel (Düren, Germany) filled with Nucleosil 100-5 C18 was used in combination with a 30 mm precolumn (same company) and filled with the same adsorbent.

Equipment: Centrifuge (Hettich Universal 30 RF [Tuttlingen, Germany]), solid phase extraction unit (Baker Vacuum manifold 12 G System [Phillipsburg, NJ, USA]), Metalblock-Thermostat (Thermolyne Modular Dri-Bath [Dubuque, IA, USA]), ultrasonic bath (Bandelein Sonorex RK 1028 H [Berlin, Germany]).

HPLC equipment consisted of a Pharmacia LKB 2150 pump (Freiburg, Germany), autosampler SIL 9A (Shimadzu, Düsseldorf, Germany), column thermostat (VDS optilab [Berlin, Germany], fluorescence detector RF 551 and integrator C-R5A (both of Shimadzu). The mobile phase was degassed by passing the mixture through a glass frit into an Erlenmeyer flask kept under vacuum. To keep the mobile phase degassed, a degasser (VDS optilab) had been installed between pump and autosampler.

The flow rate was 1 ml/min., the temperature of the column 20 °C; 20 µl of prepared samples were injected. Excitation wavelength was 330 nm, emission wavelength 460 nm. Samples of final volume were analyzed in duplicate and the mean was taken for calculations. The difference between the two data was less than 5 %. A standard solution was injected at least twice a day.

The HPLC system was calibrated by different concentrations of OTA dissolved in methanol. The concentration of the stock solution was determined spectrophotometrically (ε_{331} : 6640 cm^{-1} M^{-1}). Mobile phase: Acetonitrile/water/glacial acetic acid 45/55/1. OTA was eluted after 15.2 min.

For confirmation purposes OTA was derivatized according to ZIMMERLI and DICK (1995): An aliquot of the final sample solution taken for HPLC analysis was evaporated to dryness and 2.5 ml methanol + 100 µl of concentrated HCl were added. The mixture was kept overnight at 30 °C. After evaporation of the solvent, the residue was taken up into the mobile phase consisting of acetonitrile/water/acetic acid 70/30/0.5. OTA methylester was eluted after 6.5 min.

Determination of recovery rates: The content of a 500 g bag of each coffee type was manually mixed and 10 subsamples of 25 g (10 g in case of instant coffee) were taken from each bag. Five samples were left untreated and five spiked with OTA dissolved in methanol (0.159 and 0.130 µg/ml, resp). Spiking was carried out by dropping the solution, by means of a Hamilton syringe, onto different parts of the coffee powder which had been filled into coffee filters; care was taken not to contaminate the filter. The powder was left overnight at 45 °C before it was gently remixed and analyzed. Tea samples were spiked by the same method. To spike beer samples, OTA solutions were added to 250 ml samples, then the procedure was continued as described below.

Extraction of OTA from coffee

The toxin was extracted essentially according to STUDER-ROHR et al. (1995). The total content of a 500 g bag, in a 3 l glass flask, was mixed by hand for 5 minutes. Twenty five g were weighed into household paper filters and 500 ml of boiling distilled water added. After cooling of the filtrate, 5 g $NaHCO_3$ were added and the volume was readjusted to 500 ml with water. Ten ml were filtered through a 0.45 µm cellulose acetate filter, 5 ml were diluted with 5 ml PBS solution and passed through an immunoaffinity column at a maximum rate of 1 ml/min. The column was washed with 20 ml of distilled water (HPLC quality) and air dried by nitrogen. The toxin was slowly eluted with 6 ml of methanol/glacial acetic acid (98/2). The eluate was collected in 20 ml glass flasks, evaporated at 45 °C and the residue taken up in 500 µl of the mobile phase; 100 µl were transferred into a 500 µl vial adapted to the autosampler.

Extraction of ochratoxin from tea

Tea was extracted in boiling water. If tea samples were contained in small bags, the bags were opened and the contents mixed. Twenty five g of tea were extracted in 500 ml distilled water. As the pH of some teas has been found to be low, it was adjusted to 7.3 with NaOH before the tea was filtrated through a 0.45 µm filter. The procedure was continued as described for coffee.

Extraction of ochratoxin from beer

The toxin was extracted according to SCOTT and KANHERE (1995). About 200 ml of beer were degassed in an ultrasonic bath. One ml of a solution (a. dest. containing 15 % NaCl and 2 % $NaHCO_3$) were added to 5 ml of the degassed beer and the mixture was passed through an immunoaffinity column. The column was washed with 10 ml of a solution containing NaCl (2.5 %) and $NaHCO_3$ (0.5 %) in HPLC water, and subsequently with 10 ml of HPLC water. The toxin was eluted with 6 ml methanol. Then the procedure was continued as described for coffee.

Results and Discussion

Coffee

Roasted coffee is sold in retail shops either as beans or ground in vacuum packages. Since STUDER-ROHR et al. (1995) have shown that the mycotoxin may be distributed very inhomogeneously in raw coffee, ground coffee samples were tested exclusively. It is reasonable to assume that due to the different mixing procedures during the production of coffee powder the toxin is more homogeneously distributed in ground coffee which German consumers prefer anyhow, by the way. Samples of the brands of the five leading manufacturers in the German coffee market had predominantly been taken.

OTA in food matrices is usually analyzed by extraction with organic solvents. This procedure was not applied here as the coffee should be extracted in a household manner to get informed on the amount of OTA taken up by the consumer. It was intended, furthermore, to compare the results obtained in the present study to those published by several authors who had applied immunoaffinity columns in the extraction procedure; the columns therefore were also applied here. In all samples tested, the detection limit of the toxin, calculated on the basis of a S/N ratio of 2, was 0.3 µg toxin/kg coffee powder. In calculations of the arithmetic mean, half the detection limit was assumed for samples not containing the toxin or contaminated by toxin concentrations below 0.3 µg toxin/kg.

Reproducibility of the results and recovery of ochratoxin were checked in all coffee types tested. Samples containing no toxin or concentrations below the detection limit were spiked with ochratoxin, then several aliquots were analyzed. More than 90 % of the toxin added were recovered. In view of the high recovery rates, results have not been corrected for recovery. Reproducibility was sufficient (variation coefficient below 10 %; see Table 1).

Another preliminary test was conducted to check whether the coffee powder contained in a package unit had been sufficiently mixed thus resulting in an equal distribution of the toxin. In principle, identical concentrations were found in six aliquots taken of a mixed 500 g sample of ground coffee verifying an equal distribution of ochratoxin in the sample (Table 2).

The total results of the study including all coffee types studied are shown in Table 3 indicating most relevant data, and in Fig.1 demonstrating the frequency pattern of OTA concentrations.

About half of the roasted coffee samples with or without caffeine contained OTA at concentrations above 0.3 µg/kg. Of 'mild' coffee, less samples contained toxin concentrations above the detection limit; the mean was lower, as was the 90. percentile. Of roasted coffee with caffeine 81 %, of decaffeinated coffee 79 %, and of the 'mild' type 93 % of samples contained toxin concentrations below 1 µg/kg powder (including all samples below the detection limit); 75 %, 67 % and 80 %, resp., of the samples contained less than 0.6 g toxin per kg. The highest amount of 6.3 µg/kg was detected in a sample with caffeine. In contrast to the roasted coffees, a high proportion of instant coffee samples was contaminated by considerably higher toxin concentrations (see table). Considering the fact that less instant powder than roasted coffee is regularly taken for a cup, the in-take of OTA per cup is not much different. Of the decaffeinated instant coffee samples, however, less were contaminated and toxin concentrations were lower than in those containing caffeine. Malt coffee, a coffee substitute made of barley, which was consumed mostly at war times when real coffee was rare, is still commercially available. As cereals, too, may be contaminated by OTA, it could be expected that the toxin is present also in malt coffee. The data show that it was present indeed but only in a relatively small number of samples.

The extent to which the same coffee types of different manufacturers differ in ochratoxin contents may be of interest to consumers. It has become obvious very soon in this study that roasted coffee samples of one of the leading manufacturers were free of detectable amounts of OTA while the remaining leading brands comprised contaminated products and toxin free ones. To confirm these early results it was decided to take more samples of this manufacturer than of the others. At the time the study was concluded, only two out of 36 samples of the leading manufacturer concerned have been found to contain measurable toxin concentrations. This shows that it is possible to continuously supply practically OTA free coffee over the test period of three years.

Table 1: **Recoveries of OTA in spiked coffee, tea and beer samples**

coffee type	OTA-added	no. of samples	amount detected mean	standard deviation	recovery range	% CV
	(µg/kg)		(µg/kg)	(µg/kg)	%	
coffee roasted ground	1.04	6	0.98	0.07	85–103	7.1
coffee roasted ground decaffeinated 1st test	2.08	5	1.97	0.17	88–108	8.6
coffee roasted ground decaffeinated 2nd test	1.04	5	1.03	0.09	88–108	8.7
coffee roasted ground mild	1.04	5	0.98	0.09	82–102	9.2
instant coffee	1.27	5	1.24	0.05	94–103	4.0
instant coffee decaffeinated	1.27	5	1.24	0.02	96–100	1.6
malt coffee	1.04	5	1.03	0.04	93–103	3.9
tea type						
black tea	1.27	3	0.59	0.06	42–50	10.2
black tea*	0.52	1	0.41		79	
	1.04	1	0.94		90	
green tea	1.27	3	0.84	0.02	65–68	2.4
green tea*	1.08	1	0.99		92	
fruit tea	1.27	3	0.77	0.09	55–69	11.7
fruit tea*	1.04	1	1.04		100	
herb tea	1.27	3	0.65	0.01	50–52	1.5
herb tea*	1.04	3	1.14	0.08	118–104	7.0
children's herb tea*	1.04	2	0.93	0.08	84–95	8.6
beer type						
Pils 1st test	0.052	4	0.047	0.010	64–105	21.3
Pils 2nd test	0.052	3	0.050	0.007	80–107	14.0
Exportbier	0.051	4	0.050	0.002	92–104	4.0
Weizenbier	0.051	4	0.042	0.004	76–90	9.5
Starkbier	0.051	4	0.041	0.005	67–92	13.1
Bier alkoholfrei	0.051	4	0.048	0.002	88–100	4.2
Leichtbier	0.051	4	0.048	0.002	90–98	4.2
Diätbier	0.051	4	0.043	0.003	78–92	7.0
Malzgetränk	0.051	4	0.046	0.004	82–100	8.7

*from beverages

Table 2: **Ochratoxin A concentration in 6 samples taken from a commercial 500 g bag of coffee; its content was manually mixed before the samples, 25 g each, were taken**

sample no.	OTA concentration detected (µg/kg)
1	1.93
2	2.33
3	1.99
4	2.00
5	2.07
6	1.87
mean (µg/kg) = 2.03 standard deviation = 0.161 % CV = 7.92	

Table 3: **Ochratoxin A concentrations in samples of main coffee types consumed in Germany. Detection limit for all types: 0.3 µg/kg powder**

coffee type	total of samples	samples contam.	mean *all samples*	mean *samples contam.*	median *all sampels*	median *samples contam.*	maximum	90. percentile
		%	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
coffee roasted ground	113	46	0.61	1.15	< 0.3	0.72	6.32	1.58
coffee roasted ground decaffeinated	67	48	0.56	1.01	< 0.3	0.84	3.34	1.75
coffee roasted ground mild	60	35	0.45	1.0	< 0.3	0.64	4.75	0.84
instant coffee	52	88.5	1.83	2.05	1.06	1.16	9.47	4.27
instant coffee decaffeinated	32	59	0.59	0.89	0.36	0.76	1.8	1.59
malt coffee	33	15	< 0.3	0.65	< 0.3	0.64	0.96	0.64

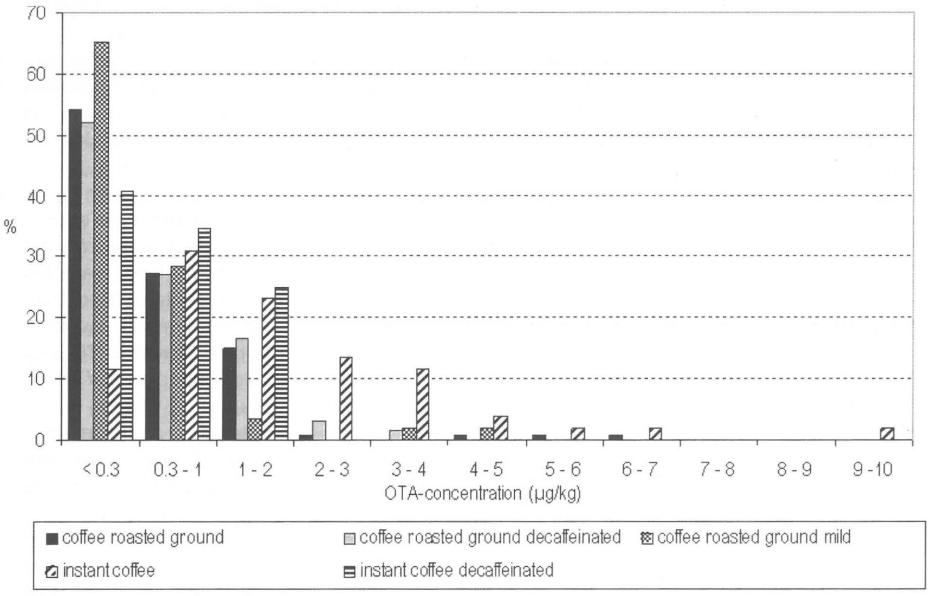

Fig. 1: Ochratoxin A frequency distribution in samples of different coffee types in the German market.

Besides the five leading companies holding a share of about 70 % of the German coffee market, several minor producers supply coffee, some of them in mainly local markets. Some of these products were comparable in quality to those of four of the leading companies, some were of lower quality regarding OTA concentrations; some, although few, of the contaminated samples contained more than 3 µg ochratoxin/kg, others were nearly OTA free.

One out of ten samples purchased in 5 health food stores contained the toxin. In view of the low number of samples taken from these stores it cannot be judged whether coffee from this source is of better quality regarding the toxin content than coffee sold by usual stores.

No differences in OTA concentrations among instant coffee of different manufacturers were found.

The fact that OTA is not, or only incompletely destroyed during roasting of green coffee beans (TSUBOUCHI et al., 1988) has led to several investigations to determine the toxin concentrations in coffee in the last ten years. Table 4 provides a summary of these earlier results. Accordingly, a considerable part of the samples analyzed by different groups contained the toxin; a comparison of the results in terms of quantity is not always possible, as the degree of contamination depends not only on natural fluctuations in the raw material, but also on the different quality – in terms of OTA levels – of the coffee supplied by different manufacturers; differences in analytical details, mainly concerning the detection limit , also play some role. Most of the earlier publications do not report any details as to differences among qualities supplied by different manufacturers. Mean values differ to some extent, depending on the concentrations assumed for samples in which no toxin has been found. However, besides these discrepancies, some agreements are obvious, too; ochratoxin levels ranged from the detection limit up to 10 µg/kg in roasted and ground coffee. In most papers, arithmetic means were between 0.5 and 1.0 µg/kg, and instant coffees with caffeine usually contained higher toxin concentrations than roasted coffee. In a recent extended study, 9 laboratories analyzed 633 coffee samples which had been selected according to the manufacturers' market share (v. d. STEGEN et al., 1997). About 50 % of the roasted coffee samples contained OTA (detection limit 0.3 µg/kg; for negative samples, half of the detection limit was taken for calculations); about 75 % of the samples were in the concentration range of 0–1 µg/kg powder, the highest concentration detected was 8.2 µg/kg and the arithmetic mean 0.8 µg/kg of caffeine containing and 0.7 µg/kg of decaffeinated roasted coffee. These data correspond well to those obtained in the present study. Regarding instant products, toxin concentrations of caffeine containing samples fluctuated over a wider range and the mean, 1.4 µg/kg, was higher than that of roasted coffee samples. This is confirmed by the present study in which lower toxin concentrations have been found in the decaffeinated instant products. Since both investigations yielded very similar results it can be assumed that OTA levels in coffee remained about the same during the last five years. As one of its most remarkable results the present study has shown that one of the leading coffee producers in the German market supplied practically OTA free roasted coffee over a longer period. Regarding instant coffee, efforts should be made towards less contaminated products.

The European Commission is discussing limits for OTA in different food. For coffee, a limit of 4 µg/kg coffee powder has been propos-ed. The present study has shown that, at least in Germany, more than 90 % even of instant coffees contained concentrations below this limit. Provided that a limit of 4 µg/kg is fixed for EU member-countries, it will be interesting to see whether toxin concentrations will remain the same or whether manufacturers will utilize the scope left to them and supply coffee of worse quality in terms of ochra-toxin concentrations.

Tea

Samples of the most favourite teas, black, green, fruit and herb tea, as well as some teas on a herb basis for children were analyzed. As the data in Table 1 show recovery rates of OTA spiked tea were lower than of spiked coffee related to tea leaves, however, from the beverage the toxin was recovered quantitatively as also shown in the table. The detection limit was 0.3 µg/kg tea leaves. Results are presented in Table 5. In black tea samples, no toxin has been found. One out of 32 samples of green tea contained OTA. Fruit tea samples did not contain detectable amounts of the toxin, while of herb teas, about 10 % of the samples were contaminated up to a maximum of 1.78 µg/kg. Surprisingly, many of the herb teas for children were contaminated, some even by higher toxin concentrations. Studies to detect the origin of OTA in the tea samples are underway; results will be published elsewhere.

Table 4: **Earlier data of ochratoxin A in coffee samples**

Author(s)	no. of samples	samples contam. %	detect. limit µg/kg	range µg/kg	mean µg/kg
MAJERUS et al. (1993)	29	3			3.0
STUDER–ROHR (1995)	40	45	0.5	0.4–7.8	
MAIERHOFER et al. (1995)	23	61	0.05	0.05–0.44	0.18
KOCH et al. (1996)	30	83	0.3	n.d.–1.0 (21) 1.0–7.54 (9)	1.43
PITTET et al. (1996)*	101	74	0.2	0.2–6.5	1.1
PATEL et al. (1997)	20	85	0.1	0.2–2.1	0.6
PATEL et al. (1997)*	80	80	0.1	0.1–8.0	1.1
BURDASPAL and LEGARDA (1998)	28	100	0.1	0.22–5.64	0.88
BURDASPAL and LEGARDA (1998)*	9	100	0.1	0.19–1.08	0.50
JØRGENSEN (1998)	11	100	0.1	0.1–0.99 (10) 3.2 (1)	0.51

* instant coffee; n.d. none detected

Beer

Many beer specialities of more or less economic importance are supplied in Germany; leaders in terms of consumption figures are *Pils, Export* and *Weizen.* A product of seasonal importance is called *Starkbier.* Specialities of less economic weight are beers containing no alcohol, *Leichtbier ('light' beer)* with reduced alcohol content, *Diätbier* reduced in its carbohydrate but not in its alcohol content, and *Malzbier* sometimes also called *Malztrunk* containing additional ingredients not complying with the German 'demand of pureness'. Samples of these, more of the strongly selling and less of the other, had been collected from producers all over Germany including smaller and less known brands of merely local importance.

Reproducibility and recovery were 80–97 %; variation coefficients were between 8 and 20 % (Table 1). A detection limit of 0.01 µg OTA/l beer had been derived from the chromatograms. For samples, in which no toxin has been found, half the detection limit was taken for calculations.

OTA concentrations detected are given in Table 6. Any type of product contained the toxin; however, of the product types ranking second, less samples were contaminated than of the most favourite types. More than 70 % of the samples of *Pils, Export-, Weizen-* und *Starkbier* contained the toxin at concentrations of about 0.03 µg/l on the average, while contaminated samples of the secondary types have been found to contain only about half that concentration. The highest concentration of 0.29 µg/l, i. e. 10 times the average, was detected in one sample of *Weizenbier.* Fig. 2 shows the frequency pattern of toxin concentrations. Accordingly, more than 90 % of all contaminated samples contained OTA concentrations of 0.01–0.1 µg/l and more than 80 % of positive samples of the most favourite products contained less than 0.05 µg/l.

Table 5: **Ochratoxin A concentrations in commercial tea in Germany. Detection limit: 0.3 µg/kg tea leaves**

tea type	total of samples	samples contam. %	mean *all samples* µg/kg	mean *samples contam.* µg/kg	median *all sampels* µg/kg	median *samples contam.* µg/kg	maximum µg/kg	90. percentile µg/kg
black tea	32	0						
green tea	32	3	< 0.3		< 0.3		1.33	< 0.3
fruit tea	32	0						
herb tea	34	9	< 0.3	1.14	< 0.3	1.21	1.78	0.4
children's herb tea	31	42	1.25	2.78	< 0.3	2.67	10.3	3.06

Table 6: **Ochratoxin A concentration in samples of different German beer types. Detection limit: 0.01 µg/l**

beer type	total of samples	samples contam. %	mean *all samples* µg/l	mean *samples contam.* µg/l	median *all sampels* µg/l	median *samples contam.* µg/l	maximum µg/l	90. percentile µg/l
Pilsbier	135	75	0.026	0.033	0.020	0.029	0.137	0.058
Exportbier	31	81	0.027	0.032	0.019	0.021	0.123	0.059
Weizenbier	30	77	0.031	0.039	0.022	0.027	0.293	0.041
Starkbier	54	83	0.031	0.036	0.023	0.027	0.126	0.082
Bier alkoholfrei	24	54	0.013	0.020	0.012	0.015	0.035	0.030
Leichtbier	8	25	0.011	0.030	< 0.010	0.030	0.047	–
Diätbier	6	67	0.013	0.017	0.015	0.017	0.019	–
Malztrunk	30	47	0.016	0.029	< 0.010	0.028	0.081	0.033

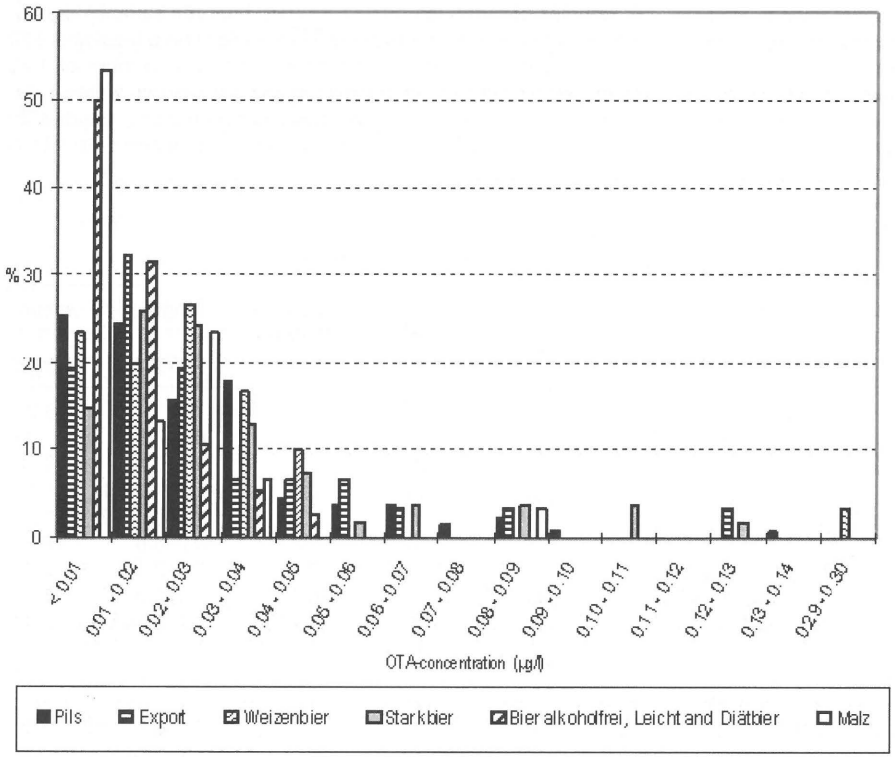

Fig. 2: Ochratoxin A frequency distribution in samples of different German beer specialities.

The number of *Pils* samples analyzed was regarded as sufficient to reveal differences in OTA levels among beers from different regions. To detect regional differences, Germany was arbitrarily divided into four parts and the mean of all samples from breweries located in one of these was calculated. Fig. 3 shows that breweries in the North and East supplied more contaminated samples with higher mean concentrations than those in the West and South. There were not enough samples from one and the same brewery taken at different times to allow a detailed analysis for differences in the products of one brewer, which would have been another interesting question to answer. Toxin concentrations of two samples from a larger well known brewery bought in 1996 and in 1997 varied from below detection limit up to 0.063 µg/l. Four beers of another well known brewery contained 0.017–0.022 µg/l.

In the last decade, concentrations of OTA in beer samples were reported by different laboratories (see Table 7). *Starkbier* obviously was more contaminated than the best selling products in which no toxin was detected. The discrepancy between these earlier data and those obtained more recently could be due to a higher toxin content of barley grown in earlier years; it is explained also by earlier detection limits

of as much as 0.05 µg/l . Most of the previous data hence no longer represent the situation of today. So our present results should be compared to the most recent data of other laboratories. LEGARDA and BURDASPAL (1998) investigated beer produced in Spain and other European countries. The mean toxin concentration of 38 Spanish beers was 0.024 µg/l and of 42 samples from other countries 0.025 µg/l. These data correspond to the results presented here. JØRGENSEN (1998) who analyzed 21 beers, most of them from Danish breweries, obtained a mean concentration of 0.049 µg/l. The slightly higher value could be due to barley grown in the North of Europe with more rain than in the South. In general, however, the beer was contaminated to about the same extent nearly independent of its origin in Central Europe. This was also confirmed by own results obtained in 7 *Pils* samples from the Czech Republic which contained 0.021 µg/l on the average. The very similar data obtained from inves-tigations in different regions of Europe lead one to assume a toxin concentration of about 0.03 µg/l to prevail in the best selling beer types.

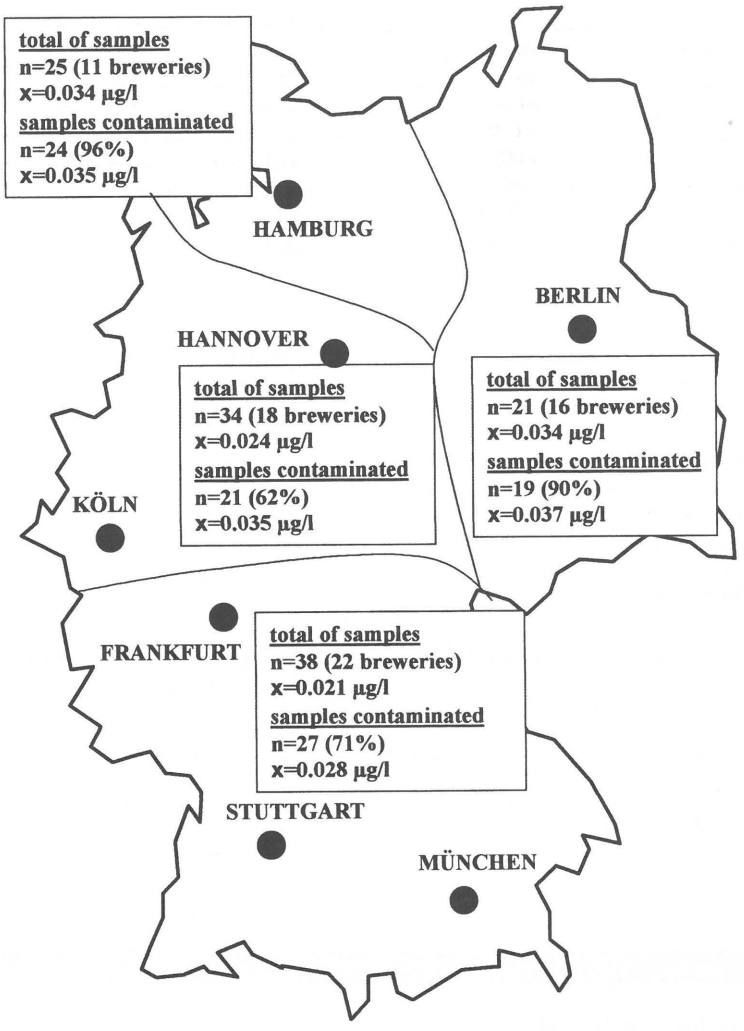

Fig. 3: Ochratoxin A levels in *Pils* from different regions of Germany. Samples collected in 1996–1998.

Summary

To determine the ochratoxin status of the German population, 357 samples of coffee, 161 of tea and 318 of beer were analyzed for their toxin content. The samples had been purchased in retail shops. Analysis was performed by applying immunoaffinity columns and the toxin was detected by HPLC. The detection limit for coffee was 0.3 µg/kg powder. About 50 % of the samples of roasted and ground coffee, with and without caffeine, contained the toxin at concentrations of 0.3–6.3 µg/kg; taking the data of all samples, a mean of 0.61 µg/kg was obtained. Of 'mild' coffee, less samples were contaminated, on the average by 0.45 µg/kg. Of instant coffee samples with caffeine, about 90 % contained the toxin in the range of 0.3–9.47 µg/kg, resulting in a mean of 1.83 µg/kg. A lower level was detected in decaffeinated instant products where the toxin has been found in less samples.

No toxin has been found in black and fruit tea (detection limit 0.3 µg/ kg tea leaves), but in 3 % of green tea, 9 % of herb tea and 42 % of children tea samples on a herb basis.

Of *Pils, Export, Weizen* and *Stark*beer samples analyzed, 70–80 % contained ochratoxin at concentrations of 0.01–0.29 µg/l, i. e. 0.03 µg/l on the average. The limit of detection was 0.01 µg/l . Lower concentrations have been found in beer without alcohol, *Leichtbier, Diätbier* and *Malzbier.*

Zusammenfassung

Im Zuge der Ermittlung des Ochratoxin-Status der deutschen Bevölkerung wurden 357 Kaffee-, 161 Tee- und 318 Bierproben aus Lebensmittelläden auf ihren Gehalt an Ochratoxin A untersucht. Zur Analyse wurden Immunoaffinitätssäulen eingesetzt; das Toxin wurde mit HPLC über seine Eigenfluoreszenz bestimmt. Bei den Kaffeesor-

Table 7: **Earlier data on ochratoxin A in beer samples**

Author(s)	main origins of beers	no. of samples	samples contam. %	detection limit µg/l	mean µg/l	range or max. concentration µg/l
EL-DESSOUKI (1992)	Germany	56	30	0.3		0.35–1.53[1]
MAJERUS et al. (1993)	Germany	66	8			0.1[2]
		40	35			0.1–1.5[1]
JIAO et al. (1994)	Germany	32	59	0.05		0.1–0.19[1]
		133	56			0.20–0.49[2]
		25	8			0.05–0.09[3]
ZIMMERLI and DICK (1995)	Switzerland	7	100	0.005	0.012 (median)	0.01–0.03[3]
SCOTT and KANHERE (1995)	Canada	41	63	0.05	0.061	0.2
THELLMANN and WEBER (1997)	Germany	11	55	0.03		0.08
GULDBORG (1997)	Denmark	9	56	0.01	0.02	0.026
JØRGENSEN (1998)	Denmark	21	100	0.001	0.049	0.16
LEGARDA and BURDASPAL (1998)	Spain	38	97	0.004	0.024	0.121
	Europe	42	100		0.025	

[1]: mostly Starkbiere; [2]: Vollbier earlier product categorie, Pils and Export included; [3]: Malztrunk

ten lag die Nachweisgrenze bei 0,3 µg/kg Pulver. Etwa die Hälfte der gemahlenen Röstkaffeeproben enthielten Toxinmengen im Bereich von 0,3–6,3 µg/kg, im Mittel aller Proben 0,61 µg. Von den „milden" Sorten waren weniger Proben kontaminiert, im Mittel enthielten sie 0,45 µg/kg. Bei den koffeinhaltigen Instantprodukten ließ sich das Toxin in nahezu 90 % der Proben nachweisen, im Mittel ergab sich eine Menge von 1,83 µg/kg. Der entkoffeinierte Instantkaffee erwies sich als weniger toxinhaltig. In den Schwarztee- und Früchteteeproben ließ sich Ochratoxin nicht nachweisen. Hingegen enthielten 3 % der Proben der Sorte Grüner Tee und 9 % der Kräuterteeproben das Toxin. Bei den auf Kräuterteebasis hergestellten Kindertees ist Ochratoxin in 42 % der Proben nachgewiesen worden. Zwischen 70 und 80 % der Pils-, Export-, Weizen- und Starkbierproben enthielten Ochratoxin im Bereich von 0,01–0,29 µg/l, im Mittel 0,03 µg/l. Alkoholfreies Bier, Leichtbier, Diätbier und Malzbier waren weniger kontaminiert.

Acknowledgement

This work was supported by the German Ministry of Health. H. Bresch is grateful to Mrs. Sybill Saupe for revision of the English manuscript.

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***Corresponding author:**
Dr. Horst Bresch, Bundesforschungsanstalt für Ernährung, Institut für Hygiene und Toxikologie, Haid-und-Neu-Straße 9, D–76131 Karlsruhe.
Tel.: ++721 6625 526, Fax: ++721 6625 453.
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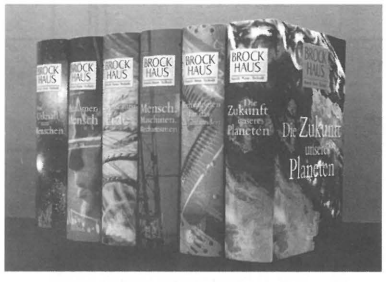

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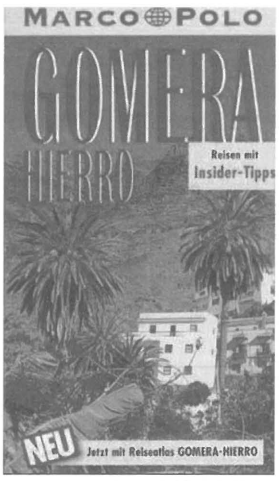

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