codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

JOINT OFFICE: Viale delle Terme di Caracalla 00100 ROME Tel: 39 06 57051 www.codexalimentarius.net Email: codex@fao.org Facsimile: 39 06 5705 4593

Agenda Item 16 (f) **CX/FAC 05/37/26**
December 2004

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON FOOD ADDITIVES AND CONTAMINANTS

Thirty-seventh Session
The Hague, the Netherlands, 25 – 29 April 2005

MYCOTOXIN CONTAMINATION IN SORGHUM

(Information submitted in response to CL 2004/9-FAC)

The following comments have been received from: Japan

JAPAN

Background

The 36th CCFAC noted that working paper could not be prepared because no data were received in response to CL 2003/13-FAC. In recognizing that sorghum was an important crop for many countries, in particular developing countries, and because of the need to move towards the establishment of maximum levels, the Committee agreed to request information on: source of contamination; type of mycotoxin involved; analytical methods and sampling procedures; consumer protection from the point of view of health; actual and potential problems in international trade; work already undertaken by other international organizations; etc. for discussion at its next Session.

On the basis of this agreement, we submit the Japanese analytical data on mycotoxins in sorghum in the GEMS/Food format.

Comments

Purpose of analysis

Japan conducts surveillance on mycotoxin levels in primary feed materials in order to reduce their levels in feeds. We have analyzed mycotoxins in imported sorghum as sorghum is one of the major feed materials used in Japan and most of sorghum grains are imported into Japan.

Method of sampling

The whole content of a silo or a truckload of sorghum is regarded as a lot. Incremental samples were collected more than several times during the transfer of sorghum from the silo or the truck to the processing

line of factory. All the sample taken as above were combined to make a primary sample of about 20 kg. The primary sample was separated to produce a secondary sample of 5 kg to be sent to the respective laboratory. A total of six laboratories were contracted to analyze mycotoxins in sorghum. The whole secondary sample was comminuted prior to chemical analysis.

Summary of methods of analysis

Aflatoxin B1

AflatoxinB1 was extracted from the comminuted sorghum by acetonitrile-water (9+1), the extract was cleaned up using Mycosep #226 multifunction clean up column, and aflatoxin B1 was determined with HPLC equipped with a fluorometric detector.
The condition of the HPLC is as follows:
Column: ODS column (250 mm×4.6 mm i.d.)
Mobile phase: Water:methanol (3+2)
Flow rate: 0.8 ml/min
Detection: excitation at 365 nm; and fluorescence measured at 450 nm

Ochratoxin A

After the comminuted sorghum was acidified with acetic acid-water (1+19) and Ochratoxin A was extracted with chloroform, the extract was cleaned up using the cartridge column (SEP-PAK) and ochratoxin A was determined with HPLC with a fluorometric detector.
The condition of the HPLC is as follows:
Column: ODS column (250 mm×4.6 mm i.d.)
Mobile phase: Acetonitrile:dilute phosphoric acid (11+9)
Flow rate: 1.0 ml/min
Detection: excitation at 337 nm; and fluorescence measured at 467 nm

Zearalenone

Zearalenone was extracted from the comminuted sorghum by acetonitrile-water (21+4), the extract was cleaned up using Mycosep #226 multifunction clean up column, and zearalenone was determined with HPLC with a fluorescence detector.
The condition of the HPLC is as follows:
Column: ODS column (250 mm×4.6 mm i.d.)
Mobile phase: Methanol:water (13+7)
Flow rate: 1.0 ml/min
Detection: excitation at 278 nm; and fluorescence measured at 460 nm
.

Deoxynivalenol and Nivalenol

The five components of group B of trichothecene mycotoxins were extracted from the comminuted sorghum by acetonitrile-water (84+16), the extract was cleaned up using Mycosep #226 multifunction clean up column, deoxynivalenol and nivalenol were derivatized and then determined with gas chromatography equipped with an electronic capture detector.
The condition of the gas chromatography is as follows:
Column: DB-35 (35%-Phenyl)-methylpolysiloxane (30 m×0.25 mm i.d., 0.25 μm film thickness)
Carrier gas: He 1.5 ml/min
Injection temp. 250 °C
Column temp. 80 °C (1min)-20 °C /min-180 °C -5 °C /min-300 °C (10min)
Detector temp. 300 °C

Fumonisin

Fumonisin B1 and B2 were extracted from the comminuted sorghum by methanol-water (3+1), the extract was cleaned up using Bond Elut LRC SAX cartridge, and fumonisin was determined with HPLC/post-column derivatization.
The condition of the HPLC is as follows:
Column: fluoridated silica gel column (30 mm×4.6 mm i.d.)

Mobile phase: Methanol:trifluoroacetic anhydride (1+1)
Flow rate: 1.0 ml/min
Post-column derivatization: o-phthalaldehyde and N-acetyl –L-cystein
Detection: excitation at 340 nm; and fluorescence measured at 450 nm

Quality assurance

- The Fertilizer and Feed Inspection Service (FFIS) in Japan has participated in the proficiency test of the FAPAS for deoxynivalenol and nivalenol in wheat.
- It conducted procedural recovery tests at 2 to 3 different concentrations of individual mycotoxins added to 3 to 4 kinds of grains or compound feeds.
- Six branch laboratories of the FFIS participate in collaborative studies organized by the FFIS for 2 to 3 analyte/matrix combinations a year. They also participate in other collaborative studies as are invited to do so.
- Whenever the FFIS develops an analytical method, it conducts a collaborative study using 2 or 3 different matrices, in cooperation with 6 to 15 laboratories.

		WHO GEMS/Food Reporting Format - Aggregate Data on Contaminant Levels in Food											
Further instructions for completing these fields is available in GEMS/Food Instructions for Electronic Submission of Data on Chemical Contaminants in Food, Revised January 2002 available at http://www.who.int/fsf/Chemicalcontaminants/index2.htm													
1	2	3	4	5	6	7	8	9	10	11	12a	12b	12c
SN	CD	CC	FD	OR	SP	REP	NOL	AQA	CON	DIM	LODMIN	LODMAX	LOQMIN
Serial no. of the record	Creation date of record	Country code	Food identifier	Food origin	Sampling period	Sample representativeness	Number of contributing laboratories	Analytical quality assurance	Contaminant	Dimension of results	LOD min.	LOD max.	LOQ min.
T1040001	15-Oct-2004	JPN	GC651	AUS	04/2001-06/2001	NP	5	IQ	021	2	0.05	0.05	0.5
T1040002	15-Oct-2004	JPN	GC651	USA	04/2001-05/2004	NP	6	IQ	021	2	0.05	0.05	0.5
T1040003	15-Oct-2004	JPN	GC651	ARG	04/2001-11/2003	NP	5	IQ	021	2	0.05	0.05	0.5
T1040004	15-Oct-2004	JPN	GC651		04/2001	NP	1	IQ	021	2	0.05	0.05	0.5
T1040005	15-Oct-2004	JPN	GC651	CHN	08/2003	NP	1	IQ	021	2	0.05	0.05	0.5
T1040006	15-Oct-2004	JPN	GC651	AUS	04/2001	NP	2	IQ	092	2	1	1	2
T1040007	15-Oct-2004	JPN	GC651	USA	04/2001-05/2004	NP	2	IQ	092	2	1	1	2
T1040008	15-Oct-2004	JPN	GC651	ARG	04/2001	NP	1	IQ	092	2	1	1	2
T1040009	15-Oct-2004	JPN	GC651	AUS	04/2001-08/2002	NP	5	IQ	070	2	20	20	50
T1040010	15-Oct-2004	JPN	GC651	USA	04/2001-05/2004	NP	6	IQ	070	2	20	20	50
T1040011	15-Oct-2004	JPN	GC651	ARG	04/2001-11/2003	NP	5	IQ	070	2	20	20	50
T1040012	15-Oct-2004	JPN	GC651	CHN	05/2003	NP	2	IQ	070	2	20	20	50
T1040013	15-Oct-2004	JPN	GC651	USA	05/2003-06/2004	NP	3	IQ	170	2	5	5	10
T1040014	15-Oct-2004	JPN	GC651	CHN	05/2003	NP	1	IQ	170	2	5	5	10

T1040015	15-Oct-2004	JPN	GC651	ARG	09/2003-11/2003	NP	3	IQ	170	2	5	5	10	
T1040016	15-Oct-2004	JPN	GC651	USA	05/2003-04/2004	NP	3	IQ	186	2	5	5	10	
T1040017	15-Oct-2004	JPN	GC651	CHN	05/2003-08/2003	NP	1	IQ	186	2	5	5	10	
T1040018	15-Oct-2004	JPN	GC651	ARG	09/2003-11/2003	NP	3	IQ	186	2	5	5	10	
T1040019	15-Oct-2004	JPN	GC651	ARG	04/2001-11/2003	NP	5	IQ	132	2	10	10	20	
T1040020	15-Oct-2004	JPN	GC651	AUS	04/2001-06/2004	NP	5	IQ	132	2	10	10	20	
T1040021	15-Oct-2004	JPN	GC651	USA	04/2001-06/2001	NP	5	IQ	132	2	10	10	20	
T1040022	15-Oct-2004	JPN	GC651	CHN	05/2003-05/2004	NP	3	IQ	132	2	10	10	20	
▯1 When means in columns 17a, 17b and 17c were calculated in accordance with Table 1 in APPENDIX 4 in the “Instruction for Electronic Submission of Data on Chemical Contaminants in Food and the Diet”, LOQs were utilized instead of LODs because of having no data of between LOD and LOQ, we calculated using 1/2 of LOQ.														

12d	13	14	15	16a	16b	17a	17b	17c	18	19	20	21	22	23	
LOQMAX	BASE	N	<	MIN	MAX	X	XL	XU	MED	90th	STDDEV	STATUS	REM	RMA	
LOQ max.	Results based on	Number of samples	Number of samples less than the LOQ	Range - minimum	Range - maximum	Mean or best estimate	Mean - lower bound	Mean - upper bound	Median or best estimate	90th Percentile	Standard deviation (Optional)	Status of data	Remarks/ References	Reference for method of analysis	
0.5	A	14	13	1	1		0.1	0.5				0	▯1		92.9%
0.5	A	32	31	2	2		0.1	0.5				0	▯1		96.9%
0.5	A	12	11	1	1		0.1	0.5				0	▯1		91.7%
0.5	A	1	1				0.0	0.5				0	▯1		100.0%
0.5	A	1	1				0.0	0.5				0	▯1		100.0%
2	A	4	4				0	2				0	▯1		100.0%
2	A	4	3	560	560		140	142				0	▯1		75.0%
2	A	1	1				0	2				0	▯1		100.0%
50	A	15	3	164	1290	290			206	428	312	0	▯1		20.0%
50	A	59	22	75	3764	532			260	1243	784	0	▯1		37.3%

50	A	11	6	615	1030	399						0	▫1		54.5%
50	A	3	3				0	50				0	▫1		100.0%
10	A	11	4	17	180	59			32	165	70	0	▫1		36.4%
10	A	2	2				0	10				0	▫1		100.0%
10	A	3	3				0	10				0	▫1		100.0%
10	A	11	7	13	98		13	19				0	▫1		63.6%
10	A	2	1	4750	4750	2378						0	▫1		50.0%
10	A	2	2				0	10				0	▫1		100.0%
20	A	13	13				0	20				0	▫1		100.0%
20	A	13	13				0	20				0	▫1		100.0%
20	A	29	20	22	532		62	76				0	▫1		69.0%
20	A	4	2	47	100	42						0	▫1		50.0%