DETERMINATION OF TRICOTHECENE MYCOTOXINS AS THEIR TRIMETHYLSILYL AND HEPTA-
FLUOROBUTYRYL DERIVATIVES IN FEEDS AND GRAINS

E. LINDFORS, S. BERG and A. RIZZO

National Veterinary Institute, P.O.Box 368, SF-00101 Helsinki, Finland

In 1982 and especially 1984 a large number of farm and fur animals and Rainbow trouts became ill in Finland, and many died after consuming certain commercial feeds. The symptomatology indicated to poisoning by Fusarium trichothecenes which chemical analysis finally confirmed in 1984 in those feeds "(ref. 1)". In 1984 dense growths of F. graminearum and F. poeae were found in feed factories, and molds were isolated and identified from production lines and grain silos.

Since 1985 we have analyzed more than 400 feed and grain samples. Trichothecenes of the T-2 type have been detected in feed only, not in the grain samples. The identified trichothecenes are deoxynivalenol (DON), 3-acetyl-deoxynivalenol (3-AcDON), nivalenol (NV), fusarenon-X (F-X), diacetoxyscirpenol (DAS), scirpentriol (SCT), T-2, HT-2 and acetyl-T-2. (Table 1).

The toxins have been determined as their trimethylsilylethers (TMS) with capillary gas chromatography and EC-detector (EC-GC). The purification procedure is quite long (Fig. 1), but it is especially necessary for feed

Table 1. Trichothecenes in feeds and grains in Finland

		Procentual occurrence									
	No of										
Year	samples	Negat	DON	3-AcDON	DAS	NV	F-X	T-2	HT-2	SCT	AC-T-2
1985	82	6	91	- ^a	18	2	0	15	10	-	-
1986	113	3	97	51	18	10	7	13	5	-	-
1987	168	2	98	66	4	2	0	11	3	1	1

Range and mean of concentration in µg/kg								
Year	DON	3-AcDON	DAS	NV	F-X	T-2	HT-2	SCT
1985	1-6300 (138)	-	7-1680 (293)	10-161 (86)	n.d. ^b	5-60 (16)	5-80 (24)	-
1986	1-4000 (120)	2-211 (11)	1-1500 (141)	1-1000 (122)	1-38 (12)	9-238 (77)	8-42 (16)	-
1987	1-16000 (190)	2-574 (29)	27-822 (305)	27-115 (85)	n.d.	23-964 (288)	5-120 (56)	781-2466 (1624)

-^a = not looked for, n.d.^b = not detected

samples which contain many different materials in addition to cereals. Analysing nine trichothecenes with different polarities means that many impurities are present in the final analytical step. It is therefore extremely necessary to use mass spectrometry for confirmation.

Determination of trichothecenes as heptafluorobutyrate (HFB) with an EC-detector is difficult owing to many interfering peaks, but determination of trichothecenes as HFB derivatives by mass spectrometry and SIM-technique offers many advantages. The purification procedure is shorter and simpler (Fig. 1), the method is more specific and the higher sensitivity of the instrument can be utilized since ions of higher mass are in a range where the background signal is lower (Fig. 2).

Fig. 1. Procedures used for (A) EC-GC determination of TMS derivatives and (B) GC-MS determination of HFB derivatives

Fig. 2. GC-MS selected ion monitoring analysis as HFB derivatives of spiked wheat sample.

REFERENCES

- 1 E. Karppanen, A. Rizzo, S. Berg, E. Lindfors and R. Aho, Fusarium mycotoxins as a problem in Finnish feeds and cereals, J. Agric. Sci. Finland, 57 (1985) 195-206.