### Levels of PCDD and PCDF Congeners in Milk from Farms Near Bolsover, UK

#### J.R. Startin, C. Wright, M. Kelly, M. Rose

CSL Food Science Laboratory, Norwich Research Park, Colney, Norwich, UK

#### and

N. Harrison

Ministry of Agriculture, Fisheries and Food, Ergon House, 17 Smith Square, London, UK

#### Introduction

The analysis of cows' milk for PCDDs and PCDFs is of particular interest because milk consumption is generally recognized as one of the main routes of exposure to these compounds for the general population and also because it can provide valuable information on the impact of localized point sources to the atmosphere and help to locate such sources. MAFF and the CSL Food Science Laboratory have been following a programme to survey PCDDs and PCDFs in milk from different locations in the UK. Results on some background and urban/industrial samples have been published previously [1,2]. This report deals with samples obtained from the area around Bolsover in Derbyshire, UK.

#### Experimental

Milk was either extracted with solvents following a modification of the AOAC Official Method of Analysis (1984) for General Multiresidues in Milk (Method 29-012) or freeze-dried and ground to provide a homogeneous powder. These methods have been shown to give equivalent results [3]. PCDDs and PCDFs were isolated by the method of Smith *et al* [4] using an automated apparatus (Fluid Management Systems) [5] and determined by HRGC/HRMS using a non-polar GC column. Further details are given elsewhere [6].

#### Results

In late 1990 samples were obtained from a number of farms in the area close to Bolsover, Derbyshire. Two farms were found to have extremely high levels of some congeners.

Table 1 gives a summary of results for milk from 30 farms in the area, excluding the farms found to be obviously affected by contamination. Some farms were sampled on different occasions and the results represent a total of 47 samples taken between October 1990 and October 1993. Most of the results are consistent with the range established previously [1,2] although one farm included in this set did show unusually high concentrations (0.5 ng/kg TEQ).

Details of the results obtained from the initial and from subsequent sampling of the farms showing high levels are given in Table 2. Following repeated and extended sampling a third farm was found on which there were high concentrations in milk from cows used to maintain a suckler herd and these reults are also included in Table 2 (Farm B). It should be noted

that the August 1992 sample from Farm C was from a single cow and had an extremely low fat content leading to a low dioxin concentration on a whole sample basis.

Comparison with Table 1 clearly shows elevation of the PCDD concentrations, but not of PCDFs, in samples from these farms taken during 1990 - 1991 and a decline over time. Further discussion of these results and the management of the incident to which they refer will be presented separately [7].

#### References

- 1. J.R Startin, M. Rose, C. Wright, I. Parker and J. Gilbert, Chemosphere, **20**, 793 (1990).
- 2. Ministry of Agriculture Fisheries and Food, Dioxins in food; Food Surveillance Paper No.31, HMSO, London (1992).
- 3. C. Wright, M. Kelly and J.R. Startin, The effects of sample storage conditions on the determination of PCDDs and PCDFs in milk, Presented at: Dioxin 92, Tampere.
- 4. L.M. Smith, D.L. Stalling and J.L. Johnson, Anal. Chem., 56, 1830 (1984).
- 5. W.E. Turner, S.G. Isaacs and D.G. Patterson Jr., Chemosphere, 25, 805 (1992).
- J.R. Startin, C. Wright, M. Kelly and A. Charlesworth, Dioxin concentrations in the blood of individuals resident on farms near Bolsover, UK, Submitted for presentation at Dioxin 94, Kyoto, Japan.
- 7. N. Harrison and J.R. Startin, Dioxins in milk: A case study on localised contamination, Submitted for presentation at Dioxin 94, Kyoto, Japan.

	mean	minimum	lower quartile	median	upper quartile	maximum
2,3,7,8-TCDD	0.05	0.01	0.03	0.04	0.07	0.14
1,2,3,7,8-PeCDD	0.09	0.02	0.05	0.07	0.10	0.42
1,2,3,4,7,8/1,2,3,6,7,8-HxCDD	0.20	0.05	0.09	0.17	0.25	0.8
1,2,3,7,8,9-HxCDD	0.07	<0.01	0.03	0.05	0.09	0.35
1,2,3,4,6,7,8-HpCDD	0.13	0.03	0.08	0.10	0.12	0.97
OCDD	0.47	0.12	0.28	0.38	0.49	2.9
2,3,7,8-TCDF	0.01	<0.01	<0.01	0.01	0.01	0.04
1,2,3,7,8-PeCDF	0.01	<0.01	<0.01	0.01	0.01	0.11
2,3,4,7,8-PeCDF	0.03	0.03	0.05	0.07	0.10	0.17
1,2,3,4,7,8-HxCDF	0.02	0.02	0.03	0.04	0.05	0.08
1,2,3,6,7,8-HxCDF	0.01	0.01	0.03	0.03	0.05	0.09
1,2,3,7,8,9-HxCDF	0.01	<0.01	<0.01	0.02	<0.01	0.02
2,3,4,6,7,8-HxCDF	0.01	<0.01	0.03	0.03	0.05	0.08
1,2,3,4,6,7,8-HpCDF	0.01	<0.01	0.02	0.02	0.03	0.07
1,2,3,4,7,8,9-HpCDF	0.01	<0.01	<0.01	0.01	<0.01	0.01
OCDF	0.01	<0.01	<0.01	0.01	0.01	0.11
Total I-TEQ	0.17	0.07	0.11	0.14	0.21	0.50

**Table 1.** Summary of concentrations (ng/kg) of PCDDs and PCDFs in whole milk from farmsin Derbyshire other than those displaying high concentrations.Means calculatedusing full value of detection limit for non-detected values.

Sampling date	October 1990		April 1991		July 1991			
	A	с	Α	С	A	B	С	
2,3,7,8-TCDD	0.98	0.78	0.51	0.34	0.33	1.7	0.35	
1,2,3,7,8-PeCDD	0.75	1.3	0.72	0.57	0.36	1.7	0.55	
1,2,3,4,7,8/1,2,3,6,7,8- HxCDD	1.8	2.3	2.1	1.4	1.2	5.0	1.4	
1,2,3,7,8,9-HxCDD	0.53	0.87	0.75	0.52	0.3	1.1	0.38	
1,2,3,4,6,7,8-HpCDD	0.18	0.14	0.48	0.72	0.19	0.45	0.18	
OCDD	0.60	0.41	0.78	1.2	0.50	2.2	0.66	
2,3,7,8-TCDF	0.02	0.02	<0.01	0.01	0.01	0.04	0.02	
1,2,3,7,8-PeCDF	0.02	0.01	<0.01	<0.01	0.01	<0.02	<0.01	
2,3,4,7,8-PeCDF	0.31	0.22	0.20	0.11	0.17	0.31	0.15	
1,2,3,4,7,8-HxCDF	0.11	0.06	0.08	0.05	0.07	0.10	0.06	
1,2,3,6,7,8-HxCDF	0.12	0.13	0.11	0.08	0.08	0.17	0.08	
1,2,3,7,8,9-HxCDF	<0.01	<0.01	<0.01	<0.01	<0.01	0.02	<0.01	
2,3,4,6,7,8-HxCDF	0.13	0.10	0.10	0.06	0.08	0.17	0.07	
1,2,3,4,6,7,8-HpCDF	0.06	0.04	0.05	0.03	0.03	0.08	0.03	
1,2,3,4,7,8,9-HpCDF	0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.01	
OCDF	0.03	0.03	<0.01	<0.01	<0.02	0.08	<0.01	
Total (TEQ)	1.8	1.9	1.3	0.90	0.77	3.4	0.90	
Total (TEQ) on fat	42	40	37	23	13	56	22	
Fat content (%)	4.2	4.7	3.5	4.0	5.9	6.1	4.1	

## Table 2. Concentrations (ng/kg) of PCDDs and PCDFs in whole milk from affected farms.

# Table 1 continued.

Sampling date Farm	August 1992			October 1992			September 1993	
	A	В	С	A	В	С	A	В
2,3,7,8-TCDD	0.13	0.69	0.01	0.12	0.94	0.06	0.07	0.16
1,2,3,7,8-PeCDD	0.06	0.42	0.02	0.06	0.58	0.05	0.03	0.07
1,2,3,4,7,8/1,2,3,6,7,8-	0.25	1.5	0.04	0.13	2.7	0.10	0.06	0.55
HxCDD								
1,2,3,7,8,9-HxCDD	0.06	0.37	0.01	0.04	0.66	0.03	0.02	0.07
1,2,3,4,6,7,8-HpCDD	0.07	0.04	0.02	0.06	0.14	0.03	0.03	0.02
OCDD	0.27	0.15	0.07	0.22	0.42	0.17	0.05	0.04
2,3,7,8-TCDF	0.01	0.01	<0.01	0.03	0.02	0.02	<0.01	0.01
1,2,3,7,8-PeCDF	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2,3,4,7,8-PeCDF	0.07	0.13	0.01	0.12	0.27	0.04	0.05	0.05
1,2,3,4,7,8-HxCDF	0.03	0.04	<0.01	0.04	0.12	0.02	0.02	0.02
1,2,3,6,7,8-HxCDF	0.03	0.05	<0.01	0.04	0.12	0.01	0.02	0.02
1,2,3,7,8,9-HxCDF	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2,3,4,6,7,8-HxCDF	0.03	0.08	<0.01	0.05	0.19	0.02	0.02	0.03
1,2,3,4,6,7,8-HpCDF	0.01	0.02	<0.01	0.02	0.05	<0.01	<0.01	<0.01
1,2,3,4,7,8,9-HpCDF	<0.01	<0.01	<0.01	<0.01	<0.02	<0.01	<0.01	<0.01
OCDF	<0.01	0.01	<0.01	<0.01	<0.02	<0.01	<0.01	<0.01
Total (TEQ)	0.23	1.17	0.03	0.24	1.8	0.11	0.13	0.29
Total (TEQ) on fat	7.4	48	4.5	8.3	72	4.8	4.1	24
Fat content (%)	3.1	2.5	0.67	2.9	2.5	2.3	3.1	1.2