DETERMINATION OF PHTHALIC ACID ESTERS IN PREMIXTURES AND FEEDING STAFFS

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Introduction:

The dialkyl or alkyl/aryl esters of 1,2-benzenedicarboxylic acid, commonly known as phthalates, are high-production-volume synthetic chemicals and ubiquitous environmental contaminants because of their use in plastics. Di-(2-ethylhexyl) phthalate is the most spread phthalate in the environment. Phthalates are animal's carcinogens and can cause fatal death, malformations, testicular injury, liver injury and reproductive toxicity in laboratory animals LATINI (2005).

From the civilization or household chemicals the ubiquitous phthalic acid ester is mentioned. This mechanism is called carry-over. In the food industry the HACCP concept is a very effective tool to control and eliminate hazards during production HEESCHEN et al. (2004).

Material and Methods:

Samples of feed dtuffs were taken from the industrial feed producers (P1 – P5) in the Silesian-Moravian Region of the Czech Republic in accordance with the National Law of Feeding Stuffs. The samples were being taken within the official inspections.

The samples of complete feeding stuffs (CF), mineral complementary feeding stuff (MCF), premixtures (M) and all of their components (C), which include additives and feed materials, were taken from CF given for laying hens (P1, C = 11) and for piglets (P2, C = 14), from MCF for pigs (P3, C = 7), from premixture for dairy cattle (P4, C = 13) and premixture for pigs (P5, C = 19).

Concentrations of phthalic acid esters was also measured.

Analysis were made by a method for the determination of di-2-ethylhexyl phthalate (DEHP) and di-n-butyl phthalate (DBP). Procedure consisted of extraction of phthalates (hexane : acetone, 1 : 1), separation of analytes from co-extracts using gel permeation chromatography in Bio-beads S-X3 gel (mobile phase dichloromethane : cyclohexane, 1 : 1), clean up of extracts or eluates with sulphuric acid and detection and quantification by HPLC (mobile phase acetonitrile : water, 99 : 1; flow 0,8 ml/min; Cogent e-Column, C 18, 150 x 4,6 mm, particle-size 5 μm) with UV detection (diode array detector) at 224 nm.

The statistical software Unistat, version 4.53 was used.

Results and Discussion:

The concentrations (mg.kg^{-1} of original sample) of di-n-butyl phthalate (DBP) and di-2-ethylhexyl phthalate (DEHP) as the sum of DBP and DEHP in samples of each component of feeding stuffs and premix at producers (P1 – P5) are shown on the table 1.

producers	P 1 (CF)	P 2 (CF)	P 3 (MCF)	P 4 (P)	P 5 (P)
component	1.09	4.06	3.71	0.38	2.07
component	0.6	1.74	0.67	0.13	1.05
component	0.62	2.41	1.06	1.52	0.85
component	2.03	4.36	0.72	1.91	0.1
component	3.15	11.03	0.73	9.71	0.35
component	1.21	131.42	0.93	0.19	0.33
component	7.96	0.23	0.66	0.08	0.37
component	0.63	0.31		0.13	0.52
component	28.49	1.32		0.84	0.06
component	38.36	1.15		0.17	0.09
component	2.3	0.31		0.12	0.5
component		1.68		0.45	* 0
component		0.91		3.08	0.24
component		1.89			2.04
component					0.42
component					0.44
component					0.48
component					3.21
component					0.22

** bellow limit of detection*
The limit of detection DBP a DEHP in fat tissues was 0,2 mg.kg^{-1}.
The limit of detection DBP a DEHP in animal and plant stuffs with lower level of fat was 0,03 mg.kg^{-1}.

The highest levels of DBP and DEHP (mg.kg^{-1} of original sample) in components, which were used for producing feeding staffs and premixtures, are shown on table 2.

Table No. 2

kind of feed	producer	component		DBP	DEHP	Sum
CF	P 1	feed material:	soy oil	19.89	8.6	28.49
CF	P 1	additives:	methionin	38.36	<0,03	38.36
CF	P 2	feed material:	fish meal	9.55	1.48	11.03
CF	P 2	feed material:	soy oil	110.96	20.46	131.42
MCF	P 3	premix of salinomycin sodium		1.96	1.74	3.71
MCF	P 3	feed material:	carbonate calcium	0.07	0.86	0.93
M	P 4	additives:	vitamin E	0.49	9.23	9.71
M	P 4	premix of flavours		0.11	2.97	3.08
M	P 5	feed material:	feed flour	1.38	0.69	2.07
M	P 5	additives:	vitamin A	<0,03	3.21	3.21

(CF) complete feeding stuffs; (MCF) mineral complementary feeding stuff; (M) premixtures
P 1 - P 5 industrial producers of feed
** bellow limit of detection*
The limit of detection DBP a DEHP in fat tissues was 0,2 mg.kg^{-1}.
The limit of detection DBP a DEHP in animal and plant stuffs with lower level of fat was 0,03 mg.kg^{-1}.

The highest concentration as a sum of DBP and DEHP was found in feed material – soy oil (131,42 mg.kg^{-1}) as component of complete feeding stuff and in additive – vitamin E (9,71 mg.kg^{-1}) as component of premixture.

Concentrations of phthalic acid esters as sum of DBP and DEHP (mg.kg^{-1}) were found (n=8) in CF (P1) 1,32 ; CF (P2) 1,55; CMF (P3) 0,45; M (P4) 0,54 and M (5) 1,04. There were evidential differences between all feeding stuffs and premixtures ($P < 0,05$).

Concentrations of phthalic acid esters, which we found in feeding stuffs and premixtures were similar as, found RASZYK et al. (1998) in combined feeds. They reported that mean concentrations of di-n-butyl phthalate (DBP) and di-2-ethylhexyl phthalate (DEHP) and the sum of DBP and DEHP were 0.207; 0.216 and 0.423 (mg.kg^{-1} of original sample). Our results are similar as were found by JAROŠOVÁ, A. – MALYSZOVÁ, J. (2005). They reported that concentrations of DEHP 0,07 – 1,77 and DBP 0,06 – 2,36 mg.kg^{-1} had been found in feeding stuffs for pigs, cattle and poultry.

Conclusion:

Results reached in this work will be served as for monitoring esters of phthalic acid in components and feeds in the Czech Republic as for development of analysis method. This conclusion is same as LATINI'S (2005) opinion, which said that the environmental phthalate monitoring should be continued and their maximum allowed concentrations should be prescribed by regulations.

The work was supported by the Czech National Agency for searching in Agriculture. Project No.: QG60066/2005.

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