Analytical From Fish Bioconcentration Studies

PROJECT NO. 9G-706-12.600 3M TECHNICAL NOTEBOOK NO. 41947 21 PAGE

Subject: Bioaccumulation of FM-3422 in Channel Catfish Date: 1/15/76

Object: A Significant problem with this work is that nowhere was it demonstrated that Solvent extracts of fish Tissue were quantitative SMRC 3-4-83

Reference:

Three groups of 5 channel catfish were exposed for 32 days to Control water, 400ml acetone on marbles, and 16g FM-3422 + 400ml Acetone sprayed on marbles in 16ℓ Tank.

The fish after 32 day exposure were dried and wt. taken

Control	Acetone Only	FM-3422 + Acetone
3.95g	4.65g	4.77g.

The three (5 lots) groups were digested with ~25ml 20% KOH at 50°C for 1 hr. The three digested samples were then diluted to ± 50ml and extracted with 10 ml N-Octanol. G.Ced for FM-3422

50ml aliquots of the tank water was extracted with 10ml octanol. also for G.C. analysis,

All samples G.C.ed see FC 1/19/76 to yield

Water - mg/ℓ FM-3422	Fish - mg/g - dried fish
Control - Nil	Control - 0.0015mg/g
Acetone - Nil	Acetone - 0.0006mg/g
FM-3422 - 0.17mg/ℓ	FM-3422 - 0.161mg/g

Signature Greg Vrospir Date: 3/8/78

If I [illegible] 2-28-78

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PROJECT NO. 99706126000 3M TECHNICAL NOTEBOOK NO. 41947 23

Subject: Date: 2/19 - 2/20/76

Object:

Reference: LR 3114 was to determine the uptake of FM-3422 by channel catfish in at 1,000 ppm tank (static) 33day Exposure Verify the fish test. Page 21

Samples submitted - Test Water (1,000ppm)

Fish were gutted — Fresh gutted wt.

- Fish ① - 3.20g
- Fish ② 3.46g
- Fish ③ 2.58g

Test water sample was extracted with 10ml Oct per 10ml H_2O

Fish were digested in 10ml each of 20% kOH by heating to ~50°C for 1hr. Then extracted with 10ml Oct.

Extracts G Ced See integrator reports 043-048

to yield Test Water 4.86mg/l

Fish ① - 0.31mg/g - Frsh gutted wt.

Fish ② - 0.24 mg/g - " " "

Fish ③ - 0.46mg/g - " " "

The test water was filtered through a Millipore 0.22µ filter and 50ml water extracted with 2ml Oct. G.Ced integrator report 049-051 to yield 0.011 mg/l.

Signature M.O. Vaupir Date: 3/8/76

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PROJECT NO. 9970612600 3M TECHNICAL NOTEBOOK NO. 41947 24
Subject: Date: 2/23 - 2/25/78

Object:

Reference: LR 3114 - The second part of the uptake bioassay was the removal of the fish from the FM-3422 tank and placement of them into ~~the~~ a tank of clean H_2O to determine the cleaning effect if any.

Samples submitted - Control Water

	Fresh gutted wt.
Recovery tank Water	
Control Fish #1	4.63g
Control Fish #2	4.80g
Exposed fish #1	3.90g
Exposed Fish #2	3.66g

Water samples extracted 2ml/10ml Oct/H_2O

Fish digest as earlier and extracted with 10ml oct.

Sample ext. G Ced Inj reports - 061-067 to

yield Control Water - <0.01mg/l FM-3422

Recovery Water - 0.033mg/l "

Fish

C-1 - 0.00067mg/g "

C-2 0.00097mg/g "

F-1 0.029mg/g "

F-2 0.031mg/g "

Signature K. O. Vroegindewey Date: 3/8/78

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PROJECT NO. 9970612600 3M TECHNICAL NOTEBOOK NO. 41947 25

Subject: Date: 2/23 - 2/24/76

Object:

Reference: Some question was raised to the validity of the 1ml/50ml Oct/H_2O extraction ratios used to obtain the lower detection limits.

A 0.5mg/l soln of FM-3422 in H_2O was made by dispersing the FM-3422 via acetone

1st, 2nd, & 3rd extractions were made with the 1ml/50ml ratio GC - Int. reports - 052-057

The yield was 1st - 86% } Total = 0.438mg/l
2nd 12% }
3rd 2%

100% recovery in 3 successive 1:50 ml extracts of H_2O

Same procedure was tried on a sat water soln of FM-3422. G.C. int. reports 058-059

To yield 1st ~~85%~~ 89% 7.5mg/l } 8.4mg/l
2nd ~~12%~~ 10% 0.84mg/l }
3rd 1% 0.06mg/l }

Signature G.O. Vrespin Date: 3/8/78

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Subject: Date: 3/8/77

Object:

Reference:

3 samples were drawn from the FM-3422 fish tank. These samples were taken at the same levels as the initial samples and will be analyzed for FM-3422

Samples ① Day 8 ② Day 8 ③ Day 8

Mo gave me 2 digested bluegill fish samples

Day 8 Control fish 1.15g Exposed Fish 0.977g.

Water samples and fish samples were taken on 3/14/77 - 14 days of exposure.

Control 3/14/77 - 3 fish wt.= 1.434g.

Exposed 3/11/77 - 3 fish wt.= 1.172g.

Fish samples were extracted with 2 - 10ml portions of ethyl Acetate and then the 2 - 10ml aliquots were diluted to 25ml. Digested fish sample were pH adjusted with H_2SO_4 conc. to pH 7-8

Fish samples G.C. Int Integrator reports - 051 - 054

Results: Control fish - No background

	Total wt. fresh fish	Total mg FM-3422	FM-3422
Exposed Fish 8 days 3/8/77 -	0.977g	0.115mg	- 0.118 mg/g
Exposed Fish 14 days 3/14/77 -	1.172g.	0.137mg	- 0.117 mg/g FM

See page 54 for 21 day Exposure.

Signature Gary Vroman Date: 3/21/77

M.T Shadroui 3/20/77

PROJECT NO. 9970612600 3M TECHNICAL NOTEBOOK NO. 46269 35

Subject: FM-3422 Uptake in Channel Cat fish Date: 2/1/78

Object: G.C. analysis of water and fish samples submitted by Al Welter for FM-3422. ~~EC~~ Electron Capture

Reference: G.C. conditions used for study: Column 6'x⅛" SS on column injection Chromosorb W A.W. 60/80 mesh CW 20m - 10% Flow: Argon methane 40 ml/min Temp.: Inj. Port 200°C Det. 300°C Oven 170°C Isothermal

Standards run on each day of analysis. Std. w/v in ethyl acetate. Water samples extracted with two portions of ethyl acetate. 10ml of sat NaCl soln added to salt out organics. 100ml water sample extracted & diluted to 10ml.
Tissue sample fine minced in ethyl acetate prior to submittal for G.C. analysis. The ethyl acetate was tranfered to volumetric flasks and subsequent washes made with E.A. then diluted to volume. The study involved Uptake - coded "U" and clearance coded "Cl".

G.C. analysis of 2/1/78

Stds.	10ppm FM-3422	1ppm
Integrator Values.	Ave. 1,261,386	126,838

Uptake

Water Sample

20hr.	Sample size	Final Vol.	ppm FM-3422	
Bottom	100ml	10ml	0.347	
Middle	100ml	10ml	0.342	0.309
Top	80ml	10ml	0.514	0.478

Fish	Wt.	Final Vol.	FM-3422 (µg/g)	
20hr.	~~10.309g.~~			
Whole 1	10.309g.	50ml	62.0	60.6
Whole 2	10.111g.	50ml	75.2	73.0
16hr				
Whole 1	13.678g.	"	53.0	48.4
Whole 2	13.185g.	"	59.6	53.6

Signature GO Vroman Date: 3/7/78

Read and Understood IT [illegible]