

Research Overview

National Water Quality Laboratory

USGS

Lakewood, CO

NRP/LEAG Meeting

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Jeff McCoy

Chief, Methods Research & Development Program

Agenda

- Quick overview of the NWQL
- Ongoing research at the NWQL
- Snapshots of a few current projects
- Future directions...

The NWQL: What we do...

- **Develop modern methods for environmental analysis**
- **Analyze water, wastewater, soil and tissue in support of international, national and state programs**
- **Collaborative research with other government agencies**

NWQL Areas of Expertise

- **Methods Research and Development**
- **Inorganic, Organic and Radiochemistry**
- **ID & Quantitation of Benthic Invertebrates**
- **Varied Matrices:**
Water, Wastewater, Sediment, Tissue
- **Admin, Support Services, QAS, IT, BDT**

Key Inorganic Chemistry Capabilities

- **Nutrients: Ortho-P, ammonia, nitrate, nitrite**
- **Metals: whole & filtered water: ICP & ICP/MS**
- **Bromide: filtered by IC**
- **Iodide, silica: filtered by colorimetric flow**
- **Fluoride: filtered by ISE**
- **Major anions: filtered by IC**
- **Radchem, Hg, As speciation**
- **Etc., etc. – see online catalog at nwql.usgs.gov**

Key Organic Chemistry Capabilities

- Pesticides
- Volatiles
- Emerging Contaminants
 - Wastewater Indicators
 - Pharmaceuticals
 - Hormones
 - Endocrine Disruptors
- High Production Volume chemicals

Biological Services

- Macroinvertebrate Sample Production
 - Subsampling & Sorting
 - Taxonomic Identification
 - ~ 800 samples/yr.
 - Key to USGS ecosystems focus



Method Development at the NWQL



Methods Research at the NWQL:

Analytical Focus

- **Improved integrative sampling technology**
 - Suspended Sediment Integrative Samplers
 - Polar Organic Chemical Integrative Sampling (POCIS)
- **Improved Sensitivity and Selectivity:**
 - Direct Aqueous Injection LC/MS/MS
 - High mass resolution TOF/MS
 - Isotope dilution MS/MS
- **Green Chemistry, Automation & Efficiency:**
 - ASE Sediment/Tissue Prep Methods
 - Replacement of Cadmium w/ nitrate reductase
 - Kone Discrete analyzer

Ongoing Research Projects (incomplete list)

- **Integrated suspended sediment samplers**
- **Pharmaceuticals in water, tissue and sediment**
- **Wastewater indicators from biosolids**
- **Persistence of emerging contaminants through the waste treatment processes**
- **Hormones in water, sediment and tissue**
- **High Production Volume chemicals in water**
- **Elimination of Cadmium with NO_3^- Reductase**
- **Silica: move from SFF to Discrete method**



Glyphosate in suspended sediment in streams from two agricultural areas of the United States

Mark Sandstrom, Max Stroppel, **Michael Meyer**,
Claire Rose, Richard Coupe, and Steven Kalkhoff

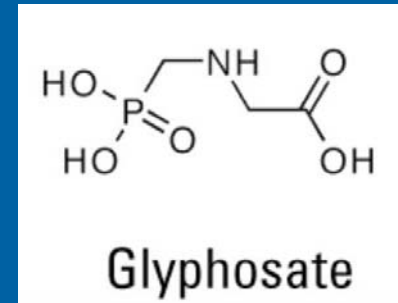
U.S. Geological Survey

(SETAC, 2008)

Introduction

- **Glyphosate-based herbicides are used for control of weeds on corn, soybeans, and cotton.** (High volume, broad geographical usage)
- **Glyphosate is strongly adsorbed by soils and stream sediment.**
- **Dissolved glyphosate has been found frequently in tile drains and streams in agricultural areas (Battaglin and others, 2005).**

Study Objectives



- Evaluate importance of suspended sediment in transport and fate of glyphosate from agricultural fields into streams
- Evaluate time-integrating sediment samplers ability to collect fine-grained suspended sediment

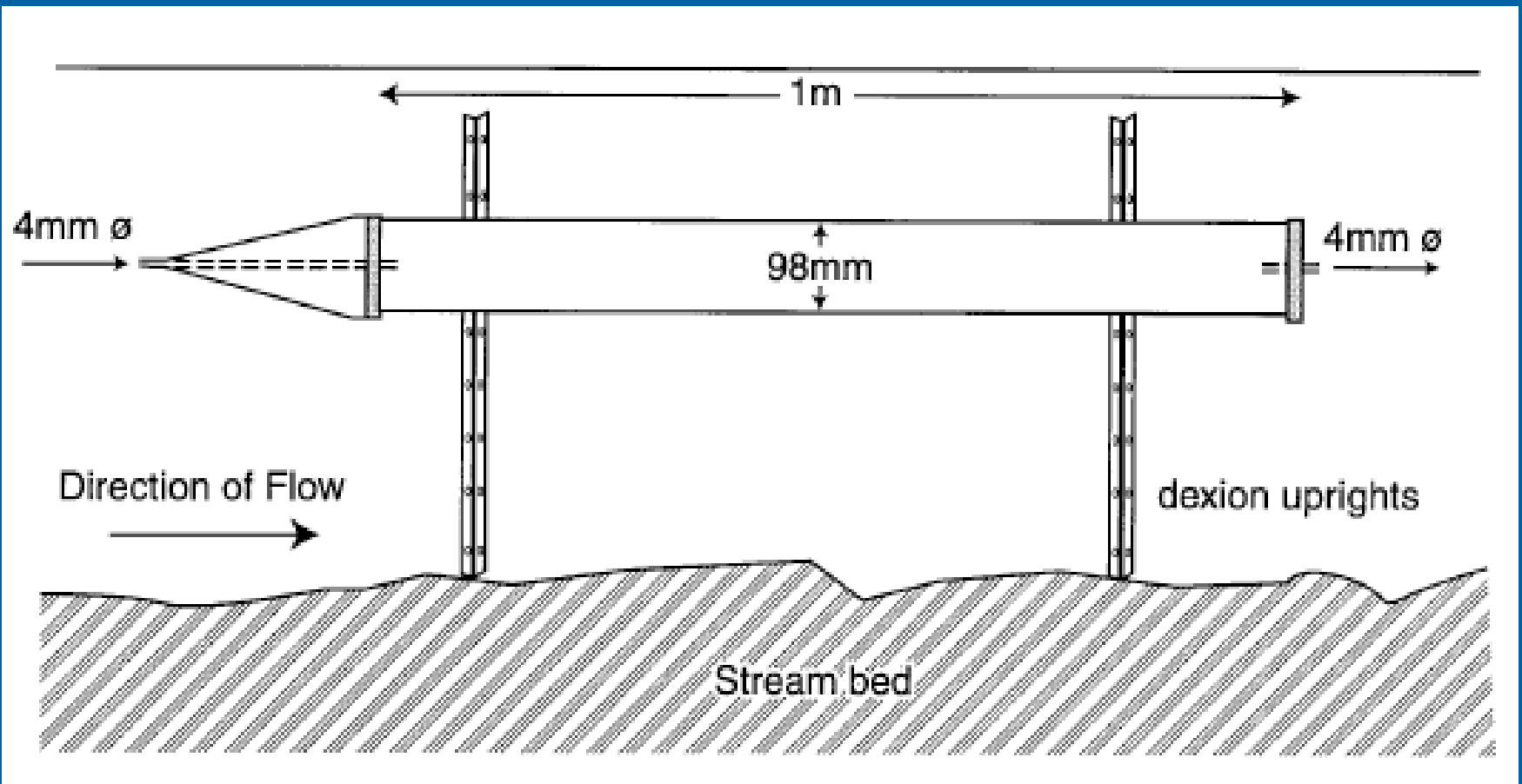
Time-integrated suspended sediment sampler for small watersheds

- Simple design based on sedimentation of particles (Phillips, et al, 2000)
- Operates unattended, no power requirements
- Composite sample collected continuously over period of days to weeks
 - Samples entire storm runoff event
- Collects sufficient mass for variety of geochemical analyses



Phillips, J.M., Russell, M.A., and Walling, D.E., 2000, Time-integrated sampling of fluvial suspended sediment: a simple methodology for small catchments: *Hydrological Processes*, v. 14, no. 14, p. 2589-2602.

Time-integrated suspended sediment sampler for small watersheds



Suspended sediment sampler operation

- **Sampler submerged in stream with inlet orientated directly into the flow**
- **Within main cylinder flow velocity is reduced by ~600 times ambient flow**
- **Reduction in flow velocity induces sedimentation**

Fraction of total glyphosate in suspended sediment

Stream Site	Sample Event	Glyphosate in sediment samplers, in ng/g	Median concentration of suspended sediment in mg/L	Calculated glyphosate in suspended sediment in ng/L	Median concentration of glyphosate in filtered stream samples, in ng/L	Glyphosate in suspended sediment, in percent of total glyphosate in water
Blairsburg, IA	1	111	44	4.9	80	6.1
	2	172	34	5.8	675	0.9
New Providence, IA	1	37	19.5	0.7	60	1.2
	2	79	204	16.1	320	5.0
Tommie Bayou, MS	1	1043	110	114.7	890	12.9
	2	437	-	-	-	-
Bogie Phalia, MS	1	1755	86	150.9	1,690	8.9
	2	1528	81	123.8	1,015	12.2

Conclusions

- Time-integrating sediment samplers were effective in the collection of suspended sediment during major storm-runoff at each location.
- Glyphosate in suspended sediment represented a small fraction (1 - 12 percent) of the total glyphosate concentration in the stream.

Fate of Antidepressants Downstream of the Boulder Creek, CO Wastewater Treatment Facility

- Ed Furlong, Melissa Schultz - NWQL
- Note: data is preliminary, research ongoing
- Matrices: water, fish brain, sediment
- Bottom line: Are differences seen due to metabolism in fish? Due to chemical property differences in water vs. sediment?

Boulder Creek Antidepressants: Comparison Between Media Downstream of WW Outfall

	Water (ng/L)	Brain (ng/g)	Sediment (ng/g)*
Fluoxetine	9	0.6	12
Norfluoxetine	4	0.9	2.3
Sertraline	3	1.5	11
Norsertaline	4	2.5	8.7
Paroxetine	4	ND	2.4
Citalopram	60	0.07	10
Bupropion	50	0.05	1.7
Venlafaxine	220	ND	21
*Provisional Results			



Land Application of Biosolids - II

Mobilization of Microconstituents from Land-Applied Biosolids via Simulated Rainfall

- Ed Furlong, (NWQL), Thomas Borch, Jessica Davis (CSU), Tracy Yager (USGS Colorado WSC)
- Coordinated with Metro Wastewater Reclamation District
- First-time application of biosolids (Roggen,CO)
- Artificial rainfall applied; runoff collected..
- Samples analyzed for:
 - Wastewater indicators
 - Pharmaceuticals, hormones
 - Nutrients, Organic carbon



Mobilization of Wastewater-Indicator Compounds From Biosolids Treated Sites - Simulated Rainfall

Elapsed time following application	1 day	8 days	35 days
Average Triclosan Concentration ug/l			
Rainfall Simulation - Beginning	0.25	0.20	0.38
Rainfall Simulation - Middle	0.33	0.34	0.39
Rainfall Simulation - End	0.31	0.32	0.50

Mobilization of Wastewater-Indicator Compounds From Biosolids Treated Sites - Simulated Rainfall

Elapsed time following application	1 day	8 days	35 days
Average DEET Concentration ug/l			
Rainfall Simulation - Beginning	0.14	0.48	ND
Rainfall Simulation - Middle	0.10	0.59	ND
Rainfall Simulation - End	0.09	0.33	ND

Future Directions in Method Development

- Collaborate with ecosystem-focused studies
 - Ecological biomagnification / metabolism studies
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- Data Mining / Automation
 - Direct Aqueous Injection

LC/MS/MS

- Pharmaceuticals
 - HPV Compounds
 - Pesticides
 - Polar Indicators
- LC/ToF MS for TICs

