

#### FHWA/IN/JTRP-2010/19

**Final Report** 

#### **EVALUATION OF TOXICITY ANALYSIS FOR FOUNDRY SAND SPECIFICATIONS**

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August 2010



# **TECHNICAL** Summary

INDOT Research

Technology Transfer and Project Implementation Information

Publication No.: FHWA/INDOT/JTRP-2010/19

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#### EVALUATION OF TOXICITY ANALYSIS FOR FOUNDRY SAND SPECIFICATIONS

#### INTRODUCTION

In the United States, over 2,300 foundries produce castings with uses ranging from automobiles to hand tools to military applications. Foundry sands are used to form molds into which molten metal is poured. This process uses sand molds, hardened onto reusable patterns by compaction and binders. These sands are reused multiple times but eventually the angular edges are rounded and the sand is no longer usable in the foundry industry. Over one hundred million tons of sand are used annually with six (6) to ten (10) million tons disposed terminally. The majority of this sand is currently placed in landfills with an EPA estimated 15% recycled.

In 2001, only ten percent of the waste sand from foundries was reused outside of the foundry industry. The remaining sands were either discarded in landfills or stockpiled on site. This discarded sand often is usable and sometimes superior to virgin materials. The benefits gained from the recycling of foundry sands are well documented for both the producers and users. These benefits include reducing the disposal cost for the foundries which in turn lowers operating costs, reducing the necessity to mine virgin materials, providing low cost alternatives for end users, and extending the construction season by providing a material with a lower freezing temperature. The U.S. Department of Transportation Federal Highway Administration estimates that contractors would save 25 to 30 percent using foundry sand over virgin material (US Dept. Transportation, 2003). Additionally, the decrease in land fill materials increases the lifetime of current landfills.

Of concern to the end users of foundry sand is the potential toxicity of recycled foundry sand (RFS) leachate. Indiana is one of the top ten foundry production states in the U.S. and has a progressive and comprehensive set of protocols in place to ensure safe and environmentally conscientious use of RFS.

Using recycled foundry sand by transportation agencies as fill and foundation material is considered advantageous for both parties, although the potential liability is a consideration in waste placement. In accordance with protection of the public from potential risk, use of RFS is limited to that material classified as Types III or IV. Classification of sand as Type III and IV foundry sand is restricted based on heavy metal content (As, Ba, Cd, Cr, Pb, Hg, Se, Ag by the Toxicity Leaching Characteristic Procedure) and secondary drinking water parameters (Cl, Cu, total CN, Fl, Fe, Mn, Ni, pH, phenols, Na, sulfate, total S, TDS, Zn by Indiana neutral leachate assessment).

INDOT Document ITM 215-02T provides the detailed procedure for Microtox assessment of waste foundry sand based on a 1998 JTRP report. Recently, INDOT was presented with concerns that mandating the Microtox test prior to the use of RFS in transportation projects was too restrictive: a) Indiana seems to be the only state that requires a biological toxicity test of any kind in the recycling of foundry sands; b) Microtox may not be suited for application to foundry sands; c) the addition of the Microtox requirement seems redundant because a TCLP test is already required; and d) few labs exist in Indiana that perform the Microtox test.

Each state has rules and regulations to identify the possible uses of RFS. Additionally, each state's Department of Transportation has published specifications for the physical and chemical characteristics that a material must meet before reuse. Thus, a review and comparison of regulations of the various states is one of the tasks of this project. We also reviewed the scientific literature to evaluate the efficacy of the Microtox test.

The overall objective of this study is to provide perspective concerning the INDOT requirement of a Microtox test for RFS.

#### FINDINGS

The review of regulations for various states suggests that Indiana falls in the middle of the range of rigor for foundry-sands testing prior to recycling; of course, many states have no foundry sand recycling program at all. Some states have minimal requirements while others are very aggressive in their testing programs. As suspected prior to this study, Indiana is the only state to require a biological toxicity test.

As part of the execution of this project, we polled 15 commercial laboratories in Indiana that analyze water and soil for their ability to provide a Microtox analysis. None of these laboratories currently provide this analysis, though some of them suggested that they once did. STL Valparaiso indicated that Microtox was available in the recent past, and they still possess the equipment. However, they no longer have the technical expertise to run the test. Two of the labs provided contact information for out-of-state laboratories analyze that for Microtox; unfortunately, these labs actually do not provide the service.

The current Microtox requirement for recycling foundry sand in Indiana is viewed by some in the industry as excessive. However, Microtox is readily defensible from a scientific perspective, and many studies suggest that Microtox should be coupled with at least one other biological test to be fully encompassing.

Strictly from viewpoint of environmental protection, the inclusion of Microtox makes sense. The test has the sensitivity to detect potentially toxic agents in recycled sand that might escape chemical analysis. The test, therefore, provides a layer of assurance that otherwise would be absent. From the perspective of the foundry industry, the Microtox test is an unneeded hurdle that could potentially block the beneficial use of spent foundry sand. Cost is one consideration, but the lack of local analytical facilities for the Microtox is particularly troublesome. Subobjectives were to a) provide a brief review of various states' rules and regulations concerning the use of Recycled Foundry Sand specifically as they pertain to Department Of Transportation projects and b) Review the scientific literature to determine the efficacy of Microtox and its applicability to RFS.

#### IMPLEMENTATION

Our recommendation is that the Microtox test be retained by INDOT, but we suggest the following:

- a) If at all possible, minimize the number of samples of foundry sand that must be tested. Periodic testing is critical to ensure protection of the environment, but in the absence of changes in foundry processes, it might be possible to reduce the frequency of sampling and testing.
- b) A consistent, readily available laboratory needs to be established to

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Research & Development, INDOT 1205 Montgomery Street West Lafayette, IN 47906 Phone: (765) 463-1521 Fax: (765) 497-1665 tnantung@indot.in.gov ensure rapid turn around of analyses and reduced costs. Two problems exist for commercial establishing a service for Microtox: the demand is low and some dedicated equipment is needed to perform the test.

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#### 16. Abstract

Byproducts from many industries have the potential to be used as construction materials, but some means is required to determine if the material is environmentally benign. Foundry sands are produced in many states and can be useful as in transportation projects. However, INDOT currently requires the use of the MICROTOX test to assess the potential toxicity of the sands, and this requirement is viewed as an unnecessary impediment by the producers of foundry sands and is a requirement not encountered in other states. Therefore, the goal of this project was to review current requirements for testing of recycled materials, determine the availability of MICROTOX testing, and to make recommendations concerning the continued use of MICROTOX as an assessment tool. Strictly from viewpoint of environmental protection, the inclusion of Microtox makes sense. The test has the sensitivity to detect potentially toxic agents in recycled sand that might escape chemical analysis. The test, therefore, provides a layer of assurance that otherwise would be absent. From the perspective of the foundry industry, the Microtox test is an unneeded hurdle that could potentially block the beneficial use of spent foundry sand. Cost is one consideration, but the lack of local analytical facilities for the Microtox is particularly troublesome. Our recommendation is that the Microtox test be retained by INDOT, but we suggest the following:

- a) Minimize the number of samples of foundry sand that must be tested possibly by reducing the frequency of sampling and testing.
- b) A consistent, readily available laboratory needs to be established to ensure rapid turn around of analyses and reduced costs. Currently, the demand is low and some dedicated equipment is needed to perform the test.

One of the potential outcomes of this project discussed during the negotiations for this project was a possible follow-up project in which we would investigate the modifications to the bioassay. This might include exploring alternatives to Microtox or simplifications of the Microtox test. We remain open to this possibility, but from the scientific point of view, such a follow-up may not be necessary. Of all the bioassays we reviewed, Microtox seemed to be the most widely used (though not for foundry sands), and we found no evidence that other bioassays were being offered routinely at commercial

labs				
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Principal Investigator (PI): M. Katherine Banks

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INDOT Strategic Goal Impact Areas (select all that the implementation of this project will impact):

Mobility Safety Economic Dev

*Economic Development Customer Service* 

Resource Management Training

Summary of Implementation Plan: The MICROTOX test has been shown to be valuable in determining potential, hidden toxicity. However, it is recognized that the lack of facilities to peform this test is a signifiacnt impediment to those wishing to recycle foundry sand. We suggest that INDOT either assists foundry operators in finding/establishing a service lab through either helping with anaytical costs for laboratories out of state or helping to establish the service at a laboratory within Indiana. If INDOT is not willing to assist in this way, we suggest that the requirement for the MICROTOX test be abandoned.

Note: If more than one implementor recommended, please fill in the information on each implementor's implementation items:

Responsible for Implementing: Evaluating the practicality of INDOT helping with the establishing of a MICROTOX lab in Indiana, assisting financially with analytical costs for out-of-state labs, or simply abandoning the MICROTOX test.

Help or resources needed for implementation (e.g., help from PI, funding, equipment, etc.): Fuinding would be required for MICROTOX instrumentation and supplies. OR Funding would be needed for deferring some of the costs for outside labs.

Name of Implementor/User: Signature:\_\_\_\_\_

Responsible for Implementing:

Help or resources needed for implementation (e.g., help from PI, funding, equipment, etc.):

Name of Implementor/User: Signature:\_\_\_\_\_

Responsible for Implementing:

Help or resources needed for implementation (e.g., help from PI, funding, equipment, etc.):

Signatures of SAC members:\_\_\_\_\_

Please send a copy of this form to the INDOT Research & Development Office and FHWA with the final report.

## **Evaluation of Toxicity Analysis for Foundry Sand Specifications**

### PART I. STATE GUIDELINES FOR RECYCLING FOUNDRY SANDS

#### **Background and Rationale**

<u>Recycling of Waste Foundry Sand</u> In the United States, over 2,300 foundries produce castings with uses ranging from automobiles to hand tools to military applications. Foundry sands are used to form molds into which molten metal is poured. This process uses sand molds, hardened onto reusable patterns by compaction and binders. These sands are reused multiple times but eventually the angular edges are rounded and the sand is no longer usable in the foundry industry. Over one hundred million tons of sand are used annually with six (6) to ten (10) million tons disposed terminally. The majority of this sand is currently placed in landfills with an EPA estimated 15% recycled. Figure 1 is a flow chart of this process.



Figure 1. The process that generates waste foundry sand (from Bastian and Alleman, 1998).

The foundry process generates several types of waste sands, but two seem to be the most prominent. The "green sands" account for roughly 90% of the sands produced, and "resin sands" are the second most prevalent. The primary difference between these types of sands is the binder

for the sand when in the mold. The binding agent for green sands is clay, comprised of 85 to 95% silica sand, 4 to 10% bentonite clay (the binding agent), 2 to 10% carbonaceous additive, and 2 to 5% water. Resin sands generally use an organic binder, although alternative systems use inorganic binders. Chemically bonded sands are generally light in color and coarser in texture than clay bonded sands.

In 2001, only ten percent of the waste sand from foundries was reused outside of the foundry industry. The remaining sands were either discarded in landfills or stockpiled on site. This discarded sand often is usable and sometimes superior to virgin materials. The benefits gained from the recycling of foundry sands are well documented for both the producers and users. These benefits include reducing the disposal cost for the foundries which in turn lowers operating costs, reducing the necessity to mine virgin materials, providing low cost alternatives for end users, and extending the construction season by providing a material with a lower freezing temperature. TheU.S. Department of Transportation Federal Highway Administration estimates that contractors would save 25 to 30 percent using foundry sand over virgin material (US Dept. Transportation, 2003). Additionally, the decrease in land fill materials increases the lifetime of current landfills.

<u>Microtox Luminescent Bacterial Assay</u> Typical Microtox protocols call for the exposure of the marine, luminescent bacterium, *V. fischeri*, to liquid samples that have been adjusted osmotically with sodium chloride. The samples often are serially diluted to identify the range of biosensitivity and to possibly determine  $EC_{50}$  or  $LC_{50}$  values. Freeze-dried *V. fischeri* are rehydrated prior to their use, temperature is controlled usually to 15 °C, adjusted a range of 6 to 8, and the salt content adjusted to approximately 2% to simulate seawater.

Modifications to the original protocol were necessary to allow application of Microtox to soils, sediments, and similar solid phases. Generally, the sediment or soil is diluted by mixing 7 g soil with 35 mL of 2% sodium chloride (osmotic adjusting solution). The slurry is mixed for a predetermined time, and samples for testing with the bacterium are obtained by transferring a measured volume of the slurry while mixing the slurry. Solids in the slurry will interfere with the luminescence measurement and add challenges to determining an absolute toxicity measurement. Therefore, it is necessary to include control samples containing solids that have the same particle distribution and similar color (uncontaminated soil of the same soil series is ideal). In some instances, the soils are heavily contaminated and require such a large dilution that the impact of the solids becomes minimal. An alternative to this approach is to prepare an extract of the solids using water or an organic extractant and using the extract directly for Microtox measurements.

<u>Testing of Foundry Sands Prior to Reuse</u> Of concern to the end users of foundry sand is the potential toxicity of recycled foundry sand (RFS) leachate. Indiana is one of the top ten foundry production states in the U.S. and has a progressive and comprehensive set of protocols in place to ensure safe and environmentally conscientious use of RFS.

Using recycled foundry sand by transportation agencies as fill and foundation material is considered advantageous for both parties, although the potential liability is a consideration in waste placement. In accordance with protection of the public from potential risk, use of RFS is limited to that material classified as Types III or IV. Classification of sand as Type III and IV foundry sand is restricted based on heavy metal content (As, Ba, Cd, Cr, Pb, Hg, Se, Ag by the Toxicity Leaching Characteristic Procedure) (Table 1) and secondary drinking water parameters

(Cl, Cu, total CN, Fl, Fe, Mn, Ni, pH, phenols, Na, sulfate, total S, TDS, Zn by Indiana neutral leachate assessment) (Table 2).

Constituent	Classification						
	Type IV	Type III	Type II	Type I			
		mg/	′L				
Arsenic	≤0.05	≤0.5	≤1.25	≤5.0			
Barium	≤1	≤10	≤25	≤100			
Cadmium	≤0.01	≤0.1	≤0.25	≤1.0			
Chromium	≤0.05	≤0.5	≤1.25	≤5.0			
Lead	≤0.05	≤0.5	≤1.25	≤5.0			
Mercury	$\leq 0.002$	$\leq 0.02$	≤0.05	≤0.2			
Selenium	≤0.01	≤0.1	≤0.25	≤1.0			
Silver	≤0.05	≤0.5	≤1.25	≤5.0			

Table 1. Indiana restricted waste criteria for parameters using TCLP (Indiana Administrative Code Section 329, Article 10, "Solid Waste Lands Disposal Facility Classification," Rule 9, Part 4. ( 329 IAC 10-9-4)).

Current INDOT requirements limit the use of recycled foundry sand (defined as a mixture of residual material used from ferrous or non-ferrous metal castings and natural sands) [INDOT Document 200-R-401] to the following:

- Recycled waste sand (RFS) derived from Type III residual sand shall not be permitted within 30 m (100 ft) horizontally, of a stream, river, lake, reservoir, wetland, or any other protected environmental resource area
- RFS from Type III or Type IV residual sand shall not be placed within 50 meters (150 ft), horizontally, of a well, spring, or other ground sources of potable water.
- RFS shall not be permitted adjacent to metallic pipes, or other metallic structures
- RFS shall not be used as encasement materials
- RSF shall not be used in MSE wall applications

In addition to the general requirements for the material, approval requirements are:

- Current MSDS and summary of specified tests
- Name of testing facility
- Dates of sampling and testing results
- Test method used for IDEM classification
- Letter from IDEM indicating waste classification of materials
- Test results for leachate
- Test results for Microtox (ITM 215)

- Stockpile sampling locations
- Gradation test results
- Hydraulic conductivity (permeability) test results
- Recycled foundry sand source certification

Table 2. Indiana restricted waste criteria for parameters using the EP Water Test (Indiana Administrative Code Section 329, Article 10, "Solid Waste Lands Disposal Facility Classification," Rule 9, Part 4. ( 329 IAC 10-9-4).

Constituent	Classification					
	Type IV	Type III	Type II	Type I		
		mg	g/L			
Barium	≤1	≤10	≤25	*		
Boron†	≤2	≤20	≤50	*		
Chlorides	≤250	≤2,500	≤6,250	*		
Copper	≤0.25	≤2.5	≤6.25	*		
Cyanide (total)	≤0.2	≤2	$\leq 5$	*		
Fluoride	≤1.4	≤14	≤35	*		
Iron	≤1.5	≤15	*	*		
Manganese	≤0.5	≤0.5	*	*		
Nickel	≤0.2	≤2	$\leq 5$	*		
Phenols	≤0.3	≤3	≤7.5	*		
Sodium	≤250	≤2,500	≤6,250	*		
Sulfate	≤250	≤2,500	≤6,250	*		
Sulfide	≤1	55	≤12.5	*		
Total dissolved Solids	≤500	≤5,000	≤12,500	*		
Zinc	≤2.5	≤25	≤62.5	*		
pH‡	≤6-9	≤5-10	<u>≤</u> 4-11	*		

<sup>†</sup>Not included in 1996 Indiana Department of Environmental Management Edition ‡Acceptable range (standard units).

\*Testing not required.

The INDOT Document ITM 215-02T provides the detailed procedure for Microtox assessment of waste foundry sand. This methodology was developed based on a 1998 JTRP report (Partridge and Alleman, 1998). More recently, INDOT was presented with concerns that mandating the Microtox test prior to the use of RFS in transportation projects was too restrictive: a) Indiana seems to be the only state that requires a biological toxicity test of any kind in the recycling of foundry sands; b) Microtox may not be suited for application to foundry sands; c) the addition of the Microtox requirement seems redundant because a TCLP test is already required; and d) few labs exist in Indiana that perform the Microtox test.

Each state has rules and regulations to identify the possible uses of RFS. Additionally, each state's Department of Transportation has published specifications for the physical and chemical characteristics that a material must meet before reuse. Thus, a review and comparison of regulations of the various states is one of the tasks of this project. We also reviewed the scientific literature to evaluate the efficacy of the Microtox test.

#### **Objectives**

The overall objective of this study is to provide perspective concerning the INDOT requirement of a Microtox test for RFS. Subobjectives are:

- Provide a brief review of various states' rules and regulations concerning the use of Recycled Foundry Sand specifically as they pertain to Department Of Transportation projects.
- Review the scientific literature to determine the efficacy of Microtox and its applicability to RFS.

#### **Approach and Methodologies**

<u>RFS Regulations</u> The evaluation of regulations from various states guiding the beneficial use of RFS was approached in three ways: 1) Information readily available via state and federal websites concerning the regulations on the use of RFS was reviewed, 2) Information from secondary sources and private industry was reviewed, and 3) Personal communication via e-mail and telephone calls was used to verify the information already gathered. Once this information was compiled, each state's regulatory process was divided into its components so as to be more easily compared to other states. The data were split\_into trends in regulations and examined with special care being given to anomalies such as extremely aggressive, lax, or novel approaches. Finally, the information was compiled and the sources indexed for easy reference.

<u>Review of the Scientific Microtox Literature</u> Our approach was to summarize existing comprehensive reviews of the Microtox procedure. The reviews were evaluated extensively and cited publications were collected. In addition, literature concerning the application of Microtox was reviewed for articles published from 2006 to 2008.

#### Results

<u>RFS Regulations</u> Examination of the regulations for the reuse of foundry sand reveals that while regulations set forth by each state's respective environmental agency are as diverse as the states from which they come, they all have the common goal "to ensure the protection of human health and the environment by identifying and minimizing potential risks of reusing industrial wastes" (EPA 2002). The majority of the programs are based upon the characterization of the waste and/or leachate and the acceptance or rejection of that waste for reuse. Table 3 summarizes the regulations from a representative group of states. The U.S. EPA document, State Toolkit for Developing Beneficial Reuse Programs for Foundry Sand (2006), compares state regulations for reuse of foundry sand. In this document, the EPA describes the reuse programs: case-by-case

studies, waste classification, and hybrid programs. For the case-by-case programs, the state reviews each request individually. In waste classification area, each state categorizes the foundry sand based on the concentration of specific elements in the leachate. In a hybrid system, some uses are reviewed individually while other requests are streamlined. Another important characteristic of reuse programs is the leachate threshold concentrations that the state will allow. These range from percentages of the Resource Conservation and Recovery Act Toxicity Characteristic (TC) levels to variations of the Drinking Water Standards (DWS).

The regulations set forth by the state environmental agencies are not the sole focus of this study; the most relevant regulations are those adopted by the departments of transportation for the protection of the land and human health. As can be seen in Table 3, not all states that allow reuse of foundry sand encourage the use of foundry sand for Department of Transportation (DOT) projects. The DOT regulations concerning the use of foundry sand range from Alabama's brief specification/definition of waste foundry sand to Iowa's extensive list of requirements. Indiana's Department of Transportation has implemented a rigorous set of tests and policies. No state other than Indiana requires a biological toxicity test.

Alabama has the least stringent Department of Environmental Management (DEM) and DOT regulations. The DEM employs the Waste Classification approach which categorizes foundry sand into one of two classifications. A completed Solid/Hazardous Waste Determination Form is also required. TCLP is used to chemically characterize the foundry sand. If the TCLP results are less than 50 percent of the TC levels for metals, then the material is useable. If the foundry sand exhibits higher levels, then it is not used. Table 4 compares the acceptable concentration levels of the various states, and Alabama's concentration limits are consistently higher than those of the remaining states. Also, Alabama imposes siting restrictions on all foundry sand use. The Alabama DOT regulations refers to foundry sand twice. The document classifies this material as "waste material consisting of burned sand with or without slag fragments. In general, this material is waste or by-product material from foundry operations".

Wisconsin has developed one document to regulate all beneficial reuse of industrial byproducts. Wisconsin uses a waste classification system to place industrial byproducts into one of five categories. Under Wisconsin Administrative Code Chapter NR 538, all industrial byproducts that are to be used must be characterized by the ASTM test method for shake extraction of solid wastes with water. Additionally, in order to qualify for category one or two, industrial byproducts must undergo a total chemical analysis. Table 4 compares the allowable concentration limits with those of the other states. Recharacterization is required at various intervals depending on the byproduct category. For use in Wisconsin roadway projects, byproducts are required to be in Category IV or higher. If depth of placement is below four feet, they must be in Category III or higher. Wisconsin also requires sitting restrictions on byproducts not in category one.

State	Program Structure	Siting Restrictions	Testing and Characterization	Basis of Leachate Thresholds	RFS in DOT Works	Source
AL	Waste Classification	Flood Plains, Wetlands, Residential Zones, 5 feet above the uppermost aquifer.	TCLP[1]	50% of RCRA TC[2] Levels	Yes	Authority: 335-13-4.26
IL	Waste Classification	N/A	ASTM D3987-85 & physical analysis	DWS	No	Authority: Title 35 Part 817
IN	Hybrid	N/A (DOT applies siting criteria)	TCLP	Variable % RCRA TC Levels	Yes	Authority: IC 13-19-3- 7
IA	Hybrid	Potable Wells, Ground water, Sur <u>gf</u> ace water, Wetlands, Floodplain	TCLP, SPLP[3] (occasionally)	90% RCRA / <= 10 times the MCL[4] for drinking water	Yes	Authority: 567 Chapter 108
LA	Case-by-Case	N/A	chemical and physical characterization	N/A	No	Authority: Title 33 Chapter 11
ME	Case-by-Case	Potable Wells, Ground Water, Surface water, Wetlands, Critical Habitat	TCLP, Total Composition	Not Specified	No	Authority: Bureau of Remediation & Waste Management
NY	Case-by-Case	N/A	TCLP	RCRA[5] TC Levels	No	Authority: 6NYCRR 360-1.5
ОН	Waste Classification	Potable Wells, Surface water, Wetlands, Floodplains, Residential	TCLP	Up to 30X state DWS[6]	Yes	Authority: DSW- 0400.007
РА	Case-by-Case	Potable Wells, Ground water, Surface water, Wetlands	TCLP, Total Composition	Variable % RCRA TC Levels	Yes	Authority: General Permit WMGR019
WI	Waste Classification	Residential Areas, water table, surface water	ASTM D3987–85 water leach test, TCLP	Variable % RCRA TC Levels	Yes	Authority: Chapter NR 538

Table 3. State-by-state comparison of the regulations pertaining to waste foundry sands.

Toxicity Characteristic Leaching Procedure
 Toxicity Characteristic
 Synthetic Precipitation Leaching Procedure
 Maximum Contaminant Level
 Resource Conservation and Recovery Act
 Drinking Water Standard

Contaminant	RCRA TC Level	AL (TCLP)	AL Iowa OH (TCLP) WI (ASTM D3987-85) †			IN (TCLP)
	(mg/L)			- state threshold (	mg/L)	
Antimony	*	*	*	*	0.012	*
Arsenic (As)	5	2.5	0.1	1	0.05	0.5
Barium (Ba)	100	50	20	40	4	10
Beryllium (Be)	*	*	*	*	0.004	*
Cadmium (Cd)	1	0.5	0.05	0.1	0.005	0.1
Chromium (Cr)	5	2.5	1	2	0.1	0.5
Iron (Fe)	*	*	*	*	1.5	*
Lead (Pb)	5	2.5	0.15	1	0.015	0.5
Manganese (Mn)	*	*	*	*	0.25	*
Mercury (Hg)	0.2	0.1	0.02	0.04	0.002	0.02
Selenium (Se)	1	0.5	0.5	1	0.1	0.1
Silver (Ag)	5	2.5	1	*	*	0.5
phenol	*	*	*	7	12	*
cyanide	*	*	*	0.4	0.4	*
fluoride	*	*	*	8	8	*

Table 4. Comparison of state limts for metals in waste foundry sands.

‡ Iowa's DOT (DOT requires it? If so when in the approval process?) requires a TCLP be performed and submitted for review (Case by case or as part of the waste classification?)

<sup>†</sup> Wisconsin requires characterization of industrial by products using the TCLP to determine its status as non-hazardous

Iowa's Environmental Protection Commission (EPC) uses a hybrid system. Solid byproducts first must be approved for reuse. The approved byproducts are then considered on a case-by-case basis for use from a pre-existing list of recycling options. To be approved for reuse, foundry sand must undergo the synthetic precipitate leaching procedure and have concentrations less than or equal to ten times the maximum contaminant level (MCL) for drinking water. Only the SPLP analytes for total metals are necessary. Additionally, a total metals test including thallium must meet the Iowa standards for soil. There are pH requirements based on depth of placement. Siting restrictions apply as well. The Iowa DOT mandates a waste generation process report including:

- the industrial origin
- quantity produced annually
- geographic location
- variability of process and characteristics

#### • recycling and disposal practices at the generation site

Additionally, this report must contain a section which includes employee health and safety requirements as well as a list of all federal and state environmental regulations which apply to the waste and its disposal. An environmental analytical testing protocol report must be filed and needs to include:

- total and leachable values of metals present
- TCLP
- bacteriology
- petroleum
- general chemistry
- fate and transport
- brief description of sampling protocol, expected waste variability, acceptable standard deviation of reported analytical data.

The Iowa DOT also requires an engineering and material properties report providing a description of the physical, chemical, mechanical and other properties of the material. The report must also contain the relevant design considerations, construction procedures, material processing requirements, and performance records. Finally, economic and cost report is required to include handling and disposal costs, fair market value, cost of using the material, and life-cycle benefits/costs. Figure 2 shows a flow chart of the evaluation process.

The Ohio EPA uses a waste classification system which classifies waste as "nontoxic" and has four categories of beneficial reuse; each of which has different requirements. A TCLP is required for waste characterization. Either the TCLP acid or a modified TCLP water solution may be used. The water solution must be used to obtain acidity, alkalinity, chlorides, cyanide, fluoride, pH, phenol, specific conductance, sulfates, and total dissolved solids. The total parameters to be evaluated are acidity, alkalinity, aluminum, arsenic, barium, cadmium, chlorides, chromium, copper, fluoride, iron, lead, manganese, mercury, pH, selenium, specific conductance, sulfates, total dissolved solids, vanadium, and zinc. Phenol and cyanide analyses are also required for spent foundry sands. In order to qualify as nontoxic material, the waste cannot have concentrations exceeding thirty times the Drinking Water Standard. Table 4 compares the allowable concentrations with those of the other states. An annual analysis of the material must be performed, but does not need to be submitted to the EPA. Siting restrictions apply. The Ohio DOT requires that foundry sands comply with EPA standards in addition to being classified by the specifications for subsurface ingestions. Additionally, there must be an engineering analysis which is to include <u>(all state DOTs have engineering criteria)</u>

- stability analysis
- stability sensitivity analysis
- total settlement analysis
- total settlement sensitivity analysis
- differential settlement analysis
- differential settlement analysis
- A moisture density curve or relative density results are required for compaction acceptance.
- Thirty days prior to sand use the location where the sand will be used, the estimated volume to be used, a summary of engineering analysis, tests, and proposed compaction acceptance must be submitted.



**Figure 2. Flowchart for the Iowa approval process for reuse of waste foundry sands.** (Image from Iowa Department of Transportation Policy & Procedure Manual, Policy 500.12)

Indiana's Department of Environment Management (IDEM) uses a hybrid system in which the waste is classified based on the constituents using the TCLP (Indiana Code 13-19-3-7). The maximum level of constituents is compared with the other states in Table 4. There are four waste categories. There is a list of preapproved uses for foundry sand with a classification of III or IV which includes direct land application, soil amendments, roads, road shoulders, and parking lots and highway use. The Indiana DOT requires a complete Material Safety Data Sheet as well as a copy of IDEM's waste classification certificate. IDEM also imposes additional siting restrictions (not sure this is correct). The general IDEM requirements are:

- Name and location of source or manufacturer,
- List of material and specification reference for the material that the approval is being requested,
- Average monthly production of the material by size, type or grade,
- Name, address and telephone number of responsible contact person,
- Facility layout or production process of the material,
- Quality parameters of the material,
- Raw material sampling and testing frequency,
- Procedures for conforming materials which provides a positive linkage between the furnished materials and the quality control test data,
- Procedures for non-conforming materials,
- Procedures for marking and tracking materials,
- Procedures for documentation maintenance,
- Finished material sampling and testing frequency,
- Procedures for reviewing and updating the source operations,
- Testing laboratory quality system,
- Names, titles and qualifications of sampling and testing personnel,
- Location and telephone number of the laboratory testing office,
- Laboratory equipment and calibration frequency,
- Test methods, procedures and laboratory equipment used for each type of material,
- Sample management describing procedures for samples identification, maintenance of the samples prior to testing, sample retention and disposal of samples,
- Testing report procedures,
- Methods used to identify improper test results and procedures followed when testing deficiencies occur,
- Statistical analysis of test results,
- Maintenance of test records.

Additionally the following are required to be submitted.

- A current MSDS (discuss validity and practicality) and summary of results of all specified tests for the previous year's production shall be submitted. No test results shall be more than two years old at time of submission.
- Name of Testing Facility
- Dates Samples were obtained
- Dates Samples were tested
- Test method used for IDEM classification
- Letter from IDEM indicating the waste classification of the materials.
- Test results for Leachate
- Test results for Microtox<sup>™</sup> in accordance with ITM 215

- Stockpile sampling locations, including depths and available historical testing results.
- Gradation test results
- Hydraulic conductivity (permeability) test results
- Recycled Foundry Sand Source Certification

The Microtox<sup>TM</sup> test is to be carried in accordance with ITM No. 215.08T (Indiana DOT Recurring Special Provisions & Plan Details document 200-R-401)

#### Leachate Characterization

Each state can use leachate characterization to determine the appropriateness of the foundry sand as a building material. As can be seen in Table 4, three of the five states evaluated chose to use TCLP for the leachate characterization. The remaining two states required the TCLP to be used in some capacity, but used alternate tests to determine the usability of the foundry sand. One possible reason behind the decision to use an alternate leachate testing is the fact that TCLP was designed to examine waste destined for landfills while others, such as the SPLP, were designed to simulate leachate from natural rainfall. The U.S. EPA states that only TCLP can be used to determine the characterization, Alabama appears to have the most lenient standards with Indiana as the strictest (of all states or of states reviewed, frequency of testing required by regulatory agency, if IDOT requires TCLP then not necessarily a true statement?). The benefits of stricter guidelines are twofold. First, Indiana is more confident in the safety and quality of the material. Second, other than use on DOT projects, the higher initial standards result in a more streamlined usage process whereby for many applications there is no further action or notification required even for direct land application. Indiana also has a biological toxicity analysis, Microtox, which is conducted on the leachate and is required for any DOT project.

#### **Siting Restrictions**

Another tool used by all states evaluated in this study is siting restrictions. These regulations restricted the placement of foundry sand from those areas where there would be the highest risk of leaching and possible contamination of groundwater. Table 5 shows the siting restrictions placed on foundry sand by various states. There are three aspects to these restrictions. All of the states have designated areas where there is either flowing or ponded water (streams, lakes, etc) or areas with a high risk assessment(residential areas, wetlands, etc). The second aspect of these regulations is the specificity with which the regulations are written. Some states such as Alabama indicate that foundry sand "may be managed in areas other than" those listed while other states such as Indiana have specific distances (e.g. 150 feet from potable water). (change in font size) The final aspect of siting restrictions is the actual distance of the mandated buffer zone. There is no clear trend as to which state is the strictest with siting restrictions. Ohio seems to have specific locations and defined distances. Indiana has a high amount of specificity with both location and distance. Alabama has few sites designated and little specificity as to what distance is required between foundry sand usage and the designated sites.

#### Anomalies

While each state is reducing foundry sand waste and using this material in projects in different ways, similarities exist between the regulations. Each state has an initial characterization of the foundry sand which is compared to a standard. Then the specific use of the sand is taken into account by way of either a preapproved list or a case-by-case determination. Finally various measures are taken to monitor the project to ensure that no unforeseen environmental problems have arisen. Yet beyond these similarities, there are a few anomalous policies. Alabama has relatively lenient regulations with TCLP and an approval from a materials engineer. Iowa has an extensive list of requirements.. Finally, Indiana has a requirement of the Microtox evaluation in addition to conservative required leachate paramaters.- Of note, no other state uses the Microtox test.

State			Sit	ing Requirements			
				Distance above			
	Flood		Residential	the uppermost	Water		
	Plains	Wetlands	zones	aquifer	Way	Well	Other
AL	Х	Х	Х	5 Feet			
OH	Х	100 Feet		5 Feet	100 Feet	300 Feet	
WI			Х	Х	Х		$\mathbf{x}^1$
IA	Х	Х		5 Feet	Х	200 Feet	2
IN		100 Feet			100 Feet	150 Feet	$\mathbf{x}^2$

Table 5. Siting requirements/restrictions associated	l with the use of	waste foundry	sand for five
states.			

X = restricted from site

Areas that need to be dewatered prior to placement
 Metallic pipes or structures, encasement material, MSE wall applications, protected environmental resource area.

State	URL for website
AL	http://www.adem.alabama.gov/Regulations/Div13/Div.%2013%20Effective%20December%2012,%202005.pdf
IL	http://www.ipcb.state.il.us/documents/dsweb/Get/Document-12195/
IN	http://www.ai.org/legislative/ic/code/title13/ar19/ch3.html#IC13-19-3-7 and Article 10 Rule 9 : <u>http://www.in.gov/idem/4996.htm</u>
IA	http://www.legis.state.ia.us/Rules/Current/iac/567iac/567108/567108.pdf
LA	http://www.deq.louisiana.gov/portal/Portals/0/planning/regs/title33/33v07-200807.pdf
ME	http://www.state.me.us/dep/rwm/rules/index.htm#rulesadmbrwm
NY	http://www.dec.ny.gov/regs/4415.html
ОН	http://www.epa.state.oh.us/dsw/policy/04_07r.pdf
ΡΑ	http://www.depweb.state.pa.us/landrecwaste/lib/landrecwaste/residual_waste/gp/wmgr019.pdf
WI	http://www.legis.state.wi.us/rsb/code/nr/nr538.pdf

# PART II. REVIEW OF THE SCIENTIFIC LITERATURE FOR MICROTOX APPLICATIONS

A database search (Web of Science) yielded over 550 scientific journal publications in which Microtox and soil, water, or sediment were studied. The earliest publication found on the subject was printed in 1983. Three exhaustive reviews of literature have been published (Doherty et al. 2001; van Beelan et al. 2003; Parvez et al. 2006) in addition to an early overview of Microtox (Galli et al. 1994). Rather than repeat the efforts of these reviews, we chose to summarize them and follow with an examination of Microtox publications from 2006 through 2008. This allowed us to search for new developments or changes in opinion about the efficacy of the Microtox test.

The experimental approach of Galli et al. (1994) was innovative in that the authors were attempting to use Microtox as a broad screening test for pesticides in remediated soils. Their rationale was that chemically analyzing for all possible pesticides and metabolites would be impractical and cost prohibitive; Microtox could be used as a biosensing test that integrates impacts of the contaminants. They tested the toxicity of pure compounds, developing  $EC_{50}$  values and the % inhibition of the bioluminescence. The  $EC_{50}$  of individual compounds was calculated using the equation:

$$\log \Gamma = \log \frac{I_0 - I_t}{I_t}$$

in which  $\Gamma$  is the ratio of the bioluminescent light lost (I<sub>0</sub> - I<sub>t</sub>) to the light remaining after exposing the bacteria for 30 minutes to a solution of a constant concentration. When various chemicals are present, one may assume that the overall effect is the result of the sum of the individual effects. Galli et al. (1994) summarized their findings about this application of Microtox as follows:

"The application of aquatic toxicity tests as an indicator for the detection of unexpected contaminants has been evaluated. Assessment of parameters such as relative sensitivity, time consumption and costs has led to the selection of the Microtox test for this special application, whereas bioassays with the water flea and green algae were less favorable. Among bacterial toxicity bioassays the Microtox test is one of the most sensitive tests (Reteuna et al., 1989; Dutka & Kwan, 1981) and toxicity data for many chemicals are available (Kaiser and Ribo, 1988)... Complex mixtures of chemicals may have a net toxicity quite different from the sum of the effects of the known toxic material present, due to synergistic or antagonistic action related to the specific nature of the toxicants, partially confirmed in this study."

Ultimately, Galli et al. (1994) recommended the use of Microtox as a tool to evaluate the residual toxicity of remediated soil, particularly when the residual contamination was so chemically complex that it defied reasonable chemical analysis.

Doherty (2001) compiled a comprehensive review of the literature up to the year 2000 concerning Microtox as a screening test for soils and sediments. His stated objective was to provide a review that "summarizes studies in which the luminescent bacterium *V. fischeri* and the Microtox Test System were used in the assessment of soil or sediment toxicity, including all naturally or artificially generated aqueous media (porewater, groundwater, leachates and elutriates), organic solvent extracts, and solid-phase material." It was concluded that solvent extraction has many potential difficulties because the solvent often is toxic to the *V. fischeri*. Aqueous extracts of soils are clearly problematic because water will remove only a fraction of most contaminants; for some sparingly soluble metals and hydrophobic organic contaminants, results using aqueous extracts would be highly misleading. Thus, Doherty recommends that tests using aqueous extracts are far less sensitive than the direct use of the solids.

Perhaps the most important aspect of Doherty's review was the analysis of several studies that attempted to correlate Microtox results with concentrations of contaminants in field samples. Wide ranges in correlation coefficients were reported, and interactions among contaminants were apparent.

This led the author to the conclusion that "no single evaluation procedure can adequately define groundwater contamination, and that future monitoring requirements should include Microtox (specifically the 100% test), chemical screening tests (TOX and TOC) and indicator parameters (chloride, specific conductance and pH)." Thus, Microtox is viewed as a potentially powerful indicator but should not be used alone.

Another review of the use of Microtox and other microbial tests specifically examined sediments (van Beelan 2003). The anaerobic nature of sediments was a point of emphasis, particularly when it comes to using aerobic bacteria to measure toxicity. When obligate aerobes are placed in an anaerobic medium, toxicity symptoms may be observed that are strictly the result of the anaerobic conditions and not due to the presence of toxic chemicals. On the other hand, anaerobic environments can generate high concentrations of natural substances that are potentially toxic to *V. fischeri*, including ammonia and hydrogen sulfide. Correcting for this problem could be problematic but has been accomplished by extracting the sediments with solvents that could ignore the natural toxicants. This approach works in some instances but the selectivity may exclude toxic compounds of interest yet enhance concentrations of toxic compounds that normally are inactivated through strong adsorption. Van Beelan's (2003) review provided an excellent overview of the practical considerations of adapting microbial tests but provided very little actual data.

Parvez et al. (2006) appear to have the most recent review of Microtox applications. They examined and summarized the variations of Microtox being used. They also discussed the chemical mechanism by which the V. fischeri become luminescent and how the change in light intensity can be used to quantify toxic responses to contaminants. Most importantly, Parvez et al. (2006) evaluated many publications relative to the successful use of Microtox. The authors discussed the strong relationship between Microtox EC<sub>50</sub> values and LC<sub>50</sub> for fathead minnow. bluefill. catfish. goldfish, guppy, killifish, rainbow trout, sheepshead minnow, zebrafish, Daphnia, algae, and intravenous LC<sub>50</sub> for mouse and rat (Kaiser 1998). Dezwart and Sloof (1983) examined the sensitivity of Microtox to 15 metals, anions, and organic compounds and determined that Microtox compared favorably with other tests. Padrtova et al. (1998) compared bioluminescent bacteria to other biological tests and found that the bioluminescent bacteria and algae were the most sensitive. The algal (algal or algae?) test was considered to be less desirable than the bioluminescent bacteria because the algae were more difficult to maintain and much slower to respond. V. fischeri was compared to Pseudomonas flourescens (Abbondanzi et al. 2003) for sensitivity to metals and organic contaminants. The organisms had similar sensitivity to metals, but the Pseudomonas flourescens was less sensitive to organics than V. fisheri. This evaluation led the authors to conclude, "Based on this literature survey we can conclude that out of the various available bioassays, Vibrio fischeri based luminescent inhibition test is more sensitive, rapid, cost effective, reproducible and without ethical problems ensuing from the use of higher organisms such as fish and rat."

Significant, relevant findings concerning the application of Microtox have been published since 2006 (Amoros et al., 2007; Antunes et al., 2007; Murakami et al., 2008; Flokstra et al., 2007). Amoros et al. (2007) examined the toxicity of glyphosate (Roundup®) in lake water as determined by a new *Aeromonas* bioassay compared to Microtox. *Aeromonas* curiously showed either no toxicity or positive responses to glyphosate at concentrations as high as 100 mg/L. Microtox EC50 was shown to range from 36 to 64 mg/L, depending upon the method used. The authors attributed the toxicity of glyphosphate to the acidity of the herbicide (pH 4.5).

Atunes et al. (2007) described the use of Microtox, Daphnia, and *Eisenia andrei* to evaluate the toxicity of soils in the vicinity of a uranium mine. Microtox and Daphnia showed no toxicity, whereas *E. andrei* showed far more sensitivity to mine runoff and/or sludge deposition. The authors attributed the lack of response by V. fisheri and Daphnia to lack of water solubility/mobility of the contaminants associated with this mine waste. Because the *E. andrei* interact directly with the solid phase, their response will be more pronounced in the case of immobile contaminants.

Murakami et al. (2008) used Microtox as an evaluation tool and compared it to the yeast estrogen screen, algal growth inhibition, and mutagen formation potential. They were testing the removal of toxins from road runoff by allowing the runoff to pass through a column of soil. Most of the assays showed a decrease in toxicity of the leachates after passing through the soil, but Microtox did not show this decrease except in very late stages of infiltration.

Flokstra et al. (2008) used Microtox to evaluate the removal of the explosives TNT and RDX from water by plant cell cultures. The authors found that the Microtox test was quite sensitive to these compounds and revealed a significant decrease in toxicity as the TNT and RDX were removed from solution by the plant cells.

The results discussed immediately above are consistent with previous results: Microtox is quite sensitive to organic contaminants, but less sensitive to metals. When evaluating toxicants that are highly sorbed to solid surfaces, it is best to use the modified Microtox that tests the solids directly rather than using an extract of any kind. Microtox probably is best used in conjunction with other biological tests, but of all the biological tests considered, Microtox would be the logical choice for a stand-alone biological test.

#### **Conclusions and Recommendations**

<u>Regulations from Other States</u> The review of regulations for various states suggests that Indiana falls in the middle of the range of rigor for foundry-sands testing prior to recycling; of course, many states have no foundry sand recycling program at all. Some states have minimal requirements while others are very aggressive in their testing programs. As suspected prior to this study, Indiana is the only state to require a biological toxicity test.

<u>Review of the Scientific Literature</u> Realizing the unique aspect Indiana's Microtox requirement, we initiated a review of the scientific literature to determine the applicability of Microtox and to evaluate whether or not it has been used successfully in various environmental contexts. The literature is fairly consistent in concluding that Microtox is useful in evaluating potential toxicity for soils, sediments, and water.

<u>Microtox Analyses by Commercial Laboratories</u> As part of the execution of this project, we polled 15 commercial laboratories in Indiana that analyze water and soil for their ability to provide a Microtox analysis. None of these laboratories currently provide this analysis, though some of them suggested that they once did. STL Valparaiso indicated that Microtox was available in the recent past, and they still possess the equipment. However, they no longer have the technical expertise to run the test. Two of the labs provided contact information for out-of-state laboratories that analyze for Microtox; unfortunately, these labs actually do not provide the service.

The Western Canada Microtox Users Committee (<u>http://www.wcmuc.com/</u>) provides information and data concerning the use of Microtox. Through their website, we were able to locate ALS Environmental Group of British Columbia that provides Microtox analysis. According to their analytical team leader, theses analyses can be obtained for a cost of \$160 (Canadian) with a turn-around time of 6 days.

<u>Recommendations</u> The current Microtox requirement for recycling foundry sand in Indiana is viewed by some in the industry as excessive. However, Microtox is readily defensible from a scientific perspective, and many studies suggest that Microtox should be coupled with at least one other biological test to be fully encompassing.

Strictly from viewpoint of environmental protection, the inclusion of Microtox makes sense. The test has the sensitivity to detect potentially toxic agents in recycled sand that might escape chemical analysis. The test, therefore, provides a layer of assurance that otherwise would be absent. From the

perspective of the foundry industry, the Microtox test is an unneeded hurdle that could potentially block the beneficial use of spent foundry sand. Cost is one consideration, but the lack of local analytical facilities for the Microtox is particularly troublesome.

Our recommendation is that the Microtox test be retained by INDOT, but we suggest the following: (discuss with Paul the combination of IDEM TCLP criteria and the MSDS, versus TCLP at time of use [IDOT], versus pre-certification, versus Microtox testing. Two entities involved in setting requirements both the State regulatory agency and the DOT.)

- a) If at all possible, minimize the number of samples of foundry sand that must be tested. Periodic testing is critical to ensure protection of the environment, but in the absence of changes in foundry processes, it might be possible to reduce the frequency of sampling and testing.
- b) A consistent, readily available laboratory needs to be established to ensure rapid turn around of analyses and reduced costs. Two problems exist for commercial establishing a service for Microtox: the demand is low and some dedicated equipment is needed to perform the test.

One of the potential outcomes of this project discussed during the negotiations for this project was a possible follow-up project in which we would investigate the modifications to the bioassay. This might include exploring alternatives to Microtox or simplifications of the Microtox test. We remain open to this possibility, but from the scientific point of view, such a follow-up may not be necessary. Of all the bioassays we reviewed, Microtox seemed to be the most widely used (though not for foundry sands), and we found no evidence that other bioassays were being offered routinely at commercial labs.

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