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Sandia National Laboratories Arsenic Treatment Technology Demonstration Program

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http://www.sandia.gov/water/arsenic

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company,
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Arsenic Water Technology Partnership Background

- Congressional Appropriation - $10M FY03 – FY05
- DOE- funded peer-reviewed, cost-shared research program to develop and demonstrate innovative technologies for removal and disposal of arsenic from drinking water
- Partner Roles
  - Bench-Scale Studies (AwwaRF)
  - Demonstration Studies (Sandia)
  - Economic Analysis/Outreach (WERC)
- Focus on small systems
  - 40% of resources directed to rural and Native American utility needs
  - Minimize costs - capital, operating, maintenance
  - Minimize residual quantities & disposal costs

Goals of Sandia Arsenic Treatment Program

1. Screen commercially-available and innovative technologies
2. Carry out pilot tests using credible test procedures to obtain performance data sufficient to predict full-scale performance
3. Develop rapid test procedures to allow widespread testing of technologies by communities

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Goal 1: Technology Evaluation

- **Sandia Arsenic Treatment Vendors Forum**
  - Open session allows Vendors to present product descriptions
  - Closed session review by Technical Evaluation Teams
  - 2003, 2004, 2005 Vendor Forums led to recommendation of innovative technologies for initial pilots and others for additional bench-scale studies
- **Other Evaluation Programs**
  - Awwa Research Foundation
    - Technical Review Committee defines research objectives
    - Grants are awarded through competitive, peer-reviewed RFP process
  - WERC Design Contest
    - WERC utilizes existing Design Contest in order to obtain innovative arsenic removal technologies.

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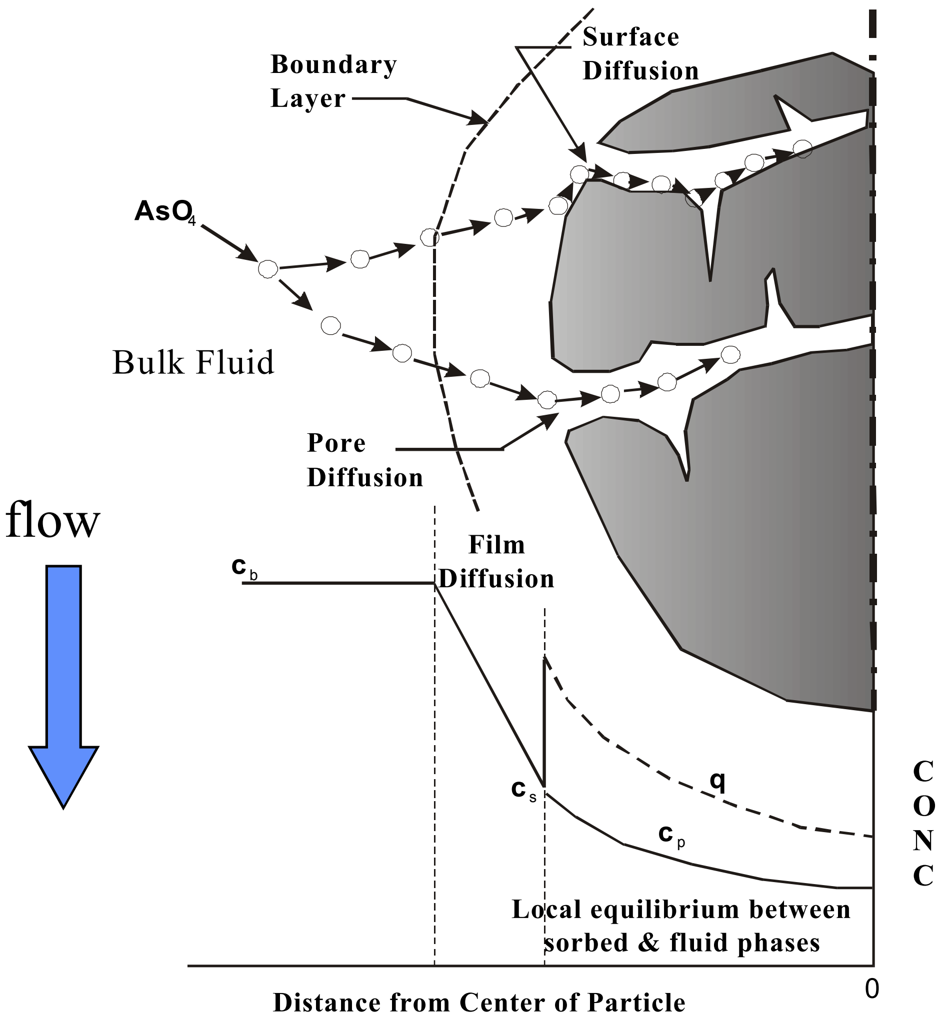
Performance of Adsorptive Media



Targets for Improvement

- Sorption equilibria
- Intraparticle diffusion rates
- Maintain flow

(after Crittendon, 2004)

Current Sorption Treatment Innovations

- **Fe, Ti, Cu, Zr or mixed metal oxides in granules formed by chemical precipitation or nanoparticle agglomeration. (e.g. AdEdge, Kemiron, Argonide, Graver)**
- **Coating granular activated carbon (GAC), strong base anion exchangers resin or polymeric ligand exchangers with nanoparticulate metal oxides. (e.g. Purolite, Resintech, Auburn University)**
- **Coating inexpensive natural media or waste products with metal oxides or other functional groups. (e.g. ADA, Virotec, Arizona State)**
- **Increased surface area and chemical selectivity based on fibrous or gel substrates coated by metal oxides or materials with sulfhydryl functional groups. (e.g. NMSU, Weber State, Drexel University)**

Goal 2: Sandia Pilot Tests

- **Side-by-side demonstrations of technologies tested by AwwaRF bench-scale program, WERC design contest, or commercial technologies vetted through Vendor Forums**
  - **Test duration: 3 – 9 months**
  - **Test size: 0.3 – 10 gpm**
  - **Different technology classes: adsorptive media, Coagulation/Filtration, membranes, electrochemical**
- **Cooperative effort between Sandia, Technology Owner and Site Owner**
- **Test Protocols developed with help from NSF International, academia, industry during 2004-2005**
- **Phase I Tests: Innovative technologies designed with vendor input**
  - **Fixed bed adsorbent media: Particle size, hydraulic loading rate, Empty Bed Contact Time**
  - **Batch systems (e.g. C/F) with vendor equipment**
- **Phase II**: evaluate newer media, pH changes, corrosivity, other effects**.**

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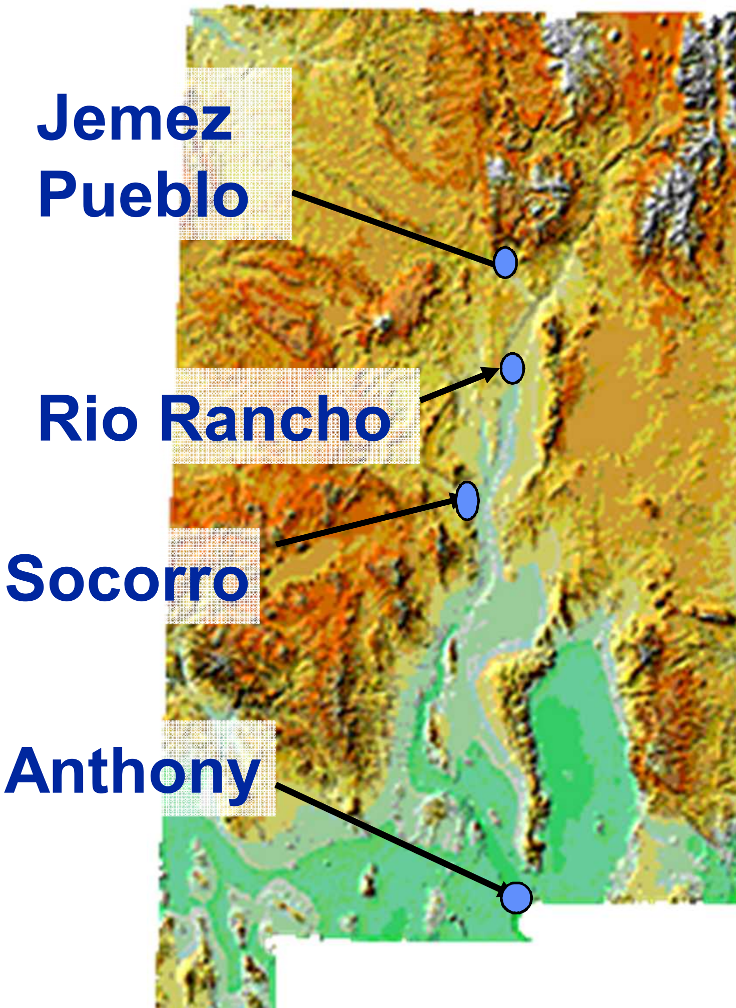
Things we look for in a pilot site

- As concentration (>10 ppb)
- Example ground water composition that will help other communities
  - pH, TDS, foulants such as Fe, Mn, silica, and organics
  - As(III)/As(V)
  - Competing ions (V, SO_4, etc.)
  - Other contaminants of concern/benefit (e.g, Ra, U, ClO_4, F)
- Small size of system to be treated (< 10,000 users)
- Community support facilitates rapid deployment
  - Water utility
  - Municipal government
- Ability to deal with residuals/treated effluent
- Rural and Native American communities that would benefit from assistance

On-going Pilots in New Mexico



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New Mexico Pilot Sites – Water Quality

Site	Total As/As(III)	V (ppb)	SO_4 (ppm)	Fe (ppm)	pH
Socorro	*42 ppb / 0 ppb*	*11*	*29*	*0.4*	*8.0*
Anthony	20 ppb / 18 ppb	2	180	0.15	7.7
Rio Rancho	19 ppb / < 1 ppb	15	100	<0.10	7.7
Jemez Pueblo	20 ppb / 19 ppb	<1	24	1.2	7.5

Site	Cond. (µS/cm)	TOC (ppm)	Ca Hard (ppm $CaCO_3$)	Alkalinity (ppm $CaCO_3$)	SiO_2 (ppm)
Socorro	*360*	*0.5*	*44*	*120*	*25*
Anthony	1380	0.8	66	180	37
Rio Rancho	630	ND	62.5	184	22
Jemez Pueblo	770	2.0	155	290	50

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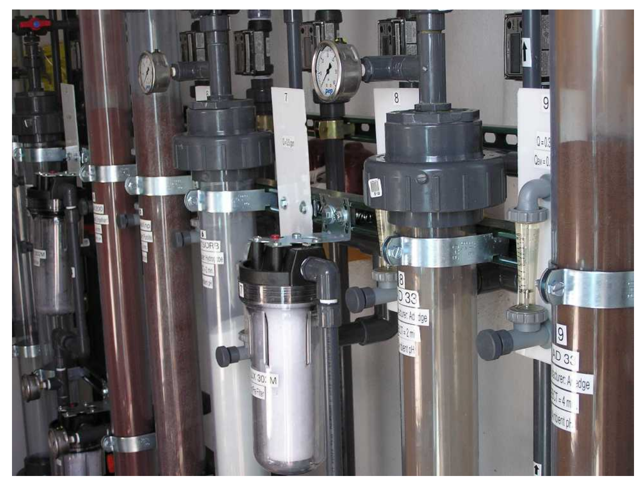
Socorro, NM

- 100% groundwater source for drinking water
- 2 warm springs (90^{o}F) provide 500 gpm, 35 – 55 ppb As(V) by gravity flow.
- Formerly site of tap for bottled water company;
- Optimal F for oral health
- Phase 1: Feb-Oct 2005
  - Tested
    - Fe oxides: AD33, ARM200
    - Resin - ArsenX^{np}
    - Ti-oxide - Metsorb
    - Zr-oxide - Isolux
  - EBCT study of AD33: 2,4,5 min



ARM200 and ArsenX^{np} found to be ‘substandard’ by vendors.

Phase II Studies in Socorro

Phase IIa:

- Capacity extension tests of spent media
  - pH adjustment by CO_2 gas
  - Interrupted flow

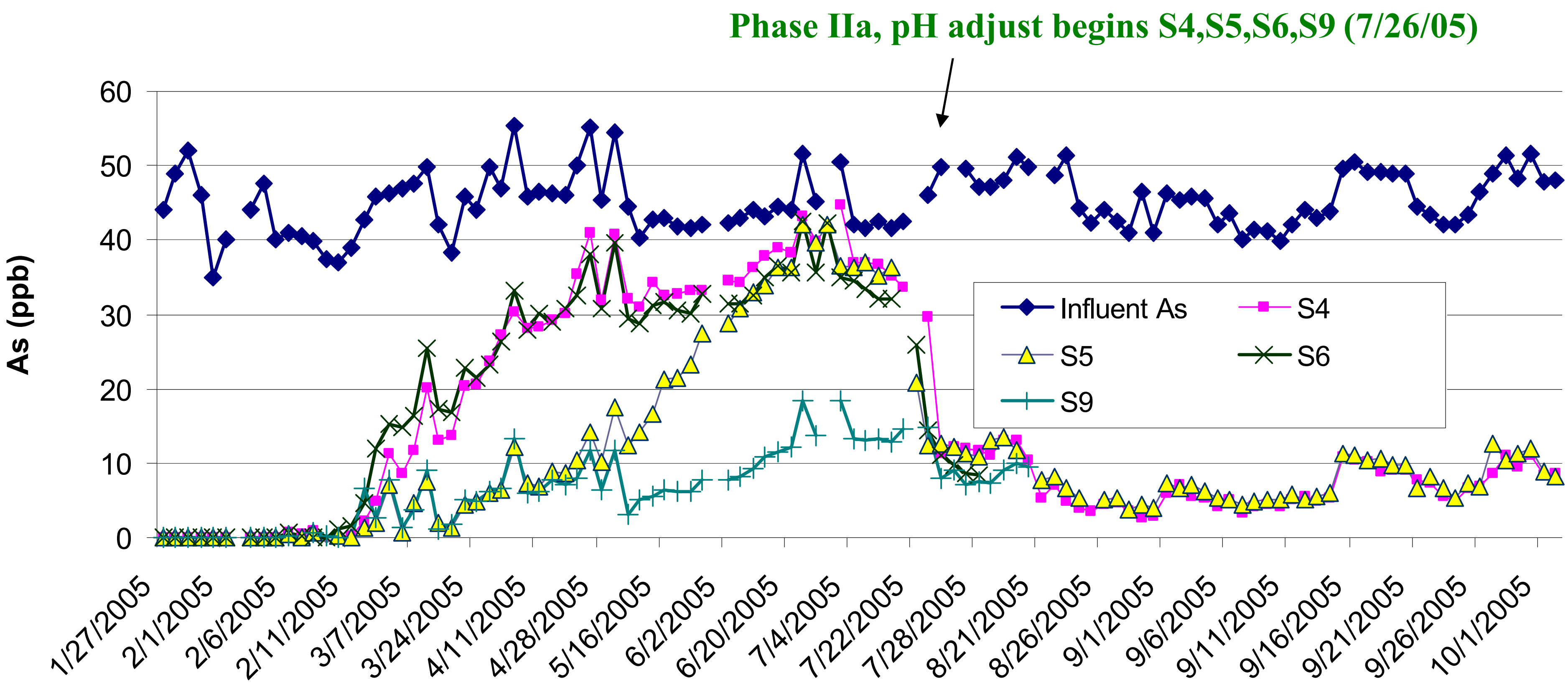
Phase IIb:

- Side-by side comparisons of 5 media at 2 pH levels

  pH = (8: ambient and 6.8:CO_2 gas)
  - ArsenX^{np} – QC'd batches
  - Isolux – larger cartridge for more 'reliable' BV
  - Kemiron – FeOx media
  - SANS – Sandia proprietary media
  - Metsorb – TiOx media
- Evaluate inadvertent effects of treatment
  - Loss of pH control and arsenic spike

Socorro Pilot Phase I and IIa Events

S4 = pre-production ARM200 (FeOx); S5 = AsXnp (defective resin); S6 = Metsorb (TiOx); S7 = Isolux (ZrOx); S9 = AD33 (FeOx)



Not a linear scale!

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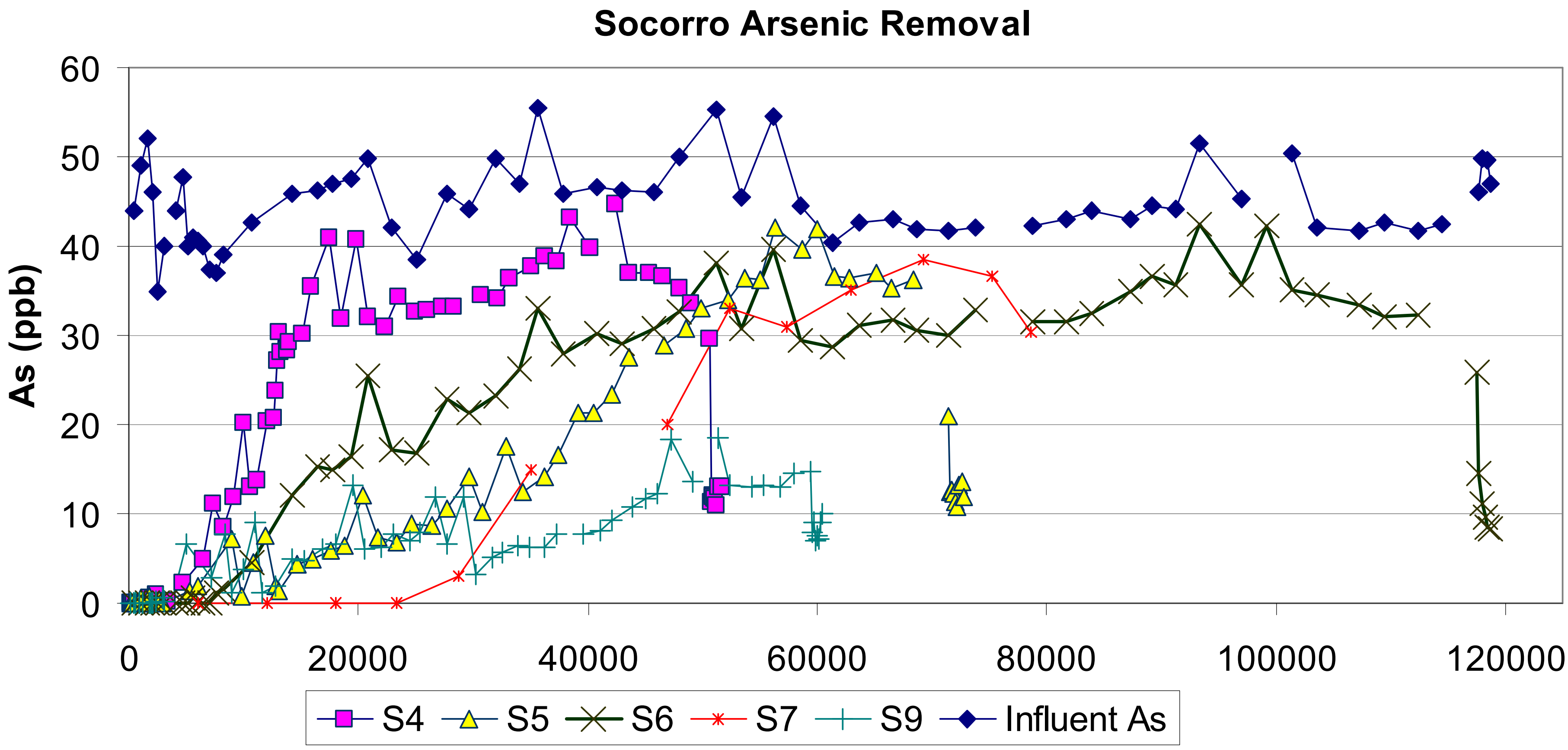
Media Performance Socorro, NM

S4 = pp ARM200 (FeOx); S5 = ArsenX^{np} (defective resin); S6 = Metsorb (TiOx); S7 = Isolux (ZrOx); S9 = AD33 (FeOx)



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Second Pilot Site: Anthony, NM (Desert Sands MDWCA)

- 100% groundwater source for drinking water
- Warm source (~85^{o}F) provides 240-270 gpm, 20 ppb As - mainly As(III).
- High sulfates, TDS
- Intermittent Flow Operation
- Media Tested
  - FeOx: AD33, ARM200, CFH12
  - ZrOx: Isolux
  - TiOx: Metsorb, Adsorbsia GTO
  - Resins: ASM-10HP, ArsenX^{np}
- Phase 2: Added new media, reloaded others
  - La, Fe, Mg-coated diatomaceous earth: Eagle Picher/NXT-2
  - FeOx-Coated GAC: Virotec/Bauxsol
  - Fe-coated silicate: ADA/Am Si
  - FeOx: Sandia/SANS
  - Reloaded ArsenX^{np} column

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Third Pilot Site: Rio Rancho, NM

- 100% groundwater source for drinking water
- Deep well (800 ft) provides 2000 gpm, 20 ppb As (mainly As V).
- Moderate sulfate, Vanadium, TDS
- Phase 1: 9/1/05-12/6/05
  - FeOx: AD33, CFH10
  - ZrOx: Isolux
  - TiOx: Adsorbsia GTO
  - Resins: ASM-10HP, ArsenX^{np}
- Phase 2: 4/3/06 start
  - FeOx: AD33, CFH12, SANS
  - ZrOx: Isolux
  - TiOx: Adsorbsia GTO
  - Resins: ASM-10HP, ArsenX^{np}
  - Other: Am Si, Bone Char
- Continuous Flow Operation

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Summary: Bed Volume Results (through 2/22/2006)

Media	Socorro BV to 10 ppb breakthrough (Ph1)	Desert Sands BV to 10 ppb (Ph1 & Ph2)	Rio Rancho BV to 10 ppb (Ph1)
ARM200 - Fe	9,000	18,000	>20,000
AD33 - Fe	26,000/43,000/42,000 (2/4/5 min EBCT)	>20,000	N/A
CFH12, CFH10	N/A	>20,000	>20,000
Isolux - Zr	32,000	>12,000	>20,000
Metsorb- Ti	13,000	>20,000	N/A
Adsorbsia GTO	N/A	>20,000	>20,000
ArseneX^{np}	27,000	>20,000 (Ph2)	>20,000
ASM-10HP	N/A	8,500	17,000
SANS	N/A	>10,000	N/A
Bauxsol/GAC	N/A	<500	N/A
Amorph Si	N/A	2,500	N/A
NXT-2 - La	N/A	*Media broke down at 2,400*	N/A

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Fourth Pilot Site: Jemez Pueblo, NM

- As levels : 20-30 ppb ; optimal F level
- Treatment plant online December 2005
- Pilot System started April 2006:

  4 systems to be tested:
  - Hungerford & Terry (Greensand Plus™)
  - Kinetico (Macrolite™)
  - Blue Water Technologies
  - Orca (Sand/Anthracite)

  Objectives:
  - Jar Studies
  - Oxidation/Filtration (no $FeCl_3$) using Cl_2, ClO_2
  - Coagulation/Filtration (2-6 ppm as Fe using $FeCl_3$)
- Opportunities for training and outreach will be important aspects of pilot test program

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Goal 3: Develop Rapid Test Methods

Reduce time and costs required to determine the most effective adsorptive treatment technology for small systems for a variety of water qualities.

Full scale treatment
12-24 months

Pilot scale
6-12 months

RSSCT & isotherm
Days-weeks

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Laboratory Studies

Objective: Compare predictions of media performance obtained from different kinds of lab tests to the results of pilot tests.

- Materials characterization
  - Pre-test and post studies, temperature-ageing studies
  - XRD, Surface area (BET), pore size distribution
  - Particle morphology and surface chemistry
  - Attrition loss
  - Post-mortem pore fluids and solids
- Batch sorption studies
  - Kinetic (15^{o}C and 40^{o}C)
  - Isotherms (linear, Freundlich, Langmuir)
- Rapid small scale column tests (RSSCTS)
  - Proportional Diffusivity (PD) and Constant Diffusivity (CD)

Are results from different tests consistent?

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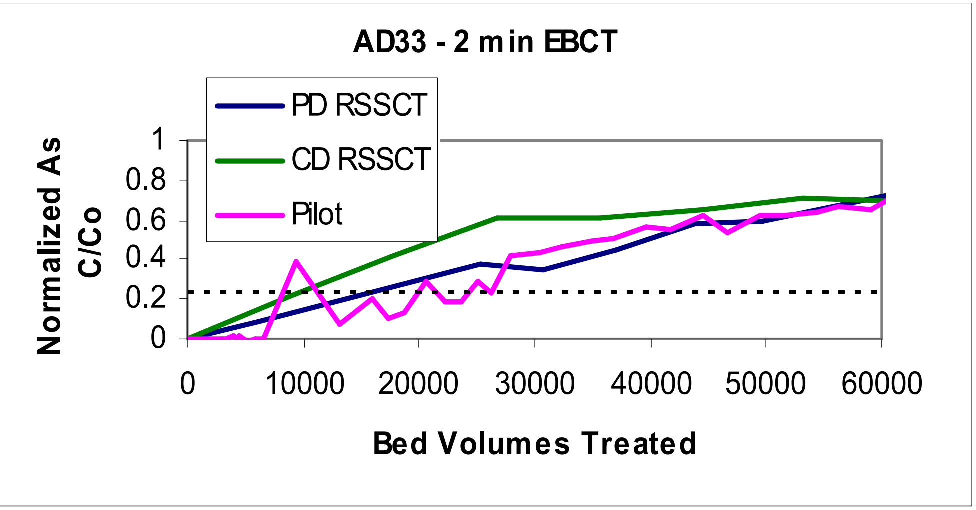
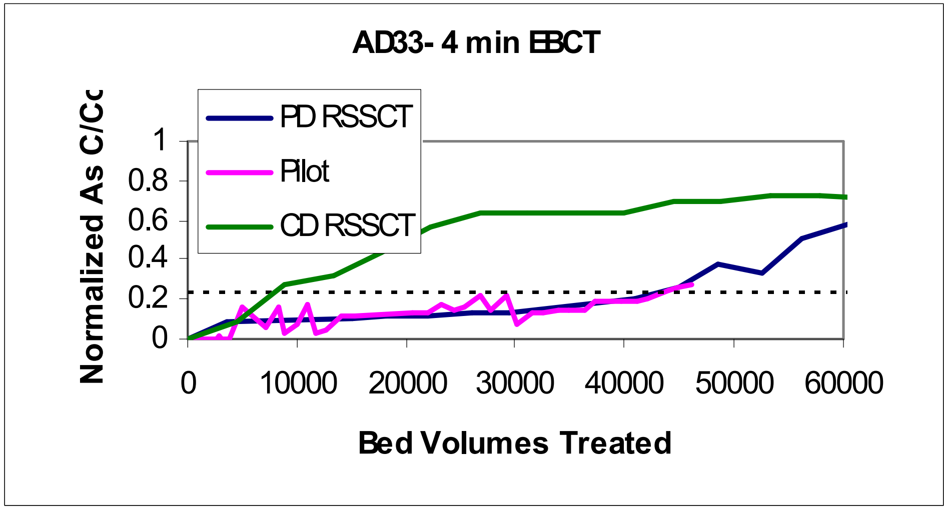
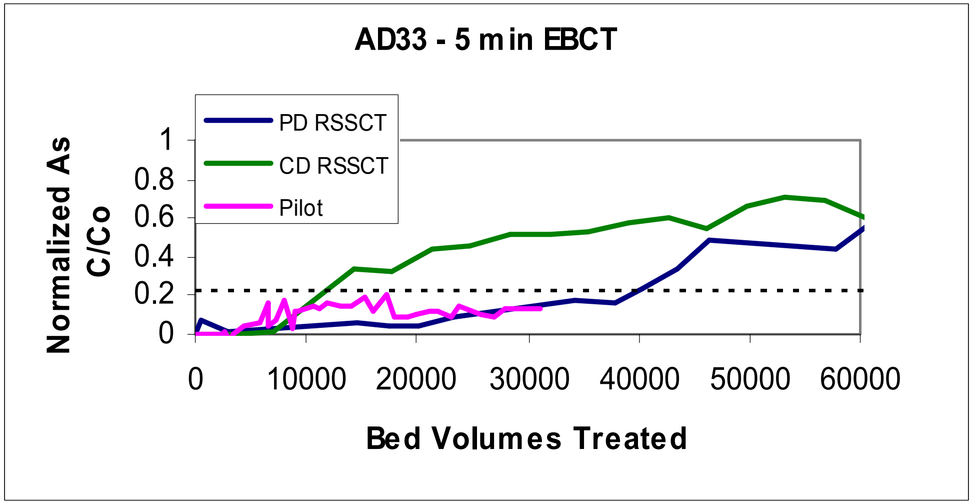
RSSCT Design and Practice

- Crush media to much smaller sizes
    - Smaller media, faster kinetics
- Reduce column diameter
    - Smaller column, higher HLR
- Apply a higher hydraulic loading rate
    - Faster HLR, smaller boundary layer, faster kinetics
    - Reduces external mass transfer resistance
- Shorter EBCT (Empty Bed Contact Time)
- Dimensional analysis and similitude
    - Attention to dimensionless parameters
- Two RSSCT designs:
    - Proportional Diffusivity: duration 2-5 weeks
    - Constant Diffusivity: duration 2-10 days

Comparison of Breakthrough for AD-33







PD results closer to Pilot.

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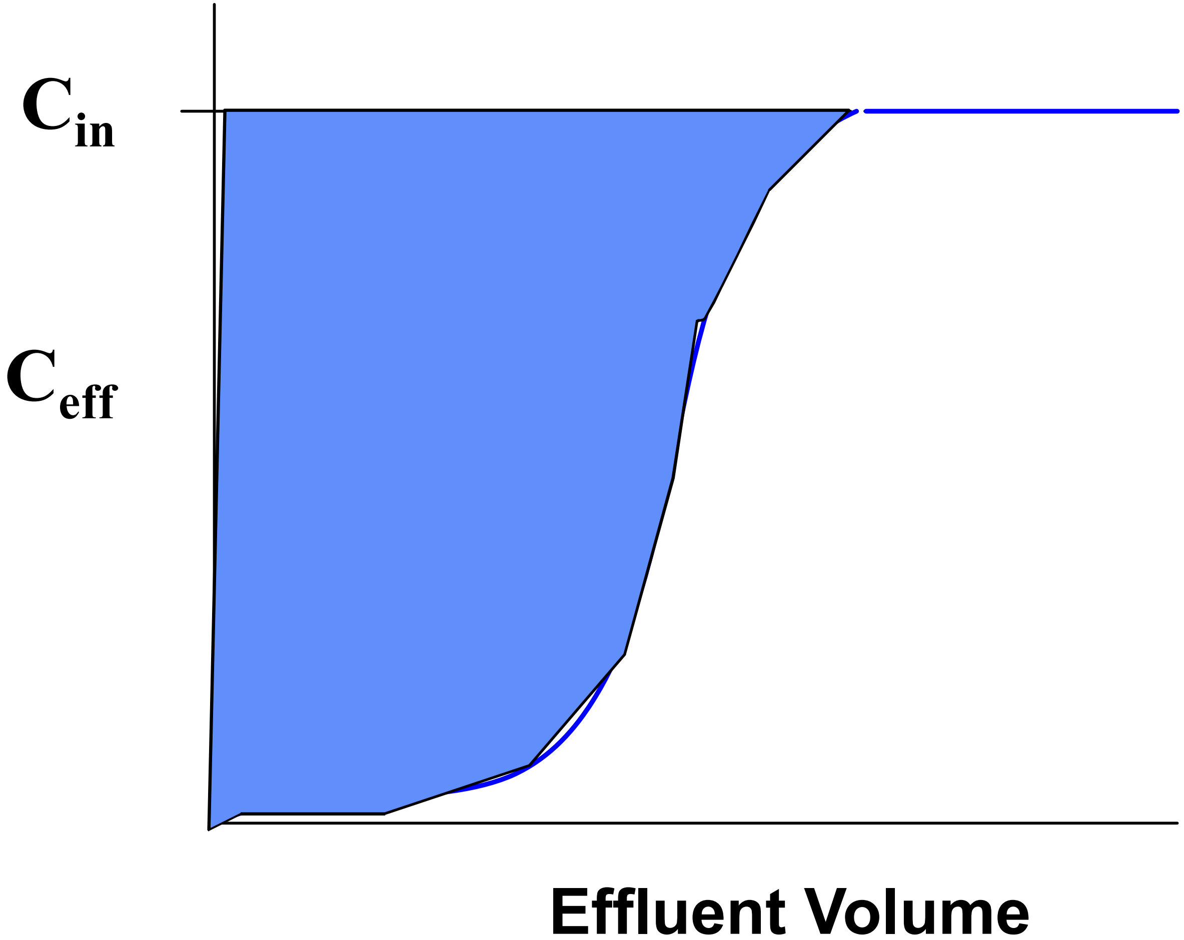
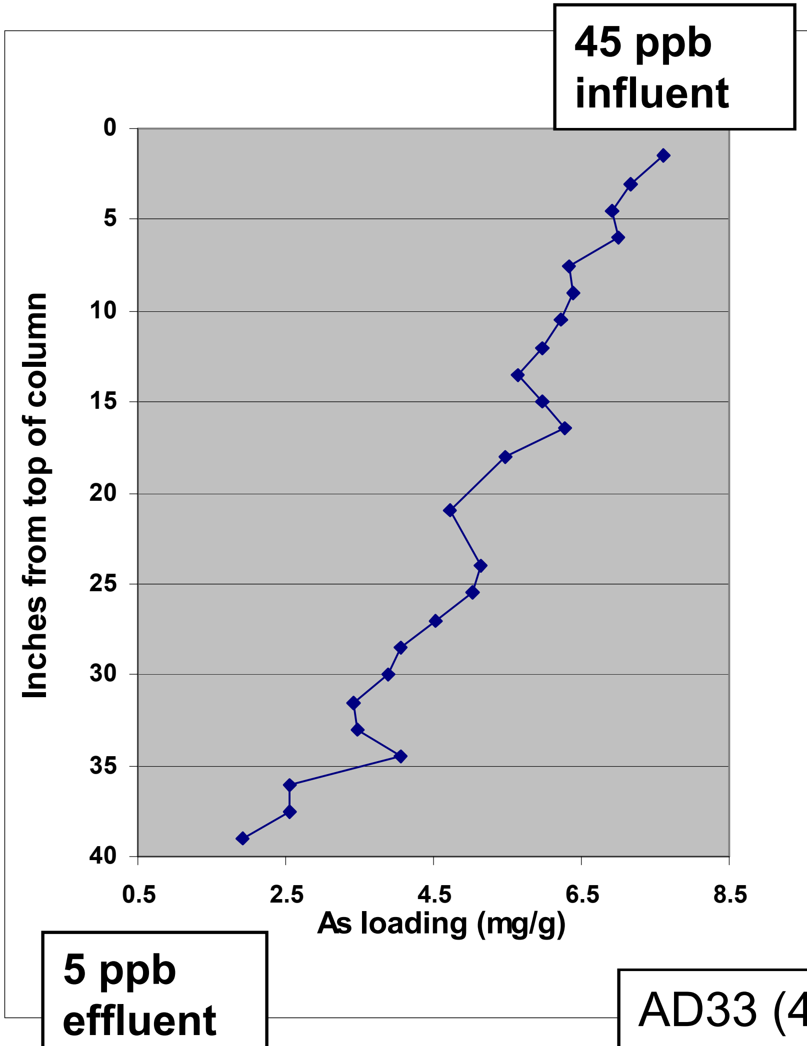
Calculation of Column Arsenic Loading Capacity



Spent Core Analyses – *in situ* K_d and capacity



AD33 (4 min EBCT)

- **Arsenic leached from 1 g samples taken every 1.5 inches.**
- **Sorption equilibria:**
- **K_d top = 7604/0.045 = 152,080 ml/g**
- **K_d bot = 1917/0.005 = 383,400 ml/g**
- **Total arsenic content**
  - **Assume As loading constant for 1.5" thick disks.**
  - **Sum media mass and As content to obtain average concentration and capacity of column.**
- **As capacity = 5.08 mg As/g media.**
- **As Capacity from mass balance on pilot effluent/influent**
- **> 4.48 mg/g As mg /g media**
- **Agreement within 10%!!**

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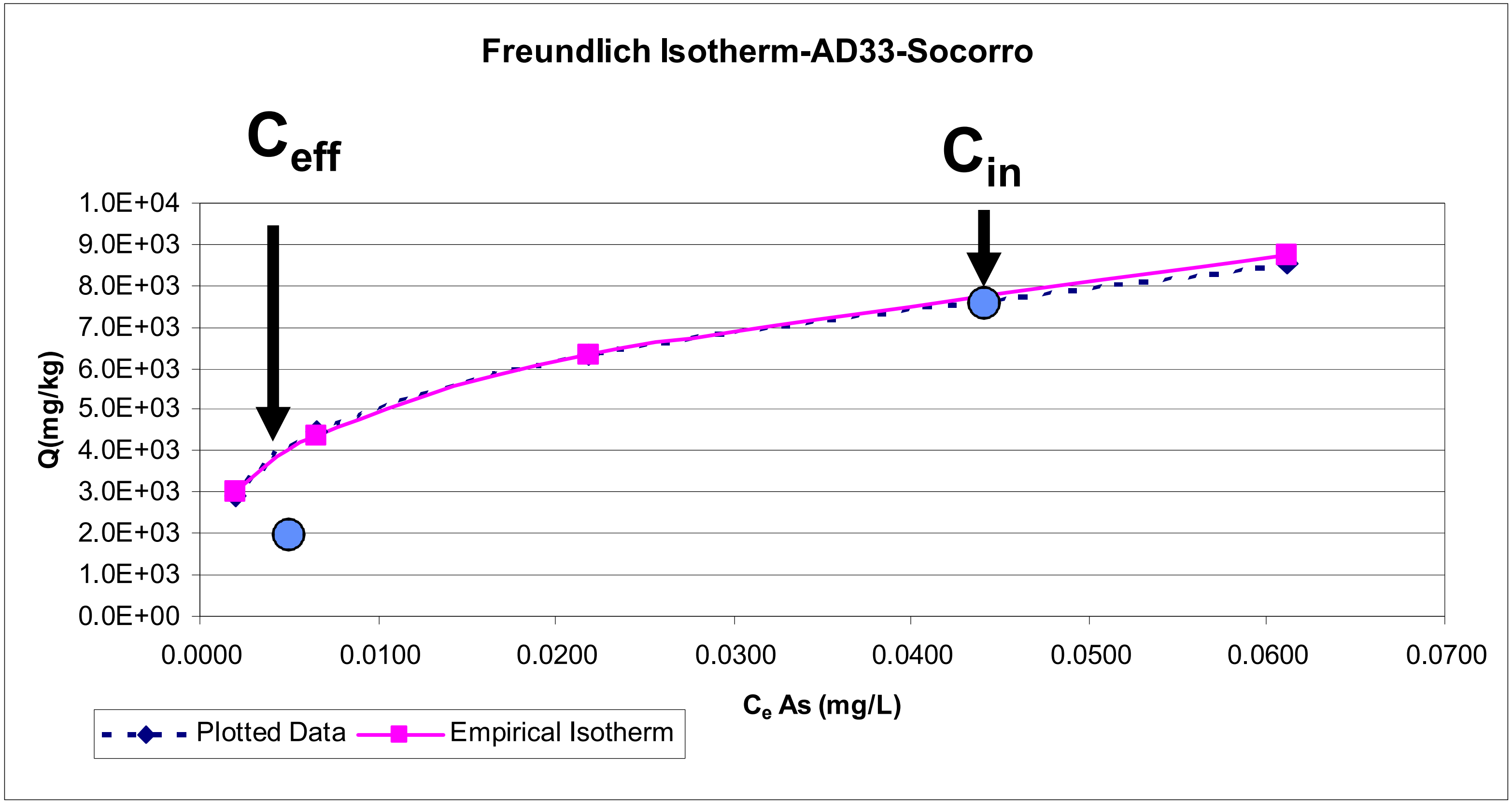
Isotherm Studies



n_F=0.3131, K_F=2.1E4

Column data

Estimates of Arsenic Sorption Capacity from Different Tests

	AD33	Pre-prod ARM200	Metsorb
BV to 10ppb (pilot)	43,000	8,600	13,000
As at 10ppb (pilot)	3.56 mg/g	0.6 mg/g	0.7 mg/g
BV to 10ppb (RSSCT)	43,000 (PD)	6000 (CD)	12,800 (PD)
As at 10 ppb (RSSCT)	3.39 mg/g (PD)	0.42 mg/g (CD)	0.69 mg/g (PD)
As at 10 ppb (Freundlich)	5.0 mg/g	3.6 mg/g	1.2 mg/g

BV = bed volumes, PD = proportional diffusivity, CD = constant diffusivity

As = capacity calculated from mass balance loading or batch test

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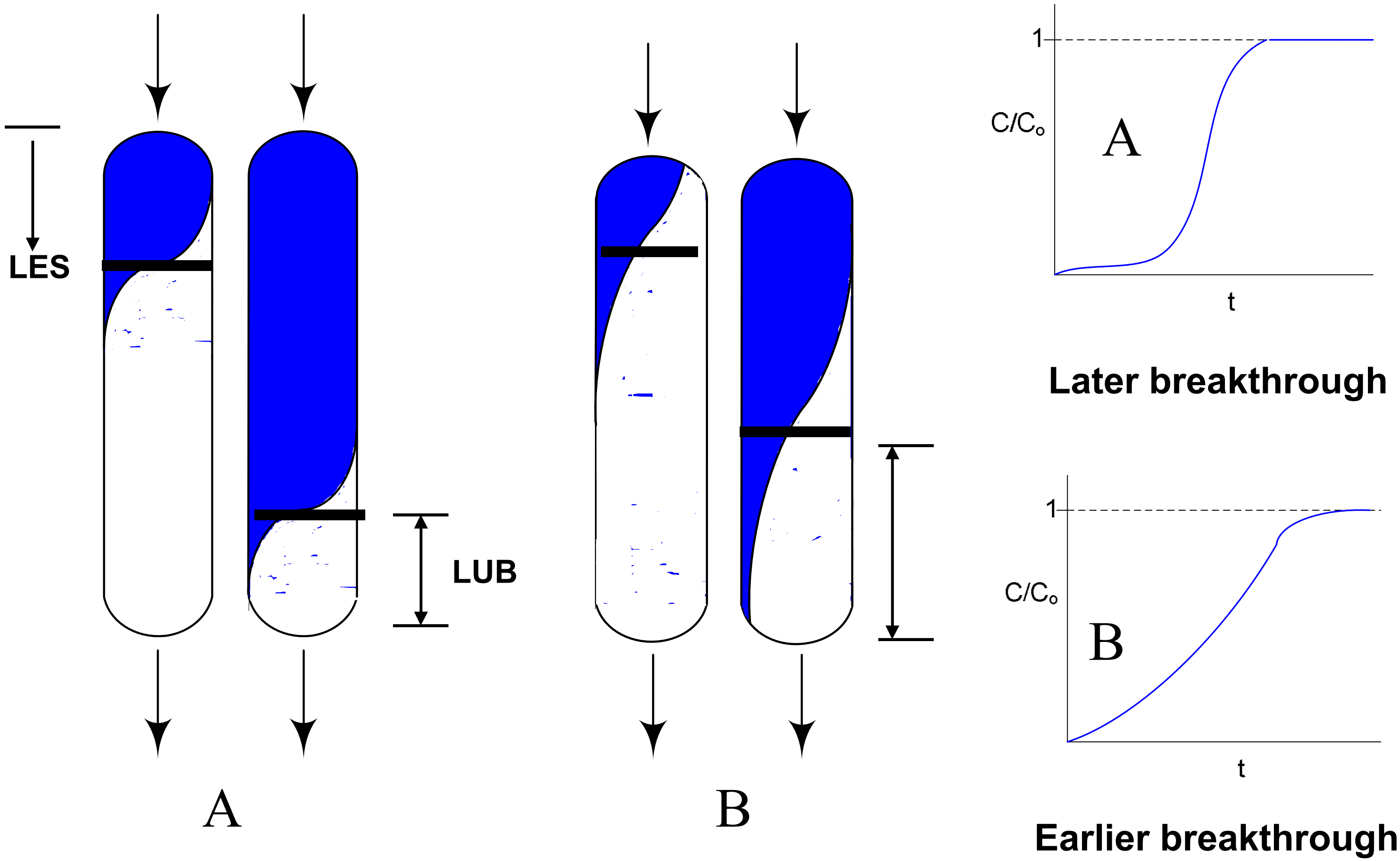
Shape of Mass Transfer Zone Determines Capacity



LES = Length of Equilibrium Bed   LUB = Length of Unused Bed

Bed Efficiencies of Sorbent Media Columns

Bed Efficiency = 10 ppb pilot capacity/45 ppb batch capacity x 100%

	AD33 (4 min)	*ARM200	*AsX^{np}	Metsorb
As at 10ppb (pilot)	3.6 mg/g	0.6 mg/g	1.4 mg/g	0.7 mg/g
As at 10 ppb (Freundlich)	5.0 mg/g	3.6 mg/g	4.6 mg/g	1.3 mg/g
As at 45 ppb (Freundlich)	7.7 mg/g	8.0 mg/g	10 mg/g	4.5 mg/g
Bed Efficiency %	47	8	14	16

* pre-production or defective media according to vendors

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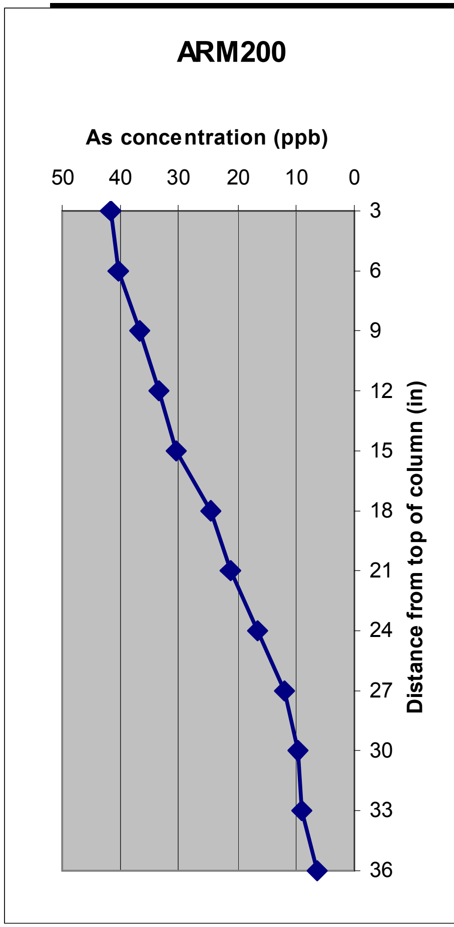
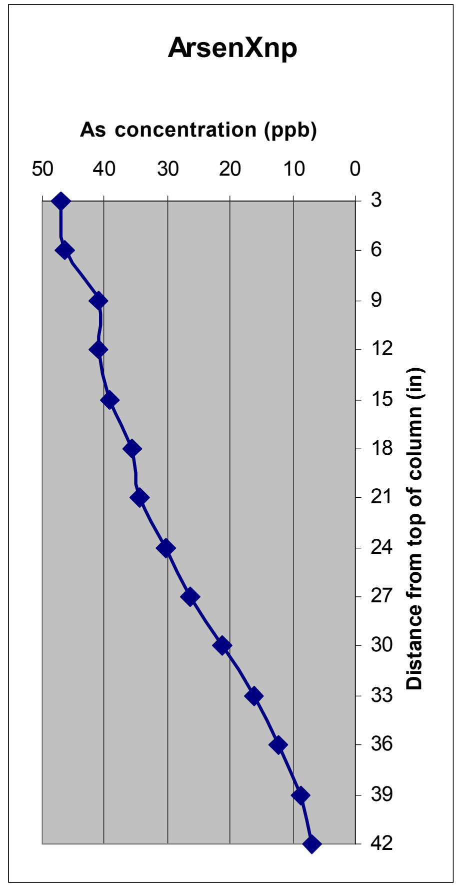
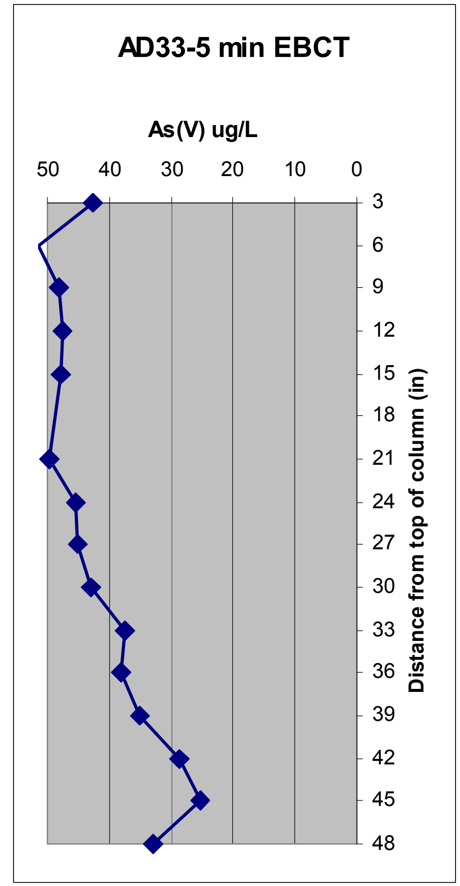
Pore Water Analyses Profiles are consistent with calculated bed efficiencies.







Efficiencies: 8% **16%** **Efficiency: 47%**

Larger Length of Unused Bed (LUB)

Smaller LUB

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Summary: Future Plans

- Technology Evaluation
  - Complete documentation of Vendors Forum and make accessible on web.
  - Evaluate media from AwwaRF and WERC programs for pilots
- Pilot Studies
  - Completion of ongoing 40+ studies at 4 pilot sites
  - ‘hedgehog’ and in-line As sensor studies in Rio Rancho
  - New pilots in Oklahoma, Navajo Reservation and Placitas, NM
- Rapid Test Method Development
  - Particle attrition and crush strength tests to ‘predict’ hydraulics
  - Replicate RSSCT studies to assess test precision
  - Mass transfer zone analysis to develop predictive tool from short-term test data

Additional slides

Technologies

For More Information:

Arsenic Partnership Website

http://www.arsenicpartners.org/

Sandia Website

http://www.sandia.gov/water/arsenic

WERC CoAsT Website

http://wercstation.nmsu.edu:8080/arsenic/AsTree.dsb

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Top Five Ranked Vendors at Forums

2003	2004	2005
Hydroglobe – TIO_2	Purolite – Hybrid resin	Purolite
MEI - ZrO_2	Engelhard - GFO	ResinTech
Kinetico	*Filtronics*	EaglePicher – La-coated DE
AdEdge - GFO	DOW – TiO_2	ADA – Coated silicate
Filtronics	ResinTech – Hybrid resin	Virotec – mixed oxides from Bauxite

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Sorptive Media Projects Funded in 2004

- **Developing a New Class of Ion Exchangers for Selective Removal of Arsenic** (Cu-polymeric ligand exchanger)
- **Agglomerated Nanoparticle Media** (TiO_2/polymeric binders)
- **Aerogel & Iron-Oxide Impregnated GAC** (composite materials from hydrophobic sol-gel precursors + Fe-Mn-GAC chemical agents)
- **High Efficiency & Cost-Effective Zirconium & Titanium-Based Nanocomposites for Removal of Arsenic from Drinking water** (doping Ti and Zr oxide sorbents to improve performnace).
- **As Removal onto Activated Carbon Preloaded w/ Surfactant-Iron Complexes** (series system: As-Fe-complexes sorb onto tailored Fe- organic-GAC bed)

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New Sorptive Media Projects Funded in 2005

- **$FeCO_3$(s) as an inexhaustible source of $Fe(OH)_3$(s) for As removal** (granular siderite packed bed)
- **Evaluation of innovative regenerable & non regenerable adsorption media for As removal** (Field-scale comparison of 2 regenerable media (AsX^{np} and Absorbtia –GTO)
- **Low-cost As removal w/ treated coal ash** (Use bottom ash as substrate for Fe-oxide coating in batch systems)
- **Metal-doped hydro-gel media for As removal & brine minimization** (Biopolymer with Fe immobilized throughout structure by coordination with carboxylate functional groups; can be dehydrated for low volume disposal)
- **Removal of As by sorption to iron-coated fibers**

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AwwaRF Phase II Sorptive Media Projects: 2006 starts

- Fe and Ti- impregnated Granular Activated Carbon
  - Team: ASU, Clemson, SolmetTex
  - Optimize Fe oxide–GAC formulation for iron coverage and arsenic removal
  - Investigate TiO_2 -impregnated GAC
  - Investigate multiple contaminant removal
    - Arsenic, uranium, SOC
- GAC Modified with Organic Carboxyl-metal Complexes
  - Pennsylvania State University
  - Develop series treatment systems for small utilities
    - Zero-valent iron source for FeOOH sorbent
    - Removal of As-Fe complex by modified GAC bed
- Polymeric Ligand Exchanger for Highly Selective and Regenerative Arsenic Removal
  - Auburn State University
  - Test DOW 3N-Cu resin in field pilot
  - optimize operating parameters (EBCT, column config.)
  - Optimize regeneration with brine

2003 and 2004 WERC Design Contests

2003: Arsenic Treatment for Small Water Delivery and Domestic Water Systems

2004: Arsenic Treatment for Domestic Water Systems

- **Teams developed and demonstrated a cost-effective treatment technology to remove arsenic from drinking water in small water delivery systems and domestic water systems.**
  - **2003**: **11 teams**: Clarkson, Clemson, Lafayette College, Mich. Tech., Montana Tech, Ohio University, SD School of Technology, Thadomal Shahani (India), Univ. ID, Univ. New Hampshire, Univ. Waterloo**.**
  - **2004: 6 teams:** Dalhousie University (Canada), LSU, Montana Tech., Ohio State University, Tufts Univ., and Widener Univ.

2005 and 2006 WERC Design Contests

2005 - Arsenic Treatment for Rural Isolated Communities

- **Develop and demonstrate a cost-effective, energy-efficient treatment technology to remove arsenic and nitrate from drinking water in the presence of other competing ions such as silica and phosphate in rural isolated communities.**
  - 11 teams: Clemson, Duke, Lafayette College, Montana Tech., NMSU, Stevens Inst. Of Tech., Univ. Manitoba, Univ. NM, Univ. Waterloo, Univ. Wyoming, Washing Univ. at St. Louis.

2006 - Arsenic Treatment for Rural Isolated Communities

  - **remove arsenic from (high TDS = 1000) ppm) challenge water**

Additional slides

Pilots

New Mexico Pilot Sites – Water Quality Summary (Average Values)

	Socorro	Anthony	Rio Rancho	Jemez Pueblo
Total As/As(III), ppb	42 / <2	20 / 18	19 / < 1	20 / 19
V (ppb)	11	2	15	<1
SiO2 (ppm)	25	37	27	50
SO4 (ppm)	29	180	100	24
Ca (ppm $CaCO_3$)	44	70	55	155
Fe (ppm)	0.04	*0.5*	0.15	*1.2*
pH	8.0	7.7	7.6	7.5
Conductivity (μS/cm)	340	*1400*	*620*	*770*
Alkalinity (ppm $CaCO_3$)	130	180	160	290
TOC (ppm)	0.5	0.8	0.5	2.0
NO_3 (ppm N)	0.2	0.0	2.0	0.0
F (ppm)	0.50	0.50	0.90	1.00

RED = above EPA Primary MCL; ***BLUE*** = above EPA secondary MCL

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Parametric Study: Socorro, NM

- **Effect of EBCT on Arsenic Removal Capacity**

Parameter	AD33		
	2 min	4 min	5 min
BV to 10 ppb	24,000	43,000	42,000
Capacity at 10 ppb, mg/g	1.95	3.56	3.47
Capacity at 35K BV, mg/g	2.59	3.01	2.92
Depletion - C/Co at 35K BV	0.50	0.15	0.12
BV at C/Co = 0.8	84,000	>270,000	>235,000
Capacity at C/Co = 0.8	4.03	> 4.62	>3.47

Media Performance in Socorro, NM

- **Arsenic Removal Capacity**

Parameter	ARM200 FeOx	Metsorb - TiOx	*AsX^{np}	Isolux ZrOx	AD33 (FeOx)
BV to 10 ppb	8,600	13,000	27,000	32,000	43,000
Capacity at 10 ppb, mg/g	0.60	0.70	1.38	1.67	3.56
Capacity at 35K BV, mg/g	1.17	1.39	1.75	1.67	3.01
Depletion - C/Co at 35K BV	0.88	0.60	0.35	0.38	0.15
BV at C/Co = 0.8	33,000	87,000	53,000	63,000	>270,000
Capacity at C/Co = 0.8	1.15	2.26	2.10	2.23	> 4.62

*AsX^{np} batch was defective

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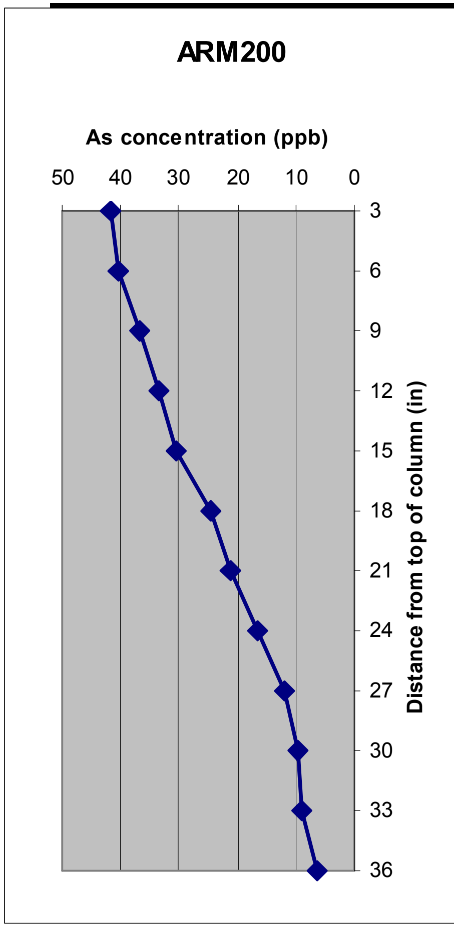
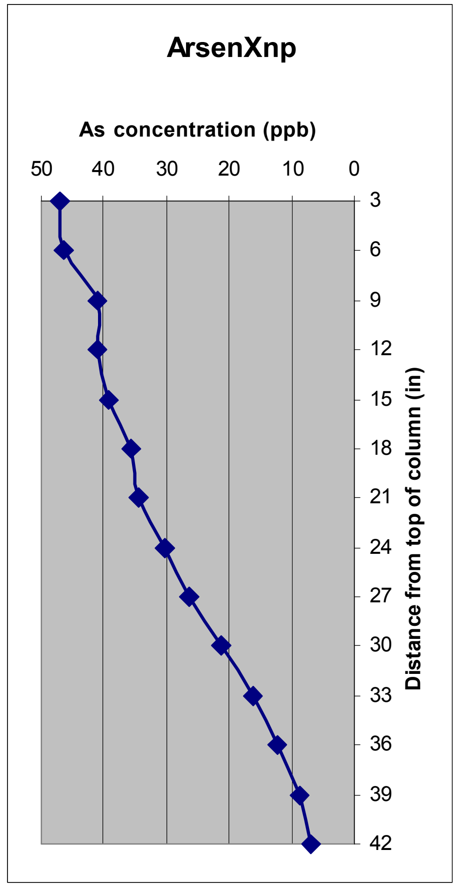
Hydraulic Test Results: Socorro, NM

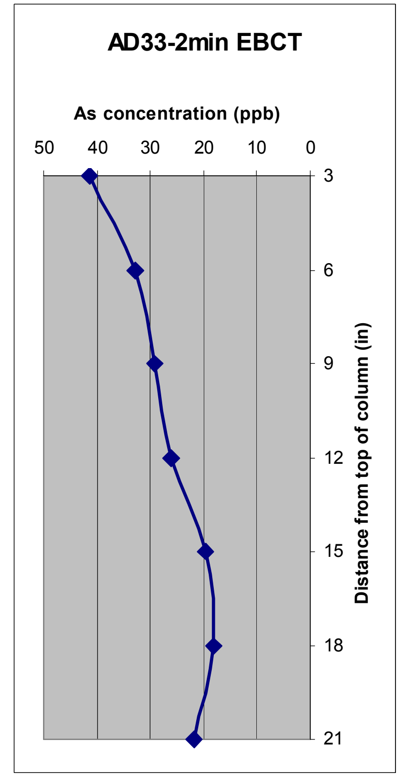
Results: Physical Observations

- Sieve Analysis: 0.8-29% media loss
- Particle Size Uniformity: All media had C_u < 5, most <2.5 (fairly uniform)
- Surface Area: Doesn't seem to control As removal – the media with the smallest surface area had the highest capacity
- Each column reacted differently to operating conditions
  - Media was lost due to backwashing
  - Media compacted throughout pilot experiment

Pore Water Analyses show homogeneous flow







1 month pH adjusted influent

After 4 months pH - adjusted influent

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Media Performance: Anthony, NM

- **Backwashing:**
  - **Some media have required monthly backwashing (~2,000 bed volumes)**
  - **Others have required bi-monthly backwashing (~4,000 bed volumes)**
  - **A few have required little or no backwashing**
- **Most media haven't compressed much (compressed<10% of original height)**
- **Can see iron oxide forming at top of TiOx media and ZrOx pre-filter**

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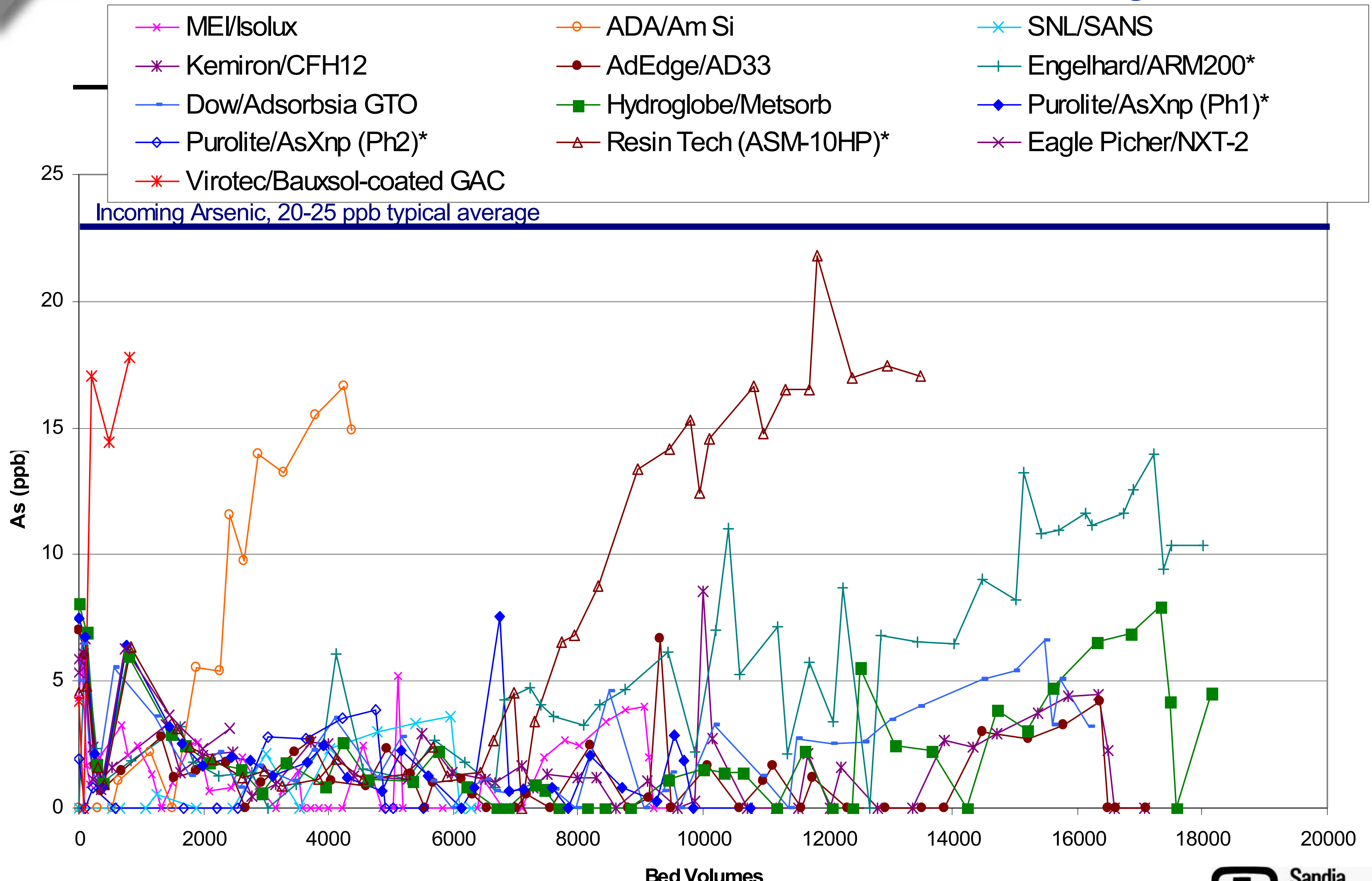
Media Performance: Anthony, NM



Indicates that the vendors' media was a pre-production batch and/or a poor quality batch of media

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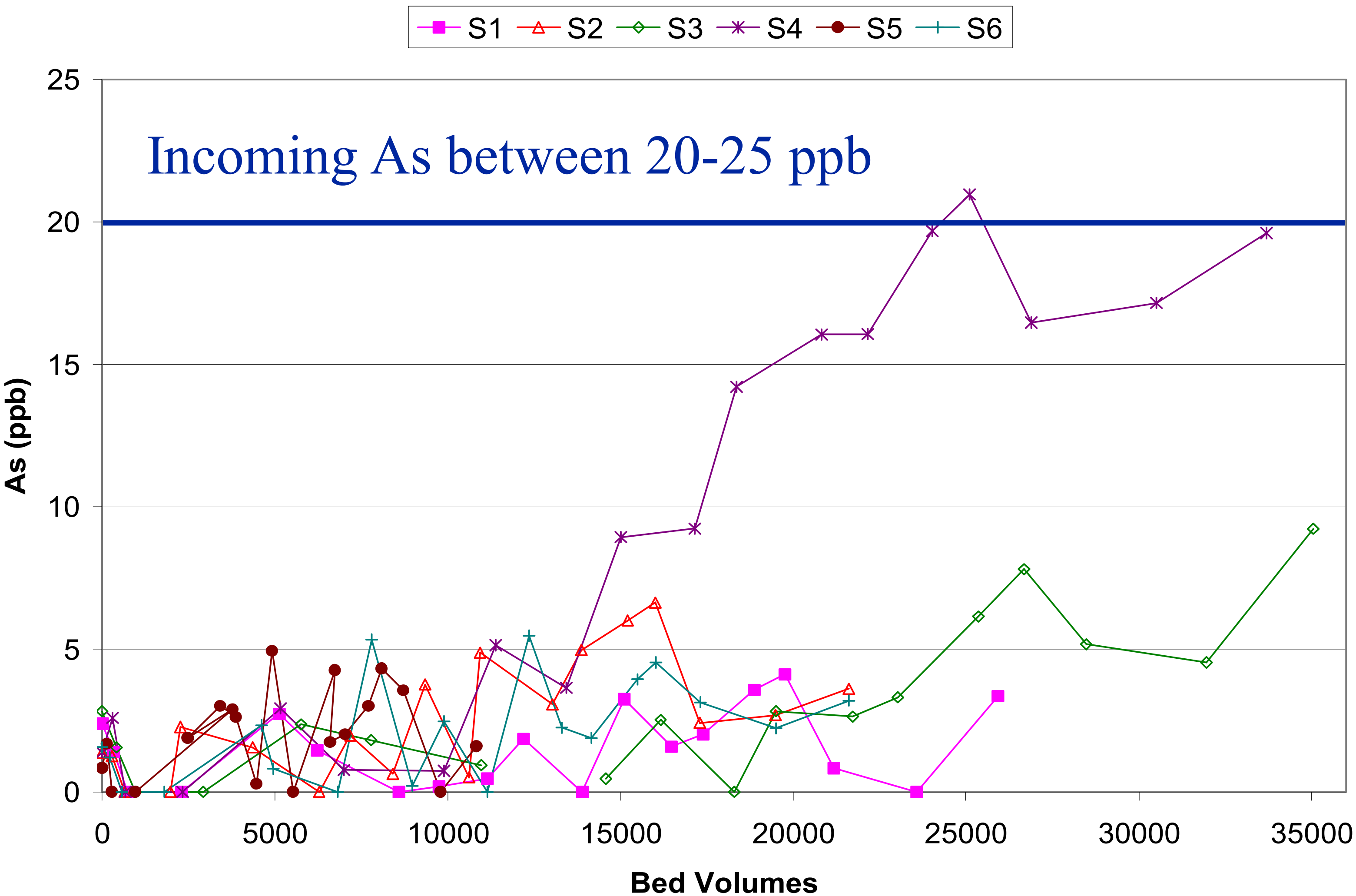
Media Performance: Rio Rancho, NM (Phase 1)

S1=AD33 (FeOx); S2 = CFH10 (FeOx); S3 =ArseneX^{np}; S4 = ASM-10HP; S5=Adsorbsia GTO (TiOx); S6=Isolux (ZrOx)



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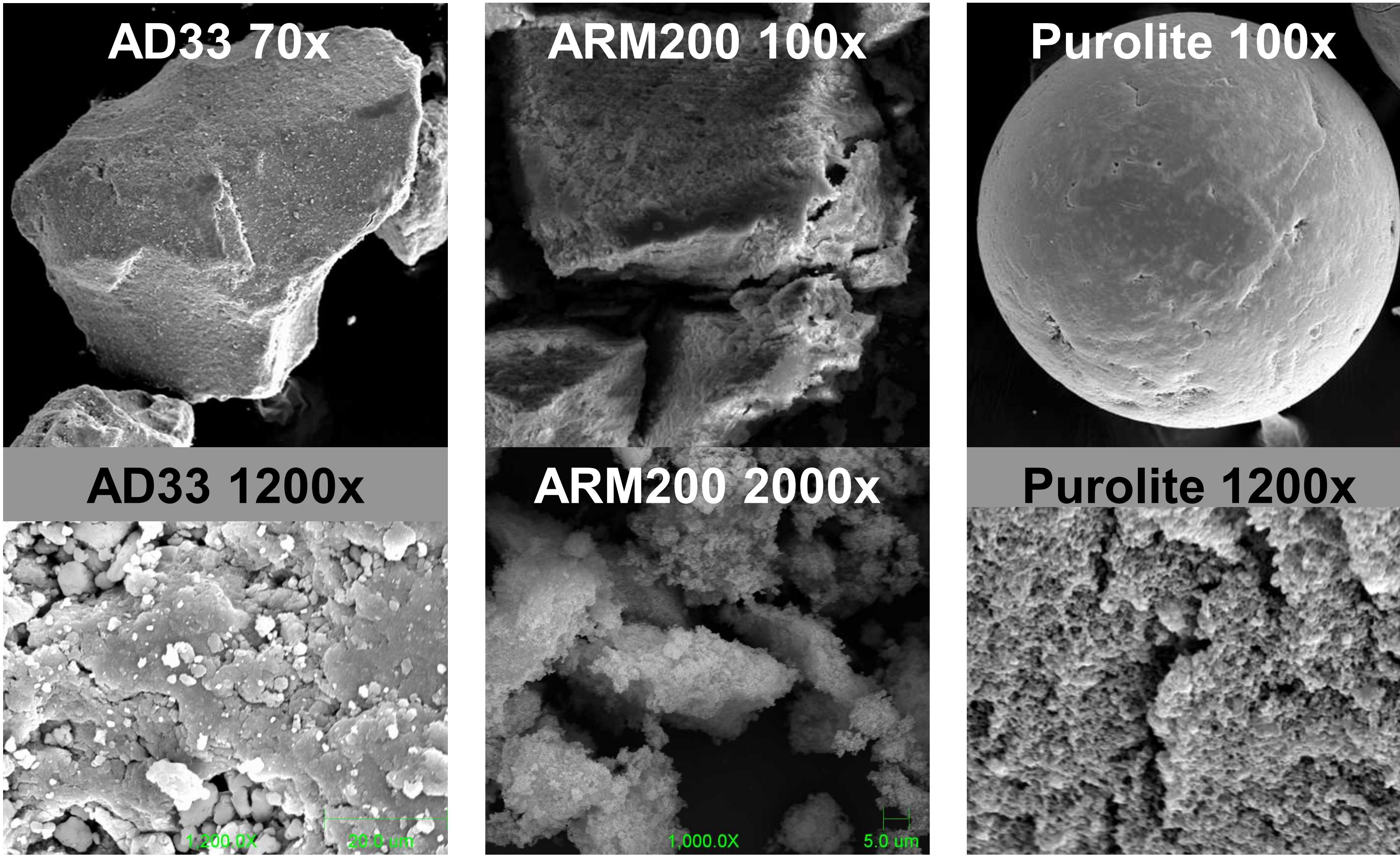
Additional Slides

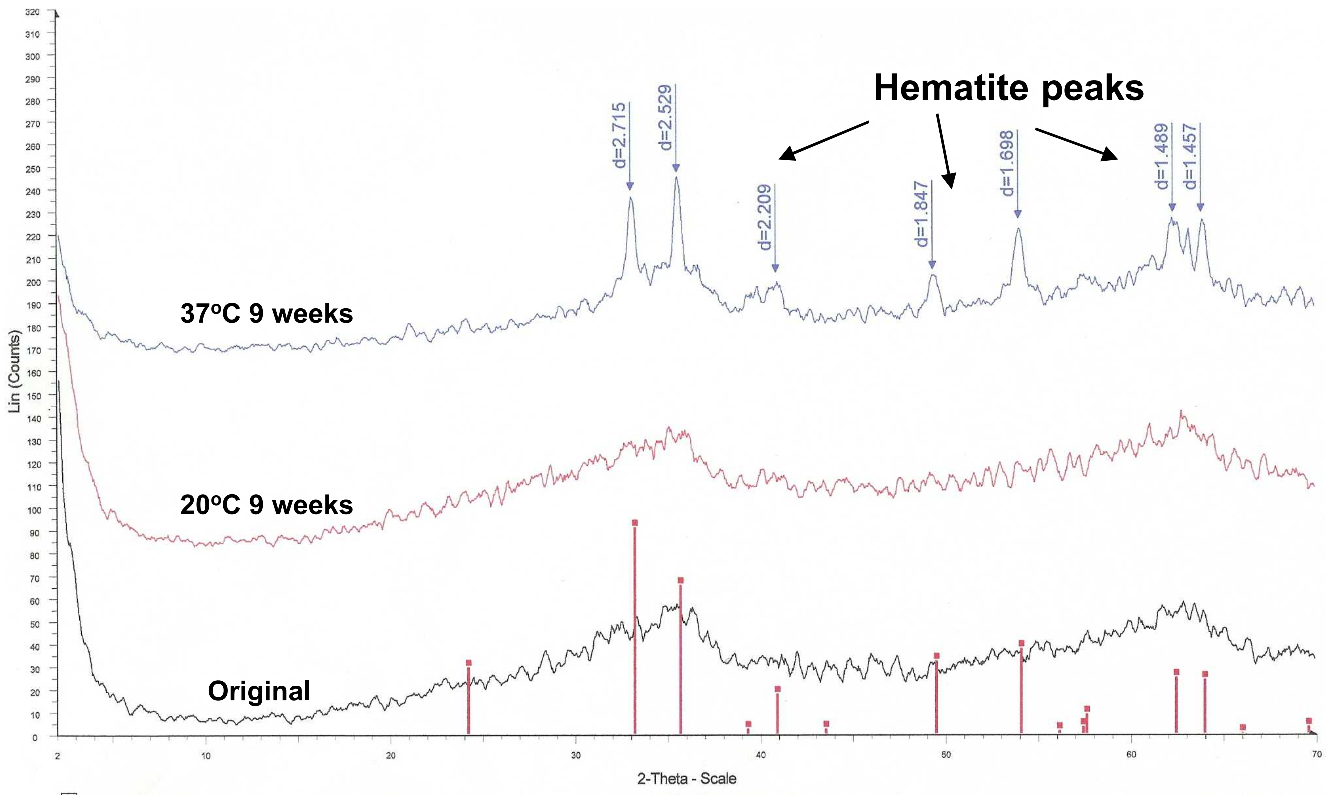
Laboratory studies

SEM Photos of Adsorption Media



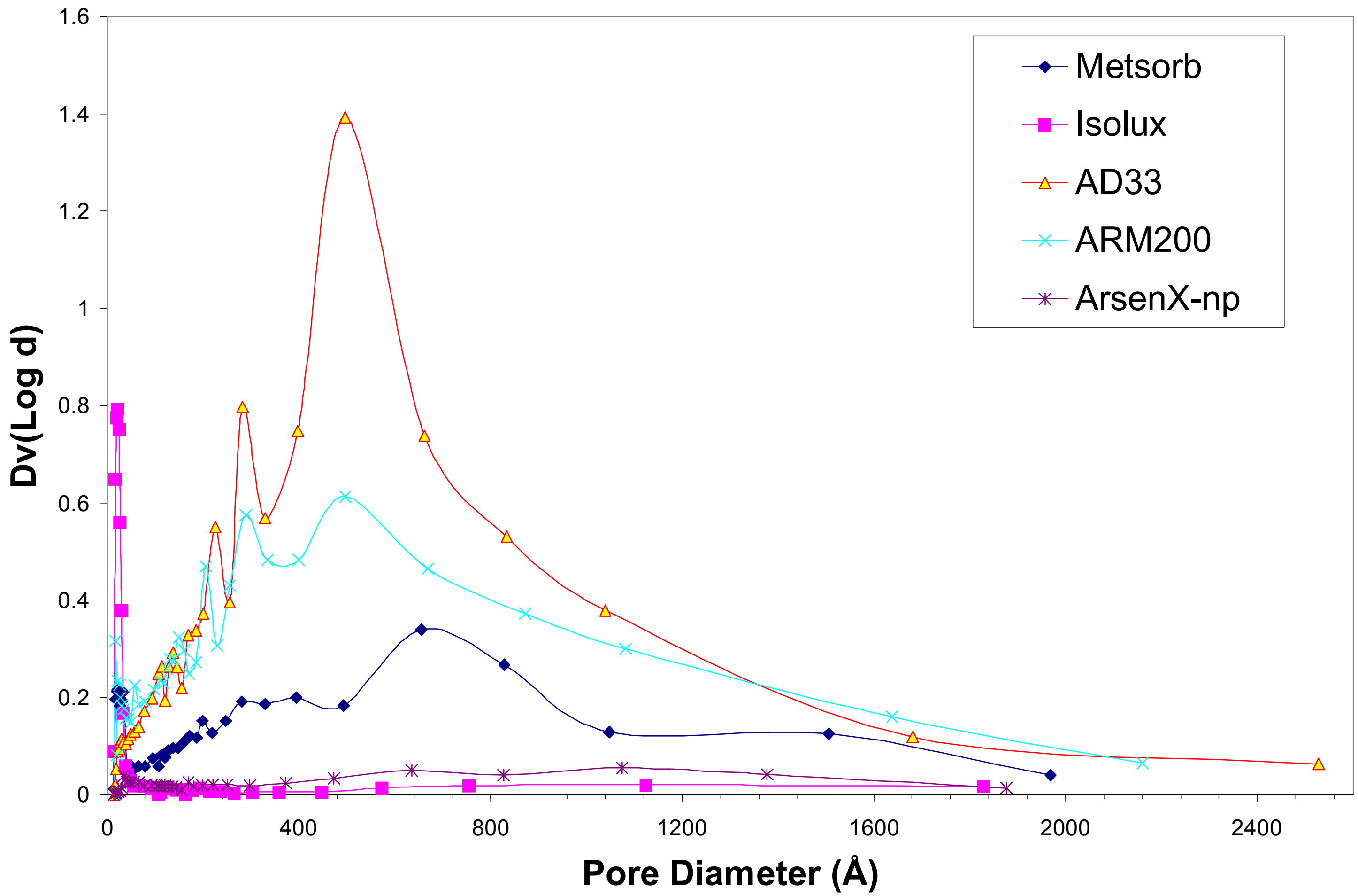
XRD Studies Used to Evaluate Potential Changes in Mineralogy of Media

ARM200



Recrystallization may impact performance.

Media have different pore size distributions



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Batch Sorption Studies

• Solution:solid (ml/g)	750-800
• Equilibration time	24 hrs (per kinetic studies)
• Particle size	325 – 400 mesh
• pH (initial)	7.7 – 8.1
• pH(final)	7.5 – 7.7
• Arsenic analysis	ICP-MS
• Isotherm fits	Langmuir and Freundlich
• Final As	3 - 80 ppb

Theoretical Scaling Relationships

Diffusivity factor (x) **Relationship between D_s and particle size**	$$\frac{D_{s,RSSCT}}{D_{s,pilot}} = \left[\frac{R_{RSSCT}}{R_{pilot}}\right]^{x}$$
Non-constant D_s (x = ?)	$$\frac{EBCT_{RSSCT}}{EBCT_{pilot}} = \left[\frac{R_{RSSCT}}{R_{pilot}}\right]^{2-x}$$
Proportional D_s (x = 1)	$$\frac{EBCT_{RSSCT}}{EBCT_{pilot}} = \left[\frac{R_{RSSCT}}{R_{pilot}}\right]$$
Constant D_s (x = 0)	$$\frac{EBCT_{RSSCT}}{EBCT_{pilot}} = \left[\frac{R_{RSSCT}}{R_{pilot}}\right]^{2}$$

10 ppb Breakthrough and Capacity