

# **MichiganTech**

## **A Summary of Proposed Changes to AASHTO M 295 Resulting from NCHRP Project 18-13 - Specifications and Protocols for Acceptance Tests of Fly Ash Used in Highway Concrete**

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## Acknowledgements

### NCHRP 18-13 Specifications and Protocols for Acceptance Tests of Fly Ash Used in Highway Concrete

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&

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## Disclaimer

The research work presented herein was performed as part of the National Cooperative Highway Research Program Project 18-13 “*Specifications and Protocols for Acceptance Tests of Fly Ash Used in Highway Concrete*”. This presentation does not necessarily indicate acceptance by the National Academies, the Federal Highway Administration, or by the American Association of State Highway Officials of the findings, conclusions, or recommendations either inferred or specifically expressed herein.

### Reference:

Sutter, L.L., R.D. Hooton, S. Schlorholtz. “NCHRP Report 749: *Fly Ash for Highway Concrete*.” National Cooperative Highway Research Program. Transportation Research Board. Washington D.C. (2013). (In press)

- Objective - recommend potential improvements to specifications and test protocols to determine the acceptability of fly ash for use in highway concrete

## NCHRP 18-13

- Characterization Study – evaluate existing specifications and classification methods for CFA
- Strength Test Study – investigate test methods for characterizing the strength activity of CFA
- Carbon Effects on Air Entrainment Study – develop test methods for characterizing the adsorption properties of residual carbon in CFA
- ASR Mitigation Study – examine test methods to evaluate use of CFA to mitigate alkali-silica reaction in concrete

## Characterization Study

- Gathered data on 100+ CFA sources
- Surveyed the SHAs to determine common sources used
- Selected 30 for comprehensive analysis
  - 17 Class F, 13 Class C
  - Selected sources from the 30 best suited for the other testing performed

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## Summary of 30 Sources

- Sum of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$ : 51.8 to 92.7%
- Calcium oxide ( $\text{CaO}$ ): 0.9 to 30.6%
- $\text{Na}_2\text{O}_e$ : 0.3 to 7.9%.
- **LOI: 0.1 to 5.6%** Also made blends to achieve higher LOI
- Fineness: 10 to 24.0%
- Strength Index (7-day test value): 75 to 112%
- Strength Index (28-day test value): 80 to 120%
- Water requirement: 93 to 100%
- Density: 2.1 to 2.8g per cubic-centimeter

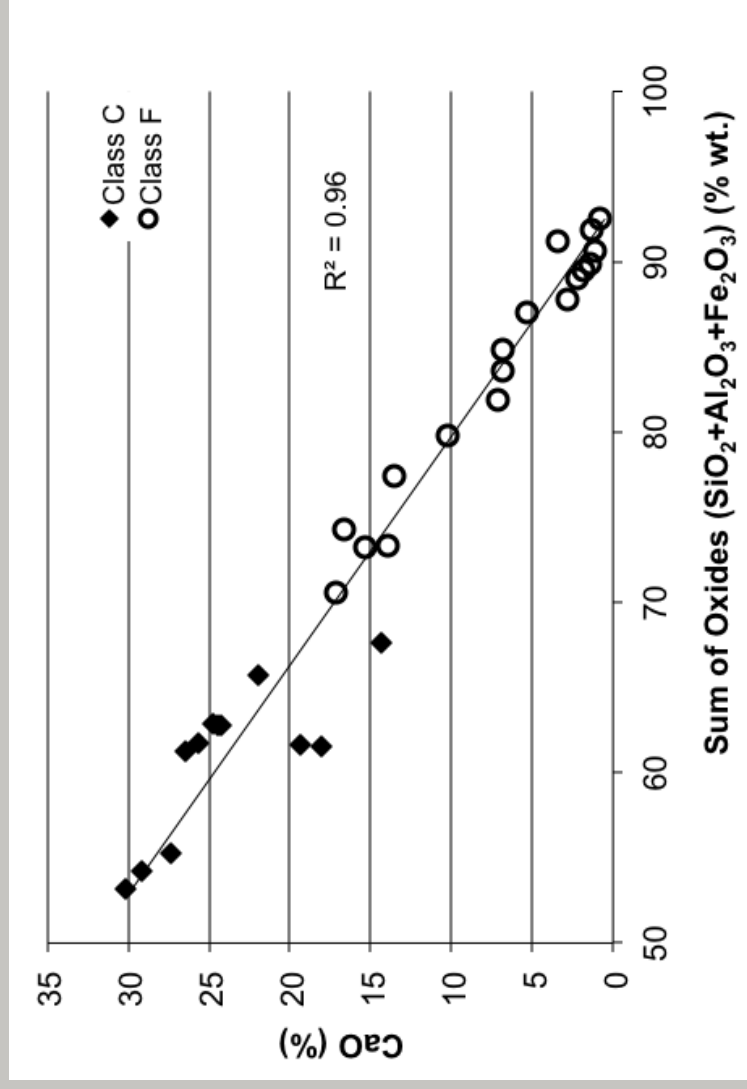
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## Characterization Study

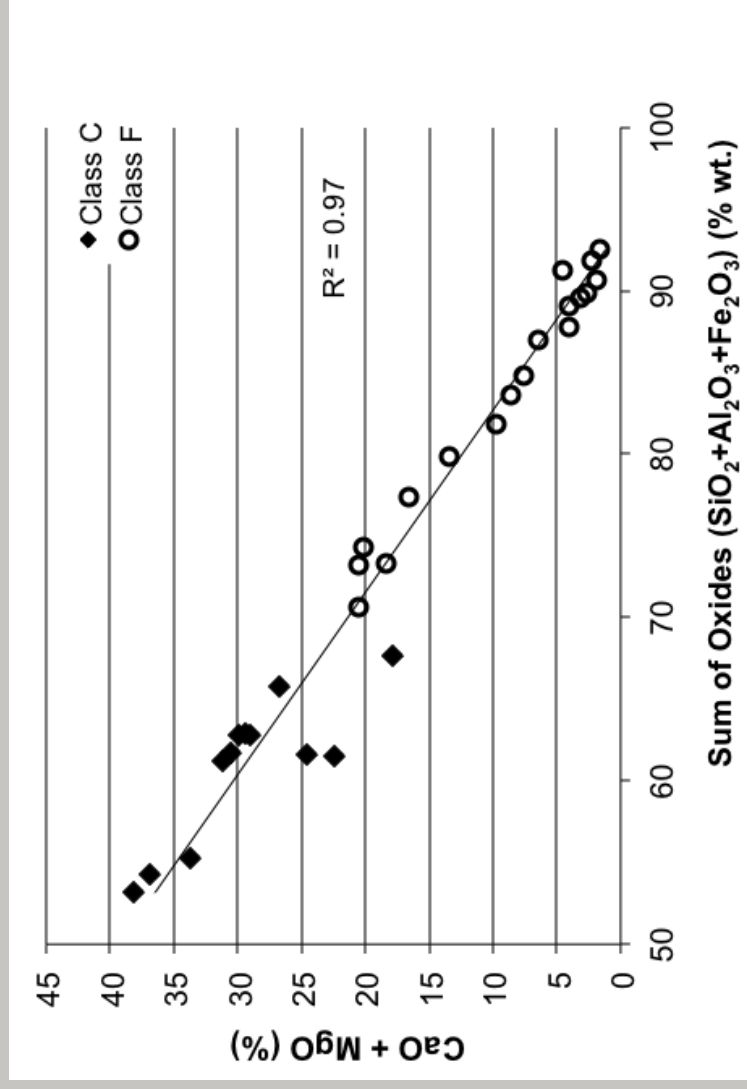
- Characterized 30 sources using ASTM C311 methods
  - All AASHTO M 295 Required and Optional Chemical and Physical Properties
  - Pozzolan Activity Index (PAI) using ASTM C311 methods
  - Qualitative XRD
- Quantitative XRD and TGA/DTA on 8 selected sources



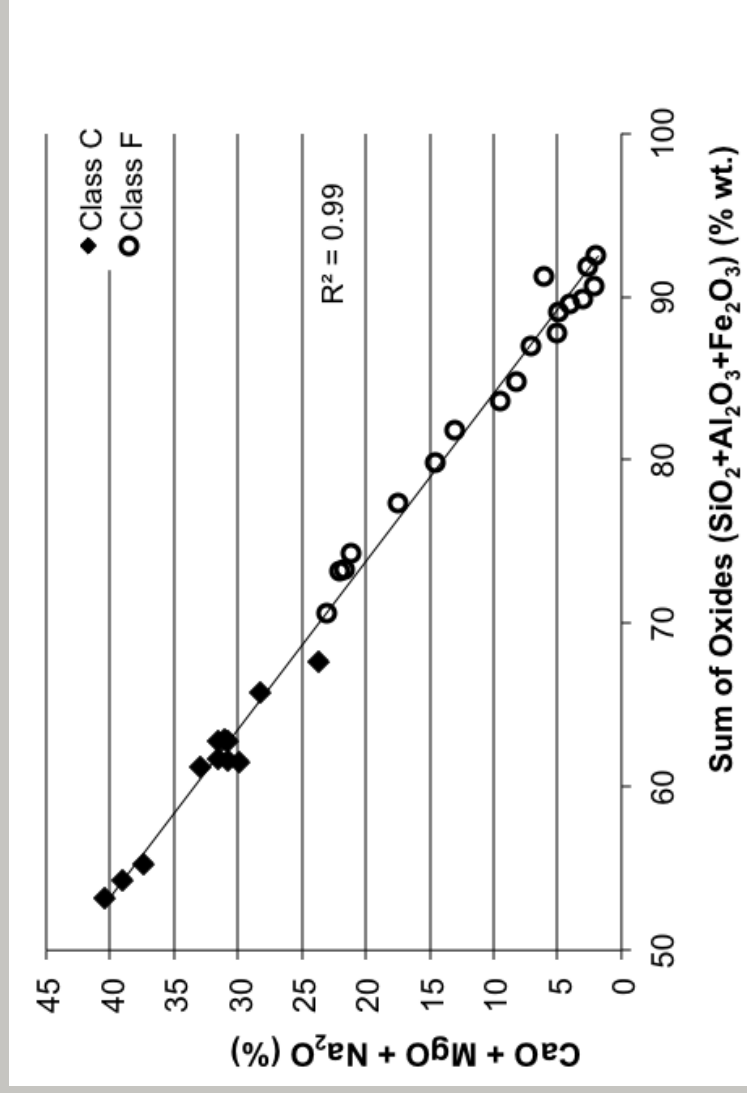
## Chemical Classification



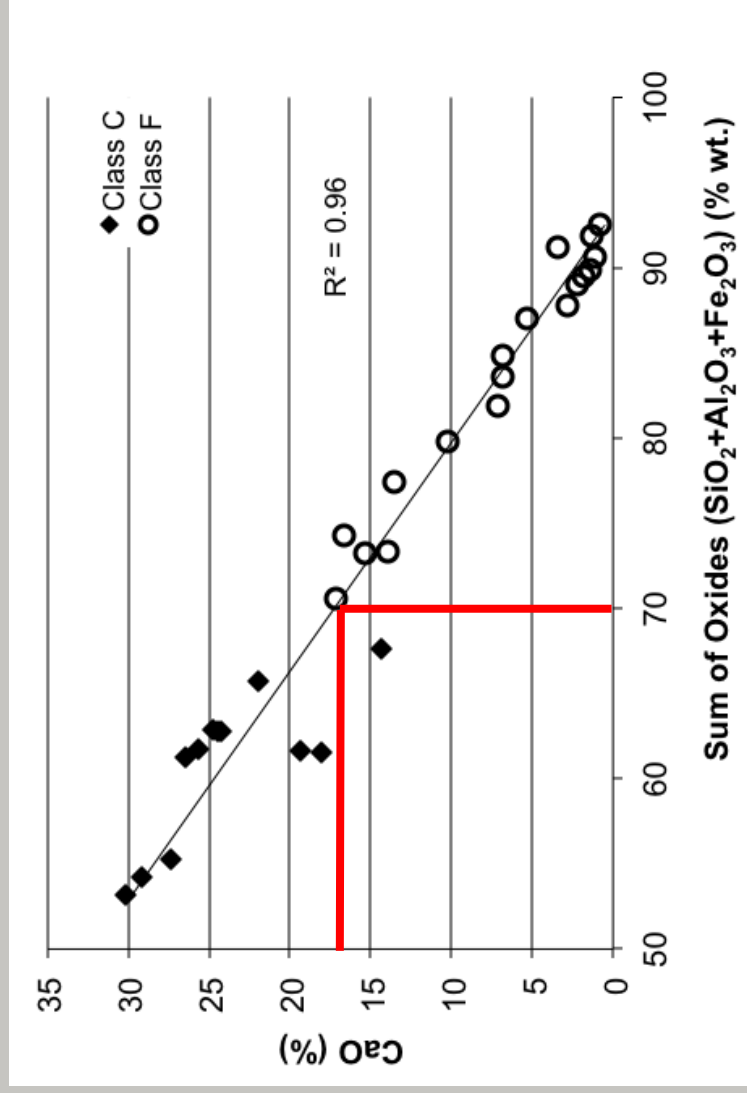
## Chemical Classification



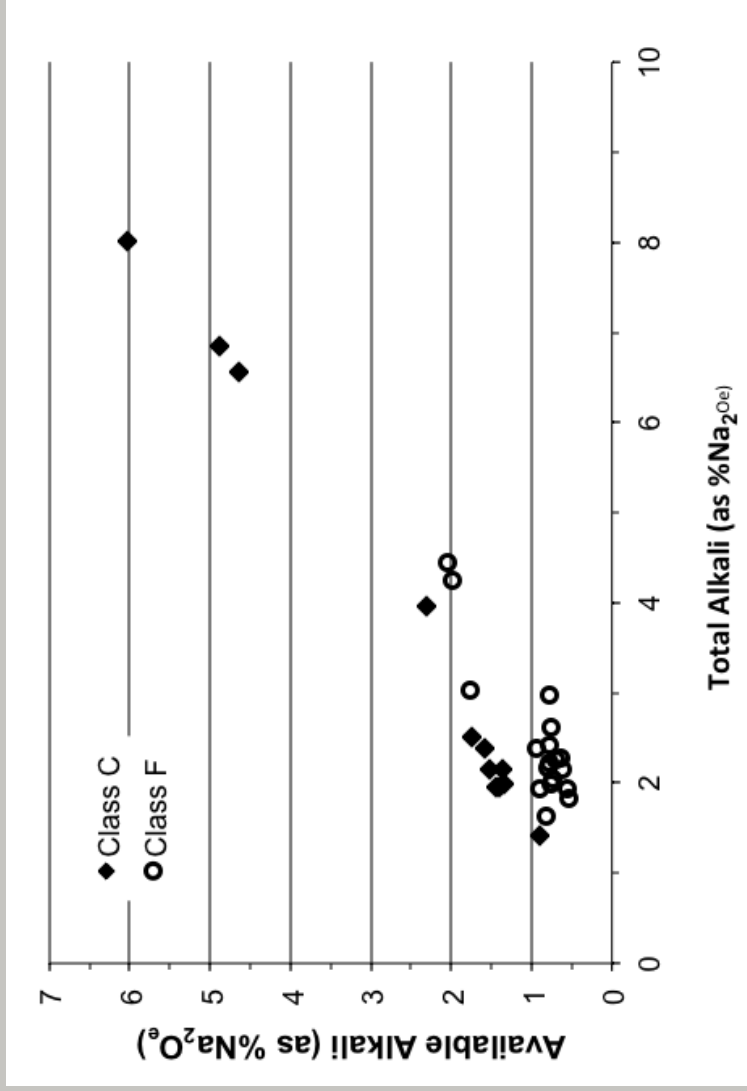
## Chemical Classification



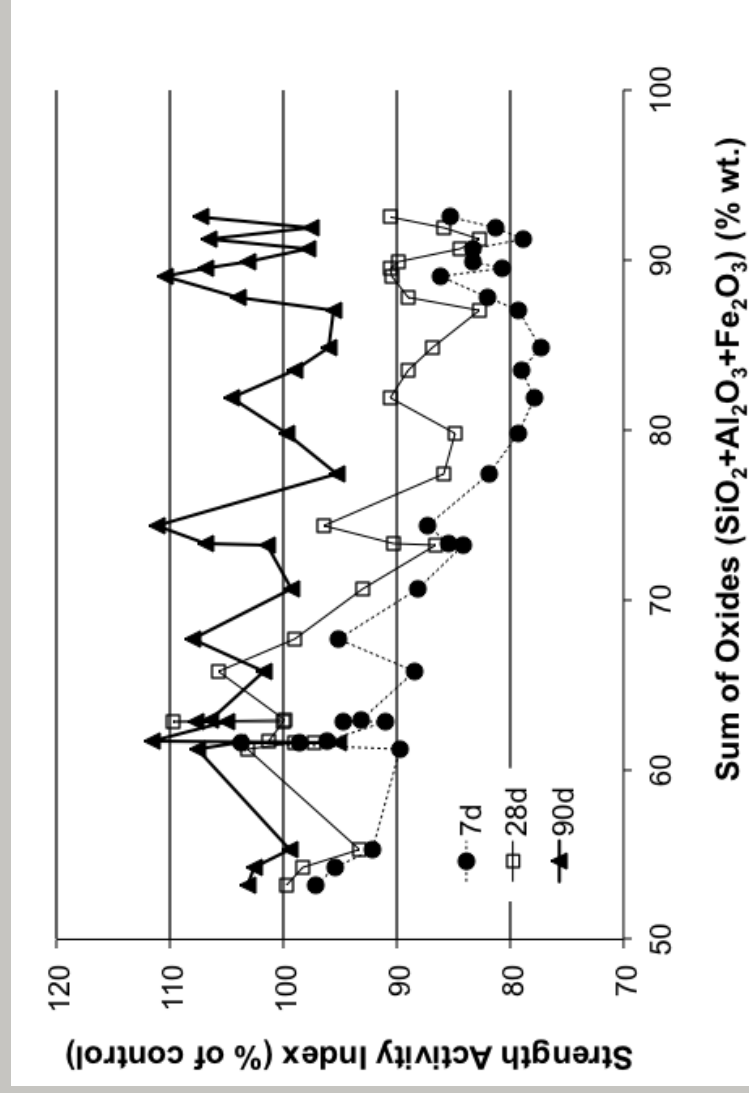
## Chemical Classification



## Available Alkali vs. Total Alkali



## Strength Activity Index



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## Strength Test Study

- Strength Activity Index is questioned as it allows inert materials to pass
- Experiments performed with non-pozzolanic quartz filler

Cement Type	Age (days)	100% Cement		20% Replacement		35% Replacement	
		Strength (psi)	SAI	Strength (psi)	SAI	Strength (psi)	SAI
PC-1	7	4554	84	3829	84	3075	68
PC-2	7	4293	79	3408	79	2640	62
PC-3	7	4090	87	3539	87	2886	71
PC-1	28	5715	84	4815	84	3945	69
PC-2	28	5526	77	4235	77	3655	66
PC-3	28	5134	85	4351	85	3307	64

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## Strength Test Study

- Evaluated the Keil Hydraulic Index
- Replace an equal percentage of the control sample cement with an inert filler
- Evaluated different fillers, replacement levels, and cements

$$\text{Keil Hydraulic Index} = \frac{a - c}{b - c} \times 100$$

Where:

**a** = the strength of 70% slag/30% portland cement at time **t**;

**b** = the strength of 100 percent portland cement at time **t**;

**c** = the strength of 70% ground quartz/30% portland cement at time **t**

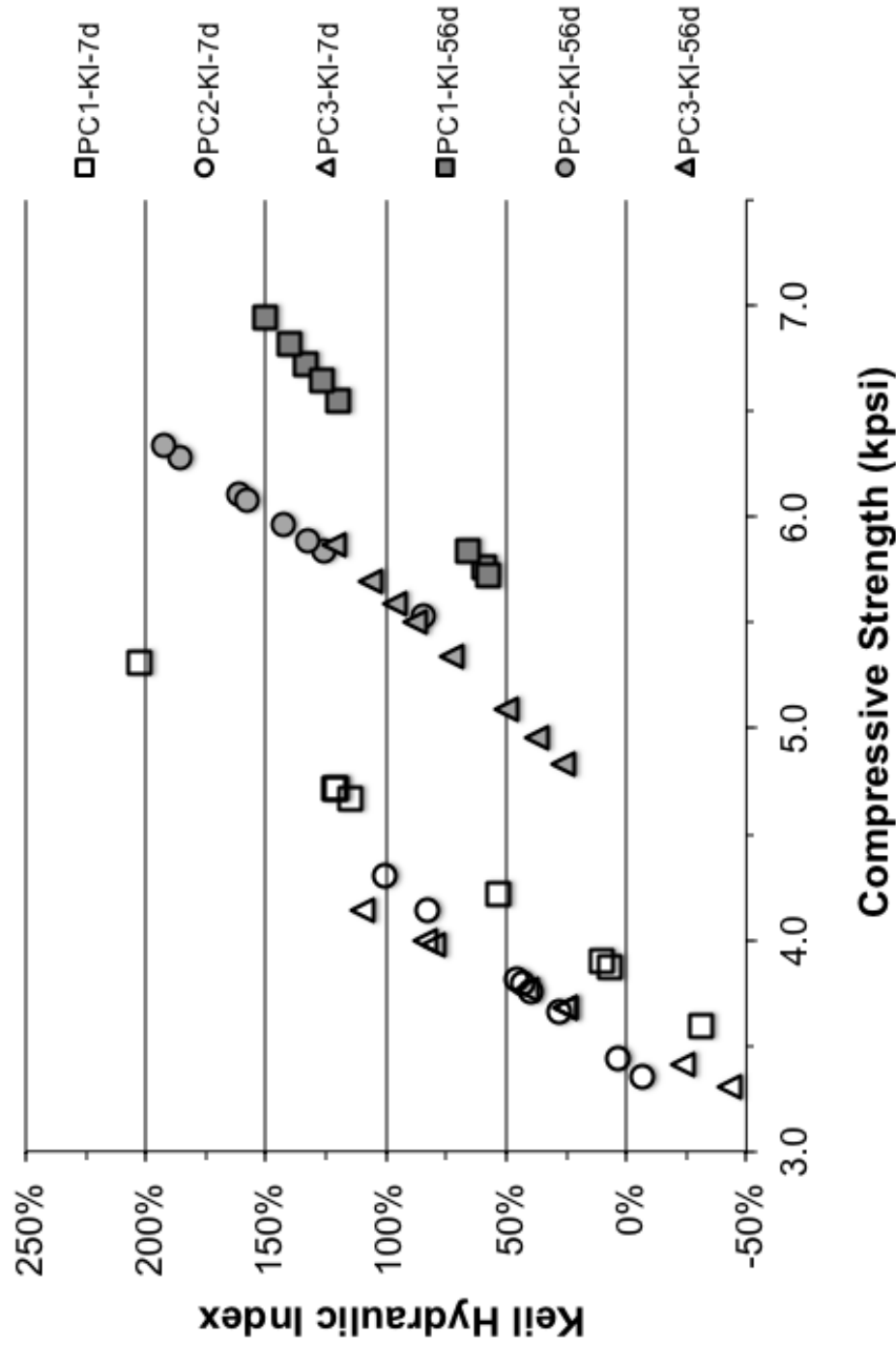
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## Keil Hydraulic Index

ID-% Replace.	KHI - 7 days (%)			KHI - 28 days (%)			KHI - 56 days (%)		
	PC-1	PC-2	PC-3	PC-1	PC-2	PC-3	PC-1	PC-2	PC-3
FA-H-20	-31	4	-43	71	91	66	60	162	88
FA-M-20	7	28	26	119	55	34	66	143	50
FA-O-20	10	-6	-24	7	73	39	57	84	26
FA-Q-20	53	44	26	135	102	109	120	185	121
FA-U-20	121	40	84	184	75	171	133	158	73
FA-X-20	115	101	80	96	30	72	127	126	96
FA-ZA-20	122	46	110	184	99	153	150	132	38
FA-ZC-20	203	83	41	138	119	130	140	193	106
FA-U-35	60	21	35	102	44	93	121	102	126
FA-X-35	89	74	110	118	68	94	78	114	82
FA-ZA-35	80	35	63	124	46	114	116	102	101
FA-ZC-35	140	45	39	83	75	82	102	99	96

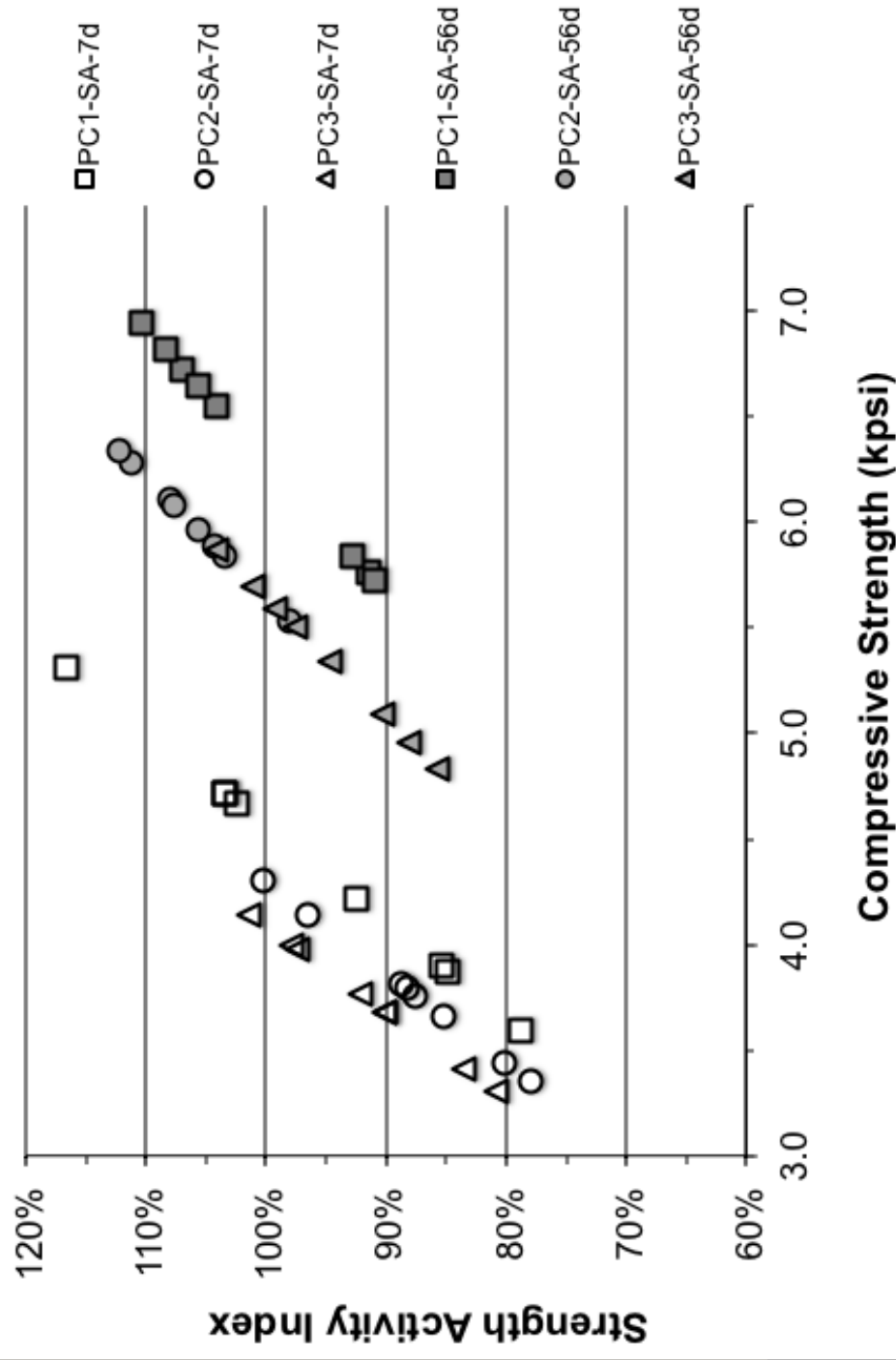
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## Keil Hydraulic Index



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## Strength Activity Index



## Strength Test Study

- *Take Aways*
  - The Keil Hydraulic Index provided a test that identified strength contribution separate from “filler” effects
  - The test was sensitive to the cement used
  - Other evaluations of the existing strength activity index showed increasing the specification limit to 85% eliminated inert materials
  - Need to change the time required for testing to accommodate some Class F ash

## Carbon Effects on Air Entrainment Study

- *Effect of Carbon on Air Entrainment*
  - The LOI test is adequate for estimating the total carbon but does not adequately identify if the carbon will effect air entrainment
  - There is a need for a test to directly determine adsorption capacity
  - The foam index test is useful at determining the interaction of the fly ash with air entrainment admixtures but has not been standardized and is not part of AASHTO M 295 or ASTM C311

## Carbon Effects on Air Entrainment Study

- Four tests evaluated:
  - Foam Drainage
  - Foam Index
  - Direct Adsorption Isotherm
  - Coal Fly Ash Iodine Number

## Foam Index Test

- Evaluated 16 published versions
- Adopted the methodology of Harris with some modifications

Harris, N. J., K. C. Hover, K. J. Folliard, and M. T. Ley. The Use of the Foam Index Test to Predict AEA Dosage in Concrete Containing Fly Ash: Part I-Evaluation of the State of Practice. *Journal of ASTM International*, Vol. 5, No. 7, 2008.

Harris, N. J., K. C. Hover, K. J. Folliard, and M. T. Ley. The Use of the Foam Index Test to Predict AEA Dosage in Concrete Containing Fly Ash: Part II-Development of a Standard Test Method: Apparatus and Procedure. *Journal of ASTM International*, Vol. 5, No. 7, 2008.

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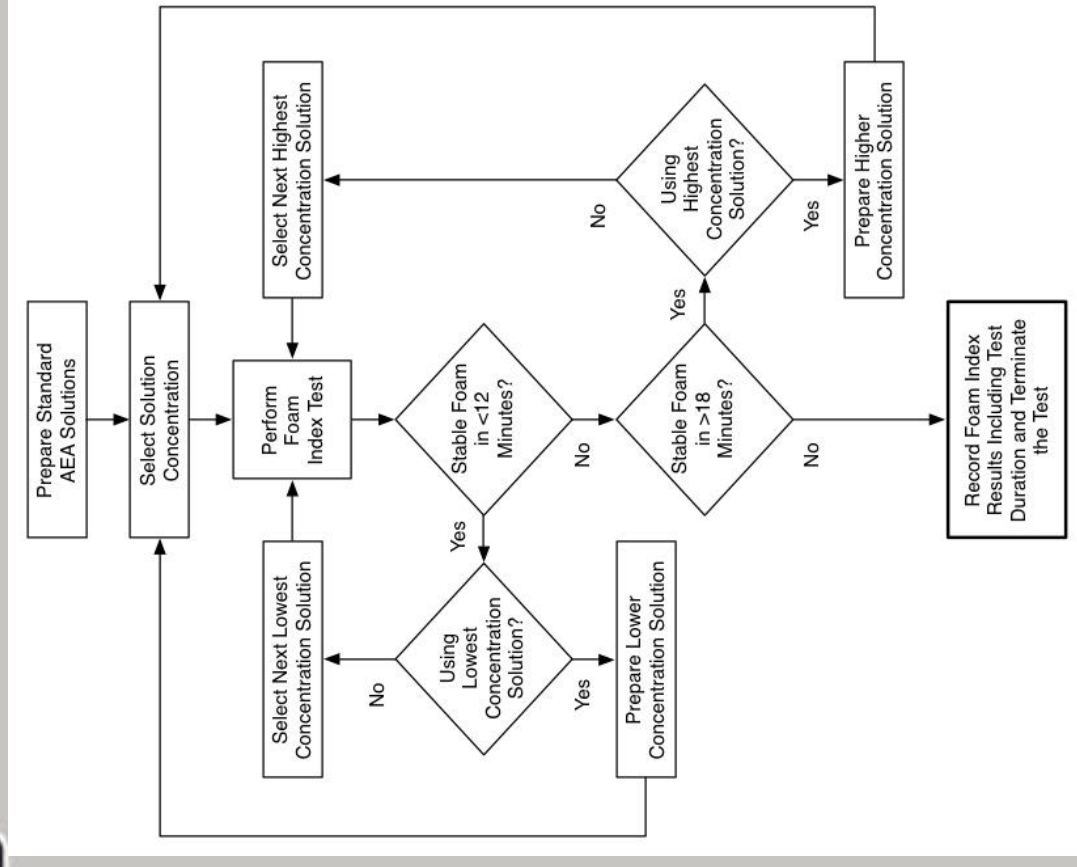
## Foam Index Test

- 2 g ash, 8 g cement
- 25 mL water
- Add AEA solution drop-wise
  - 5 % vol. AEA / Water solution
  - (0.02 mL/drop)
- Shaken, not stirred
- Look for a stable foam
- Repeat...

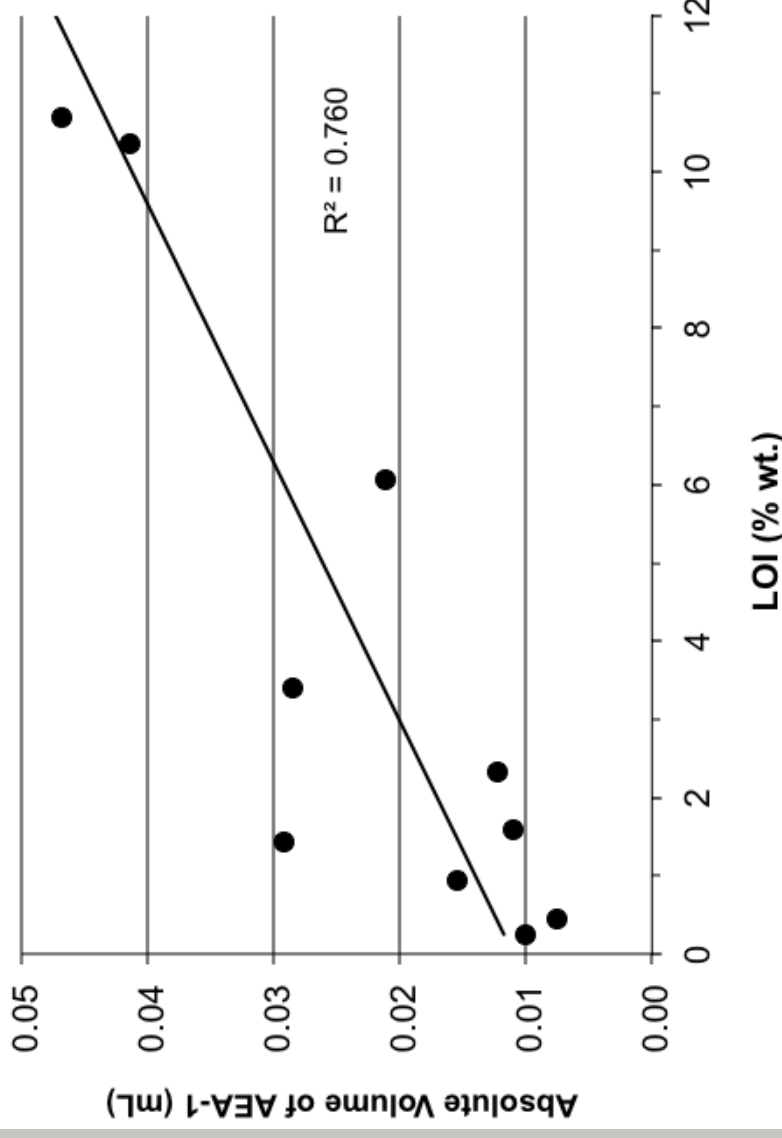


## Foam Index Test

- Vary Solution Strength
  - 2, 6, 10, 15 %vol. AEA
- Achieve uniform contact time
  - 12 to 18 minutes
- Determine total AEA added
  - Foam Index



- Benefits
  - Cheap & Easy
- Issues
  - Not achieving equilibrium
  - Not quantitative
  - Subjective
    - Agitation?
    - What is a stable foam?



# Adsorption Based Tests

- Adsorption characterized by an adsorption isotherm
- Multiple adsorption models and isotherms

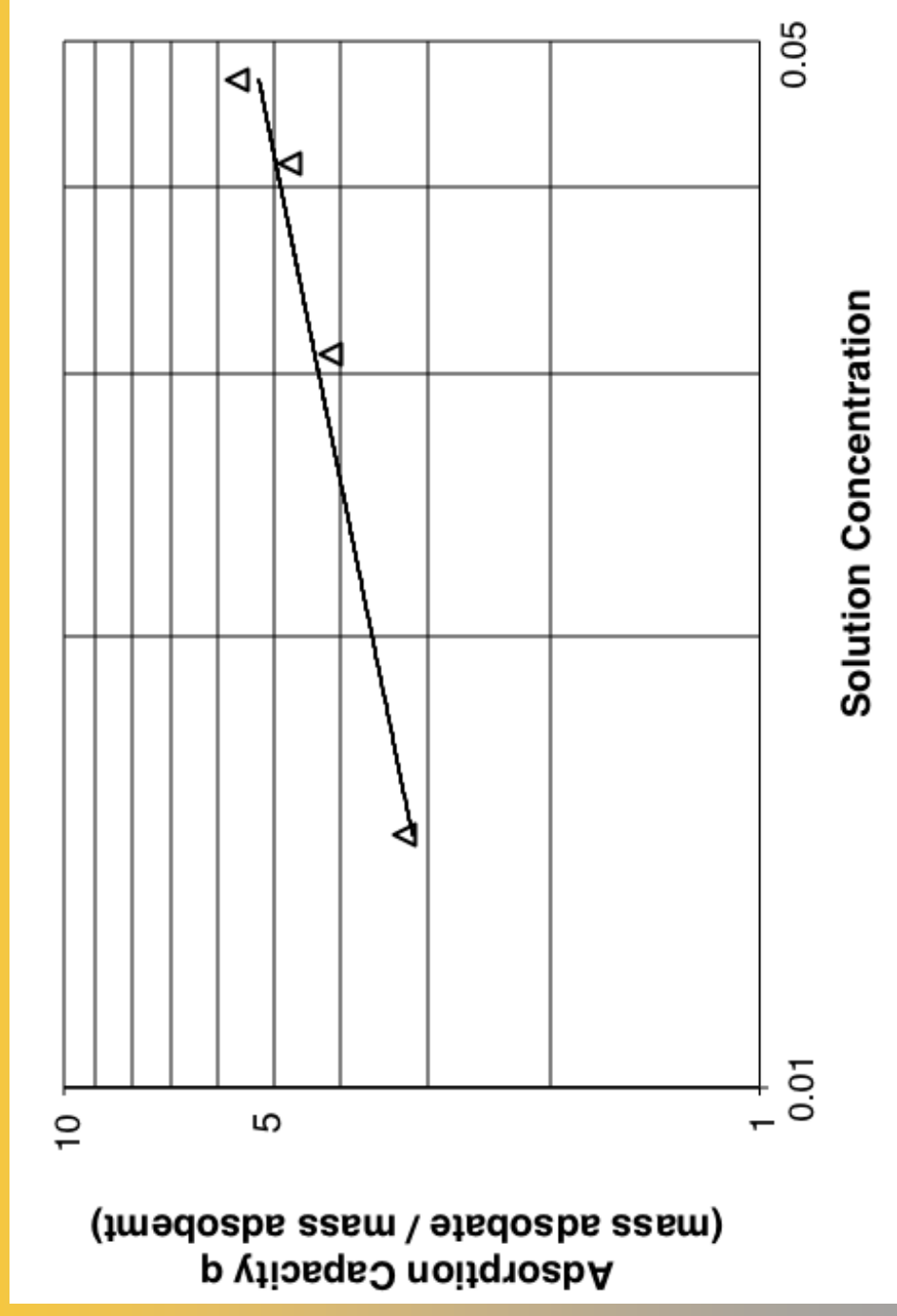
- Freundlich Isotherm

$$q = K \cdot C^{1/n}$$

- $q$  = mass of adsorbate adsorbed per unit mass of adsorbent, mg/g
- $K$  = Freundlich isotherm capacity parameter, (mg/g) (L/mg)<sup>1/n</sup>
- $C$  = Solution concentration, mg/L
- $1/n$  = Freundlich isotherm intensity parameter, dimensionless

# Freundlich Isotherm

Slope =  $1/n$     Intercept =  $\log K$



# Direct Adsorption Isotherm

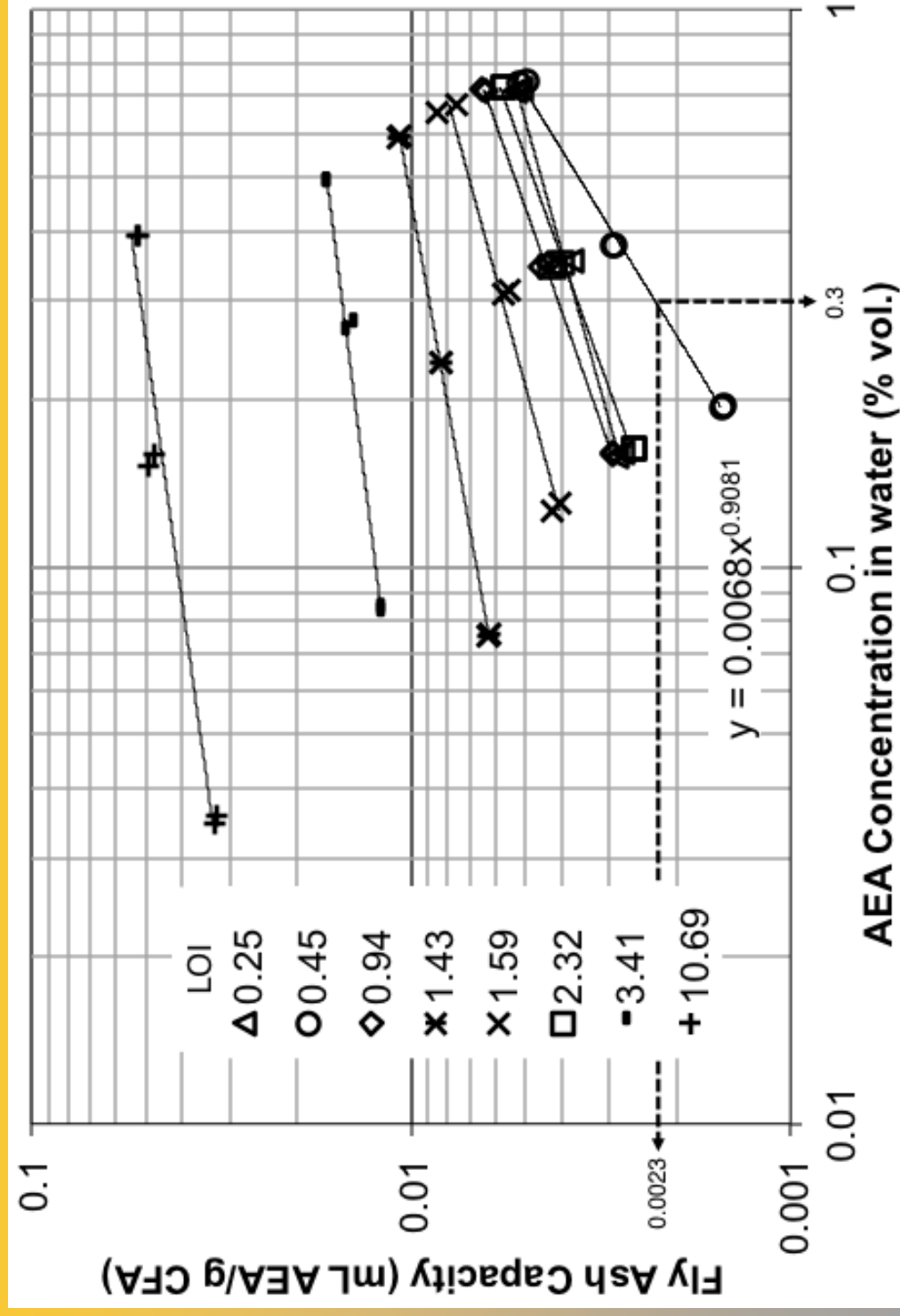


Designation: D3860 – 98 (Reapproved 2008)

## Standard Practice for Determination of Adsorptive Capacity of Activated Carbon by Aqueous Phase Isotherm Technique<sup>1</sup>

- Based on existing ASTM test method with modifications:
  - Modified procedure for determining solution concentration
    - COD test versus spectroscopic methods
  - Needed to account for the contribution of cement

# Direct Adsorption Isotherm determines AEA adsorption “capacity”



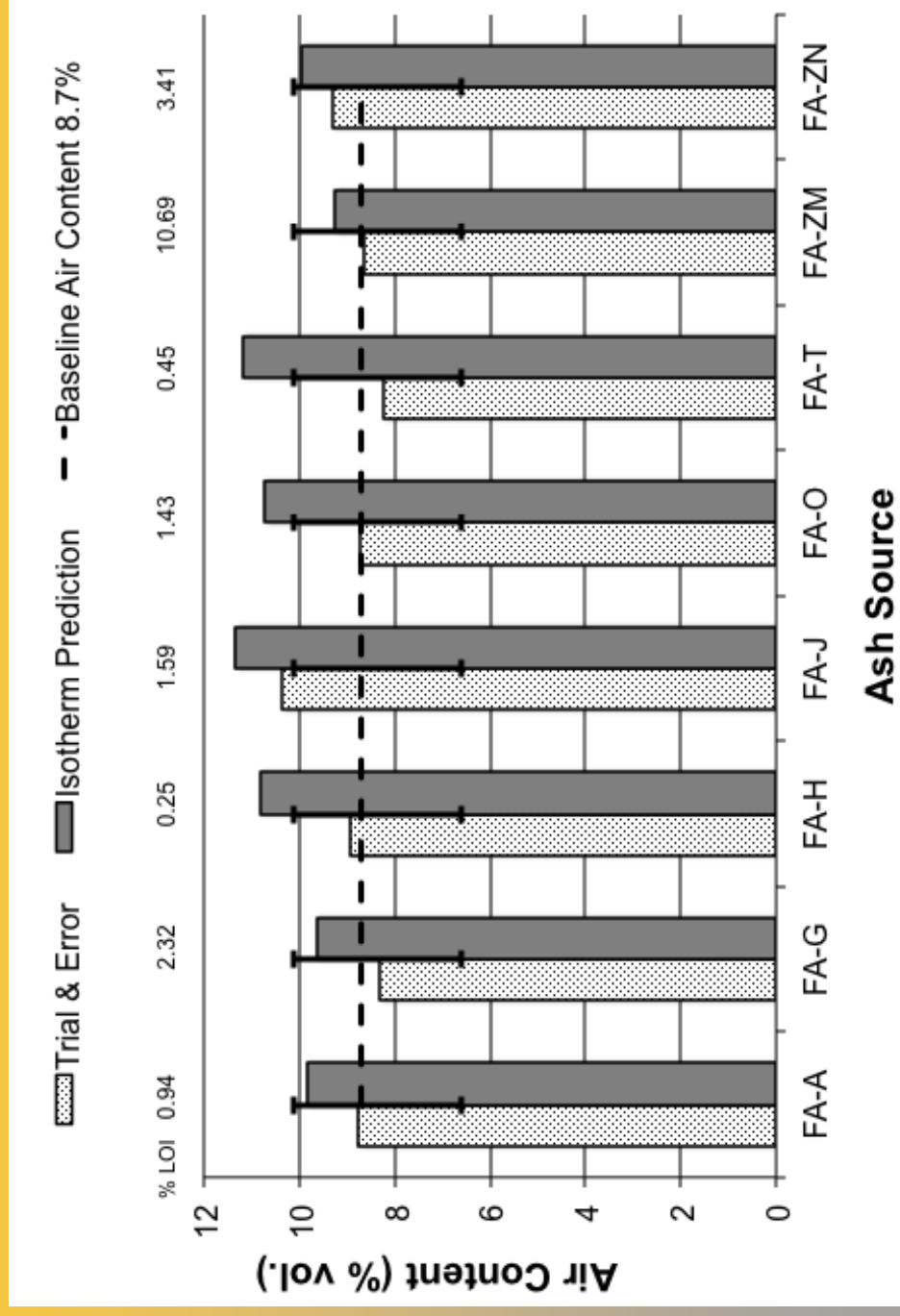
# Direct Adsorption Isotherm

- Measures the adsorption capacity of the ash AND the adsorption capacity of the AEA
- Can be used to estimate AEA dosage
- Simple execution
  - Scales
  - Beakers & Stir Plate & Filtration
  - COD Kits & Colorimeter



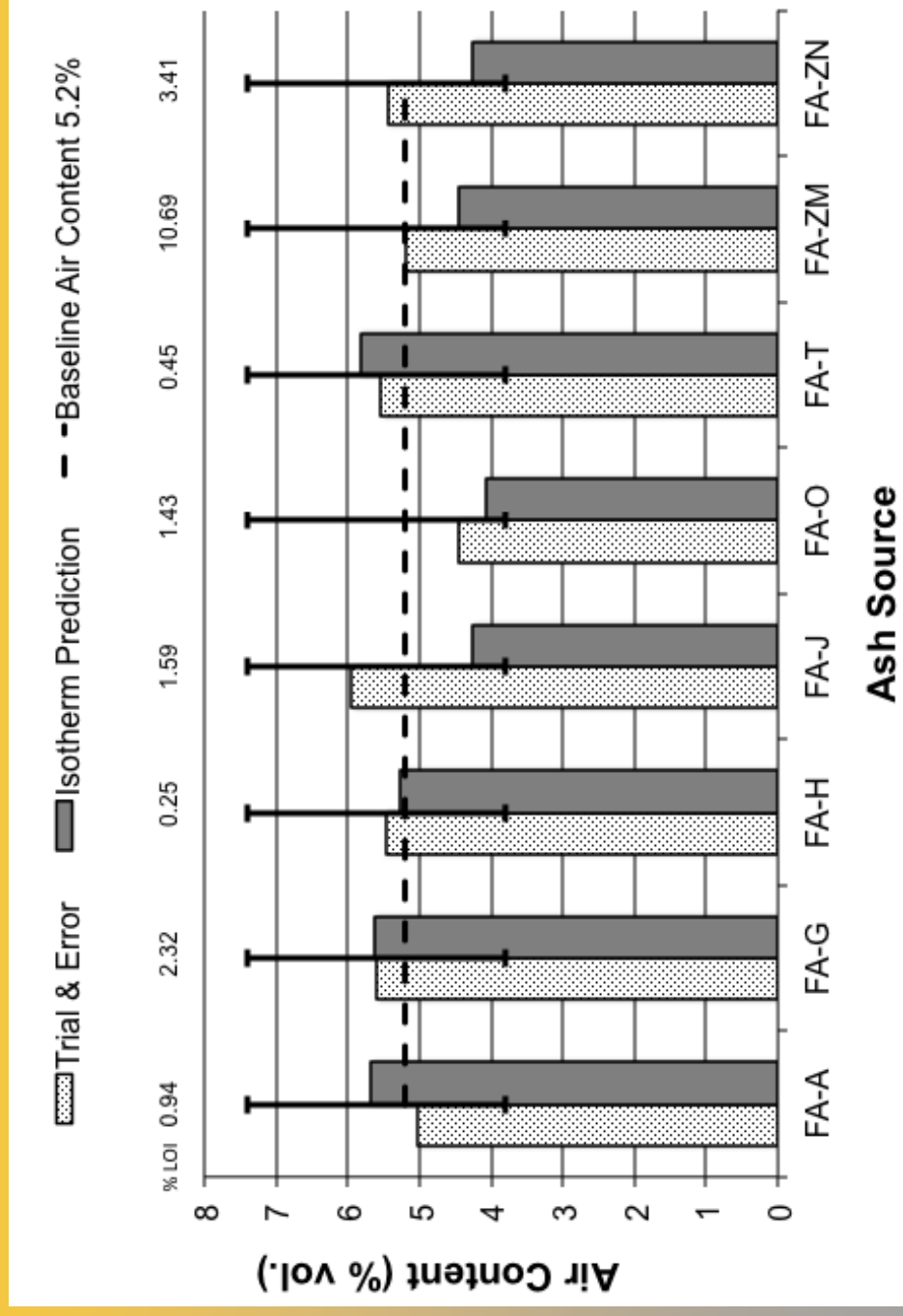
# Direct Adsorption Isotherm

Vinsol resin



# Direct Adsorption Isotherm

Alpha olephin sulfonate



# Coal Fly Ash Iodine Number

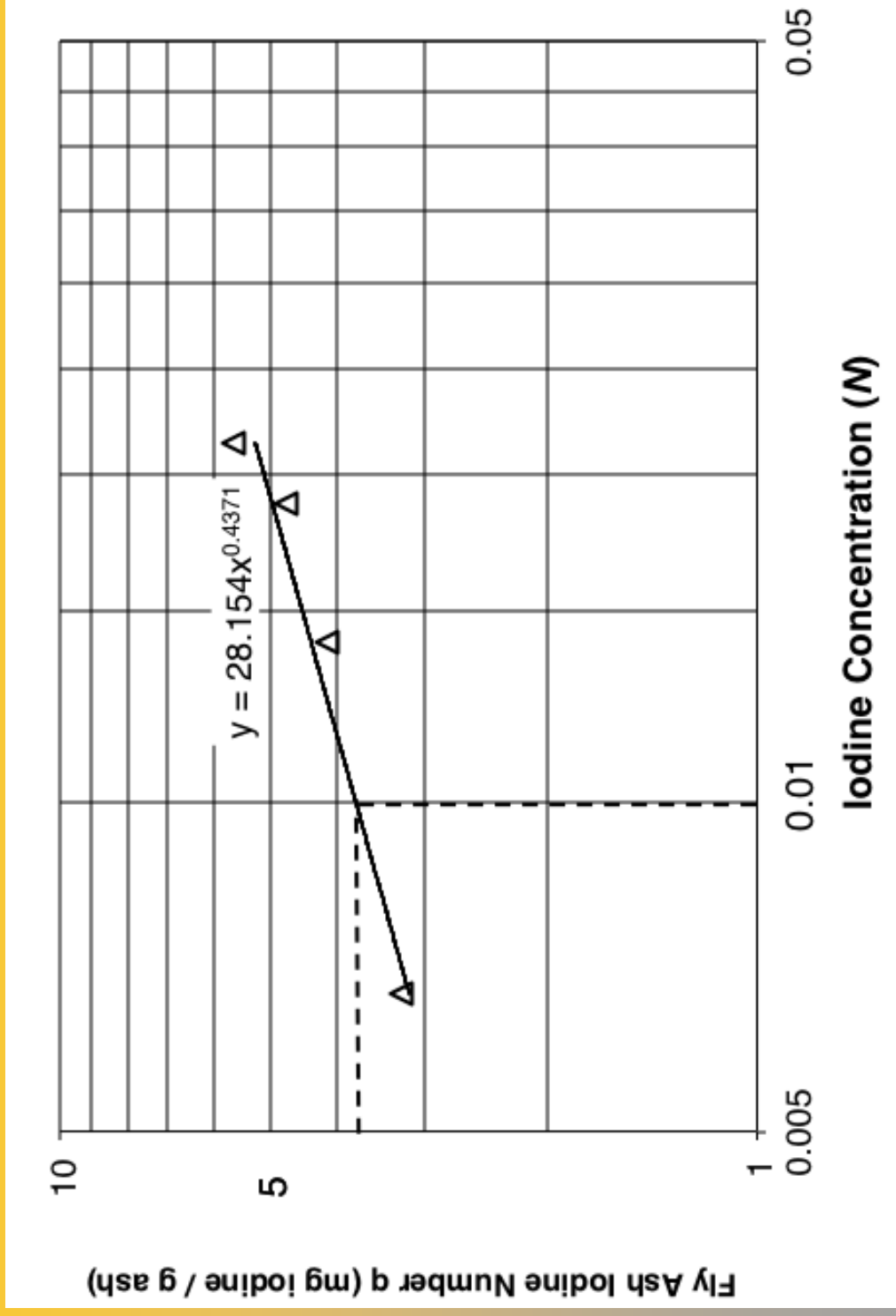


Designation: D4607 – 94 (Reapproved 2006)

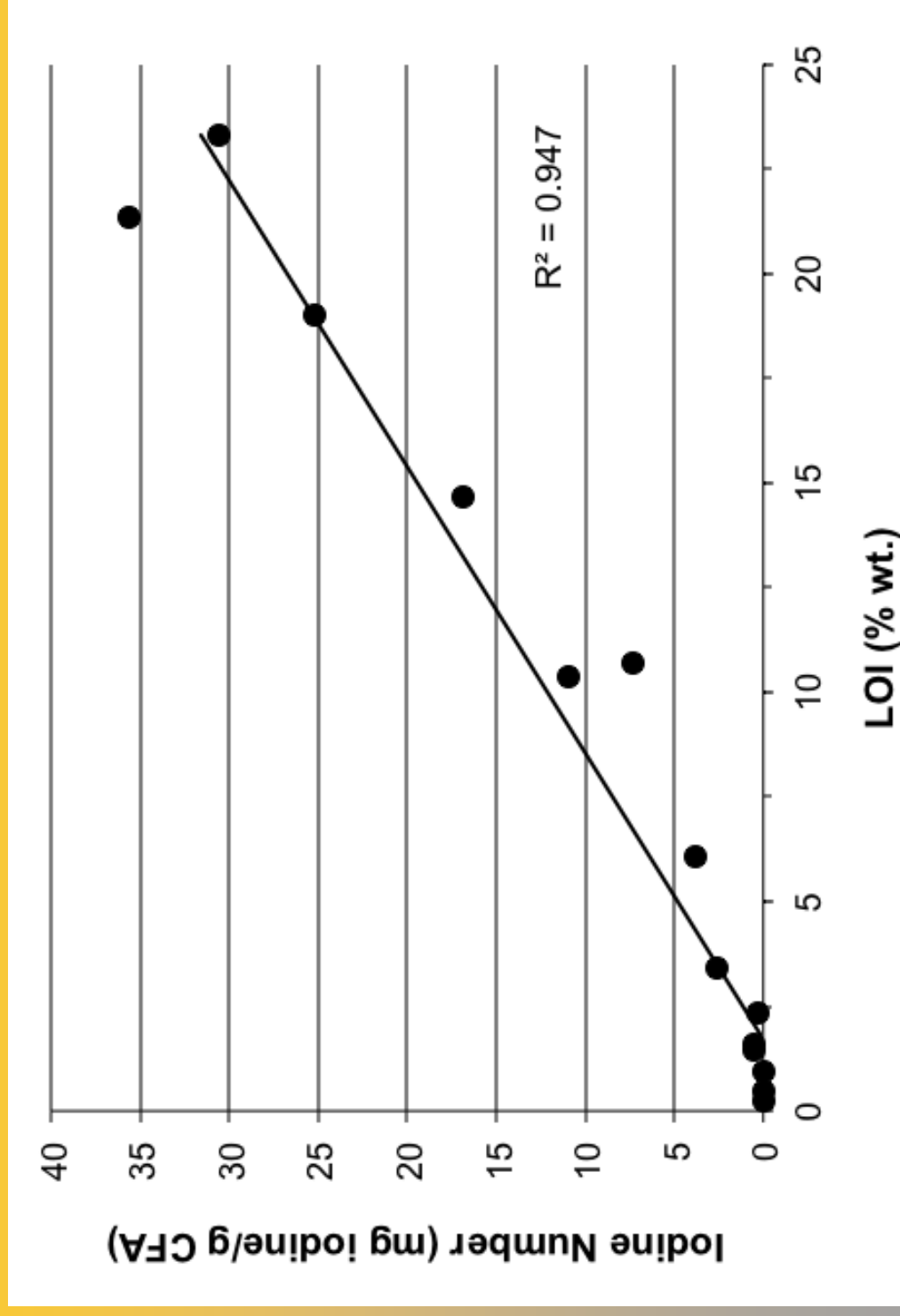
## Standard Test Method for Determination of Iodine Number of Activated Carbon<sup>1</sup>

- Based on existing ASTM test method with modifications:
  - HCl treatment to acidify the ash and remove SO<sub>3</sub>
  - Initial solution strengths modified (0.025 N vs 0.1 N)
  - Target concentration for determining capacity differs from published test method (0.01 N vs 0.02)

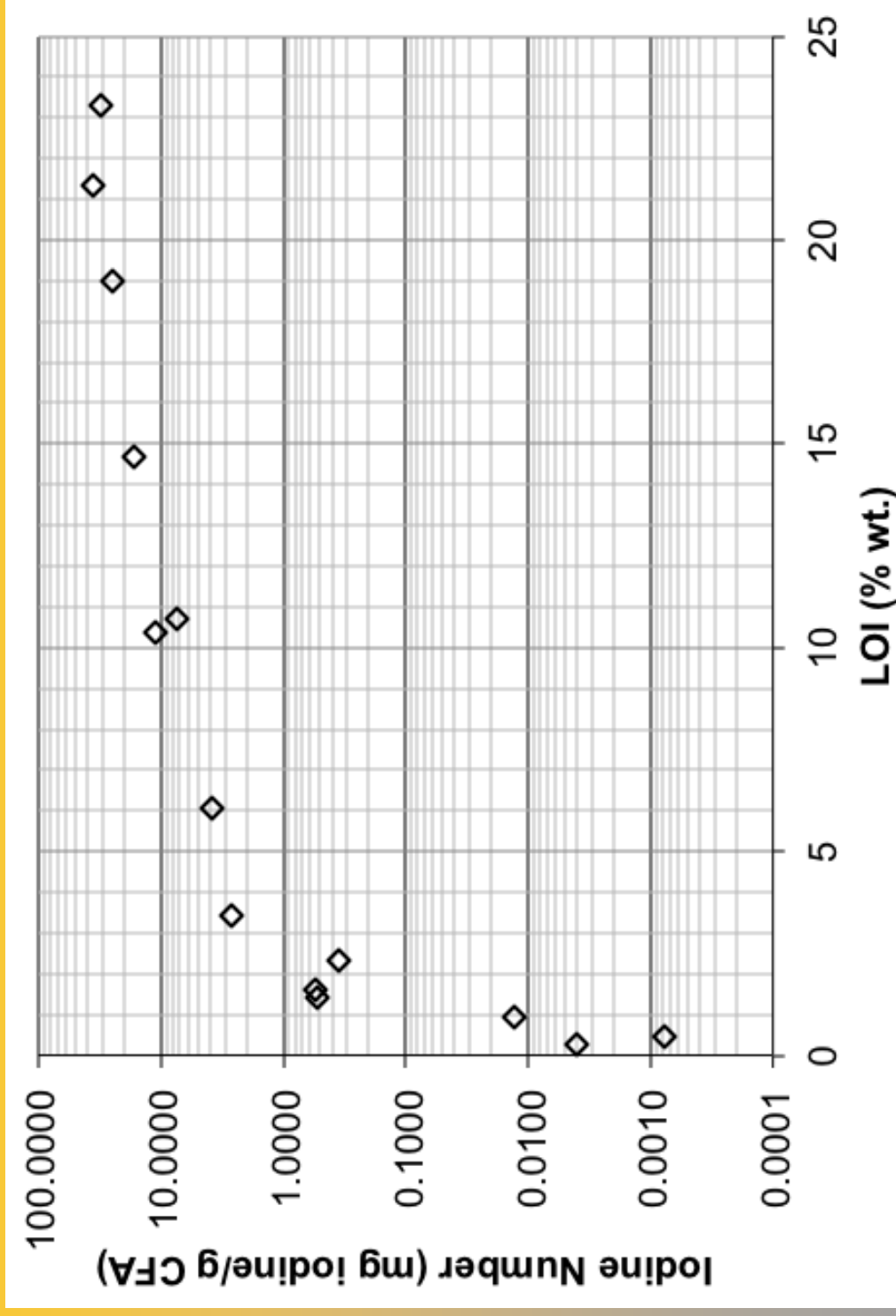
# Coal Fly Ash Iodine Number



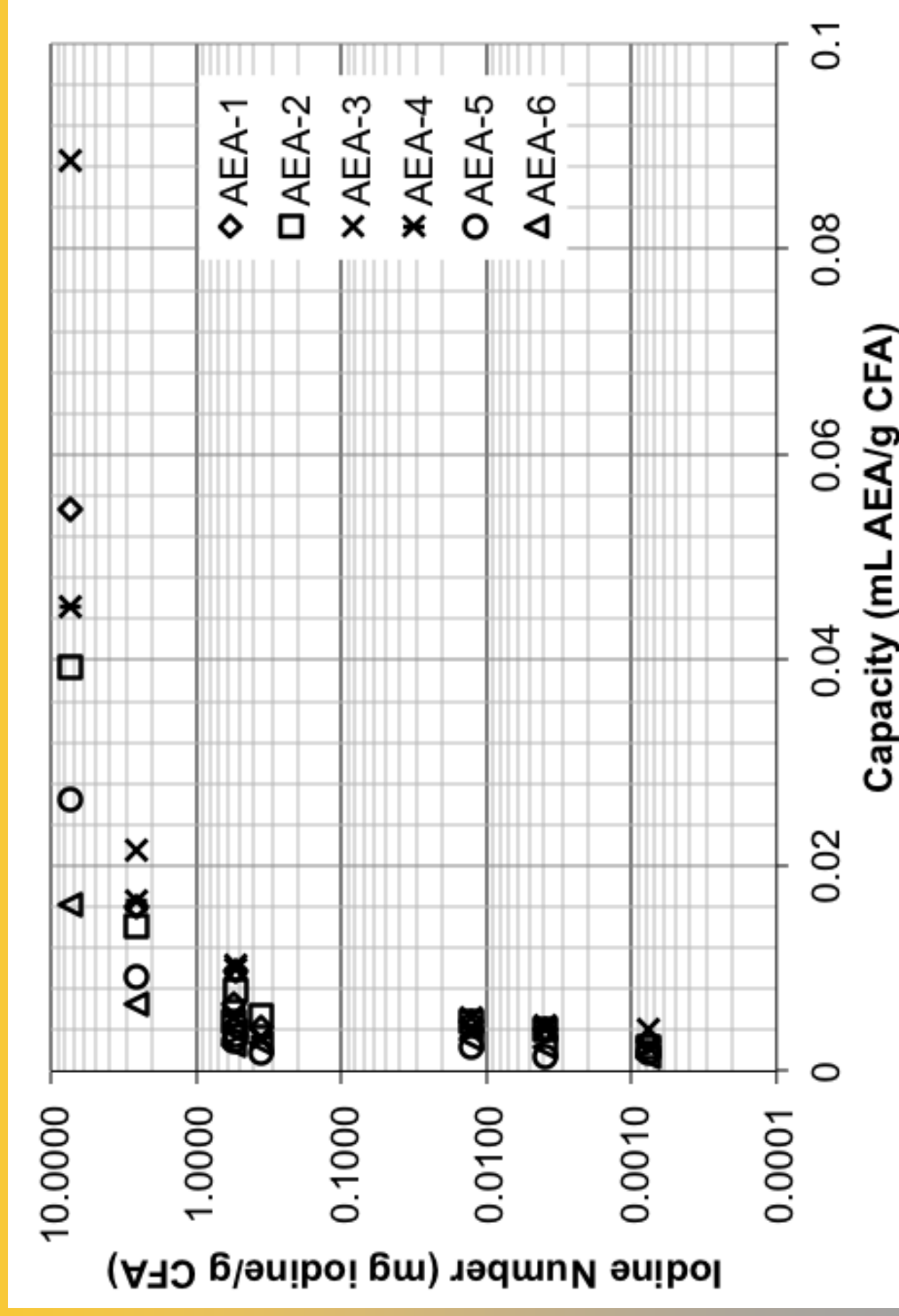
# Coal Fly Ash Iodine Number



# Coal Fly Ash Iodine Number



# Iodine Number vs. Capacity



# Coal Fly Ash Iodine Number

- Measures the adsorption capacity of the ash
- Does not account for the adsorption capacity of the AEA
- Simple execution
  - Scales
  - Beakers & Stir Plate & Filtration
  - Titration



## Carbon Effects on Air Entrainment Study

- *Take Aways*
  - Publish a standardized version of the foam index test that provides a uniform test time and mechanical agitation
  - Specify use of the the coal fly ash iodine number to evaluate ash adsorption potential
  - Specify use of the direct adsorption isotherm test to evaluate fly ash – air entrainer combinations

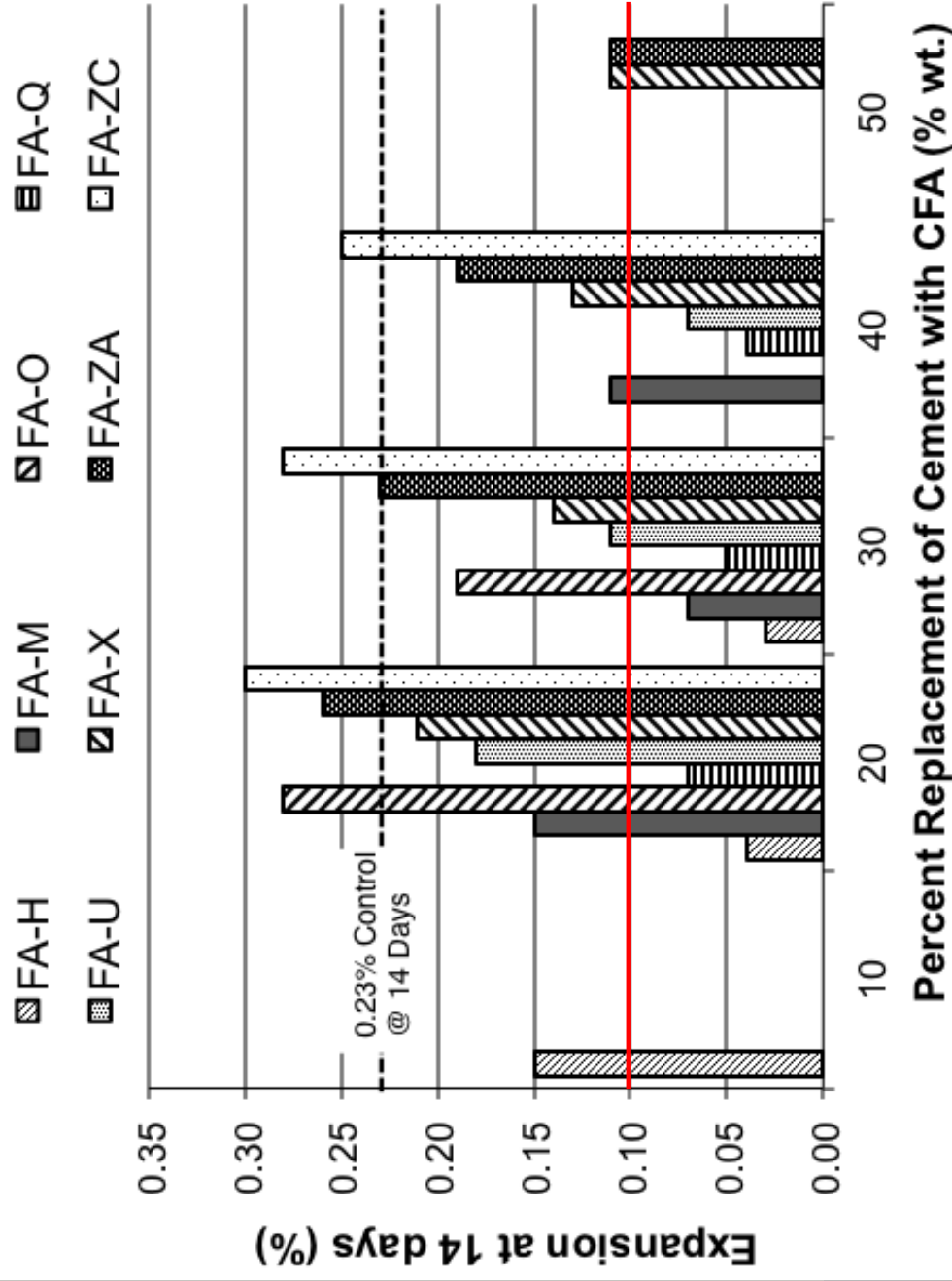
## ASR Mitigation Study

- Evaluate protocols for applying existing test methods (ASTM C1567 and ASTM C1293)
- Evaluate the Alkali Leaching Test (Shehata and Thomas, 2006) and correlate with the results of ASTM C1293 and ASTM C1567
- The alkali leaching test is used to determine the free alkalis available to be leached from a particular combination of cement and fly ash



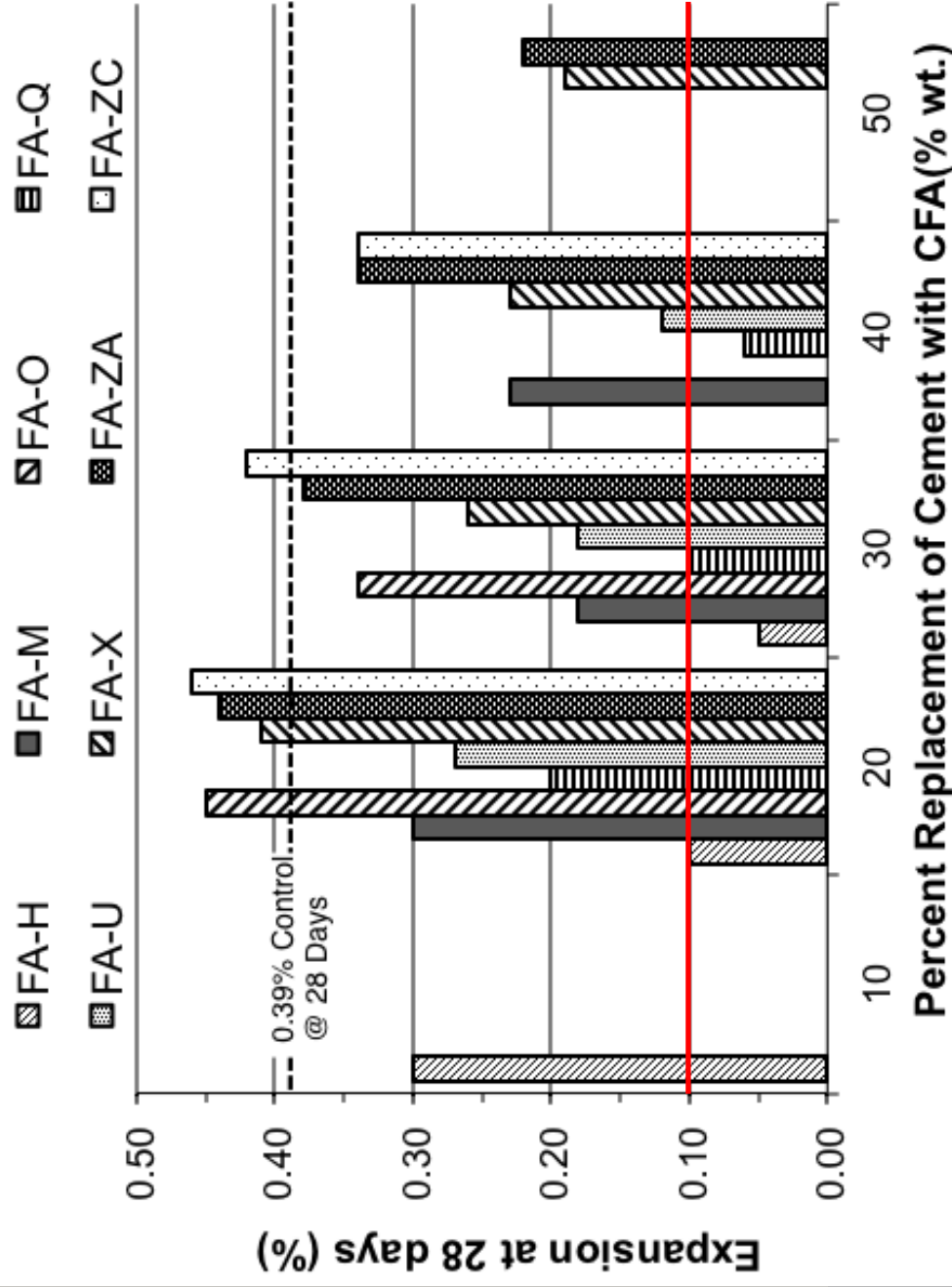
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## ASTM C1567 – 14 days



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## ASTM C1567 – 28 days



## ASR Mitigation Study

- *Take Aways*
  - Confirmed the AASHTO PP-65 limits of 0.1% expansion @ 14 days for ASTM C1567
  - Provided data showing a 28-day limit on ASTM C1567 does not correlate with ASTM C1293
  - Alkali Leaching Test – no clear threshold of alkali release was identified that correlated with a 0.04% ASTM C1293 expansion

## Recommended Changes to AASHTO M 295

- Add a maximum sum of the oxide limit (i.e., 70%) to the Class C classification
- Report CaO, MgO, Na<sub>2</sub>O, and K<sub>2</sub>O
- Adopt the use of the Iodine Number Test and the Direct Adsorption Isotherm Test under Optional Chemical Requirements

## Recommended Changes to AASHTO M 295

- Raise the the Strength Activity Index to 85% of control  
BUT allow the material to be qualified at 7, 28, OR 56  
days
- Delete the available alkali limit
- Delete use of ASTM C441 (Pyrex Glass Test) and adopt  
ASTM C1567 with a 14 day limit of 0.1%



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Questions?

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