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International Forensic Research Institute
School of Integrated Science and Humanity

The State of Data in Glass Analysis

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Outline

1. Introduction to the aims of glass evidence and analysis
2. Glass, the material
3. Application of analytical chemistry tools for glass examinations (~ 500 publications in support)
4. Efforts to standardize the measurement tools
    1. Inter-laboratory exercises
    2. Consensus building
5. Existing Collections/Databases for glass data and samples
6. Reporting results without **overstating or understating** the significance of the evidence

Statisticians are on a mission ......

SIZING UP THE EVIDENCE

Statisticians are on a mission to reverse a legacy of junk science in the courtroom

By Kelly Servick

Science, 11 March **2016**, 1130-1132.

"Forensic scientists have often overstated the strength of evidence from tire tracks, fingerprints, bullet marks and bite marks"

Charlotte PD (Tim French)

1997 Cadillac STS (K11)

S. Montero, A. Hobbs, T. French and J.R. Almirall, “Elemental Profiling of Glass Fragments by ICP-MS as Evidence of Association: Analysis of a Case”, *Journal of Forensic Sciences,* **2003,** 48(5) 1101-1107.

Sources and thickness ranges of the known samples.

Sample	Thickness (in inches)	Vehicle Source
K1	0.1610”- 0.1615”	1999 Chevrolet Tahoe
K2	0.1515”- 0.1520”	2001 Ford Van
K3	0.2231”- 0.2240”	1993 Chevrolet Beauville
K4	0.1510”- 0.1516”	1999 Ford Explorer
K5	0.1628”- 0.1634”	1996 Jeep Cherokee
K6	0.1508”- 0.1518”	1989 Ford Econoline Van
K7	0.1395”- 0.1401”	1998 Jeep Wrangler
K8	0.1604”- 0.1610”	1999 Chevrolet Tahoe
K9	0.1354”- 0.1360”	1998 Ford Ranger
K10	0.1878”- 0.1881”	1988 Oldsmobile Touring Sedan
K11	0.1916”- 0.1926”	1998 Cadillac STS
K12	0.1915”- 0.1924”	1997 Cadillac STS
K13	0.1526”- 0.1530”	1993 GMC Sierra
K14	0.1279”- 0.1285”	1994 Ford Ranger
K15	0.1628”- 0.1640”	2000 Dodge Dakota

S. Montero, A. Hobbs, T. French and J.R. Almirall, “Elemental Profiling of Glass Fragments by ICP-MS as Evidence of Association: Analysis of a Case”, *Journal of Forensic Sciences,* **2003,** 48(5) 1101-1107.

Suspect Overalls back and front

S. Montero, A. Hobbs, T. French and J.R. Almirall, “Elemental Profiling of Glass Fragments by ICP-MS as Evidence of Association: Analysis of a Case”, *Journal of Forensic Sciences,* **2003,** 48(5) 1101-1107.

Likelihood Ratio (LR) Estimation (Source and Activity)

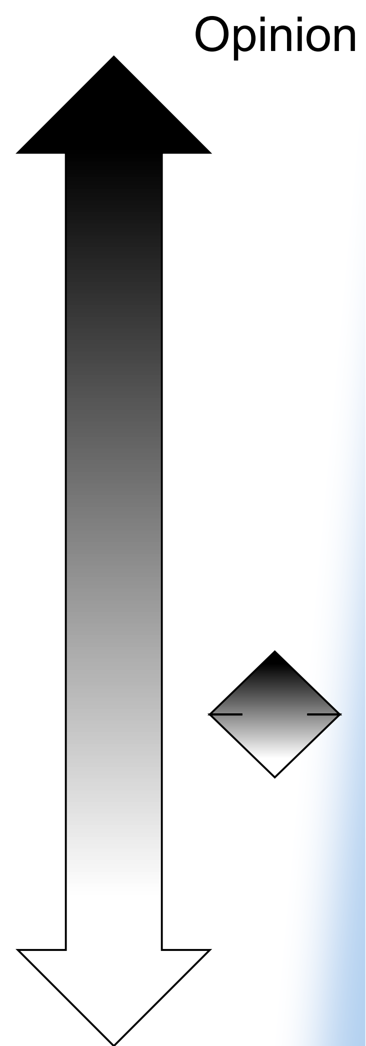
Association scale:	Equivalent LR
Association: Identification	∞
Association: Very Strong Evidence	1,000 – 10,000
Association: Strong Evidence	10 – 100
Association: Some evidence	1 – 10
Inconclusive (no support for either proposition)	1
Evidence of no association	0.1
Strong evidence of no association	0.001
Very strong evidence of no association	0.000001
Elimination:	0

J. Almirall and T. Trejos, Analysis of Glass Evidence (Chapter 6) *in Forensic Chemistry: Fundamentals and Applications, J. Siegel, Ed.* ***2015****. Wiley and Sons (in press)*

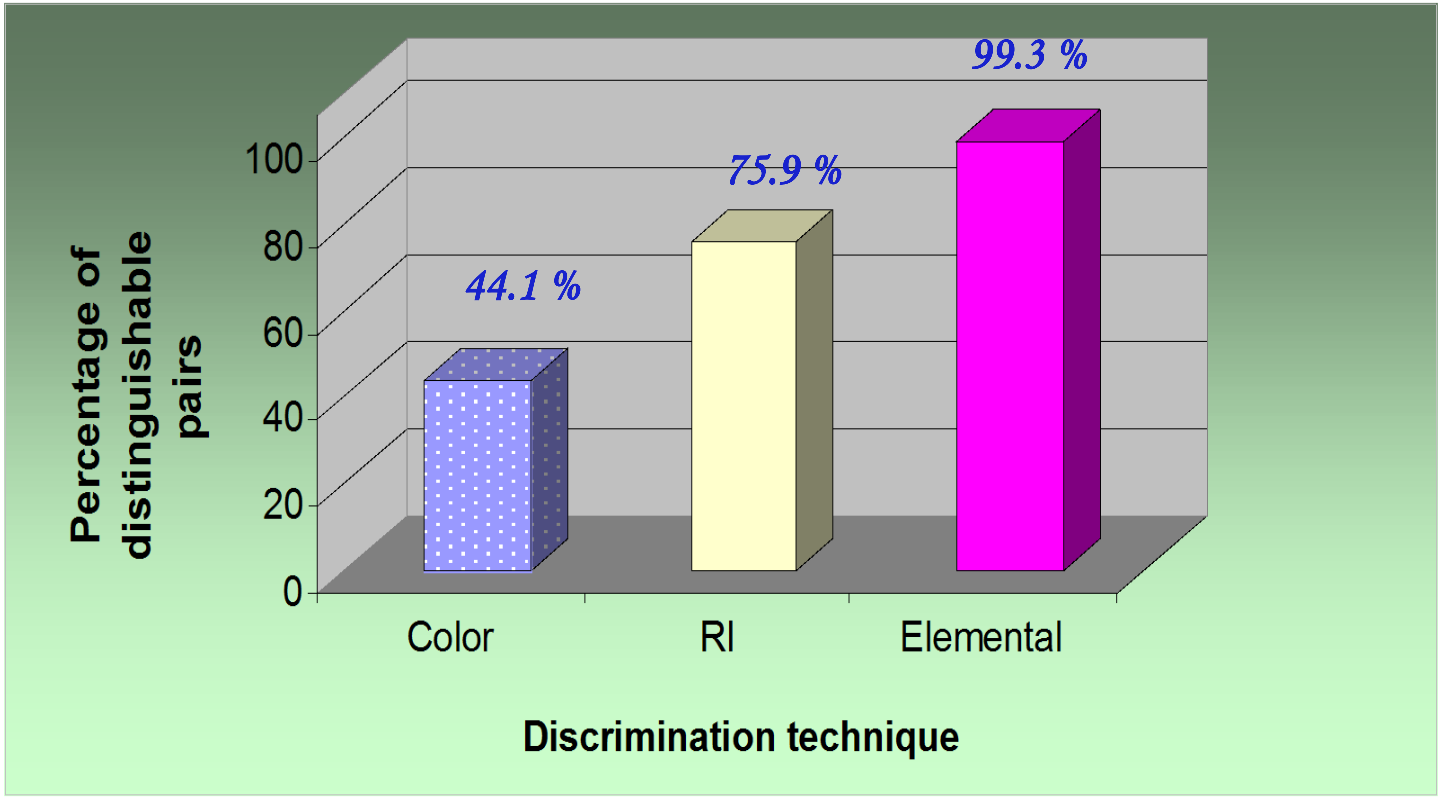
Case Scenario # 1

A suspect is apprehended close to the scene, and **no glass** is found. **(related to activity level)**

Association scale:	Equivalent LR
Association: Identification	**∞**
Association: Very Strong Evidence	**1,000 – 10,000**
Association: Strong Evidence	**10 – 100**
Association: Some evidence	**1 – 10**
Inconclusive (no support for either proposition)	**1**
Evidence of no association	**0.1**
Strong evidence of no association	**0.001**
Very strong evidence of no association	**0.000001**
Elimination:	**0**



Results of the pairwise comparisons for all 17 <u>known fragments</u> compared to each other for a possible 136 total comparisons.

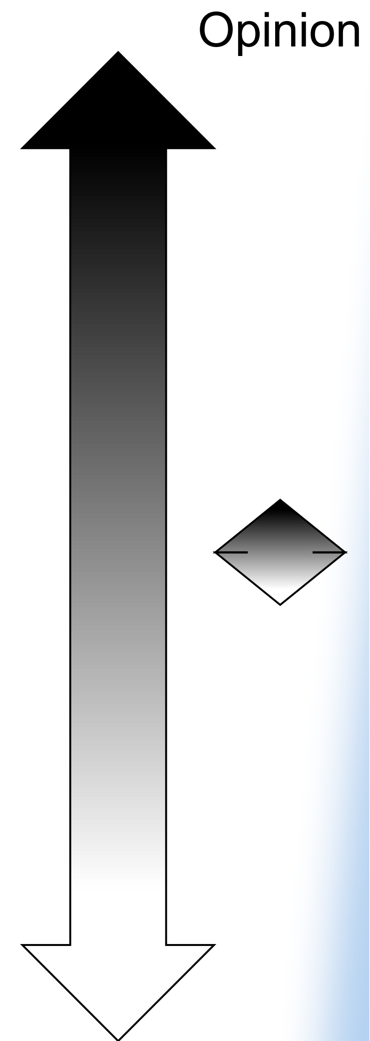


S. Montero, A. Hobbs, T. French and J.R. Almirall, "Elemental Profiling of Glass Fragments by ICP-MS as Evidence of Association: Analysis of a Case", *Journal of Forensic Sciences,* **2003,** 48(5) 1101-1107.

Case Scenario # 2

A suspect is apprehended close to the scene, and **only one glass fragment** is found and it is found indistinguishable by RI to one of the Known sources.
(related to activity level and to source level)

Association scale:	Equivalent LR
Association: Identification	**∞**
Association: Very Strong Evidence	**1,000 – 10,000**
Association: Strong Evidence	**10 – 100**
Association: Some evidence	**1 – 10**
Inconclusive (no support for either proposition)	**1**
Evidence of no association	**0.1**
Strong evidence of no association	**0.001**
Very strong evidence of no association	**0.000001**
Elimination:	**0**



Summary results of the comparisons by elemental composition (ICP-MS) and by classical methods (density, thickness, color and refractive index with Emmons double variation).

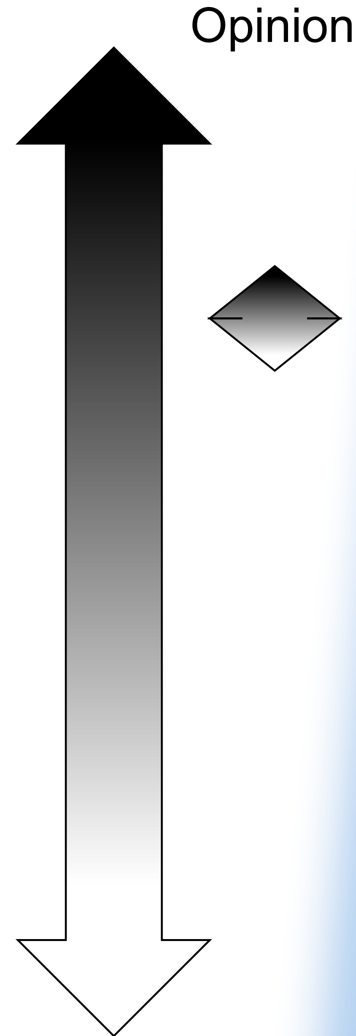
Samples associated by elemental composition	Pairs associated by classical methods
K2 and Qb	K2 and Qb
K5 and Qn	K5 and Qn
K7 and Qi	K7 and Qi
K15 and Qa	K15 and Qa
K4 and K8c	K4 and Qd
K2 and Qc	K6 and Qo
K7 and Qh	K7 and Qo
K7 and Qm	K13 and Qc
Qa and Qk	
Qb and Qc	
Qh and Qi and Qm	
Qd and Qg and Qj	

S. Montero, A. Hobbs, T. French and J.R. Almirall, "Elemental Profiling of Glass Fragments by ICP-MS as Evidence of Association: Analysis of a Case", *Journal of Forensic Sciences,* **2003,** 48(5) 1101-1107.

Case Scenario # 3

A suspect is apprehended close to the scene, and **>30 glass fragments** were found and 25 of the 30 fragments were indistinguishable by elemental composition to four (4) different K sources. **(related to activity level and to source level)**

Association scale:	Equivalent LR
Association: Identification	**∞**
Association: Very Strong Evidence	**1,000 – 10,000**
Association: Strong Evidence	**10 – 100**
Association: Some evidence	**1 – 10**
Inconclusive (no support for either proposition)	**1**
Evidence of no association	**0.1**
Strong evidence of no association	**0.001**
Very strong evidence of no association	**0.000001**
Elimination:	**0**

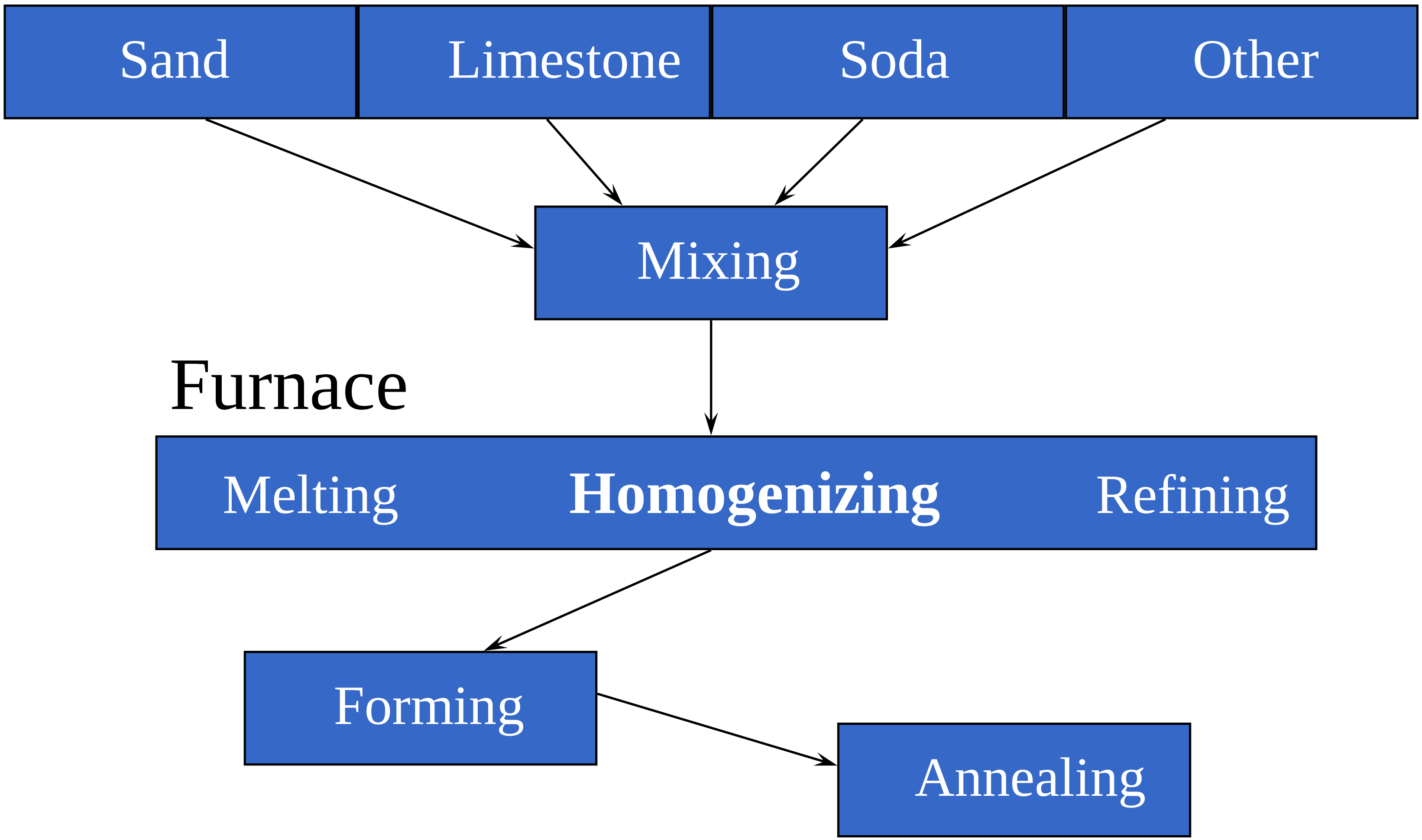


How homogeneous is glass? It depends….

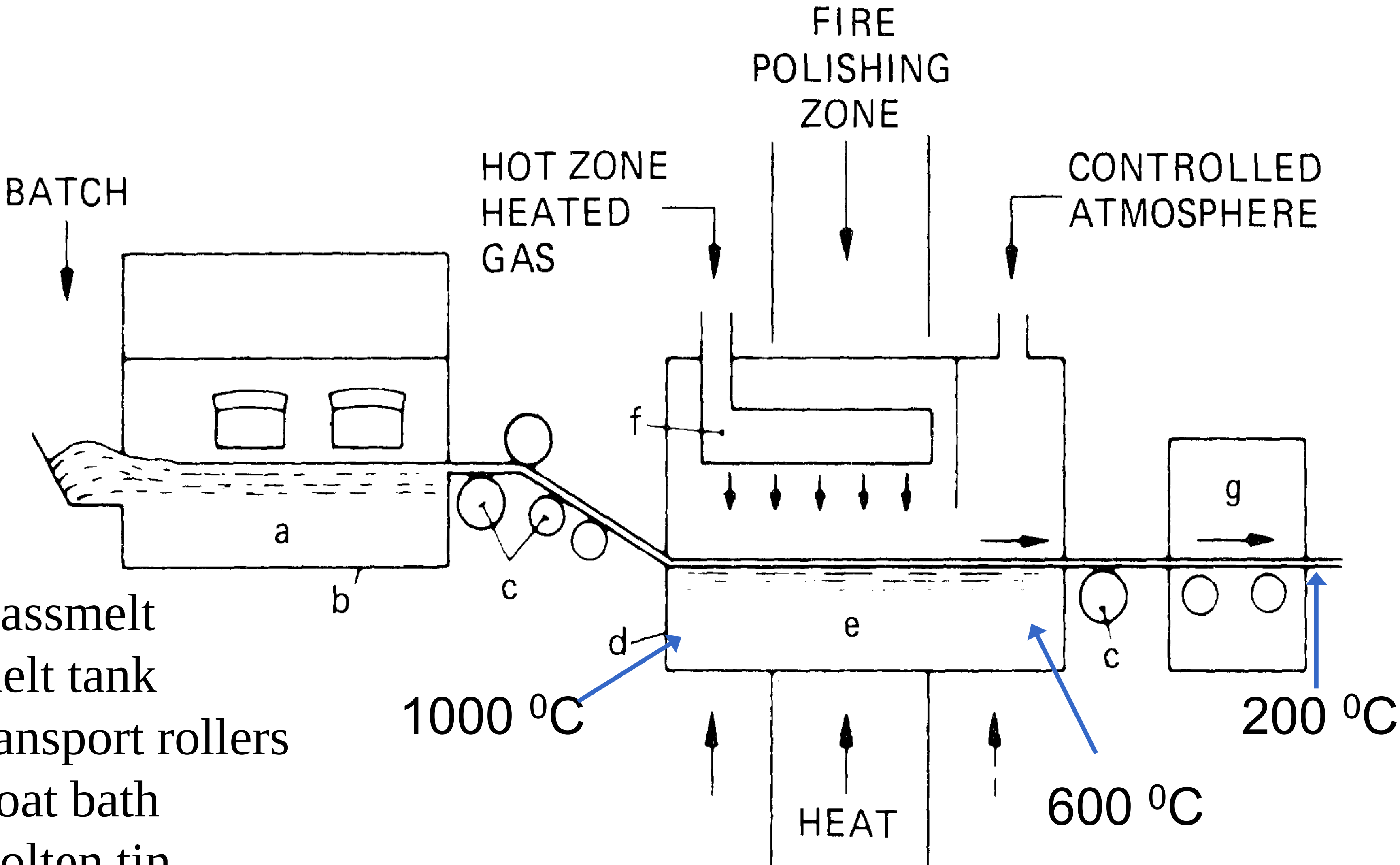
2-foot thick 1000 lb pieces of optical glass created at NBS.

www.nist.gov/digitalarchives

Glass Manufacture



“Float glass” manufacturing process



a - glassmelt
b - melt tank
c - transport rollers
d - float bath
e - molten tin

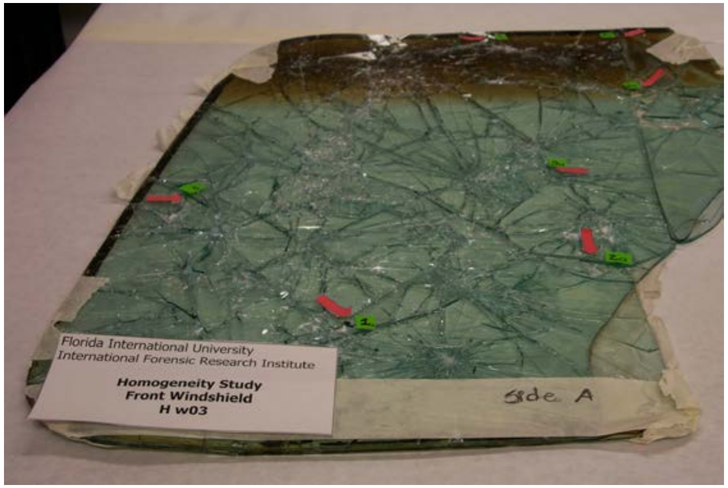
How different is the glass composition based on source?

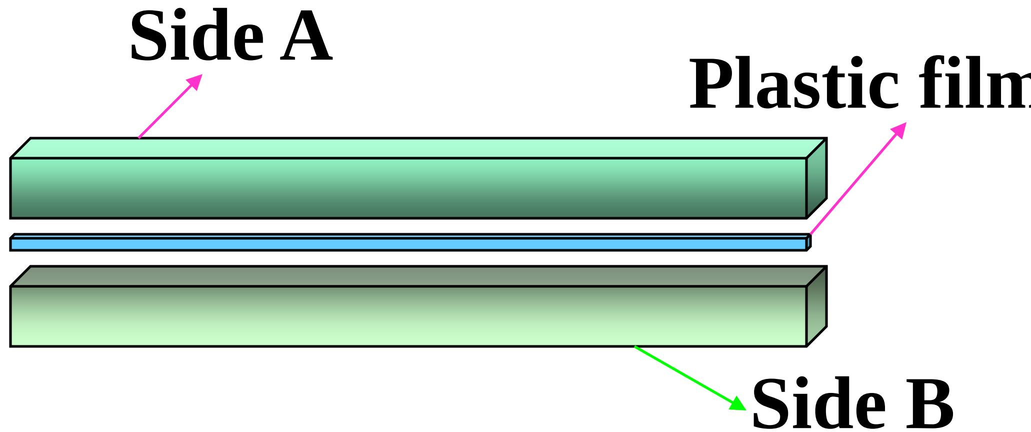
Sources of variation in analytical data from glass

1. Measurement (quality of measurement)
   - precision
   - accuracy
   - uncertainty
   - limits of detection
2. Within-sample (same "source" --- sample heterogeneity)
   - manufacturing protocols
   - nature of sample
3. Between-sample (Differences between "sources")
   - diversity of sources for raw materials
   - diversity of manufacturing sources and formulas
   - quantity of materials manufactured with the same composition
   - temporal variation in composition of materials

and others: ie. Inappropriate sampling, insufficient # of measurements, etc.

Windshields





28 samples from each windshield:

✓14 samples A
(7 replicates and 7 dif. Fragments)

✓14 samples B
(7 replicates and 7 dif. Fragments)

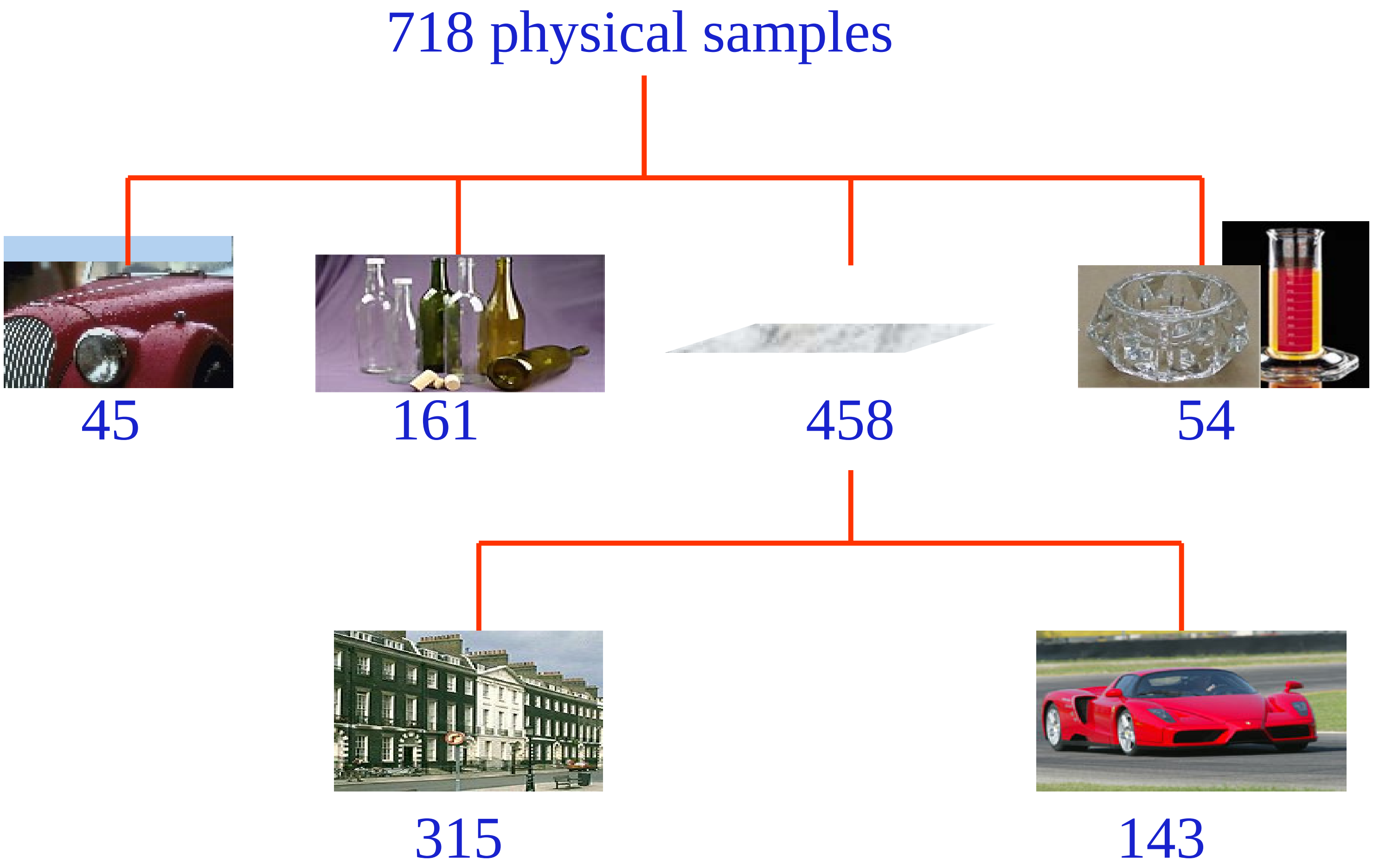
Trejos T, Almirall JR. Sampling strategies for the analysis of glass fragments by LA-ICPMS. Part I. Microhomogeneity study of glass and its application to the interpretation of forensic evidence,, Tatanta, **2005:** 67, 388-395.
Trejos T, Almirall JR. Sampling strategies for the analysis of glass fragments by LA-ICPMS Part II. Sample size and shape considerations, Tatanta, **2005**:67, 396-401.

Float Glass (Architectural): Manufacturers Survey

- A single plant (Cardinal FG, Portage, Wisconsin) sampled from 1997 to 2001 for a total of 190 samples of which 97 were sampled during 24 hours

- 36 float glass plants across the U.S. sampled from 94-96 for total of 125 samples

FIU Glass Database (ICP-MS data)



Discrimination of glass comparisons using LA-ICP-MS

Glass Subset	CFS *1	Headlamp *1	Container*1	Automobile*2
# of samples	46	45	45	41
# comparison pairs	**1035**	**990**	**990**	**820**
Discrimination power (LA-ICP-MS)	**99.7%**	**100%**	**100%**	**99%**
% false inclusions	0.3%	0%	0%	1.0%*

 # comparison pairs
n(n-1)/2

% DISC = 100* (1- IP/CP)

[1] *Trejos T., Montero S. and Almirall J.R., J. of Analyt. and Bioanalyt. Chem.,* ***2003****, 376, 8: 1255-1264.*
[2] *Naes B., Umpierrez S., Ryland S., Barnett C. and Almirall J.R., Spectrochimica Acta. B.,* ***2008****, 63 ,1145-1150.*

Discrimination/Association studies

Authors	Year	Technique	Samples (*n*)	Discrimination (%)
Suzuki, Y.; Sugita, R.; Suzuki, S.; Marumo, Y.	2000	RI & ICP-MS	16	**100** (120/120)
Schenk, E. R.; Almirall, A. R.	2012	LA-ICP-OES	41	**99.5** (816/820)
Bridge, C. M.; Powell, J.; Steele, K. L.; Sigman, M. E.	2007	RI	91	**66.1** (753/1122)
		RI & LIBS	91	**87.2** (978/1122)
		RI & LA-ICP-MS	64	**98.8** (658/666)
Naes, B. E.; Umpierrez, S.; Ryland, S.; Barnett, C.; Almirall, J. R.	2008	LA-ICP-MS	41	**99.4** (815/820)
		uXRF	41	**99.0** (812/820)
		LIBS	41	**98.9** (811/820)
Weis, P.; Ducking, M.; Watzke, P.; Menges, S.; Becker, S.	2011	LA-ICP-MS	62	**99.9** (1889/1891)
Koons, R. D.; Peters, C. A.; Rebbert, P. S.	1991	RI (n_C, n_D, n_F)	81	**94.5** (3062/3240)
		RI & ICP-AES	81	**99.9** (3238/3240)
Duckworth, D. C.; Morton, S. J.; Bayne, C. K.; Koons, R. D.; Montero, S.; Almirall, J. R.	2002	RI & ICP-MS	76	**99.1** (2532/2556)
Becker, S.; Gunaratnam, L.; Hicks, L.; Stoecklein, W.; Warman, G.	2001	RI, SEM-EDS, XRF, ICP-MS	6	**100** (12/12)
Trejos, T.; Montero, S.; Almirall, J.	2003	LA-ICP-MS	46	**97.9** (1028/1035)
Stoecklein, W., Kubassek, E., Fischer, R., Chadzelek, A.	2009	ICP-MS	60 (30 plants)	**100** (1830/1830)
Trejos, T.; Montero, S.; Almirall, J.	2003	LA-ICP-MS	45	**99.3** (969/990)

Selected Analytical Chemistry studies

Author	Year	Technique	Samples	Conclusions
Alexander, M. L.; Smith, M. R.; Hartman, J. S.; Mendoza, A.; Koppenaal, D. W.	1998	LA-ICP-MS	5	UV laser leads to smaller particle sive, better accuracy, and better precision
Duckworth, D. C.; Bayne, C. K.; Morton, S. J.; Almirall, J.	1999	Solution ICP-MS	1	Study to identify source and magnitude of variation using ANOVA; potentially discriminating elements (46 total) were selected based on precision (RSD < 10%) and accuracy (bias < 10%
Horn, I.; Gunther, D.	2003	LA-ICP-MS	3	Study on influence of carrier gas (Ar, He, N) on particle size and transport efficiency using 193 and 266 nm laser; He leads to smaller particle size though this is more pronounced for 193nm
Latkoczy, C.; Becker, S.; Ducking, M.; …. Almirall J.	2005	LA-ICP-MS	9	NITE-CRIME: matrix-matched standards are required to produce comparable results (i.e. precision and accuracy) between laboratories; FGS1 and FGS2 suitable as calibration standards
Trejos, T.; Almirall, J.	2003	LA-ICP-MS	3	Effect of fractionation: minimal fractionation for all elements except U and Th (266nm laser); however, fractionation of U and Th did not affect precision
Umpierrez, S.; Trejos, T.; Neubauer, K.; Almirall, J.	2006	DRC LA-ICP-MS	1	DRC (methane) LA-ICP-MS to resolve Fe; LOD 2 orders of magnitude lower when compared to LA-ICP-MS (no reaction gas) and 1 order of magnitude lower when compared to solution ICP-MS
Berends-Montero, S.; Wiarda, W.; de Joode, P.; van der Peijl, G.	2006	LA-ICP-MS	124	Method validation: LOD ≤3 ppm (except Ti and K), %RSD and % bias <10 (except Ti and K for NIST 1831); 10 elements used in total
Trejos, T.; Almirall, J.	2005	LA-ICP-MS	104	Micro-homogeneity study: architectural glass is homogeneous within window pane; for automotive windshields, there may be elemental differences between the two sides of glass separated by a plastic film; container glass was also found to have inherent heterogeneity. Thus, the heterogeneity of the K must be well characterized for case work
Trejos, T.; Almirall, J.	2005	LA-ICP-MS	2	Effect of sample size and shape on elemental composition: using ANOVA followed by Tukey's HSD, no significant difference was found on elemental concentration for samples of various sizes and shapes; this cannot be generalized for fragments smaller than 0.1x0.2 mm

Round Robin 2

	Type 1 Error Rate (%) False Exclusion		Type 2 Error Rate (%) False Inclusion		
	RR2	RR4	RR2	RR3	RR4
Comparison Interval ±4* standard deviation (minimum 3% RSD)	**0** (0/19)		**0** (0/19)		
T-Test (Welch's Modification) 95% confidence, Bonferroni correction	**52.6** (10/19)		**0** (0/19)		
T-Test (assuming equal variance) 95% confidence, Bonferroni correction	**36.8** (7/19)		**0** (0/19)		
Equivalence Test θ calculated with known	**78.9** (15/19)		**0** (0/19)		
Equivalence Test θ calculated with Cardinal glass	**36.8** (7/19)		**0** (0/19)		
Equivalence Test θ calculated with FIU Database	**36.8** (7/19)		**0** (0/19)		
Hotelling's T^2 Assuming equal covariance	**25.0** (1/4)		**0** (0/6)		
Hotelling's T^2 No assumptions	**50.0** (3/6)		**0** (0/6)		

K1 and **Q1** from the same source

Q2 manufactured 2 years and 8 months before (same manufacturing plant)

Round Robin 3

	Type 1 Error Rate (%) False Exclusion		Type 2 Error Rate (%) False Inclusion		
	RR2	RR4	RR2	RR3	RR4
Comparison Interval ±4 * standard deviation (minimum 3% RSD)				**11.9** (15/126)	
T-Test (Welch's Modification) 95% confidence, Bonferroni correction				**4.0** (5/126)	
T-Test (equal variance) 95% confidence, Bonferroni correction				**4.0** (5/126)	
Equivalence Test θ calculated with known				**0.8** (1/126)	
Equivalence Test θ calculated with Cardinal glass				**14.3** (18/126)	
Equivalence Test θ calculated with FIU Database				**25.4** (32/126)	
Hotelling's T^2 Assuming equal covariance				**23.8** (5/21)	
Hotelling's T^2 No assumptions				**38.2** (13/34)	

K1, K2, Q1, Q2 and **Q3** all from different source (same manufacturing plant, but each manufactured between 2 weeks to 3 years and 4 months apart

Round Robin 4

	Type 1 Error Rate (%) False Exclusion		Type 2 Error Rate (%) False Inclusion		
	RR2	**RR4**	**RR2**	**RR3**	**RR4**
Comparison Interval ±4 * standard deviation (minimum 3% RSD)		**27.5** (33/120)			**0** (0/60)
T-Test (Welch's Modification) 95% confidence, Bonferroni correction		**70.0** (84/120)			**0** (0/60)
T-Test (equal variance) 95% confidence, Bonferroni correction		**67.5** (81/120)			**0** (0/60)
Equivalence Test θ calculated with known		**100** (120/120)			**0** (0/60)
Equivalence Test θ calculated with Cardinal glass		**64.2** (77/120)			**0** (0/60)
Equivalence Test θ calculated with FIU Database		**31.7** (38/120)			**0** (0/60)
Hotelling's T^2 Assuming equal covariance		**50.0** (10/20)			**20.0** (2/10)
Hotelling's T^2 No assumptions		**67.9** (19/28)			**26.7** (4/15)

K1, K2, Q2 and **Q3** from the same source

Q1 manufactured 2 weeks before (same manufacturing plant)

Error rates for collections using LA-ICP-MS data

FIU Collection 104 samples, 3 replicates BKA Collection 62 samples, 6 replicates	Type 2 Error Rate (%) False Inclusion	Type 2 Error Rate (%) False Inclusion	Type 2 Error Rate (%) False Inclusion
CFS Collection – 82 samples from casework, 9 replicates	**FIU[1]** Florida International University	**BKA[2]** Bundeskriminalamt	**CFS[3]** Centre of Forensic Science
Comparison Interval ±4 * standard deviation (minimum 3% RSD)	**0.3** (36/10712)	**0.1** (2/1891)	**0.1*** (7/6642)
T-Test (Welch's Modification) 95% confidence, Bonferroni correction	**2.2** (117/5356)	--	--
T-Test (equal variance) 95% confidence, Bonferroni correction	**0.5** (29/5356)	**0** (0/1891)	--
Equivalence Test θ calculated with known	**1.9** (206/10712)	--	--
Equivalence Test θ calculated with Cardinal glass	**0.02** (2/10712)	--	--
Equivalence Test θ calculated with FIU Database	**2.6** (277/10712)	--	--

* The minimum % RSD used differed for each element (4 or 5%)

1. Trejos, T.; et al, *Journal of Analytical Atomic Spectrometry*, **2013**
2. Weis, P.; et al. *Journal of Analytical Atomic Spectrometry,* **2011**
3. Dorn, H.; et al. *Canadian Society of Forensic Science Journal,* **2015**

Current Glass Collections/Databases

Laboratory	# of Samples	Types of Samples	RI or Elemental	Use Database?
Netherlands Forensic Institute(NFI)	1400	Architectural, Automotive, Container, Other	Elemental	"We use the database for calculation of likelihood ratios"
Centre of Forensic Sciences (Toronto)	>2300	Casework and Survey, Architectural, Automotive	Both	"Use modified ±4 s to compare question sample to database"
Bundeskriminalamt (Germany)	>600	Casework, Architectural, Automotive, Container	Elemental	"To assess the probability of a match between two randomly chosen glass samples from our case data collection" (< 0.1 %).
Florida Department of Law Enforcement (FDLE)	>700	Casework	Both	"Using database to aid in interpretation"
FBI	600	Automotive	Elemental	No
FIU	>700	Automotive, Architectural, Container, Other	Both	No

FIU database is available from TSWG (jeff.huber.ctr@cttso.gov)

E2927 method – Type 1 and type 2 error rates for interlab trials (RR2, RR3 and RR4 where RR4 uses atypical heterogeneity)

Match criteria	Type 1 error rate (%)			Type 2 error rate (%)		
	Test 2	**Test 3**	**Test 4**	**Test 2**	**Test 3**	**Test 4**
Range	42	-	81	0	0	0
t-test .05	74	-	93	0	1	0
t-test .01	53	-	84	0	1	0
t-test Bonf.	53	-	69	0	2	0
±2s	53	-	85	0	0	0
±2s (s>3%)	26	-	75	0	0	0
±3s	42	-	66	0	2	0
±3s (s>3%)	0	-	47	0	2	0
±4s	26	-	42	0	5	0
±4s (s>3%)	**0**	**-**	**28**	**0**	**5**	**0**
±5s	11	-	30	0	9	0
±5s (s>3%)	0	-	18	0	11	0
±6s	11	-	27	0	12	0
±6s (s>3%)	0	-	13	0	15	0
Equivalence	**74**	-	**100**	0	1	0

Type 1 error
Failure to associate samples with common origin was observed in ***RR4, with higher type I error*** *rates associated to* ***heterogeneity*** *of the sample source*

Type 2 error:
Failure to discriminate samples that originated from different sources was observed only for samples that originated from the ***same plant*** *manufactured* ***2 weeks apart*** *(RR3)*

T Trejos, et al, Forensic analysis of glass by μ-XRF, SN-ICP-MS, LA-ICP-MS and LA-ICP-OES: evaluation of the performance of different criteria for comparing elemental composition, *J. Anal. At. Spectrom.*, **2013**, 28, 1270-1282.

Bias and precision found in SRM NIST 1831 from inter-laboratory study and reported in E2927.

Element	Reported value, $\mu g g^{-1}$	Average, $\mu g g^{-1}$ D	Bias %	Repeatability-within s_r (%)	Reproducibility-between s_R (%)
Li	5.00 A	5.3	7.0	**5.1**	**5.6**
Mg	21200 B	23900	13	**1.1**	**10**
Al	6380 B	6400	0.3	**1.1**	**9.3**
K	2740 B	2690	-1.8	**2.3**	**7.2**
Ca	58600 B	58000	-1.0	**2.6**	**3.9**
Fe	608 B	500	-18	**2.7**	**22**
Ti	114 B	130	14	**2.6**	**7.0**
Mn	15.00 C	13.1	-13	**1.8**	**2.4**
Rb	6.11 C	6.0	-1.8	**2.4**	**3.8**
Sr	89.12 C	85	-5.0	**2.0**	**4.6**
Zr	43.36 C	36	-17	**2.2**	**6.8**
Ba	31.5 C	30.0	-4.4	**2.6**	**6.7**
La	2.12 A	2.2	4.2	**2.6**	**6.7**
Ce	4.54 C	4.4	-3.1	**2.6**	**3.8**
Nd	1.69 A	1.8	4.1	**2.3**	**7.1**
Hf	1.10 C	0.96	-13	**3.7**	**8.5**
Pb	1.99 C	1.8	-11	**5.0**	**6.7**

Data from 7 participant laboratories using different manufacturer LA and ICP-MS instruments

Products of NIJ-funded inter-laboratory trials

JAAS

Journal of Analytical Atomic Spectrometry

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PAPERS

1270

Forensic analysis of glass by μ-XRF, SN-ICP-MS, LA-ICP-MS and LA-ICP-OES: evaluation of the performance of different criteria for comparing elemental composition

Tatiana Trejos, Robert Koons, Peter Weis, Stefan Becker, Ted Berman, Claude Dalpe, Marc Duecking, JoAnn Buscaglia, Tiffany Eckert-Lumsdon, Troy Ernst, Christopher Hanlon, Alex Heydon, Kim Mooney, Randall Nelson, Kristine Olsson, Emily Schenk, Christopher Palenik, Edward Chip Pollock, David Rudell, Scott Ryland, Anamary Tarifa, Melissa Valadez, Andrew van Es, Vincent Zdanowicz and Jose Almirall*

Anal Bioanal Chem

DOI 10.1007/s00216-013-6978-y

RESEARCH PAPER

Cross-validation and evaluation of the performance of methods for the elemental analysis of forensic glass by μ-XRF, ICP-MS, and LA-ICP-MS

Tatiana Trejos · Robert Koons · Stefan Becker · Ted Berman · JoAnn Buscaglia · Marc Duecking · Tiffany Eckert-Lumsdon · Troy Ernst · Christopher Hanlon · Alex Heydon · Kim Mooney · Randall Nelson · Kristine Olsson · Christopher Palenik · Edward Chip Pollock · David Rudell · Scott Ryland · Anamary Tarifa · Melissa Valadez · Peter Weis · Jose Almirall*

Research article

Signal-to-noise ratios in forensic glass analysis by micro X-ray fluorescence spectrometry

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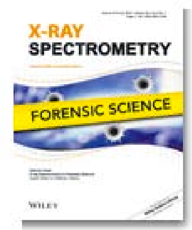
Issue



X-Ray Spectrometry

Special Issue: X-ray Spectometry in Forensic Science

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ASTM E2926 - 13

Standard Test Method for Forensic Comparison of Glass Using Micro X-ray Fluorescence (μ-XRF) Spectrometry

ASTM E2927 - 13

Standard Test Method for Determination of Trace Elements in Soda-Lime Glass Samples Using Laser Ablation Inductively Coupled Plasma Mass Spectrometry for Forensic Comparisons

Significance of Elemental Analysis from Trace Evidence

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Elemental Analysis in Forensic Science: Practice

"*Elemental analysis methods are used (**should/shall be**) when other methods of comparison fail to distinguish two glass fragments as having different sources.*"

SWGMAT Guidelines on Elemental Analysis of Glass; 2004

http://www.swgmat.org/Elemental%20Analysis%20of%20Glass.pdf

SEM-EDS is not recommended due to limitations in sensitivity for detection of trace elements (MDL ~ 1000 ppm)
uXRF, solution/digestion ICP-MS and LA-ICP-MS are methods of choice in operational forensic laboratories.

Of the ~ **111** trace evidence laboratories completing the **2013 CTS glass examination**, **31 labs** reported using XRF and **11 labs** reported using ICP-MS or LA-ICP-MS, (**43/111 or only 39%** follow SWGMAT Guidelines).

Six (6) incorrect responses included 1 SEM-EDS and labs with no elemental analysis

Forensic LA-ICP-MS or LIBS labs in the U.S.	Forensic LA-ICP-MS or LIBS labs around the world
FBI Laboratory (CFRSU)	Dubai Police, United Arab Emirates (UAE)
Sacramento County Forensic Laboratory	National Forensic Science Service, Seoul (Korea)
Texas Department of Public Safety	National Research Institute of Police Science (Japan)
Iowa State University/Ames Laboratory	Health Sciences Authority Forensic Lab (Singapore)
Tennessee Bureau of Investigation FSD	Beijing Police Forensic Science Lab (China)
U.S. Customs and Border Protection, DHS	Madrid Federal Police (Spain)
Homeland Security Investigation Lab, DHS	Netherlands Forensic Institute (The Hague)
New Jersey State Police Forensic Laboratory	Forensic Science Institute (BKA, Germany)
South Carolina Law Enforcement Division	State Forensic Labs in Germany (LKAs)
Virginia Department of Forensic Sciences (LIBS)	RCMP, (Ottawa, Canada)
Food and Drug Administration Forensic Labs	Barcelona Guardia Civil (Spain)
U.S. EPA Forensic Laboratory	South Africa Police Services Lab (Pretoria, South Africa)
Several other LIBS installations in the US	Australian Federal Police (Canberra, Australia) (LIBS)
Florida International University, IFRI Lab	Brazilian Federal Police Forensic Laboratory, and more

LA-ICP-MS installed instruments in Forensic Laboratories

~ 30 forensic labs around the world

12 forensic labs in North America

…and, what about significance?

What we say in the report and in testimony will vary depending on the conditions of the particular case.

- The instrumental method(s) used in the comparison
- Number of fragments found that are indistinguishable
- Number of different sources found indistinguishable
- Location of the recovered fragments (ie., shoe?)
- How common or uncommon is the glass? ✓
  - ie. How many car windows have the same composition?
- Other domain-relevant context……

Likelihood Ratio (LR) Estimation (Source and Activity)

Association scale:	Equivalent LR
Association: Identification	∞
Association: Very Strong Evidence	1,000 – 10,000
Association: Strong Evidence	10 – 100
Association: Some evidence	1 – 10
Inconclusive (no support for either proposition)	1
Evidence of no association	0.1
Strong evidence of no association	0.001
Very strong evidence of no association	0.000001
Elimination:	0

J. Almirall and T. Trejos, Analysis of Glass Evidence (Chapter 6) *in Forensic Chemistry: Fundamentals and Applications, J. Siegel, Ed.* ***2015***. *Wiley and Sons (in press)*

Acknowledgments

The Des Moines Register, July 24, 2003, p. 5B

Scientists see glass forensics making strides

By STACI HUPP
REGISTER AMES BUREAU

Ames, Ia. — The tiniest fragments of evidence gathered from crime scenes could make big strides in putting vandals, thieves and killers behind bars, Ames scientists say.

Technology developed in Ames enables crime laboratories to match microscopic shards of glass found in a suspect's hair, shoes or home with glass at a crime scene.

It's a step up from older methods that require larger glass samples and more time. Researchers also believe it's exact enough to erase reasonable doubt in many criminal court cases.

"It's picking up speed as people are adopting the technology and hearing more about it," said Jose Almirall, a Florida International University researcher who trained criminalists from 15 states at the U.S. Department of Energy's Ames Laboratory on Wednesday.

Almirall and his research team have used the technique to help solve crimes in several southern states, but they hope it catches on.

The technology helped nab a Miami man who drove away after he struck and killed a pedestrian with his car, Almirall said. The driver told police his car had been stolen, but experts matched glass fragments found in the man's clothes hamper and bathtub with the car's windshield.

Criminalists call it a real-life example of the crime-solving seen on CBS-TV's "CSI."

But Wednesday's workshop was all science and no drama. Crime lab experts watched as instructors inserted pieces of glass into a machine, then saw the process unfold on computer monitors. State officials spent $600 to send two Iowa criminalists to the workshop.

Here's how the technology works: Lasers zap glass fragments, which evaporate into particles that have the consistency of cigarette smoke. Then a gas sweeps the particles into a hot plasma that allows criminalists to see a unique chemical makeup.

The technique is so precise that criminalists are able to trace the fragments to a glass manufacturer, Almirall said.

Sam Houk, an Ames lab researcher who helped develop the technology as an ISU graduate student in the 1970s, said it could become a new frontier in forensics. Houk sees other uses for it such as analyzing paint chips and the elements in lead bullets.

The equipment costs hundreds of thousands of dollars, but federal grants could cover some of the expenses. About 4,000 machines around the world are capable of the forensics technique, Houk said. But the equipment traditionally has been set aside for more high-profile work, such as spotting radioactive elements used for nuclear weapon development.

Nick Gerhardt of the Missouri State Highway Patrol's criminal lab was impressed.

Small glass samples "can be really hard to work with," said Gerhardt, who has analyzed glass and paint samples from crime scenes. "There are definitely times when this may be the only way of discriminating."

On the Web

For more information on the inductively coupled plasma-mass spectrometry (ICP-MS) technique, go to: http://www.epsci.ameslab.gov/etd/technologies/projects/icpms/icpms.html

RODNEY WHITE/THE REGISTER

Changed view: Tatiana Trejos, foreground, of Florida International University, helps teach a glass forensics workshop at the U.S. Department of Energy's Ames Laboratory on Wednesday.

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- Tricia Hoffman and Ruthie Corzo

Summary of Errors Involved in Hypothesis Testing

Inference Based on Sample Data	Real State of Affairs: H_0 is True	Real State of Affairs: H_0 is False
H_0 is True	Correct decision	Type II error P (Type II error) = β
H_0 is False	Type I error Significance level = α *	Correct decision Power = $1-\beta$

*Term α represents the maximum probability of committing a Type I error