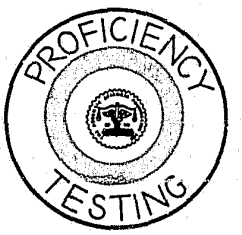
LABORATORY PROFICIENCY TESTING PROGRAM

REPORT NO. 17

METAL EXAMINATION



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LABORATORY PROFICIENCY TESTING PROGRAM

REPORT NO. 17

METAL EXAMINATION

PROJECT STAFF

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Statistical presentations prepared by:

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Vienna, Virginia

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Points of view or opinions stated in this document are those of the authors and do not necessarily represent the official position or policies of the U.S. Department of Justice.

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FOREWORD

The analysis summarized in this report is the seventeenth of a series that will be made in conjunction with this proficiency testing research project.

In the course of this testing program participating laboratories will have analyzed and identified different samples of physical evidence similar in nature to the types of evidence normally submitted to them for analysis.

The results for Test Number Seventeen are reflected in the charts and graphs which follow.

The citing of any product or method in this report is done solely for reporting purposes and does not constitute an endorsement by the project sponsors.

Comments or suggestions relating to any portion of this report or of the program in general will be appreciated.

April 1977

BACKGROUND

This laboratory proficiency testing research project, one phase which is summarized in this report, was initiated in the fall of 1974.

This is a research study of how to prepare and distribute specific samples; how to analyze laboratory results; and how to report those results in a meaningful manner. Information is being collected for research and statistical purposes only. Such information will not be revealed or used for any other purpose. Information furnished by any person or agency identifiable to any specific person or laboratory will not be revealed or used for any purposes, other than the research and statistical purposes for which it was obtained.

Participation in the program is voluntary. Accordingly, invitations have been extended to 239 laboratories to share in the research. It is recognized that all laboratories do not perform analyses of all possible types of physical evidence. Thus, in the data summaries included in this report, space opposite some Code Numbers (representing specific laboratories) may be blank, or marked "No Data Returned."

Additional evaluations of individual tests will be published in a separate report.

The Project is under the direct control of the Project Advisory Committee whose members' names are listed on the Title Page. Each is a nationally known criminalistic laboratory authority.

Supporting the Project Advisory Committee in their efforts is the Forensic Sciences Foundation with additional support from the Collaborative Testing Service, Inc., Vienna, Virginia in the area of statistical presentation.

SUMMARY

In this test, each of 239 laboratories were sent three metal samples which were referred to as Items A, B, and C. Participants were asked: (1) Could Items A, B or C have a common origin? (2) What tests were employed to answer Question 1? (3) Report any elemental data. (4) Report those elements which were sought but not found.

Of the 239 laboratories, 68 laboratories responded with data, 82 indicated they do not perform metal analysis, and 89 did not responded. This represents a participation rate of 43%.

The information contained in the tables consists of the following:

Table 1 - Supplier's Characteristics
Table 2 - Responses to Question 1
Table 3 - Frequency of Reported Methods
Table 4 - Summary of Elements Found
Table 5 - Summary of Laboratory Results
Table 6a & 6b - Supplementary Tables Regarding Individual Laboratory Results.

ANNEX A

LAB CODE _____

FIGURE 1.

☐ CHECK HERE (AND RETURN) IF YOU DO NOT PERFORM METAL EXAMINATION

DATE RECEIVED IN LAB_____

DATE PROCESSED IN LAB_____

DATA SHEET
PROFICIENCY TESTING PROGRAM

TEST #17
METAL EXAMINATION

Items A, B, and C represent metal samples submitted in connection with a criminal case.

1. a) Could Items A and B have a common origin?

☐ Yes

☐ No

☐ Inconclusive

b) Could Items A and C have a common origin?

☐ Yes

☐ No

☐ Inconclusive

c) Could Items B and C have a common origin?

☐ Yes

☐ No

☐ Inconclusive

2. What tests were employed to answer Question 1? (Please be specific, e.g. emission spectroscopy, energy dispersive X-Ray, etc.) Use page 4 if additional space is required.

a._____

b._____

c._____

d._____

3. Please report any elemental data (both qualitative and quantitative) developed in the analysis of Items A, B, and C. Report quantitative data in either % by weight or ppm. Indicate which instrumental techniques identified each element reported.

ITEM A			ITEM B			ITEM C		
Element	Instrument	Quantity	Element	Instrument	Quantity	Element	Instrument	Quantit

4. If particular elements were sought but found not to be present in Items A, B, and C, please indicate those elements below.

ITEM A		ITEM B		ITEM C	
Element	Instruction	Element	Instruction	Element	Instruction

5. Additional Comments:

Table 1

Supplier's Characteristics

Item A: National Bureau of Standards Standard Reference Material 362, AISI 94B17 Steel (Modified)

Items B & C : National Bureau of Standards Standard Reference Material 19G, Acid Open Hearth Steel, 0.2% Carbon

The chemical composition (nominal weight percent) of the materials is as follows:

	C	Mn	P	S	Si	Cu	Ni	Cr	V	Mo	W	Co	Ti	As
Item A	.160	1.04	.014	.038	.39	.50	0.59	.30	.040	.068	(.20)	.30	(.084)	(.079)
Items B&C	0.223	.554	.046	.033	..186	.093	.066	.374	.012	.013	-	0.012	0.027	-

	Sn	Al	Nb	Ta	Zr	N	B	Pb	Sb	Bi	Ag	Se	Te	Ce
Item A	(.016)	(.086)	(.28)	(.20)	(.21)	(.0040)	(.0025)	(.0006)	(.013	(.006)	(.0009)	(.001)	(.001)	(.002)
Items B&C	0.008	.031	0.026	-	-	-	-	-	-	-	-	-	-	-

	La	Nd	Ca	Mg	Zn	Pr	Ge	O	H	Au	Hf
Item A	(.0005)	(.0005)	(.0003)	(.0007)	(.001)	(.0003)	(.002)	(.001)	(<.0005)	(<.00006)	(.0040)
Item B&C	-	-	-	-	-	-	-	-	-	-	-

Table 2

Response to Question 1

QUESTION 1a) Could Items A and B have a common origin?
b) Could Items A and C have a common origin?
c) Could Items B and C have a common origin?

Response	Number of Labs Giving Response	%
NNY	49	72.1%
NNI	6	8.8%
NNN	6	8.8%
YYY	2	2.9%
III	2	2.9%
NYN	1	1.5%
No Response	2	2.9%
TOTAL	68	99.9%

Question	Responses		
	Yes	No	Inconclusive
1a) Could A and B have a common origin:	2	62 (93.9%)*	2
1b) Could A and C have a common origin?	3	61 (92.4%)*	2
1c) Could B and C have a common origin?	51 (77.3%)*	7	8

*Labs giving no response not included in this percent calculation.

Note: Responses above are coded as three letters, with each letter corresponding to a possible response to one of the three questions asked in Question 1. For example, NNY would correspond to No for 1a, No for 1b, Yes for 1c

Table 3

Frequency of Reported Methods

Method	Number of Labs Reporting Use of this Method	Percentage of Responding Labs Reporting Use of this Method
Emission Spectroscopy	40	58.8%
Energy Dispersive X-ray	25	36.8%
Microscopic Examination	11	16.2%
Chemical Tests	11	16.2%
X-ray Fluorescence	7	10.3%
Magnetic	7	10.3%
Macroscopic Exam	5	7.4%
X-ray Diffraction	2	2.9%
Atomic Absorption	2	2.9%
NAA	1	1.5%
UV-Visible Spectrophotometry	1	1.5%

Table 4

Frequency of Reported Elements

Elements	Number of Labs Reporting Presence of Element in Item A	Number of Labs Reporting Presence of Element in Items B & C
Iron	54	54
Nickel	47	38
Manganese	46	48
Chromium	45	48
Copper	43	39
Titanium	23	19
Cobalt	21	12
Zirconium	21	2
Niobium	21	11
Aluminum	20	20
Silicon	19	19
Molybdenum	14	14
Tin	13	12
Magnesium	11	11
Silver	9	6
Arsenic	9	4
Calcium	6	6
Lead	6	5
Vanadium	6	5
Zinc	5	6
Antimony	4	4
Tungsten	3	2
Carbon	2	1
Bromine	2	2
Lanthanum	2	2
Tantalum	2	1
Potassium	2	2
Palladium	1	1
Phosphorus	1	1
Sulfur	1	1
Bismuth	1	0
Germanium	1	1
Cesium	1	1

Table 5

Summary of Laboratory Responses

LAB CODE			044		058					194*												
RESPONSE	010		NNI		NNY					NNY												
METHOD	NNY		E.S.		EDX					Macrosc.												
USED	E.S.		X-ray		HCl;HNO_3		071*		169	Microsc.		197		210		214		219	220*			
			Spec.		UV-		NNY		NNY	X-ray		NNY		NNY		NNY		No	NNY			
					Visible		E.S.		E.S.	Fluor.		E.S.		E.S.		EDX		Resp.	X-ray	Fluor.		
ELEMENT FOUND	A	B&C	A	B&C	A	B&C	A	B&C	A B&C	A	B&C	A	B&C	A	B&C	A	B&C	A B&C	A	B&C		
C					X	X																
Mn	X	X	X	X			X	X		X	X			X	X	X	X		X	X		
P																						
S																						
Si							X	X														
Cu			X	X						X	X								X	X		
Ni	X	X					X	X		X	X	X	X	X	X	X	X		X	X		
Cr					X	X	X	X		X	X	X	X	X	X	X	X		X	X		
V																						
Mo							X	X		X	X	X	X	X	X							
W																						
Co	X						X	X				X	X	X	X							
Ti			X				X	X						X	X				X	X		
As																						
Sn																						
Al			X	X										X	X							
Nb										X	X					X						
Ta																						
Zr										X						X			X			
N																						
B																						
Pb										X	X											
Sb																						
Bi																						
Ag	X																					
Se										X	X											
Te																						
Ce																						
La																						
Nd																						
Ca																						
Mg	X	X																				
Zn	X																					
Pr																						
Ge																						
O																						
H																						
Au																						
Hf																						
K																						
Fe	X	X	X	X			X	X		X	X	X	X	X	X	X	X		X	X		
Cd										X	X											
Pd										X	X											
Br										X	X											

* See Table 6b for elements sought but not found

** See Table 6a for methods used

See note in Table 2 for an explanation of the three letter response under the lab codes above.

Table 5 (continued)

LAB CODE RESPONSE METHOD USED	232 NNY EDX	292 NNY SEM/ EDX	318* NNY * *	323 NNY E.S.	330 NNY EDX	333 NNY 1.Micro 2.EDX	338 NNN E.S.	341 NNY EDX	367 NNY 1.Solub, 2.E.S,	372 NNY * *	395* NNY * *
ELEMENT FOUND	A B&C	A B&C	A B&C	A B&C	A B&C	A B&C	A B C	A B&C	A B&C	A B&C	A B&C
C											X
Mn	X X	X X				X X	X X X		X X		X X
P							X X X				
S		X									
Si		X X					X X X				
Cu	X X	X				X X	X X X		X X	X	
Ni	X X	X	X X			X X	X X X		X X	X	X X
Cr	X X	X X	X X			X X	X X X	X	X X	X X	X X
V							X X X		X X		X
Mo							X X X		X X		
W											
Co			X						X X		X X
Ti							X X X		X X		X X
As	X					X X					
Sn							X X X				X X
Al							X X X		X X		X X
Nb	X X					X X		X X			
Ta											
Zr	X							X	X X		
N											
B											
Pb									X X		
Sb											
Bi											
Ag											
Se											
Te											
Ce											
La		X X									
Nd											
Ca											
Mg									X X		X X
Zn						X					
Pr											
Ge											
O											
H											
Au											
Hf											
K											
Fe	X X	X X	X X			X X	X X X	X X	X X	X X	X X
Cd											
Pd											
Br											

* See Table 6b for elements sought but not found

** See Table 6a for methods used

Table 5 (continued)

LAB CODE RESPONSE METHOD USED	412* NNY E.S.		429 III		435* NNY * *		436 NNY 1.Micro 2.Chem. 3.E.S.		459 NNY EDX		472 NYN E.S.		481 NNY 1.Micro 2.Magnet 3.E.S.		514 NNY EDX		515* NNY 1.Micro 2.EDX		532 NNY 1.X-ray Fluor. 2.SEM/EDX	
ELEMENT FOUND	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C
C																				
Mn	X	X			X	X	X	X		X	X	X	X	X	X	X	X	X	X	X
P																				
S																				
Si	X	X			X	X							X	X					X	X
Cu	X	X			X	X	X	X	X		X	X	X	X	X	X	X	X	X	X
Ni	X	X			X	X					X	X	X	X	X		X		X	
Cr	X	X			X	X					X	X	X	X	X	X	X	X	X	X
V																				
Mo	X	X									X	X	X	X	X	X				
W					X	X														
Co					X	X							X	X						
Ti					X		X	X			X	X	X	X			X	X	X	X
As					X	X														
Sn	X	X			X	X							X	X						
Al	X	X									X	X	X	X						
Nb					X	X			X											
Ta					X	X														
Zr					X				X						X		X			
N																				
B																				
Pb	X	X					X	X												
Sb					X	X	X	X												
Bi																				
Ag							X	X					X	X						
Se																				
Te																				
Ce																				
La																				
Nd																				
Ca																			X	X
Mg													X	X						
Zn																				
Pr																				
Ge																				
O																				
H																				
Au																				
Hf																				
K																				
Fe					X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Cd																				
Pd																				
Br																				

* See Table 6b for elements sought but not found

** See Table 6a for methods used

Table 5 (continued)

LAB CODE	543		565*		616		617*		620		624		647		664*		677		681		705	
RESPONSE	YYY		NNN		NNY		NNY		NNY		NNY		NNY		NNY		NNY		NNI		NNY	
METHOD USED	1.Macro 2.Magnet 3.E.S.		E.S.		EDX		EDX		EDX		EDX		E.S.		X-ray Spec.		E.S.		Particle Size E.S.		1.Micro 2.E.S.	
ELEMENT FOUND	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C
C																						
Mn							X	X	X	X	X	X	X	X	X	X			X	X		
P																						
S																						
Si																			X	X		
Cu							X	X	X	X	X	X	X	X					X	X		
Ni							X		X	X	X		X	X	X				X	X		
Cr							X	X	X	X	X	X	X	X		X			X	X		
V													X	X					X	X		
Mo																			X	X		
W																						
Co			X	X									X	X					X			
Ti													X	X					X	X		
As							X				X	X										
Sn																			X	X		
Al													X	X					X	X		
Nb							X		X		X	X			X				X	X		
Ta																						
Zr							X		X		X				X							
N																						
B																						
Pb									X													
Sb																						
Bi																						
Ag													X	X								
Se																						
Te																						
Ce																						
La																						
Nd																						
Ca																						
Mg													X	X								
Zn							X		X													
Pr																						
Ge																						
O																						
H																						
Au																						
Hf																						
K																						
Fe							X	X	X	X	X	X	X	X	X	X			X	X		
Cd																						
Pd																						
Br																						

* See Table 6b for elements sought but not found

** See Table 6a for methods used

Table 5 (continued)

LAB CODE				787		847				
RESPONSE	763*	770	772	NNY	823	836*	NNY	850	854	882
METHOD	NNY	NNI	III	1.E.S.	NNY	NNI	1.Macro	YYY	1.Micro	NNI
USED	E.S.	1.Solub.	E.S.	2.EDX	1.Wet	Atom.	2.Micro	EDX	2.Magnet	E.S.
		2.E.S.		3.Atomic	Tests	Spec.	3.Magnet			
				Absorp.	2. E.S.		4.X-Ray Fl.			

ELEMENT FOUND	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C
C																		
Mn	X	X					X	X	X	X	X	X	X	X			X	X
P																		
S							X	X										
Si	X	X					X	X			X	X			X		X	X
Cu	X	X			X	X X	X	X	X	X	X	X	X	X			X	X
Ni	X	X					X	X	X		X	X	X				X	X
Cr	X	X					X	X	X	X	X	X	X	X	X		X	X
V																		
Mo	X	X																
W													X					
Co									X	X	X	X						
Ti	X	X									X	X						
As													X	X				
Sn											X	X	X					
Al	X	X					X	X			X	X			X			
Nb													X					
Ta																		
Zr													X					
N																		
B																		
Pb													X	X				
Sb																		
Bi									X									
Ag					X	X		X			X	X						
Se																		
Te																		
Ce																		
La																		
Nd																		
Ca									X	X	X	X						
Mg									X	X	X	X						
Zn											X	X						
Pr																		
Ge																		
O																		
H																		
Au																		
Hf																		
K																		
Fe	X	X			X	X X	X	X	X	X	X	X	X	X			X	X
Cd																		
Pd																		
Br																		

* See Table 6b for elements sought but not found

** See Table 6a for methods used

Table 5 (continued)

LAB CODE			891	895		905*	912	915*	971 **	
RESPONSE	887	890	NNY	NNI	897	NNY	NNY	NNY	NNY	972*
METHOD USED	NNN E.S.	NNY 1.Magnet 2. E.S.	Acid Digest E.S.	1. E.S. 2.Atom Absorb	NNY E.S.	EDX	Solub. E.S.	EDX E.S.	1.E.S, 2.Micro 3.Chem.	NNY 1.EDX 2.Micro
ELEMENT FOUND	A B&C	A B&C	A B&C	A B&C	A B&C	A B&C	A B&C	A B&C	A B&C	A B&C
C										
Mn	X X X	X X	X X	X X		X X	X X	X X	X	X X
P										
S										
Si	X X X			X X		X X	X X	X X	X	
Cu	X X X	X X	X X	X X	X X	X		X X	X	X X
Ni	X X X	X X	X X	X X	X X	X X	X X		X	X X
Cr		X X	X X	X X		X X	X X	X X	X	X X
V	X X X									
Mo				X X					X	X
W							X X			
Co							X X			
Ti	X X X	X		X X	X X				X	
As						X				X
Sn	X X X		X X	X X						
Al		X X	X X	X X			X X	X X		
Nb						X		X		X X
Ta										
Zr						X		X		X
N										
B										
Pb				X X						
Sb										
Bi	X		X				X			
Ag										
Se										
Te										
Ce										
La										
Nd										
Ca	X		X X							X X
Mg	X X X			X X						
Zn				X X	X X					X
Pr										
Ge										X X
O										
H										
Au										
Hf										
K										
Fe	X X	X X	X X	X X	X X	X X	X X	X X		X X
Cd										
Pd										
Br										X X
Cs						X X				

*See Table 6b for elements sought but not found

**See Table 6a for methods used

Table 5 (continued)

LAB CODE	978		984		986		988		991	
RESPONSE	NNY		NNY		NNN		NNN		NNY	
METHOD USED	1.Macro 2.Chem 3. E.S.		EDX		1. E.S. 2.X-ray Fluor.		1. EDX 2. Chem		1. E.S. 2. Solubility	
ELEMENT FOUND	A	B&C	A	B&C	A	B&C	A	B&C	A	B&C
C										
Mn	X	X	X	X	X	X X	X	X X	X	X
P										
S										
Si	X	X								
Cu	X	X	X	X	X		X	X X	X	X
Ni	X	X	X		X		X	X X	X	X
Cr	X	X		X	X	X X	X	X X		
V										
Mo										
W										
Co									X	
Ti									X	X
As	X									
Sn	X	X							X	X
Al	X	X								
Nb					X		X	X X		
Ta							X			
Zr			X		X		X		X	X
N										
B										
Pb									X	X
Sb										
Bi										
A										
Ce										
La							X	X X		
Nd										
Ca					X	X X				
Mg	X	X							X	X
Zn										
Pr										
Ge										
O										
H										
If										
Fe	X	X	X	X	X	X X	X	X X	X	X
Cd										
Pd										
Ir										

* See Table 6b for elements sought but not found.

** See Table 6a for methods used

Table 6a

Supplemental Table for Reported Methods

Lab Code	Methods Reported
318	1. Emission Spectroscopy 2. EDX 3. Magnetism 4. Reflected Light Microscopy 5. Acid Solution - Insoluble Residue 6. X-ray Diffraction
372	1. X-ray Diffraction 2. Emission Spectroscopy 3. X-ray Fluorescence 4. Microscopic--magnetic
395	1. Emission Spectrograph--no matrix 2. Emission Spectrograph--Li_2CO_3 matrix 3. Plasma Emission Spectrometer 4. Solubility
435	1. Emission Spectroscopy 2. Wavelength Dispersive X-ray Fluorescence 3. NAA

Table 6b

Elements Reported Sought but not Found to be Present

Lab Code	Item A	Item B	Item C
071	V, W	V, W	V, W
194		Zr	Zr
220		Zr	Zr
318	C, B, Li, Be	C, D, Li, Be	C, D, Li, Be
395	Mo, Cu, W, Pb, Zn	Mo, Cu, W., V, Pb, Zn	Mo, Cu, W, V, Pb, Zn
412	Zr, Zn, V, Nb, Ti, Ag, Mg, W, P, B	Zr, Zn, V, Nb, Ti, Ag, Mg, W, P, B, Co	(B and C same)
435	Br, Na, Sc, Ba, Zn	Br, Na, Sc, Ba, Zn, Zr, Ti	(B and C same)
515		Nb, Zr	Nb, Zr
565		Co	
617	K, Ca, Sc, Ti, V, Co, Zn, Ga, Ge, Se, Br, Kr, Rb, Sr, Y, Mo, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At, Rn, Fr, Ra, Ac, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf, Es, Fm, Md, No, Lw	Same as A except Zr, Nb, were also not found Also Zn was found	
664	Cr	Ni, Zr, Nb (same for B and C)	
681	B, P, Pb	B, P, Pb, Co (same for B and C)	
763	Ag, B, Ba, Be, Bi, Cd, Mg, Na, Zn, Ph, Sn, W	B and C same as A except Co also not found	

Table 6b (continued)

Lab Code	Item A	Item B	Item C
836	As, B, Sb, Hg, W, Be, Ge, Cd, Tl, Pb, Ba, V, Na, Sr, Zr	B and C same as A except Bi, also not found	
890	Mo	Mo	Mo
905	Ti, Sc, V, Ag, Cd, Xe, Ba, Na	Cu, Ti, Sc, V, Zr, Nb, Ag, Cd, Xe, Ba	(B and C same)
915	Be, Na, Mg, K, Ca, Ti, Mo, V, Co, Ta, W, Ge	B and C same as A except Zr and Nb also not found	
972	Zn, V, W, Bi, Pb, Ti, K, In, La, Co, Ga, Ba	V, W, Bi, Pb, Ti, K, In, La, Co, Ga, Ba, Mo, Zr	(B and C same)

END