# LABORATORY PROFICIENCY TESTING PROGRAM

# REPORT NO. 16 PAINT EXAMINATION



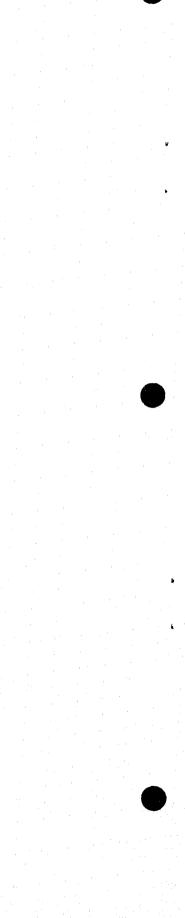


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#### **FOREWORD**

The analysis summarized in this report is the sixteenth 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 sixteen 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.

February 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 238 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 188 laboratories were sent three paint samples which were referred to as Items A, B, and C. Participants were asked: (1) Could Items A or C have common origin with B? (2) What information did you develop to arrive at your conclusions in Question 1? (3) What methods and instruments were used:

Of the 188 laboratories, 102 laboratories responded with data, 5 indicated they do not perform paint analysis, 79 did not respond, and 2 were unable to complete the test. This represents a participation rate of 56%.

The information contained in the six tables consisted of the following:

Table 1 - Supplier's Characteristics

Table 2 - Referee Laboratory Results

Table 3 - Responses to Question 1

Table 4 - Frequency of Reported Methods

Tables 5a and 5b - Comparison of Item B to Items A and C

Table 6 - Summary of Laboratory Results

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#### **ANNEX A**

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

DATE	RECEIVED IN LAB
DATE	PROCESSED IN LAB

LAB CODE

## DATA SHEET PROFICIENCY TESTING PROGRAM

## TEST # 16 PAINT EXAMINATION

	Item B represents a pa A and C represent samp	les found on the	clothing of tw	o different	suspects.	I tems
_ C	ould Items A or C have com	mon origin with	3?	• 1		
		ITEM A	ITEM C		•	
	YES			•		•
	NO					
	INCONCLUSIVE					

2. What information (qualitative and quantitative) did you develop to arrive at your conclusions in Question 1? Please check all appropriate boxes and provide values where applicable.

In the left hand column indicate the sequence (1,2,3 etc.) in which the tests were run. Indicate with an asterisk (\*) the point where a conclusion was reached, even though subsequent tests were performed for confirmatory purposes.

Sequence of Testing	ITEM A	ITEM B	ITEM C
DEMSITY STUDIES			
EMISSION SPECTROSCOPY (Specify Elements Identified)			
FLUORESCENT STUDIES			
INFRARED ANALYSIS			
MACROSCOPIC EXAMINATION			
MICROSCOPIC EXAMINATION			
PYROLYSIS G-C			
SOLUBILITY TESTS (Specify Solvents Used)			
THIN LAYER CHROMATOGRAPHY			
UV SPECTROPHOTOMETRY			
X-RAY DIFFRACTION			
X-RAY FLUORESCENCE (Count Ratio)			
OTHER (SPECIFY)			
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•		•
• •		
	Method:	
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4.	1. Additional Comments:	
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Please specify the information developed with each of the methods and instruments checked in Question 2. (Example: Solubility tests using HCl,  $\rm H_2SO_4$ , Acetone and  $\rm HNO_3$ ). Please provide specific and complete responses. Attach additional sheets if necessary.

Method:

DATA SHEETS MUST BE RECEIVED AT THE FOUNDATION OFFICE BY AUGUST 9,1976

Table 1

#### Supplier's Characterization of Samples

The paints were drawn at six mils wet film on glass to yield approximately 120 square inches for each sample. The three samples consist of the following:

Content	A	Sample B	<u> </u>
TiO <sub>2</sub>	3.0 lbs.	3.0 lbs.	2.0 lbs.
Zn0			1.0 lbs.
Solids Soya Alkyd		3.6 lbs.	3.6 lbs.
Solids Acrylic Alkyd	3.6 lbs.		

All have traces of Iron, Zinc, Lead and Cobalt.

Samples A, B, and C could not have common origin with each other.

#### Table 2

#### RESULTS OF THE REFEREE LABORATORY

I. Response to Question 1

No for Item A No for Item C

#### II. Methods and Results

- 1. Macroscopic Examination A, B, C similar in color
- 2. Microscopic Examination A, B and C are all single layer, but A is more brittle than B or C
- 3. Solubility Tests Unable to detect any significant differences between samples A, B and C
  - A. H<sub>2</sub>SO<sub>4</sub> Turns deep golden yellow; entire pigment dissolves
  - B. Acetone, DMSO, DMF, ChCl3, HNO3, M-cresol, butyrolactone, Benzyl alcohol, MeCl2 - Yellow pigment dissolves; residue grayfth in color
- 4.\* Pyrolysis G-C Test samples B and C very similar. One high boiling compound present in A in 2-3 times the quantity as a compound having the same retention time in samples B and C.
- 5.\* X-ray Fluorescence Sample C has a very high concentration of zinc whereas A and B have none. Also, C has a slightly higher amount of lead where A and B display only a trace of lead.

Table 3
SUMMARY OF RESPONSES TO QUESTION 1

Response	Number of Labs Giving Response	% of Total Labs
No for Items A and C	67	65.6%
Yes for Item A and No for Item C	11	10.7%
No for Item A and Yes for Item C	11	10.7%
Yes for Items A and C	<b>3</b>	2.9%
Inconclusive for Item A and		
No for Item C	3	2.9%
No for Item A and Inconclusive		
for Item C	5	4.9%
Inconclusive for Items A and C	2	2.0%

Table 4
FREQUENCY OF REPORTED METHOD USED TO ANSWER QUESTION 2

Question 2: What information did you develop to arrive at your conclusion?

<u>Method</u>	Number of Reported Uses of This Method	% of Responding Labs Using This Method
Microscopic Examination	95	93.1%
Macroscopic Examination	88	86.3%
Solubility Tests	87	85.3%
Pyrolysis G-C	61	59.8%
Infrared Analysis	48	47.1%
Emission Spectroscopy	35	34.3%
Fluorescent Studies	31	30.4%
X-ray Fluorescence	22	21.6%
X-ray Diffraction	14	13.7%
Thin Layer Chromatography	14	13.7%
UV Spectrophotometry	8	7.8%
Density Studies	4	3.9%
Visible Spectrophotometry	2	2.0%
Microchemical	2	2.0%
EDAX	1	1.0%
Energy Dispersive Spectroscopy	1	1.0%
Thermogravimetric Analysis	1	1.0%
Polarizing Microscopy	1.0	1.0%
Scanning Electron Microscope	· 1	1.0%
Spectral Reflectance	1	1.0%
GC of Binder Extract	1	1.0%

Table 5a

#### Comparison of Item A and Item B by the Eight Most Frequently Reported Methods

<u>Method</u>	Total Number of Labs Reporting Comparison of Item A and Item B by This Method.	Number of Labs Reporting They Could Differentiate Item A and Item B by This Method.	Number of Labs Reporting They Could Not Differentiate Item A and Item B by This Method.
Macroscopic Exam	73	12	61
Microscopic Exam	85	11 julius 1	74
Solubility Tests	75	24	51
Pyrolysis G-C	53	50	<b>3</b>
Infrared Analysis	42	22	20
Emission Spectroscopy	27	6	21
Fluorescent Studies	25	0	25
X-ray Fluorescence	17	. <b>8</b>	9

Table 5b

#### <u>Comparison of Item B and Item C</u> by the <u>Eight Most Frequently Reported Methods</u>

<u>Method</u>	Total Number of Labs Reporting Comparison of Item B and Item C by This Method.	Number of Labs Reporting They Could Differentiate Item B and Item C by This Method.	Number of Labs Reporting They Could Not Differentiate Item B and Item C by This Method.
Macroscopic Exam	73	<b>5</b>	68
Microscopic Exam	82	10	82
Solubility Tests	69	14	55
Pyrolysis G-C	49	17	32
Infrared Analysis	34	<b>9</b>	25
Emission Spectroscopy	32	31	<b>1</b>
Fluorescent Studies	25	12	13
X-ray Fluorescence	20	20	0

Table 6

Detailed Summary of Laboratory Responses

Lab Code	Sequence of Testing	Test	Item A Item B	Item C
<u>Code</u> 003	1	Macroscopic Exam	A slightly more brittle than B an	nd C
NN	2 3	Microscopic Exam Solubility Tests	No discernible differences betwee 7 reagents used, A different from by HNO3 reaction	
	4 5*	Fluorescent Studies X-ray Fluorescence	No differences between A, B, C Pb/Fe = .31	Ti/Fn = .85
			A different from B	C different from A and B
009 NN	1 2 3	Macroscopic Exam Microscopic Exam Solubility Tests	No differences observed Shiny Dull H <sub>2</sub> SO <sub>4</sub> Darker Brown than B or C	Dull Diphenyl benzidene C-Turns purple A and B do not
	<b>4 5</b>	Pyrolysis G-C X-ray Fluorescence	Does not match B	Does not match B
013 NN	1 2 3 4	Macroscopic Exam Microscopic Exam Solubility Tests TLC		
	5 6 7*	UV Spectrophotometry Pyrolysis G-C IR		
020 NI	1 2 3	Macroscopic Exam Microscopic Exam Pyrolysis G-C	A slightly darker than B or C A, B, C indistinguishable	
046 NN	1)	Macroscopic Exam Microscopic Exam	A, B, C light green slight stria- no striations tions	Multiple layers appears to have been on wood
	3*	Solubility Tests	4 reagents KOH - insol. KOH - sol. HNO3 - insol. HNO3 - sl. sol.	KOH - Sol. KNO3 - sl. sol.
	4* 5*	Infrared Analysis Emission Spectroscopy	slightly different than B No Fe	Similar to B Fe present
050 NN	1 2 3 4	Solubility Tests Macroscopic Exam Microscopic Exam H <sub>2</sub> SO <sub>4</sub>	A, B, C similar A, B, C similar A, B, C similar A, B, C similar Black upon heat- Yellow ing solution	Black upon heating
056 IN	1 2	Macroscopic Exam Microscopic Exam	A similar to B Similar to B	C similar to B Slight difference
	3*	X-ray Fluorescence	Cl:Ti = .46	from B Ti:Zn = .85
	4	Solubility Tests	Zn:Ce = .07 Zn:C1 = .12 A similar to B	C different rate of rx. w/HNO3
061 NN	1 2 3 4* 5*	Macroscopic Exam Microscopic Exam Solubility Tests Infrared Analysis X-ray Fluorescence	A, B, C single-layered A, B, C similar A different from B and C B different from	yellowish green n C

<sup>\*</sup> indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
YY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

Lab Code	Sequence of Testing	<u>Test</u>	<u>Item A</u>	<u>Item B</u>	<u>Item C</u>
068 NN	1 2 3	Macroscopic Exam Microscopic Exam Solubility Tests	A, B, C similar, A, B, C 4 layers A not completely sol. in Alc-KOH	of gold Sol. In.	Sol. In. KOH
	4 5*	Infrared Anaylsis Emission Spectroscopy	A, B, C similar		Zn present in C
072 YN	1 2 3 4*	Macroscopic Exam Microscopic Exam Solubility Tests Emission Spectroscopy			
080 NN	1 2 3 4*	Macroscopic Exam Microscopic Exam Solubility Tests	A, B, C similar A, B, C similar A, B, C differen A, B, C differen		
	5* 6	Pyrolysis G-C X-ray Diffraction TLC	A and B similar A, B, C similar		C different
• 1	7 8	Emission Spectroscopy Fluorescent Studies	A and B similar A, B, C similar		C different
122 NN	1 2 3	Macroscopic Exam Microscopic Exam Solubility Tests	A, B, C green pa A, B, C single la A, B, C similar		
	4* 5*	Pyrolysis G-C X-ray Fluorescence	A different Ti/Zn = .90 Ti/Cu = .63 Ti/Mn = .70	B and C match .54 .42 .60	.4 .31 .52
128 NN	1 2 3 4* 5.	Macroscopic Exam Microscopic Exam Solubility Tests Infrared Analysis X-ray Fluorescence	A, B, C olive ye A, B, C single 1 A, B, C similar A, B, C differen	ayer	
134 NN	1* 2 3 4 5	Macroscopic Exam Microscopic Exam Solubility Tests Fluorescent Studies UV Spectrophotometry	A, B, C different A different A, B, C different A, B, C similar A, B, C differen	B and C similar t	
167 NN	1 2 3 4 5 6*	Macroscopic Exam Microscopic Exam Solubility Tests UV Spectrophotometry Infrared Analysis Emission Spectroscopy	A, B, C similar A, B, C similar A, B, C similar A, B, C have sam A different	e max. B and C similar No Zn	Zn Present
	7* 8	Pyrolysis G-C Thermogravimetric Analysis	A different A, B, C similar	B and C similar	
170 NY	1 2 3 4 5*	Macroscopic Exam Microscopic Exam UV Light Solubility Tests Pyrolysis G-C	A different	B and C are the	same
178 NN	1 2 3 4* 5*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Emission Spectroscopy	Ti, Fe, Si, Al, 1	in color B and C similar C slightly diffe	C conta( ( )
	6	X-ray Fluorescence	and B A similar to B		elements or A and B + 2n C similar to B
	· -	and the second s			but with Zn

<sup>\*</sup> indicates the point where a conclusion was reached

NN		Indicates	No for A and No for C
NY			No for A and Yes for C
YN		Indicates	Yes for A and No for C
YY		Indicates	Yes for A and Yes for C
IN	6.5	Indicates	Inconclusive for A and No for C
NI		Indicates	No for A and Inconclusive for C

Lab <u>Code</u>	Sequence of Testing	<u>Test</u>	Item A
194 NN	1 2	Microscopic Exam Solubility Tests	A, B, C yellowgreen
	3 4*	Infrared Analysis Density Studies	Inconclusive Bromoform-Benzene 6.0-4.0 5.5-4.5 5.0-5.0 A, B, C different
199 NN	1 2 3	Macroscopic Exam Microscopic Exam X-ray Fluorescence	A, B, C single layer, mustard color Ti/Pb = 13.65
	4*	Pyrolysis G-C	A and B different
212	1 .	Macroscopic Exam	Brittle flake Pliable Flake Pliable Flake A, B, C gold color
	2 3 4 5* 6*	Microscopic Exam Fluorescent Studies Solubility Tests Emission Spectroscopy Density Studies	A, B, C similar A, B, C similar A, B, C similar A, B, C similar A and B similar A and B different B and C similar
228 NN	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Emission Spectroscopy	A, B, C similar A possibly different from B and C A, B, C similar A different from B B different from C
247 NN	1 2 3 4* 5	Macroscopic Exam Microscopic Exam Solubility Tests Infrared Analysis Pyrolysis G-C Emission Spectroscopy	A, B, C similar A, B, C similar A, B, C similar A, B, C different A different from B A and B similar Mg, Ti, Si, Mn, C also contains Zn Pb, Fe, Ca, Ae C different
251	1	Macroscopic Exam	Fine splintered B and C large regular piece chips
NY	2 3 4 5 6 7	Microscopic Exam Micro chemical Solubility Test TLC UV Spectrophotometry Infrared Analysis	A = B = C A ≠ B = C A ≠ B = C A = B = C A = B = C A ≠ B = C
254 NN	1 2 3 4*	Macroscopic Exam Microscopic Exam Solubility Tests Emission Spectroscopy	
266 NY	1 2 3	Microscopic Exam Solubility Tests Pyrolysis G-C	A, B, C dissimilar A, B, C similar A different B and C similar
272 NN	1 2 3 4* 5*	Macroscopic Exam Microscopic Exam Solubility Tests Fluorescent Studies Pyrolysis G-C	A, B, C similar A, B, C similar Inconclusive Absorbs Absorbs Yellow Fluores- A different from B and C cence
278 NN	1 2 3 4	Macroscopic Exam Microscopic Exam Solubility Tests Emission Spectroscopy	A, B, C similar A, B, C similar A, B, C similar A, B, C different

<sup>\*</sup> indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
ΥY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

Lab Code	Sequence of Testing	Test	Item A	Item B	Item C
289 NN	1 2 3 4* 5	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Emission Spectroscopy	A, B, C similar A, B, C similar A, B, C Similar A, B, C differen A and B similar		C different
297 NN	1 2 3* 4 5	Microscopic Exam Pyrolysis G-C X-ray Diffraction Infrared Analysis Emission Spectroscopy	A, B, C similar Acrylic TiO <sub>2</sub> Acrylic A and B contain	Alkyd TiO2 Alkyd Mn,Pb,Mg,Si, Fe,Al,Ca,Ti	Alkyd Ti02/and ZnO Alkyd C also contains Zn
301		Unable to complete test			
305		Unable to complete test			
308 NN	1 2 3*	Macroscopic Exam Microscopic Exam X-ray Fluorescence	Inconclusive Inconclusive A and B trace Zn		C - Zn major element
317 NN	1 2 3 4*	Macroscopic Exam Microscopic Exam Fluorescent Studies Emission Spectroscopy			C different in Zn content
	5* 6	Pyrolysis G-C Solubility Tests	A different from A different from		ar contains
330 NN	1 2 3* 4 5 6*	Macroscopic Exam Microscopic Exam Pyrolysis G-C Solubility Tests Infrared Analysis X-ray Fluorescence			
333 YY	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Fluorescent Studies Solubility Tests Infrared Analysis		rbed on between A, B, on between A, B,	
344 NN	1 2 3 4 5* 6	Macroscopic Exam Fluorescent Studies Microscopic Exam Solubility Tests Density Studies Emission Spectroscopy	A, B, C contain A and B dark pur A, B, C similar Very soluble Level-bottom A and B have no	Least Soluble Level-middle	No fluorescence Soluble Level-Top Zn present in C
345 NN	1 2 3 4* 5. 6*	Macroscopic Exam Microscopic Solubility Tests Infrared Analysis TLC Pyrolysis G-C	A, B, C similar A, B, C similar A, B, C similar A, B, C all have A, B, C similar A ≠ B	differences; def	inîtely C≠B
347 NN	1 2 3 4 5 6	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Infrared Analysis Fluorescent Studies TLC	A, B, C similar A, B, C similar A, B, C similar A, B, C all diff A, B, C differen A, B, C similar A, B, C similar		

 $\ensuremath{\star}$  indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NÝ	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
YY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NT	Indicates No for A and Inconclusive for C

	Lab Code	uence Testing	Test	Item A	Item B	Item C
	357 NN	1 2 3 4 5 6 7	Macroscopic Exam Microscopic Exam Polarizing Microscope Solubility Tests Infrared Analysis X-ray Diffraction Emission Spectroscopy	A, B, C similar TiO2 A and B contain	TiO2 Mn, Mg, Si, Ti, Co, Al, Ca, Fe, Pb, Cu	TiO <sub>2</sub> and ZnO C contain Zn in addition
		8*	Pyrolysis G-C	A, B, C different		
	370 NN	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C X-ray Fluorescence	A, B, C similar A, B, C similar A different A different	B and C similar B and C similar B contains more 7 C also contains	
	375 NN	1 2 3* 4	Macroscopic Exam Microscopic Exam Solubility Tests Infrared Analysis	A, B, C similar A, B, C similar A, B, C different A different from		B and C distinquishable
	386 NN	1 2 3* 4*		A = B = C A = B = C A different	B and C different	B≂C
	395 NY	1 2 3 4* 5*	Macroscopic Exam Microscopic Exam Solubility Tests Infrared Analysis Pyrolysis G-C	A, B, C similar A different A different	B and C similar B and C similar	
	399 NN	1 2 3* 4 5 6 7	Microscopic Exam Pyrolysis G-C Solubility Tests Infrared Analysis	A, B, C similar A, B, C similar Gross differences B different from A, B, C different A, B, C different A, B, C no fluore	A or C	
	401 IN	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Solubility Tests Infrared Analysis Emission Spectroscopy	A, B, C lemon yel A, B, C similar Inconclusive A, B, C similar A and B inconclus		C different
	409 NN	1 2 3* 4 5*	Macroscopic Exam Microscopic Exam Pyrolysis G-C Infrared Analysis X-ray Fluorescence	A, B, C yellow-gr A, B, C single la A different A, B, C alkyd ena Trace Cu No Zn	reen yer B and C similar mel, TiO2 present No Cu Trace Zn	No Cu High conc. Zn
	415 NN	1 2 3*	Macroscopic Exam Solubility Tests EDAX	A, B, C similar A, B, C similar A and B similar	D and Coimile	High Zn concentration
	120	4*	Pyrolysis G-C	A different	B and C similar	
-	420 NN	1 2 3 4 5	Macroscopic Exam Microscopic Exam Fluorescent Studies X-ray Diffraction Solubility Tests	A, B, C similar A, B, C similar A and B do not fl Slight difference A different from	e in A and B	C fluoresces

 $<sup>\</sup>star$  indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN.	Indicates Yes for A and No for C
YY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NT	Indicator No for A and Inconclusive for C

Lab	Sequence			
Code	of Testing	Test	Item A Item B	Item C
430 YN	1 2 3* 4* 5 6	Macroscopic Exam Microscopic Exam Fluorescent Studies Solubility Tests Pyrolysis G-C Infrared Analysis	Inconclusive A, B, C similar Absorbs Absorbs	Yellow
448 NN	1 2 3* 4*	Macroscopic Exam Microscopic Exam Solubility Tests Emission Spectroscopy	A, B, C similar A, B, C similar A different from B A and B contain Mu, Mg, Al, Si, Ti, Ca	C also contains Zn
	5 6	Infrared Analysis Fluorescent Studies	A, B, C similar No fluorescence in A, B, C	
457 NN	1* 2 3 4 5*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Emission Spectroscopy	A more brittle than B or C A slightly different than B or C A, B, C similar A different from B or C A and B contain Mg, Al, Fe, Si	C also
	6	Scanning Electron Microscope	Pb, Mn, Ti A and B similar	contains Zn C contains Zn not in A, B
461 II				
475 NN	1 2 3 4* 5* 6	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C X-ray Diffraction Emission Spectroscopy	B slightly different than A and C A, B, C similar A, B, C similar A different B and C similar A and B similar A and B contain Al, Ca, Mg, Pb, Si, Ti	C different C also contains Zn
496 NI	1 2 3 4 5 6	Macroscopic Exam Microscopic Exam Fluorescent Studies Solubility Tests Pyrolysis G-C Density Studies	A and B slightly soluble in HNO3	
504 YY	1 2 3*	Macroscopic Exam Microscopic Exam Solubility Tests	A, B, C same A, B, C same A, B, C same - used H2SO4, NH4ON, Ethyl Acetate Ketone	CHCR3, Dioxane , Methyl Ethyl
507 NN	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Energy Dispersive Spectroscopy	A, B, C pea-green enamel A, B, C similar A, B, C similar  Zn, Al, Si, S Mg, Al, Si, Ti Major, Fe Ti Major, Fe	Mg, Si, Ti Major, Fe
513 NN	1 2	Microscopic Exam Fluorescent Studies	A and B have no fluorescence	C fluoresces
	3	X-ray Fluorescence	A and B similar	slightly C different
	<b>4 5</b>	Infrared Analysis Pyrolysis G-C	A and B similar A and B different	in Zn
523 NN	1 2 3 4 5	Microscopic Exam Solubility Tests Infrared Analysis Pyrolysis G-C Emission Spectroscopy	A, B, C indistinguishable A, B, C similar Small difference between A and B A different A and B contain Ti, Si, Mg, Mn, Pb, Cu	B and C similar B and C similar C also contains Zn

<sup>\*</sup> indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
YY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

Lab Sequence of Testing Test Item A Item B  545 1 Macroscopic Exam Fine thin chips One irregular chip Yellow-green Yellow-green A and B one layer, yellow-green, smooth surfaces  3* Fluorescent Studies A and B different from C Some differences in A, B, C Similar A and B contain Si, Mg, Mn, Ti, Pb  7 Infrared Analysis A and B similar A and B same	Item C Small thin Yellow-green Same as A and B except has pro- minent ridges C also contains Zn
Chip Yellow-green Yellow-green Yellow-green A and B one layer, yellow-green, smooth surfaces  3* Fluorescent Studies A and B different from C Some differences in A, B, C A, B, C similar Emission Spectroscopy A and B contain Si, Mg, Mn, Ti, Pb Infrared Analysis A and B similar	Yellow-green Same as A and B except has pro- minent ridges C also contains
Yellow-green Yellow-green A and B one layer, yellow-green, smooth surfaces  3* Fluorescent Studies A and B different from C Solubility Tests Some differences in A, B, C A, B, C similar Emission Spectroscopy A and B contain Si, Mg, Mn, Ti, Pb Infrared Analysis A and B similar	Same as A and B except has prominent ridges  C also contains
4 Solubility Tests Some differences in A, B, C 5 TLC A, B, C similar 6 Emission Spectroscopy A and B contain Si, Mg, Mn, Ti, Pb 7 Infrared Analysis A and B similar	C also contains
564 1 Macroscopic Exam  NN 2 Microscopic Exam 3 Solubility Tests 4 Fluorescent Studies 5* X-ray Fluorescence Definite differences in Zn conte	n <b>t</b>
584 1 Macroscopic Exam A, B, C similar, A slightly differences A, B, C similar A, B, C similar A, B, C similar A, B, C on differences A, B, C on differences A, B, C on differences A different B and C similar A and B similar	erent C different
594 1 Macroscopic Exam A, B, C yellow-green paint NN 2 Microscopic Exam A, B, C 1-layer 3 Solubility Tests	
4* Pyrolysis G-C A different from B	C different from B in quantity
597 1 Macroscopic Exam B flat, A and C shiny NN 2 Microscopic Exam B flat, A and C shiny 3* Solubility Tests B different from A and C w.r.t. 4 Infrared Analysis Inconclusive 5 TLC Inconclusive 6 Emission Spectroscopy B different from A and C	color eluted
599 1 Fluorescent Studies A, B, C negative YN 2 Macroscopic Exam A, B, C similar 3 Microscopic Exam A, B, C single layer 4 Solubility Tests A and B different from C in acet. 5 Emission Spectroscopy A and B contain Pb, Mg, Al, Fe	one C also contains Zn
608 1 Macroscopic Exam A, B, C similar in color NN 2 Microscopic Exam No differences noted 3* Solubility Tests A different B and C slight! 4 Pyrolysis G-C	y different
5 Emission Spectroscopy A and B contain no Zn 6 Infrared Analysis A, B, C similar	C containIn
NN 2 Microscopic Exam A, B, C similar NN 2 Microscopic Exam A, B, C non metallic, similar A, B, C non metallic, similar A, B, C have no fluorescence A, B, C similar A, B, C similar A, B, C different A, B, C different A, B, C different A, B, C different	
632 1 Macroscopic Exam A, B, C similar NN 2* X-ray Fluorescence Ti/Pb = 4.4 Ti/Pb = 7.5	C contains more Zn than A or B
3 Solubility Tests A and B similar 4 Infrared Analysis A and B similar	

<sup>\*</sup> indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
YY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

Lab	Sequence				•
<u>Code</u>	of Testing	Test	Item A	Item B	<u>Item C</u>
637 NN	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Fluorescent Studies Solubility Tests Pyrolysis G-C	A, B, C simi A, B, C simi A, B, C same A, B, C simi A different	lar lar	C slightly
	6	X-ray Diffraction	A, B, C simi	Tar	different from B
646 NI	1 2* 3*	Miscroscopic Exam Solubility Tests Pyrolysis G-C	A, B, C simi A different A different	lar B and C simil C slightly di	
648 NN	1 2 3* 4	Macroscopic Exam Microscopic Exam Solubility Tests Fluorescent Studies	A and B diff	erent from C	C different from A, B
	<b>5</b> 6	Emission Spectroscopy Infrared Analysis	A and B diff A different		(10111 7)
652 NN	1 2 3* 4* 5 6* 7	Macroscopic Exam Microscopic Exam Fluorescent Studies Infrared Analysis X-ray Diffraction Pyrolysis G-C Solubility Tests Emission Spectroscopy	A, B, C simi A, B, C simi A and B simi A, B, C diff TiO2 A, B, C diff A, B, C simi A and B simi	lar lar erent TiO2 erent lar	C dissimilar TiO <sub>2</sub> & ZnO C has conc. of
		Emission operation	71 dina 5 5 min		Zn
655 NI	1 2 3 4 5	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C TLC	A, B, C indi A, B, C simi A different A, B, C simi	B and C simil	ar
675 YY	1 2*	Macroscopic Exam Solubility Tests	A, B, C same A, B, C same		
677	1 .	Macroscopic Exam	Fractures in	splinters	B and C not in splinters
NY	2 3* 4 5	Microscopic Exam Solubility Tests TLC Infrared Analysis	A, B, C simi A insol. H <sub>2</sub> S A, B, C simi A, B, C modi	004 B and C sol. lar	•
700 YN	1 2	Macroscopic Exam Fluorescent Studies	A and B dark	: Aks.	C- weak yellow fluorescence
	3	Solubility Tests	A and B simi	lar	C slightly different
	4*	Emission Spectroscopy	A and B cont	ain Ti, Mg, Si, Fe, Pb, Al	C contains Zn also
702	1	Macroscopic Exam	A, B, C yell flexible	ow - A long, thin	fragments, B and C
	2 3 4* 5 6	Microscopic Exam Solubility Tests Infrared Analysis TLC UV Spectrophotometry	A, B, C sing A, B, C simi A ≠ B = C A = B = C A ≠ B = C		C wrinkled surface
706 YN	1 2* 3	Macroscopic Exam Microscopic Exam Solubility Tests	A, B, C same A and B same A and B same	t <sub>ar</sub> in the second of the	C different C different

<sup>\*</sup> indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
YY	INdicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

Lab <u>Code</u>	Sequence of Testing	<u>Test</u>	Item A Item B	<u>Item C</u>
711 NY	1 2 3	Microscopic Exam Solubility Tests TLC	A, B, C gold color A, B, C similar	
717	4* 1	Pyrolysis G-C	A different from B and C	
717 NY	2 3 4*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C		
719 NY	1 2 3* 4	Microscopic Exam Solubility Tests Pyrolysis G-C Infrared Analysis	A, B, C similar A different from B and C	
730 NI	1 2 3 4* 5	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C Infrared Analysis	inconclusive on B and C	
747 IN	1 2 3 4 5*	Macroscopic Exam Microscopic Exam Fluorescent Studies Solubility Tests X-ray Fluorescence	A, B, C same color A, B, C same color, layer structu A, B, C have no fluorescence No significant differences Low - Zn Low - Zn High - Ti High - Ti	ure High - Zn Low - Ti
	6	GC of Binder Extract	Minor differences between A and E	
761 YN	1 2*	Macroscopic Exam Fluorescent Studies	A and B gold A and B no fluorescence	C greenish-gold C slight fluores- cence
	3 4 5* 6	Microscopic Exam Solubility Tests Infrared Analysis TLC	A and B single layer enamel A and B same A and B same A and B match	cence
763 NN	1 2 3*	Macroscopic Exams Microscopic Exam Solubility Tests	A, B, C green A, B, C green, single layer A different B and C same \$2NH in H2SO4	
	4* 5 6 7	X-ray Diffraction Emission Spectroscopy Pyrolysis G-C Infrared Analysis	A and B similar Ti Ti A different B and C similar A, B, C similar	C different Ti & Zn
768 YN	2 3 4* 5 6 7	Macroscopic Exam Microscopic Exam Solubility Tests Emission Spectroscopy Infrared Analysis UV Spectrophotometry Pyrolysis G-C	A, B, C similar A, B, C similar A, B, C similar A and B same A, B, C similar A, B, C similar Inconclusive	C different
775 YN	1 2*	Microscopic Exam X-ray Fluorescence	No differences noted A and B contain Ti, Pb, Fe, Zn, Mn	C contains Zn,
	3 4 5	Solubility Tests Infrared Analysis X-ray Diffraction	No changes in H <sub>2</sub> SO <sub>4</sub> , HNO <sub>3</sub> for A, A and B same A and B rutile	Ti, Fe, Pb B, C
793	a 7.	Microscopic Exam	A and B single layer, yellow- green	C two layers, yellow-green
NN	2 3*	Infrared Analysis Pyrolysis G-C	A, B, C different A, B, C different	30110M-9166H

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NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
ΥY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

Lab <u>Code</u>	Sequence of Testing	Test	Item A Item B	Item C
803	1	Microscopic Exam	A, B, C yellow green; B not gloss	sy, A and C
NN .	2 3*	Fluorescence Pyrolysis G-C	glossy A, B, C no fluorescence A different from B	B and C same Contains Zn
	4	X-ray Fluorescence	A and B same B and C differen	
836 NN	1 2 3 4 5* 6 7*	Macroscopic Exam Microscopic Exam Solubility Tests TLC X-ray Fluorescence Infrared Analysis Pyrolysis G-C	A, B, C yellow paint, glossy find A, B, C single layer A, B, C react the same A, B, C same Zn/Pb = .053 Zn/Pb = .232 A, B, C similar A different B and C same	Ish Zn/Pb = 14.2
849 NY	1 2 3 4* 5 6 7	Macroscopic Exam Microscopic Exam Solubility Tests Visible Spectrophotometry TLC Infrared Analysis Pyrolysis G-C	A different flaking pattern from A, B, C similar	B and C B and C similar
855 NN	1 2 3* 4*	Macroscopic Exam Microscopic Exam Solubility Tests Emission Spectrophotometry	A, B, C similar A, B, C similar A different from B in alc - KOH B - no Zn	C and B similar C - contains Zn
857 NN	1 2 3* 4	Macroscopic Exam Microscopic Exam Infrared Analysis Solubility Tests	A, B, C similar A and B pliable A, B, C all different	C brittle
872 NN	1 2 3* 4 5*	Infrared Analysis Emission Spectroscopy	A, B, C similar in color A, B, C similar A different from B and C A different from B Mg, Pb, Si, Mg, Pb, Mn, Si, Al, Cu, Fe, Al, Ti, Ca	B similar to C Mg, Pb, Mn, Si, Fe, Al, Cu, Fn, Na, Ti, Ca
877 YN	1 2 3 4*	Microscopic Exam Pyrolysis G-C	A, B, C similar in color A, B, C similar in layer number a A, B, C consistent A and B - no fluorescence	C - yellow
	5 6*	Solubility Tests Infrared Analysis	A and B similar A and B similar	fluorescence
895 NN	1 2 3	Macroscopic Exam Microscopic Exam X-ray Fluorescence	A, B, C yellow paint A, B, C yellow paint High in Zn Low in Zn	Contains more Zn than A
902 II	1 2	Infrared Analysis Pyrolysis G-C	A = B = C Insufficient sample for conclusion	on .
910 NY	1 2 3* 4 5*	Macroscopic Exam Microscopic Exam Solubility Tests Fluorescent Studies Pyrolysis G-C	A, B, C same appearance A - smooth surface B and C - 1 A different from B No fluorescence in A, B, C A different from B	rough surface B and C same B and C same

#### \* indicates the point where a conclusion was reached

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
YY	Indicates Yes for A and Yes for C
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NI	Indicates No for A and Inconclusive for C

Lab Code	Sequence of Testing	<u>Test</u>	Item A Item B	Item C
917 NN	1 2 3 4*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C	No significant differences No significant differences No significant differences A different from B	Slight differ- ence in 8 & C
	5* 6 7	Emission Spectroscopy X-ray Diffraction Visible Spectrophotometry	A and B TiO <sub>2</sub> A, B, C similar	C TiO <sub>2</sub> and ZnO
927 NN	1 2 3 4* 5*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C X-ray Fluorescence	A, B, C green A, B, C single layer A, B, C similar A different from B A and B - Ti, Pb, Fe, Ti/Zn > 1 Zn (*race)	B and C similar C - Ti, Fe, Pb Zn (strong) Ti/Zn < 1
928 NN	1 2 3* 4* 5	Macroscopic Exam Microscopic Exam Pyrolysis G-C X-ray Diffraction Solubility Tests	A, B, C similar color A, B, C similar color A different from B TiO <sub>2</sub> TiO <sub>2</sub> A, B, C similar	B similar to C TiO <sub>2</sub> + ZnO
936 NN	1 2* 3 4*	Microscopic Exam Infrared Analysis Pyrolysis G-C X-ray Fluorescence	A, B, C similar A different from B A different from B	B and C same B and C same B and C different
942 YN	1 2 3 4	Macroscopic Exam Fluorescent Studies Microscopic Exam Infrared Analysis	A, B, C similar A and B no fluorescence A and B similar A and B same	C fluorescent
967 NN	1 2 3* 4* 5	Macroscopic Exam Solubility Tests Pyrolysis G-C Emission Spectroscopy X-ray Diffraction	A, B, C similar A and C compare to B A different from B A and B similar (No Zn) A different from B	B and C compare C - Zn present B and C similar
972 NN	1 2 3 4*	Macroscopic Exam Microscopic Exam Solubility Tests Pyrolysis G-C	A, B, C yellow paint A, B, C shiny surface A, B, C similar A grossly different from B	B and C different

NN	Indicates No for A and No for C
NY	Indicates No for A and Yes for C
YN	Indicates Yes for A and No for C
ΥY	Indicates Yes for A and Yes for C
IN	Indicates Inconclusive for A and No for C
NI	Indicates No for A and Inconclusive for C

<sup>\*</sup> indicates the point where a conclusion was reached



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