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**SPECIAL RE-REVIEW
FINAL DETERMINATION
DECLASSIFICATION CONFIRMED**

BY 2C Borden DATE 6-12-79
BY W.A. Snyder DATE 11-1-79

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October 23, 1947

R. E. Curtis
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ANALYTICAL REQUIREMENTS OF THE 234-5 PROJECT

NOT UCN1

Classification Cancelled and Changed To

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By Authority of RM Pac

May 1973

By J.H. Wells 11-21-94

Verified By J.E. Savely 11-23-94

At this time it is desirable that preliminary estimates be made of the man-power, equipment, space, layout and facility requirements of the analytical section for the 234-5 Project. To arrive at these requirements a rough estimate of the required analyses, their scope and their frequency has been compiled. This is expected to serve as a basis for the Laboratories Division to outline their space and equipment requirements as soon as possible and to assemble the required personnel for operation later.

On this project an unusual burden will be placed on spectrographic type analyses. To acquaint all concerned with the scope desired of these analyses, the first part of this letter is devoted to outlining supposedly crucial elements, to listing typical element concentrations at various points in the process, and to outlining several types of analyses with the estimated sensitivity.

Crucial elements encountered in the process are classed as light impurities or as metallurgical impurities. The light elements are undesirable due to the α , n reaction with plutonium. The metallurgical impurities are undesirable since they change the physical properties of plutonium. In Table I are listed the light elements undesirable in the finished product and the approximate tolerances (in ppm of plutonium) acceptable for each, assuming all other elements listed are absent. Included in Table II is a list of the undesirable metallurgical impurities with the tolerances (again expressed in ppm of plutonium) for each element, assuming the other elements are absent.

When plutonium was first shipped from Hanford to Los Alamos, the consumer listed the tolerance limits for impurities which could not be exceeded in the Hanford Sample Can. These are given in Table III. Analyses were run on many incoming Sample Cans at Los Alamos during the first shipments. The results of these analyses are listed in Table IV and are the averages of the numbers of analyses also listed.

At this time, information is very incomplete regarding the impurities present at various points in the existing Los Alamos process but some analyses are available on two early runs and have been tabulated in Table V. Impurities are expressed in ppm when the plutonium exists as an oxalate, as a fluoride and as the metal.

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To obtain analyses of the above impurities, at least three methods have been used, the direct copper spark method, the cupferron procedure and the pyroelectric procedure. The elements detected and the limits of sensitivity for each method are listed in Tables VI, VII, and VIII.

A close survey of Tables I through VIII will indicate the element impurities of concern in the process, approximate concentrations at several points in the process, methods by which the desired sensitivity can be obtained and the relative importance of each analysis as assigned at Los Alamos.

In Table IX are listed all of the samples now anticipated for control of the 234-5 Process, their sources, analyses desired, potential analytical procedures, major impurities anticipated, and maximum and anticipated number of samples per month. This analytical program may be increased somewhat by the necessity for following initial production runs more closely and by the demands of the development laboratory.

TABLE I - Undesirable Light Elements in Finished Product and Their Tolerances

Element	Tolerance (ppm of Pu)
Li	120
Be	4.3
B	17
C	2,500
O	5,100
F	52
Na	330
Mg	320
Al	910
Si	4,700
S	250,000
Ca	100,000

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TABLE II - Undesirable Metallurgical Impurities in Finished Product and Their Tolerances

Element	Tolerance (ppm of Pu)
Al	40
Si	100
Ga	100*
In	350
Tl	600

*Exclusive of any added

TABLE III - Tolerance limits for Impurities in the
Hanford Sample Can (1, 2, 3, 4)

<u>Element</u>	<u>Tolerance</u>
Li	1.0%
C	Footnote (3) below
N	1.0% (other than NO_3^-)
O	1.0% (as peroxide)
F	0.01%
Na	5.0%
Mg	1.0%
Al	1.0%
Si	0.005%
P	1.0% (as H_3PO_4)
S	1.0% (as H_2SO_4)
Cl	5.0%
K	5.0%
Ca	1.0%
Fe	2.0%
Zn	0.5%
Br	5.0%
Rb	1.0%
Sr	1.0%
Zr	0.5%
Cb	0.01%
Sn	0.01%
I	0.01%
Cs	1.0%
Ba	1.0%
La	5.0%
Hf	0.5%
Ta	0.01%
Th	0.5%
U	0.5%
Np	0.001%

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Notes:

- (1) 160 gm/liter plutonium concentration between 1 and 2 M in nitric acid concentration.
- (2) To contain less than 1 mc of beta and 1 mc of gamma radiation per gram of plutonium.
- (3) Each shipment to contain less than 10 mg. of suspended solids.
- (4) Minimum plutonium concentration 95% by weight exclusive of H^+ , NO_3^- and H_2O . Other 5% can be made up of any other elements in the periodic table if they do not exceed the values listed in the table and if they do not precipitate alone or with other elements.

TABLE IV - Average Analyses of Inlet Plutonium (1)

<u>Element</u>	<u>Concentration (ppm)</u>	<u>Number Analyses Performed</u>
Li	< 3	29
Be	< 0.4	29
B	2	18
Na	320	29
Mg	200	183
Al	100	32
Si	1,500	22
P	1,470	46
SO ₄ ⁻	24 (2)	26
K	900	28
Ca	200	168
Sc	< 600	21
Cr	5,000	182
Mn	200	29
Fe	9,400	182
Co	< 15	29
Ni	3,000	180
Cu	80	19
Zn	200	26
As	< 4,000	163
Sr	4	29
Zr	< 1,000	164
Ag	10	6
Cd	1,600	39
In	< 30	15
Sn	2,000	36
Sb	< 100	21
Bi	< 13	29
Pb	32,000	183
Ce	< 50	29
Hg	< 50	19
Th	< 150	15
Pb	130	29
Bi	800	17
Th	< 4,000	158
U	100	24
Solids	740	23

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Notes:

1. Based on 184 Sample Cans
2. SO₄⁻ expressed as percent by weight of plutonium.

TABLE V - Impurities Found at Three Points on
Two Early Los Alamos Runs

Element	Oxalate (ppm)	Fluoride (ppm)	Metal (ppm)
Li	< 0.46	-	< 0.8
B	< 0.46	< 0.27	< 0.09
P	< 0.3	-	0.4
Na	170	-	17
Mg	64	< 5	2
Al	23	~ 3,900	< 5
Si	325	-	~ 300
P	< 44	-	-
K	< 42	-	< 34
Ca	340	< 62	< 11
V	< 150	-	< 30
Cr	< 4.6	< 9	< 2
Mn	< 4.6	< 5	< 2
Fe	212	-	124
Co	< 4.6	< 17	< 9
Ni	< 46	65	< 7
Cu	< 30	-	< 60
Zn	-	< 55	170
As	44	9	-
Sr	24	< 2	1.6
Cd	< 46	< 27	< 9
Pb	< 30	-	-
Sn	175	< 27	< 45
Sb	< 150	-	< 60
Bi	41	27	< 2
La	~ 810	~ 2,955	~ 830
Co	< 46	< 44	125
Hg	46	< 130	< 17
Pb	< 30	< 33	< 9
Ba	< 100	-	< 100
Th	< 150	-	-
U	< 130	-	15

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TABLE VI - The Detectable Elements and the Sensitivity of the Direct Copper Spark Procedure of Spectrographic Analysis

Element	Sensitivity (ppm)
Li	200
B	10
Be	200
Mg	20
Al	200
Si	2,000
Ca	20
V	1,000
Cr	400
Mn	400
Co	1,000
Ni	1,000
Zn	2,000
Sb	20
As	1,000
Se	1,000
Br	1,000
I	20
La	1,000
Co	2,000
Ru	200
Re	2,000
Pb	2,000
Bi	1,000
Tl	2,000
Cd	2,000

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TABLE VII - The Detectable Elements and The Sensitivity of The Cupferron Procedure of Spectrographic Analysis

<u>Element</u>	<u>Sensitivity (ppm)</u>
Li	2
Be	0.2
Na	2
Hg	2
Al	2
K	20
Ca	10
Gr	2
Mn	2
Os	10
Fe	10
Si	2
Co	5
Ba	2
La	2
Ce	20
Pb	10

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TABLE VIII - The Detectable Elements and The Sensitivity of The Pyroelectric Procedure of Spectrographic Analysis

<u>Element</u>	<u>Sensitivity (ppm)</u>
Si	8
P	300
Cu	30
Ge	8
Ag	< 6
Te	16
Sn	15
Sb	100
Tl	30

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TABLE IX - Anticipated 234-f Process Control Analyses

Source	Analyses Desired	Potential Procedure (see footnotes)	Major Impurities	Samples per Month	
				Maximum Possible	Antici- pated
PROCESS STREAMS					
Diluted Sample Can Solution	Pu	1, 2		175	175
	Solids	4, 5	Si, P, Cr, Fe, Zn, Sn	175	10
	Soluble Impurities	3	La, Pb, Cr, Ni, Sn, Si, Na, Mg, B, Al, U, Zr, Co	175	175
	P, Pb, B	5		175	175
	U	6		175	175
	S	13		175	175
Purification Supernates	Pu	2		250	22
	Fe	7		22	22
	Al	8		22	22
	Impurities	3	La, Si, Co, Sn, Na, Mg	170	13
Condensate from Dry Chemistry	Pu	2		22	13
Plutonium Oxide	Pu	2		22	13
	Impurities	3		22	13
Plutonium Fluoride	Pu	2		22	13
	Impurities	3		22	13
Reduction Bomb Wash	Pu	2		22	22
Buttons Cleaning Soln.	Pu	2		22	13
Plutonium Buttons	Impurities	3	La, Si, Zn, Co, Fe, Na, B	60	60
Finished Plutonium	B, Ga, S, O, O	4		15	15
	Si	3a		15	15
	Impurities	5b	Al, Be, Cu, Li Mg, Na	15	15
Final Piece Cleaning Solution	Pu	2		22	13
Recovery Supernates	Pu	2		22	22
Recovery Concentrates	Pu	2		22	22
Recovery Solutions	Pu	2		75	75
	B	8		75	22

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TABLE IX (Continued)

Source	Analyses Desired	Potential Procedure (see footnotes)	Major Impurities	Samples per Month	
				Maximum Possible	Antici- pated
Recovery Recycle	Pu	1		15	15
	Impurities	3 ③		15	15
Sample Can Washes	Pu	2		22	22
PROCESS REAGENTS					
HI	Molarity	7		22	11
Oxalic Acid	Molarity	4		22	11
Oxalic-Nitric Wash	Molarity	4, 8		22	11
Nitric Acid	Molarity	8		44	22
Thiosulfate	Molarity	7		22	11
HCl	Molarity	8 ③		22	11
Glycol Sol. tion	Molarity	12		22	21
Phosphoric Acid Soln.	Molarity			22	11
KOH	Molarity	8		22	11
NaOH	Molarity	8		22	11
ESSENTIAL MATERIALS					
HI	Impurities	5		10	3
	Sp. Gr. % Free I ₂	4, 7		10	3
Oxalic Acid	Impurities	3		10	3
Nitric Acid	Impurities	3		10	2
Cylinder O ₂				22	11
Cylinder HF	H ₂ O, organic material			22	11
Calcium	3	3		10	2
	5	20		10	3
	Impurities	3	Mn, Al, Si, Mg Be, Ni, Fe, Co, Ag, Pb	10	2
Iodine	Organic Matter			10	3
	Impurities	3		10	2
Magnesium	Impurities	4	Si, Fe, Al, Ca, B	10	2
Argon	O ₂ , H ₂ , N ₂			22	11
HCl	Molarity	8		10	3
Witchhazel	Impurities	3	B, Mg, Al, Si, Ca, Cr, Mn, Fe, N, Cu, Zn, Ag, In, Sn, Hg, Pb	5	2
Holium	O ₂ , H ₂			22	11
Ni(CO) ₄				22	11
Phosphoric Acid	Molarity			10	3
Bromobenzene	Density	12		22	11
KOH	Cl			10	2
	Impurities	3		10	3
NaOH	Purity			10	2

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MM:dg

1. Potentiometric
2. Radio assay
3. Spectrophotophic
- a. Copper spark
- b. Cupferron
- c. Pyroelectro
4. Gravimetric
5. Colorimetric
6. Fluorimetric
7. Oxidation - reduction
8. Neutralization
9. Methanol distillation
10. Kjeldahl
11. H₂S liberation
12. Density

Notes:

TABLE IX (Continued)