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1. O.H. [redacted] - K. Woods
2. R.B. Richards
3. F.W. Albaugh
4. R.S. Bell
5. O.F. Hill - R.H. Reas
6. K.M. Harmon
7. V.R. Cooper
8. P.E. Collins - R.E. Isaacson -
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From: P.E. Collins - Leader
 234-5 Building, Process Assistance
 Separations Technology Unit
 Technical Section
 by R.E. Isaacson - Engineer *R.E.I.*

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Rocky Flats C.G.
J.E. Savely
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PURITY DATA ON REDOX START-UP MATERIAL
THROUGH THE 231 AND 234-5 BUILDINGS

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DECLASSIFIEDINTRODUCTION

Redox effluent for production was first processed in the 231 Building on February 4, 1952. Samples of process streams were taken after each purification step to evaluate the adequacy of purification processes in the 231 and 234-5 Buildings and to provide close control of this material.

Data regarding pile residence histories, radiation surveys of "Sample Cans", reduction yields, C/Q values, and product purity after purification steps were accumulated and are presented in this report to establish preliminary data which may be used to evaluate future process changes.

SUMMARY

1. Purity of the product obtained from Redox effluent proved adequate to meet specifications (See Tables V and VI).
2. Purification factors for the major impurities are given below to indicate the magnitude of purification achieved. (Purification factors less than 2 are omitted inasmuch as the spectro-chemical analyses are considered to be accurate within $\pm 2X$)

<u>Impurity</u>	<u>Purification Factor* for</u>		
	<u>2 Peroxide Precipitations</u>	<u>1 Oxalate Precipitation</u>	<u>Reduction of PuF₄ to Metal</u>
Al	150	--	3
Ba	15	--	10
Bi	--	4	--
Ca	7	--	3
Cr	10	35	--
Fe	10	10	--
La	20	--	--
Mn	--	10	--
Na	NaOH added	45	40
Ni	5	3	--
Pb	3	5	3
Sb	--	3	--
Zn	3	--	--

*Based on Table V

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3. The C/Q value for an average of 40 buttons (calculated from B-1 analyses) produced on the RM and RG Lines are as follows:

<u>Origin</u>	<u>234-5 Process Line</u>	<u>Max.</u>	<u>Ave.</u>	<u>Min.</u>	<u>Ave. C/Q for Mg.</u>	<u>Ave. for others</u>
S-plant	RM	1.951	0.935	0.435	0.676	0.259
S-plant	RG	1.711	0.794	0.252	0.591	0.203
T-plant	RG	0.981	0.540	0.276	0.338	0.202

The average C/Q value for magnesium is included to indicate that the major portion of the C/Q value is due to Mg which is introduced during reduction of PuF_4 in an MgO crucible. Concentrations of Mg vary excessively, 20 to 500 parts per million parts plutonium. Omitting Mg, a fair comparison of light element impurities and their contribution to the C/Q value results.

4. Reduction yields in the RG Line, calculated from

$$\frac{(\text{wt. of metal} - (70 - 58 + \text{Recycle}))}{(\text{wt. of PuF}_4 \text{ charged} \times \frac{239}{315})} \times 100, \text{ averaged } 98.11\% \text{ for 120 runs}$$

reduced from processed Redox effluent as compared to 97.74% for 200 runs reduced from material from the bismuth-phosphate process during the same period.

TABLES

Spectro-chemical analyses of S-Plant material are included in Tables I to IV to indicate the purification achieved by the major purification steps. The corresponding sample codes are:

1. P-1, - analyses of Redox effluent.
2. AT, - analysis of the product after the Isolation Building treatment of 2 peroxide precipitations.
3. DC-1, - analysis of the plutonium oxalate cake.
4. B-1, - analysis of the metal button reduced from plutonium fluoride.

Table I includes 7 batches showing items 1, 2, 3, and 4 above.

Table II includes 9 batches showing items 2, 3, and 4 above.

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Table III includes 10 batches showing items 2 and 3 above.

Table IV includes 4 batches showing items 2 and 3, and 3 batches showing items 3 and 4 above.

Tables I - IV also includes pile residence in MWD, 231 Building "Sample Can" surveys in MR/hr., fluoride to metal reduction yields, and C/Q values for each batch.

Table V includes the maximum, average, and minimum impurity concentration in parts per million parts plutonium for 7 P-1, 28 AT, 48 DC-1, and 40 B-1 analyses.

Table VI includes maximum, average, and minimum impurity concentrations in parts per million parts plutonium for metal reduced from PuF_4 in the RM Line.

1

Redox Bat NUMBER	M.W.D.	231. BATCH NUMBER	SURVEY MR/HR	X UN NUMBER	Y-RUN NUMBER
S-02-02-L-26	260	12-B-126	4	12-2-187	12-2-103
S-02-02-L-27	260	12-B-127	1	12-2-188	12-2-103
85% S-02-02-L-28	260	12-B-128	4	12-2-189	12-2-103
15% S-02-02-L-27	"	12-B-142	1 1/2	12-2-173	12-2-96
61% S-02-02-L-29	260	12-B-143	4	12-2-200	12-2-110
39% S-02-02-L-28	"	12-B-146	4	12-2-203	12-2-115
S-02-03-L-10	553	12-C-104	5	12-3-103	12-4-3

BATCH	MR/HR	X UN	Y-RUN	YIELD	2. Mg	CODE	SAMPLE	A _g	A ₁	B	B _a	B _e	B
B-126	4					P-1		1600	1600	1600	1600	1600	1600
						AT		100	120	1.1	10	10.05	5
						DC-1		5	1.50	20	20	10.2	1
B-127	1					P-1		1600	1600	500	10.2		
						AT		1	1.20	1.2	10	10.03	5
						DC-1		1	1.50	10	50	10.2	5
B-128	4					P-1		1600	1600	508	10.2		
						AT		1	1.13	1.2	5	10.03	5
						DC-1		1	1.50	10	50	10.2	5
						B-1		30	0.8			10.05	
						DC-1		2	1.50	5	20	10.2	2
						B-1		30	0.8			10.05	
B-129	1					P-1		1600	1600	500	10.2		
						AT		1	1.20	1.2	10	10.03	5
						DC-1		1	1.50	10	50	10.2	5
						B-1		30	0.8			10.05	
B-130	1					P-1		1600	1600	500	10.2		
						AT		1	1.20	1.2	10	10.03	5
						DC-1		1	1.50	10	50	10.2	5
						B-1		30	0.8			10.05	
B-143	4					P-1		5000	5000	130	13.08		
						AT		2	30	1.2	20	10.03	2
						DC-1		2	1.50	5	50	10.16	2
						B-1		120	0.4			10.03	
						P-1		63000		200	10.05		
						AT		20	13	1.2	10	10.05	5
						DC-1		1	1.50	2	100	10.16	
						B-1		10	0.5			10.02	
B-146	4					P-1		63000		200	10.05		
						AT		20	13	1.2	10	10.05	5
						DC-1		1	1.50	2	100	10.16	
						B-1		10	0.5			10.02	
						P-1		25000		150	10.05		
						AT		1	1.20	1.1	15	10.2	
						DC-1		2	1.20	5	50	10.1	
						B-1		120	2.0			10.05	
C-104	5					P-1							
						AT		1	1.20	1.1	15	10.2	
						DC-1		2	1.20	5	50	10.1	
						B-1		120	2.0			10.05	

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REDOX TCH NUMBER	MWD	231 BATCH NUMBER	SURVEY MPHR	RUN NUMBER	Y-RUN NUMBER
S-02-03-L-5	553	12-C-94	5		
				12-3-93	
					12-4-1
66% S-02-03-L-6	553	12-C-97	3		
34% S-02-03-L-7	"	"	"	12-3-96	
					12-3-54
S-02-03-L-8	553	12-C-100	2		
				12-3-99	
					12-3-54
85% S-02-03-L-7	553	12-C-101	2		
15% S-02-03-L-9	"	"	"	12-3-100	
					12-4-1
S-02-03-L-9	553	12-C-102	2		
				12-3-101	
					12-4-3
S-02-03-L-9	553	12-C-103	2		
				12-3-102	
					12-4-5
S-02-03-L-10	553	12-C-104	5		
				12-3-103	
					12-4-3
S-02-03-L-11	553	12-C-106	2		
				12-3-105	
					12-4-15
52% S-02-03-L-15	566	12-C-132	1		
48% S-02-03-L-19	"	"	"		
				12-4-19	
					12-4-15

Y-RUN NUMBER	REDUCTION YIELD	S ₂ VALUES		SAMPLE CODE	SPE ^{TR}									
		Σ	Mg		Ag	Al	B	Ba	Be	Bi	Ca	Cd	Ce	Cr
				A.T.	L1	L20	L1	5	L0.08	L5*	20	50	L800	200
				DC-1	L2	1000	10	200	L0.05	L4	1000	L80	L500	40
12-4-1	99.2%	0.79	0.63	B-1		L20	0.5		L0.08		8			5
				A.T.	L1	L20	1	10	L0.1	L5	20	100	L500	200
				DC-1	L1	L20	5	L80	L0.05	1	L100	L50	L300	L1
12-3-54	99.0%	0.82	0.63	B-1		L20	L2		L0.03		20	2		
				A.T.	L1	L20	L1	L5	L0.2	2	20	L50	L1200	100
				DC-1	L1	L20	2	50	L0.1	*	L100	L160	L1000	2
12-3-54	99.0%	0.82	0.63	B-1		L20	L2		L0.05		20			2
				A.T.	L1	L20	L1	20	L0.1	5*	L100	50	L800	200
				DC-1	L1	L20	2	50	L0.1	*	L100	L160	L1000	2
12-4-1	99.2%	0.79	0.63	B-1		L20	0.5		L0.08		8			5
				A.T.	L1	L20	L1	L5	L0.2	5	20	L50	L1200	100
				DC-1	L1	L20	5	L50	L0.1	2*	L100	L160	L1000	2
12-4-3	99.9%	0.83	0.63	B-1		L20	2		L0.05		20			5
				A.T.	L1	L20	L1	L5	L0.2	5	20	L50	L1200	100
				DC-1	L1	L20	5	L50	L0.1	L2	L100	L160	L1000	L5
12-4-5	98.3%	0.89	0.63	B-1		L20	2.2		L0.05		30			10
				A.T.	1	L20	L1	L5	L0.2	2	20	L200	L1200	500
				DC-1	2	20	5	50	L0.1	5	L100	50	L1000	20
12-4-3	99.9%	0.84	0.63	B-1		L20	2		L0.05		20			5
				A.T.	1	L20	L1	20	L0.2	2	30	L200	L1200	500
				DC-1	L1	L20	5	L30	L0.05	L1	30	L50	L300	2
12-4-15	100.7%	1.71	1.56	B-1		L20	0.4		L0.05		50			5
				A.T.	61000	L20	L1	20	L0.08	2	50	61000	L300	200
				A.T.	L20	L20	L1	20	L0.08	2	L30	50	L300	200
				DC-1	L1	L20	10	L50	L0.03	L2	20	L50	L200	L5
12-4-15	100.7%	1.71	1.56	B-1		L20	0.4		L0.08		50			5

ANALYSIS

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Y-RUN	NUMBER	YIELD	REDUCTION	2.0	SAMPLE	CODE	Ag	Al	B	Ba	Be	Bi	Ca	Cd
2-3-88	12-3-50	98.3%	Ungrainy	B-1										
				DC-1	L1	1100	2	50	10.8	1.1	100	1.50		
				AT	L1	120	1	5	10.13	1	50	1.50		
2-3-89	12-3-49	77.6%		B-1										
				DC-1	L1	120	1	2	10.13	2	50	1.50		
				AT	L1	120	1	2	10.08	5	30	1.50		
2-3-90	12-3-51	99.9%		B-1										
				DC-1	L1	120	5	1.50	10.05	5	1100	1.50		
				AT	L1	120	1	5	10.03	15*	20	50		
2-3-91	12-3-52	98.9%		B-1										
				DC-1	L1	120	2	1.50	10.05	1.1	1100	50		
				AT	L1	120	1	10	10.1	1.5	20	100		
2-3-92	12-3-53	99.6%		B-1										
				DC-1	L1	120	5	1.50	10.05	1.1	1100	1.50		
				AT	L1	120	1	20	10.1	5*	1100	50		
2-3-93	12-3-54	99.6%		B-1										
				DC-1	L1	120	5	1.50	10.05	1.1	1100	1.50		
				AT	L1	120	1	1.5	10.2	2	20	1200		
2-3-94	12-4-2	98.0%		B-1										
				DC-1	L1	120	2	1.50	10.1	1.1	1100	1.50		
				AT	L1	120	1	1.5	10.2	2	20	1200		
2-3-95	12-4-8	99.5%		B-1										
				DC-1	L1	120	5	100	10.05	1.2	30	1.50		
				AT	L1	120	1	40	10.05	1.2	30	1.50		

ELECTRO-CHEMICAL ANALYSIS

Ca	Cd	Ce	Cr	Cu	Fe	Hg	In	K	La	Li	Mg	Mn	Na	Ni	P	Pb
50	L50	L500	200	10	1500	P*	L10	20	L5	L1	50	200	L500	500	200	50
100	L50	L500	2	50	150		L5	5	L30	L1	100	2	150	50	L100	5
30	L50	L500	200	20	1500	P*	L10	20	L50	L1	50	200	L500	500	200	50
100	L80	L500	2	20	50		L5	200	L80	5	20	5	250	50	L100	L5
50	L50	L500	200	10	1000	P*	L10	50	L50	L1	50	200	5000	200	200	50
100	50	L300	L2	200	50		L5	L1	L80	L1	20	5	50	50	L10	L5
50	L50	L500	200	10	1000	P*	L10	50	L50	L1	50	200	5000	200	200	50
100	L50	L1600	2	10	50		L5	100	L250	2	20	10	250	100	L100	L5
20	50	L800	200	5	3000	P*	L10	100	L130	L1	50	50	4x10 ⁴	200	200	50
100	50	L1600	2	5	50		L5	1	L250	L1	20	10	55	100	L100	L5
20	100	L500	200	10	1800	P*	L10	50	L80	L1	50	100	2500	500	500	50
100	L50	L500	L1	10	65		L10	L1	L50	L1	L20	2	2.5	20	L100	L5
100	50	L300	200	5	1800	P*	L10	100	L130	L1	50	100	15000	200	200	50
100	L50	L300	2	10	50		L5	5	L50	L1	20	10	250	50	L100	L5
20	L50	L1200	100	1	700	*	L10	100	L200	L1	20	50	4x10 ⁴	50	100	L10
100	L50		L2	10	50		L5	2		1	50	5	150	20	L100	L5
20	L200	L1200	500	50	1400	*	L10	50	L200	L1	50	200	4x10 ⁴	200	500	20
100	L50	L1000	20	10	50		L20	5	L100	L1	10	5	250	20	L100	L5
0	L50	L200	1000	10	1000		L20	200	L800	L2	200	100	5000	L40	1000	200
0	L50	L300	130	20	61000		L20	10	L50	L1	100	200	50	80	1000*	*

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REMARKS

6	Sb	Si	Sn	Sr	V	Zn	Ti	F
50	L100	5	L1	L8	L100	L50	L200	
5	L100	10	L1	L8	L100	L50	L200	
50	L100	5	L1	L8	L100	L50	L200	
5	L100	10	L1	L8	L100	L50	L200	
50	L100	5	L1	L8	L100	L50	L200	
5	L100	5	L1	L8	L100	L50	L200	
50	L100	5	L1	L8	L100	L50	L200	
5	L100	5	L1	L8	L100	L50	L200	
50	L100	5	L1	L8	L100	L50	L200	
5	L100	5	L1	L25	L100	L50	L640	
50	P*	5	L1*	L13	L100	L100	L300	
5	L100	5	L1	L25	L100	L50	L640	
50	P*	5	L1*	L8	L100	L100	L200	
5	L100	5	L1	L13	L200	L100	L300	
50	P*	5	L1*	L13	L100	L100	L300	
5	L100	5	L1	L8	L100	L50	L200	
10	P*	5	L1	L20	L100	L100	L500	
5	L100	5	L1		L100	L50		
20	L100	5	L1	L20	L100	L100	L500	
5	L100	5	L2	L25	L100	L50	L640	
20	L200	20	L2	L8	L100	L100	L200	
*	*	50	L5	L5	L100	L50		

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TABLE III

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BATCH NUMBER	X-F V	Y-RUN NUMBER	REDUCTION %	VALUES	CODE	Ag	Al	B	Ba	Be
2-C-126	2	12-3-111	99.5%	Not Available	AT	1	1.25	1.1	10	1005.7
2-C-127	5	12-4-1	99.0%	Not Available	DC-1	1.1	1.20	1.0	1.80	1005.7
2-C-150	3	12-4-17	99.0%	Not Available	AT	1	1.20	1.1	2	1005.7
2-C-153	3	12-4-37	98.0%	Not Available	DC-1	1.1	1.20	1.0	1.80	1005.7
2-C-154	"	12-4-38	98.0%	Not Available	AT	1	1.20	1.1	2	1005.7
2-C-182	4	12-4-65	98.6%	0.68	AT	10	1.20	1	20	101.1
2-C-183	5	12-4-66	98.6%	0.68	AT	11	1.20	1.1	20	101.1
2-C-196	4	12-4-78	99.4%	0.74	AT	120	1.20	0.1	1015	

SPECTRO CHEMICAL ANALYSIS

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REMARKS

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TABLE IV

TABLE V RANGE OF IMPURITIES AFTER VARIOUS PURIFICATION STEPS

HW-25834

(From Spectro-Chemical Analyses Reports)

Sample Code No. Samples	P-1			A.T.			RGX-DC-1			RGY-B-1		
	7			28			48			40		
Impurity	Max.	Ave.	Min.	Max.	Ave.	Min.	Max.	Ave.	Min.	Max.	Ave.	Min.
Ag				1000	49.4	L1	5	1.4	L1		L1	
Al	25000	5686	1600	400	39.7	L13	1000	56.1	L2	30	19.8	L1
As				L2	L1	L1	L200	L17.7	L1		L1	
B				2	1.3	L1	20	6.6	L2	2.30	0.84	L0.10
Ba	1600	444	L50	L250	28.4	2	200	52.7	L10		L5	
Be	L0.2	L0.09	L0.03	L0.02	L0.105	L0.03	L2.0	L0.12	L0.03	L0.13	L0.05	L0.02
Bi				500	30.5	L1	50	7.9	L1			
Ca	1000	350	130	250	49.5	L13	1000	122	20	200	37	L8
Cd	L250	L110	L50	G1000	111.3	L30	L300	62.1	30		L50	
Ce	L1000	L473	L130	L1200	548.6	L130	L1600	L547	L200		L130	
Cr	10000	3129	300	1600	365.7	100	130	8.9	L1	20	5.9	L1
Cu				1000	49.4	1	200	20.8	1		12.5	
Fe	S-10 only											
	10000			3000	1353	700	G1000	140.5	50	1400	197.4	80
Hg	L6400	L3050	L1300	L2000	L1333	L800			L4?*			
In	L400	L213	L130	L400	L38.2	L10	L40	L21.0	L5		L10	
K				500	104.6	20	200	53.1	L1		15	
La	G2X10 ⁴	3204	50	L800	153.6	L20	1000	146	30	800	105	L20
Li	25	14.3	5	L25	13.7	L1	5	11.3	L1	L1	L1	L1
Mg	160	88.6	50	200	52.1	20	200	32.2	5	500	196	50
Mn	400	125	50	200	104.8	20	20	11.6	L2	200	39	10
Na	5000	3560	20	LX10 ⁴	2.02X10 ⁴	2500	3000	452	10	50	11	L10
Ni	3000	997	400	500	203	L20	200	63	L20	200	57	L20
P				1000	402	L100	1000*	163	L100		100	
Pb	L640	L393	L200	L1000	129	L10	L640	27.4	L4		10	
Sb				L200	L120	L100	L200	130	L100		L100	
Si				20	5.8	L5	50	13.4	L5	20	7	L5
Sn				L10	L1.5	L1	L20	16.2	L1		L1	
Sr	L64	L14	L3	L20	L10	L2	L25	L17	L2		L2	
V				L100	L100	L100	L200	L127	L100		L100	
Zn	L1000	330	L30	1000	114	L50	L200	L92	L50		L50	
Tl	L640	L374	L200	L1000	L352	L130	L1000	L313	L30		L130	

SYMBOL

MEANING

NUMERICAL VALUES = PARTS PER MILLION PARTS PLUTONIUM

L

LESS THAN

G

MORE THAN

* Interference, results questionable.

DECLASSIFIED

DECLASSIFIEDTABLE VI

RM LINE METAL IMPURITIES
(From Spectro-Chemical Analyses Reports)

41 RMX-B-1 Analyses

<u>Impurity</u>	<u>Max.</u>	<u>Ave.</u>	<u>Min.</u>
Ag	L1	L1	L1
Al	L50	L19	L1
As	L100	L48	L1
B	1.9	0.7	L0.1
Ba	L100	39	L5
Be	L0.1	L0.06	L0.03
Bi	L5	L1.8	L1
Ca	500	51.3	8
Cd	L80	50	L30
Ce	L800	391	L200
Cr	100	15	L1
Cu	100	18	1
Fe	325	156	L50
In	L20	L14.2	L10
K	L80	10	L1
La	200	48	L8
Li	L5	L1.2	L1
Mg	500	227	100
Mn	500	97	10
Na	80	13	L5
Ni*	500	183	20
P	200	113	L100
Pb	20	10	L5
Sb	L100	L100	L100
Si	10	6	L5
Sn	20	9	L1
Sr	L13	7	L5
V	L100	L100	L100
Zn	L100	L60.4	L50
Tl	L320	L181	L130

* Does not include RMX-12-4-2 which had 2000 ppm Ni due to processing in an inconel boat.