Atlantic Richfield Hanford Company
Richland, Washington 99352

ARH-9

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LABORATORY QUALITY CONTROL PROGRAM
STANDARDS AND REFEREES

D. J. Rochon

December 1, 1967

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LABORATORY QUALITY CONTROL PROGRAM:
STANDARDS AND REFEREES

D. J. Rochon

Technical Services Section
Chemical Processing Division
Atlantic Richfield Hanford Company
Richland, Washington

December 1, 1967

This report was prepared for use
within Atlantic Richfield Hanford Co.
in the course of work under
Atomic Energy Commission Contract AT(45-1)-2130

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I. PROGRAM DESCRIPTION

The products of a laboratory are measurements. An integral part of laboratory operation is the quality control program to evaluate the precision and accuracy of the measurements produced.

Electronic data processing (EDP) programs have been developed to speed the computation of precision and accuracy statements. The Laboratory Standards and Referee EDP Program is designed for treating data from process samples and from standard solutions simulating process samples. The Plutonium Finishing Analytical EDP Program is designed for making precision and accuracy statements on emission spectrographic data and will be discussed in detail as an appendix to this document. The multicomponent analytical program (MAP) is designed to treat data from gamma emitting isotopes.

The ARHCO Laboratory Standards and Referee EDP Program as administered by the Standards Laboratory is capable of handling 999 different analytical methods for each of ten laboratories. There are three laboratorians presently participating and approximately 100 methods are being monitored.

Standard synthetic mixtures simulating plant streams are prepared by the Standards Laboratory and issued to the participating laboratories. Five percent of the work load in a control laboratory is assigned to analyzing standard solutions. From the results of the analyses by laboratorians and chemists, the average percent recovery and the standard deviation is computed to give a measure of the accuracy and the precision of an individual observation at the 95 percent confidence level.

Another five percent of the work load is assigned for referee samples. An authentic plant sample, chosen at random, is analyzed by two analysts on succeeding shifts. It must be emphasized that this does not yield a "between analysts" variance, but simply the variance observed on the plant samples as run in the laboratory. Sources of variance other than the analyst may exist; concentration of the sample due to evaporation (a time variance) or solids present in the sample are examples of these other sources of variance.

At monthly and quarterly intervals, the data is processed into a primary report consisting of tabulations of values and the resultant statistical measures. A summary page listing the laboratory methods analyzed and their monthly average and standard deviations is published with the primary report. The summary page includes a listing of flyers and reruns and indicates significant shifts in the average and precision of a given method.

At quarterly intervals, a more concise report is issued summarizing the standard and referee statistical data.

The Laboratory Standards and Referee EDP Program has been written in sub-routine form so that additions to the program may be made.

The following detailed discussion is written as an aid to those who consult the reports.

II. MECHANISM OF THE PROGRAM

A. Input Data

1. Work Load for Standards and Referees

Referees and standards are intended to amount to about ten percent of the work load in a control laboratory--five percent for each. Where greater control is necessary, more standards and referees may be run. Fewer standards and referees may be run for samples of little importance.

2. Scheduling of Standard and Referee Analyses

A schedule indicating the number of standards or referees to be run is called a "frequency sheet" and is issued to the laboratories by the Standards Laboratory each month. (See Figures Ia and Ib).

A tabulation of current standard values is kept in each control laboratory. A master list of IBM codes issued is kept in the Standards Laboratory. (See Figure II)

3. Standard Samples

Standard uranium solutions are made from high purity UO_3 . This is designated as natural uranium and has low metallic impurities. It contains a trace of NO_3^- , which is readily removed by washing with water.

Uranium standard solutions are made from weighed amounts of UO_3 , but because of variable moisture content of the powder, the solutions are characterized by drying weighed samples (with the aid of HF) and igniting to U_3O_8 .

Plutonium nitrate solutions are made from plant (Purex) product. The product is filtered, fumed with sulfuric acid in a carefully controlled manner to remove nitrate and then is reduced by TiCl_3 and titrated with standard ceric solution to obtain a standard value.

A standard value for total alpha count is also determined for plutonium nitrate solutions. A calculated correction for americium growth is applied to the total alpha each month.

Neptunium standards characterized by alpha count are also used.

Standard synthetic mixtures based on these materials and simulating plant streams are prepared and issued. Standard solutions are packaged and weighed. The weight is again checked before issuing the package as a guard against possible evaporation loss.

Standards for the spectrochemical laboratory are also prepared and issued. Results on these are reported in the P.F.A. Quarterly Report.

Standard "gamma" samples are also used, though a separate program, Multicomponent Analytical Program, "MAP", covers most of these.

Standards are also made for such items as iron by spectrophotometer, nitric acid by "HPH", and for "falling drop" specific gravity.

4. Recording of Laboratory Results

New cards have been designed for reporting results of analyses. 7-3/8 X 3-1/4 IBM cards are used. Referee cards are white with a salmon edge. Standard cards are buff with a gray deckle edge. Examples follow.

Both the standard and the referee card have space for the initials of those approving the results. These cards must be filled out without error, as the data must be taken from them by key punch operators who are instructed to never try to correct or interpret entries on a card. After the cards have been filled out and approved, they should be returned to the Standards Laboratory, 222-S Building, 200-W Area, on a weekly basis. A random selection of fifteen of each standard and referee cards will be made to determine the correctness of entries. In the event an error is found, the entire week's cards will be returned to the originator for corrections.

Midnight the twentieth day of the month is the cut-off date for standards and referee data. By the second working day after the twentieth, all cards for the month are sent to the data control operation for key punching. A data transmittal form accompanies the cards for subsequent submission to Computer Sciences Corp., Federal Building, Richland.

The new standard key-punch size data cards are perforated so that they may be sorted if desired. For example, all the cards covering given accountability points could be segregated either before or after the monthly report is issued.

USE CAPITAL LETTERS
PRINT CLEARLYPUREX STANDARDS PROGRAM

DATE ISSUED _____ SHIFT _____ OBSOLETE DATE _____

METHOD	CODE	STD.	NO./MO.
DZ 1A-0	048	D 5	2
FES-1A	038	FES 1A	1
FES 1B	039	FES-1B	1
HC-1A (Cell #1 & 2)	025	HC-1A	Run 1/day on Swing
HPH 1B	029	D 5	2
HV-1A	031	HV-1A	2
HV 3C	033	L-9	2
NPA 5A	014	3 XF	3
NPA 40A	105	3 AW	2
NPA 6A	010	K6-2	1
PUA 30A	007	K6 2	1
PUA 6B 1	001	L 9	3
PUA 6B 4	003	D-5	3
PUX 3A	009	L-9	3
PUA 20A	006	F 15 2	1
UF 1B	017	UFSTD2	1
UF 1C	019	USTD1C	1
UX-2A	013	K6-1	3
TB 1A	046	TBP	1
FEB-1B	040	1 BX	1
HV 1A	035	1 BX	1
NPA 40B	012	F 15 1	1
DZ 1A	042	AQ.STD3	5
DZ 1A	043	AQ.STD4	5
NPA 5A	016	3 XW	3
NPC	020	3 XN	Run as in- structed

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FIGURE 1b

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234-5 REFEREE FREQUENCY SHEET

DATE ISSUED	SHIFT OBSOLETE
-------------	---------------------

[illegible]

MASTER LIST IEM CODES

222-S UO₃ LABORATORY

<u>IEM Code</u>	<u>Standard Material</u>	<u>Bldg.</u>	<u>Vol. of Std. (ml)</u>	<u>Shipping Freq.</u>	<u>Method</u>	<u>No. Std. Per Mo.</u>	<u>Sample Refereed</u>	<u>No. Referees Per Month</u>
141								
142								
143								
144								
145								
146								
147	C-1	R	2-30 ml	15th	XRE	**Use only if UDS method is out		
148	C-1	R	2-30 ml	15th	UDS	9		
149	C-1	R	2-30 ml	15th	UD-1A	9		
150								
151	C-4	R	30 ml	15th	XRE	4		
152	NA-STD	R	30 ml	15th	NAJ-1B	Run as directed		
153	C-4	R	SEE CODE 151		H-1A	4		
154								
155	FES-1D	R	30 ml	15th	FES-1D	4		
156								
157	SULFUR	R	30 ml	15th	SS-2A	4		
158	C-1	R	SEE CODE 149		HPH-1B	9		
159	S-LOW	R	30 ml	15th	SS-2A	4		
160	C-1	R	SEE CODE 149		DW-1A	9		
161	C-1S	R	17 ml	15th	NPA-5F	4		
162	C-1S	R	17 ml	15th	PUA-20C	4		

163

164

When filling out a card, print alphabetical letters and numerals. Exercise care that (1) 7 and 4 are written plainly, (2) "A" is printed as (A) and not (A), which can be misinterpreted as "H", (3) "U" is printed as (U) and not (U), which can be misinterpreted as "V". Mark alphabetical letters to distinguish them from numerals; for example:

One (/) and I (I)

Two (2) and Z (Z)

Zero (O) and 0 (0)

a. Description of the standard card:

B		CODE		METHOD		DATE			STANDARD NO.		MATERIAL		
1	2	3	4	5	6	7	8	9	10	11	12	13	
P025HC-1A						16	17	18	19	20	21	22	
HC-1A						23	24	25	26	27	28	29	
SHIFT		P.R. NO.		NEG		STD VALUE		EXP.		NEG		RESULT	
30	31	32	33	34	35	36	37	38	39	40	41	42	43
44		45		46		47		48		49		50	
51		52		53		54		55		56		57	
58		59		60		61		62		63		64	
65		66		67		68		69		70		71	
72		73		74		75		76		77		78	
79		80		81		82		83		84		85	
86		87		88		89		90		91		92	
93		94		95		96		97		98		99	
100		101		102		103		104		105		106	
107		108		109		110		111		112		113	
114		115		116		117		118		119		120	
121		122		123		124		125		126		127	
128		129		130		131		132		133		134	
135		136		137		138		139		140		141	
142		143		144		145		146		147		148	
149		150		151		152		153		154		155	
156		157		158		159		160		161		162	
163		164		165		166		167		168		169	
170		171		172		173		174		175		176	
177		178		179		180		181		182		183	
184		185		186		187		188		189		190	
191		192		193		194		195		196		197	
198		199		200		201		202		203		204	
205		206		207		208		209		210		211	
212		213		214		215		216		217		218	
219		220		221		222		223		224		225	
226		227		228		229		230		231		232	
233		234		235		236		237		238		239	
240		241		242		243		244		245		246	
247		248		249		250		251		252		253	
254		255		256		257		258		259		260	
261		262		263		264		265		266		267	
268		269		270		271		272		273		274	
275		276		277		278		279		280		281	
282		283		284		285		286		287		288	
289		290		291		292		293		294		295	
296		297		298		299		300		301		302	
303		304		305		306		307		308		309	
310		311		312		313		314		315		316	
317		318		319		320		321		322		323	
324		325		326		327		328		329		330	
331		332		333		334		335		336		337	
338		339		340		341		342		343		344	
345		346		347		348		349		350		351	
352		353		354		355		356		357		358	
359		360		361		362		363		364		365	
366		367		368		369		370		371		372	
373		374		375		376		377		378		379	
380		381		382		383		384		385		386	
387		388		389		390		391		392		393	
394		395		396		397		398		399		400	
401		402		403		404		405		406		407	
408		409		410		411		412		413		414	
415		416		417		418		419		420		421	
422		423		424		425		426		427		428	
429		430		431		432		433		434		435	
436		437		438		439		440		441		442	
443		444		445		446		447		448		449	
450		451		452		453		454		455		456	
457		458		459		460		461		462		463	
464		465		466		467		468		469		470	
471		472		473		474		475		476		477	
478		479		480		481		482		483		484	
485		486		487		488		489		490		491	
492		493		494		495		496		497		498	
499		500		501		502		503		504		505	
506		507		508		509		510		511		512	
513		514		515		516		517		518		519	
520		521		522		523		524		525		526	
527		528		529		530		531		532		533	
534		535		536		537		538		539		540	
541		542		543		544		545		546		547	
548		549		550		551		552		553		554	
555		556		557		558		559		560		561	
562		563		564		565		566		567		568	
569		570		571		572		573		574		575	
576		577		578		579		580		581		582	
583		584		585		586		587		588		589	
590		591		592		593		594		595		596	
597		598		599		600		601		602		603	
604		605		606		607		608		609		610	
611		612		613		614		615		616		617	
618		619		620		621		622		623		624	
625		626		627		628		629		630		631	
632		633		634		635		636		637		638	
639		640		641		642		643		644		645	
646		647		648		649		650		651		652	
653		654		655		656		657		658		659	
660		661		662		663		664		665		666	
667		668		669		670		671		672		673	
674		675		676		677		678		679		680	
681		682		683		684		685		686		687	
688		689		690		691		692		693		694	
695		696		697		698		699		700		701	
702		703		704		705		706		707		708	
709		710		711		712		713		714		715	
716		717		718		719		720		721		722	
723		724		725		726		727		728		729	
730		731		732		733		734		735		736	
737		738		739		740		741		742		743	
744		745		746		747		748		749		750	
751		752		753		754		755		756		757	
758		759		760		761		762		763		764	
765		766		767		768		769		770		771	
772		773		774		775		776		777		778	
779		780		781		782		783		784		785	
786		787		788		789		790		791		792	
793		794		795		796		797		798		799	
800		801		802		803		804		805		806	
807		808		809		810		811		812		813	
814		815		816		817		818		819		820	
821		822		823		824		825		826		827	
828		829		830		831		832		833		834	
835		836		837		838		839		840		841	
842		843		844		845		846		847		848	
849		850		851		852		853		854		855	
856		857		858		859		860		861		862	
863		864		865		866		867		868		869	
870		871		872		873		874		875		876	
877		878		879		880		881		882		883	
884		885		886		887		888		889		890	
891		892		893		894		895		896		897	
898		899		900		901		902		903		904	
905		906		907		908		909		910		911	
912		913		914		915		916		917		918	
919		920		921		922		923		924		925	
926		927											

- 6-16 = Analytical method code; for example, PUA-6B
- 17-22 = Date--Fill in all six spaces, e.g. March 7, 1967, would be written 03-07-67 and December 21, 1967, would be written 12-31-67.
- 23-28 = Identifying number of standard sample
- 29-34 = Identifying symbols for material being analyzed
- 35 = Shift, i.e. A, B, C, D
- 36 = D, S, G, for Days, Swing and Graveyard
- 37-41 = Payroll number of analyst
- 42 = Used for negative value such as minus lbs/gal where a negative sign (-) is entered.
- 43-47 = Numerical value of standard value is entered here with the exponential value in 48-49.
- 48-49 = Exponent associated with numerical value in 43-47
- 0.135 would be written with 1.35 in spaces 43-47 and -1 in spaces 48-49.
- 5.97×10^3 would be written with 5.97 in spaces 43-47 and 03 in spaces 48-49.
- 50 = Provision for negative result
- 51-55 = Numerical value obtained as a result of analyzing the standard
- 56-57 = Exponent associated with value observed in 51-55
- 58 = A 1 is entered to designate a first rerun and a 2 for a second rerun, etc.
- 59-72 = Space for special notes by an analyst
- 80 = A "Z" is printed here to distinguish current data from deletion or update entry.

b. Referee Card

FZ183

B		CODE				METHOD				DATE						SERIAL NO.				MATERIAL									
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
R	Z	1	8	3		P	U	A	-	6	B	-	2																

SHIFT		O. ANALYST PR NO.		NEG		REPORTED RESULT		EXP.		
35	36	37		41	42	43		47	48	49

SHIFT		REF. ANALYST		NEG		REF. RESULT		EXP.		
50	51	52		56	57	58		62	63	64

CORR		RR		T A G			
65	66	67					79

RANGE	
80	
Z	

APPROVED

CALCULATIONS

SAMPLE SIZE

A-6300-010 (2-67)

Figure (IV) - The referee card, white with salmon edge, is preprinted and prepunched before use in the laboratories.

The referee card consists of 80 spaces divided as follows:

- 1 = "R" for referee
- 2 = "P", "R" or "Z" for Purex, Redox or Z-Plant respectively
- 3-5 = Identifying data processing code
- 6-16 = Analytical method code, e.g. UF-1B
- 17-22 = Date, use all six spaces as discussed under Standard Card
- 23-28 = Serial number identifying the plant sample being run by two analysts
- 29-34 = Identifying symbols for the material being determined, such as sampling point or tank number.
- 35 = Shift, e.g. A, B, C, D, etc.

- 36 = D, S, G, for Days, Swing or Graveyard respectively
- 37-41 = Original Analyst's payroll number
- 42 = Provision for a negative result
- 43-47 = Numerical value of the original reported result
- 48-49 = The exponent associated with the numerical value reported in 43-47
- A value of 0.135 would be entered as 1.35 in spaces 43-47 and -1 in spaces 48-49
- A value of 5.75×10^{13} would be entered as 5.75 in spaces 43-47 and 13 in spaces 48-49
- 50-56 = Referee analyst's shift and payroll number
- 57 = Provision for a negative result
- 58-64 = Referee result in exponential notation
- 65 = A 1 is entered if, as a result of the referee analysis, a corrected report was made to the processing operation.
- 66 = A 1 is entered for a first rerun, a 2 is entered for a second rerun, etc.
- 67-79 = Provision for comments
- 80 = A "Z" is entered to distinguish the original data card from delete and update cards

c. LSR Input Data Transmittal Form

The LSR Input Data Transmittal Form (Figure V) is a multipurpose form. Its primary function is to supply a date and control parameter card for the completed data. This card controls the program flow through its many options and insures that the correct tape files are mounted. The format as laid out on the form is as follows:

- 1-6 = Master File and Long Term File identification date. This date must match with the date recorded on the tape files. The file date is the date the tape was created and is listed on the most recent LSR Primary Report. If columns 1-6 are left blank, no Master File or Long Term

LSR INPUT DATA TRANSMITTAL																																																																																													
SENDER					DEPARTMENT																																																																																								
PLANT MAILING ADDRESS					PHONE			DATE																																																																																					
DESCRIPTION AND TYPE OF INPUT DATA																																																																																													
JOB NO. LSR		TYPE OF RUN: ☐ MONTHLY ☐ QUARTERLY ☐ RERUN			INPUT SOURCE: ☐ PUNCHED CARDS ☐ KEYPUNCH ☐ PARTIAL ☐ COMPLETE																																																																																								
DATE AND CONTROL PARAMETER CARD																																																																																													
<div style="display: flex; justify-content: space-between; align-items: flex-start;"> <div style="text-align: center;"> M M D D Y Y <table border="1" style="border-collapse: collapse; width: 100px;"> <tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr> <tr><td style="text-align: center;">1</td><td></td><td></td><td></td><td></td><td style="text-align: center;">6</td></tr> </table> </div> <div> REQUESTED LSR MASTER FILE DATE. </div> </div> <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>REPORT PERIOD DATES:</p> <div style="display: flex; justify-content: space-between;"> <div style="text-align: center;"> M M D D Y Y FROM <table border="1" style="border-collapse: collapse; width: 100px;"><tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr><tr><td style="text-align: center;">7</td><td></td><td></td><td></td><td></td><td style="text-align: center;">12</td></tr></table> </div> <div> TO <table border="1" style="border-collapse: collapse; width: 100px;"><tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr><tr><td style="text-align: center;">13</td><td></td><td></td><td></td><td></td><td style="text-align: center;">18</td></tr></table> </div> </div> </div> <div style="width: 45%;"> <p>QUARTERLY RUN FLAG:</p> <div style="display: flex; align-items: center;"> ☐ PUNCH "Q" IF QUARTERLY </div> </div> </div> <div style="text-align: center;"> <p>LIST MASTER FILE (OPTIONAL):</p> <div style="display: flex; justify-content: space-between; align-items: flex-start;"> <div style="text-align: center;"> ☐ PUNCH "L" FOR LIST. 20 </div> <div> PERIOD FROM <table border="1" style="border-collapse: collapse; width: 100px;"><tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr><tr><td style="text-align: center;">21</td><td></td><td></td><td></td><td></td><td style="text-align: center;">26</td></tr></table> </div> <div> TO <table border="1" style="border-collapse: collapse; width: 100px;"><tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr><tr><td style="text-align: center;">27</td><td></td><td></td><td></td><td></td><td style="text-align: center;">32</td></tr></table> </div> </div> </div> <div style="text-align: center;"> <p>DELETE PERIOD FROM MASTER FILE (OPTIONAL):</p> <div style="display: flex; justify-content: space-between; align-items: flex-start;"> <div> DELETE PERIOD DATES.....FROM <table border="1" style="border-collapse: collapse; width: 100px;"><tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr><tr><td style="text-align: center;">33</td><td></td><td></td><td></td><td></td><td style="text-align: center;">38</td></tr></table> </div> <div> TO <table border="1" style="border-collapse: collapse; width: 100px;"><tr><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td><td style="width: 20px; height: 20px;"></td></tr><tr><td style="text-align: center;">39</td><td></td><td></td><td></td><td></td><td style="text-align: center;">44</td></tr></table> </div> </div> </div> <p>NOTES:</p> <ol style="list-style-type: none"> (1) ALL DATES LESS THAN 10 MUST BE PRECEDED BY A ZERO, E.G. 010867. (2) IF THE LIST OPTION IS NOT USED THE MASTER FILE LIST WILL COVER ONLY THE REPORT PERIOD DATES. (3) THE DELETE OPTION SHOULD BE USED ONLY WHEN THE DATA FOR A GIVEN PERIOD IS NO LONGER NEEDED. 																1					6							7					12							13					18							21					26							27					32							33					38							39					44
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CONTROL INFORMATION AND INSTRUCTIONS																																																																																													
<p>1. THE DATE AND CONTROL PARAMETER CARD AS PUNCHED ABOVE IS THE FIRST CARD OF ALL SUBMITTED DATA.</p>																																																																																													

File will be mounted when the program runs, but new Master and Long Term Files will be generated and saved for the next run.

7-12 = Start date for the report period, e.g. -7-21-67

13-18 = End date for the report period, e.g. 08-20-67

19 = Quarterly report flag. If a quarterly report is requested, a "Q" should be input in this column, otherwise it is left blank.

20 = Master File List flag. If a listing of the Master File covering other than the report period is desired, an "L" should be input in this column, otherwise it is left blank.

21-26 = Start date for period to be listed from the Master File

27-32 = End date for period to be listed from the Master File

At various intervals it may be determined that the Master File contains data that is out of date and is no longer needed. The option to delete this material is provided in columns 33-44. Even though the Master File can save five years data, it is desirable to limit the data span to between twelve and eighteen months. The reason for this is that the program takes longer to run with more data on the file.

33-38 = Start date for period to be deleted from the Master File

39-44 = End date for period to be deleted from the Master File

In addition to the preparation of the Date & Parameter Card, the Input Data Transmittal form provides the customer the option of sending the data to CSC for keypunching or submitting punched cards. These cards may be input either as a partial submission or a complete month's data by checking the appropriate squares under "Input Source". For a partial submission of cards, no date parameter card is required.

B. Computation and Reporting of Results

There are three routine reports issued from the LSR Program output. The first is called the primary report and is issued on 8½ X 14" paper from the EDP printer. The other routine reports

are derived from this. The second report, issued on duplimats from the EDP printer, is a special quarterly summary report. The third routine report is a one-page stenographic report called "CPD Analytical Control Data Corrections to be Applied to Accountability Source Data". This report uses data taken from the primary report.

1. The Primary Report

The primary report consists of four page types: (1) The LSR Master File Page, (2) The Results Page, (3) The Detail Page, and (4) The Summary Page. In addition to these four page types, an error report is issued if any data card contains an illegal program entry. All delete and update records processed are printed on a separate report page; and for the quarterly run, the quarterly titles are listed.

The LSR Master File page (Figure VI) is a tabulation of all data from standard and referee cards covered by the control dates listed on the Input Transmittal Form. (See Figure V)

Column 1	= Record Number. Each data card is assigned a record number after the new data has been merged with the old and sorted according to Referee or Standard, Building, EDP Code, and date. Therefore, the record numbers will change from month to month as more data is compiled.
Column 2	= "R" for referee data, "S" for standards data
Column 3	= "P", "R", or "Z" for Purex, Redox, or Z-Plant, respectively
Column 4	= EDP code number assigned to a particular method and building
Column 5	= Analytical method
Column 6	= Date
Column 7	= Serial number for referee data and standard number for standards data
Column 8	= Material identification code
Columns 9 and 10	= Original analysts's shift and payroll number
Column 11	= Assigned analytical value of the standard or original analytical value of referee

LSR MASTER FILE FILE DATE 10-10-67

UNCLASSIFIED

RCRD	CODE	METHOD	DATE	SER/STD	MAT.	S	O/ANL	RSLT/STD	S	R/ANL	RSLT	CR	RR	COMMENTS
34	R P	1	PUA-6B-1	062767	4519	L-10	AS	61765	6.2700+13	CG	60360	6.3400+13	-0	-0
35	R P	1	PUA-6B-1	062967	4796	L-10	CG	61547	8.9500+13	BS	61581	8.8900+13	-0	-0
36	R P	1	PUA-6B-1	071667	6227	L-10	DS	61119	9.1800+13	CD	60360	9.2200+13	-0	-0
37	R P	1	PUA-6B-1	071767	6367	L-10	DS	61119	9.1000+13	BG	61765	9.2800+13	-0	-0
38	R P	1	PUA-6B-1	072967	7719	L-10	AD	60689	7.2700+13	BS	61521	7.4000+13	-0	-0
39	R P	1	PUA-6B-1	072967	7719	L-10	DG	61765	6.8400+13	AD	61291	7.2000+13	1	1
40	R P	1	PUA-6B-1	080767	8672	L-10	CS	61521	8.1200+13	CS	60415	7.9200+13	-0	-0
41	R P	1	PUA-6B-1	080967	8865	L-10	BD	61536	8.2800+13	DS	61768	8.0200+13	-0	-0
42	R P	1	PUA-6B-1	081467	9394	L-10	DS	61768	7.9000+13	BG	61524	7.9500+13	-0	-0
43	R P	1	PUA-6B-1	090967	676	L-10	BG	61581	7.0500+13	CD	60360	7.2200+13	-0	-0
44	R P	1	PUA-6B-1	091067	759	L-10	BG	61581	7.5200+13	CD	60360	7.4000+13	-0	-0
45	R P	1	PUA-6B-1	091167	851	L-10	BG	61581	7.1400+13	DS	61119	7.4500+13	-0	-0
46	R P	1	PUA-6B-1	091267	921	L-10	DS	61119	7.6200+13	BG		7.7200+13	-0	-0
47	R P	1	PUA-6B-1	091967	1830	L-10	DD	60511	7.9900+13	AS	60689	7.9400+13	-0	-0
96	S P	1	PUA-6B	062367	15B4D	L-9	BG	61521	8.4100+13			8.2200+13	0	-0
97	S P	1	PUA-6B	062367	15B4D	L-9	BG	61536	8.4100+13			8.2200+13	0	1
98	S P	1	PUA-6B	062867	15B4E	L-9	BS	61581	8.4100+13			8.0100+13	0	-0
99	S P	1	PUA-6B	062867	15B4E	L-9	BS	61581	8.4100+13			7.9500+13	0	1 REASON-UNKNOWN
100	S P	1	PUA-6B	062967	15B4E	L-9	CG	60360	8.4100+13			8.2600+13	0	-0
101	S P	1	PUA-6B	062967	15B4E	L-9	DD	61119	8.4100+13			8.1650+13	0	-0
102	S P	1	PUA-6B	063067	15B4E	L-9	CG	60360	8.4100+13			8.2300+13	0	-0
103	S P	1	PUA-6B	071167	15B4F	L-9	AG	61483	8.4100+13			8.0100+13	0	-0 UNKNOWN
104	S P	1	PUA-6B	071667	15B4F	L-9	BG	61524	8.4100+13			8.5900+13	0	-0
105	S P	1	PUA-6B	071767	15B4F	L-9	CD	60360	8.4100+13			8.4600+13	0	-0
106	S P	1	PUA-6B	071967	15B4H	L-9	AS	61584	8.4100+13			8.1800+13	0	1
107	S P	1	PUA-6B	072567	15B4I	L-9	CG	61547	8.4100+13			8.2000+13	0	-0
108	S P	1	PUA-6B	072567	15B4I	L-9	AS	61543	8.4100+13			8.2900+13	0	-0
109	S P	1	PUA-6B	072867	15B4I	L-9	BS	61521	8.4100+13			8.3800+13	0	-0
110	S P	1	PUA-6B	080367	27B4G	L-9	DG	61765	8.0500+13			8.1400+13	0	-0
111	S P	1	PUA-6B	080467	27B4G	L-9	BD	61581	8.0500+13			8.0500+13	0	-0
112	S P	1	PUA-6B	080567	27B4G	L-9	CS	60334	8.0500+13			8.1400+13	0	-0
113	S P	1	PUA-6B	080767	27B4G	L-9	BD	61536	8.0500+13			8.0100+00	0	-0
114	S P	1	PUA-6B	081067	71B4A	L-9	AG	61273	8.9200+13			8.8800+13	0	-0
115	S P	1	PUA-6B	081167	71B4A	L-9	CD	61547	8.9200+13			8.7200+13	0	-0
116	S P	1	PUA-6B	081267	71B4A	L-9	CD	61547	8.9200+13			8.8300+13	0	-0
117	S P	1	PUA-6B1	090867	71B4C	L-9	CD	60360	8.9200+13			8.7000+13	0	-0
118	S P	1	PUA-6B-1	090367	71B4C	L-9	DS	61119	8.9200+13			8.8100+13	0	-0
119	S P	1	PUA-6B-1	091967	71B4C	L-9	AG	61543	8.9200+13			8.8500+13	0	-0
120	S P	1	PUA-6B	091267	15B4J	L-9	BG	61742	8.4200+13			8.0700+13	0	-0 ANALYST-ERROR
121	S P	1	PUA-6B-1	091367	15B4J	L-9	BG	61742	8.4200+13			8.0500+13	0	1 ANALYST-ERROR
122	S P	1	PUA-6B-1	091067	15B4J	L-9	CG	60693	8.4200+13			8.1500+13	0	-0
123	S P	1	PUA-6B-1	091467	15B4J	L-9	CG	61273	8.4200+13			8.3300+13	0	-0
222	R P	2	US-1	062167	3797	US-m3	BG	61521	3.3800+00	CD	61547	3.3100+00	-0	-0
223	R P	2	US-1	062267	3910	US-1	CG	61521	3.3400+00	DG	60511	3.6300+00	1	1
224	R P	2	US-1	062267	3910	US-1	CG	61521	3.3400+00	CD	61547	3.6800+00	-0	-0

Page 17

ARR-9

ISOHEM LABORATORY STANDARDS AND REFEREE REPORT 2225 LABORATORY RESULTS PAGE DATE COMPUTED 10-10-67
 EOP CODE 149 METHOD UD-1A EQUIPMENT PIECE C-1 FOR PERIOD 06-21-67 TO 09-20-67

	REFEREE		STANDARDS	
	CURRENT	LONG TERM	CURRENT	LONG TERM
ITEMS SUBMITTED	0		18	
NUMBER AVERAGED	0	0	13	7
NUMBER OF RERUNS	0		0	
CORRECTED TO PLANT	0			
NUMBER OF FLYERS	0		5	
% AVERAGE RANGE	0.0000	0.0000	% AVERAGE RECOVERY 1.0036+02	1.0054+02
VARIANCE	0.0000	0.0000	5.5339-03	2.5000-01
% STANDARD DEVIATION	0.0000	0.0000	7.4390-02	5.0000-01
% PRECISION OF SINGLE OBSERVATION 95% LEVEL	0.0000		(+ OR -) 1.6210-01	
			% PRECISION OF AVG (+ OR -) 4.4957-02	
F VALUE	0.0000	AT 95 LEVEL	4.5176+01	GAIN SIG AT 95 LEVEL
		T VALUE	-9.5160-01	GAIN N/S AT 95 LEVEL

REFEREE DATE - - LONG TERM VALUES WERE ESTABLISHED
 TEST WAS SIGNIFICANT STANDARDS DATE 09-29-67 F TEST WAS SIGNIFICANT

Figure VII

Results Page of LSR Primary Report

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Columns

- 12 and 13 = Referee analyst, shift and payroll number
- Column 14 = Numerical value of analysis of standard or value of referee analysis
- Column 15 = Corrected result reported to processing operation
- Column 16 = Number of reruns
- Column 17 = Any comments by the analyst are reported here.

The results page of the primary report lists averages and statistical values on the submitted data. For each EDP code number for a laboratory there is a results page and a detail page. The EDP code number is used to correlate a sample or standard with the analytical method used. There are 999 permissible codes for each laboratory. (See Figure VII)

- Line 1 In this line appears the name of the report, the laboratory, and the computed data.
- Line 2 The period covered by the report is shown.
- Line 3 The electronic Data Processing (EDP) code number is given. The analytical method code appears next, followed by the equipment piece number. The equipment piece number provides a means for identifying a plant sample and is usually a tank number or a sample point. Where no equipment piece is involved, some other notation is used, such as a repeated method code.
- Line 5 Headings for referee and standard columns appear here. The term "referee" here designates a data category comprised of all original and referee analyses as presented on the "referee" card. "Standards" refers to the completed assay of a standard sample as written on the "standard" card.
- Line 6 Current and Long Term headings for standards and referees appear on this line. "Current" refers to all submitted data covered in the reporting period noted in Line 2.

"Long Term" refers to data accumulated subsequent to a statistically significant change in the "referee" precision, or "standard" bias or precision. This data serves as a basis of comparison for detecting gains or losses in precision in the current data by the "F" test, a description of which appears for Line 28.

- Line 8 The number of acceptable standard and referee cards submitted is noted here.
- Line 10 The number of pieces of data used in the statistical computations is noted as the "number averaged". This number may differ from the number submitted, due to reruns and flyers being excluded from referee computations and second reruns and flyers being eliminated from computations on standards data.
- Line 12 The number of reruns is noted here.
- Line 13 When a referee analysis has shown the original value to be in error, a corrected value is submitted to the processing operation. Such occasions are noted as "corrected to plant".
- Line 15 The number of "flyers" is noted on this line. These are statistical outliers and mistakes, such as incorrect exponents. The routine for discarding flyers is given later. (Page 26)
- Line 18 The percent average range for referees is the average of the percent ranges, which is defined to be:

$$\% \text{ Range} = \frac{(\text{Original} - \text{Referee})}{\text{Original}} \times 100$$

The sign of the individual ranges for a referee is inconsequential. The percent recovery on a standard is defined to be:

$$\% \text{ Recovery} = \frac{\text{Observed Value}}{\text{Standard Value}} \times 100$$

The average percent recovery appears in the standards column.

- Line 20 The variance for an individual analysis appears on this line. For a referee, the variance is computed as the square of the standard deviation. The variance for an individual analysis of a standard is computed as

$$S^2 = \frac{\sum (X - \bar{X})^2}{N - 1} \quad \frac{X^2 - (\sum X)^2 / N}{N - 1}$$

Line 22 The standard deviation is shown here. For referee data, an unbiased estimate of the standard deviation of an individual analysis is computed by multiplying the average percent range by 0.886 (Introduction to Statistical Analysis, Dixon and Massey, 2nd Edition, 1957, p. 404, Table A-8, *b(1)).

The standard deviation of an individual analysis of a standard is the square root of the variance.

Line 24 The percent precision of a single observation at the 95 percent confidence level for standard and referees is computed by multiplying the standard deviation by a factor from the "t" table (See Table I). The degrees of freedom are the number of observations for the referees, but referees and originals are counted as pairs; therefore, this degree of freedom is lost due to replication.

Line 26 The precision of the average recovery is the precision of a single observation at the 95 percent confidence level (Line 24) divided by the square root of the number averaged. For the given reporting period, the average recovery, with 95 percent confidence, would fall between the extremes of the precision of the average.

Line 28 The "F" value for both standards and referees is computed as the ratio of the current and long term variances, with the variance with the larger numerical value in the numerator. The computed value is compared with the value found in the .05 "F" table using the number averaged minus one as the degrees of freedom. The "F" value will be followed by a notation of "gain" or "loss" over the long term data and will also be noted as to significant (Sig.) or not significant (N/S).

Example:

$$\frac{S^2 \text{ current}}{S^2 \text{ Long Term}} = \frac{42.337}{7.259} = 5.83 \quad \frac{\text{Of current}}{\text{Of Long Term}} = \frac{115}{9}$$

From the Table F = 2.75. As 5.83 is greater than the table value, a loss in precision significant at the 95 percent confidence level has occurred. In other words, the current data are significantly less precise than the long term data.

TABLE 1

<u>df</u>	<u>t.975</u>
1	12.706
2	4.303
3	3.182
4	2.776
5	2.571
6	2.447
7	2.365
8	2.306
9	2.262
10	2.228
11	2.201
12	2.179
13	2.160
14	2.145
15	2.131
16	2.120
17	2.110
18	2.101
19	2.093
20	2.086
21	2.080
22	2.074
23	2.069
24	2.064
25	2.060
26	2.056
27	2.052
28	2.048
29	2.045
30	2.042
40	2.021
60	2.000
120	1.980
∞	1.960

If more than 20 pieces of data are current when a significant change in variance occurs, then this number will be input as the long term number averaged. If there are fewer than 20 pieces of current data when a significant change occurs, subsequent data will accumulate, lot by lot, until more than 20 observations comprise the long term number averaged.

Line 30

On standards data a "t" test is performed to determine if a significant change in the average recovery has occurred. In order to estimate the degree of freedom in the "t" test, it is necessary to know if the "F" test was significant or not.

If the "F" ratio was not significant, the "t" test is computed as follows:

$$t = \frac{\bar{X} \text{ Long Term} - \bar{X} \text{ Current}}{S_p \sqrt{\frac{1}{N} + \frac{1}{N}}}$$

Where: $\bar{X}$ = Average recovery

N = Number of long term observations

n = Number of current observations

S_p = Standard deviation of pooled variance

and the pooled variance, S_p^2 is found as follows:

$$S_p^2 = \frac{(n-1)S^2_{\text{Current}} + (N-1)S^2_{\text{Long Term}}}{n + N - 2}$$

The degrees of freedom equals $n + N - 2$.

If the "F" ratio was significant, the "t" test is computed as

$$\frac{\bar{X} \text{ Long Term} - \bar{X} \text{ Current}}{\frac{S^2_{\text{Long Term}}}{N} + \frac{S^2_{\text{Current}}}{N}}$$

The degrees of freedom for "t" in this case is calculated from:

$$\frac{\left[\left(\frac{S^2_{\text{Long Term}}}{N} \right) + \left(\frac{S^2_{\text{Current}}}{n} \right) \right]^2}{\frac{\left(\frac{S^2_{\text{Long Term}}}{N} \right)^2}{N-1} + \frac{\left(\frac{S^2_{\text{Current}}}{n} \right)^2}{n-1}}$$

The "t" value is compared with the value appearing in the "t" table at the 95 percent level. A significant (Sig.) or not significant (N/S) gain or loss is recorded.

Lines 32 & 33 The dates of the last significant change in referee or standard variance or in the standard average are given here. On these dates, the Long Term data was replaced.

c. The Detail Page of the Primary Report

For each EDP code number for a laboratory there is a "detail" page as well as a results page. A listing of each individually computed referee range and each individual standard percent recovery is listed. (See Figure VIII)

- Line 1 The name of the report, the laboratory submitting the data and the compilation date are shown here.
- Line 2 The period covered by the report is given.
- Line 3 The EDP code number, analytical method, and material being assayed (equipment piece) and abbreviations used for column headings are noted.
- Line 7 Headings for standard or referee data appear here. Referee data is listed first. If no referee data is available, the standards data is listed.
- Line 8 Column headings for referee data listing are shown. The data listing consists of a serial number; material analyzed; original analyst's payroll number, shift, and result; the percent range for each referee pair; the date; NA for not averaged; CR for correction reported; RR for rerun; and comments. In the NA column a 0 is printed for acceptable data and a 1 appears if

SOCHEM LABORATORY STANDARDS AND REFEREE REPORT 202A LABORATORY DETAIL REPORT DATE COMPUTED 10-10-67
FOR PERIOD 06-21-67 TO 09-20-67

DP CODE 31 METHOD HV-1A EQUIPMENT PIECE J5A ABBREVIATIONS. MATL-MATERIAL, PR-PAYROLL, E-EXPONENT
NA-NOT AVERAGED (FLYERS), RR-RERUNS
CR-CORRECTION REPORTED

REFEREE DATA				ORIGINAL ANALYST		REFEREE ANALYST				% RANGE	DATE	NA	CR	RR	COMMENTS
SERIAL	MATL	PR NO	SHIFT	RESULT	E	PR NO	SHIFT	RESULT	E						
5967	J5A	60361	DS	2.7300+00		61291	AG	2.0700+00		24.176	07-14-67	1	1	1	ANALYST-ERROR
8772	J-5A	61521	CS	1.9100+00		61547	AG	1.9100+00		.000	08-09-67	0	-0	-0	
523	J5A	61543	AG	1.8600+00		61842	CD	1.8600+00		.000	09-08-67	0	-0	-0	
1653	J5A	61246	CG	1.9900+00		60415	DD	1.9800+00		.503	09-18-67	0	-0	-0	
6475	J5A	61119	DS	2.2000+00		61536	BG	2.2200+00		.909	07-18-67	0	-0	-0	
6022	J-5A	61765	AG	2.0200+00		60360	CD	2.0000+00		.990	07-14-67	0	-0	-0	
8595	J5A	61536	ED	1.9300+00		61521	CS	1.9000+00		1.554	08-07-67	0	-0	-0	
7481	J5A	61584	DD	1.9600+00		61521	BS	1.9200+00		2.041	07-27-67	0	-0	-0	
9119	J5A	61547	CD	1.9200+00		61768	DS	1.8800+00		2.083	08-11-67	0	-0	-0	
804	J5A	61119	DS	1.8900+00		61581	BG	1.8500+00		2.116	09-11-67	0	-0	-0	
1240	J-5A	60360	CC	1.9600+00		60689	AS	2.0100+00		2.551	09-14-67	0	-0	-0	
5967	J5A	60361	DS	2.7300+00		61765	AG	2.1000+00		23.077	07-14-67	1	-0	-0	ANALYST-ERROR

STANDARD DATA				ANALYST		STANDARD		% RECOVERY	DATE	NA	RR	COMMENTS
SERIAL	MATL	PR NO	SHIFT	RESULT	E	VALUE	E					
49J	HV-1A	61119	DD	1.5840+00		1.5990+00		99.062	06-24-67	0	-0	
49J	HV-1A	61581	BS	1.5840+00		1.5990+00		99.062	07-02-67	0	-0	
49L	HV-1A	60415	DD	1.5600+00		1.5590+00		100.064	09-15-67	0	-0	
49J	HV-1A	61765	AS	1.6040+00		1.5990+00		100.313	06-26-67	0	-0	
49L	HV-1A	60693	CG	1.5650+00		1.5590+00		100.385	09-16-67	0	-0	
49K	HV-1A	61547	CG	1.6080+00		1.5990+00		100.563	07-27-67	0	-0	
49K	HV-1A	61521	CS	1.6100+00		1.6000+00		100.625	07-30-67	0	-0	
49K	HV-1A	61524	BG	1.6100+00		1.6000+00		100.625	08-13-67	0	-0	
49K	HV-1A	61521	CS	1.6100+00		1.5990+00		100.688	08-07-67	0	-0	
49J	HV-1A	61536	BD	1.6100+00		1.5990+00		100.688	07-11-67	0	-0	
49K	HV-1A	61584	AD	1.6120+00		1.5990+00		100.813	07-28-67	0	-0	
49-K	HV-1A	61765	DS	1.6200+00		1.5990+00		101.313	08-02-67	0	-0	
49J	HV-1A	60360	CC	1.6200+00		1.5990+00		101.313	07-15-67	0	-0	
49L	HV-1A	61742	BS	1.5900+00		1.5600+00		101.923	08-28-67	0	-0	
49J	HV-1A	61360	CC	1.6300+00		1.5990+00		101.939	07-17-67	0	-0	
49L	HV-1A	60693	CG	1.6200+00		1.5590+00		103.913	08-21-67	1	-0	
49K	HV-1A	61547	AG	1.6900+00		1.5990+00		105.691	08-03-67	1	-0	UNKNOWN
49L	HV-1A	61765	AS	1.6000+00		1.5590+00		107.761	09-16-67	1	-0	

the sample is a rerun or a flyer. The CR column notes those occasions where a referee analysis brought about a correction of the original value reported to the processing operation. The RR column lists the number of reruns.

When the listing of referee data has been completed, all the standards data for that code are listed.

The data listing for standards consists of the serial number of the standard; the material; the analyst's payroll number, shift and result; the standard value; the computed percent recovery; the date; NA for data that has not been averaged; RR for rerun data; and comments. In the NA column a 0 is printed for accepted data and a 1 appears for flyers or second reruns.

All the data is listed with excluded reruns first and the remaining data listed in order of magnitude of percent range for referees and percent recovery for standards. Flyers and wide range values are easily noted at the top and bottom of the columns.

Flyers

In the past, the philosophy has been to err in the direction of retaining data. Flyers were discarded based on a flyer limit set from a year's data. Only one pass was made to discard flyers, and the 99.9 percent confidence level was used. Present practice includes deleting all flyers by making as many computations as necessary to delete them and using the 90 percent confidence level.

The present system for determining flyers is divided into two sets for both the referee and the standard data. One set is for twenty-six or more observations, and the second deals with from three to twenty-six observations.

Referee Flyers

(A) Twenty-six or more observations:

- (1) Discard all data with a percent range equal to or greater than 100 percent.
- (2) Discard flyers on remaining data based on a 90 percent acceptance "q" factor. (See Table 2)

(a) Compute average percent range = $\bar{X}$

- (b) Compute $S = (0.886)\bar{X}$
 - (c) Discard data where percent range is greater than $(q\sqrt{2})S$
 - (d) Repeat a, b, and c until there are no more flyers or until only two observations remain.
- (3) Record the number of flyers.
- (B) Three to twenty-six observations:
- (1) Discard all data with a percent range equal to or greater than 100 percent.
 - (2) Discard flyers on remaining data, using the gap over range test at the 90 percent acceptance level, repeating until no further flyers occur or until only two pieces of data remain. See Table 3 for gap over range.

Standards Flyers

- (A) Twenty-six or more observations:
- (1) Discard any results as flyers with less than one percent recovery or greater than 200 percent recovery.
 - (2) Discard flyers on remaining data using a 90 percent acceptance "q" factor. (Table 2)
 - (a) Compute average percent recovery = $\bar{X}$
 - (b) Compute $S^2 = \frac{\sum X^2 - \frac{(\sum X)^2}{N}}{N-1}$
 - (c) Compute S
 - (d) Discard all results above and below $\bar{X} \pm qs$
 - (e) Repeat until no more flyers occur or until only two results remain.
- (B) Three to twenty-six observations:
- (1) Discard any results as flyers with less than one percent or greater than 200 percent recovery.

TABLE II

<u>90 Percent Acceptance of Factor</u>			Two-sided normal t
<u>n</u>	<u>90% Ac</u>		<u>q</u>
26	99.596		2.875
30	.649		.920
35	.699		.967
40	.737		3.008
50	.790		.075
75	.860		.194
100	.895		.276
150	.930		.389
200	.947		.467
400	.974		.649
600	.992		.752
1000	.995		.878

TABLE III

Gap Over Range Table

* CRITICAL VALUES AND CRITERIA FOR TESTING FOR EXTREME VALUES

α N	.30	.20	.10	.05	.02	.01	.005	Criterion
3	.684	.781	.886	.941	.976	.988	.994	$r_{10} = \frac{x_n - x_{n-1}}{x_n - x_1}$
4	.471	.560	.679	.765	.846	.889	.926	
5	.373	.451	.557	.642	.729	.780	.821	
6	.318	.386	.482	.560	.644	.698	.740	
7	.281	.344	.434	.507	.586	.637	.680	
8	.318	.385	.479	.554	.631	.683	.725	$r_{11} = \frac{x_n - x_{n-1}}{x_n - x_2}$
9	.288	.352	.441	.512	.587	.635	.677	
10	.265	.325	.409	.477	.551	.597	.639	
11	.391	.442	.517	.576	.638	.679	.713	$r_{21} = \frac{x_n - x_{n-2}}{x_n - x_2}$
12	.370	.419	.490	.546	.605	.642	.675	
13	.351	.399	.467	.521	.578	.615	.649	
14	.370	.421	.492	.546	.602	.641	.674	$r_{22} = \frac{x_n - x_{n-2}}{x_n - x_3}$
15	.353	.402	.472	.525	.579	.616	.647	
16	.338	.386	.454	.507	.559	.595	.624	
17	.325	.373	.438	.490	.542	.577	.605	
18	.314	.361	.424	.475	.527	.561	.589	
19	.304	.350	.412	.462	.514	.547	.575	
20	.295	.340	.401	.450	.502	.535	.562	
21	.287	.331	.391	.440	.491	.524	.551	
22	.280	.323	.382	.430	.481	.514	.541	
23	.274	.316	.374	.421	.472	.505	.532	
24	.268	.310	.367	.413	.464	.497	.524	
25	.262	.304	.360	.406	.457	.489	.516	

* Taken from "Processing Data for Outliers" by W. J. Dixon. Biometrics 9:1 pages 74-89 March 1953.

- (2) Discard remaining flyers, using 90 percent acceptance gap over range or until only two pieces of data remain.

d. The Summary Page

A summary page is supplied for each laboratory. An illustration is provided in Figure IX. The building number of the laboratory and the date the program was executed are given as a heading to this report. Referee and standard results are summarized in separate columns. The EDP cards, methods, and sample identification are listed in numerical order of EDP code.

The referee summary report lists the number of pieces of data used for statistical computations under the heading of number averaged. In the next column appears the number of flyers for a given method. The number of reruns not included in the statistical data and the number of corrections reported to the processing operation due to the sample being refereed are shown in the next two columns. The sum of the number averaged, number of flyers, and number of reruns indicates the total number of analyses reported.

For convenient comparisons, the current and Long Term average percent range and percent standard deviation are listed in columns side by side. The results of the "F" test for precision of analyses is shown with a "G" or "L" for Gain or Loss and "S" or "NS" for statistically significant or Not Significant.

The standards summary records the number averaged, the number of flyers, and the number of second reruns. The sum of these three columns is the number of pieces of data submitted for computation. The current and long term percent recovery and percent standard deviation are again listed side by side for comparison convenience. The results of both the "F" test and the "t" test appear in the column headed FT and TT. A "G" or "L" represents either a gain or a loss and "S" or "NS" appears for statistically significant or not significant.

The abbreviated column headings are defined at the bottom of the page.

Occasionally errors arise in the primary report due to improper entries on the data card or illegible writing which leads to a key punch error. These errors need to be removed from or corrected in the master file; otherwise, erroneous long term values would occur and would

be retained in the quarterly report. Two nonroutine cards titled "Delete or Update Cards" have been provided to correct these results in this program. The Update Cards for Referees and Standards are illustrated in Figures X and XI.

In making any changes on the Master File, a match must be made on the update card and the master file between the record number obtained from the latest master file listing, the code for standard or referee, the building code, the method code, and the date.

The standard and referee update cards are to be filled out in the same manner as the original data cards with the corrected information input on the update card.

In order to delete an entry from the master file, it is only necessary to enter the appropriate data in card columns 1 through 5, 17 through 22, and 74 through 79. A "D" should appear in Column 79 if the data is to be deleted. No entry should be input in Column 79 for an update.

2. The Quarterly Report

The Quarterly Report is issued in two parts. The first part is a three-month compilation of the primary reports. The second part is received as a duplimat for use in reporting laboratory performance in a concise report. The format of the quarterly duplimat is illustrated in Figure XII.

Two headings that appear in this report are not found in the primary report. These are DET-N for determination, indicating the entity being determined, and analytical method, giving the category of the analytical practice, e.g. spectrophotometric or colorimetric analyses.

The precision of an individual analysis at the 95 percent confidence level is listed in percent for the referees.

The average percent recovery and the precision of the average in percent are shown for standards. The precision for a single observation may be obtained by multiplying the precision of the average by the square root of the number of samples in the number averaged column.

Since information appears in the quarterly duplimat report that is not available in the primary report, a Title Card (Figure XIII) is needed for each EDP Code number. These cards may be submitted as either new title data or as an update to existing titles. In the event an EDP code is deleted, the title card may also be deleted.

234-5 LABORATORY STANDARDS AND REFEREE SUMMARY REPORT

DATE COMPUTED 10-10-67

UNCLASSIFIED

EDP CODE	METHOD	SAMPLE	REFEREE PROGRAM								STANDARDS PROGRAM							
			NO	NO	NO	NO	AVG	%	RANGE	%	STD	DEVIATION	FT	NO	NO	NO	%	STD
			AVG	FLY	RR	CR	CURRENT	LONG	T	CURRENT	LONG	T		AVG	FLY	RR	CURRENT	LONG
175	PUA-6D	CAX	25	5	6	1	8.2347	15.6315		7.298	13.947	G S		36	1	0	103.620	106.227
176	PUA-6B-3	MT	22	4	4	3	1.9973	3.5035		1.770	3.105	G S		0	0	0	.000	.000
180	PUA-6B	COP	0	0	0	0	.0000	.0000		.000	.000			31	1	0	100.058	99.940
181	PUA-6D	AAP	24	4	5	1	5.3668	4.6455		4.756	4.326	LNS		0	0	0	.000	.000
182	PUA-6D	CWX	24	-0	0	0	3.8659	4.3698		3.426	3.917	GNS		0	0	0	.000	.000
183	PUA-6B	RCR	11	2	5	1	3.4983	4.4414		3.100	3.967	GNS		33	2	0	101.184	101.111
185	PUX-4A-1	PU-200	2	-0	0	0	.3526	.8535		.313	.771	GNS		47	1	0	99.926	99.599
192	DW-2A	SPGSTD	24	3	6	2	.2512	.3916		.223	.347	GNS		95	4	2	100.058	99.993
193	DV-2A-2	CAX	23	3	4	1	.1817	.4534		.161	.402	G S		0	0	0	.000	.000
194	CD-SPEC	NA-FL	0	0	0	0	.0000	.0000		.000	.000			21	-0	0	106.355	99.631
195	HV-3E	PU-200	23	3	3	2	1.9889	1.9916		1.763	1.775	GNS		53	-0	0	100.124	99.761
196	CD-SPEC	NA-CL	0	0	0	0	.0000	.0000		.000	.000			19	2	0	95.892	96.800
197	FES-1B	FESTD	2	-0	0	0	.6329	1.0256		.561	1.245	GNS		9	2	0	101.931	102.557
198	CZ-1B	CARBON	14	-0	4	0	17.9576	13.7350		15.914	12.172	LNS		17	-0	0	92.995	96.596
199	CF-1B	USTD	1	-0	0	0	.0000	12.1651		.000	11.364	GNS		28	-0	0	98.879	100.196
200	F-1A	GA	0	0	0	0	.0000	.0000		.000	.000			14	-0	0	100.350	102.561
201	X-RAY	MT	3	-0	2	1	5.3236	6.1057		4.718	5.411	GNS		0	0	0	.000	.000
202	X-RAY	CAIS	23	-0	4	1	1.3497	1.2934		1.196	1.157	LNS		23	1	0	100.783	99.767
204	M-SPEC	PU-239	0	0	0	0	.0000	.0000		.000	.000			18	1	0	100.052	100.004
205	M-SPEC	PU-240	0	0	0	0	.0000	.0000		.000	.000			17	2	0	99.895	100.164
206	M-SPEC	PU-241	0	0	0	0	.0000	.0000		.000	.000			18	1	0	95.506	95.245
207	M-SPEC	PU-242	0	0	0	0	.0000	.0000		.000	.000			18	1	0	101.010	100.963
208	M-SPEC	U-234	0	0	0	0	.0000	.0000		.000	.000			2	2	0	120.000	140.000
209	M-SPEC	U-235	0	0	0	0	.0000	.0000		.000	.000			2	2	0	100.505	100.713
210	M-SPEC	U-2360	0	0	0	0	.0000	.0000		.000	.000			2	2	0	114.286	122.857
211	M-SPEC	U-238	0	0	0	0	.0000	.0000		.000	.000			4	-0	0	99.998	99.997
214	AEA	PU-RA	9	-0	0	0	3.0925	1.2861		2.741	1.140	LNS		60	1	0	98.678	98.849
215	AEA	AM-RA	5	-0	0	0	2.4087	4.3025		2.135	3.813	GNS		59	2	0	100.292	100.450
216	PUA-2UE	PUAM	25	2	3	2	6.7263	25.9596		5.961	23.005	G S		26	2	0	96.635	98.515
217	CF-1B	RCR	0	0	0	0	.0000	.0000		.000	.000			39	-0	0	109.393	120.325
218	CF-1C	U-ORG	0	0	0	0	.0000	.0000		.000	.000			9	1	0	107.453	115.112
220	CA-2	PU-200	1	-0	0	0	.1575	.1575		.140	.140	GNS		39	2	0	100.168	100.159
221	LK-2	CAIS	0	0	0	0	.0000	.0000		.000	.000			1	-0	0	.000	.000

SYMBOLS FLY-FLYERS RK-RERUN'S CR-CORRECTIONS REPORTED FT-F TEST TT-T TEST G-GAIN L-LOSS N-NOT S-SIGNIF

FIGURE IX

Summary Page of ICR Primary Report

1000-10

1	2	3	4	5	6	METHOD					DATE				SERIAL NO				MATERIAL											
R																														

SHIFT	O. ANALYST PR NO.			NEG	REPORTED RESULT			EXP		
35	36	37		41	42	43		47	48	49

SHIFT	REF. ANALYST			NEG	REF. RESULT			EXP		
50	51	52		56	57	58		62	63	64

CORR	RR
65	66

RECORD NO	
74	79

80
U

79 = D FOR DELETE - OTHERWISE BLANK
 DELETE OR UPDATE CARD FOR LSR MASTER FILE
 A-6300-012 (3-67)

FIGURE X

The referee Delete or Update Card is printed on a white card with a yellow edge.

1	2	3	4	5	6	METHOD					DATE				STANDARD NO				MATERIAL											
S																														

SHIFT	P.R. NO.			NEG	STD VALUE			EXP	NEG	RESULT			EXP			
35	36	37		41	42	43		47	48	49	50	51		55	56	57

RR
58

RECORD NO	
74	79

80
U

79 = D FOR DELETE - OTHERWISE BLANK
 DELETE OR UPDATE CARD FOR LSR MASTER FILE
 A-6300-013 (3-67)

FIGURE XI

The standard Delete or Update Card is printed on white with a blue edge.

SP CODE	METHOD CODE	MATERIAL	DET-ON	ANALYTICAL METHOD	NO	AVG	% RECOVERY	PRECISION	95% LEVEL
1	REF; STD.	PUA-6B-1	L-9 L-10 L-9	ALPHA RADIOASSAY ALPHA RADIOASSAY	14 18		99.558	OF INDIV. OF AVG.	3,883 % .660 %
2	REF; STD.	US-1A	D-5DIL D-5DIL	U SPECTROPHOTOMETER U SPECTROPHOTOMETER	29 99		100.633	OF INDIV. OF AVG.	7,279 % .248 %
3	STD.	PUA-6B-4	D-5	ALPHA RADIOASSAY	19		98.574	OF AVG.	9.127 %
4	STD.	PUA-6B-5	D-5DIL	ALPHA RADIOASSAY	54		99.630	OF AVG.	.395 %
5	REF.	SR-4X-W	E-5	SR BETA COUNT	15			OF INDIV.	11.441 %
6	REF; STD.	PUA-20A	F-15, 16, 17 F-15-2	PU RADIOASSAY PU RADIOASSAY	6 9		97.536	OF INDIV. OF AVG.	26,544 % 4.785 %
7	REF; STD.	PUA-30A	K-6 K6-2	PU RADIOASSAY PU RADIOASSAY	6 10		97.254	OF INDIV. OF AVG.	18,954 % 3.283 %
8	REF.	CEG-2A-W	E-5	CE SCAN	18			OF INDIV.	19.940 %
9	REF; STD.	PUX-3A	L-9 L-10 L-9	PU X-RAY PU X-RAY	14 23		100.760	OF INDIV. OF AVG.	.869 % .488 %
10	STD.	NPA-6A	K6-2	NP RADIOASSAY	8		92.673	OF AVG.	3.242 %
12	STD.	NPA-40B	F-15	NP RADIOASSAY	5		82.509	OF AVG.	3.402 %
13	REF; STD.	UX-2A	K-6 K-6	U X-RAY U X-RAY	15 22		100.472	OF INDIV. OF AVG.	.991 % .182 %
14	REF; STD.	NPA-5A	3XF 3BY 3XF	NP RADIOASSAY NP RADIOASSAY	13 19		109.316	OF INDIV. OF AVG.	5,065 % 10.410 %
15	REF; STD.	NPA-40A	3AW 3AW	NP RADIOASSAY NP RADIOASSAY	6 13		86.839	OF INDIV. OF AVG.	18.997 % 3.215 %
16	STD.	NPA-5A	3XW	NP RADIOASSAY	23		99.278	OF AVG.	1.049 %
17	STD.	UF-1R	UFSTD2	U FLUORIMETER	7		85.034	OF AVG.	17.786 %
18	REF.	UF-1R	L-10	U FLUORIMETER	6			OF INDIV.	48.414 %

Any modification to titles on file must have matching values in columns 1 through 5, and if the title is to be deleted, a "D" must appear in column 41.

CODE			MATERIAL										DETH								
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
ANAL. METHOD																					
23																				40	41
																					80
																					T

1 = R OR S, FOR REFEREE OR STANDARD

2 = BUILDING R P OR Z

41 = D FOR DELETE - OTHERWISE BLANK

THIS CARD IS FOR MAKING ENTRIES OR DELETIONS FOR QUARTERLY LSR DUPLIMAT TITLES.

A-6200-015 (3-67)

FIGURE XIII

The Title Card is printed on white with a green edge.

3. The Bias Report

The "Bias" Report is formally known as "CPD Analytical Data Corrections to be Applied to Accountability Source Data". Figure XIV illustrates this report. This report shows the average percent recoveries, the number of standards averaged, and the precision of the average at the 95 percent confidence level on those standard solutions that monitor accountability points.

It is accepted that actual routine laboratory results may have some error or bias and may be corrected accordingly. Laboratory values should be divided by the decimal equivalent of the percent average recovery to compensate for the error.

A preliminary report is issued on or before the first of each month covering data for the preceding period from the 21st to the 21st calendar day. The performance on standards is considered consistent enough so that an average for that period can safely represent a calendar month.

In both the preliminary and final reports, the samples monitored are designated by an equipment piece number. The EDP code and method are listed for each sample being monitored by a standard.

The complete report is issued when data on a D-5 (D11) standard is available from the 202-A Laboratory. This data includes the recovery on an americium standard so that a correction is given in a footnote to the report.

Special data reduction reports requests, such as listings by date or by shift, should be referred to the CSC programmer in charge of maintaining the LSR program. If any problems are involved, the technical systems specialist of Management Systems Operation should be contacted.

Any new referee or standard items may be entered into the LSR Program by assignment of an available EDP code number, filling out data cards, and transmitting these cards in the usual manner for the primary report. For a new code to appear in the quarterly report, a "title" card must be submitted. This new entry will not have long term data available for "F" to "t" tests. As data is accumulated, the long term file generates long term data as described for the primary report.

FIGURE XIVCPD Analytical Control Data Corrections
to be Applied to Accountability Source Data

<u>Bldg.</u>	<u>Sample</u>	<u>Code</u>	<u>Method</u>	<u>N</u>	<u>% Average Recovery and Precision of Avg. (95)</u>
234-5	CCP, Hood 4 (1)	220	DK-2	11	100.57 $\pm$ 0.67
		192	SpG	25	99.96 $\pm$ 0.10
	RCR	183	AT	13	100.58 $\pm$ 1.05
		192	SpG	25	99.96 $\pm$ 0.10
URO	C-1, X-1, X-2	149	UD-1A	5	100.31 $\pm$ 0.10
	X-36, X-37	160	SpG	6	100.11 $\pm$ 0.03
	X-19, X-30				
Purex	L-9	001	AT	9	96.77 $\pm$ 1.06
		009	X-ray	8	99.28 $\pm$ 0.52
		042	SpG	11	100.01 $\pm$ 0.09
	K-6, UNH trailer Tube 4	013	X-ray	13	98.51 $\pm$ 0.65
		043	SpG	14	99.93 $\pm$ 0.04
	D-5 DIL	004	AT	18	99.66 $\pm$ 0.88
		023	UC	4	101.77 $\pm$ 0.40
					(2)

Notes:

N Number of data observed

(1) Apply correction only when sample is greater than 30 g/l.

(2) A 30.4 percent correction for AmCm should be applied.