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TO: To Those Listed Below

FROM: M. T. Kelley

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For The Atomic Energy Commission

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CONFIDENTIALABSTRACT

Charts and summaries of the control results for Departments 3290 and 4270 of the Analytical Chemistry Division are presented for the first quarter of this year. The control programs in Department 3290 remained essentially the same as those for previous periods. In general, the precision was maintained in these laboratories and such biases as were found to exist were eliminated. In Part I of this report, the control work for Department 3290 is presented and discussed.

The control work in Department 4270 was reviewed and re-evaluated, during this period, to determine what changes are necessary to improve the effectiveness of the control program. The results of this review are discussed and the reasons for certain revisions are presented in Part II of this report.

Provision has been made for the presentation of experimental data which is pertinent to, but is not a part of, the control program. Experimental data for the determination of molybdenum, uranium and vanadium as well as for the analysis of NaZrF_5 are given in Part III of this report.

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JANUARY THROUGH MARCH, 1956

INTRODUCTION

In the growth and development of a Quality Control Program, such as that now in effect in the laboratories of the Analytical Chemistry Division, the fundamental purpose of the program sometimes becomes distorted or even forgotten. Certain misconceptions arise which cause misunderstandings and controversies. Recognizing that this control program has been in effect for over five years and that the scope has been greatly expanded to meet the changing needs of the laboratories, a re-statement of the fundamental purposes as well as a discussion of certain phases of the program seems to be in order.

Perhaps the most general misconception is that a control program which is based on the analysis of standard materials will serve to establish the precision of test results for all types of samples that are analyzed by a particular method; that is not always the case. Actually, a control program that is based on the analysis of standard materials serves two fundamental purposes which are:

- (1) To establish the precision of analytical methods, and
- (2) To determine the existence of a bias in a method.

These two functions should not be confused because the precision of a method, under normal conditions, is constant; whereas, a bias is a variable factor within the method which should be eliminated as soon as possible after it is detected.

After the precision of a method has been established by means of standards, and biases have been eliminated, the reproducibility of test results for a particular type of sample can be ascertained from replicate analyses of a typical material. The number of replicates should not exceed four, but each determination should be made at a different time, preferably on a different day. From this data the precision for other types of samples can be established.

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Other functions of the control program are:

- (1) To assist the supervisor in the maintenance and control of the quality and quantity of the work in the laboratory, and
- (2) To alert supervision to the development of an "out-of-control" condition or of a bias.

In order to determine whether all variables within a method are properly controlled, standard materials and samples should be tested at the same time. The test results for the standards should be reported for control purposes and should be shown on the control charts for that analysis. From this data, the supervisor can determine whether remedial action is necessary to maintain the precision and accuracy of the test results. The analysis of standards when samples are not being tested should not be considered as control data and ordinarily should not be reported for that purpose. Since standards are to serve as an independent control of a method, the use of this material for calibration purposes is not recommended.

When standards are tested to determine whether a measuring device is functioning properly, whether reagents have deteriorated, or whether some other variable of the method is under control, the results are not properly considered to be control data. The information derived from the tests, however, is pertinent to the control program and should be reported. Data of this type should not be shown on control charts.

The analysis of standards for the sole purpose of providing data for a control chart is a waste of time because such data cannot be used for control purposes. A chart of such data is not a control chart but merely a graphic presentation of test results which were obtained from the analysis of standards. Control charts are intended to provide a visual means of evaluating the day-to-day variation in a particular method of analysis to ensure that the precision of the operations of the laboratory is being maintained. The use of data which is not directly related to the analysis of samples, therefore, is incompatible with the basic purpose of the control charts.

A Quality Control program to be effective must be conducted independently and without bias, the limitations of the control techniques must be recognized, and such aids as control standards and control charts must be properly used.

Inasmuch as the Quality Control Program for the Analytical Chemistry Division is maintained in two departments, 3290 and 4270, which are separated geographically, certain changes have been made so that the information derived from this program will be more readily available to the supervisors of these departments. Therefore, for convenience and to ensure that the control charts are accessible at all times to the supervisors of department

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3290, the control charts will be maintained, the control data will be tabulated, and the necessary computations will be made within that department. Except for this change in the procedure for handling of the data for department 3290, the operation of the control program for the division will continue in the usual manner.

During this quarter, 3,145 determinations were evaluated for control purposes. The distribution, by laboratory, of these results is given in Table I. A similar distribution of the results for the fourth quarter of 1955 is also included in this table.

Table I

Distribution of Control Determinations

<u>Laboratory</u>	<u>Determinations Evaluated for Control Purposes</u>	
	<u>Fourth Quarter 1955</u>	<u>First Quarter 1956</u>
<u>Dept. 4270</u>		
Bldg. 9201-2	192	235
Bldg. 9204-1	179	179
Bldg. 9733-4	68	439 60 474
<u>Dept. 3290</u>		
Bldg. 3019	1,813	1,877
Bldg. 3508	50	54
Bldg. 3550	204	240
Bldg. 4500	401	432
General Analytical	66	2,534 68 2,671
Grand Total	2,973	3,145

A slight increase in the number of determinations that were reported for control purposes in the laboratories of Department 3290 was noted for this period, but in general, the control program for these laboratories remained essentially the same as in previous quarters. Summaries of these test results are given in Tables I-1 through I-11. These same results are shown graphically in Figures 1-1 through 1-17. In general, little change in precision was noted, although trends to high and low values were observed during the quarter. A discussion of the control data for the laboratories of Department 3290 is given in Part I of this report.

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During this period, the control work in the various laboratories of Department 4270 was reviewed and re-evaluated to determine whether the control programs for these laboratories were commensurate with their needs.

As a result of this re-evaluation, a different type of program was recommended for the determination of uranium in the fluorometric range; this will be discussed later. An expansion of the control program in Building 9733-4 to include the spectrophotometric determination of Mo, Al, and V, as well as the volumetric determination of Al, V, and U was proposed. At this time, only the spectrophotometric determination of molybdenum has been included in the control program. The others will be added as soon as possible.

The programs in the laboratories of Buildings 9201-2 and 9204-1 were not revised. Although new standards were provided for use in Building 9201-2 about the first of the year, and the control solutions in Building 9204-1 will be replaced sometime in April or May, this does not constitute a revision in the control program because the standards are replaced periodically in all laboratories.

The use of a solid material as a control standard in Building 9201-2 was suggested because many solid materials are being tested by this laboratory. The control program will be expanded to include such a standard, possibly, during this next quarter.

In Part II of this report, the results of the control tests for department 4270 are summarized and discussed. Summaries of the data are given in Tables II-1 and II-2. These same results are shown graphically in Figures 2-1 through 2-9.

As was mentioned earlier, when standards are tested to determine whether a measuring device or some other variable in a method is functioning properly, the results are not considered to be control data; but the information derived from these tests is pertinent to the control program. The data and the decisions that are reached as a result of such tests do have an important place in a Quality Control Program. For this reason, if data of this type is reported it will be evaluated and presented in Part III of this and future issues of the "Statistical Quality Control" Report for the Analytical Chemistry Division.

In Part III of this report, experimental data for the determination of molybdenum, vanadium, and uranium has been evaluated. The results of the evaluations are given and discussed.

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Part I: Department 3290

B. J. Ginocchio, J. H. Edgerton and L. T. Corbin

Building 3019

The control results which were reported by the laboratory in Building 3019 for the determination of thorium and uranium, in the fluorometric and colorimetric ranges, are summarized in Tables I-1 through I-4. These results are shown graphically in Figures 1-1 through 1-4.

Thorium. The precision of the control results for the determination of thorium at two concentration levels remained the same as that observed for earlier periods. The negative bias that existed during the last quarter for the determination of thorium at the lower concentration level of ~ 0.3 mg/ml was eliminated early in this quarter. This is shown by the control charts of Figure 1-1.

The existence of a positive bias was found in the determination of thorium at a concentration level of ~ 266 mg/ml during January and February of this quarter. This positive bias was eliminated in March and a trend to low values was observed. This is confirmed by the control charts of Figure 1-2.

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CONTROL CHARTS, 1956

Bldg. 3019

Fig. 1-1. Thorium; Thoron-Colorimetric Method. Low Thorium Concentration.

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CONTROL CHARTS, 1956

Fig. 1-2. Thorium; Thoron - Colorimetric Method. High Thorium Concentration.

Bldg. 3019

CONFIDENTIALTable I-1Thorium by the Colorimetric, Thoron MethodSummary of Control Results

Building 3019

Quarter 1956	Shift	Concentration Range mg/ml	Number Determi- nations N	Number Determi- nations Outside L ₂	Limit of Error		Bias
					L ₁ Per Cent	L ₂	
First	A	0.29 to 0.32	132	8	2	2	None
	B		134	10	2	2	None
	C		138	6	2	2	None
	D		138	8	2	2	None
1955 Fourth	Combined		526	28	2	2	Negative
1956 First	Combined		542	32	2	2	None
First	A	241 to 266	134	25	2	2	Positive
	B		136	17	2	2	Positive
	C		136	9	2	2	Positive
	D		138	12	2	2	Positive
1955 Fourth	Combined		528	53	2	2	None
1956 First	Combined		544	63	2	2	Positive

L₁ = Experienced this periodL₂ = Established on basis of prior experience

Uranium by the Fluorometric Method. A trend to low values was noted during this quarter for the determination of uranium in the fluorometric range, but a bias was not observed. The precision achieved in previous quarters was maintained and is summarized in Table I-2 and shown graphically in Figure 1-3.

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Fig. 1-3. Uranium; Fluorometric Method.

Bldg. 3019

CONTROL CHARTS, 1956

CONFIDENTIALTable I-2Uranium by the Fluorometric MethodSummary of Control Data

Building 3019

<u>Quarter</u>	<u>Concentration Range μg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u> Per Cent	<u>L₂</u>	
Fourth, 1955	0.6 to 13.	231	19	10	10	None
First, 1956		251	37	10	10	None

L₁ = Experienced this periodL₂ = Established on basis of prior experience

Uranium by the Colorimetric, Thiocyanate Method. A comparison of the data for this quarter with that obtained during the fourth quarter of 1955 indicates that the precision has been maintained during this quarter. This comparison of data is given in Table I-3.

Table I-3Uranium by the Colorimetric, Thiocyanate MethodSummary of Control Data

Building 3019

<u>Quarter</u>	<u>Concentration Range μg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u> Per Cent	<u>L₂</u>	
Fourth, 1955	1.2 to 5.4	528	37	4	4	None
First, 1956		540	27	4	4	None

L₁ = Experienced this periodL₂ = Established on basis of prior experience

CONTROL CHARTS, 1956

Fig. 1-4. Uranium; Thiocyanate, Colorimetric Method.

Bldg. 3019

CONFIDENTIALBuilding 3508

Uranium by the Colorimetric, Thiocyanate Method. The precision established in the third and fourth quarters of 1955 for the determination of uranium in the colorimetric range was maintained during this quarter as shown in Figure 1-5. A trend to low values was observed during this quarter, but no bias was found to exist. This is confirmed by the summary of control results that is given in Table I-4.

Table I-4Uranium by the Colorimetric, Thiocyanate MethodSummary of Control ResultsBuilding 3508

<u>Quarter</u>	<u>Concentration Range mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u>	<u>L₂</u>	
				<u>Per Cent</u>		
Fourth, 1955	1.2 to 5.4	50	1	2	4	None
First, 1956	..	54	2	2	4	

L₁ = Experienced this period

L₂ = Established on basis of prior experience

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CONTROL CHARTS, 1956

Fig. 1-5. Uranium; Thiocyanate, Colorimetric Method.

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In Tables I-5 through I-7 the control results, for thorium at two concentration levels and for uranium in the colorimetric and potentiometric ranges, are summarized. This data is shown graphically in Figures 1-6 through 1-9.

Thorium by the Colorimetric, Thoron Method. During this quarter, the precision of the thorium results at both concentration levels was maintained, as shown in Figures 1-6 and 1-7. A trend to low values of thorium in the high concentration level, ~266 mg/ml was observed, although no bias existed.

Table I-5Thorium by the Colorimetric, Thoron MethodSummary of Control Results

Building 3550

Quarter	Concentration Range mg/ml	Number Determi- nations N	Number Determi- nations Outside L ₂	Limit of Error		Bias
				L ₁ — Per Cent	L ₂	
Fourth, 1955	0.20 to 0.32	58	3	2	2	None
First, 1956		68	0	2	2	None
Fourth, 1955	240 to 266	58	4	2	2	None
First, 1956		68	1	2	2	None

L₁ = Experience this period

L₂ = Established on basis of prior experience

Uranium by the Colorimetric, Thiocyanate Method. An improvement in precision, as shown in Figure 1-8 was noted for the determination of uranium during this period as compared to that obtained during the fourth quarter of last year. The improvement in precision is confirmed by the summary of the control results that is given in Table I-6.

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CONTROL CHARTS, 1956

Fig. 1-6. Thorium; Thoron-Colorimetric Method. Low Thorium Concentration.

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Fig. 1-7. Thorium; Thoron-Colorimetric Method. High Thorium Concentration.

Bldg. 3550

CONFIDENTIALTable I-6Uranium by the Colorimetric, Thiocyanate MethodSummary of Control Results

Building 3550

<u>Quarter</u>	<u>Concentration Range mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u>	<u>L₂</u>	
Fourth, 1955	1.2 to 5.4	30	2	4	4	None
First, 1956	.	50	1	3	4	None

L₁ = Experience this periodL₂ = Established on basis of prior experience

Uranium by the Potentiometric, Ferric Sulfate Titration Method. It is apparent from the summary of the control results for the determination of uranium by the potentiometric titration method that the precision during this period is comparable to that obtained during the previous quarter. This comparison of data is given in Table I-7. No bias was found to exist as shown in Figure 1-9.

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Fig. 1-8. Uranium; Thiocyanate, Colorimetric Method.

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CONFIDENTIALTable I-7Uranium by Potentiometric, Ferric Sulfate Titration MethodSummary of Control Results

Building 3550

<u>Quarter</u>	<u>Concentration Range mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u>	<u>L₂</u> Per Cent	
Fourth, 1955	. 30 to 33.	58	1	0.2	0.2	None
First, 1956		54	0	0.2	0.2	None

L₁ = Experience this periodL₂ = Established on basis of prior experienceCONFIDENTIAL

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CONTROL CHARTS, 1956

Fig. 1-9. Uranium; Potentiometric Titration Method.

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In Figures 1-10 through 1-13, the control results are shown for thorium, at two concentration levels and for uranium in the fluorometric and colorimetric ranges. These data are also summarized in Tables I-8 through I-10.

Thorium by the Colorimetric, Thoron Method. During the month of February, negative biases were found to exist for thorium at both concentration levels. This negative bias was eliminated early in March as shown in Figures 1-10 and 1-11. The precision attained in the previous quarter was maintained as shown in the summary given in Table I-8.

Table I-8Thorium by the Colorimetric, Thoron MethodSummary of Control ResultsBuilding 4500

<u>Quarter</u>	<u>Concentration Range mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u> Per Cent	<u>L₂</u>	
Fourth, 1955	0.29 to 0.32	106	16	2	2	None
First, 1956		122	11	2	2	None
Fourth, 1955	241 to 266	110	12	2	2	None
First, 1956		122	3	2	2	None

L₁ = Experienced this period

L₂ = Established on basis of prior experience

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Fig. 1-10. Thorium; Thoron-Colorimetric Method.
Low Thorium Concentration

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Fig. 1-11. Thorium; Thoron - Colorimetric Method. High Thorium Concentration.

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Uranium by the Fluorometric Method. The precision of the results for the determination of uranium by the fluorometric method as summarized in Table I-9 is comparable to that achieved during the fourth quarter of last year. No bias was found to exist, see Figure 1-12.

Table I-9Uranium by the Fluorometric MethodSummary of Control Results

Building 4500

<u>Quarter</u>	<u>Concentration Range mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u>	<u>L₂</u>	
Fourth, 1955	0.6 to 13.	59	3	10	10	None
First, 1956		62	0	10	10	None

L₁ = Experienced this period

L₂ = Established on basis of prior experience

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CONTROL CHARTS, 1956

Fig. 1-12. Uranium, Fluorometric Method.

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Uranium by the Colorimetric, Thiocyanate Method. A summary of the control data as given in Table I-10 shows that the precision attained during this quarter was again 2 per cent which is comparable to the precision achieved during the last year. As shown in Figure 1-13, there was a slight trend to positive values the month of January but this trend was eliminated and no bias was found to exist.

Table I-10Uranium by the Colorimetric, Thiocyanate MethodSummary of Control Results

Building 4500

<u>Quarter</u>	<u>Concentration Range mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u>	<u>L₂</u>	
Fourth, 1955	1.2 to 5.4	126	2	3	4	None
First, 1956		126	2	2	4	None

L₁ = Experience this periodL₂ = Established on basis of prior experienceCONFIDENTIAL

CONTROL CHARTS, 1956

Fig. 1-13. Uranium; Thiocyanate, Colorimetric Method.

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CONFIDENTIALGeneral Analysis Laboratory - Building 3550

Control data for the determination of chromium, iron, nickel, and uranium is summarized in Table I-11. These results are also shown graphically in Figures 1-14 through 1-17.

An improvement in precision was achieved during this quarter for the four colorimetric determinations. The number of determinations this quarter was not sufficient to warrant changing the control limits, but if this improved precision is maintained the next quarter, it is recommended that the control limits of 2 per cent be substituted for the control limits of 4 per cent that are currently in use.

Table I-11Summary of Control Results for Chromium, Iron, Nickel, and Uranium

General Analysis Laboratory - Building 3550

(First Quarter, 1956)

<u>Element and Method</u>	<u>Approximate Concentration mg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>		<u>Bias</u>
				<u>L₁</u> — Per Cent	<u>L₂</u>	
Cr - Diphenylcarbazide	0.20	12	0	3	4	None
Fe - Orthophenanthroline	0.15	26	0	2	4	None
Ni - Dimethylglyoxime	0.20	20	0	2	4	None
U - Thiocyanate	1.00	22	2	2	4	None

L₁ = Experience this periodL₂ = Established on basis of prior experienceCONFIDENTIAL

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CONTROL CHARTS, 1956

Fig. 1-14. Chromium; Diphenyl Carbazide Colorimetric Method.

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Fig. 1-15. Iron; Orthophenanthroline Colorimetric Method.

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CONTROL CHARTS, 1956

Fig. 1-16. Nickel; Dimethyl Glyoxime Colorimetric Method.

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CONTROL CHARTS, 1956

Fig. 1-17. Uranium; Thiocyanate, Colorimetric Method.

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Table II-1

Summary of Control Results

Building 9201-2

<u>Determination and Method</u>	<u>Concentration Range μg/ml</u>	<u>Number Determi- nations N</u>	<u>Number Determi- nations Outside L₂</u>	<u>Limit of Error</u>			<u>Control Chart No.</u>
				<u>L₂</u>	<u>L₁</u>	<u>Quarter</u>	
					<u>First</u> 1956	<u>Fourth</u> 1955 Per Cent	
Cr - Diphenylcarbazide	100 - 400	69	1	4	4	4	2-1
Fe - Orthophenanthroline	50 - 400	61	2	4	4	4	2-2
Ni - Dimethylglyoxime	150 - 750 <u>mg/ml</u>	59	0	4	4	4	2-3
U - Automatic Titration - K ₂ Cr ₂ O ₇	.2 - 3.	46	9	0.5	1	0.8	2-4

L₁ = Experience this periodL₂ = Established from prior experience

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Part II: Department 4270

R. L. McCutchen and C. D. Susano

Building 9201-2

As a part of the review and re-evaluation of the over-all control program for Department 4270, the control work in the laboratory of Building 9201-2 was examined. As a result of this examination, it was suggested that the number of results which are reported for control purposes be decreased by a factor of two and that a solid material be provided as a control standard; the solid standard to be tested in addition to, rather than in place of, the standard solutions which are analyzed for control purposes.

In the past, two results were reported for each constituent in the control solution. Since these results were usually obtained on the same day, and the second value was essentially the same as the first, it was recommended that only one determination per constituent be made. By eliminating the duplicate determinations the number of results will be decreased by a factor of two and yet sufficient information will be available to maintain control of the various analytical methods.

A large quantity of a solid material, which is similar in composition to samples that are being analyzed, was prepared for use as a control standard. As soon as values are established for the various components of this material, it will be used for this purpose in the laboratory of Building 9201-2.

Early in this quarter, new control solutions were substituted for those which had been tested during the past eight or nine months. The results for iron, nickel and chromium in these new solutions, were "in-control" and no biases were observed, see Figures 2-1 through 2-3. The precision in all cases, was essentially the same as that attained in previous periods, see Table II-1. Thus, it can be concluded that the program in this laboratory is being conducted independently and without bias.

The erratic results that were noted for the volumetric determination of uranium in previous quarters were also observed during this period, see Figure 2-4 and Table II-1. Although these erratic results could not, in every case, be related to the performance of the automatic titrator, the consensus of opinion seemed to be that the electrode in the instrument was the source of the trouble. Consequently, toward the end of March, the zinc-ceric sulfate method was substituted for the automatic titrator-dichromate procedure.

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Fig. 2-1. Chromium; Diphenyl Carbazide Method.

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Fig. 2-2. Iron; Orthophenanthroline Method.

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Fig. 2-3. Nickel; Dimethyl Glyoxime Method.

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CONTROL CHARTS, 1956

Fig. 2-4. Uranium, Automatic Titration - $K_2Cr_2O_7$.

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Table II-2

Summary of Control Results

Building 9204-1

Determination and Method	Concentration Range μg/ml	Number Determi- nations N	Number Determi- nations Outside L ₂	Limit of Error			Control Chart No.
				L ₂	L ₁		
					Quarter		
					First 1956 Per Cent	Fourth 1955	
Cr - Diphenylcarbazide	10 - 100	37	2	4	4	4	2-5
Cu - Diethyldithiocarbamate	150 - 250	33	2	4	4	2	2-6
Fe - α-α'-bipyridyl	4 - 17	35	0	4	4	4	2-7
Ni - Dimethylglyoxime	10 - 300	37	1	4	4	4	2-8
	<u>mg/ml</u>						
U - Zinc - K ₂ Cr ₂ O ₇	35 - 42	37	1	0.5	0.5	0.5	2-9

L₁ = Experience this periodL₂ = Established from prior experience

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Although the program for the laboratory in Building 9204-1 was reviewed during this quarter, no revisions were recommended. The number of determinations that are reported for control purposes rarely exceeds 15 per week or three results per constituent per week. In a typical week about 60 to 125 samples of uranyl sulfate and three standard solutions will be tested for uranium and such minor constituents as chromium, copper, iron and nickel. Consequently, the work in this laboratory amounts to less than three per cent of the total work for the week. This amount has been found to be sufficient to maintain the precision and accuracy of the test results.

The precision of the results for chromium, copper, iron, nickel and uranium, as shown in Figures 2-5 through 2-9 for the quarter, was comparable to that attained during the previous year. This observation is confirmed by the summary of these results which is given in Table II-2.

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Fig. 2-5. Chromium; Diphenyl Carbazide Method.

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Fig. 2-6. Copper; Diethyldithiocarbamate Method.

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Fig. 2-7. Iron; α - α' Bipyridyl Method.

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Fig. 2-8. Nickel; Dimethyl Glyoxime Method.

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CONTROL CHARTS, 1956

Fig. 2-9. Uranium; Jones Reductor - $K_2Cr_2O_7$ Titration.

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After the control work in the laboratory of Building 9733-4, Room 7, had been reviewed, it was apparent that certain revisions were necessary; particularly with regard to the fluorometric determination of uranium. Certain changes were also recommended for the determination of uranium in the colorimetric range.

Uranium by the Fluorometric Method. Toward the end of last year, a question was raised as to the reliability of the data from which the precision of the fluorometric method for the determination of uranium was derived. At that time, the data appeared to be valid. On investigation, however, a lack of independence and a definite bias was evident. This condition apparently developed through the use of control standards to obtain calibration data as well as through a misconception as to the purpose of a control program.

When it became apparent that the control data for the fluorometric determination was not reliable, the control work was suspended while a more thorough examination of the fluorometric method was made. The details of some of these studies and experiments are given in Part III of this report. As a result of these studies, a different type of program was proposed for the fluorometric determination of uranium.

In the revised program, provisions are made for a series of standard solutions of uranyl nitrate which are to be used only to obtain calibration data. Actually these standards will be required about once-a-month because the flux, which is an integral part of the fluorometric method, is prepared in small batches that are consumed in about 3 or 4 weeks. Consequently, the calibration standards will be used only when a new batch of flux is prepared.

The control standards for the fluorometric method will consist of several solutions of uranyl nitrate which will be tested along with and in the same manner as the samples. The uranium content of these standards will be adjusted so that the fluorescence of the uranium phosphor will be measured on different scales of the fluorophotometer. Each time a control solution is tested, the uranium content will be determined only once instead of twice. The results of these tests will be used for control purposes and will be shown on the chart for this analysis.

In order to establish the precision for different types of samples, the uranium content of a typical material will be determined 2 or 4 times on different days by the fluorometric method. This data will be evaluated to establish the reproducibility of test results for this particular type of material. Periodically, as materials of this type are submitted for

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analysis, duplicate or quadruplicate determinations will be made for one sample to ensure that the precision is being maintained. Although this data will be evaluated for control purposes, it will not be shown on a control chart.

The revised control program for the fluorometric determination of uranium will include, (1) a series of standards to be used for calibration, only, (2) a series of standards to be tested for control purposes and (3) the analysis of typical samples to establish the precision for different materials that are tested by this method. The first step in this revised program was initiated during the last few days of March. The second phase will be instituted during the early part of April. As soon as it can be established that the control results are "in-control" and without bias, the third phase of this program will be undertaken.

Uranium by the SnCl_2 -Thiocyanate Method. When the control program for the determination of uranium by the SnCl_2 -Thiocyanate Method was examined, it was found to be commensurate with the needs of the laboratory. A lack of independence in the control results was discovered, however, when it was found that control standards were used to calibrate the method. Consequently, it was recommended that standard solutions of uranyl nitrate be provided for calibration purposes, and that a different series of standard solutions be used for control purposes. The use of the control standards for purposes of calibration in this case was not serious. The two different series of standard solutions have been prepared and will be tested in the laboratory during this next quarter. The analysis of samples to provide information as to the reproducibility of test results for various types of samples will also be included in the control program for the determination of uranium by the colorimetric method.

An evaluation of the control data for this quarter is not presented herein because only 14 results were reported for the determination of uranium by the SnCl_2 -Thiocyanate Method. This data will be combined and evaluated with that secured during the next quarter.

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As a part of the over-all review of the control programs for Department 4270, the possible need for such a program in the laboratory of Building 9733-4, Room 8, was investigated. Subsequently, a program was proposed which will include the spectrophotometric determination of aluminum, molybdenum, thorium and vanadium, as well as the volumetric determination of aluminum, uranium and vanadium.

It is anticipated that control data will be available for all these constituents by the end of the next quarter; only molybdenum was determined for control purposes during the latter part of this quarter. Since only a small amount of data was available this quarter for the determination of molybdenum by the ethyl acetate-thiocyanate method, it will be included in the summary of the control data for the next period.

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Part III

Evaluation of Experimental Data Pertinent to Quality Control

R. L. McCutchen and C. D. Susano

INTRODUCTION

Experimental data which are pertinent to, but not a part of, the Quality Control Program have been evaluated for the determination of molybdenum, uranium and vanadium. The results of these evaluations are presented here because it is recognized that information of this sort is vital to a Quality Control Program.

An evaluation of the preliminary tests which have been made to establish the composition of a sample of NaZrF_5 so that it can be used as a control standard in the laboratory of Building 9201-2 is also included.

Molybdenum by the Thiocyanate Method^(1,2). The reproducibility of calibration factors, for the determination of molybdenum by the ethyl acetate - thiocyanate method, was established by a statistical evaluation of the calibration data which had been obtained since the first of the year. Calibration factors were calculated from the absorbancy at 470 mμ of a 3-ml portion of a standard solution, $\text{Mo} = \sim 10 \mu\text{g/ml}$, which had been processed and extracted in the same manner as the test samples. A new calibration factor was derived for each group of three samples or 25 per cent of the test results were obtained for standard samples. When the reproducibility of the calibration factors was found to be 5 per cent, the use of an average factor of 0.092 mg/A was recommended. If this average factor is used, the precision of the test results will be maintained while the number of samples that can be tested in a given period of time will be increased by 25 per cent.

The effect of iron on the spectrophotometric determination of molybdenum by the thiocyanate method was investigated by E. C. Lynn in Building 9733-4, Room 8. After the results of this experiment were analyzed, it was concluded that iron in quantities of 20 mg or less do not interfere with the spectrophotometric determination of molybdenum.

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Preparation of a Standard Sample of NaZrF₅ for Control Purposes (3,4).
A quantity of NaZrF₅, designated No. 499, was secured for use as a standard sample in the laboratory of Building 9201-2. Before it could be used for this purpose, however, it was necessary to establish the concentration of the various constituents in the material. The concentrations of fluoride, chromium, iron, nickel, sodium, uranium and zirconium were determined by appropriate spectrophotometric, volumetric and gravimetric methods after the original sample had been pulverized in a hammer-mill; the particle size, after this treatment, was equal to or smaller than that obtained when a material is ground to pass a 200-mesh sieve.

Values were established for the concentration of the different components of NaZrF₅, but the reproducibility of the test results was not commensurate with the precision usually associated with these analyses, see Table III-1. Before this sample of NaZrF₅ could be used as a standard sample, therefore, it was necessary to ascertain the reason for the erratic test results and to devise some means of eliminating such results.

Table III-1Analysis of NaZrF₅Evaluation and Summary of Test Results ForChromium, Fluoride, Iron, Nickel, Sodium, Uranium and Zirconium

(95 Per cent probability level)

<u>Determination and Method</u>	<u>Number of Determinations N</u>	<u>Average $\bar{X}$ Per Cent</u>	<u>Limit of Error of One Determination L.E. Per Cent</u>
Fluoride- Pyrohydrolysis			
NaOH Titration	50	42.1	3
Zirconium- Gravimetric,			
Mandelic Acid	56	38.1	2
Sodium- Gravimetric			
Zinc Uranyl Acetate	55	10.6	10
Uranium- Volumetric			
Automatic Titrator-K ₂ Cr ₂ O ₇	57	8.9	30
Iron- Spectrophotometric			
Orthophenanthroline	55	230 ppm	30
Chromium- Spectrophotometric,			
Diphenyl Carbazide	55	70 ppm	35
Nickel- Spectrophotometric,			
Dimethylglyoxime	56	75 ppm	80

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Soon after the program was initiated to establish the concentration of various constituents in the sample of NaZrF_5 , it was suggested that the pulverized sample was not homogeneous. Since the solid had been reduced to a very fine powder in a hammer-mill and appeared, visually, to be uniform, the possible heterogeneity of the sample was not given serious consideration. At this time, however, it became necessary to determine whether the poor reproducibility of the test results could be attributed to the heterogeneity of the sample.

In a second series of tests, a portion of the finely ground NaZrF_5 was thoroughly mixed before samples were withdrawn for analysis. The precision of this data was then compared with that obtained in the earlier experiments. In all but one case, the precision for the mixed material was better by a factor of 2 or more, see Table III-2. Consequently, the theory that the original material was heterogeneous must be accepted. Before this sample of NaZrF_5 can be used for control purposes, it must be thoroughly mixed and re-analyzed.

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Analysis of NaZrF₅: Precision of Test Results For
Mixed and Unmixed Material
 (95 Per cent probability level)

<u>Determination and Method</u>	<u>Material Treatment</u>	<u>Number of Determinations N</u>	<u>Average $\bar{X}$ Per Cent</u>	<u>Limit of Error of One Determination L.E. Per Cent</u>
Fluoride: Pyrohydrolysis NaOH Titration	Mixed	4	42.75	1
	Unmixed	8	42.39	2
Zirconium: Gravimetric Mandelic Acid	Mixed	4	38.00	0.2
	Unmixed	6	38.30	2
Sodium: Gravimetric Zinc Uranyl Acetate	Mixed	4	10.00	2
	Unmixed	7	10.27	5
Uranium: Volumetric Automatic Titrator-K ₂ Cr ₂ O ₇	Mixed	4	9.02	4
	Unmixed	7	9.27	10
Iron: Spectrophotometric Orthophenanthroline	Mixed	4	199 ppm	4
	Unmixed	8	240 ppm	10
Chromium: Spectrophotometric Diphenylcarbazide	Mixed	3	60 ppm	4
	Unmixed	6	62 ppm	10
Nickel: Spectrophotometric Dimethylglyoxime	Mixed	4	60 ppm	35
	Unmixed	8	110 ppm	45

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Uranium by the Fluorometric Method(5,6). In order to determine the reproducibility of fluorescent measurements on the 1-, 10- and 100-scale of the Photovolt fluorophotometer, a series of experiments were performed with solutions of uranyl nitrate.

The reproducibility of the test results for each of the three scales of the fluorophotometer was determined from the fluorescent measurements. The precision which was established for the three scales of the fluorophotometer is presented in Table III-3.

Table III-3

Fluorometric Determination of Uranium: Reproducibility of
Test Results as a Function of the Scale of
the Fluorophotometer

(95 Per cent probability level)

<u>Scale</u>	<u>Number of Tests N</u>	<u>Average</u>		<u>Limit of Error of One Determination L.E. Per Cent</u>
		<u>D</u>	<u>U μg/ml</u>	
1	4	51	215	10
10	4	5.6	235	5
100	4	0.64	275	15

The reproducibility of fluorescent measurements for the 1-, 10- and 100-scale of the fluorophotometer was found to be 10, 5, and 15 per cent, respectively.

The calibration data for two batches of flux, designated "A" and "B", were evaluated to ascertain the reproducibility of the fluorescent measurements from which the calibration factors are derived and to determine whether the precision of the two sets of data was significantly different.

Since each batch of flux is calibrated with standard solutions of uranium, the reproducibility of the fluorescent measurements for different batches of flux can be ascertained. To determine if the reproducibility of the fluorescent measurements for flux "A" is better than that for flux "B", the variance of the calibration data for each batch of flux was calculated. The variances were then compared by means of the variance ratio or "F" test. The results of this test are given in Table III-4.

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Fluorometric Determination of Uranium
Evaluation of the Calibration Data for Two Batches of Flux
Variance Ratio or "F" Test

Instrument Number	Flux	Number of Determi- nations N	Variance		"F" Values	
			S ²	Ratio	Experimental	Theoretical ⁽¹⁾
I	A	34	15.7			
	B	38	30.4			
				$\frac{30.4}{15.7}$	1.94	1.75 (DF ₁ = 37; DF ₂ = 33)
II	A	34	17.8			
	B	39	53.5			
				$\frac{53.5}{17.8}$	3.00	1.75 (DF ₁ = 38; DF ₂ = 33)

(1) 95 Per cent probability level

In both cases, the experimental "F" value was larger than the theoretical value, "F_t"; therefore, the difference in the reproducibility of the fluorescent measurements for the two batches of flux is considered to be significant. As a result of this evaluation it is apparent that the reproducibility of the fluorescent measurements for Flux "A" is significantly better than that for Flux "B".

Since two fluorophotometers, designated I and II are available for use in the laboratory the reproducibility of the calibration factors for the two batches of flux as well as the established factors are given in Table III-5 for both instruments. The calibration factors which are given in the table were used to determine the uranium content of samples.

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Table III-5
Fluorometric Determination of Uranium
Precision of the Calibration Factors for Two Batches of Flux
 (95 Per cent probability level)

<u>Instrument Number</u>	<u>Flux</u>	<u>Number of Determi- nations N</u>	<u>Calibration Factor "F"</u>	<u>Precision of the Factor</u>	
				<u>Standard Deviation</u>	<u>Limit of Error of One Determination Per Cent</u>
I	A	34	8.0	0.32	10
	B	38	7.3	0.44	15
II	A	34	5.4	0.21	10
	B	39	5.0	0.35	15

Although it is obvious that the two batches of flux are different, the reason for this difference has not been established. The constituents of the flux* and the manner in which they were combined and mixed were apparently the same for both batches of flux; nevertheless, the two batches of flux are different.

* Composition of the flux -

K₂CO₃ - 1040 grams

Na₂CO₃ - 800 grams

KF - 190 grams

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Vanadium by the Volumetric Method⁽¹⁾. The vanadium content of samples, in which the quantity of vanadium is 75 to 100 mg, is usually determined by titration with 0.1 N FeSO_4 . For smaller quantities of vanadium, or when the amount of sample that is available for the analysis is limited, the concentration of the titrant is decreased to 0.02 N. The test portion, in either case, is adjusted so that 15 to 20 ml of the titrant is required.

Inasmuch as the concentration of vanadium in the samples, which are being tested in the laboratory, Building 9733-4, Room 8, is low and the quantity of sample available for testing is small, the less concentrated titrant is used. The precision of the volumetric method at this lower concentration of vanadium was estimated to be five per cent. An evaluation of the data, which was derived from the analysis of a standard solution of vanadium, indicates that the precision for vanadium in quantities of 15 to 20 mg is about 3 per cent. The concentration of vanadium in the standard solution is 5.17 ± 0.02 mg/ml.

The data, which were evaluated to determine the precision of this method and to establish the concentration of vanadium in the standard sample, were obtained by testing a portion of the standard solution each time that samples were analyzed. If, in the opinion of the supervisor, the value obtained for the standard sample was not satisfactory, the samples which had been tested at the same time as the standard, were all re-analyzed. In this way, both the precision and accuracy of the test results were maintained.

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2. Ibid, Preliminary Report ECL-5 and RLM-10.
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