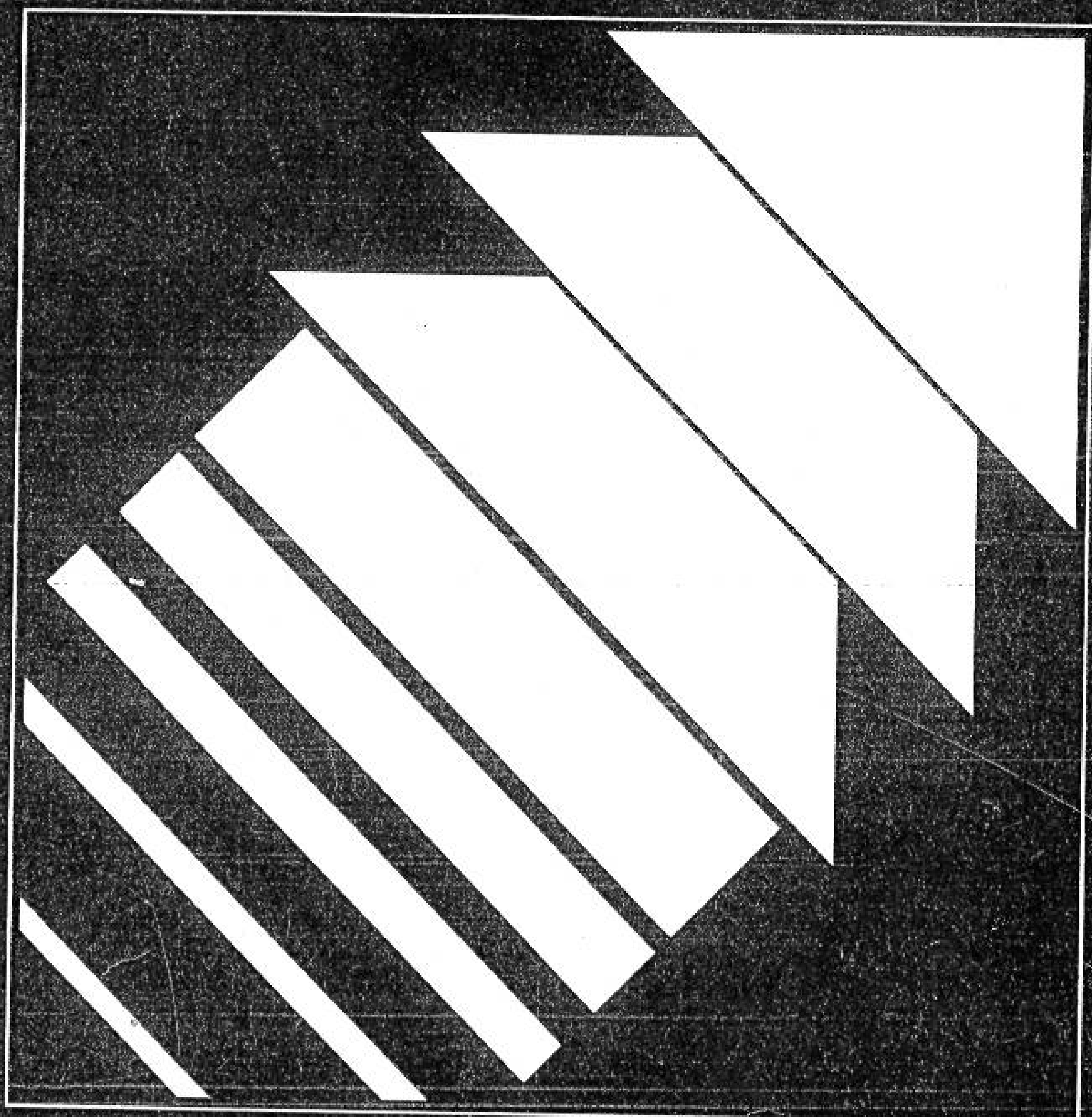
NATIONAL
URANIUM
TAILINGS
PROGRAM

URANIUM TAILINGS REFERENCE MATERIALS

NUTP-2E



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C.W. Smith H.F. Steger W.S. Bowman

National Uranium Tailings Program

NUTP-2E

CANMET

Canada Centre for Mineral and Energy Technology

Centre canadien de la technologie des minéraux et de l'énergie

Available in Canada through

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or by mail from

Canadian Government Publishing Centre
Supply and Services Canada
Ottawa, Canada K1A 0S9

Catalogue No. M39-15/1984E — Canada: $4.75
ISBN 0-660-11757-6 — Other countries: $5.70

Price subject to change without notice

Disponible en français

FOREWORD

There are currently about 130 million tonnes of mine wastes (tailings) produced from the mining and milling of uranium ore that are on, or near, the surface in Canada. At public hearings and other forums held in connection with proposals for new uranium production facilities, concern has been expressed about the impact that these wastes may have on the environment in the future. Government regulatory agencies believe that there may be long-term environmental and health consequences associated with uranium tailings that are not yet fully understood.

As a result of a year-long study, undertaken by a group of experts from government, industry, and the universities, into the possible long-term problems of uranium tailings management, the decision was taken by the Federal Government to launch an R & D program to assist in clarifying the nature and extent of problems that may arise in the future. It is anticipated that the results of such a program will be of use to the Atomic Energy Control Board and to provincial regulatory agencies in the establishment of criteria for the decommissioning and abandonment of uranium tailings. To this end, the National Uranium Tailings Program of CANMET has been funded to the extent of $9.5 million for a period of five years.

The main objective of this Program is to provide a scientifically credible information base that will assist government regulatory agencies in making confident, knowledgeable decisions, and in establishing criteria for the long-term protection of the environment and human health. The scientific information base will, among other things, describe the chemical and physical processes that involve tailings constituents and their movement along pathways from the tailings into the biosphere.

The National Uranium Tailings Program's activities, which have been entirely contracted out, have included research into the processes involved in the behaviour and movement of contaminants from tailings, field sampling and measurements, chemical analyses, and predictive modelling. As an important part of the Program output, manuals have been prepared that incorporate the results of this work.

This manual has been prepared for the purpose of making available up-to-date information and data that will be of interest and practical use to those directly concerned with operational aspects of uranium tailings management and regulation.

W.G. Jeffery
Director General
Canada Centre for Mineral and Energy Technology

PREFACE

Quality assurance in the production of data is indispensable to the success of the National Uranium Tailings Program. In order to provide as much assistance as possible in ensuring high quality chemical analyses for radionuclides and other constituents in a uranium tailings matrix, standard reference materials were prepared to cover the range of tailings composition encountered in Canada. These reference materials will be used to maintain a high level of quality control for all work performed by contract on behalf of the Program, and will also be available to all others that may find them useful in verifying the performance of their own equipment and procedures.

As additional reference materials, relevant to the Program are certified in the future, supplements to this manual will be prepared and published.

ACKNOWLEDGEMENTS

At the beginning of the Program, Lloyd Dalton and Clint Smith of CANMET Chemical Laboratories served as Scientific Authorities on behalf of NUTP in the exchange of analyses with participating laboratories. This responsibility has subsequently been assumed by Mr. Roy John, Project Manager, Measurements, of the National Uranium Tailings Program Office. The laboratories that participated in the exchange program are listed in the body of the manual. The efforts of the following CANMET staff in organizing and operating the project in developing the reference standards are gratefully acknowledged: C.W. Smith, H.F. Steger and W.S. Bowman.

V.A. Haw
Director
National Uranium Tailings Program

URANIUM TAILINGS REFERENCE MATERIALS

by

C.W. Smith, H.F. Steger* and W.S. Bowman**

SYNOPSIS

Samples of uranium tailings from Bancroft and Elliot Lake, Ontario, and from Beaverlodge and Rabbit Lake, Saskatchewan, have been prepared as compositional reference materials at the request of the National Uranium Tailings Research Program. The four samples, UTS-1 to UTS-4, were ground to minus 104 μm, each mixed in one lot and bottled in 200-g units for UTS-1 to UTS-3 and in 100-g units for UTS-4. The materials were tested for homogeneity with respect to uranium by neutron activation analysis and to iron by an acid-decomposition atomic absorption procedure.

In a *free choice* analytical program, 18 laboratories contributed results for one or more of total iron, titanium, aluminum, calcium, barium, uranium, thorium, total sulphur, and sulphate for all four samples, and for nickel and arsenic in UTS-4 only. Based on a statistical analysis of the data, recommended values were assigned to all elements/constituents, except for sulphate in UTS-3 and nickel in UTS-4.

The radioactivity of thorium-230, radium-226, lead-210, and polonium-210 in UTS-1 to UTS-4 and of thorium-232, radium-228, and thorium-228 in UTS-1 and UTS-2 was determined in a radioanalytical program composed of eight laboratories. Recommended values for the radioactivities and associated parameters were calculated by a statistical treatment of the results.

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RÉSIDUS D'URANIUM, MATÉRIAUX DE RÉFÉRENCE

par

C.W. Smith, H.F. Steger* et W.S. Bowman**

SYNOPSIS

Des échantillons de résidus d'uranium provenant de Bancroft et d'Elliot Lake en Ontario et de Beaverlodge et de Rabbit Lake en Saskatchewan ont été préparés comme matériaux de référence de composition connue à la demande du Programme national de recherche sur les résidus d'uranium. Chacun des quatre échantillons, UTS-1 à UTS-4, a été broyé à une granulométrie inférieure à 104 µm et mélangé pour former un lot. Les résidus UTS-1 à UTS-3 ont été mis dans des bouteilles de 200 g et l'UTS-4 a été mis dans des bouteilles de 100 g. On a vérifié l'homogénité des matériaux quant à l'uranium par la méthode d'activation neutronique et quant au fer total par une méthode de décomposition acide et de spectrométrie d'absorption atomique.

En vertu d'un programme analytique de *libre choix*, 18 laboratoires ont soumis des résultats pour un, ou plusieurs des éléments/constituants suivants: fer total, titane, aluminium, calcium, baryum, uranium, thorium, soufre total et sulfate pour les quatre matériaux, et nickel et arsenic pour l'UTS-4 seulement. Après une analyse statistique des données, on a assigné des valeurs recommandées à tous les éléments/constituants, sauf au sulfate pour l'UTS-3 et au nickel pour l'UTS-4.

On a déterminé la radioactivité des isotopes, thorium-230, radium-226, plomb-210, et polonium-210 pour les quatre matériaux, et thorium-232, radium-228, et thorium-228 pour l'UTS-1 et l'UTS-2, dans le cadre d'un programme radioanalytique auquel huit laboratoires ont participé. Les valeurs recommandées pour les activités des isotopes et des paramètres associés ont été calculées à l'aide d'une analyse statistique des données.

**Chercheurs scientifiques, Laboratoires des sciences minérales, CANMET, Énergie, Mines et Ressources Canada, Ottawa, K1A 0G1.*

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No.

INTRODUCTION

The preparation of compositional reference materials from several uranium tailings samples, typical of existing Canadian tailings sites, was initiated under the auspices of the National Uranium Tailings Research Program (NUTRP) in late 1982 (1). These were intended for use in quality-control functions for both chemical and radiochemical analytical measurements with the National Measurement Program of NUTRP. The reference materials were also to be made available through the Canadian Certified Reference Materials Project (CCRMP) for more general use by the analytical community.

CCRMP currently offers many compositional reference ores, concentrates, and related products, typical of Canadian deposits and not, in general, available from other sources for use in analytical laboratories associated with mining, metallurgy, and the earth sciences (2). Among these are uranium ores from Elliot Lake, Ontario, and Beaverlodge, Saskatchewan, of which four, DL-1a, DH-1a, BL-4a and BL-5, have been certified for radium-226 activity (3).

This report describes the preparation and characterization of the four uranium tailings samples: UTS-1, UTS-2, UTS-3 and UTS-4. The description of their nature, physical preparation, and estimation of the consensus values and related statistical parameters for use as reference materials follows.

The results of the analytical program to establish the concentration of total iron, titanium, aluminum, calcium, barium, uranium, thorium, total sulphur, sulphate, nickel and arsenic in the tailings samples are presented in "Part A" of this report.

The results of the radioanalytical program to determine the activity of thorium-230, radium-226, lead-210, and polonium for all four samples, and also for UTS-1 and UTS-2, thorium- 232, radium-228 and thorium-228, are presented in "Part B".

NATURE AND PREPARATION

UTS-1

The uranium tailings sample for UTS-1 was donated to NUTRP and CCRMP in October 1982 by Madawaska Mines Limited located near Bancroft, Ontario. The mine is in a belt of meta-gabbro and amphibolite. The ore is part of a zone of irregular pegmatitic granite dykes, with metasomatic phases. Uranium is present as uraninite and uranothorite with minor uranophane (4). The ore is comminuted to 50% minus 74 μm and is leached at pH = 1.6 to 1.8 with sulphuric acid and sodium chlorate under agitation with compressed air with a 24 h retention period. The mill tailings slurry is treated with lime to pH = 9 before disposal (5).

UTS-2

The tailings sample for UTS-2 was donated by Rio Algom of Elliot Lake, Ontario. The ore is a pebble conglomerate. The pebbles are mainly quartz with some chert. The matrix is a sericitic, feldspathic quartzite containing about 10% pyrite on a whole ore basis (6). The comminuted ore is leached at 60-70°C at a concentration of 40 g/L sulphuric acid under agitation with compressed air for approximately 40 h. The mill tailings slurry is treated with limestone and lime to raise the pH to 10 before disposal (7).

UTS-3

The tailings sample for UTS-3 was donated by Eldorado Nuclear Limited at Beaverlodge, Saskatchewan. The orebody consists of complexes of disseminations and stringers, lenses and veins of pitchblende in reddish-brown mylonitized oligoclase saturated with dusty hematite. Pitchblende is the sole radioactive mineral present. Minor amounts of carbonates and pyrites occur along with the pitchblende (8). The pyrites are separated as a concentrate by flotation at pH 10 (sodium carbonate) using isopropyl xanthate. This concentrate is leached with 100 lb/ton (44 kg/1000 kg) of sulphuric acid and 2.5 lb/ton (1.1 kg/1000 kg) of sodium chlorate. The uranium is precipitated with magnesium hydroxide and sent to the carbonate leaching circuit.

The slurry of flotation tailings and the above precipitated uranium in 100 g/L dissolved carbonate and sulphate salts is leached for four days at 90°C. Oxidation of the uranium and residual sulphide minerals is accomplished by sparging with 99.5% oxygen. The mill tailings are disposed of directly (9).

UTS-4

The tailings sample for UTS-4 was provided by the Extractive Metallurgy Laboratory of CANMET which had previously prepared it from ore donated by, and using the recovery process of, Eldor Mines (formerly Gulf Minerals Canada Limited) at Rabbit Lake, Saskatchewan. The host rock for the uranium mineralization is a siliceous dolomite that has been highly altered and fractured. The ore consists of a high-grade zone of such mineralization in the centre of a brecciated zone, grading to low grade in the lesser brecciated perimeter (10). The comminuted ore is leached with sulphuric acid and sodium chlorate in seven stages whereby pH and temperature are changed from 2.5-2.8 and 30-34°C to 1.6-1.7 and 48-52°C, respectively. The leach retention time is approximately eight hours. The mill tailings are neutralized with lime before disposal (10).

The four uranium tailings samples were dried at 80°C and dry-ground in November-December 1982 to pass a 104-μm sieve. The powdered tailings samples were each tumbled in one lot in a 570-L conical blender for 15 h and bottled. Approximately 340 kg of each of UTS-1, UTS-2 and UTS-3 were bottled in 200-g units. For UTS-4, approximately 134 kg were bottled in 100-g units. The four tailings samples were found to be sufficiently homogeneous for uranium by a neutron activation analytical technique and for total iron by an acid dissolution-atomic absorption spectrometric procedure to qualify as reference materials. Results of the confirmation of the homogeneity of UTS-1, UTS-2, UTS-3 and UTS-4 are reported in Appendix A.

The particle size analysis of the tailings samples is given in Table 1.

Table 1 – Particle size analysis of uranium tailings samples (wet screen)

Size of fraction (μm)	UTS-1	UTS-2	UTS-3	UTS-4
	mass %			
− +104	0.1	0.03	<0.01	<0.01
−104 + 74	47.8	40.9	54.8	19.7
− 74 + 55	17.6	15.6	16.5	13.1
− 55 + 37	7.0	6.9	8.3	5.7
− 37	27.5	36.6	20.4	61.5

The concentration of the elements/constituents and the activity of the selected radioisotopes in the uranium tailings samples were determined by interlaboratory programs involving eighteen and eight laboratories, respectively. All participants except one for each program performed the requested analytical work under contract to CANMET with arrangements made by Supply and Services Canada (Tables A-1 and B-1).

ESTIMATION OF CONSENSUS VALUES AND 95% CONFIDENCE LIMITS

The estimation of the consensus value and related statistical parameters by the following procedure is performed only after outlying values are removed. The criteria for removal is peculiar to each interlaboratory program and as such is discussed in Parts A and B.

A one-way analysis of variance technique was used to estimate the consensus value and its variance. This approach considers the results of the described certification program to be only one sampling out of a universal set of results. The analytical data were assumed to fit the model (11)

$$x_{ij} = \mu + y_i + e_{ij}$$

where x_{ij} = the j^{th} result in set i,

μ = the true consensus value,

y_i = the discrepancy between the mean of the results in set i ($\overline{x}_{i.}$) and μ, and

e_{ij} = the discrepancy between x_{ij} and $\overline{x}_{i.}$

It is assumed that both y_i and e_{ij} are normally distributed with means of zero and variances of ω and σ^2, respectively. The significance of ω^2 is detected by comparing the ratio of between-set mean squares and within-set mean squares with the F statistic at the 95% confidence level and with the appropriate degrees of freedom.

The consensus value of the assumed model is estimated by the overall mean $\overline{x}_{..}$:

$$\overline{x}_{..} = \sum_i^k \sum_j^{n_i} x_{ij} \Big/ \sum_i^k n_i$$

where n_i = the number of results in set i and

k = the number of sets.

The value of σ^2 is estimated by s_1^2 which is given by

$$s_1^2 = \sum_i^k \sum_j^{n_i} (x_{ij} - \overline{x}_{i.})^2 \Big/ \sum_i^k n_i - k$$

The value of ω^2 is estimated by

$$\omega^2 = (s_2^2 - s_1^2) \Big/ \frac{1}{k-1} \left(\sum_i^k n_i - \sum_i^k n_i^2 / \sum_i^k n_i \right)$$

where

$$s_2^2 = \sum_i^k n_i (\overline{x}_{i.} - \overline{x}_{..})^2 \Big/ k - 1$$

The variance of the overall mean is given by

$$v[\overline{x}_{..}] = \left(\sum_i^k n_i^2 / (\sum_i^k n_i)^2 \right) \omega^2 + \left(1/\sum_i^k n_i \right) \sigma^2$$

and the 95% confidence limits for $\overline{x}_{..}$ are

$$\overline{x}_{..} \pm t_{0.975,\,(k-1)} \sqrt{V[\overline{x}_{..}]}$$

It should be noted that 95% confidence limits denote that if the interlaboratory program were performed 100 times, it would be expected that the overall mean in 95 would fall within the prescribed limits.

The average within-set standard deviation, σ_A, is a measure of the average within-bottle precision as determined by the analytical methods used. The implication exists, therefore, that a laboratory using a method of average or better reproducibility should obtain individual results for a given certified element with a repeatability that is at least comparable to the reported value of σ_A.

The consensus values for the elements/constituents and the radioactive isotopes are reported in Tables 2 and 3 respectively and are given recommended value status.

Table 2 – Recommended values for elements/constituents

Element/ constituent	UTS-1	UTS-2	UTS-3	UTS-4
	mass %			
Fe (total)	4.87	3.20	3.25	2.62
Ti	0.54	0.16	0.23	0.24
Al	6.24	2.71	5.80	6.29
Ca	5.24	0.42	4.03	1.75
S (total)	1.00	3.23	0.23	1.80
sulphate	2.64	0.84		5.21
	µg/g			
barium	324	464	212	65
uranium	49	56	513	1010
thorium	138	174	10.0	15.4
arsenic				38

Table 3 – Recommended activity values for radioactive isotopes

Isotope	UTS-1	UTS-2	UTS-3	UTS-4
	Bq/g			
Th-230	3.6	4.4	11.3	22.9
Ra-226	3.67	5.6	13.3	36.6
Pb-210	3.25	4.55	12.6	32.4
Po-210	3.1	4.4	11.8	30.8
Th-232	0.68	0.85		
Ra-228	0.68	1.0		
Th-228	0.71	0.92		

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CONFIRMATION OF HOMOGENEITY

CONFIRMATION OF HOMOGENEITY

The homogeneity of the uranium tailings samples was confirmed by Chemex Laboratories Ltd., North Vancouver, British Columbia, (Contract 15SQ.23440-2-9146) by analyzing for uranium and total iron in three separate subsamples for each of 19 bottles selected from a stock of 1655 bottles for UTS-1, UTS-2 and UTS-3 and of 1280 bottles for UTS-4. The stock of UTS-1 to UTS-3 was divided into 18 lots of 87 bottles and a 19th lot of 89 bottles; the stock of UTS-4 was divided into 18 lots of 67 bottles and a 19th lot of 74 bottles. In each case, the code number of the first bottle was selected at random out of the first lot. The code numbers of the remaining 18 bottles were given by the code number of the preceding bottle plus the number of bottles in each of the first 18 lots.

Uranium was determined by an instrumental neutron activation analytical technique. Total iron was determined by atomic absorption spectrometry after multi-acid sample decomposition. The results are shown in Tables 4 to 7.

A one-way analysis of variance techniques was used to assess the homogeneity (11). Herein, the ratio of the between-bottle to within-bottle mean square is compared with the F statistic at the 95% level of probability. There was no statistical evidence for 'bottle-to-bottle inhomogeneity' with respect to uranium in all four tailings samples or with respect to iron for UTS-1 and UTS-2.

The iron results for UTS-3 and UTS-4 did indicate some bottle-to-bottle inhomogeneity. This however does not imply that the inhomogeneity is physically significant particularly in view of the contradictory results observed for uranium. Moreover, a detected inhomogeneity, statistical, physical or both, does not necessarily disqualify a candidate reference material from its intended use provided that its magnitude is acceptable in comparison with the overall uncertainty in the recommended value for the element of interest. The between-bottle standard deviation for iron in UTS-3 and UTS-4 was calculated to be 0.026 and 0.015% for the overall means of 3.12 and 2.60% Fe, respectively. The detected inhomogeneity therefore gives rise to relative uncertainties of 0.83 and 0.58% for UTS-3 and UTS-4, values which CCRMP concluded would be acceptable in comparison with the between-laboratory relative uncertainty expected from the results of the interlaboratory program. The latter can be calculated to be 0.089 and 0.073% Fe for UTS-3 and UTS-4, i.e., the relative certainty is 2.74 and 2.79%, respectively, thereby demonstrating UTS-3 and UTS-4 to be sufficiently homogeneous for use as reference materials.

Table 4a – Confirmation of homogeneity of UTS-1 for uranium

Bottle No.	Uranium (µg/g) Individual			Mean
40	54	53	48	51.7
127	47	49	51	49.0
214	51	49	50	50.0
301	53	52	50	51.7
388	49	49	48	48.7
475	52	49	51	50.7
565	50	50	54	51.3
649	51	48	48	49.0
736	50	49	51	50.0
823	49	49	47	48.3
910	52	49	49	50.0
997	52	48	48	49.3
1084	52	49	45	48.7
1171	48	48	49	48.3
1258	48	49	52	49.7
1345	51	51	49	50.3
1432	50	50	53	51.0
1519	49	47	51	49.0
1606	51	47	49	49.0

Overall mean = 49.8

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	3.595
Within bottles	38	3.614
Total	56	
Calculated F statistic =	0.995	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for uranium

Table 4b – Confirmation of homogeneity of UTS-1 for total iron

Bottle No.	Total iron (%) Individual			Mean
40	4.75	4.74	4.83	4.77
127	4.93	4.75	4.86	4.85
214	4.80	4.80	4.76	4.79
301	4.83	4.82	4.82	4.82
388	4.81	4.75	4.73	4.76
475	4.77	4.76	4.76	4.76
565	4.83	4.83	4.77	4.81
649	4.78	4.85	4.82	4.82
736	4.86	4.81	4.76	4.81
823	4.77	4.83	4.81	4.80
910	4.84	4.86	4.88	4.86
997	4.83	4.82	4.82	4.82
1084	4.79	4.90	4.90	4.86
1171	4.75	4.77	4.89	4.80
1258	4.86	4.81	4.77	4.81
1345	4.82	4.80	4.81	4.81
1432	4.78	4.77	4.78	4.78
1519	4.80	4.78	4.88	4.82
1606	4.83	4.81	4.87	4.84

Overall mean = 4.81

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	2.645×10^{-3}
Within bottles	38	1.861×10^{-3}
Total	56	
Calculated F statistic =	1.421	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for iron

Table 5a – Confirmation of homogeneity of UTS-2 for uranium

	Uranium (µg/g)			
Bottle No.	Individual			Mean
72	53	54	55	54.0
159	52	53	56	53.7
246	50	57	58	55.0
333	55	56	54	55.0
420	55	55	55	55.0
507	57	57	54	56.0
594	56	57	55	56.0
681	55	56	55	55.3
768	54	55	57	55.3
855	55	55	57	55.7
942	57	57	55	56.3
1029	55	58	58	55.7
1116	55	54	55	54.7
1203	54	56	54	54.7
1290	54	55	57	55.3
1377	56	56	55	55.7
1464	54	57	56	55.7
1551	56	55	57	56.0
1638	56	53	54	54.3

Overall mean = 55.2

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.558
Within bottles	38	2.368
Total	56	
Calculated F statistic =	0.658	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for uranium

Table 5b – Confirmation of homogeneity of UTS-2 for total iron

	Total iron (%)			
Bottle No.	Individual			Mean
72	3.05	3.05	3.14	3.08
159	3.08	3.07	3.15	3.10
246	3.09	3.12	3.05	3.09
333	3.07	3.09	3.14	3.10
420	3.11	3.08	3.01	3.07
507	3.04	3.05	3.08	3.06
594	3.09	3.09	3.05	3.08
681	3.03	3.13	3.05	3.07
768	3.04	3.08	3.09	3.07
855	3.09	3.03	3.04	3.05
942	3.10	3.13	3.19	3.14
1029	3.15	3.15	3.12	3.14
1116	3.11	3.10	3.04	3.08
1203	3.10	3.12	3.11	3.11
1290	3.05	3.16	3.07	3.09
1377	3.12	3.07	3.14	3.11
1464	3.06	3.07	3.10	3.08
1551	3.08	3.13	3.07	3.09
1638	3.07	3.04	3.06	3.06

Overall mean = 3.09

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.888×10^{-3}
Within bottles	38	1.349×10^{-3}
Total	56	
Calculated F statistic =	1.400	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for iron

Table 6a – Confirmation of homogeneity of UTS-3 for uranium

Bottle No.	Uranium (%) Individual			Mean
43	.0513	.0515	.0519	.0516
130	.0517	.0522	.0527	.0522
217	.0513	.0500	.0501	.0505
304	.0521	.0485	.0494	.0500
391	.0507	.0521	.0520	.0516
478	.0506	.0513	.0518	.0512
565	.0515	.0521	.0497	.0511
652	.0516	.0521	.0513	.0517
739	.0517	.0510	.0524	.0517
826	.0517	.0502	.0497	.0505
913	.0511	.0516	.0515	.0515
1000	.0505	.0516	.0507	.0510
1087	.0508	.0504	.0518	.0510
1174	.0517	.0510	.0511	.0513
1261	.0518	.0514	.0512	.0515
1348	.0531	.0513	.0510	.0518
1435	.0513	.0522	.0521	.0519
1522	.0513	.0497	.0497	.0502
1609	.0517	.0525	.0518	.0520

Overall mean = .0513

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.117×10^{-6}
Within bottles	38	6.628×10^{-7}
Total	56	

Calculated F statistic = 1.685

F.95 (18,38) = 1.883

Null hypothesis of no difference between bottles is accepted for uranium

Table 6b – Confirmation of homogeneity of UTS-3 for total iron

Bottle No.	Total iron (%) Individual			Mean
43	3.15	3.17	3.18	3.17
130	3.20	3.15	3.11	3.15
217	3.16	3.10	3.13	3.13
304	3.07	3.10	3.11	3.09
391	3.13	3.07	3.14	3.11
478	3.10	3.05	3.13	3.09
565	3.06	3.05	3.08	3.06
652	3.09	3.08	3.06	3.08
739	3.20	3.20	3.13	3.18
826	3.15	3.11	3.15	3.14
913	3.13	3.15	3.15	3.14
1000	3.19	3.15	3.10	3.15
1087	3.06	3.16	3.14	3.12
1174	3.09	3.11	3.14	3.11
1261	3.10	3.06	3.10	3.09
1348	3.12	3.19	3.16	3.16
1435	3.14	3.07	3.10	3.10
1522	3.13	3.15	3.13	3.14
1609	3.13	3.10	3.10	3.11

Overall mean = 3.12

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	2.960×10^{-3}
Within bottles	38	9.649×10^{-4}
Total	56	

Calculated F statistic = 3.068

F.95 (18,38) = 1.883

Null hypothesis of no difference between bottles is rejected for iron

Table 7a – Confirmation of homogeneity of UTS-4 for uranium

Bottle No.	Uranium (%) Individual			Mean
51	.1079	.1076	.1057	.1071
119	.1057	.1069	.1056	.1061
187	.1066	.1069	.1076	.1071
255	.1063	.1055	.1054	.1057
323	.1019	.1051	.1063	.1044
391	.1064	.1065	.1048	.1059
459	.1060	.1060	.1077	.1066
527	.1092	.1065	.1076	.1078
595	.1060	.1082	.1065	.1069
663	.1060	.1075	.1036	.1057
731	.1076	.1064	.1066	.1069
799	.1067	.1045	.1066	.1059
867	.1045	.1060	.1063	.1056
935	.1064	.1066	.1068	.1066
1003	.1063	.1072	.1058	.1064
1071	.1075	.1051	.1078	.1068
1139	.1070	.1069	.1065	.1068
1207	.1071	.1056	.1065	.1064
1275	.1062	.1059	.1043	.1055

Overall mean = .1063

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.772×10^{-6}
Within bottles	38	1.250×10^{-6}
Total	56	

Calculated F statistic = 1.418

F.95 (18,38) = 1.883

Null hypothesis of no difference between bottles is accepted for uranium

Table 7b – Confirmation of homogeneity of UTS-4 for total iron

Bottle No.	Total Iron (%) Individual			Mean
51	2.64	2.61	2.63	2.63
119	2.63	2.61	2.58	2.61
187	2.62	2.62	2.60	2.61
255	2.57	2.59	2.60	2.59
323	2.58	2.59	2.55	2.57
391	2.58	2.60	2.58	2.59
459	2.56	2.55	2.59	2.57
527	2.58	2.57	2.59	2.58
595	2.55	2.58	2.60	2.58
663	2.59	2.61	2.62	2.61
731	2.59	2.61	2.63	2.61
799	2.63	2.61	2.61	2.62
867	2.64	2.62	2.60	2.62
935	2.65	2.60	2.58	2.61
1003	2.59	2.60	2.57	2.59
1071	2.59	2.61	2.62	2.61
1139	2.61	2.63	2.63	2.62
1207	2.61	2.58	2.58	2.59
1275	2.61	2.61	2.64	2.62

Overall mean = 2.60

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.040×10^{-3}
Within bottles	38	3.491×10^{-4}
Total	56	

Calculated F statistic = 2.980

F.95 (18,38) = 1.883

Null hypothesis of no difference between bottles is rejected for iron