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EVALUATION OF THE BORON CONTENT IN FUEL PLATES FABRICATED FOR THE FIRST CORE LOADING OF THE ARMY SM-1 REACTOR (FORMERLY APPR-1)

J. H. Cherubini
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Printed in USA. Price $0.50. Available from the
Office of Technical Services
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Washington 25, D.C.

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ORNL-3249
UC-25 – Metals, Ceramics, and Materials
TID-4500 (16th ed.)

Contract No. W-7405-eng-26

METALLURGY DIVISION

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DATE ISSUED

FEB - 8 1962

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ABSTRACT

The austenitic stainless steel-clad plate-type fuel element used in the SM-1 reactor contains a dispersion of 25.16 wt % UO_2 and 0.13 wt % B_4C dispersed in type 302B stainless steel. Subsequent to fabrication of the first fuel element loading for this reactor, an investigation was conducted to determine the magnitude of boron losses which occurred during sintering of the powder metallurgically prepared fuel compacts. The objective in this case was to determine the total boron losses in the completely processed and finished fuel plate. This was done by dissolving fuel plates in their entirety and chemically analyzing for boron. The accuracy of the analytical methods used was first established by analyzing powder mixtures containing the exact quantities of UO_2, B_4C, and stainless steel representative of a fuel plate. A negative bias was shown to exist. The conclusion was that the accuracy of the analysis of boron in the SM-1 fuel plate was $^{+0}_{-10}$%. Subsequent dissolution of 18 plates and results of the boron analyses revealed losses ranging from 9 to 50%. The average boron loss in the Core I loading was estimated to be 22%.

INTRODUCTION

The fuel component designed for service in the 10-Mw SM-1 reactor at Fort Belvoir, Virginia, consists of 18 composite fuel plates assembled in the manner illustrated in Fig. 1. Each fuel element contains, nominally, 515 g of U^{235} and 2.5 g of natural boron. Boron is added to aid control of the high initial reactivity of the heavily loaded core, and indirectly permits continuous operation of the reactor at full power for a period of 15 months without refueling.[1] The boron is incorporated in the

[1] J. E. Cunningham and R. J. Beaver, "Stainless Steel-Uranium Dioxide Fuel Components for Army Package Power Reactor," Nuclear Metallurgy V, 30, IMD Special Report Series No. 7, The Metallurgical Society of AIME, New York, 1958.

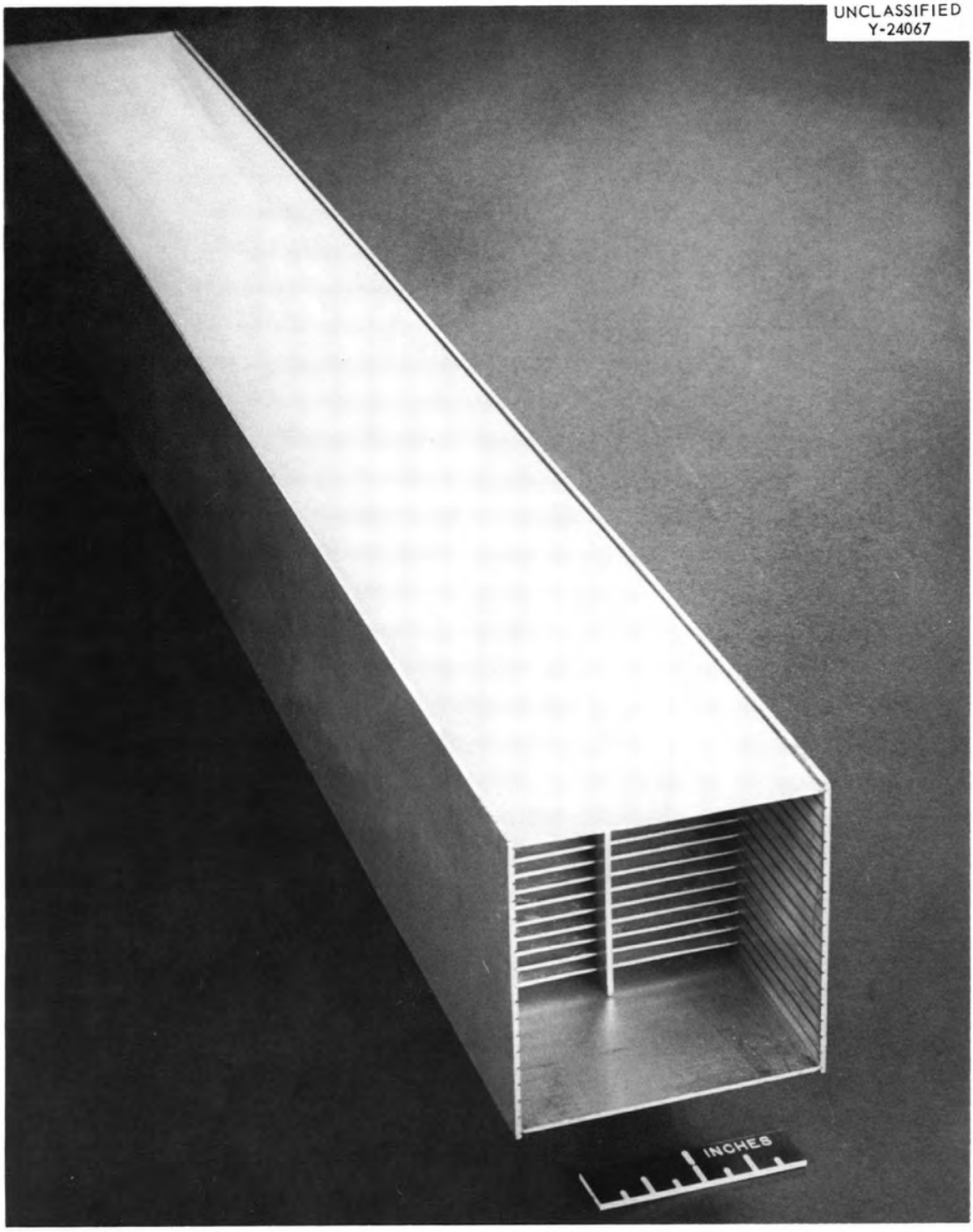

Fig. 1. Fuel Element Body of Eighteen-Plate Stationary SM-1 Fuel Element.

25 wt % UO_2–stainless steel fuel section of the plate as the compound B_4C, utilizing powder metallurgy processing. The quantity added to each plate is 0.188 g (ref 2). Subsequent to manufacturing the first core loading, a preliminary critical experiment indicated that the elements did not contain the specified quantity of boron.[3] Although previous work had indicated losses as high as 10% when sintering fuel compacts in hydrogen at 1175°C and the boron loading adjusted accordingly, it was conceivable that additional losses may have occurred during processing of the fuel plates. An investigation was initiated to determine the boron content in a number of finished fuel plates by complete dissolution of each plate followed by chemical analysis for boron. From these results, the average boron loss could be obtained and an estimate made of the actual boron content in the first fuel element loading of the SM-1 reactor. To accomplish this objective, the assistance of two outside laboratories, The Martin Company, Baltimore, Maryland, and the L. Pitkin Company, New York, New York, was enlisted to confirm the accuracy of chemical analyses for boron in stainless steel. Subsequently, The Martin Company also cooperated in the dissolution and analyses of several of the finished fuel plates. Concurrent with this program, The Martin Company conducted a critical experiment as an alternative method for predicting the boron loss in the Core I fuel elements.[4] This permitted a comparison of the average total boron contained by two independent methods.

EXPERIMENTAL PROCEDURE

Two laboratories in addition to the Oak Ridge National Laboratory (ORNL) were selected to analyze mixtures of known quantities of B_4C, UO_2, and stainless steel powders. These were: (1) The Martin Company (Nuclear Division), and (2) the L. Pitkin Company. No samples containing UO_2 were

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[2]R. J. Beaver et al., Specifications for the Army Package Power Reactor (APPR-1) Fuel and Control Rod Components, ORNL-2225 (July 24, 1957).

[3]Personal communication with Captain L. D. Crocker, Army Reactors Branch, AEC, Washington, D. C.

[4]J. F. O'Brien, Some Aspects of a Boron Determination for APPR-1, MND-E-1718-1 (Feb. 1959).

sent to the L. Pitkin Company because of lack of licensing agreement with the Atomic Energy Commission. It was assumed that the presence of UO_2 would not appreciably influence accuracy of the analytical methods for boron, and that results from the L. Pitkin Company would be valid.

Twelve standard samples were prepared by dispensing preweighed charges of 0.1880 g B_4C, 35.09 g depleted UO_2, and 203.8 g type 304B stainless steel powder into a weighed container. The quantity of stainless steel was based on the total grams of stainless steel in the finished composite plate, including both the stainless steel matrix of the fuel core and the cladding and framing material. Pertinent data of these samples are listed in Table 1.

Table 1. Composition of Standard Powder Mixtures Prepared for Establishment of Accuracy of Chemical Methods in Analyzing for Boron in Stainless Steel

	ORNL and The Martin Company		L. Pitkin Company	
Material	Weight (g)	Concentration (wt %)	Weight (g)	Concentration (wt %)
B_4C	0.1880	0.078	0.1880	0.092
B*	0.1444	0.060	0.1444	0.071
UO_2	35.09	17.22	0.00	0.00
Stainless Steel**	203.8	Balance	203.8	Balance

*Based on 75.86 wt % B in B_4C and 8.5×10^{-4} wt % B in stainless steel.

**Low-carbon type 302B stainless steel.

The sizes of the B_4C, UO_2, and stainless steel powders were -325, -170 +325, and -100 mesh, respectively. The B_4C selected was identical with that used in the fuel elements for the first core loading of the SM-1 reactor. Boron in this compound was established as 75.86 wt % by wet chemical analyses. Depleted UO_2 was used in those mixtures containing a fuel compound. The stainless steel powder was a low-carbon type 302B, whose composition includes 2.5 wt % Si. The boron content of the steel, 8.5×10^{-4} wt %, was based on two analytical chemical results at ORNL and one by the L. Pitkin Company. The boron content of the synthetic standard powder sample of 144.4 mg therefore includes both the boron in

the boron carbide and the boron in the stainless steel. Quantities in each sample are tabulated in Appendix I. The three laboratories each analyzed four samples; thus, results of twelve samples were available for evaluation. Analytical chemistry procedures used at The Martin Company and ORNL for determination of boron are presented in Appendix II. Those used at the L. Pitkin Company were not made available.

The reported boron contents of the standard samples were statistically analyzed to compare the results of three laboratories and to establish an arbitrary value of analytical accuracy. The "F" (variance ratio) test was applied to the data to mathematically ascertain if the analytical schemes of the laboratories yielded comparable results.[5]

Eighteen SM-1 stationary-type fuel plates, which had been rejected from the first core loading for failure to meet dimensional specifications, were dissolved and the total boron in each plate determined by the analytical chemical procedures identical to those employed for boron analysis of the standard powder specimens. These plates had been fabricated at 1150°C, cold reduced, and annealed in hydrogen for 3 hr at 1125°C in accordance with the manufacturing specifications.[2] Prior to destructively analyzing the plates, they were delivered to The Martin Company for reactivity measurements in their Zero Power Critical Facility. This experiment was conducted in an endeavor to correlate boron loss predicted by reactivity measurements with boron-loss data obtained by analytical chemistry methods.

DISCUSSION OF RESULTS

The uncertainties associated with predicting the exact quantity of boron in the standard powder mixtures are tabulated in Appendix III. It was concluded that these samples contained 144.4 mg B_4C ± 2%.

The boron results obtained from the analytical chemical work of the three laboratories are listed in Table 2. Both ORNL and The Martin Company

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[5]Communication with D. A. Gardiner, ORNL Mathematics Panel.

Table 2. Boron Content of the Standard Samples as Analytically Determined

Sample	Analytical Laboratory	Boron Found (mg)	Deviation from Theoretical* (mg)	(%)
R-1	ORNL	136.1	-8.3	-5.7
R-2	ORNL	136.2	-8.2	-5.7
R-3	ORNL	139.0	-5.4	-3.7
R-4	ORNL	141.2	-2.2	-1.5
R-5	The Martin Company	146.0	+1.6	+1.1
R-6	The Martin Company	133.0	-11.4	-7.9
R-7	The Martin Company	140.0	-4.4	-3.0
R-8	The Martin Company	130.5	-13.9	-9.6
R-25	L. Pitkin Company	144.2	-0.2	-0.1
R-26	L. Pitkin Company	147.0	+2.6	+1.8
R-27	L. Pitkin Company	145.2	+0.4	+0.3
R-28	L. Pitkin Company	143.9	-0.5	-0.3

$$^{*}\frac{-144.4 + \text{B found}}{144.4} \times 100$$

average data show a negative bias of 6 and 10%, respectively, indicative of boron losses of this magnitude by the analytical methods used. The L. Pitkin Company's results revealed an accuracy of ± 2%. Their specimens did not contain any UO_2 but their accuracy cannot be definitely attributed to this condition. A statistical treatment, described in Appendix IV, in which the "F" (variance ratio) test at a 95% significance level was used did not reveal a significant difference between the results of any of the laboratories. This test does not predict the overall accuracy of any laboratory but does demonstrate that the interlaboratory analyses are comparable. Correspondingly, the subsequent results of The Martin Company and ORNL in chemically analyzing for boron in the finished SM-1 fuel plate were assumed to be comparable.

Eighteen finished SM-1 fuel plates were completely dissolved and chemically analyzed — five by The Martin Company and thirteen by ORNL. The boron results are summarized in Table 3 and their distribution plotted in Fig. 2. Detailed data are listed in Appendix V. The method of

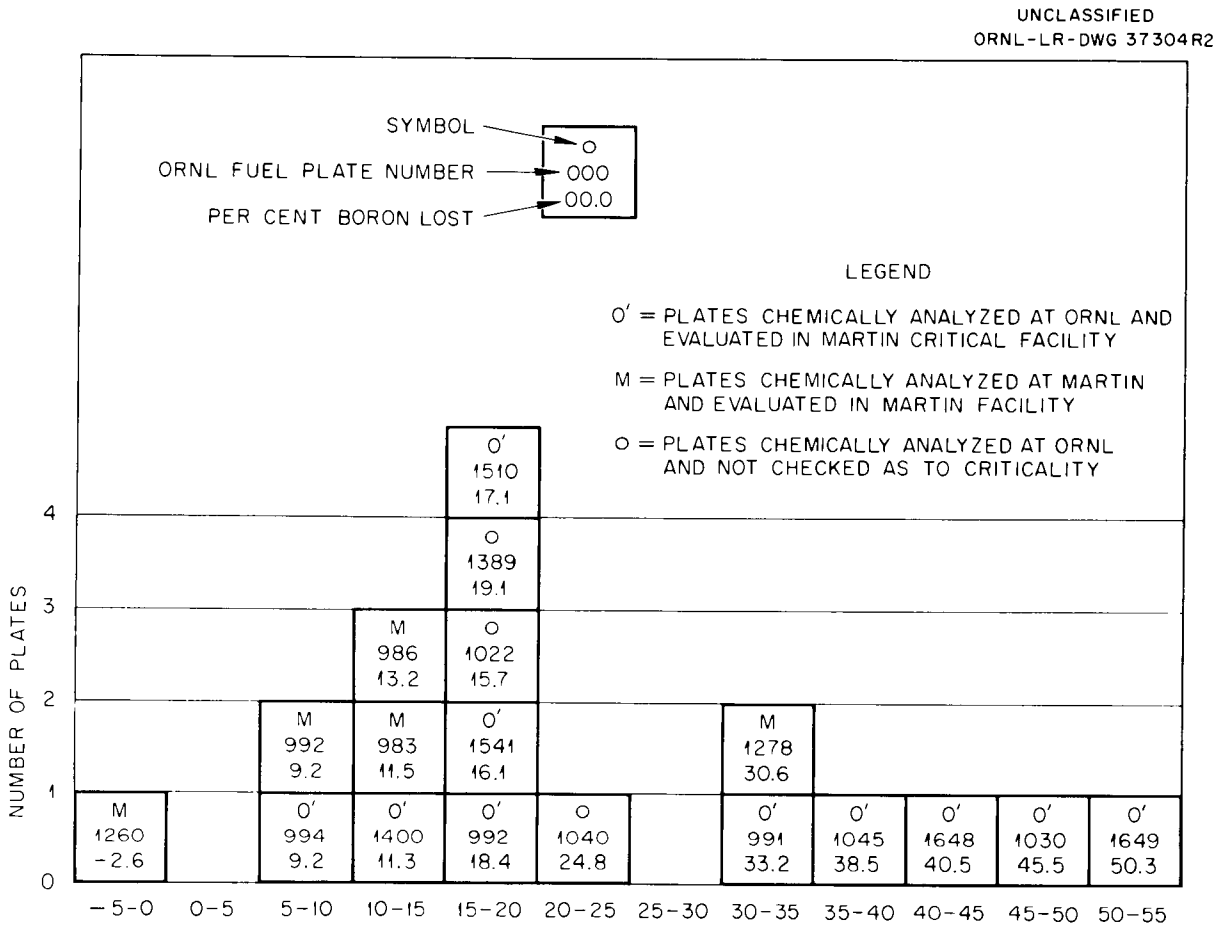

Fig. 2. Range of Boron Loss (%).

Table 3. Summary of the Results of the Boron Determination of SM-1 Core I Fuel Plates

Plate No.	Analytical Laboratory	Boron Reported (mg)	Deviation from Theoretical Loading of 144.4 mg B* (mg)	(%)
991	ORNL	96.7	-47.7	-33.2
992	ORNL	118.2	-26.2	-18.4
994	ORNL	131.5	-12.9	-9.2
1022	ORNL	122.0	-22.4	-15.7
1030	ORNL	79.0	-65.4	-45.5
1040	ORNL	108.9	-35.5	-24.8
1045	ORNL	89.1	-55.3	-38.5
1389	ORNL	117.1	-27.3	-19.1
1400	ORNL	128.5	-15.9	-11.3
1510	ORNL	119.9	-24.5	-17.1
1541	ORNL	121.5	-22.9	-16.1
1648	ORNL	86.2	-58.2	-40.5
1649	ORNL	72.0	-72.4	-50.3
983	The Martin Company	127.8	-16.6	-11.5
986	The Martin Company	125.3	-19.1	-13.2
992	The Martin Company	131.2	-13.2	-9.2
1260	The Martin Company	148.7	+4.3	+2.6
1278	The Martin Company	99.4	-45.0	-30.6

$$^{*}\frac{-144.4 + B \text{ found}}{144.4} \times 100$$

analyzing these data is described in Appendix VI. This analysis predicts the average boron content in the fuel plates to be 112.4 mg with an associated σ of 20.85 mg or 112.4 mg ± 43.99 mg with 95% confidence. The average boron loss was 22% although losses as high as 50.3% were reported. In comparison, a criticality test in The Martin Company Critical Facility indicated an average boron loss in the range of 25 to 32%.[4]

CONCLUSIONS

The boron content of mixtures of 35.09 g of UO_2, 0.1880 g of B_4C, and 203.8 g of type 304B stainless steel powder can be ascertained within limits of $^{+0}_{-10}$% by analytical chemistry methods.

The average boron loss in the fabricated and annealed fuel plates in Core I of the SM-1 reactor is estimated to be 22%. This value may be compared to an average loss of 27% as determined from fuel element worth data obtained in reactor critical experiments measured.

At a 95% confidence level, the quantity of boron in any randomly selected SM-1 Core I fuel plate is predicted to be 112.3 ± 43.7 mg.

ACKNOWLEDGMENT

The authors wish to express their appreciation to D. A. Gardiner of ORNL who was instrumental in selecting and describing the statistical methods involved; W. Laing and M. Hill of ORNL who contributed effort and time to the chemical aspects of the problem; and Dr. Bell at L. Pitkin Company and J. F. O'Brien at The Martin Company who coordinated the efforts of their respective companies in this program.

APPENDIX

APPENDIX I

INDIVIDUAL COMPOSITION OF THE STANDARD SAMPLES

Sample	B_4C (mg)	UO_2 (g)	Type 304B Stainless Steel
R-1	188.0	35.0887	203.7980
R-2	188.0	35.0899	203.7993
R-3	187.9	35.0899	203.7995
R-4	188.2	35.0901	203.7992
R-5	188.1	35.0896	203.7996
R-6	187.9	35.0893	203.7994
R-7	188.2	35.0897	203.7985
R-8	187.8	35.0899	203.7988
R-25	188.2	*	203.7985
R-26	188.0	*	203.7993
R-27	187.9	*	203.7992
R-28	187.9	*	203.7990

*Samples did not contain UO_2.

APPENDIX II

ANALYTICAL CHEMISTRY PROCEDURES FOR DETERMINATION OF BORON IN STAINLESS STEEL

I. The Martin Company's Method for Determination of the Boron Content of Stainless Steel-UO_2-B_4C Cermets and Stainless Steel-Boron Alloys

A method has been developed for the chemical analysis of the stainless steel-UO_2-B_4C cermets and stainless steel-boron alloys for boron content. The method is based on the removal of interfering cations by means of ion-exchange resins. Of the anions likely to be present, only CO_2 (picked up from the atmosphere) interferes in subsequent boron titrations. This CO_2 is removed when the slightly acid solution is boiled. Since boron escapes with the steam when an acid solution containing boron is boiled, a reflux arrangement is used consisting of a 300-ml Erlenmeyer flask fitted to a 50-cm air condenser with standard taper fittings. Both flask and condenser are made of Corning No. 7280 (boron-free) glass. After all interfering cations and anions are removed, the boron is titrated potentiometrically.

A 3-g sample is boiled in a mixture of acids under reflux conditions in boron-free glassware. Any insoluble residue is filtered off on quantitative grade filter paper. The filtrate is set aside (Solution A). The paper and residue are ashed at low heat in an electric muffle furnace and fused with Na_2CO_3 in a platinum crucible at 900°C. The melt is taken up in an excess of dilute H_2SO_4 at room temperature, boiled under reflux conditions in boron-free glassware, and added to Solution A. The volume of the combined solutions is adjusted to 500 ml. After thorough mixing, a 50-ml aliquot is pipetted into a 400-ml beaker of boron-free glass. The pH is adjusted to 1.90 with CO_2-free NaOH. This solution is passed through a prepared 65-cm bed of Amberlite IR-120 cation-exchange resin at a flow rate of one drop per second. A rinse, consisting of 150 ml of H_2O, is now passed through the bed at a flow rate of one drop per second. The resulting solution, now free of iron, nickel, chromium, and uranium, is collected in 400-ml beakers of boron-free glass. The pH is adjusted to seven with CO_2-free NaOH, then two drops of 1:4 H_2SO_4 are added. The

solution is boiled under reflux conditions in boron-free glassware, cooled to room temperature, then titrated potentiometrically using mannitol and a standard NaOH solution.

A trial run was made on five synthetic test samples by this method. To four of these samples, a known quantity of boron was added (in solution form, by means of a pipette, because such small quantities of solid boron cannot be weighed accurately on a gramatic balance). No boron was added to the fifth sample and it was run as a blank. The results of these five analyses are shown below. The percentages are based on a sample weight of 3 g.

Sample No.	Per Cent Boron Added	Per Cent Boron Found	Deviation	Per Cent Deviation
1	0.100	0.104	+0.004	4.0
2	0.167	0.172	+0.005	3.0
3	0.333	0.330	-0.003	0.9
4	0.500	0.490	-0.010	2.0
5(blank)	0.000	0.011	-	-

Average Deviation 2.5%

The method appears to be highly accurate, compared to published results by other methods, especially when the small percentage of boron present is considered.

II. ORNL's Method for Determination of Boron in Stainless Steel Fuel Plates (Microvolumetric Titration)

A. Dissolution

1. Cut the fuel plate into 2-in.-wide strips and put about 125-g portions into 2-liter quartz or Vycor reflux flasks.

2. Add aqua regia (3% HCl–1% HNO_3) in 25-ml portions through the top of the reflux condenser. Continue the addition of the acid mixture while heating at medium heat until approx 250 ml has been added. (Time, approx 4.0 hr.)

3. Cool the flask in a water bath and transfer the liquid portion into another 2-liter flask. While heating under reflux, add approx 25 ml of aqua regia followed by 100 ml of HCl in 25-ml portions to the transferred liquid. Reflux for about 1 hr.

4. Add aqua regia to the residue in the original flask and continue to heat until all the metal has dissolved.

5. Cool the flasks in a water bath and add an equal volume of water.

6. Pour the acid insolubles and the acid soluble portions into large plastic centrifuge cones. Centrifuge and decant the clear supernate. Wash the solids several times with water. Combine all liquid portions at this time. The total volume will be about 6 liters.

7. Fuse the acid insolubles in 2- to 4-g portions with 20 g of NaOH in nickel crucibles. Dissolve the melt with water in polyethylene containers. Acidify the dissolved melt with HCl and stir it for 10 min in order to remove any dissolved CO_2.

8. Combine the separate fused portions but do not mix with the acid soluble portion. The volume will be about 3 liters.

B. Separation

Cations which may interfere in the subsequent titration are removed on a Dowex 50W resin column (50 to 100 mesh). The columns are 20 cm long, 10-mm-ID, and the resin is added to a height of 8 to 9 cm. This column size will remove the cations from 100 mg of stainless steel. The resin, however, must be conditioned with NaOH, washed with water, converted to the acid form with 6 N HCl, and finally washed with water before use.

1. The resin column should be in the acid form and washed free of excess HCl. Dilute an aliquot of the sample (containing not more than 100 mg of stainless steel) with three times its volume of water and pass this solution through the prepared resin column.

2. Wash the column with small portions of water and catch all effluent to a total volume of 30 ml. This portion is now ready for titration of the boron.

C. Titration

The aliquots are titrated by the null-point method using a grounded Beckman Model G pH meter and a 1 cc/in. micrometric syringe filled with 0.05 N NaOH. With this instrumentation, a factor of 0.61 μg B/dial division is obtained.

Potassium oxalate is added before titration in order to complex any cations which may have come through the resin column.

1. Add 2 ml of 1 M potassium-oxalate solution to the column effluent and raise the pH of the solution to 6.5 to 7.0 by dropwise addition of 1 M NaOH.
2. Start a flow of nitrogen gas over the sample beaker and, using the micrometric buret, titrate to a pH of 8.0.
3. Add 4 ml of 10% mannitol solution and wait for 30 sec.
4. Titrate back to pH 8.0 with the micrometric buret.

D. Calculations

A factor is established by titration of blanks and 50 μg B standards. Each should have been adjusted to the same acidity with the same kind of acid as the samples and also put through the resin column.

$$\text{Factor, } \mu\text{g B/division} = \frac{\mu\text{g B in aliquot}}{\text{standard titration} - \text{blank titration}} .$$

This factor is usually 0.610 μg B/division.

Per Cent Boron in Sample =

$$\frac{(\text{Total divisions} - \text{blank})(\text{Factor})(\text{dilution factor})}{(10)(\text{sample weight, mg})} .$$

The L. E. at 95% confidence level for titration of standards is 1%. The L. E. at 95% confidence level for samples carried through the entire procedure is thought to be 5%.

APPENDIX III

FACTORS CONSIDERED IN DETERMINING BORON CONTENT IN SYNTHETIC SAMPLES

1. Boron content of SM-1 grade B_4C as determined at ORNL: 75.86% (av)
2. Boron content of stainless steel powder: 0.001% (ORNL)
   0.0007% (L. Pitkin Company)
3. Nominal boron content per standard sample:

B_4C contribution	0.1880 (0.7586) =	142.62 mg
Matrix contribution	203.8 (8.5×10^{-6}) =	1.73 mg
	Total	144.35 mg

4. Weighing error:

   B_4C contribution (see Appendix I) $\pm 4 \times 10^{-4}$ (0.7586) = ±0.3 mg

   Matrix contribution Negligible
5. Uncertainty in absolute boron content of constituents:

   B_4C contribution 0.1880 (1.5×10^{-2}) = ±2.8 mg

   Matrix contribution Negligible
6. Theoretical boron content of standard sample:

   144.4 ± 3.1 mg

   (144.4 mg used in subsequent calculations).

APPENDIX IV

INTERLABORATORY COMPARISON OF DATA OF ACCURACY OF ANALYTICAL CHEMISTRY IN DETERMINING BORON BASED ON THE "F" TEST

Let X_i = Reported boron content per standard sample less the calculated value (144.4 mg).

Subscripts refer to the participating laboratory: O is ORNL, P is L. Pitkin Company, and M is The Martin Company.

Sample	X_O	Sample	X_P	Sample	X_M
1	-8.3	25	-0.2	5	+1.6
2	-8.2	26	+2.6	6	-11.4
3	-5.4	27	+0.8	7	-4.4
4	-3.2	28	-0.5	8	-13.9
ΣX_O =	-25.1	ΣX_P =	2.7	ΣX_M =	-28.1

$$\text{Let } G = \frac{(\Sigma X_O + \Sigma X_P + \Sigma X_M)^2}{12} = 254.8$$

Source of Error	df*	Sum of Sq	Mean Sq = $\frac{\text{Sum of Sq}}{\text{df}}$
A = Among Labs	2	$\frac{(\Sigma X_O)^2 + (\Sigma X_M)^2 + (\Sigma X_P)^2}{4} - G$	MS(A) = 72.12
W = Within Labs	9	$\Sigma X_i^2 - G$	MS(W) = 19.05
Total	Σ_{df} = 11		

$$F = \frac{MS(A)}{MS(W)} = 3.79$$

Since $F < 4.26$, a significant difference among the participating laboratories cannot be detected at the 95% level.

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*Degrees of freedom.

APPENDIX V

REPORTED BORON IN SM-1 CORE I FUEL PLATES ANALYZED IN TOTO AT ORNL

Plate No.	Aliquot	Boron (mg) Acid Soluble Fraction	Insoluble Fraction
991	a	25.92	73.01
	b	27.12	68.87
	c	25.44	70.00
	d	24.92	71.63
992	a	24.21	94.53
	b	22.38	94.12
	c	24.75	93.56
	d	25.11	94.12
994	a	35.22	96.08
	b	36.30	95.84
	c	35.22	95.72
	d	35.58	95.84
1030	a	48.78	33.74
	b	44.55	32.46
	c	46.38	33.05
	d	44.01	32.85
1045	a	28.74	60.12
	b	28.56	61.67
	c	27.81	60.86
	d	28.17	60.39
1400	a	27.27	99.74
	b	28.74	100.65
	c	29.28	101.57
	d	26.91	99.83
1510	a	33.12	85.74
	b	32.94	87.11
	c	33.12	86.10
	d	34.59	86.75
1541	a	35.59	86.84
	b	36.06	85.10
	c	35.49	86.19
	d	34.95	--

APPENDIX V (Continued)

Plate No.	Aliquot	Boron (mg) Acid Soluble Fraction	Insoluble Fraction
1648	a	16.83	69.45
	b	13.17	71.28
	c	19.23	69.09
	d	14.82	69.09
	e	18.12	
1649	a	17.94	55.82
	b	14.64	56.00
	c	16.29	56.55
	d	14.82	56.00

APPENDIX VI

CALCULATED BORON CONTENT OF RANDOMLY SELECTED FUEL PLATES

Let T_i = boron content in any plate, then the average boron content per plate is:

$$\overline{T} = \sum_{i=1}^{m} \frac{T_i}{n} .$$

Let σ = estimated standard deviation, and $t_i = T_i - \overline{T}$.

Then $\sigma = \left[\frac{\sum_{i=1}^{m} t_i^2}{n - 1} \right]^{1/2}$.

Employing the data presented

$\overline{T}$ = 112.4 mg B/plate

σ = 20.85 mg B/plate with 17 deg of freedom

T_i = 112.4 ± 44.0 mg B with 95% confidence.

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