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NONDESTRUCTIVE DETERMINATION
OF U^{235} CONTENT OF SNAP FUEL RODS
VOL. III - EQUIPMENT DESCRIPTION AND
PRODUCTION QUALITY CONTROL APPLICATIONS

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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NONDESTRUCTIVE DETERMINATION
OF U^{235} CONTENT OF SNAP FUEL RODS
VOL. III - EQUIPMENT DESCRIPTION AND
PRODUCTION QUALITY CONTROL APPLICATIONS

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ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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ABSTRACT

An autoradiometric test method and associated production inspection equipment have been developed for nondestructively measuring the incremental and total U^{235} content in cylindrical SNAP fuel rods. The determination is accomplished by measuring the intensity of the 185 kev gamma photo peak from U^{235} and comparing this value with a reference standard of known composition. A prototype single channel analyzer scintillation counting system and special fuel rod scanning mechanism are described. A statistical evaluation of the autoradiometric and destructive chemical methods disclosed a correlation coefficient between the two methods to be 0.976. The precision of the autoradiometric method was 0.023 wt % uranium when the test samples were fully enriched 10% U-Zr SNAP fuel rods.

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I. INTRODUCTION

A nondestructive test method and associated equipment have been developed for determining the incremental and total U^{235} content in cylindrical SNAP fuel rods. This determination was necessary

- 1) to assure that there was the correct total amount of U^{235} in each fuel rod, and

- 2) to assure that there were no localized areas high in U^{235} which could result in hot-spots during reactor operation.

Chemical analyses of end samples had previously been relied upon to determine the total amount of uranium in the fuel rods, but the chemical data were, of course, subject to the usual limitations of destructive analysis. In addition, the U^{235} content had to be calculated from isotopic analyses by a mass spectrograph in a remotely located laboratory. Localized dilution of U^{235} with natural uranium could not be determined by the end sampling plan. Clearly, a method was required to measure the incremental and total U^{235} content in each fuel rod. The nondestructive system, called the Autoradiometric Uranium Analyzer (AUA), has successfully satisfied the above requirements. The AUA is the culmination of a long term three part effort, the first two portions of which are summarized in the following section.

Figure 1. U^{235} Gamma Spectrum,
Showing Position of Lower Level
Discriminator of Single Channel
Analyzer

Figure 2.
Special Collimator
Design

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II. FEASIBILITY STUDY AND SYSTEM DESIGN

An initial study was conducted by Nelson, Greenfield, and Koontz⁽¹⁾ to determine whether an autoradiometric counting technique could be applied to the SNAP fuel rods, which ranged from 9/16 to 1-1/4 in. OD and up to 16 in. long. The material used for all of the fuel rods was a 10% uranium (93% U^{235})-zirconium alloy which was hydrided to an effective H/Zr ratio of about 1.7. The concept of determining the U^{235} content of fuel was not new, since this method has been extensively employed in the quality control testing of flat fuel elements, using equipment ranging from simple G-M instruments to sophisticated scintillation counters with elaborately automated sample handling mechanisms and computers for high speed data reduction. However, the autoradiometric method has seldom been applied to cylindrical fuel rods.

Work performed by Nelson, Greenfield and Koontz showed that the autoradiometric method of determining total and incremental U^{235} in fuel rods could be performed on a production quality control basis. The proper positioning of a pulse-height analyzer base line was determined (see Figure 1), a special scintillation counter collimator was designed to permit subsurface as well as surface sampling (see Figure 2), and counting parameters were established by applied statistical analysis.^(2,3) Results of the feasibility study were used in the design of a prototype production system for detecting and recording the intensity of the gamma radiation emitted by the U^{235} in the fuel alloy and for mechanically handling and scanning SNAP fuel rods.

Figure 3. Block Diagram Electronic System

Figure 4. Nuclear Instrumentation Console

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III. APPARATUS

The prototype production system for SNAP fuel rods includes an electronic gamma radiation detection and recording section, a scanning mechanism, and reference standard fuel rods. The complete system is known as the Autoradiometric Uranium Analyzer (AUA).

A. ELECTRONIC SECTION

The electronic gamma radiation detection and recording section is a single channel analyzer, assembled from commercially available components. A block diagram and photograph are shown in Figures 3 and 4. Principal elements of the gamma radiation detection and recording section are as follows:

A lead collimator as shown in Figure 2 admits the gamma radiation from a finite portion of the U^{235} in the fuel rod to the detector. The fuel-side aperture in the collimator is 1/4 in. wide and 1 in. long, with the 1-in. dimension oriented along the axis of the fuel rod. The detector side of the collimator has been shaped to permit the sampling of not only the surface but also the center portion of the fuel rod.⁽¹⁾ Collimator height may be adjusted to maintain a constant distance between the fuel-side opening in the collimator and the surface of fuel rods of varying diameters.

The NaI (Tl) scintillation crystal, in which the gamma photons from the sample interact with the orbital electrons by photoelectric absorption, Compton scattering, or pair production, and produce light pulses the intensities of which are proportional to the energy of the incident gamma radiation.

A photomultiplier tube, connected optically to the scintillation crystal, converts the light pulses into an electrical signal. The photomultiplier tube also amplifies the signal to provide input to the linear amplifier of the spectrometer. A simple cathode follower circuit acts as an impedance match between the photomultiplier and the linear amplifier.

The pulse height analyzer provides a means of selecting pulses with a narrow range of amplitudes for input to the linear count rate meter, to the recycling digital scaler, and to a totalizing scaler. The pulse height analyzer base line is adjusted to exclude all pulses from gamma photons with energies less than 130 kev, and to accept all energies above 130 kev.

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a. Overall View

7568-18105

b. Closeup of Scintillation Probe

Figure 5. AUA Scanning Mechanism

The linear count rate meter accepts pulses from the analyzer and converts these pulses into a panel meter reading and to a voltage to drive a strip-chart recorder, which in turn provides a graphic record of circumferential and axial uranium distribution.

The automatic recycling digital scaler, which is controlled by an electronic timer, activates the digital printer every 50 sec with a signal, the amplitude of which is proportional to the U^{235} content in a given 1-in. length of fuel rod.

The totalizing scaler, controlled by an electro-mechanical timer, provides data proportional to the total U^{235} content of that portion of the fuel rods which has a uniform diameter.

B. SCANNING MECHANISM

The AUA scanning mechanisms, as shown in Figure 5, was designed

- 1) to hold bare fuel rods and completed elements of varying dimensions up to 1-1/4 in. OD and 16 in. long at a constant distance from the radiation detector,
- 2) to rotate the fuel rods at a constant speed,
- 3) to translate the radiation detector assembly at a constant rate from one end of the fuel rod to the other, and
- 4) to shut off the scanning mechanism at the end of a scanning run.

Sets of special collets were designed

- 1) to tightly grip each end of the fuel rods so that the rods could be rotated without slipping,
- 2) to maintain the fuel rods in axial alignment,
- 3) to permit rapid insertion and removal of rods, and
- 4) to achieve all of the above without obscuring more than about 1/4 in. of the ends of the fuel rods.

The variable speed drives on the rotating and translating mechanisms were adjusted to rotate the fuel rods at a speed of 1 revolution per 50 sec and to translate the detector assembly on the traversing carriage at a linear speed of 1 in. per 50 sec. Hence, the detector assembly advances one inch axially as

the fuel rod rotates once, which results in a helical scanning path on the surface of the fuel rod. Constant scanning and rotating speeds are maintained by employing constant voltage transformers (saturable reactors) and flywheels on each of the two variable speed drives. Tachometers are provided to monitor the speed settings. Two switches permit the rotating and translating functions to be activated independently. A locking knob permits the traversing carriage to be engaged or disengaged from the translating lead screw by means of a split nut device. The entire AUA scanning mechanism is assembled on a rigid base to maintain alignment of the drive units, and to permit the entire mechanism to be readily moved from one location to another.

C. REFERENCE STANDARDS

Reference standard fuel rods have been provided for each of the two fuel rod sizes which have been analyzed to date. The standards measure nominally 9/16 and 1-1/4 in. OD and about 4 in. long. They are of the same nominal chemical composition as SNAP fuel rods (fully enriched hydrided 10% U - 90% Zr). Since the AUA is sensitive to the dimensions as well as the chemical and isotopic composition of fuel rods, separate reference standard fuel rods are required for any change in these fuel rod parameters. In the future, additional problems may arise from residual fission product activity if reprocessed uranium is employed in fuel alloys.

The present reference standards were selected by comparing the AUA count rates of a series of fuel rods. A statistical evaluation showed that two fuel rods from the series exhibited count rates within 1% of each other, and could be employed interchangeably. Since these two rods were considered to be identical, one was retained as a reference standard and the second rod was destroyed for chemical analyses. The method of sample preparation plus the results of the "round robin" chemical analyses may be found in Appendix I.

D. INITIAL SYSTEM TESTS

The entire AUA system was tested and evaluated prior to release to Quality Control personnel. Included in the evaluation were component reliability, system stability, and defect resolution tests. The first two tests were accomplished by counting the reference standard and background at least once per day for several months. The electronic system was left on continuously. Several

failures in the scaler decades occurred, and the time base of the electronic timer drifted. These problems were satisfactorily corrected. It was found that instrument stability was affected by hourly line voltage fluctuations. This was partially corrected through the use of an electronic line voltage stabilizer. Defective electronic components requiring replacement plus adjustments in the instrument settings seriously affected the long term stability of the system. As a result, it was decided to accept small changes in instrument sensitivity by referring all fuel rod analyses to the reference standards, which were to be counted at the beginning and end of every day's analyses, and before and after every fourth fuel rod scanned in any one day.

The ability of the system to resolve localized areas, which were high or low in U^{235} , was checked experimentally. One test utilized a piece of fully enriched uranium foil cemented to the outside surface of a fuel rod to simulate a potential "hot" spot. The foil measured 1/32 in. thick, 1/4 in. wide and 1/2 in. long. To the same rod a piece of 1/16-in. thick lead measuring 3/8 in. wide by 1-1/2 in. long was attached to simulate an area which was low in uranium. The long dimension of each of the attached foils was oriented parallel to the axis of the rod. Results of this test are shown in Figure 6a. Note that the lead foil was long enough to have been detected on two successive revolutions of the fuel rod. Resolution of smaller areas were not considered, since the fuel rods were to be tested after having been extruded. Hence, inhomogeneities would be considerably elongated in the direction of extrusion. Such an inhomogeneous fuel rod section was detected at the end of one extrusion. The AUA trace, as shown in Figure 6b, indicates that there was a 33% decrease in U^{235} from one end of the fuel rod to the other (right to left) and that there was a 19% circumferential difference in U^{235} content. Cyclic peaks and valleys represent circumferential inhomogeneity; from one peak to the next denotes one revolution of the fuel rod.

a. Resolution Test

b. SNAP 8 Rod with
Heterogeneous U²³⁵
Distribution

c. SNAP 8 Rod with
Homogeneous U²³⁵
Distribution

Figure 6. Scanning Records

IV. PRODUCTION TESTING PROCEDURE

During production runs, fuel rods have been tested with the AUA using the following procedure.

A. COUNTING THE REFERENCE STANDARD

The reference standard for the particular type of fuel rod being scanned is counted prior to the first rod, after every fourth rod, and after scanning of the last rod for that day. To achieve uniform results, it is mandatory that the scintillation probe be located over the same portion (e. g., the center) of the reference standard, which is continuously rotated during the counting period to minimize the effects of any circumferential inhomogeneity in uranium content. The counting period of the reference standard is the same as that used for the fuel rods. Resulting counts from the digital printer and the total count scaler are recorded in the AUA Daily Log Sheet (Table 1, Appendix).

B. COUNTING FUEL RODS

Fuel rods are counted in much the same manner as the standard. However, each fuel rod is scanned from end to end and a chart recorder trace is obtained in addition to the data from the recycling timer and the total count scaler. When scanning, the fuel rods are continuously rotated at 1 revolution per 50 sec and the scintillation probe is translated axially at a rate of 1 in./50 sec. After counting a fuel rod, the strip chart as shown in Figure 6c is removed, identified, and attached to the tape from the digital recorder. Data from the digital recorder plus the reading on the total count scaler are entered in the AUA Daily Log Sheet (Table 1). The entire scanning period, including set-up time, is approximately 15 to 20 minutes for a 16-in. long fuel rod.

C. CALCULATION OF INCREMENTAL AND TOTAL U^{235}

The weight percent U^{235} in any one inch length of fuel rod is calculated by comparing the count rates produced by the fuel rod with the average count rate from the reference standard. Over the range of interest, it has been found that a linear relationship exists between count rates and U^{235} content, as shown in Equation 1.

$$\frac{U_F}{U_{Std}} = \frac{I_F}{I_{Std}} \quad \dots(1)$$

where:

U_F = Weight percent U^{235} in a 1-in. length of fuel rod.

U_{Std} = Weight percent U^{235} in the reference standard.

I_F = Count rate from any 1-in. length of fuel rod.

I_{Std} = Average 50-sec count rate from the reference standard.

Equation 1 is applicable for calculating maximum and minimum values of U^{235} in a fuel rod. Average values of the fuel rod are calculated by substituting data from the total count scaler in Equation 1 for the digital recorder data. In like manner, any isolated portion of the strip chart recorder trace may be converted to weight percent U^{235} by using the average count rate value from the reference standard strip chart recorder trace.

D. CALCULATION OF TOTAL URANIUM

Total uranium concentrations are calculated by dividing the U^{235} isotopic concentration into any of the above weight percent U^{235} values, as shown in Equation 2.

$$\text{wt \% } U^{\text{Total}} = \frac{U_F}{U^{235} \text{ isotopic ratio}} \quad \dots(2)$$

Example:

Weight percent U^{235} (U_F) in fuel is 9.20.

Isotopic concentration of U^{235} in uranium fuel is 93.2% or 0.932.

The weight percent of total uranium in the fuel is:

$$\text{wt \% } U^{\text{Total}} = \frac{9.20 \text{ wt \% } U^{235}}{0.932} = 9.87$$

V. EVALUATION OF THE AUTORADIOMETRIC METHOD

An evaluation test was performed to compare the uranium contents in SNAP 8 fuel rods by the autoradiometric, x-ray fluorescence, and wet chemical methods. Twenty fuel rod samples with total uranium contents ranging from about 6.0 to 10.5 wt % were employed. The initial test called for twenty samples to be examined for five consecutive days by the autoradiometric method, and then by the x-ray fluorescent method. In these tests, the autoradiometric method scanned or sampled the circumference (side) of a 1-in. length of rod at the center of each rod, while x-ray fluorescence was employed to examine an area about 3/8 in. diameter at one end of each rod. Statistical evaluation of the resulting data from these tests showed a correlation coefficient of only 0.307. The reason for this poor correlation was presumed to lie in the fact that the autoradiometric method scanned the side of the rod, while the x-ray fluorescence method examined one end. A slight axial (end to end) nonuniformity in each sample rod could account for the difference in uranium values from the two analytical methods.

A second series of correlation tests were designed to overcome the shortcomings of the first series by

- 1) confining the sampling area to one end of each fuel rod,*
- 2) by randomizing the order in which the samples were examined,
- 3) by conducting the x-ray fluorescent determinations early in the morning (a time of low interference) as well as during regular daytime working hours.

After completion of these tests, each of the 20 fuel rods was sampled for wet chemical analysis.

The results of the second series of tests were highly significant. A summary of the statistical evaluation of the data disclosed the following:

*The AUA was modified to examine the end only with a 3/8 in. collimator

<u>Test Methods</u>		<u>Correlation Coefficient</u>	<u>Remarks</u>
1.	Autoradiometric and x-ray fluorescence (daytime)	0.914	High degree of relationship
2.	Autoradiometric and x-ray fluorescence (3 to 6 a.m.)	0.976	No significant difference between methods
3.	Autoradiometric and wet chemistry	0.896	High significant correlation

In addition to these results, statistical studies disclosed the following values for the precision of the test methods:

<u>Methods</u>	<u>Precision (wt % U)</u>
Autoradiometric	0.023
X-ray fluorescence (3 to 6 a.m.)	0.109
X-ray fluorescence (daytime)	0.216

Regression curves and additional details of the statistical evaluation may be found in Appendix II.

VI. DISCUSSION OF PRODUCTION TESTING RESULTS

The AUA system has been successfully operated for the past two years to check the U^{235} homogeneity and content of all SNAP fuel rods fabricated at AI. Even before absolute values for the reference standards had been established, useful homogeneity data were obtained and recorded. Since all of these values were related to individual reference standards, these relative numbers (counting data) were converted to actual U^{235} values when the lengthy analyses of the reference standards had been completed.

In addition to its use in determining the total and incremental U^{235} in bare fuel rods, the AUA has been continuously employed as a final check on the identity of all loaded fuel elements. Since both fully enriched and natural uranium fuel rods are processed simultaneously, there is a possibility of errors occurring in loading of fuel rods. The AUA rapidly determines the isotopic identity of fuel elements by a single comparative count of all completed fuel elements. Practice has shown that enriched fuel elements produce a count rate which is about 24 times greater than fuel elements containing natural uranium fuel rods. A single 1-min counting period is normally sufficient for this inspection procedure. Inspection records show no incidence of erroneous isotopic identification of fuel rods.

These advances are relatively new, and despite the capabilities of the AUA in determining U^{235} directly, specifications still appear with requirements for total uranium and isotopic concentration as a means of arriving at U^{235} content. This type of requirement is a throw-back to the time when wet chemical or x-ray fluorescent methods followed by mass spectrographic analyses were the only techniques available for determining U^{235} content. Displacing an established practice is often a slow and painful process, but eventually it is anticipated that the AUA, which nondestructively measures U^{235} directly on a 100% basis, will supplant all requirements for indirect destructive analyses of a small fraction of total number of production fuel rods.

VII. CONCLUSIONS

1. The autoradiometric method is a rapid, precise, and economical nondestructive method of measuring the incremental and total U^{235} content of all cylindrical SNAP fuel rods.
2. Precision of the method has been calculated to be 0.023 wt % U. The accuracy is dependent on reference standards, one of which is required for each size and composition of fuel rod to be tested.
3. The linear rate of scanning SNAP fuel rods is about 1 in./min. Inspection rate is about three rods per hour including a check of the reference standard.
4. The autoradiometric method provides a rapid means of determining the isotopic identity of the fuel rods contained in completed SNAP fuel elements.

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APPENDIX I

CHEMICAL ANALYSIS DATA FOR REFERENCE STANDARD

Tables 1 and 2 list the chemical analyses of the SNAP 8 AUA Reference Standard Fuel Rod. For total uranium analyses, aliquots were submitted to three separate laboratories. Three distinct analytical methods were employed. For uranium isotopic analyses, two laboratories submitted mass spectrographic results.

The solid fuel samples were prepared for analysis by crushing and grinding followed by dissolution in HCl-HF and HNO₃ acids. The original samples were then divided into 4 equal aliquots for use by each of the laboratories participating in the "round robin" analyses.

TABLE 1
COMPILATION OF TOTAL URANIUM ANALYSES

Vendor	AUA Reference			Standard Fuel Rod	
	Metals and Controls Inc.	Union Carbide Oak Ridge	Atomics International	Atomics International	Average*
Analytical Method	X-Ray Spectrographic	Potentiometric Titration	Volumetric	X-Ray Spectrographic	
Weight Percent Uranium	9.93	9.81	10.11	10.09	
	9.93	9.78	9.86	9.97	
	9.87	9.77	9.95	10.09	
			9.99	10.11	
				10.14	
				10.09	
				10.04	
				10.12	
				10.04	
				9.95	
				10.04	
				10.13	
				10.13	
				10.09	
Average	9.91	9.79	9.98	10.07	9.90

*The significantly greater variations in the AI x-ray fluorescence method as compared to the volumetric method excluded it as being valid.

TABLE 2
 COMPILATION OF WEIGHT PERCENT U^{235} ANALYSES
 STANDARD FUEL ROD

Vendor	Union Carbide	Phillips - NRTS	Average
Analytical Method	Mass Spectrometry	Mass Spectrometry	
Weight Percent U^{235}	92.44 92.39	92.49	92.46
Average	92.42	92.49	

APPENDIX II

CORRELATION DATA AND REGRESSION CURVES FOR CHEMICAL AND AUA DATA

This appendix contains a statistical evaluation of correlation tests of twenty SNAP 8 fuel rods by the autoradiometric, x-ray fluorescence (conducted between 3 a.m. and 6 a.m., as well as during regular daytime working hours), and wet chemical methods. Included are calculated values for correlation coefficients, regression analyses, precision for determining wt % uranium, and calculations of variance.

1. The following correlation studies were made:

a) The calculated correlation coefficient between uranium determinations by the autoradiometric method and the x-ray fluorescence method of analysis, as performed during the daytime, was 0.914, thus indicating a high degree of relationship between the two methods of determining uranium.

b) The calculated correlation coefficient between uranium determinations by the autoradiometric method and the x-ray fluorescence method of analysis, as determined during the period from 3 to 6 a.m., was 0.976. This correlation, along with the correlation listed in a), indicated that the two methods of determining uranium can produce essentially the same results and thus should be able to be used interchangeably.

c) The calculated correlation coefficient between the wet chemical analysis method of determining uranium and the autoradiometric method was 0.896. This correlation coefficient is highly significant.

2. Various regression studies were made to determine the estimated relationship between the three methods of determining uranium. The results of these regression studies are shown on Figure 7.

3. The following general statistical studies were made:

a) Estimates of the precision of the autoradiometric and the x-ray fluorescence method of determining uranium are as follows: the precision of the autoradiometric method of determining uranium was 0.023 wt % U; the precision of the x-ray fluorescence method of determining uranium as analyzed during the period 3 to 6 a.m. was 0.109 wt % U; the precision of the x-ray fluorescence method of determining uranium determined during the daytime was 0.216 wt % U.

a. Showing Relationship Between
 Autoradiometric and X-Ray,
 Daytime

b. Showing Relationship Between
 Autoradiometric and X-Ray
 3 to 6 a.m.

c. Showing Relationship Between
 Chemical Analyses and
 Autoradiometric

Figure 7. Regression Analyses

Tests have been made which show the variance (precision squared) of uranium determinations by the autoradiometric method were significantly smaller than the variance of the uranium determinations by the x-ray fluorescence method. These tests also show that the variance of the uranium determinations by the x-ray fluorescence method, as made during the period of 3 to 6 a.m., was significantly smaller than the x-ray analyses made during the daytime. The above tests were made at the 0.01 significance level (99% confidence level). However, it must be noted that both methods of determining uranium were not utilized in the normal manner for the uranium determinations required for this experiment, thus it is possible that the estimates of the precision (variance) are higher than would normally be expected.

b) Several tests were made to determine if there was a significant difference in the amount of uranium analyzed by the three different methods. These tests showed that the x-ray fluorescence method generally showed less uranium than did the wet chemical analysis method. Also, there was no significant difference between the amounts of uranium analyzed by the autoradiometric method and the wet chemical method. The above tests were made at the 0.10 significance level (90% confidence level). In addition, a test, made at approximately the 0.20 significance level (80% confidence level) showed that the autoradiometric method generally shows less uranium than the wet chemical method.

TABLE 3

TYPICAL AUA DAILY LOG SHEET

DATE 4-17-63

SNAP

OPERATOR L. F. Brown

1	2	3	4	5	6	7	8	9	
Rod Number	Total Counts 11 runs	Average Count Rate	Maximum Count Rate	Minimum Count Rate	Maximum wt % U ²³⁵	Minimum wt % U ²³⁵	Average wt % U ²³⁵	Accept	Reject
604-2 Std	921,049	83,847							
614-3	924,452		84,839	83,509	9.57	9.41	9.50	X	
614-4	923,045		84,490	82,873	9.54	9.35	9.49	X	
617-6	923,212		84,876	83,043	9.58	9.36	9.49	X	
619-6	916,665		84,927	82,754	9.59	9.35	9.42	X	
604-2 Std	920,662	83,697							
620-5	917,543		84,047	82,373	9.51	9.31	9.43	X	
600-1	901,447		82,312	80,687	9.31	9.13*	9.26		X
618-5	924,005		84,904	83,284	9.60	9.43	9.50	X	

*Below tolerance