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SUBJECT: Evaluation of Gamma-Ray Attenuation Techniques for
Measuring the Density and Homogeneity of Thorium
Oxide Slurries Circulating at 300°C

To: Distribution

From: M. Richardson and A. S. Kitzes

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TABLE OF CONTENTS

I.	Introduction.....	1
A.	Scope of investigation.....	1
B.	Berman and Harris technique.....	1
II.	Determination of pulse height distribution.....	2
A.	Indirect measurement-integral bias curve using decimal scaler.....	3
B.	Direct measurement of pulse amplitude distribution using a single channel analyzer.....	6
III.	Comparison of indirect and direct methods for determining pulse amplitude distributions.....	7
IV.	Theory.....	8
V.	Method used in measurements.....	9
A.	Source and tube carrier for horizontal pipe used on 200A loop 3-in. pipe.....	10
B.	Carrier used on vertical pressurizer on 200A loop, 6 in. schedule 160 pipe.....	10
VI.	Discussion of experimental results using decimal scaler.....	10
VII.	Results using single channel analyzer.....	13
VIII.	References.....	15
X.	Appendix A - description of instruments.....	16

TABLE OF FIGURES

1. Decimal counter with phototube and recorder attached.....	17
2. Single channel analyzer.....	18
3. Integral bias curves of Co^{60} and Ce^{137}	19
4. Integral bias curve taken in position on slurry blanket mock-up.....	20
5. Cobalt 60 spectrum taken with single channel analyzer.....	21
6. Cesium 137 spectrum taken with single channel analyzer.....	22
7. Effect of excess high voltage on crystal discrimination of Co^{60} spectrum.....	23
8. Gamma scanning equipment for horizontal pipe.....	24
9. Sketch of single screw lift.....	25
10. Path length for gamma measurement - 3 in. pipe.....	26
11. Carrier for phototube and source on vertical pipe.....	27
12. Sketch of twin screw lift.....	28
13A. Recorder traces of gamma beam transmission through 3 in. pipe, 200A loop, during operation.....	29
13B. Transmission curves taken in 200A loop during operation.....	30
14. Calibration curve for Th density at 300°C taken on 200A loop 3 in. pipe.....	31
15. Density effect due to temperature change on gamma transmission.....	32
16. Traces taken during slurry withdrawals from 200A loop. Concentration reduction from 1200 g Th/kg H_2O to 820 g Th/kg H_2O	33
17. Settling rates.....	34
18. Sketch of 200A loop pressurizer.....	35
19. Longitudinal profile of 200A loop pressurizer showing geometry effect on gamma transmission.....	36
20. Plot of rise in slurry-water interface due to one addition 200A loop pressurizer.....	37

TABLE OF FIGURES (continued)

21. Traces of series of additions to 200A loop pressurizer.....	38
22. Single channel analyzer trace of 3 in. pipe and 1000 g Th/kg H ₂ O - Both Co ⁶⁰ peaks.....	39
23. Single channel analyzer trace of 3 in. pipe 1.173 MEV photopeak. Single peak sweep circuit.....	40
24. Single channel analyzer trace of 3-in. pipe 200A loop. 1.173 MEV photopeak without sweep circuit.....	41
25. Block diagram of Hewlett Packard scaler Model 522B.....	42
26. Block diagram of single channel analyzer.....	43
27. Special single peak sweep circuit.....	44

SUMMARY

The feasibility of using gamma-ray attenuation techniques was demonstrated in determining the density and homogeneity of slurries circulating at 300°C. The method appears to have general utility in the engineering studies of slurry because the equipment is mounted externally and does not interfere with the system under investigation.

Two types of detector systems were evaluated for routine service. The decimal counter-recorder system was found more adaptable for rugged operations, whereas the single channel analyzer was found to be more accurate and flexible.

INTRODUCTION

It is the purpose of this report to summarize the results of tests to determine the feasibility of measuring the density and homogeneity of circulating slurries by gamma-ray transmission. The data presented are preliminary and the equipment needs are to be checked further against slurries of known density and homogeneity. The data do indicate, however, the usefulness of the method in obtaining measurements not easily attainable by other methods.

Berman and Harris³ have evaluated the technique of using gamma-ray transmission to measure the uniformity of solid materials. They discuss, in detail, the various factors which influence the precision of the method and also describe the equipment used in their studies at Los Alamos. They found that the precision of the method was a function of such factors as: (a) composition of material, (b) the quality and strength of the radiation source, (c) the technique used to measure the radiation intensity, and (d) the constancy of alignment between the source and radiation detector.

Determination of Pulse-Amplitude Distribution

The successful application of gamma radiation to the problem of measuring small differences in density depends primarily on the reliability and sensitivity of the detection system.^{7,4,2} The need for these characteristics arises because the detection equipment must sort with unchanged sensitivity a pulse which arrives very shortly after the occurrence of a previous pulse. Both Geiger-Muller tubes and scintillation crystals are suitable detectors when coupled with a linear amplifier and a pulse height analyzer. In general, the G-M tubes have the advantage of simplicity, but the scintillation crystals have much higher gamma sensitivity. The equipment used in these studies to measure the attenuation of the gammas from a radioactive source in passing through a suspension of thorium oxide solids in water is shown in Figures 1 and 2. Both set-ups are essentially the same and consist of a scintillation detection head, a regulated high voltage supply, a collimated gamma source (not shown in Figures), a linear amplifier, pulse height discriminator, and instrumentation to determine and record the pulse-amplitude distributions.

Two general methods, one direct and the other indirect, were employed to determine the pulse-amplitude distributions. In both methods the detection of the gamma rays emitted by a radioactive source was accomplished by the use of a sodium iodide crystal containing a trace of thallium. In the crystal the gamma ray energy is transferred to recoil electrons which cause the emission of visible light as they dissipate their energy. This light is then collected by the photosensitive cathode of a photomultiplier tube which converts it to an electrical signal and amplifies the signal by a large factor. The magnitude of the voltage pulse produced by the anode of

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the photomultiplier tube is also proportional to the energy of the event occurring in the NaI crystal.¹ Cobalt-60 with two high energy components (1.173 MEV and 1.332 MEV) was used as the gamma source in a majority of the tests. In the equipment used for these experiments, the pulses (Co^{60}) appearing at the output of the photomultiplier tube are of the order of 250 millivolts.

Integral - Bias Curve by Indirect Measurement

The indirect method consisted of measuring with the equipment shown in Figure 1, during a given time interval, the number of pulses whose amplitudes were greater than a particular value. A plot of this number as a function of energy level is called the integral-bias curve. Examples are shown in Figure 3 for Cobalt-60 and Cesium-137. The pulse-amplitude distribution, if needed, can then be obtained by taking the derivative of the integral-bias curve. Since each photo-tube has its own characteristics, it was necessary to obtain an integral-bias curve for each tube to determine the optimum setting for the pulse height discriminator.

The optimum pulse height setting was obtained from the integral bias curve by noting the range in which plateaus or changes in the slope of the curve occurred. For sources with multiple components, distinct plateaus are evident if the energies of the gammas are widely different; however, if the energies are approximately the same as with Co-60, it is difficult to separate the contribution of each gamma so that a change in slope rather than a plateau is obtained on the integral bias curve. Referring to Figure 3, it can be seen that the plateau is more definitive for the Cesium source and only a change in slope is indicated for the Co-60 source. Cobalt-60 has two components whose energies are fairly close together, whereas, Cesium-137

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has only a monoenergetic gamma of 0.662 MEV.

Several other factors such as the applied voltage across the scintillation heat and the background radiation were found important in establishing the integral-bias curve. The plateaus will shift up or down in the discriminator depending on the particular gain setting and high voltage applied to the tube.

In practice, a faster method for determining the pulse height setting is used. This method entails the use of another source (other than Co^{60} which is used in the normal gamma scanning) of slightly lower energy than the lower Co^{60} peak (1.173 MEV). Ce^{137} is used for the lower energy (0.66 MEV) source. The oscilloscope is coupled to the linear amplifier in the following manner: (1) from the amplifier output the signal is transmitted to the oscilloscope vertical input, (2) from the pulse height selector output the signal is transmitted to the external horizontal trigger circuit. The oscilloscope will then trigger only on pulses which are actually being counted. By placing the Ce^{137} in front of the crystal, the 0.66 MEV pulses are readily seen on the oscilloscope (assuming the pulse height selector is set low enough). By raising the pulse height selector voltage this peak can be cut out so it will no longer trigger the oscilloscope or the counter. By this method one knows that the counter is blocked to any pulses below the 0.66 MEV level. Those pulses which are counted will then be at a minimum above 0.66 MEV. The difference in counts per minute between the 0.66 MEV level and 1.173 MEV (the lower Co^{60} peak) can be further increased by adjusting the pulse height selector and watching the signal on the oscilloscope until only the dominating Co^{60} pulses remain.

The gain setting on the amplifier must not be disturbed once this setting is established as the bias will shift with a change in voltage. Ideally,

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the second source would be of an energy level closer to Co^{60} than Ce^{137} , however, as Ce^{137} was readily available it was used in this case.

In Figure 4, the effect of the high voltage supply and the contributions of the thorium oxide gammas to the total count are shown for the Cobalt-60 source. The change in slope is more discernible and occurs over a wider range of pulse height selector settings with the high voltage set at 0.9 kv than at 0.92 kv. These data illustrate the importance of the stability of the high voltage supply because the resolution and response of the photomultiplier tube are both affected by the voltage across it. The data also indicate that the optimum voltage for each tube should be determined experimentally before the unit is put into service to minimize variations in the observed count rate.

In most cases, the background contribution to the total counting rate was found to be negligible, provided that the number and the energies of the background gamma-photons were not in the range of the source. Referring to Figure 4, it can be seen that satisfactory pulse-amplitude discrimination was obtained even though the radiation contributions from the thorium decay chain whose energies range from 0.04 to 2.615 MEV was 25% of the total counting rate. Thallium-208 and Bismuth-212 with energies above 1.0 MEV were the major contributors. In this test, a 181 millicurie Cobalt-60 source was located in the center of a 5-ft diameter steel sphere filled with a slurry containing 1500 lbs of thorium oxide suspended in water. The detector was located on the perimeter of the sphere. With more favorable geometries and less concentrated thorium oxide slurries, the contribution of the thorium gammas was found insignificant.

To monitor the counting rate, the scintillation head, counter and associated equipment was checked periodically against a standard cobalt source (approximately 10^8 disintegrations per minute) in a well-shielded pig, (shown in Figure 1). The scintillation head was placed on the source in the pig and minor shifts or drifts were corrected with the amplifier fine gain control.

As a precaution against overloading the amplifier, the oscilloscope was connected to the amplifier output. As the amplifier became overloaded, the signal on the oscilloscope became distorted with the high energy peaks being flattened out. Under this condition, the response of the amplifier was no longer linear.

Direct Measurement of the Pulse-Amplitude Distribution with a Single Channel Differential Analyzer

Pulse-amplitude distribution spectra were also obtained by direct measurement with a differential pulse-amplitude discriminator, or single channel analyzer, which counts, during a time interval, the number of pulses whose amplitudes lie between two energy levels separated by a "window" or channel width corresponding to the energy range of interest.

In essence, the analyzer contains two discriminators. The pulse is disregarded unless its height is larger than the setting of the lower level discriminator. The pulse is also rejected if the pulse height is large enough to exceed the settings of both the upper and lower level discriminators by an anti-coincidence circuit connecting the output of the two discriminators. An output pulse is therefore produced only if the input pulse is large enough to exceed the lower level but not so large as to reach the upper level. The output pulses are usually recorded by an appropriate scale or rate meter.

The instrument used in these studies was previously described⁷ and is

shown in Figure 2. The instrument is flexible; it can be operated either as a differential analyzer or as a count rate meter. Also incorporated in the design of the instrument is a circuit for scanning a particular energy peak. This is accomplished as shown in Figure 27, by a single peak sweep circuit which sweeps the "window" back and forth across any selected peak, both the amplitude and the frequency of the sweep can be controlled.

The analyzer was also calibrated against the same standard Cobalt-60 source used to check the decimal counter meter. The window width (ΔE) was found to be constant within 1% for all values of E between zero and 80 volts and any value of "window" width between zero and five volts could be obtained.

Figures 5 and 6 show the characteristic spectra¹ of Cobalt-60 and Cesium-137 obtained with the single channel analyzer using the differential pulse-amplitude discriminators.

Comparison of the Indirect and Direct Methods for Determining the Pulse-Amplitude Distributions

Both the integral-bias and differential measurement methods for determining pulse-amplitude distributions were time-consuming procedures. However, of the two methods, the differential method was simpler and the equipment more flexible in that a specific gamma of a particular source could be monitored to give the best results in a particular application. Because of the complexity of the instrument, maintenance on the analyzer was high and frequent calibration was required to insure reproducibility of the data.

The operation of the analyzer was found to be sensitive to: (a) the high voltage supply to the photomultiplier tube, (b) temperature changes which affected the scintillation head and the electronic gear in the instrument case, (c) gain setting in the linear amplifier circuit and (d) window width (ΔE

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settings).

The stability of the high voltage supply and the determination of the proper choice of voltage to the photomultiplier tube in the scintillation head was extremely important in the operation of the analyzer. A variation of $\pm 1\%$ in the voltage shifted or obscured the characteristic peak of the gamma source. Figure 7 shows the complete spectrum for Co-60 with the applied voltage set at 1150 v. The peaks, although well defined, are found in the low energy range; whereas, the characteristic well-defined peaks, reported in the literature for Co-60¹ and shown in Figure 5 were reproduced by lowering the applied voltage to 870 v.

The three other factors affecting the sensitivity of the instrument were investigated only qualitatively. Increased air circulation through the instrument case and forced air circulation around the scintillation head aided in the reproducibility of the data. The gain and "window" width settings were determined by monitoring the output from the A-1 amplifier for linearity with a cathode-ray oscilloscope. Once these settings were established, they were not changed during the course of the tests.

In contrast, the equipment employed to obtain the integral-bias curves was simple, rugged, extremely stable, easy to operate with minimum maintenance, but required a source which emitted gammas with energies higher than the radiation from the background material.

Gamma Ray Densitometer Measurements

A. Theory

A heterogeneous system may be treated as a series of homogeneous media if a highly collimated, essentially monoenergetic source is used and the mass absorption coefficient for any phase is assumed to be independent of its

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position in the system.² Thus the expression used to calculate the absorption of gamma radiation for several homogeneous media in series can be used for heterogeneous systems as follows:

$$I = I_0 e^{-\sum \mu_L L}$$

where $\mu_L = \rho (\mu_m f)_i$

and

I_0 = incident radiation intensity

L = absorber thickness

ρ = density of slurry, g/cc

μ_m = mass absorption coefficient, cm^2/gm

f = weight fraction of components in system

i = i th component

B. Procedure

Determination of the numerical value of I_0 was obtained by means of "zero" measurements using the equipment shown in Figures 8 and 9. A gamma beam (Co-60) was projected through air directly into the detection system at a fixed distance from the source, and this source-detector geometry was maintained when the gamma beam was projected through the piping. The gamma beam and scintillation head were both collimated; the beam was collimated to 1/8 in. in diameter and the head to approximately 3/8 in. in diameter.

Transmission scans were made using a constant speed power-driven table and adjusting the speed of the table to the source strength and instrument response. By calibrating the chart and table speeds against fixed

positions on the pipe, it was possible to compare data by simply imposing one trace on top of the other.

The detection system shown in Figure 1 was used to measure the attenuation of the gamma beam because of its ruggedness, fast response, and low maintenance.

C. Scanning Set-ups

1. Three inch pipe with wall thickness of 0.406 inches

In Figures 8 and 9, the arrangement for scanning the 3 inch pipe is shown. The table was moved vertically at a controlled speed by means of a 1/4 hp motor coupled to a vari-drive speed reducer. A reversing switch reversed the rotation of the motor thus changing the direction of table movement.

In scanning the 3 inch pipe, it was found that reproducible scans were obtained with a 63 millicurie, cobalt-60 source using a table speed of 2 inch/min. Figure 10 shows the path layout for traversing the pipe, the distance between the source and head being fixed at 14 inches.

2. Six inch pipe with wall thickness of 0.718 inches

Figures 11 and 12 show the set-up for scanning the 6 inch schedule 160 pressurizer. A twin screw powered by an air-driven motor was used to raise and lower the two tables. The distance between the source and head was 18 inches and the table speed was 6 inches per minute using a 181 millicurie cobalt-60 source. Because of the thickness of the pipe walls, the collimating hole was conically-shaped to increase the intensity of the radiation without increasing the source strength.

D. Discussion of Experimental Results Using Decimal Scaler

The feasibility of using gamma absorption techniques for determining

the density gradients and homogeneity of slurries in thick-walled containers was demonstrated by scanning horizontal lengths of pipe in which thorium oxide slurries of various concentrations were circulated at temperatures from ambient to 300°C. Typical profiles are shown in Figures 13A and 13B. Using these data and including data from other scans, a calibration curve relating the transmission ratios to slurry density at 300°C, is shown in Figure 14 for the equipment used as a densitometer.

The asymmetry of the profiles of air and water shown in Figures 13A and 13B are probably due to the pipe geometry through which the gamma beam was projected, the time lag due to instrument response, possible misalignment of source and tube.⁸ At a slurry concentration of 500 g Th/kg H₂O and higher, the attenuation of the gammas through the slurry over-shadowed the geometry effect, resulting in more symmetrical profiles.

Figure 15 shows the profiles for 750 g Th/kg H₂O slurry as it was cooled from 290°C to 100°C. Qualitatively, no gross change in the handling characteristics of the slurry can be seen as a result of the temperature change.

In Figure 16, data are presented to show that gamma absorption techniques can be used to detect concentration gradients in circulating slurries. The data were obtained as the circulating slurry concentration was reduced from 1200 g Th/kg H₂O to 500 g Th/kg H₂O by withdrawing slurry from two different sampling stations. Both sampling stations, 180° apart, were located approximately 10 feet upstream from the position where the scans were made. One sample point was flush-mounted to the top of the pipe, and the other one was flush-mounted to the bottom of the pipe. Referring to

Figure 16, shifts in the profile of the upper half of the pipe are apparent, indicating a decrease in concentration, with each slurry withdrawal from the top sampling station and essentially no change in concentration in the bottom 3/8-in. to 1/2-in. of pipe. However, a gross change in concentration was observed in this bottom section of pipe when slurry was withdrawn from the bottom sampling station, and the skewness in the transmission profile disappeared, indicating that homogeneous flow was re-established.

E. Other Applications for Gamma-ray Attenuation

The gamma-ray attenuation technique was also used to detect the slurry-water interface in the pressurizer of the 200A-loop and to indicate, in situ, the behavior of slurries when circulation in horizontal pipes was stopped.

1. Monitoring Slurry Behavior

In Figure 17, data are presented to illustrate the potentialities of the method in estimating settling rates and settled bed densities under actual operating conditions. Based on these data, shifts of approximately 5% in the transmission profiles are clearly discernible between flow and non-flow conditions, especially in the central scanning region where the influence of the metal is minimal. It should therefore be possible to establish the settling behavior of the particular slurry under investigation and to estimate the settling rate by choosing an appropriate position on the pipe, such as the midpoint, to observe the change in the transmission ratio as a function of time, provided the test conditions can be maintained constant.

These data also indicate that settled bed densities can be estimated by comparing the "steady-state" profile with ones obtained by direct calibration in an identical geometry for similar slurries of varying concentrations. For example, a settled bed density of 1400 g Th/kg H₂O is indicated by com-

paring the 100-minute profile shown in Figure 17 with the profiles presented in Figure 13B.

It was also possible to follow the resuspension of settled solids by monitoring the center-line transmission ratio to reach a constant value.

2. Monitoring Slurry-water Interface

Gamma ray absorption has been used successfully to measure levels in storage and surge tanks.^{4,5} Essentially the same techniques were demonstrated in monitoring the slurry-water interface in the pressurizer of the 200A loop. The geometry through which the gamma rays were projected is shown in Figure 18 and the effect of this complex geometry on the gamma ray transmission is shown in Figure 19. The profile in Figure 19 was obtained by projecting the radiation from a 181-millicurie-Cobalt-60 source through the empty 6 inch diameter pipe with a wall thickness of 0.78 inches. Even with this complex geometry changes in the slurry-water interface were detected as seen in Figure 21. The sudden breaks in the curves of Figure 21 indicate increased transmission.

Results Using Single Channel Analyzer

As pointed out previously, the single channel analyzer was not used in routine service because of its extreme sensitivity and high maintenance requirements. Data were obtained, nevertheless, to show that the instrument can be used as a densitometer. Figure 22 shows the attenuation of the two major peaks of Cobalt-60 due to various thickness of steel and slurry containing 1000 g Th/kg H₂O.

Figure 23 illustrates the profile obtained by monitoring the 1.173 MEV peak of Cobalt-60 with the single peak sweep circuit. A typical pro-

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file shown in Figure 24 was obtained by manually changing the position of the table every few seconds. The response of the single channel analyzer was too slow to permit continuous scanning of the pipe, as was accomplished with the decimal-counter system.

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Appendix A. Description of Instruments Used in These Investigations

I. Decimal scaler and recorder.

- A. A collimated, shielded, scintillation head, which includes a sodium iodide (The) crystal (1" x 1-1/2"), a phototube, and a preamplifier.
- B. A regulated high voltage supply (Victoreen Model 653).
- C. An A-1-D linear amplifier with band width, gain, and pulse-height selector (ORNL Model Q-1326).
- D. A Hewlett-Packard electronic counter (Model 522-B). (Figure 25).
- E. Bristol recorder (Model 1Phlx570-51-E-57x-N6-G27X).

II. Single Channel Analyzer

- A. A collimated scintillation head which includes NaI crystal (1"x1-1/2"), a multiplier tube, and preamplifier.
- B. High voltage supply (Victoreen Model 653).
- C. Linear Amplifier (Atomic Instrument Co., Model 218).
- D. Single Channel Analyzer (ORNL Model Q 1473). (Figure 26)
- E. Single peak sweep circuit. (Figure 27).
- F. Brown recorder Model 753X-12V-Y-6A1.

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Fig. 1 Decimal Counter With Phototube and Recorder Attached

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Fig. 2 Single Channel Analyzer

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Effect of Excess High Voltage on
Crystal Discrimination of Co^{60} Spectrum

Fig. 7

KEE 20 X 20 TO THE INCH 359-10 1/2 G
KEUFFEL & ESSER CO. MADE IN U.S.A.

Fig. 8 Gamma Scanning Equipment For Horizontal Pipe

ORNL-LR-DWG.-24994
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Sketch of Single Screw Lift

Position	2T	2T + L
0	1.8	0
1	1.2	1.4
2	1.0	2.25
3	0.90	2.6
4	0.812	2.7
5	0.9	2.6
6	1.0	2.25
7	1.2	1.4
8	1.8	0

3" Sch 80 S.S. Pipe

Path Length for Gamma Measurement - 3" Pipe

Fig. 10

981-030

Fig. 11 Carrier For Phototube And Source On Vertical Pipe

ORNL-LR-Dwg.-24996
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Sketch of Twin Screw Lift

Fig. 13A Recorder Traces of Gamma Beam Transmission Through
3 in. Pipe, 200A Loop, During Operation

Fig. 13B

Fig. 13B

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Th Slurry Density at 300°C

Calibration Curve for Th Density at 300°C

Taken on 200 A Loop - 3" Pipe

Fig. 14

ORNL-LE-DMG.-25000
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Fig. 16

Sketch of 200A Loop Pressurizer

Longitudinal Profile of Pressurizer showing
Geometry Effect on Gamma Ray Transmission

Fig. 20

Fig. 21 Traces of Series of Additions to 200A Loop Pressurizer

Fig. 22 Single Channel Analyzer Trace of 3 in. Pipe and 1000 g Th/kg H₂O
Both Co⁶⁰ Peaks

Fig. 23 Single Channel Analyzer Trace of 3 in. Pipe. 1.173 MEV Photopeak.
Single Peak Sweep Circuit

Fig. 24 Single Channel Analyzer Trace of 3 in. Pipe 200A Loop.
1.173 MEV Photopeak Without Sweep Circuit

BLOCK DIAGRAM OF MODEL 522B HEWLETT-PACKARD COUNTER

Fig. 25

BLOCK DIAGRAM OF THE SINGLE CHANNEL ANALYZER

Fig. 26

- S_1 F S_2 SVC
 IV 5.8Y

B. CONTROL PANEL

SVC SCANNING
VOLTAGE
CONTROL

Fig. 27