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Hydrodynamics of Three-Phase Slurry
Fischer-Tropsch Bubble Column Reactors

Quarterly Technical Progress Report
for the Period 1 October 1988 - 31 December 1988

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January 27, 1989

Prepared for the Pittsburgh Energy Technology Center,
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TABLE OF CONTENTS

I.	Abstract	1
II.	Objective and Scope of Work	2
III.	Summary of Progress	4
IV.	Detailed Description of Technical Progress	6
	A. Task 3 - Measurement of Hydrodynamic Parameters by Conventional Techniques	6
	A.1. Overview of Bubble Column Operations	6
	B. Task 4 - Application of a Gamma Radiation Density Gauge for Determining Hydrodynamic Parameters	8
	B.1. Measurement of Absorption Coefficients	9
	B.2. Two-Phase Experiments	10
	B.3. Three-Phase Experiments	12
	B.4. System Modifications and Improvements	15
	B.5. Evaluations of the Americium-241 and Cesium-137 Sources	16
V.	Nomenclature	21
	Tables and Figures	22

I. Abstract

During the past quarter, we have made an effort to resolve problems we have been having with our slurry pump. We have not been able to get a commitment from Gelber Pumps that replacement parts for the pump will be provided free of charge. We located an alternative pump and are in the process of ordering it. The modifications of the expansion and calibration units for both, the small and large stainless steel columns, were completed during this quarter. Some tests were conducted with a slurry containing 20 wt.% iron oxide in water, to determine the amount of solids settling in the small column circuit following the modifications. Operational problems prevented us from obtaining any conclusive results from these tests.

We devoted considerable effort during this quarter on testing and calibrating the nuclear density gauge apparatus. Absorption coefficients for various media were measured. Trial runs with two-phase and three-phase cold systems (air-water and air-water-sand systems) were used to identify potential problems with the operation of the nuclear density gauge apparatus. Some theory associated with the determination of phase hold-ups for the two- and three-phase systems is presented. Based on the trial runs, several improvements in the calibration procedure and in the instrumentation were made. We ordered and received two single channel analyzers during this period. These will enable us to count gamma rays of the desired energy level with minimal interference from stray radiation. We established criteria for evaluating radioactive sources and found that the low energy Americium-241 source was unsuitable for our application. We have decided to replace it with a new Cobalt-60 source. Our license permitting the use of radioactive materials has been amended to include the use of the Cobalt-60 source.

II. Objective and Scope of Work

The overall objective of this contract is to determine the effects of bubble column diameter, solids loading and particle size, and operating conditions (temperature, gas and liquid flow rates) on hydrodynamics of slurry bubble columns for Fischer-Tropsch synthesis, using a molten wax as the liquid medium. To accomplish these objectives, the following specific tasks will be undertaken.

Task 1 – Project Work Plan

The objective of this task is to establish a detailed project work plan covering the entire period of performance of the contract, including a detailed program schedule, analytical procedures, and estimated costs and manhours expended by month for each task.

Task 2 – Design and Construction of the Experimental Apparatus

The existing glass and stainless steel columns (0.051 m and 0.229 m in diameter, 3 m tall) that were constructed under our previous DOE contract (DE-AC22-84PC70027), will be modified and additions made in order to study the effect of continuous upward liquid flow. After the procurement of equipment and instrumentation, and construction of the unit is completed, a shakedown of test facilities will be made to verify achievement of planned operating conditions.

Task 3 – Measurement of Hydrodynamic Parameters by Conventional Techniques

In this task, the effects of operating conditions (liquid and gas superficial velocities, temperature), gas distributor, column diameter, and solids concentrations and particle size on hydrodynamic parameters in the glass and stainless steel columns will be determined. All experiments will be conducted using nitrogen at atmospheric pressure.

The hydrodynamic parameters that will be determined as a function of the independent variables mentioned above are: average gas hold-up, axial solids distribution, axial gas hold-up, flow regime characterization, and qualitative information on bubble size distribution.

Task 4 – Application of a Gamma Radiation Density Gauge for Determining Hydrodynamic Parameters

The objective of this task is to determine hydrodynamic parameters for the three-phase system using a nuclear density gauge apparatus. A movable assembly mechanism and positioning racks for the two nuclear density gauges and detectors will be designed and constructed. Following the interfacing of the apparatus with an on-line micro-processor, the gauges will be calibrated using pure components (liquid wax and solid particles), and with known proportions of liquid and solid. After calibration, the following parameters will be obtained from experiments in the large stainless steel column: axial gas hold-up, axial concentration of solids, and qualitative information on flow regimes and bubble size distributions.

III. Summary of Progress

During the past quarter, we have made a continuous effort to resolve problems that we have been having with our slurry pump. We received replacement parts from Gelber Pumps for the existing slurry pump, and were invoiced for them even though our understanding was that the parts would be provided free of charge. We have not yet resolved this issue with Gelber Pumps. In the meantime we have located a new pump from Tuthill Corporation, which appears to be more suitable for our application.

We completed modifications of the expansion and calibration units for both, the small and large stainless steel columns during this quarter. These modifications were made to minimize problems with solids settling that we had encountered in our earlier runs. We attempted to test the modified small column circuit using a slurry containing 20 wt.% of 20-44 μm iron oxide in water. A Sandpiper pump with a surge suppressor was used to conduct the test in the continuous mode of operation. Results from this test were inconclusive due to the large amount of iron oxide that settled in the surge suppressor. Some effort was also devoted to the replacement of worn out valves and fittings in the small column circuit.

We initiated the testing and calibration of the nuclear density gauge apparatus during this quarter. The procedure for measuring absorption coefficients for different media was streamlined. We then measured absorption coefficients for water, sand, silica and iron oxide. Problems with reproducibility were encountered during the initial measurements. It was discovered that stray radiation from the Americium-241 source was interfering with the measurements made with the Cesium-241 source. This problem was rectified by adding a collimator to the Cesium detector. This modification greatly improved the quality of signals obtained from the Cesium detector.

Preliminary tests were made with two-phase (air-water) and three-phase (air-water-sand) systems to check the ability of the nuclear density gauges to predict phase hold-up

values. Tests with the two-phase system were successful and we were able to obtain gas hold-ups that compared favorably with those obtained using DP cells. A theory associated with the determination of hold-ups from experimental data obtained with two- and three-phase systems has been developed. Results from the three-phase tests were not satisfactory. This was probably due to poor absorption coefficients for the solid phase (sand), and partly due to problems with the positioning of the nuclear density gauge apparatus. We improved the procedure used to obtain absorption coefficients for the solid phase by fabricating a stirred tank where the solids could be mixed with a liquid and kept suspended while measurements are made. We also designed and installed a system that accurately positioned the nuclear density gauges at the exact same axial and radial positions. Two single channel analyzers were ordered and received during this quarter. These instruments will enable us to count gamma rays of the desired energy level without interference from stray radiations. By using this setup, we expect to achieve improvements in the quality of signals from the two detectors.

We established criteria for evaluating the existing radioactive sources. This was done to check the suitability of these sources to our application. The sources were checked for source strength, sensitivity to differences in hold-up structures, and response time to changes in the flow field. Beam widths of the sources were also measured. Our tests showed that the high energy Cesium-137 source is suitable for our application. However, the low energy Americium-241 source had several problems. It is too weak for our application, its response time is too high, and the beam from this source was significantly wider than that from the high energy source. We then proceeded to evaluate other sources and subsequently decided to replace the Americium-241 source with a 35 mCi Cobalt-60 source. We have amended our license permitting the use of radioactive materials to include the use of the Cobalt-60 source.

IV. Detailed Description of Technical Progress

A. Task 3 – Measurement of Hydrodynamic Parameters by Conventional Techniques

A.1. Overview of Bubble Column Operations

During the past quarter, we have made a continuous effort to resolve problems that we have been having with our slurry pump. At the beginning of the quarter, we were informed by Gelber Pumps that following their own tests, they found an alternative material for construction of the pump's idle gear. This new material was expected to reduce wear and tear of the pump internals when used with our system. It was our understanding that the idle gear and other replacement parts would be provided free of charge. However, when the parts were finally received in early December, we were invoiced for all parts shipped. We have not yet resolved this issue with Gelber Pumps. In the meantime, we initiated a search for an alternative pump that would be suitable for our application. A suitable candidate for our application is a pump manufactured by The Pump Division of Tuthill Corporation (Chicago). It is a lobe type positive displacement pump that can be operated at variable speeds. We are in the process of acquiring bids from other pump manufacturers as well, and plan to place an order for the new pump as soon as we receive the complete technical information and bids from three vendors.

We completed modifications of the expansion and calibration units for both, the small and large stainless steel columns. These modifications (details were given in the Technical Progress Report for the period 1 July-30 September, 1988) are expected to minimize problems with solids settling that were encountered in our earlier runs. The modified units were reinstalled in the respective column circuits. A low temperature Sandpiper pneumatic diaphragm pump was placed in the feed line to the small column. A Warren-Rupp surge suppressor, located downstream to the pump, was used to dampen the pulses in the fluid discharged from the pump. The DP cells were serviced and

reinstalled on the small stainless steel column. Following the completion of the various modifications, experiments were performed to determine the amount of solids settling in the small column circuit. The tests were made in the continuous mode of operation using a slurry containing 20 wt.% of 20-44 μm iron oxide particles in water. Results from the test, which was conducted over a two day period, were inconclusive due to the large amount of iron oxide that settled in the surge suppressor. The solids concentration measured in the column was between 5 and 7 wt.% over the two day period. After the test, the system was disassembled for inspection and only 6% of the solids charged were found in the various lines and units. The majority of these were located in the expansion unit and the drain line directly below the storage tank. Negligible amount of solids were found on the distributor (less than 1% of the amount recovered) and none were found in the storage tank or the calibration unit.

During the past quarter, some effort was also devoted to the replacement of worn out valves and fittings in the small column circuit. We initiated work on fabrication of an oxygen trap that will be installed in the gas inlet line. This will increase the purity of the nitrogen gas by removing any oxygen and thus eliminate the possibility of the wax oxidizing during experiments.

B. Task 4 – Application of a Gamma Radiation Density Gauge for Determining Hydrodynamic Parameters

During the past quarter, we initiated the testing and calibration of the nuclear density gauge apparatus. This process included the measurement of absorption coefficients for different media, reproducibility checks, trial runs with two-phase and three-phase systems, selection of appropriate settings for the nuclear density gauge electronics, and evaluation of the suitability of the existing two sources for our application. Some of these steps involved trial and error procedures. The various tests and resulting improvements are presented here in chronological order. Some theory associated with the estimation of phase hold-ups is also presented.

We made several preliminary tests with the nuclear density gauge system to assess its performance. These initial tests were conducted using the two-phase air-water system. The value of testing with a two-phase system as opposed to a three-phase system, is that the volume fractions of the different phases can be determined using just one detector. Thus, measurements can be made independently with each of the detectors and results compared. During these tests we discovered that the detector for the low energy Americium-241 source was malfunctioning and had to be replaced. Following this, we were still unable to obtain comparable results from the two detectors for the air and water volume fractions. This is because stray radiation from the low energy Americium-241 source reached the Cesium detector, distorting the signal from the detector. Table 1 shows gas hold-ups obtained using the Cesium-137 source with and without the Americium-241 source present. The influence of stray radiation on the results can clearly be seen. The % relative deviations between gas hold-ups obtained with and without the Americium source are between 14-23%. To alleviate this problem, a two inch diameter by two inch long steel cylinder, with a 1/4 inch diameter hole drilled through it was added to the Cesium detector. This served as a collimator and prevented

stray radiation, emitted from the Americium source, from reaching the Cesium detector. This modification greatly improved the quality of the signal obtained from the Cesium detector, and this was reflected in the results obtained from subsequent tests with the two-phase system.

B.1 Measurement of Absorption Coefficients

Once the system was made functional, absorption coefficients of water were obtained for the Americium and Cesium sources. The procedure used for measuring the absorption coefficient of water or of any other material is briefly described here. A plexiglass calibration chamber with four compartments, each one inch thick, is used to hold the material for which absorption coefficient is to be measured. The chamber is positioned between the source and the detector in an orientation which allows the transmission of the radiation through all four compartments. Measurements were first made with the chamber empty, and the amount of radiation transmitted through the chamber is measured for a given source (I_0). A selected number of compartments are then filled with the medium under consideration and the radiation transmitted is measured (I). The absorption coefficient (μ) is then estimated by

$$\mu = -\frac{1}{\rho d} \ln \left(\frac{I}{I_0} \right) \quad (1)$$

where d is the thickness of medium (1 to 4 inches depending upon the number of compartments that are filled). Absorption coefficients for water with the two sources were estimated using 1, 2 and 4 inch layers of water. Each radiation measurement is an average of at least 3000 point measurements of the count rate. The Metrabyte data acquisition system is used to take these measurements. The absorption coefficient for water with the Cesium source was $0.0817 \text{ cm}^2/\text{g}$, and with the Americium source was $0.1447 \text{ cm}^2/\text{g}$, with relative deviations from three measurements being less than 1% for each case. The relative deviation from three measurements for absorption coefficient

for water with the Cesium source was around 3.5% before the collimator was installed. The absorption coefficient is influenced somewhat by the settings on the density gauge apparatus (electronic settings), positioning of the detector with respect to the source, etc. Even though this influence is small, we plan to estimate the absorption coefficients for each phase (solid or liquid) before each series of experiments so as to minimize the overall error.

B.2. Two-Phase Experiments

Several tests were performed in the large stainless steel column with the air-water system. The volume fractions of the gas and liquid phases were estimated for various gas flow rates. The measurements were made at a height of approximately 18 inches above the distributor at three radial positions (center, 2" left of the center, and 2" right of the center). Data were obtained at these locations with each of the two sources. The expression for the radiation transmitted through the column in the presence of the two phases is

$$I = I_0 \exp[-d(\mu_l \alpha_l + \mu_g \alpha_g) \rho_{sl}] \quad (2)$$

where the weight fractions of the two phases are related by

$$1 = \alpha_l + \alpha_g \quad (3)$$

and the slurry density is given by

$$\rho_{sl} = \frac{1}{\frac{\alpha_l}{\rho_l} + \frac{\alpha_g}{\rho_g}} \quad (4)$$

Recognizing that the weight fraction of the gas phase is negligible when compared to the weight fraction of the liquid phase, and that the absorption coefficient for the gas phase is significantly smaller than that for the liquid phase, the term $\mu_g \alpha_g$ can be eliminated from eqn. 2. Substitution of the expression for the weight fraction of gas phase from

eqn. 3 into eqn. 4 yields

$$\rho_{sl} = \frac{1}{\frac{\alpha_l}{\rho_l} + \frac{(1-\alpha_l)}{\rho_g}} \quad (5)$$

Substitution of eqn. 5 into eqn. 2 and elimination of the term $\mu_g \alpha_g$ gives

$$l = l_o \exp \left[\frac{-d\mu_l \alpha_l}{\frac{\alpha_l}{\rho_l} + \frac{(1-\alpha_l)}{\rho_g}} \right] \quad (6)$$

Eqn. 6 can now be solved for the liquid weight fraction to obtain

$$\alpha_l = \frac{1}{1 + \frac{d\rho_g \mu_l}{\ln(l/l_o)} + \frac{\rho_g}{\rho_l}} \quad (7)$$

The gas phase weight fraction can now be estimated using eqn. 3. The density of the gas phase can be obtained using the ideal gas law

$$\rho_g = \frac{(P)(MW)}{(R)(T)} \quad (8)$$

Hold-up of the liquid phase is then obtained by

$$\epsilon_l = \frac{\alpha_l \rho_{sl}}{\rho_l} \quad (9)$$

and for the gas phase by

$$\epsilon_g = 1 - \epsilon_l \quad (10)$$

Hold-ups were also estimated by differential pressure measurements made using DP cells. Table 2 shows gas hold-up values obtained with the two sources. At the center location, hold-up values obtained using the Americium-241 source are very similar to those obtained using the Cesium-137 source, with relative deviations being less than 2%. However, the agreement is relatively poor at the two off-center locations, with relative deviations as high as 14.3%. This is probably due to the poor performance of the Americium-241 source (see Section B-5). Hold-up values obtained using the two gauges independently (i.e. radial averages for the three values for each gauge)

compare favorably with those obtained using the DP cells. These results are shown in Table 3. These results also show that despite poor agreement between hold-up values obtained with the two sources at the two off-center locations, the average of the three measurements for the individual sources are very similar.

B.3 Three-Phase Experiments

Following the successful tests with the two-phase system, we initiated tests with a three-phase system. Air and water were used as the gas and liquid phases, respectively, and sand (< 30 mesh) was used as the solid phase. Before studying the three-phase system, we attempted to measure absorption coefficients for the solid phase (sand) using the calibration chamber (described earlier). During these measurements we encountered problems with reproducibility of the absorption values. The absorption coefficient for sand varied from test to test. We believe this was caused by the differences in the manner in which the sand was packed in the calibration chamber for the different tests. Since there were air gaps between the individual sand particles we were not measuring the true absorption coefficient for sand. In order to determine the reliability of the absorption coefficient value for sand (an average of several values was used), we filled one section of the calibration chamber with sand (1" thick layer) and two sections with water (2" thick layer). For this configuration, where the phases were not in direct contact with one another, we were able to determine volume fractions of sand and water within 2 percent (relative) of the actual values, which is reasonably good.

We then conducted tests using a three-phase system (air, water and sand) in the large stainless steel column. Measurements were made at a height of 18 inches above the distributor at three radial positions (center, 2" left of the center, and 2" right of the center) using a slurry that had approximately 10 wt.% solids (sand) by weight. Data were obtained with each of the two sources at each radial position. A parallel set of measurements of the gas hold-up and solids concentration, made using DP cells and

slurry withdrawal, served as the base case. The equations used to estimate the hold-ups for the various phases are developed here. The expressions for the radiation transmitted through the column in the presence of the three phases are

$$I_H = I_{oH} \exp[-d(\mu_{H\ell}\alpha_\ell + \mu_{Hs}\alpha_s + \mu_{Hg}\alpha_g)\rho_{sl}] \quad (11)$$

for the high energy source and

$$I_L = I_{oL} \exp[-d(\mu_{L\ell}\alpha_\ell + \mu_{Ls}\alpha_s + \mu_{Lg}\alpha_g)\rho_{sl}] \quad (12)$$

for the low energy source, where the weight fractions of the two phases are related by

$$1 = \alpha_s + \alpha_\ell + \alpha_g \quad (13)$$

and the slurry density is given by

$$\rho_{sl} = \frac{1}{\frac{\alpha_s}{\rho_s} + \frac{\alpha_\ell}{\rho_\ell} + \frac{\alpha_g}{\rho_g}} \quad (14)$$

In formulating the expressions for intensities (eqns. 11 and 12) we have assumed that the three phases are in effect arranged as three layers with their surfaces normal to the path of the beam (i.e. similar to resistances in series). However, the actual arrangement of the three phases may not conform to this assumption (e.g. similar to resistances in parallel, or a series-parallel combination). The effect that the different assumptions have on results will be discussed in a later report. Based on the arguments presented earlier, we can once again eliminate the terms $\mu_{Hg}\alpha_g$ and $\mu_{Lg}\alpha_g$ from eqns. 11 and 12, respectively. Solving eqn. 13 for the gas phase weight fraction and substituting this into eqn. 14, gives upon rearrangement

$$\rho_{sl} = \frac{1}{\frac{\alpha_s(\rho_g - \rho_s)}{\rho_g \rho_s} + \frac{\alpha_\ell(\rho_g - \rho_\ell)}{\rho_g \rho_\ell} + \frac{1}{\rho_g}} \quad (15)$$

Substitution of eqn. 15 into eqns. 11 and 12 and elimination of the terms $\mu_{Hg}\alpha_g$ and $\mu_{Lg}\alpha_g$ gives

$$I_H = I_{oH} \exp \left[\frac{-d(\mu_{H\ell}\alpha_\ell + \mu_{Hs}\alpha_s)}{\frac{\alpha_s(\rho_g - \rho_s)}{\rho_g \rho_s} + \frac{\alpha_\ell(\rho_g - \rho_\ell)}{\rho_g \rho_\ell} + \frac{1}{\rho_g}} \right] \quad (16)$$

for the high energy source and

$$I_L = I_{oL} \exp \left[\frac{-d(\mu_{L\ell}\alpha_\ell + \mu_{Ls}\alpha_s)}{\frac{\alpha_s(\rho_g - \rho_s)}{\rho_g\rho_s} + \frac{\alpha_\ell(\rho_g - \rho_\ell)}{\rho_g\rho_\ell} + \frac{1}{\rho_g}} \right] \quad (17)$$

for the low energy source. Eqns. 16 and 17 can now be solved simultaneously to obtain an expression for the liquid weight fraction

$$\alpha_\ell = \frac{\rho_\ell [a \ln(I_L / I_{oL}) - b \ln(I_H / I_{oH})]}{(\rho_g - \rho_\ell) [a [-d\mu_{L\ell} - \ln(I_L / I_{oL})] - b [-d\mu_{H\ell} - \ln(I_H / I_{oH})]]} \quad (18)$$

where

$$a = \left[-d\mu_{Hs} - \ln(I_H / I_{oH}) \frac{\rho_g - \rho_s}{\rho_g\rho_s} \right] \quad (19)$$

$$b = \left[-d\mu_{Ls} - \ln(I_L / I_{oL}) \frac{\rho_g - \rho_s}{\rho_g\rho_s} \right] \quad (20)$$

Similarly, the expression for the solid weight fraction is given by

$$\alpha_s = \frac{\rho_s [a' \ln(I_L / I_{oL}) - b' \ln(I_H / I_{oH})]}{(\rho_g - \rho_s) [a' [-d\mu_{Ls} - \ln(I_L / I_{oL})] - b' [-d\mu_{Hs} - \ln(I_H / I_{oH})]]} \quad (21)$$

where

$$a' = \left[-d\mu_{H\ell} - \ln(I_H / I_{oH}) \frac{\rho_g - \rho_\ell}{\rho_g\rho_\ell} \right] \quad (22)$$

$$b' = \left[-d\mu_{L\ell} - \ln(I_L / I_{oL}) \frac{\rho_g - \rho_\ell}{\rho_g\rho_\ell} \right] \quad (23)$$

The gas phase weight fraction can now be estimated using eqn. 13. Hold-ups for the three phases can be calculated using equations similar to eqn. 9.

Results from the three-phase tests were not satisfactory. Results obtained at the center of the column using the nuclear density gauge were in good agreement with the base case (9.7 wt.% solids, and 7.5% gas hold-up), however, results from the two off-center radial locations were not good. To the left of the center, 7.4 wt.% solids and a gas hold-up of 2% was determined using the density gauge, whereas to the right, solids concentration was determined to be 1 wt.% and the gas hold-up was 8%.

B.4 System Modifications and Improvements

The above tests with a three-phase system indicated several problems with our calibration and experimental procedures. An improvement in the procedure used to measure absorption coefficients for the solid phase was necessary. We believe that the poor results obtained when the column was scanned along the radial direction, were partly due to problems with the positioning of the nuclear density gauge apparatus. Since the column is curved, the amount of radiation transmitted through the empty column would be a function of radial position. This is because the distance traveled by the beam through the column wall varies with radial position (curvature effect). Thus, if measurements are not made at the exact same location where the empty column readings were taken, errors will be introduced into the analysis. A description of the new procedure used to estimate absorption coefficients for the solid phase, and details of the density gauge positioning system are given next.

We built a small stirred tank for estimating absorption coefficients of solids. The tank has a square cross-section (6" x 6") and is 18" tall, and is made of stainless steel. A stirrer is used for mixing the contents of the tank. A slurry withdrawal port located on the side of the tank is used to withdraw slurry samples for determining the weight percent of solids. We also acquired two single channel analyzers (SCA) for the nuclear density gauge system. The SCA will allow us to count gamma rays of the desired energy level. We can thus configure the SCA for a given source-detector unit so that gamma rays from only that source will be validated by the SCA, stray rays from any other source will be rejected. By using this set-up, we expected to achieve improvement in the quality of signal from each of the detectors. The stirred tank was positioned between the Cesium-137 source and its detector, such that the beam from the source passed through the tank at approximately the same height as that of the slurry withdrawal port. We determined absorption coefficients for sand (< 30 mesh), silica (0-5 μm), and

iron oxide (0-5 μm) using this set-up. Water was used as the liquid medium in these tests. Solids loadings of 10, 20 and 30 wt.% were used. Solids suspension with the large sand particles was poor with slurry samples showing a solids concentration that was only half of that expected. There was no problem in suspending the smaller silica and iron oxide particles. Results from three measurements with each solids type using the Cesium-137 source are shown in Table 4. The absorption coefficients obtained were 0.2072, 0.1722 and 0.3277 for sand, silica and iron oxide, respectively. The relative deviations in absorption coefficients for each solids type were less than 4%.

We designed and installed a system that accurately positioned the nuclear density gauges at the exact same axial and radial positions. The system consisted of magnetic switches that triggered electronic relays which cut power to the axial and radial drive motors once the gauges were in position. The magnetic switches were placed at predetermined axial and radial locations, corresponding to the points where measurements were to be made. Additional switches were also placed at axial and radial extremities to prevent the gauges from slipping off the tracks. One relay was installed in the axial drive motor power circuit and one in the radial drive motor power circuit. Bypass switches, one in each circuit, were installed to override the magnetic switches so that once measurements at a given location were completed the gauges could be moved to the next location. This system was tested successfully and it greatly improved the accuracy of our measurements.

B.5 Evaluations of the Americium-241 and Cesium-137 Sources

Following the completion of the necessary modifications and streamlining of the calibration procedures, we proceeded with the evaluations of the two sources that we currently have. There are several factors that need to be considered in the evaluation process. These include the source strength, sensitivity to differences in hold-up structures, response time to changes in the flow field, and beam width measurements. The

source strength must be sufficiently high so that the beam can penetrate the three-phase dispersion even at high solid hold-ups and still give a high enough count rate so that the noise to signal ratio is small. The sensitivity of the source is determined by the percent change in the count rate between transmission through a dispersion having high gas hold-up and low solids hold-up, and transmission through a dispersion having low gas hold-up and high solids hold-up. It follows from this that sensitivity would be influenced greatly by the absorption coefficients for the three phases with a given radioactive source. The count rate at a given location is determined using an average of at least 3000 point measurements of the count rate. It would be desirable that each of these measurements reflect the condition of the dispersion in the column. For this to happen the count rate must change as rapidly as the hold-up structure changes. This is particularly important when fluctuations in the hold-up are to be measured (e.g. for bubble size or frequency measurements). The response time or time constant for the source should therefore be as low as possible. The beam width is an important parameter when point measurements are being made. A wide beam would not be sensitive to changes taking place on a micro scale, e.g. passage of small bubbles, whereas a narrow beam would be better suited. It would be difficult to obtain reliable measurements at different radial locations using a source with a wide beam because part of the beam would be lost when the location is furthest from the center of the column. Also, with a wider beam, the probability of stray radiations from such a source interfering with measurements from the second source would increase.

We conducted several experiments that provided information on the suitability of the two sources to our application. In the first set of experiments, count rate was measured with the source at different radial locations and the column empty. This was done with both sources. The second set of experiments involved the measurement of the radial count rate profiles for both sources with the column filled with water. Figures

1 and 2 show count rate profiles for the low energy Americium-241 source and the high energy Cesium-137 sources, respectively. For each location the count rate with the column empty (I_0) and that with the column full (I) were used to determine the thickness of the layer of water through which the beam traverses. A modified form of eqn. 1 was used to determine this distance

$$d = -\frac{1}{\rho \ell \mu_{\ell}} \ln\left(\frac{I}{I_0}\right) \quad (24)$$

The absorption coefficient of water was obtained from previous measurements. Figures 3 and 4 compare the actual distances (based on chord lengths for the circular column) with distances obtained using eqn. 24 for the low and high energy sources respectively. These results show that the count rate for the high energy source is significantly higher than that for the low energy source, which is as expected based on the strengths of the two sources. However, for a full column the count rate for the low energy source when located at the center position is around 150 (Figure 1), which is very low and the noise to signal ratio for this count rate would be relatively high. This is not the case with the high energy source (Figure 2), where the lowest count rate for a full column is around 3500 at the center position. Sensitivity of the two sources to changes in hold-up structures can be estimated by comparing count rates at the center location for the empty and full column. For the Americium-241 source, the count rate with the column empty is 3000 and with the column full it is 150, a 95% change in the count rate. Whereas, for the Cesium-137 source, the count rate with the column empty is 21,500 and with the column full it is 3500, a 84% change in the count rate. Thus, the sensitivity of the low energy Americium-241 source is marginally higher than that for the high energy Cesium-137 source. This is not surprising considering that the beam from the low energy source is almost completely absorbed by the liquid medium. The time constants for radioactive sources are a function of the count rate. A low count rate

translates to a large time constant and vice versa. For a count rate of 150 (low energy source with the column filled with water), the time constant for obtaining a count rate with a 0.5% standard deviation is around 120 seconds, whereas for a count rate of 3500 (high energy source with the column filled with water), the time constant for the same accuracy in measurement is 4 seconds. The high energy source would therefore have a quicker response to changes in the flow field. When the standard deviation is increased to 5%, the time constants for the two sources are 1.2 and 0.04 seconds, respectively. Widths of the beams emitted by the two sources were measured at two locations, near the source and near the detector. For the low energy source, the beam width near the source was 0.92" and near the detector the beam was 1.68" wide. The values were 0.39" and 0.80", respectively, for the high energy source. These experiments show that the high energy source appears to satisfy the criteria for a good source very well, however, the low energy source would not work satisfactorily. The low energy source fails on several counts. It is too weak for our application, the time constant is very high, and the beam is significantly wider than the beam from the high energy source. Narrowing the beam for the low energy source using a collimator would only decrease the count rate further. The problem with the low energy source is evident from results shown in Figure 3. The low energy source underpredicts the distance through the column for all radial positions, except at the center. The error between the actual and estimated distances increases with distance from the center of the column and is as high as 45-50% at around 3" away from the center. Whereas, results from the high energy source (Figure 4) show an excellent agreement between actual and estimated distances, with an error of only 0.5-1.0% at 3" away from the center of the column.

Based on our findings from the source evaluation studies, we decided to replace the low energy Americium-241 source with a new source that would better suit our requirements. After looking into the different sources available, we decided to obtain a

35 mCi Cobalt-60 source. This source has an half-life of five years and emits 2 gamma rays per disintegration, one with an energy of 1.14 and the other with an energy of 1.33 meV. Thus a 35 mCi Cobalt-60 source has activity that is similar to a 70 mCi source which emits only one gamma ray per disintegration (such as the Cesium-137 source). The higher count rate should give us relatively low noise to signal ratios. Safety requirements, source pricing and availability were taken into consideration while selecting the new Cobalt source. We have subsequently amended our license permitting the use of radioactive materials to include the use of the Cobalt-60 source.

V. Nomenclature

a, b, a', b' terms defined by eqns. 19, 20, 22 and 23, respectively

d distance traveled by the beam when passing through the measurement volume (cm)

I intensity of radiation as measured by the detector (counts/s)

MW molecular weight of the gas (g/g mole)

P pressure (atm)

R gas constant, 82.056 cm³.atm/g mole

T temperature (°K)

Greek letters

α phase weight fraction

ϵ phase hold-up

μ absorption coefficient (cm²/g)

ρ density (Kg/m³)

Subscripts

g gas

H related to the high energy source

ℓ liquid

L related to the low energy source

o empty measurement volume

s solids

s ℓ slurry

Table 1. Gas hold-up values for the air-water system with the Cesium-137 source without collimator.

Air Pressure (psig)	Gas Hold-up (%)		% Rel. Dev.
	Americium-241 Present	Americium-241 Absent	
40	14.9	13.0	13.6
60	20.2	16.3	21.4
80	25.2	20.0	23.0

Table 2. Gas hold-up values for the air-water system with the Cesium-137 (collimated) and Americium-241 sources.

Air Pressure (psig)	Gas Hold-up (%)		% Rel. Dev.
	Americium-241	Cesium-137	
Center:			
20	8.5	8.6	1.2
40	13.1	12.9	1.5
60	15.8	16.0	1.3
2" Right:			
20	6.5	7.5	14.3
40	10.0	10.5	4.9
60	12.0	12.6	4.9
2" Left:			
20	5.7	6.4	11.6
40	10.2	9.7	5.0
60	12.4	11.6	6.7

Table 3. Comparison of gas hold-ups from nuclear density gauge (NDG) measurements with those from DP cell measurements.

Air Pressure (psig)	Gas Hold-up (%)		
	Americium-241	Cesium-137	DP Cell
20	6.9	7.5	6.2
40	11.1	11.0	10.4
60	13.4	13.4	12.9

Table 4. Absorption coefficients for solids with Cesium-137 source.

wt.% Solids	Sand	Silica	Iron Oxide
10	0.2036	0.1741	0.3315
20	0.2012	0.1728	0.3268
30	0.2168	0.1697	0.3250
Mean	0.2072	0.1722	0.3277
% std. dev.	4.0	1.3	1.0

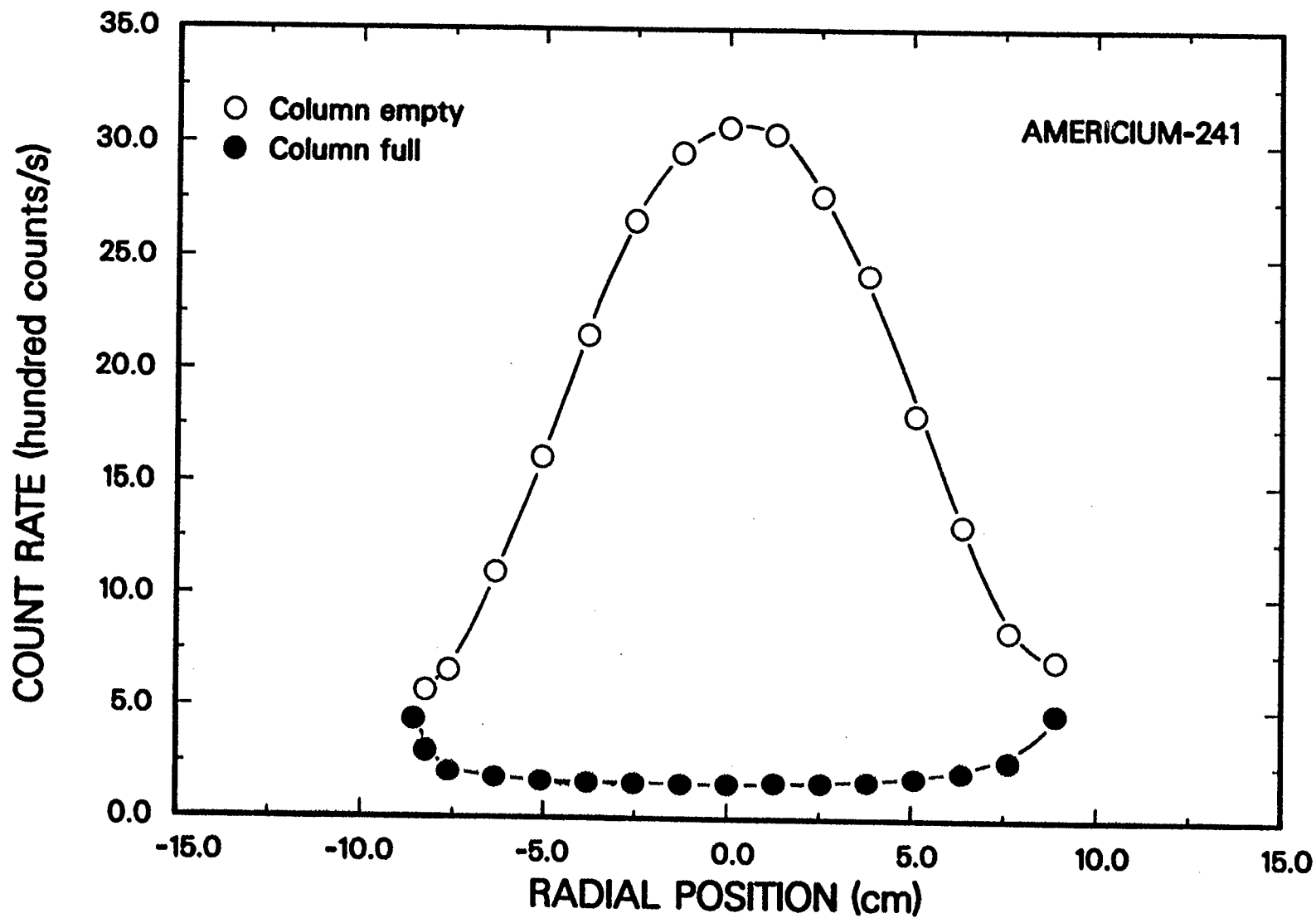


Figure 1. Intensity profiles from radial scans of the column when empty, and when filled with water (low energy source).

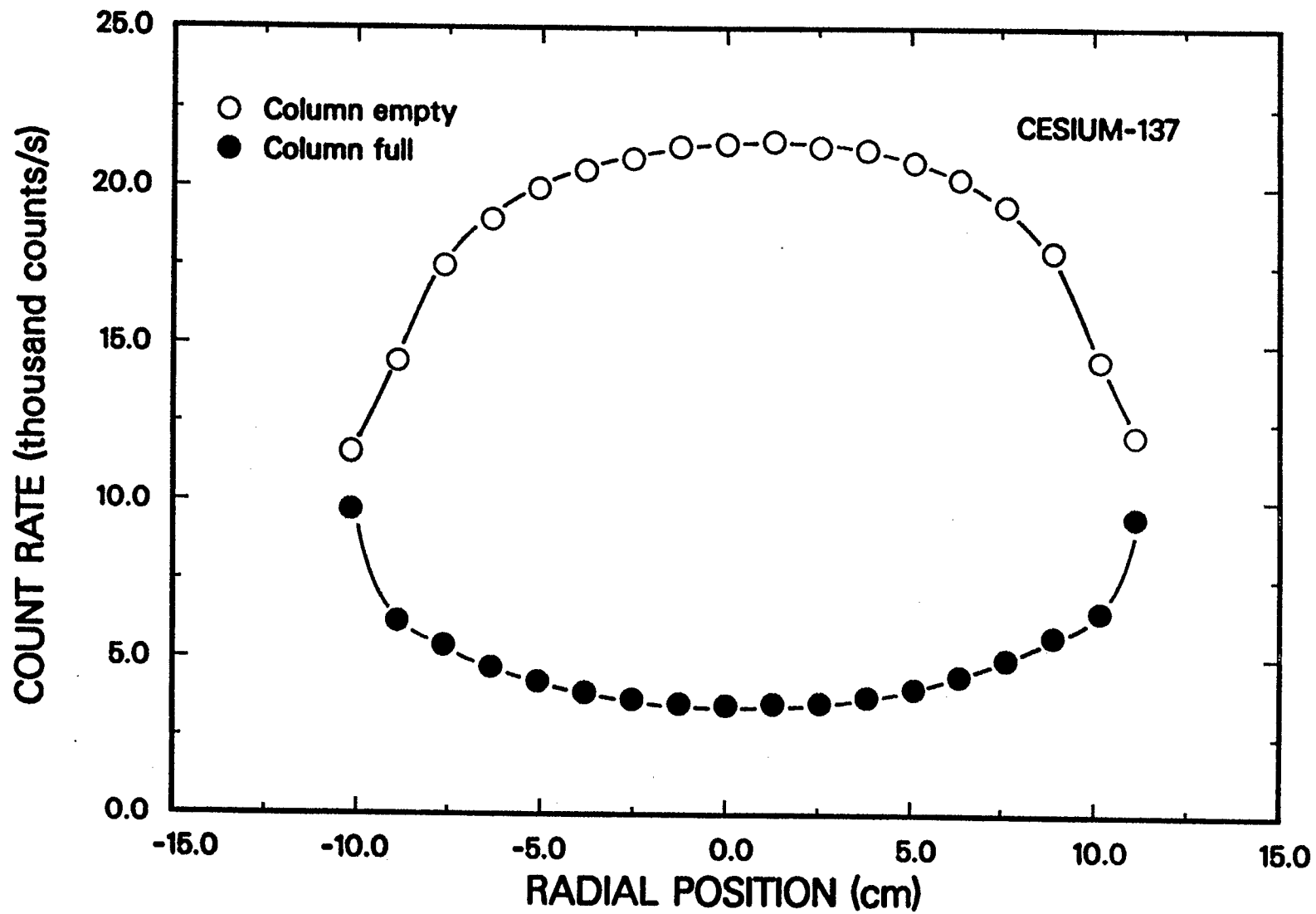


Figure 2. Intensity profiles from radial scans of the column when empty, and when filled with water (high energy source).

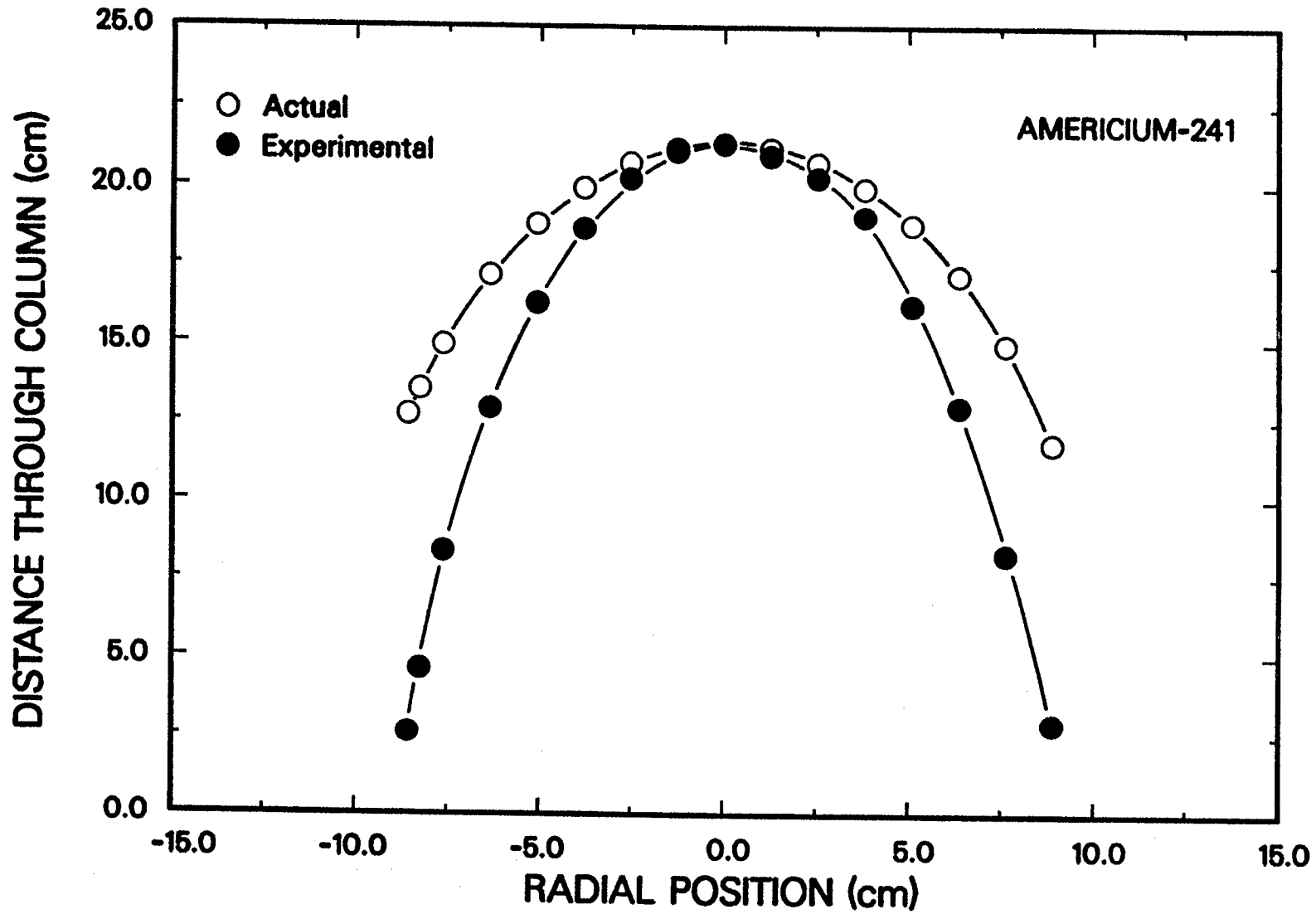


Figure 3. Comparison of actual beam path length with experimental values obtained using the low energy source and with column filled with water.

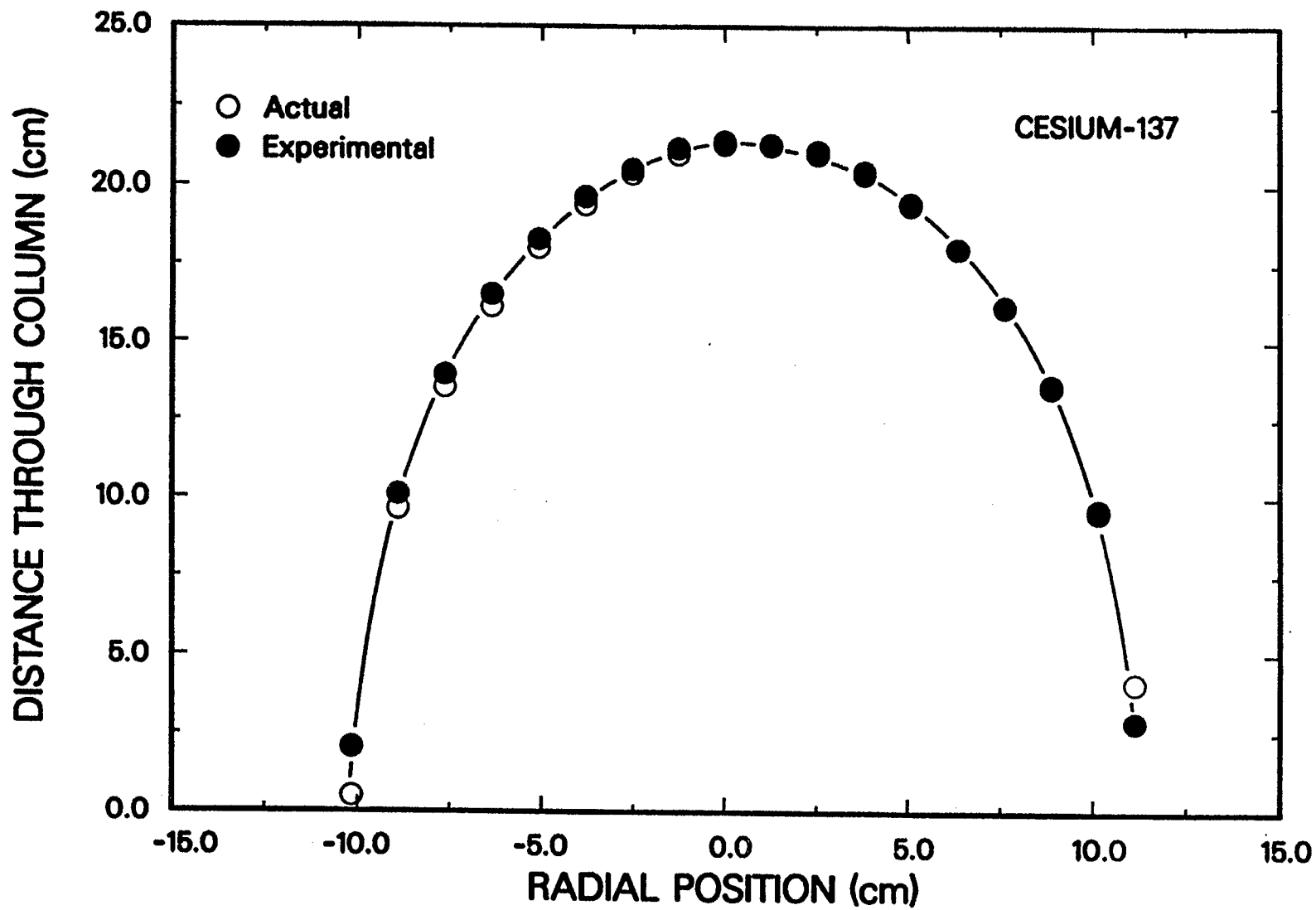


Figure 4. Comparison of actual beam path length with experimental values obtained using the high energy source and with column filled with water.