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NATIONAL BUREAU OF STANDARDS REPORT

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Progress Report
on
PROPERTIES OF DENTAL AMALGAM MADE FROM SPHERICAL PARTICLES

by

Neil C. Demaree

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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PROPERTIES OF DENTAL AMALGAM MADE FROM SPHERICAL PARTICLES

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PROPERTIES OF DENTAL AMALGAM MADE FROM SPHERICAL PARTICLES

Abstract

Experimental dental amalgams were prepared for the first time using alloys in the form of spherical particles 2 to 150 microns in diameter rather than the irregular particles normally employed. By suitable selection and blending of particle sizes physical properties of the amalgams were controlled. Increase in particle size increased setting time and setting expansion and decreased residual mercury content. Particle sizes between 15 and 50 microns in diameter gave maximum strengths. Physical properties of some of the experimental amalgam alloys were equal or superior to the properties of any amalgam alloys now in use. The use of spherical particles provides a new approach to the investigation of the setting reactions of dental amalgam.

1. INTRODUCTION

1.1 Statement of the Problem

The dental amalgams are the most widely used material for the treatment of dental caries; in fact more than three-fourths of all dental restorations are made of the silver amalgam type alloys. These facts indicate the relative importance of this type of material and the basic justification for any research, the object of which is the improvement of the alloys used for dental application. The amalgams in general use today may contain as many as five metals (silver, tin, copper, zinc, and mercury). This makes a rather complex alloy, the basic metallurgy of which is not completely understood.

This study is confined to one phase of this problem, namely, the effect of particle size of the alloy before mixing with the mercury on the physical properties of the resulting amalgam. Previous studies on the effect of particle size have been made using alloys which contained the conventional type of particles. These particles are in the form of filings which are made by machining a cast ingot on a lathe, adjusted to give certain size cuts. The particles obtained from these cuts may be reduced by ballmilling or other methods to give the desired distribution of particle sizes. It is evident this procedure produces particles having a wide variation in shape and size which makes the determination of surface area and particle size most difficult. In order to simplify this factor for this study, it was decided to prepare the alloy into small spherical particles and study the effect of particle size when the surface is a definite function of the particle diameter. Comparative data on three widely used commercial alloys are used for comparison of physical properties that have dental significance, namely, strength, flow, dimensional change on setting, and mercury content.

The available literature does not contain any previous attempts to study the effect of particle size by employing spherical-shaped particles. The approach used in this study appears to be conducive to a simplification of the problem. From a clinical point of view, it offers a new and unique feature that may be very helpful in the production of an improved dental amalgam restoration.

1.2 Previous Work

Since early investigation of amalgam, many have recognized and commented on the effect of particle size. One author [1] has commented that the greatest single difference between various amalgam alloys currently available may be the particle size and shape. Although the importance of particle size is generally realized, relatively little work has been done on this particular aspect of amalgam research.

Gray [2, 3] in 1921 was one of the first to investigate the effect of particle size. He demonstrated this effect by measuring the dimensional change of amalgam during setting. The various particle sizes were obtained by sieving, and his study showed a general decrease in expansion as the particle became smaller. He concluded that as the particle size is made progressively finer, the expansion curve undergoes the same sort of transformation that accompanies increase of trituration time. His investigation also led to the conclusion that particles should not be necessarily the same size, but by suitable blending of alloy particles that differ in size, it is possible to control the dimensional changes in the amalgam filling.

In 1942, the effect of particle size on dimensional change was also studied by Jarabak [4]. His particle sizes were obtained by sieving four alloys with 100, 200, and 325 mesh sieves. He also observed a decrease in expansion as the particles became smaller. The decrease was not particularly uniform, but in most instances showed the greatest sensitivity between particles retained on 200 and on 325 mesh sieves.

Crowell and Phillips [5] felt that the important consideration was not the size of the particles in terms of its volume but rather in terms of its surface area. Their study was made on the effect of specific surface (actual surface area per unit weight of particles) not only upon the dimensional change but also on setting time, compressive strength, and flow. Samples of alloy containing widely different specific surfaces were produced by varying the thickness of the particles which was determined by the feed of the cutting tool. They found that properties of particles of a given thickness cut from different areas of the same ingot varied. From tests made on cuttings of varying thickness from adjoining areas of the ingot, they reported that as the surface area was increased, the expansion, setting time, and flow all decreased. The compressive strength increased as the surface area was increased, with the one-hour compressive strength showing the greatest amount of increase. They concluded that the expansion was a linear function of the surface area and that the setting time plotted against the reciprocal of the relative surface area approached a straight line.

Coy and Liebig [6] reported some tensile strengths of amalgam alloys having different particle sizes. The specimens made from the comminuted alloy had a much higher tensile strength at the end of one day than the coarsely cut alloy, but showed only a slight difference after five days. They also demonstrated that the initial particle size and shape have a large influence upon the volume changes of the hardening amalgam. Specimens made from particles retained by a 200-mesh sieve showed a high expansion, the particles that passed a 200-mesh sieve showed only a slight expansion, while particles sorted by elutriation showed a contraction.

A number of authors [7, 8, 9] have commented on the importance of particle size, the need for more extensive research, and the possibility of including it in the specifications for dental amalgam. Smith [8] has commented as follows:

> More research of a fundamental kind, initiated from different view points, will eventually enable a sound specification to be made and so applied that the dentist will at least be able to rely with confidence on an exact reaction rate, time of treatment, and dimensional change, from his amalgam alloy particles.

2. EXPERIMENTAL PROCEDURE

2.1 Materials Used

An experimental spherical alloy was prepared for this investigation. The chemical composition of the alloy was designed to be typical of popular amalgams currently in use and to fall within the limits established by the American Dental Association Specification No. 1 for Dental Amalgam.

Ingots of the experimental alloy were prepared in a covered graphite crucible using a Westinghouse radio-frequency generator as a source of heat. After removal of the cooled ingot from the crucible, the surface was cleaned by immersion in dilute nitric acid for five minutes. The 500 gm ingots were approximately 4-½" long by 1-1/4" in diameter. The analysis of the silver, tin, copper, and zinc used is given in Tables 1, 2, 3, and 4.

The ingots were made into spherical particles by the Federal Mogul Division, Ann Arbor, Michigan, by a patented atomization process [10]. Atomization was done in two heats. The initial, or dummy heat, using two ingots of alloy, was done to clear the equipment of contaminants. The second heat, using three ingots, produced 1366 grams of spherical alloy which was an approximate weight loss of 10% of the original alloy. The two heats were received after separation by passing them through a No. 325 mesh sieve.

Three commercial amalgam alloys were selected as controls from the American Dental Association list of certified materials. These alloys cover a range of particle sizes and setting times. Table 5 shows the brand names, manufacturers, and batch numbers of the alloys. The chemical compositions of the control and experimental alloys are given in Table 6.

Two compositions are listed for the experimental alloy, the batch formula and the composition determined by analysis. Trace elements of Fe, Al, Mn, Ni, Pb, and Si are largely responsible for the 0.4% not accounted for in the analysis. Contaminants in the atomization equipment are believed to be responsible for their presence. It is thought that these trace elements were not in sufficient quantity to influence the results of this study.

2.2 Separation of the Experimental Alloy into Size Ranges

In order to study the effect of particle size on the physical properties of amalgam, it was imperative that a satisfactory separation of the spherical alloy into individual size ranges be obtained.

Three methods of particle size determination most commonly used are sieving, elutriation, and microscopic examination. Sieving can be accomplished rapidly and accurately to as low as 74 microns. Elutriation has its greatest usefulness for particle sizes ranging from 5 to 100 microns, and microscopic sizing can be done for the range of a few tenths of a micron to about ten microns [11].

Sieving was selected as the method of separation for particles having a diameter greater than 74 microns, and elutriation was used as the method of separation for particles having a diameter less than 74 microns.

Microscopic and visual examination of the as received alloy revealed the need for a cleansing procedure before separation. Therefore, a rather extensive washing was done to accomplish the following three objectives: the removal of any surface contaminants such as grease or oil, the elimination of cohesiveness between the large and very small particles, and the removal of very fine particles, small enough to float on the washing medium.

The plus 325-mesh fraction was put through a series of washings using water, ethyl alcohol, acetone, and ether in that order. The sequence in which the washing media were used also removed the water. The minus 325-mesh fraction was also put through this series of washings, with the exception of acetone, but in reverse order. Since this fraction was being separated by water elutriation, the washing was continued during separation, and the alloy was not dried until completion of the separation procedure.

After drying the plus 325-mesh fraction, it was passed through three sieves, No. 100, No. 140, and No. 200 mesh. A portion of the U. S. Standard Sieve Series covering the range of this particular separation is given in Table 7. The amount that was retained on the 100-mesh sieve, which included particles with a diameter greater than 149 microns and a small amount of trash in the form of oxides, was discarded. The largest fraction, that which passed through a 100-mesh sieve but was retained on a 140-mesh sieve, was designated size range 8 and contained particles with a diameter of 105 microns to 149 microns. In a like manner size range 7 was determined by the amount of alloy that passed a 140-mesh sieve but was retained on a 200-mesh sieve. This gave a size range with particles having a diameter of 74 to 105 microns.

The alloy passing the 200-mesh sieve and the minus 325 mesh fraction were separated by means of elutriation.

An elutriator was constructed as shown in Figure 1. The apparatus consisted essentially of two separation chambers, two collectors, and a flow regulating system. A standard side-arm flask was attached to a removable capillary tube by a short piece of rubber tubing. The capillary was then connected to a constant pressure head, also by rubber tubing. The pressure head contained two connections, one to a source of water and the other to an overflow. A glass tube, A, approximately 14 inches in length to the point of constriction and with an inside diameter of 1-1/4 inches, containing a stop-cock at the lower constricted end, was fitted into the mouth of the flask through a rubber stopper. A second tube, B, about the same length as A but having an inside diameter of 1-$\frac{1}{2}$ inches, was fitted into the top of tube A. Just below the connecting stopper, the constricted portion of tube B was flared to form a bell-shaped opening. A combination of rubber and glass tubing connected the top of tube B to a collection beaker.

The rate of flow of water into the system was controlled by using capillaries of varying lengths and inside diameters and by changing the height of the constant pressure head. Table 8 gives the calibration of a continuous series of 35 flow rates obtained by using seven different capillary tubes at five different heights of the pressure head. These flow rates, ranging from 0.52 ml/sec to 16.10 ml/sec, were determined by measuring the time required to collect a known volume of water using a given capillary-pressure head height combination.

It was observed that air bubbles carried the alloy particles over into the collecting beaker without obtaining separation. Therefore, before starting the separation, the apparatus was completely filled with water in order to remove all air. By placing a pinch-clamp on the tubing connecting the pressure head and capillary, and closing the stop-cock on tube A, the capillary could be changed without losing water from the system.

Using the capillary and pressure head height to produce the desired flow rate, about 30 grams of the alloy were placed in tube B. The alloy settled to the bottom of tube A, since the closed stop-cock prevented the particles from entering the side-arm collection flask. The pinch-clamp and the stop-cock were opened in that order permitting the known flow rate of water to circulate through the elutriator. This created a turbulent movement of the alloy particles in tube A. As the water passed into tube B, a smooth laminar flow was produced by the bell-shaped nozzle. The smallest particles were carried into a collection beaker through the tubing connecting the top of tube B to the beaker. The flow was allowed to continue until no particles could be observed leaving the top of tube B. The water supply was then cut off, allowing the remaining particles in tubes A and B to settle into the side-arm flask.

At a flow rate of 8.68 ml/sec, particles with diameters of 50-74 microns collected in the side-arm flask. This was size range 6. Particles with diameters less than 50 microns were carried over into the collection beaker. This fraction was re-elutriated at a slower flow rate. In a similar manner as described for size range 6, five additional fractions containing progressively smaller particles were collected by decreasing the flow rate.

A summary of results obtained from heat number two is given in Table 9.

The same drying procedure used for the particles separated by sieving was also used for the particles separated by elutriation, namely, washing three times each in alcohol, acetone, and ether in that order. In addition, the three smallest size ranges were placed in a desiccator under a vacuum for 24 hours. As a check on the possibility of any remaining water, a weighed sample of size range 1 was placed in an oven at 149° centigrade for four hours. After removal from the oven, the weight was again determined. The 0.1 mg weight change was within the weighing error of the balance used.

The diameter of the particles in each size range was measured microscopically. Within a selected field of the microscope, the diameters of eight to ten of the smallest particles were measured and the average taken. The largest particles of the same size range were determined in a like manner. There was a certain amount of overlapping of particle sizes between size ranges (± 2 microns). However, the separation obtained was considered satisfactory for the purpose of this study. The average particle diameter of each size range was taken as the middle of the range on the assumption that each size range contained an even distribution of particle sizes. For size range 1 the average particle diameter would be greater than the two microns listed in Table 9, since particle sizes of one micron and less were lost in the separation procedure. Using an average particle diameter of three microns for size range 1 and assuming constant volumes, this represents an increase of about 40 times the surface area of size range 8.

Figures 2, 3, and 4 show particles of the spherical alloy from size ranges 1, 5, and 8. Particles from control B representing the finest cut, and particles from control C representing the coarsest cut conventional type alloy used in this study, can be seen in Figures 5 and 6. All pictures were made at the same magnification of 132X and a scale showing 200 microns is drawn on each.

2.3 Specimen Preparation

All test specimens of amalgam were made following a uniform technique, with the exception of trituration time.

An 8:5 mercury alloy ratio was used for all alloys. Each mix consisted of 0.64 gram of mercury and 0.4 gram of alloy as weighed on a torsion balance to the nearest ten mg. These amounts were placed in a Caulk capsule with plastic pestle, and the start of trituration of the first mix was used for measurement of the age of the specimen. Two capsule loads were used for each compressive strength and flow specimen, whereas three to four capsule loads were required for the setting change specimens.

Trituration was done mechanically using a Crescent (Wig-l-bug) amalgamator. Normal operating speed of this particular amalgamator, with a loaded capsule, was 3330 ± 20 cycles per minute as determined by means of a General Radio "Strobatac type 631-b". Time of trituration was ten seconds per mix for the experimental alloy, and the time recommended by the manufacturer was used for the three control alloys. These times for alloys A, B, and C were 7, 10, and 13 seconds respectively. Trituration time was measured by the automatic timer on the Crescent amalgamator.

After trituration each mix was placed in a squeeze cloth, rolled and divided into three equal portions. Mercury was expressed from each increment of the mixed amalgam by squeezing it through the cloth with hand and pliers immediately before condensing in a steel mold. The mold used for the compressive strength and flow specimen was 4 mm in diameter by 8 mm in length, and the mold used for the setting change specimen was 5 mm in diameter and 10 mm in length.

Condensation of all specimens was accomplished by hand packing. A 2.5 mm Sweeney plugger was used with 15 ± 1 pounds force. This corresponds to about 2000 psi. Condensation force was measured with a Starrett pressure-gage indicator. Using this pressure, eight to ten thrusts were made on each increment of the mixed amalgam. Excess mercury was removed during packing by using pluggers with diameters just slightly less than the diameters of the molds. Increased force was applied with the larger pluggers, but pressure did not exceed 2000 psi. Each mix of a specimen was condensed in the mold before starting the trituration of the following mix. The top of the specimen was cut off with a razor blade before removal from the mold.

The preparation of all specimens was completed within six to eight minutes from the start of trituration of the initial mix. All specimen preparation, storage and testing was done in a room with controlled temperature of 73°F ± 2°F and relative humidity of 50% ± 4%.

Due to the great difference in particle size between the largest and smallest size range of the experimental alloy, minor deviations from a more standardized procedure had to be adopted.

The rapid setting of the small particles and the condensing properties of the very large particles gave the most trouble in this respect.

2.4 Testing Procedure

The physical properties determined on the amalgam specimens made from the experimental alloy and the controls were dimensional change during setting, flow, and compressive strength. Residual mercury analysis was also done on a number of the specimens after they were first subjected to tests to determine compressive strength or setting changes.

2.4.1 Setting Changes

Using a dental interferometer the setting changes were measured as the amount of dimensional change occurring during hardening of an amalgam cylinder one cm in length.

Setting changes were determined on six specimens made from each of the size ranges of the spherical alloy and the controls. The fiducial reading was taken at ten minutes from the start of specimen preparation, and the final reading was taken when no further setting change was observed on the dental interferometer. All values reported represent total dimensional change. Intermediate readings were taken on all specimens to determine the approximate time required for the total setting change to take place. Time intervals varied for these intermediate readings, depending upon the material being tested. However, 15, 30, and 45 minutes, 1, 2, 4, 6, and 24-hour readings were taken on most specimens. After the first 24 hours, readings were taken at one-day intervals.

The mercury content was intentionally varied on the setting change specimens of size range 8 to determine the effect of mercury content on dimensional change for a given particle size. This was accomplished by squeezing more mercury from the plastic mix of amalgam with the pliers and by increasing the condensation pressure during specimen preparation. All setting change values are reported to the nearest 0.25 micron per centimeter.

2.4.2 Flow

Flow as used here is defined as the percentage of shortening in length of a cylinder of prescribed dimensions subjected to a given compressive stress for a specific time and temperature.

Flow was determined on six specimens made from each of the eight size ranges of experimental alloy and the three control alloys. The tests were made according to the American Dental Association Specification No. 1 for Dental Amalgam Alloy (First Revision). Before making the tests, the three flow instruments were calibrated to produce the specified stress.

Additional modified flow tests were made starting with one-week-old specimens. These values were determined on three specimens made from each of size ranges 1, 3, 5, 6, 7, and 8 and the three controls. For size range 8, flow was determined taking the initial reading one month from the time of preparation. All flow values are reported to the nearest tenth of one percent.

2.4.3 Compressive Strength

Compressive strength is defined [12] according to ASTM as "The maximum compressive stress which a material is capable of sustaining. Compressive strength is calculated from the maximum load during a compression test and the original cross-sectional area of the specimen".

Cylindrical specimens four mm in diameter and eight mm in length were prepared for the compressive strength tests. An Instron testing machine, applying a uniform rate of strain, was used to determine compressive strength, and a head speed of 0.010 in/min was used as the loading rate for all specimens. The load cell was calibrated with a known weight, and the specimens were subjected to testing with the machine operating over a 0-2000 pound load capacity.

Compressive strength values were obtained on one, two, six, and 24 hours, one week, and one month-old specimens. Three to six specimens of a given size range or control were used to obtain the compressive strength values at these time intervals. All values are reported to the nearest 100 psi.

2.4.4 Residual Mercury

Residual mercury was determined as the total mercury content of the hardened amalgam specimens. The technique used was a modification of the method of Crawford and Larson [13]. In this method, the amount of mercury is determined as the weight loss of the sample rather than by collecting the mercury.

The specimens on which the mercury was determined, ranging from 150 to 200 mg, were weighed into porcelain combustion boats previously dried to constant weight. The boats were placed into a pyrex tube which had been sealed at one end and could be placed in a horizontal, electrically heated tube furnace. When the boats were in place, the other end of the tube was closed with a rubber stopper having a 3-way stop-cock. The stop-cock was connected to a vacuum line and a tank of nitrogen. The tube was alternately evacuated to 5 to 10 mm of mercury and flushed with nitrogen. This procedure was repeated at least three times before the tube was finally closed in the evacuated condition. The tube was then slid into the furnace. In the area of the boats, the temperature was approximately 500°C, and the samples were heated for 1-$\frac{1}{2}$ hours. The vaporized mercury condensed on the cool portion of the tube remaining outside the furnace. The tube was then removed from the furnace and allowed to cool to room temperature before releasing the vacuum. The condensed mercury was removed mechanically before taking the combustion boats out of the tube for reweighing.

Two crushing strength specimens and two setting change specimens for each size range and the controls were used for the mercury determination. A random selection of the specimens was made independent of the compressive strength and setting change values previously determined. Each specimen was crushed in a steel mortar and pestle until it would pass a 100-mesh sieve. All specimens were weighed to 0.2 mg.

Prior to doing the mercury analysis of the amalgam specimens, calibration runs were made using pure tin, dental alloy and dental alloy plus a known amount of mercury. The results showed the calibration good to 0.2 mg, with the main source of variation being due to the small sample size and weighing error. All residual mercury values are reported to the nearest tenth of a percent.

2.4.5 Blending of Particle Sizes

Flow, setting change, 24-hour compressive strength and mercury content were determined on each of four mixtures of particle sizes. The first mixture contained all particle sizes that would pass through a 325-mesh sieve. The maximum particle size was about 45 microns, and the particle size distribution was determined by the atomization process.

The composition of the other three mixtures were determined by assuming close-packed spheres and calculating the particle size and number of particles necessary to fill the spaces created by the first three sizes, assuming the largest particle size to be one unit. Results of these calculations gave particle sizes with a ratio of 1.0, 0.414, 0.19, and 0.08 and, a weight percentage ratio of 89.3, 6.2, 3.6, and 0.9 respectively.

Using this calculated weight percentage, two additional mixtures were prepared. The second contained size ranges 8, 5, 4, and 2, and the third contained size ranges 6, 4, 3, and 1. A fourth mixture was made using the same size ranges as the second but doubling the amount of the three smaller sizes. This reduced the weight percent of size range 8 from the calculated 89.3 to 78.6. This fourth range was made with the idea of increasing the probability that the three smaller sizes would occupy the calculated space. These four mixtures are designated A, B, C, and D, respectively. The composition of these mixtures are given in Table 10. Values reported for all tests on each of these mixtures were determined from three specimens.

2.4.6 Variation of Mercury Alloy Ratio and Trituration Time

The three physical properties and mercury content were determined on specimens made from size range 4 and control alloy C, varying the mercury alloy ratio and trituration time. This was done to determine the effect upon the physical properties and to compare the sensitivity of spherical particles with that of the conventional filings, when manipulation was varied. One flow specimen, two setting change specimens, and three 24-hour compressive strength specimens were made from size range 4 using a 1:1 and a 4:1 mercury alloy ratio. Tests on specimens using an 8:5 ratio had been determined previously. The same number of specimens as given above were made from size range 4 and control C, increasing the trituration to three times that which had been used throughout the investigation. For size range 4 this was 30 seconds and for control C 39 seconds.

Mercury analysis was done on five specimens selected from the group varying the mercury alloy ratio and on five specimens from the group with increased trituration time.

3. RESULTS

3.1 Setting Change

The effect of particle size on dimensional change during setting was in agreement with results of previous investigations [2, 3, 4, 5]. As shown in Figure 7, an increase in particle size resulted in an increase in expansion and in the length of time required to attain final set. The smallest size range showed 7.75 u/cm contraction, whereas the size range containing the largest particles showed an expansion in excess of 200 u/cm.

Total setting change and approximate time required for this change are shown in Table 11 for both the experimental alloy and controls. Size ranges 1 through 4 showed a final contraction with very little change being observed after the first two to four hours. Size range 5 was the only size range that showed an initial contraction followed by a subsequent expansion, typical of setting change curves for conventional type alloys. The 3.75 u/cm setting change for this size range was completed within about six hours. The amount of setting change increased very rapidly with size range 6, which required about 18 hours to expand 23.75 u/cm. The 46 u/cm expansion observed for size range 7 required approximately 42 hours for completion, while setting changes for size range 8 continued to show expansion for as long as 14 days.

Figure 8 is a plot of setting change versus time for three of the specimens of size range 8 with intentionally varied residual mercury content. The specimen that had the lowest mercury content, obtained by increasing the condensation pressure, gave only 78 u/cm expansion within 4 days. The specimen with the highest mercury content gave an expansion of 236 u/cm and required two weeks for completion. The condensation pressure of 2000 psi used in the preparation of all other specimens of this investigation produced 192 u/cm expansion. Setting change for the first 24 hours differed for the three specimens, but the following expansion took place at a constant rate of about 18 microns per day.

3.2 Flow

The effect of particle size on the flow of amalgam is given in Figure 9. One curve shows the percentage of flow determined at the end of 24 hours, and the other curve represents the percentage of flow determined on one week old specimens.

The results of the 24-hour tests show little difference in size ranges 2 through 5, but there is a significant difference as each extreme in size is approached. This increase was considerably greater for the largest particle size than for the smallest one. The specimens made from size range 8 broke under the initial 3550 stress when applied at three hours. However, starting with specimens one week old size range 8 showed 2.3% while there was no significant difference in any of the other size ranges. Using specimens one month old, the flow value of size range 8 was further decreased to 1.2%.

A comparison of flow for the spherical alloy and the three controls can be seen in Table 12. Controls B and C showed about twice the amount of flow at the end of 24 hours as size ranges

2 through 5. The flow of control A was about the same as the spherical particles for these size ranges at both 24 hours and one week. Standard deviations are given in Table 12 for all values determined from six specimens.

3.3 Compressive Strength

The effect of particle size on compressive strength is shown in Figure 10. The maximum strength was obtained for size range 4 (15 to 30 microns) at time intervals of one, six, and 24 hours, and one month. However, the difference in strength between size ranges 4 and 5 after six hours was not significant.

The decrease in strength as each extreme was approached was greater for the largest particle size range than for the smallest. The 24-hour strength for size range 8 was very low, showing only 11,000 psi, and even after one month this size range had a compressive strength of 6,000 psi less than size range 1. This was true although size range 8 showed 17.5% lower mercury content. The 55,000 psi obtained for size range 4 at one month was over 20,000 psi higher than for size range 8.

A comparison of compressive strength for the experimental alloy and controls can be seen in Table 13. Size ranges 3 through 6 showed higher strength than control alloys B and C at all times tested and had higher early strengths than all three of the commercial alloys. After six hours control A was somewhat stronger than the experimental alloy, but it was at least 10,000 psi stronger than the other controls at all times.

Early strength for the first five size ranges showed unusually high values, ranging from 23,000 to 32,000 psi at the end of one hour. Table14 gives the relative strength of the alloys using the one-week strength as a basis of comparison. All size ranges with the exception of 7 and 8 were within 10% or less of the one week's strength after 24 hours, and ranges 3, 4, and 5 were at least 90% of their 24-hour strength as early as six hours. The first five size ranges gained strength more rapidly than the control alloys as shown by the percent of strength attained at the end of one hour.

3.4 Residual Mercury

Mercury analysis determined on two setting change specimens and two compressive strength specimens from each size range showed a decrease in total mercury content as the particle size was increased. The second curve in Figure 7 shows this effect of particle size on mercury content with the mercury content ranging from 40.1 for the largest particles to 57.6 percent for the smallest size particles. The first curve in Figure 7, representing dimensional change, produced an opposite trend as the particle size was increased.

Size range 4, which gave the highest compressive strength and the least amount of flow, had an average mercury content of 48.5%. Control A, which showed superior physical properties to the other two controls, had an average mercury content value of 41.5%. Table 15 shows the average value, determined from four specimens, for each size range and control. The standard deviation is indicated to show the spread of results. Duplicate tests were run on ten specimens picked at random, and an average of the difference obtained for duplicate tests of ten specimens showed 0.16% variation.

3.5 Effect of Blending Particle Sizes

The physical properties determined for four mixtures of particle sizes (Table 10) are given in Table 16. Properties for size ranges 4, 6, and 8 are also included so that the pronounced effect of blending particle sizes can be seen by comparing the physical properties of a single size range with those obtained for the mixtures.

Mixture A, which was the minus 325 fraction of the unseperated alloy with the particle size distribution determined by the atomization process, gave properties which were not significantly different from those obtained for size range 4.

Mixture B, containing 89.3% of size range 8 and 10.7% of smaller size ranges, showed the following improvements over amalgams made only from the large size particles.

1. The flow was reduced to 1.9% as compared to the specimens of size range 8 that broke under the load of the flow test.

2. The setting expansion was decreased from 200 u/cm to 19.25 u/cm and was completed within 24 hours.

3. The 24-hour strength was increased from 11,000 psi to 51,500 psi, which was more than 15,000 psi higher than the strength determined for size range 8 at the end of a month.

4. The total mercury content was reduced from 40.1 to 32.3%.

Mixture D, which contained twice the amount of each of the three smaller size ranges contained in mixture B showed an additional decrease in flow, setting expansion, and mercury content.

The properties determined for mixture C, which contained 89.3% by weight of size range 6, were superior to properties of this size range.

3.6 Effect of Varying Mercury Alloy Ratio and Trituration Time

Results of physical properties determined on size range 4 and control C, varying the mercury alloy ratio and trituration time, are shown in Tables 17 and 18. Examination of these data demonstrate the comparative effects on the physical properties of these two types of alloy, produced by these manipulation variables.

Tripling the recommended trituration time affected the conventional type of alloy to a much greater extent than the spherical alloy. Size range 4 gave about twice the amount of contraction when triturated for 30 seconds, but there was no significant change in the other properties. However, when control C was subjected to 39 seconds trituration time the amount of contraction was increased four times the value determined using the 13 seconds recommended time. The flow was increased 2.3%, and the mercury content was increased 4.0%. There was no significant difference in the compressive strength.

Increasing the mercury alloy ratio from 1:1 and 4:1 did not affect the properties of control C to the extent of that produced by increased trituration time. The setting changes appeared to be the most sensitive to variation in manipulation for both types of alloys. The expansion increased slightly as the mercury alloy ratio was increased. The variations obtained for the other physical properties were probably within the experimental error.

4. DISCUSSION

The results of this study indicate that particle size of dental alloy is one of the controlling factors influencing the physical properties of dental amalgam.

As was shown in Figure 7, particle size had a marked effect on both dimensional change during hardening and residual mercury content. There is general agreement in the literature on the effect of particle size on dimensional change. Results of this study also confirm the fact that an increase in particle size gives an increase in expansion. The opposite effect of particle size on residual mercury content may be somewhat contrary to the accepted beliefs of some, since many dentists always associate an increased expansion with an increased mercury content. As shown by the standard deviations of the residual mercury determinations in Table 15, this study has separated the effect of particle size from mercury content.

However, as was shown in Figure 8, when particle size was kept constant the residual mercury content became a controlling factor in dimensional change. A 10% reduction in mercury content reduced the amount of expansion occurring during hardening by about one third. No correlation was made with residual mercury content for either flow or compressive strength. The available time and limited number of specimens did not permit a further investigation of this particular aspect of the study.

The significant increase in flow determined at the end of 24 hours for the larger particle sizes can no doubt be accounted for by the incomplete hardening of the amalgam specimens, since setting changes were observed to occur for as long as two weeks. After the setting reaction was apparently completed, all particle sizes gave a flow value of about 1%. The slight differences could be attributed to experimental error and to surface irregularities of the ends of the specimens. This would indicate that the specification flow test is primarily a measure of early strength and rate of setting.

This study has shown that maximum strength was obtained with intermediate particle sizes. The incomplete hardening would also account for the decreased strength of the larger particle sizes when tested at early time intervals. However, the strength of the largest particle size was still relatively weak at the end of one month, when the setting reaction apparently was completed. The large amount of expansion, in excess of 200 u/cm, observed for the largest particle size during setting may have been partly responsible for the decreased strength of size 8. One explanation for the decreased strength of the smallest particle size may have been the high residual mercury content. According to Swartz and Phillips [15], there is a significant decrease in strength as the residual mercury content is increased above approximately 55%. The high one-hour strength determined for all particle sizes under 50 microns is most likely related to the rapid rate of hardening as shown by the early completion of dimensional change (Table 11), rather than to a high eventual strength.

Although the primary objective of this investigation was to study the effect of particle size, a comparison of physical properties obtained for the spherical alloy with those obtained for the conventional type alloy indicates that the spherical alloy may have certain advantages to offer. The higher early compressive strength and the lesser amount of flow obtained for the spherical alloy are advantages that would result clinically in an improved amalgam restoration.

Results obtained from testing mixtures of particle sizes (Table 16) certainly suggest that the physical properties of amalgam can be controlled by suitable blending of particle sizes.

Considering the shape of the particles for the two different type alloys, it would seem reasonable to expect the spherical alloy to be less sensitive to manipulation variables. As was shown in Table 17, the increased trituration time affected the physical properties of the conventional filings to a greater extent than it did for the spherical particles. One explanation for this is the possibility that a greater reduction in particle size was obtained for the commercial alloy when subjected to the increased trituration.

From the manufacturer's point of view, one additional advantage of this spherical alloy may be the simplification of manufacture. It would appear that dental alloy made by an atomization process could be accomplished with greater ease and less cost than the methods now employed.

The results of this study indicate that spherical alloy has much to offer both from a clinical aspect and a research point of view. Certainly it offers a new approach to the investigation of dental amalgam which may be quite helpful in obtaining a better understanding of basic principles involved in the setting reactions of the alloy and mercury.

5. SUMMARY AND CONCLUSIONS

An experimental dental alloy has been prepared into spherical form and separated into eight size ranges by means of sieving and elutriation. Using standardized testing procedures, physical properties have been determined on amalgam specimens made from three commercial alloys used as controls, individual size ranges of the spherical alloy, and mixtures of the individual size ranges. Data of this study showed that particle size affected dimensional change during setting, residual mercury content, flow and compressive strength in the following ways:

1. An increase in particle size increased the amount of expansion during setting and the length of time needed to attain final set.

2. An increase in particle size decreased the residual mercury content.

3. Particle size had no significant effect on flow after hardening had been completed.

4. Particle sizes between 15 and 50 microns in diameter gave the maximum strength at all time intervals tested, and all particle sizes under 50 microns gave high early strengths.

Laboratory tests have shown that amalgam can be made from spherical alloy with physical properties as good or better than the alloys currently in use. Results of this study have shown the main advantages of the spherical alloy over the conventional type alloy to be as follows:

1. Apparent simplification of manufacture.

2. Control of physical properties by suitable blending of particle sizes.

3. Less sensitivity to manipulation variables.

4. Less flow.

5. Higher early compressive strength.

This initial work with spherical dental alloy indicates the need for further study and investigation of amalgam employing alloy in this form.

The author wishes to express his sincere appreciation to Mr. William Sweeney, Chief, and to Mr. Duane Taylor, Metallurgist, Dental Research Section, National Bureau of Standards, under whose direction and guidance the work of this problem was conducted, to Mr. George Dickson, Physicist, and to Miss Claire Burns, Chemist, Dental Research Section, for their invaluable aid and criticism, and others, who, in one way or another, made this work possible.

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TABLE 1

COMPOSITION OF THE SILVER* USED FOR THE PREPARATION OF THE EXPERIMENTAL ALLOY AS DETERMINED BY SPECTROCHEMICAL ANALYSIS

Ag . . . . Major Constituent

	Range of Impurities (%)
Cu	0.1 - 1.0
Pb	0.01 - 0.1
Sn	0.01 - 0.1
Al	0.001 - 0.01
Ca	0.001 - 0.01
Fe	0.001 - 0.01
Mg	0.001 - 0.01
Mn	0.001 - 0.01
Pd	0.001 - 0.01
Pt	0.001 - 0.01
Si	0.001 - 0.01
Cr	0.0001 - 0.001

Tested for but not detected: As, Au, B, Ba, Be, Bi, Cd, Co, Ga, Ge, Hf, Hg, In, Ir, La, Mo, Ni, Rh, Ru, Sb, Sr, Ta, Te, Th, Ti, Tl, V, Y, Zn, and Zr.

*Obtained from Chemistry Division, National Bureau of Standards.

TABLE 2

COMPOSITION OF THE TIN* USED FOR THE PREPARATION OF THE EXPERIMENTAL ALLOY AS DETERMINED BY THE MANUFACTURER

Assay (SN) 99.9%

	Maximum Limit of Impurities (%)
As	0.0001
Cu	0.002
Fe	0.01
Pb	0.005
Zn	0.005

*Baker and Adamson, Reagent Tin metal (sticks) Lot No. R020.

TABLE 3

COMPOSITION OF THE COPPER* USED FOR THE PREPARATION OF THE EXPERIMENTAL ALLOY AS DETERMINED BY THE MANUFACTURER

Assay (Cu)	100.0%
Insoluble in HNO_3	0.008
Sb and Sr (as Si)	0.003
As	0.0003
Fe	0.0005
Pb	0.001
Mr	0.0003
P	0.0002
Ag	0.0005

*J. T. Baker Chemical Co., Reagent copper (heavy foil .005 in.) Lot No. 20442.

TABLE 4

COMPOSITION OF THE ZINC* USED FOR THE PREPARATION OF THE EXPERIMENTAL ALLOY AS DETERMINED BY THE MANUFACTURER

Assay (Zn)	99.9%

Maximum Limit of Impurities (%)

As	0.00001
Fe	0.010
Pb	0.010

*Baker and Adamson, Reagent Zinc Metal (sticks) Lot No. N353.

TABLE 5

AMALGAM ALLOYS USED AS CONTROLS

	Alloy	Manufacturer	Batch No.
A	New True Dentaloy	S. S. White Dental Mfg. Co.	12260078
B	Aristaloy	Baker and Co., Inc.	591234
C	Twentieth Century Regular	The L. D. Caulk Co.	31E60

TABLE 6

CHEMICAL COMPOSITION OF AMALGAM ALLOYS USED

Alloy	Composition (%)			
	Silver	Tin	Copper	Zinc
Experimental (batch formula)	71.0	26.0	2.5	0.5
(analysis)	70.6	26.1	2.5	0.4
*Control A	70.8	25.8	2.4	1.0
*Control B	69.0	26.7	3.8	0.5
*Control C	70.0	26.0	3.5	0.5

*Data obtained by the Dental Research Section, National Bureau of Standards (different batch numbers).

TABLE 7

PORTION OF U. S. STANDARD SIEVE SERIES*

Sieve No.	Sieve Opening (inches)	Sieve Opening (mm)
100	0.0059	0.149
120	0.0049	0.125
140	0.0041	0.105
170	0.0035	0.088
200	0.0029	0.074

*Taken from Federal Specification RR-S-366b for Sieves, Standard for Testing Purposes [11].

TABLE 8

CALIBRATION OF FLOW RATES

Capillary	Capillary Length and I.D.	Pressure Head Level	Time*	Flow Rate**
No.	mm	No.	seconds	ml/sec
1	Length - 75.0 I.D. - 0.5	1	1268	0.52
		2	1103	0.60
		3	976	0.68
		4	877	0.75
		5	805	0.82
2	Length - 300.0 I.D. 1.0	1	755	0.87
		2	650	1.02
		3	571	1.16
		4	505	1.31
		5	456	1.45
3	Length - 100.0 I.D. - 1.0	1	425	1.55
		2	387	1.71
		3	353	1.87
		4	330	2.00
		5	308	2.14
4	Length - 25.0 I.D. - 1.0	1	290	2.28
		2	263	2.51
		3	240	2.75
		4	222	2.97
		5	206	3.20
5	Length - 25.0 I.D. - 1.0 (I.D. enlarged both ends)	1	170	3.88
		2	150	4.40
		3	130	5.08
		4	115	5.74
		5	103	6.41
6	Length - 400.0 I.D. - 2.0	1	92	7.17
		2	83	7.95
		3	76	8.68
		4	70	9.43
		5	65	10.15
7	Length - 50.0 I.D. - 2.0 (I.D. enlarged one end)	1	60	11.00
		2	54	12.22
		3	49	13.47
		4	45	14.67
		5	41	16.10

*Time required to collect 660 ml of water.
**Calibration of flow rates ±0.02 ml/sec.

TABLE 9

SEPARATION RESULTS OF THE EXPERIMENTAL DENTAL ALLOY (HEAT NO. 2)

Size Range No.	Method of Separation	Determination of Size Range*		Size Range Diameter (microns)	Average Particle Diameter (microns)	Amount of Each Size Range (gms)	Percent of total Amount** (%)
		Flow rate (ml/sec)	Sieves (Mesh No.)				
1	Elutriation	P - 0.52		0 - 4	2.0	114.0	9.3
2	Elutriation	R - 0.52 P - 0.82		4 - 8	6.0	138.0	11.2
3	Elutriation	R - 0.82 P - 1.55		8 - 15	11.5	146.5	11.9
4	Elutriation	R - 1.55 P - 3.88		15 - 30	22.5	326.0	26.6
5	Elutriation	R - 3.88 P - 8.68		30 - 50	40.0	221.0	18.0
6	Elutriation and Sieving	R - 8.68	P - 200	50 - 74	62.0	98.0	8.0
7	Sieving		R - 200 P - 140	74 - 105	89.5	62.5	5.1
8	Sieving		R - 140 P - 100	105 - 149	127.0	66.5	5.4

*R-Particle sizes retained for a given sieve no. or retained in side-arm flask for a given flow rate.
P-Particle sizes passing through a given sieve no. or passing over into collection beaker at a given flow rate.
**4.5% lost in cleaning and separation procedure.

TABLE 10

PARTICLE SIZE COMPOSITION OF MIXTURES

Mixture	Size Range (weight percent)							
	1	2	3	4	5	6	7	8
*A	12.1	14.6	15.5	34.5	23.4	-	-	-
B	-	0.9	-	3.6	6.2	-	-	89.3
C	0.9	-	3.6	6.2	-	89.3	-	-
D	-	1.8	-	7.2	12.4	-	-	78.6

*Minus 325 mesh, as received, distribution of particle sizes determined by atomization process.

TABLE 11

RESULTS OF SETTING CHANGE TESTS FOR EXPERIMENTAL ALLOY AND CONTROLS

Alloy	Setting Change (u/cm)	S.D. (u/cm)	Time Required for Change
<u>Experimental</u>			
Size Range 1	- 7.75	2.1	2 - 4 hours
2	- 5.75	1.0	2 - 4 hours
3	- 5.25	0.7	2 - 4 hours
4	- 2.75	1.4	2 - 4 hours
5	3.75	0.8	6 - 8 hours
6	23.75	2.0	18 hours
7	46.0	1.5	42 hours
8	200.0	-	14 days
<u>Conventional</u>			
Control A	1.25	2.1	6 - 8 hours
B	6.0	1.2	6 - 8 hours
C	-4.75	2.5	6 - 8 hours

S.D. Standard Deviation $= \sqrt{\dfrac{\Sigma(x - \bar{x})^2}{n - 1}}$

TABLE 12

RESULTS OF FLOW TESTS FOR EXPERIMENTAL ALLOY AND CONTROLS

Alloy	No. of Specimens	Flow (24 hrs) %	S.D. %	No. of Specimens	Flow (1 wk) %
Experimental					
Size Range 1	6	1.9	0.5	3	1.0
2	6	1.3	0.3	-	-
3	6	1.2	0.1	3	0.7
4	6	0.9	0.5	-	-
5	6	1.1	0.1	3	0.6
6	6	2.2	0.4	3	0.9
7	6	5.1	1.1	3	1.0
*8	3	Broke	-	3	2.3
Control A	6	1.1	0.3	3	0.9
B	6	2.4	0.4	3	1.4
C	6	3.0	0.8	3	2.4

S.D. Standard deviation.

*Two specimens one month old gave an average of 1.2% flow.

TABLE 13

RESULTS OF COMPRESSIVE STRENGTH TESTS FOR EXPERIMENTAL ALLOY AND CONTROLS

Alloy	Compressive Strength (psi x 1000)*						S.D.
	1 Hr	2 Hr	6 Hr	24 Hr	1 Wk	1 Mo	
Experimental							
Size Range 1	24.3	30.3	36.5	39.3	41.9	38.4	2.6
2	23.8	-	-	42.9	42.7	-	2.5
3	28.4	41.4	46.6	47.9	47.8	50.6	1.2
4	32.1	46.4	49.7	50.8	52.6	55.3	1.2
5	23.1	37.6	48.6	48.9	53.0	53.7	2.1
6	-	-	30.4	43.7	47.6	47.6	1.3
7	-	-	-	35.3	41.2	42.7	2.9
8	-	-	-	11.2	31.3	32.0	3.9
Control A	21.5	36.1	51.8	52.2	58.0	60.1	1.0
B	-	16.6	38.1	40.3	46.2	-	1.0
C	-	-	-	41.4	45.7	-	2.0

S.D. Standard deviation (calculated, using values determined at all time intervals for a given size or control)

$$= \sqrt{\frac{\Sigma (x - \bar{x})^2}{n - \text{No. Avgs.}}}$$

* Strengths determined from tests made on 3 to 6 specimens, using a head speed of 0.010 in/min.

TABLE 14

EARLY COMPRESSIVE STRENGTH (PERCENTAGE OF ONE WEEK'S STRENGTH ATTAINED AT EARLIER TIME INTERVALS)

	Percent of One Week's Strength				
	1 Hr	2 Hr	6 Hr	24 Hr	1 Wk
Experimental					
Size Range 1	57.9	72.3	86.9	93.7	100
2	55.7	-	-	100.5	100
3	59.4	86.5	97.3	100.2	100
4	61.0	88.2	94.5	96.7	100
5	43.6	71.0	91.6	92.2	100
6	-	-	63.9	91.7	100
7	-	-	-	85.8	100
8	-	-	-	35.8	100
Control A	37.1	62.3	89.3	89.9	100
B	-	36.0	83.5	87.3	100
C	-	-	-	90.7	100

TABLE 15

RESULTS OF RESIDUAL MERCURY DETERMINATIONS FOR THE EXPERIMENTAL ALLOY AND CONTROLS

Alloy	Residual Mercury	S.D.
	(weight %)	(weight %)
Experimental		
Size Range 1	57.6	1.5
2	54.5	1.2
3	52.4	0.9
4	48.5	2.3
5	43.3	1.4
6	41.4	0.7
7	40.4	0.8
8	40.1	1.7
Control A	41.5	1.2
B	44.5	1.2
C	48.2	3.1

TABLE 16

EFFECT OF BLENDING PARTICLE SIZES

Alloy	Flow (%)	Setting Change (u/cm)	24-Hr Strength (psi x 1000)	Residual Mercury (weight %)
Mixture A	1.0	- 2.0	48.0	48.1
Mixture B	1.7	19.25	51.5	32.3
Mixture C	1.6	9.0	48.2	37.6
Mixture D	1.3	7.5	45.8	28.8
Size Range 4	0.9	- 2.75	50.8	48.5
Size Range 6	2.2	23.75	43.7	41.4
Size Range 8	Broke	200.0	11.2	40.1

TABLE 17

EFFECT OF VARYING TRITURATION TIME

Alloy	Trituration Time	Flow (%)	Setting Change (u/cm)	24-Hr Strength (psi x 1000)	Mercury Content (%)
Size Range 4	Recommended	0.9	- 2.75	50.8	48.5
	3 x Rec.	0.9	- 6.0	48.4	48.5
Control C	Recommended	3.0	- 4.75	41.4	48.2
	3 x Rec.	5.3	-19.5	42.4	52.5

TABLE 18

EFFECT OF VARYING MERCURY ALLOY RATIO

Alloy	Hg/Alloy Ratio	Flow (%)	Setting Change (u/cm)	24-Hr Strength (psi x 1000)	Mercury Content (%)
Size Range 4	1:1	0.9	-3.5	49.7	47.0
	8:5	0.9	-2.75	50.8	48.5
	4:1	1.2	-1.0	48.0	47.9
Control C	1:1	2.5*	-6.0*	38.7*	48.4*
	8:5	3.0	-4.75	41.4	48.2
	4:1	2.0	2.0*	42.1*	50.1*

*Data from Sweeney and Burns [14].

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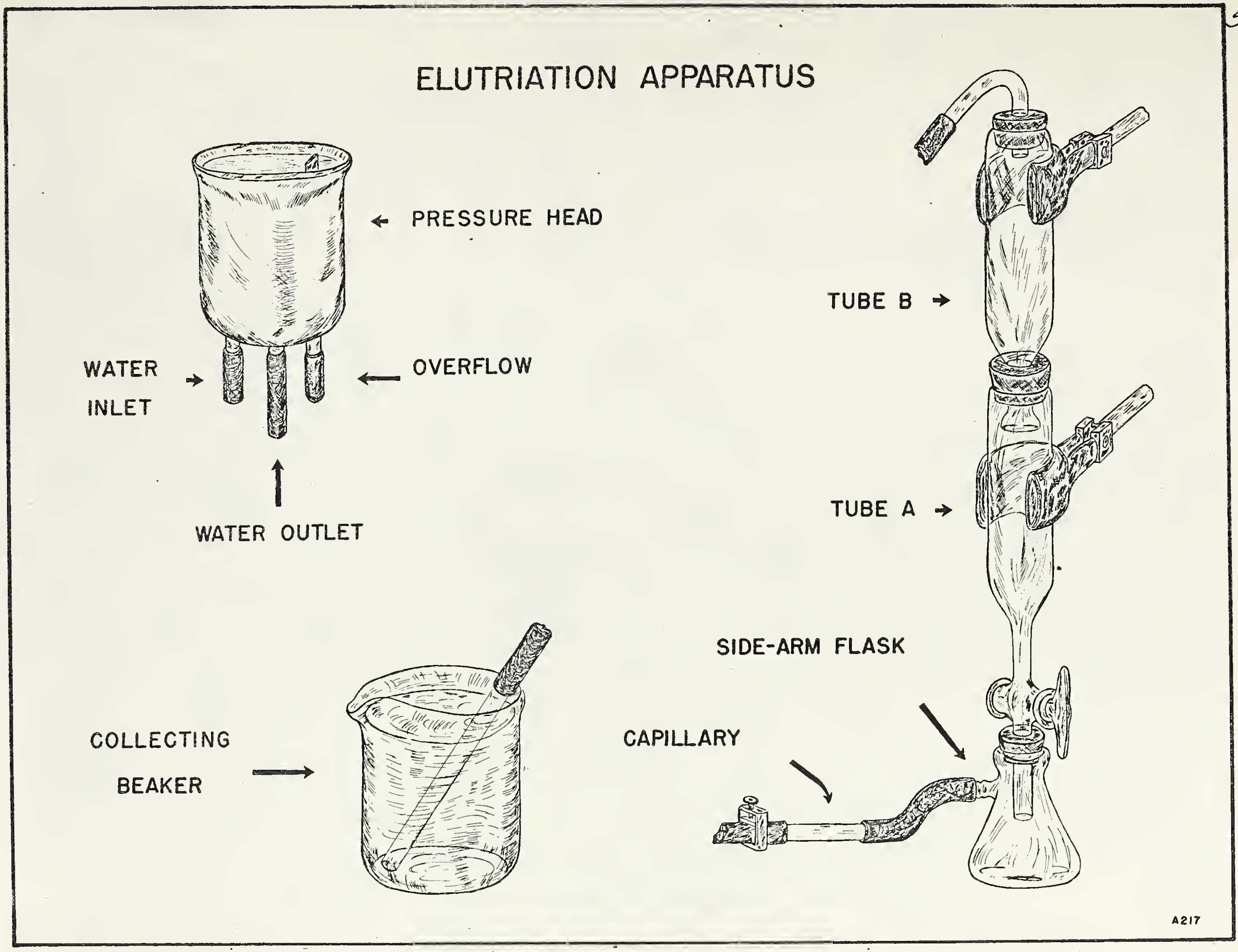

Figure 1. Drawing of Elutriation Apparatus.

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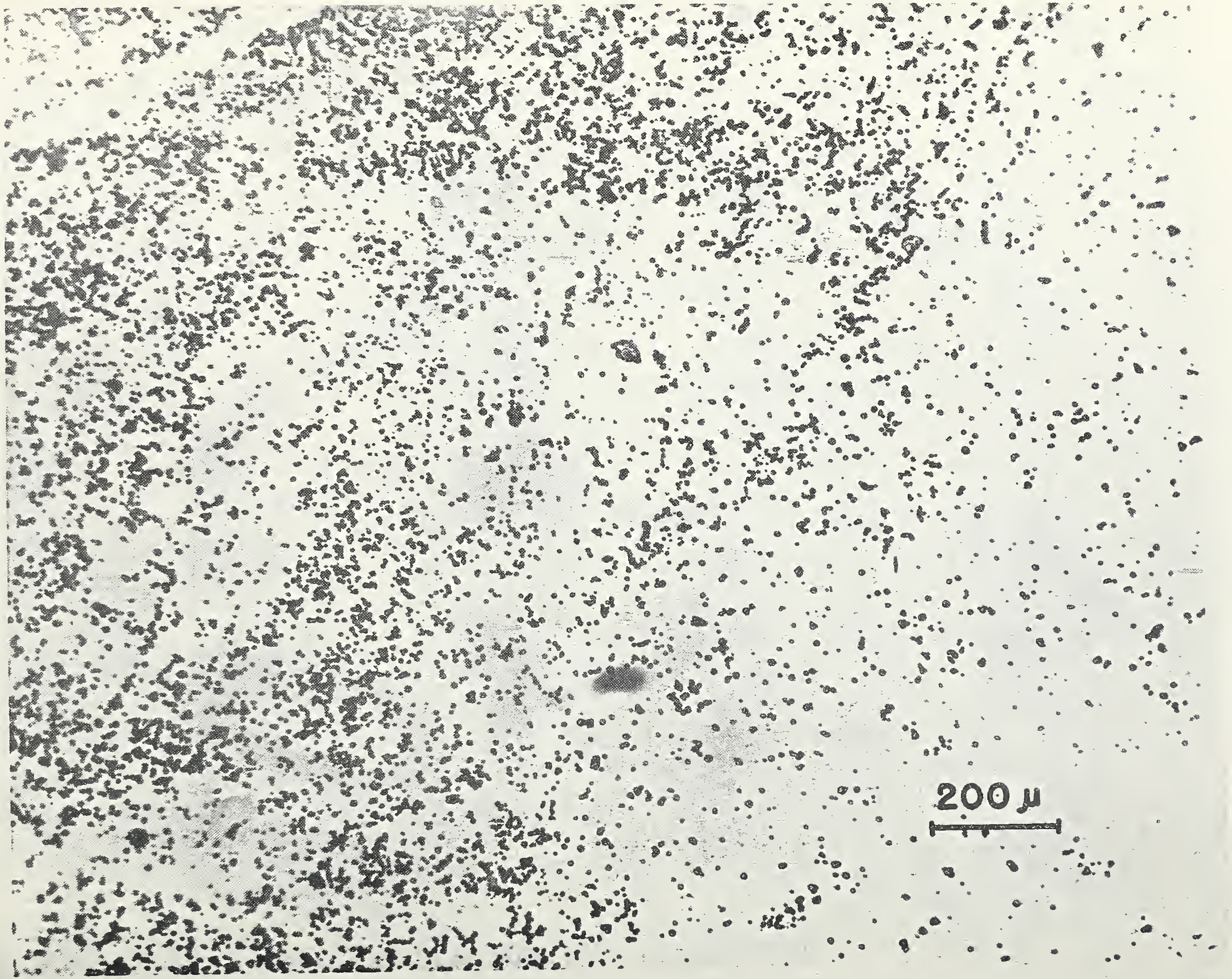

Figure 2. Spherical Alloy Particles of Size Range 1.

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Figure 3. Spherical Alloy Particles of Size Range 5.

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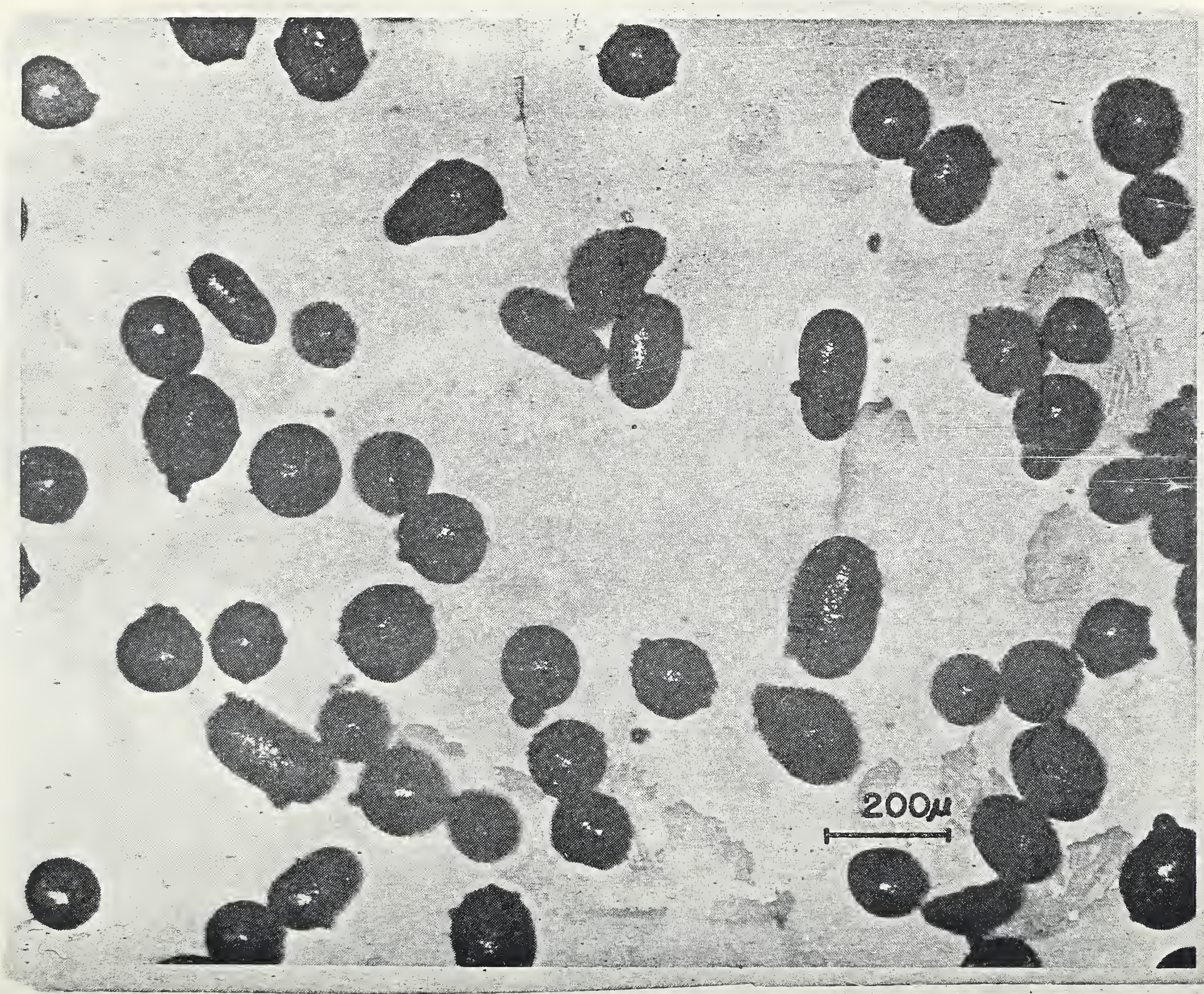

Figure 4. Spherical Alloy Particles of Size Range 8.

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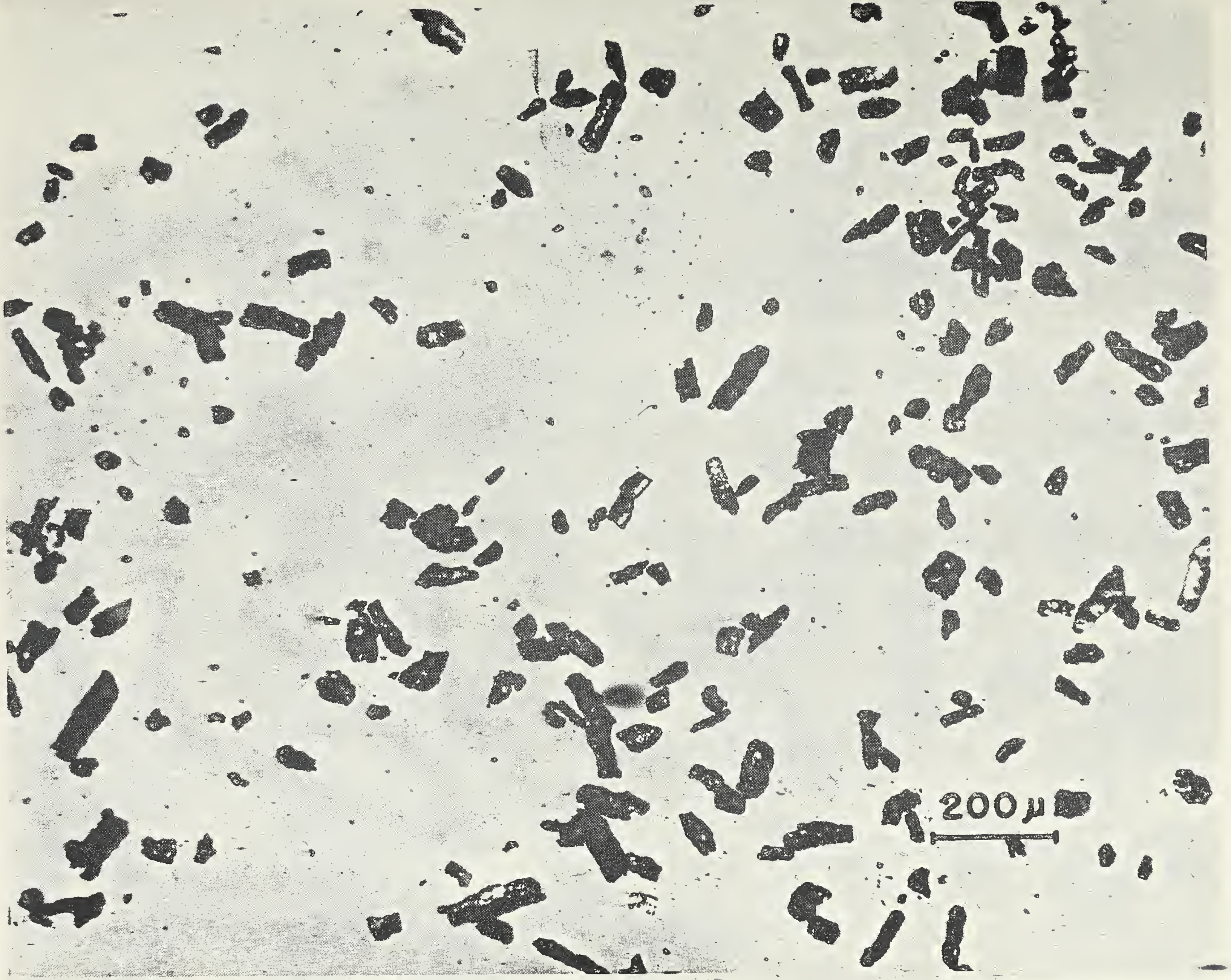

Figure 5. Fine-cut Dental Alloy Filings (Control B).

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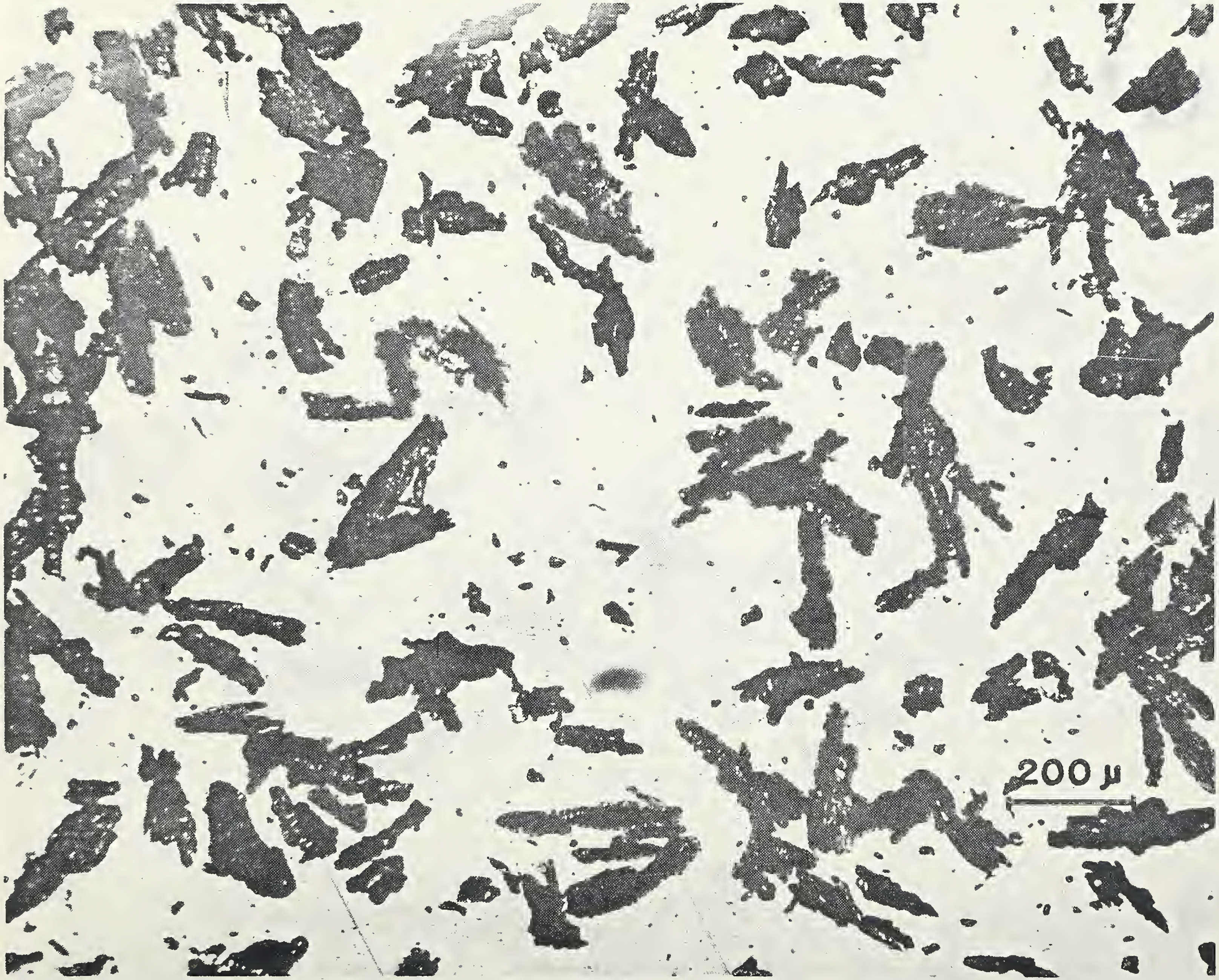

Figure 6. Coarse-Cut Dental Alloy Filings (Control C).

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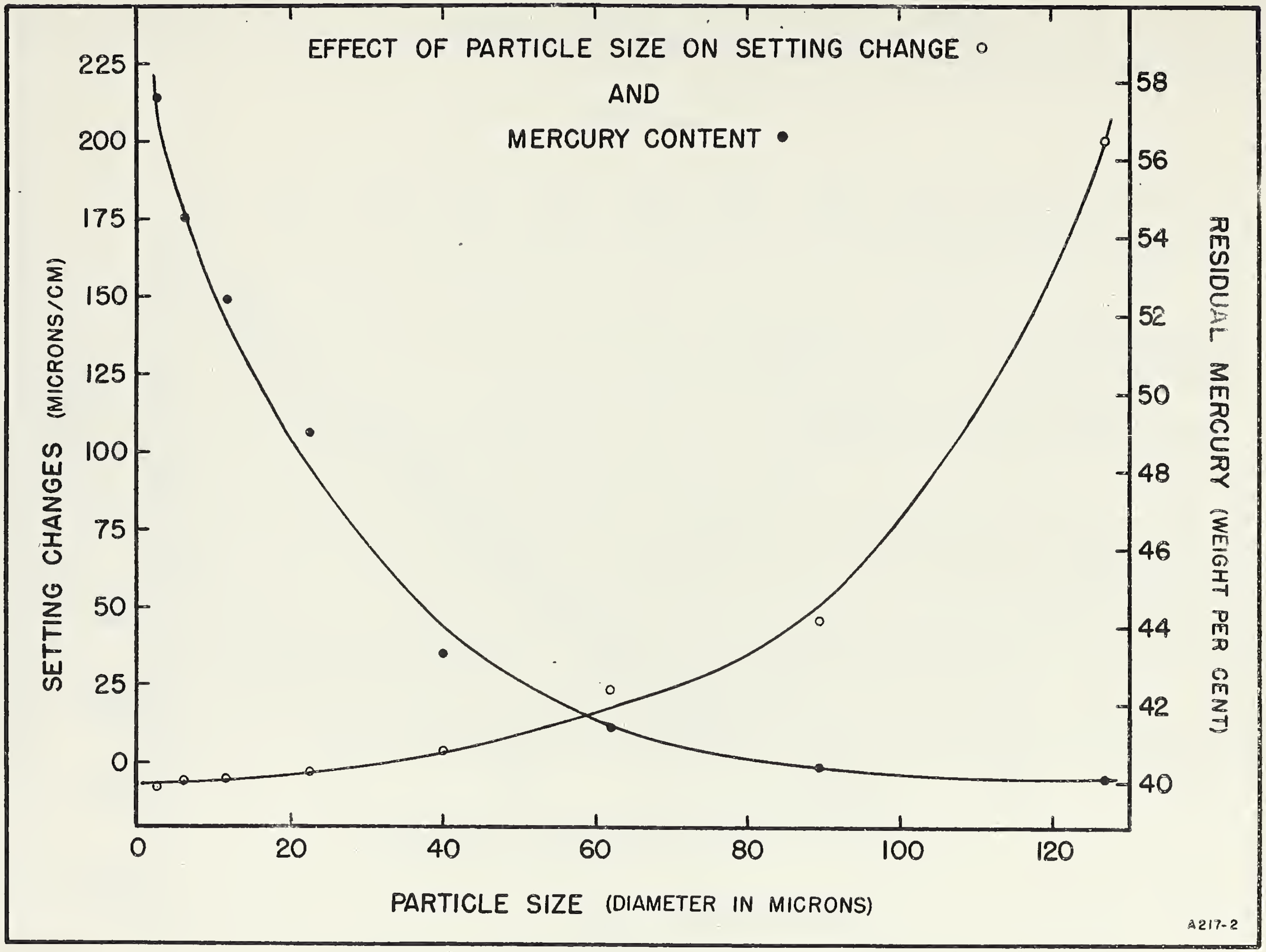

Figure 7. Effect of Particle Size on Setting Change and Mercury Content.

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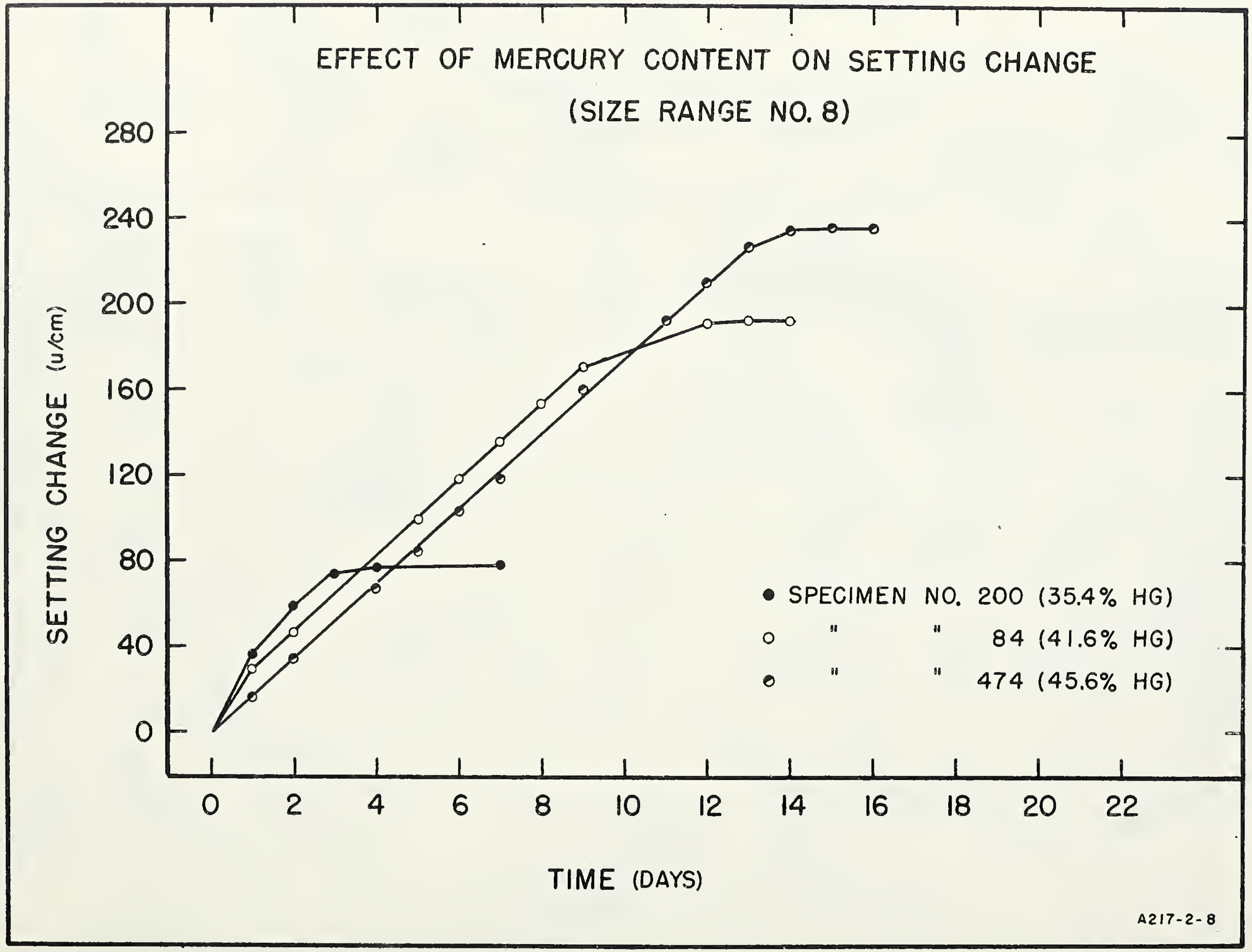

Figure 8. Effect of Mercury Content on Setting Change.

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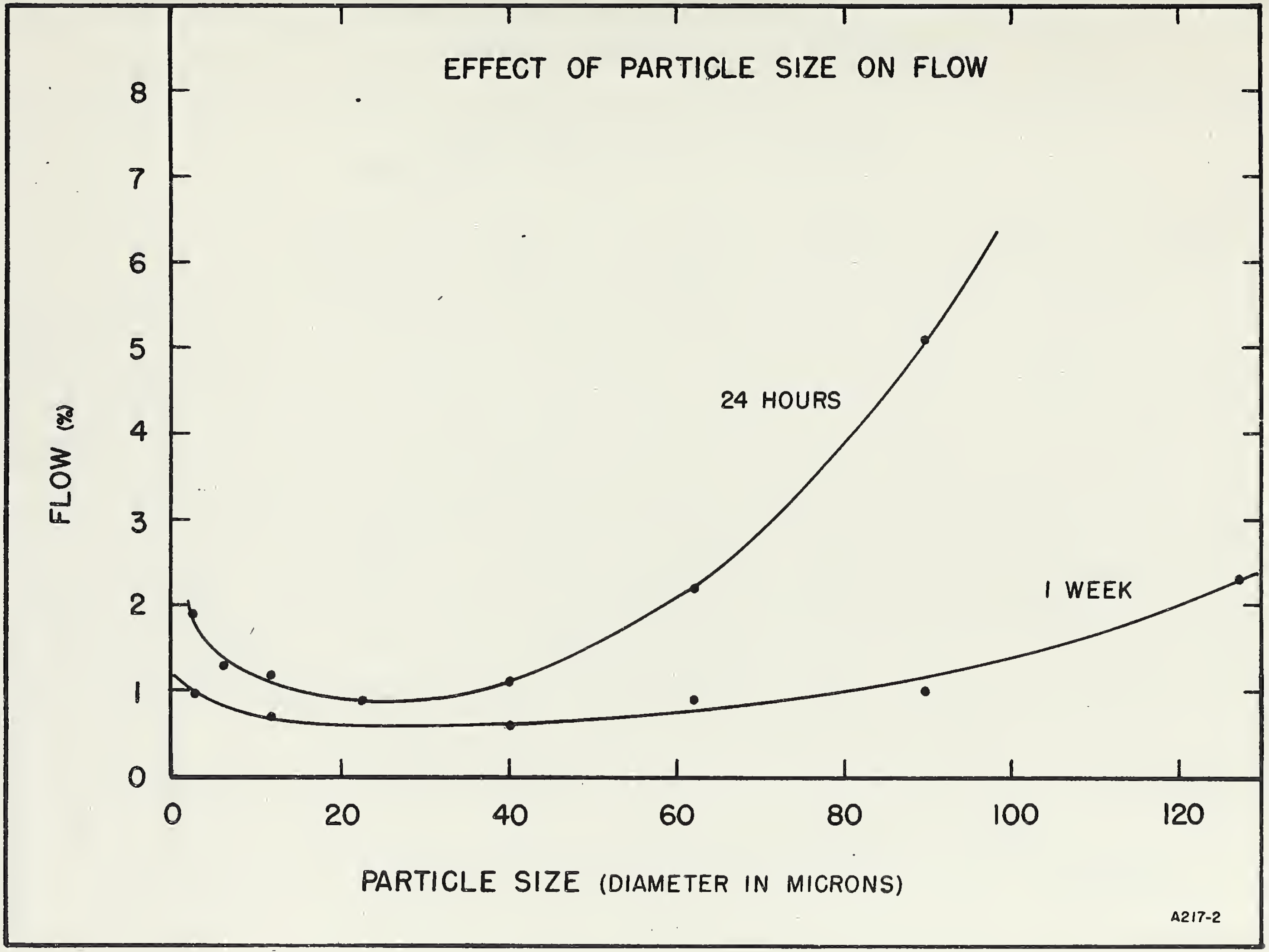

Figure 9. Effect of Particle Size on Flow.

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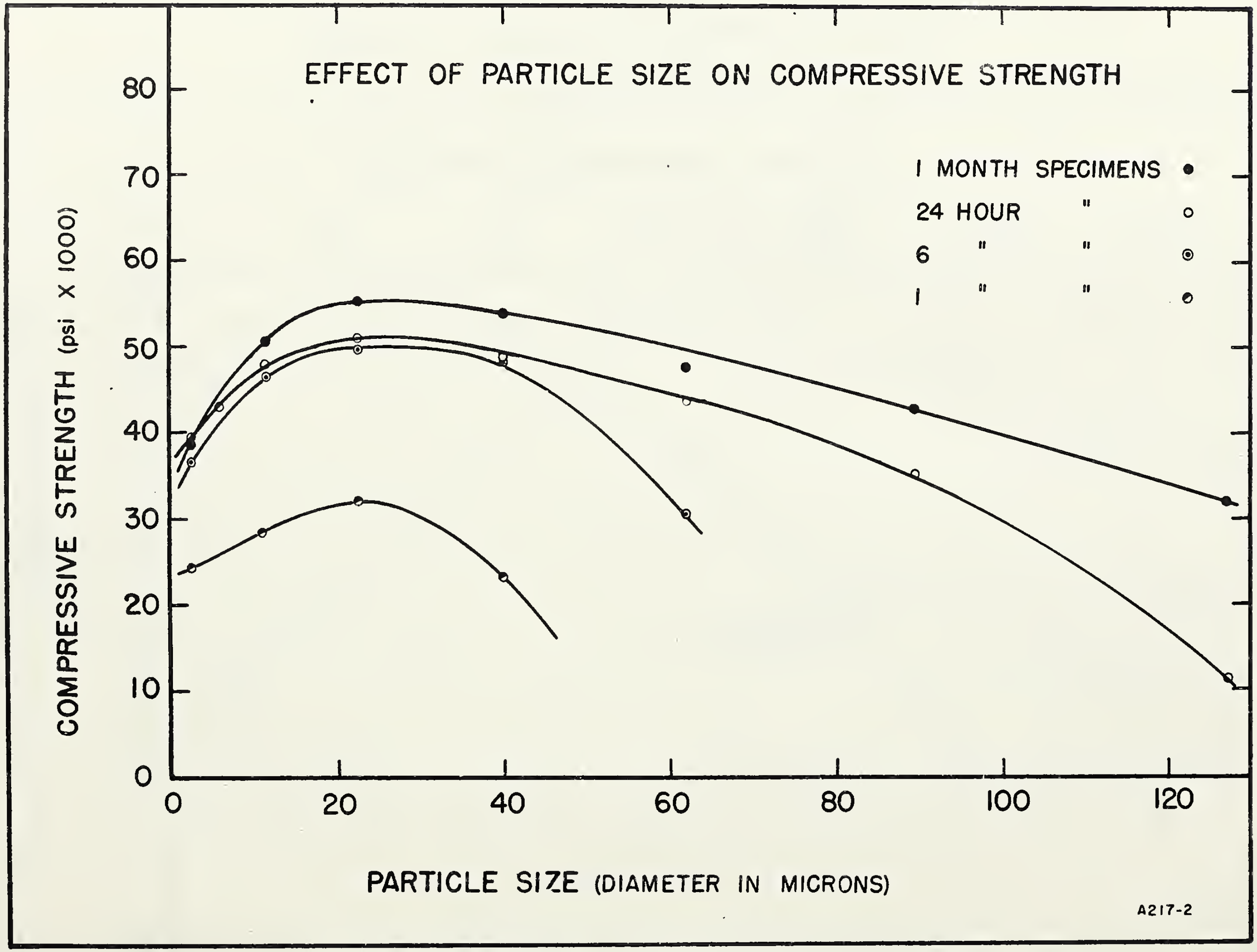

Figure 10. Effect of Particle Size on Compressive Strength.

U. S. DEPARTMENT OF COMMERCE
Luther H. Hodges, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards at its major laboratories in Washington, D.C., and Boulder, Colorado, is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section carries out specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant publications, appears on the inside of the front cover.

WASHINGTON, D.C.

Electricity. Resistance and Reactance. Electrochemistry. Electrical Instruments. Magnetic Measurements. Dielectrics.

Metrology. Photometry and Colorimetry. Refractometry. Photographic Research. Length. Engineering Metrology. Mass and Scale. Volumetry and Densimetry.

Heat. Temperature Physics. Heat Measurements. Cryogenic Physics. Equation of State. Statistical Physics.

Radiation Physics. X-ray. Radioactivity. Radiation Theory. High Energy Radiation. Radiological Equipment. Nucleonic Instrumentation. Neutron Physics.

Analytical and Inorganic Chemistry. Pure Substances. Spectrochemistry. Solution Chemistry. Standard Reference Materials. Applied Analytical Research.

Mechanics. Sound. Pressure and Vacuum. Fluid Mechanics. Engineering Mechanics. Rheology. Combustion Controls.

Organic and Fibrous Materials. Rubber. Textiles. Paper. Leather. Testing and Specifications. Polymer Structure. Plastics. Dental Research.

Metallurgy. Thermal Metallurgy. Chemical Metallurgy. Mechanical Metallurgy. Corrosion. Metal Physics. Electrolysis and Metal Deposition.

Mineral Products. Engineering Ceramics. Glass. Refractories. Enameled Metals. Crystal Growth. Physical Properties. Constitution and Microstructure.

Building Research. Structural Engineering. Fire Research. Mechanical Systems. Organic Building Materials. Codes and Safety Standards. Heat Transfer. Inorganic Building Materials.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering. Mathematical Physics. Operations Research.

Data Processing Systems. Components and Techniques. Digital Circuitry. Digital Systems. Analog Systems. Applications Engineering.

Atomic Physics. Spectroscopy. Infrared Spectroscopy. Solid State Physics. Electron Physics. Atomic Physics.

Instrumentation. Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

Physical Chemistry. Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Molecular Kinetics. Mass Spectrometry.

Office of Weights and Measures.

BOULDER, COLO.

Cryogenic Engineering. Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

Ionosphere Research and Propagation. Low Frequency and Very Low Frequency Research. Ionosphere Research. Prediction Services. Sun-Earth Relationships. Field Engineering. Radio Warning Services.

Radio Propagation Engineering. Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

Radio Standards. High Frequency Electrical Standards. Radio Broadcast Service. Radio and Microwave Materials. Atomic Frequency and Time Interval Standards. Electronic Calibration Center. Millimeter-Wave Research. Microwave Circuit Standards.

Radio Systems. High Frequency and Very High Frequency Research. Modulation Research. Antenna Research. Navigation Systems.

Upper Atmosphere and Space Physics. Upper Atmosphere and Plasma Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.


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