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BJORKSTEN RESEARCH LABORATORIES, INC.

Madison, Wisconsin

Contract DA 18-108-AMC-171(A)

U.S. ARMY EDGEWOOD ARSENAL
EDGEWOOD ARSENAL, MARYLAND

FINAL REPORT

DEVELOPMENT OF ANTI-AGGLOMERATION METHODS FOR DRY POWDERS

Prepared by

Luther L. Yaeger
Risto P. Lappala
Joel N. Lipscomb

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FOREWORD

This is the Final Report on work performed under Contract DA 18-108-AMC-171(A). It describes the work performed during the period July 1, 1963 through February 28, 1964.

The research was performed at Bjorksten Research Laboratories, Inc., Texas Division, Houston, Texas, with Mr. Luther L. Yaeger as Project Leader and Messrs. Risto P. Lappala and Joel N. Lipscomb as contributing technical personnel. Administratively the project was managed from Bjorksten Research Laboratories, Inc., Madison, Wisconsin.

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OBJECTIVE

To investigate a new technique capable of forming a non-agglomerating powder of presized solid chemical agents and simulants as follows:

(1) Para-aminobenzoic acid;

(2) Saccharin;

(3) Resorcinol;

(4) CN;

(5) BZ.

Work on the last two classified agents to be performed at Edgewood Arsenal, Maryland, only in the event that the successful processing of the first three can be accomplished.

Processing to consist of spray-drying the specified materials.

The materials to be spray-dried to have at least 90% of the particles, by weight, in the size range of 0.75 to 10 microns in diameter.

Study the properties of these compounds to determine:

(1) Which antistatic additives will be incorporated in the material;

(2) The best qualified solvents for the spray-drying process; and

(3) The flow characteristics of these powdered materials before processing as well as after incorporation of the antistatic additive.

Comparative tests to be made of each compound to determine if the flow characteristics have been improved by the incorporation of the antistatic additives.

SUMMARY

Saccharin (ortho-benzoic sulfimide) and para-aminobenzoic acid (PABA) were successfully spray dried to provide particles less than 10 microns in diameter from solvent systems of 70% acetone, 30% carbon tetrachloride (by volume). One hundred grams each of PABA and saccharin with and without antistatic agent added to solvent solution were prepared in a spray-dried form with 90% of the particles in the size range of 0.75 to 10 microns in diameter.

Solutions of resorcinol were also dried from appropriate solvent systems but the dried product was found to be too hygroscopic to handle under ordinary atmospheric conditions.

A spray dryer was designed and constructed with sufficient adaptability and simplicity to permit the evaluation of different atomization devices, hot gas-spray mixing procedure, powder collection and separation equipment. Rapid disassembly for thorough cleaning was a built-in feature.

An electrostatic precipitator was devised to collect a large portion of the minor particles not retained by the cyclone separator.

An air separator for further classifying the collected particles was built and evaluated.

Among the atomization devices used were: spinning disc, centrifugal pressure nozzle, impingement and pneumatic. The pneumatic nozzle proved to be the most successful of the devices.

DISCUSSION

A. The Theory of Spray Drying

In discussing the theory of spray drying and the advantages and disadvantages of various types of atomization devices, we quote widely from "Atomization and Spray Drying," W. R. Marshall, Jr., published by the American Institute of Chemical Engineers, Library of Congress Catalog No. 54-12941. Direct quotations are so noted.

Spray drying is accomplished by atomization of a highly dispersed liquid state in a high temperature gas zone, followed by rapid evaporation and drying of the droplets. The three equally important operations involved are (1) atomization, (2) spray-gas mixing, and (3) drying of liquid drops, followed by the removal and collection of dry product. As the three processes occur virtually simultaneously, the drying operation can be controlled by the operation which requires the greatest time to perform. Should the mixing of the gas and the spray require considerably greater time than the evaporation process, some of the advantages of a short drying time are lost. With nonuniform atomization, the large drops will control the overall drying time and the small particles may be subject to overheating because of their more rapid evaporation rates and shorter drying times. 1/ An extensive bibliography on atomization compiled by DeJuhasz 2/ is evidence of the considerable literature on the subject.

Spray drying has several principle advantages over other drying methods. Certain property and quality values of the product may be controlled and varied: A particle shape approximating a sphere, usually hollow, sometimes solid, with a density and particle size which may be varied in a given range is obtainable; it is also possible to preserve the quality of the product which may otherwise be destroyed by overheating.

The four different methods of atomizing liquids are:

1. Atomization by means of centrifugal or swirl type pressure nozzles,

2. Pneumatic or gas-stream atomization in which a jet of liquid is disintegrated by a high velocity gas stream,

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1/ Marshall, W. R., Jr., "Atomization and Spray Drying."
2/ DeJuhasz, J. J. and Meyers, W. E., "Bibliography of Sprays," The Texas Co., New York (1953).

3. Spinning disc atomization wherein a liquid is broken up by discharging it as high velocity from the periphery of a rapidly rotating disc,

4. Impingement atomization wherein two liquid jets impinge or a single jet impinges on a solid surface.

Atomization by means of supersonic and subsonic vibrations has been considered as well as atomization by high voltage electrical energy.

The degree to which atomization increases the surface area for heat and mass transfer is best understood by studying the curve shown in Figure 1. 3/ The surface area created is inversely proportional to the diameter of the atomized drop. Figure 1 is a plot of the ratio of new to original surface for various fractional values of the diameter of the original liquid mass in spherical form.

B. Consideration of Purchased Equipment Vs. In-House Assembly

Investigation of the various types and sizes of experimental spray dryers available commercially, both from the consideration of initial cost and the high cost of making even relatively simple changes or adaptations to existing equipment, influenced our decision to assemble at minimum cost from easily obtainable standard parts a versatile, easily adaptable spray dryer which would allow the use of cocurrent or countercurrent air flow or the substitution of any of the four methods of atomization.

The fact that commercial spray dryers are relatively inflexible clearly indicated the need for designing and fabricating our own apparatus which would be readily convertible. For example, a commercial spray dryer designed for fine atomization is generally incapable of producing a coarse product, and a spray dryer designed for a thermally stable material may not be at all suited to a temperature-sensitive product. A study of the droplet size distribution obtained by the four methods of atomization shows very little information is available in the less-than-ten-micron range, and that probably the greatest difficulties lie in measuring the less-than-ten-micron range.

The initial cost of a typical laboratory size spray dryer comercially obtainable was found to be about $5,000 for the basic dryer. A centrifugal atomizer for this dryer was quoted at $800 with a price of $130 for each separate wheel to provide different droplet sizes.

3/ Marshall, op. cit.

C. Design and Construction of Spray Drying Apparatus

1. Spray Drying Apparatus

The completely assembled spray drying apparatus ready for an experimental run is shown in Figure 2.

A totally enclosed room, Figure 3, was constructed by covering a wood framework of 2 x 4's with Type 55 Clear Griffolyn* fabric film. This provided a separate room which was sufficiently translucent to admit light and at the same time eliminate the presence of extraneous dust and foreign materials. A screen door with an additional two layers of outing flannel fastened over the screen filters out dust and at the same time allows a free passage of air.

2. Heater Shell

A heater shell was fabricated of 1808 stainless steel as shown in Figure 4. To provide the large heating area, #303 stainless steel strips were bent as shown in the sketch and bolted to "Chromalox" strip heaters and the interior of the shell as shown in the cross-section detail, Figure 5. One end of the heater was necked down to fit three-inch ducting. The other end of the shell was made to allow the insertion of a shop vacuum cleaner bag, as shown, which serves as an easily replaceable, efficient and low cost air filter.

To increase the efficiency of the heater, the shell was then covered with a one-inch blanket of fiber glass and then enclosed in a shell of 0.175-inch aluminum sheeting to prevent tearing of the fiber glass mat.

3. Drying Chamber Construction Details

A number of types or kinds of shells were considered for the drying chamber. The prices ranged from approximately $100 for a stainless steel drum to $350 for a fabricated shell of stainless steel. For $15, two spun aluminum shells with the dimensions shown in the sketch were obtained from a metal spinner.

The bottom of the aluminum shell was provided with a hole 10-1/2 inches in diameter. A 10-3/4-inch "Mirro" aluminum ring salad mold was fastened as shown in the photograph to the shell with mirror clips and sheet metal screws.

A 60° fabricated stainless steel cone was fitted with a tubulature at one end and a transparent viewing window on one side, Figure 6.

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*T. M. Griffolyn Co., 6813 Dixie Drive, Houston, Texas

General Electric RTV-11 silicone gaskets were poured in place on all of the mating surfaces of the components. RTV-11 was used to provide dust-impervious gaskets that would withstand the relatively high temperatures encountered during the spray drying operation, Figures 7 and 8.

Screw clamps with fastening dogs were attached to hold the drying chamber in place and allow for ease of removal.

4. Spray Forming or Atomization Apparatus

Initial trials made by using a centrifugal spinning disc type of atomizer, Figures 9 and 10, indicated that the particle size obtainable at a speed of 8,000 rpm was not sufficiently small.

A spinning disc type of atomizer, while providing trouble-free and efficient atomization, did not give sufficient breakup of the solution for the small particle desired.

A bronze gear pump with connections of copper tubing providing pressure to a centrifugal pressure nozzle corroded sufficiently to clog the system. The bronze pump and copper tubing was replaced with a system using noncorrosive materials and no moving parts to provide the necessary pressure.

Figure 11 illustrates a welded stainless steel vessel with 1/4-inch Swagelok fittings silver brazed in place. The copper fittings were replaced with either stainless steel or steel and the copper tubing replaced by polyethylene tubing with sufficient wall thickness to withstand the pressure.

For operation, the stainless steel vessel was filled about half full of the solvent solution of the material to be dried. The filling vent was then plugged and air pressure applied to the space above the liquid to force the liquid out through the nozzle as required. To minimize clogging of the very fine slit which provides the tangential flow of the liquid for the centrifugal spray nozzle, a simple fuel filter, such as used on internal combustion engines, was provided.

Excessive corrosion of the zinc diecasting in the fuel filter by materials being dried subsequently made it necessary to dispense with the fuel filter. The solutions were carefully filtered prior to use to minimize clogging.

Trials proved it best to introduce the atomized solution from the side rather than from the top of the drying chamber, either cocurrent or countercurrent to the flow of hot drying gases. Figure 12 shows the pneumatic nozzle in place while operating. To allow for control and replication of drying conditions, a flowmeter was provided in the liquid line, Figure 13.

Attempts to preheat the solvent solution before atomization caused a rapid buildup of material at the nozzle and frequent clogging. Variations of the "Bete" impingement nozzle, Figure 14a, in which atomization or liquid attenuation is achieved by directing a jet at high velocity against a solid target were tried and found difficult to control.

Atomization in which a compressible fluid such as air or steam is used to disintegrate a liquid jet is called pneumatic atomization. Application of this method of spray drying has been restricted for the most part to experimental spray dryers and special atomization devices for creating test sprays. Pneumatic atomizers are more suitable than the other types of atomizers for producing very fine sprays but large amounts of energy are required per unit of surface area created.

A pneumatic spray nozzle designated as 1A (fluid nozzle-1650, air nozzle-69) was obtained from the Spraying Systems Co., Bellwood, Illinois and found the most satisfactory for providing fine particles in spite of its very low capacity. A sketch of the cross-section of such a nozzle is shown in Figure 14b.

5. Powder Separation Section

From Western Precipitation Corporation, Los Angeles, California, we obtained a standard Multiclone Type 61F, Size 1, cyclone type separator, Figure 15. A fitting was fastened to the bottom of the separator to allow for use of a standard one-quart wide mouth Mason jar for collection of the dried product, Figure 16.

An electrostatic sampler (Model F) made by Mine Safety Appliances was loaned by the sponsoring agency to collect the fines not retained in the cyclone separator. The sampler proved very efficient for small volumes of air but did not have sufficient capacity for use with our production.

An electrostatic precipitator of larger capacity was assembled in the laboratory and is shown in cross-section in Figure 17.

Although the electrostatic precipitator removed considerable fines from the air stream, a certain percentage still passed out through the exhaust. Recovered powder yields averaged 30 to 40% of the input charge.

For the collection of extremely fine particles the carbon black industry, for instance, customarily uses bag filters of glass fabric and other industries use bag filters of other suitable fabrics. An attempt was made to recover the fines lost through the exhaust in the laboratory spray dryer by using bag filters of cotton twill and glass fabric. These bags proved successful only for a short time as the bags soon clogged and drastically reduced the porosity and air flow thus affecting the drying characteristics of the dryer.

6. Blower and Miscellaneous Control Parts

A high pressure #70650 blower delivering about 290 cfm was obtained from the W. W. Grainger Company and connected to the dust collection or powder separation section by a fabricated sheet metal adaptor. Four-inch aluminum ducting was provided to the electrostatic precipitator and from the precipitator as an exhaust to the outside.

Suitable electrical outlets, plugs and switches were provided to allow for independent operation of any of the electrical components of the system. Dial thermometers were inserted into the ducting system for the measurement of pertinent temperatures.

A Dwyer Flex-Tube Manometer #25 was attached to the blower system to give an indication of the air pressure and air flow involved.

A complete list of parts used in spray drying equipment is provided in the appendix.

D. Control Test on Commercial Equipment with the Experimental Material

Arrangements were made to run control tests on a Nerco-Niro portable spray dryer.

This spray dryer uses a centrifugal atomizer with a turbine wheel driven at 30,000 rpm by an air turbine (this atomizer alone was quoted at a price of $930 with extra turbine wheels quoted at $130 each). The complete dryer was quoted at $4,700 with any accessories or changes extra.

A 20% solution of resorcinol and water was used for the initial trial. In all cases the maximum speed of 30,000 rpm was used on the atomizer with a feed rate of approximately 10 cc per minute. The first run appeared to be discolored; consequently, the inlet and outlet temperatures were lowered. At the lower inlet temperature on the second run the material had to be fed in at an extremely low rate and still appeared to be discolored although not as much as the initial run. As a result, two subsequent runs were made using a solvent system consisting of 60% methyl alcohol and 40% water to provide a 20% solution of resorcinol. This allowed further reductions in the inlet temperature and obviated the discoloration occurring when material was dried from a water solution only.

At a five-day meeting of the American Institute of Chemical Engineers held in Houston in December 1963, Mr. Robert R. Freeman of Biochemical Processes, Inc., New York, presented a paper describing a specialized pneumatic-liquid atomizing nozzle which has been successfully used for spray drying of heat-sensitive materials in a very fine particle size. As a result of a discussion with Mr. Freeman, arrangements were made to have a small quantity of PABA dried in their

experimental apparatus for comparison with materials dried in the experimental apparatus. Microscopic examination of the material dried in the apparatus by Biochemical Processes indicates that the average particle size is not sufficiently small to justify further investigation of this nozzle.

E. Microscopic Examination of the Spray Dried Materials

Spray drying produces a product consisting of approximately spherical particles which are more or less hollow, depending on the material and on certain operating variables. Hollow particles are the rule, solid particles the exception. Duffie and Marshall 4/ studied and photographed a large variety of spray-dried materials and in all cases found a structure of spherical shape. The spherical shapes are often deformed or shriveled.

Figures 18-26 are photomicrographs of some typical spray-dried materials and are explained by the legends and in the text.

A micrometer stage scale, Figure 26, divided into units of 0.01 millimeters was photographed at the same magnification as used for the spray-dried particles and may be used to obtain some idea of the relative size of the particles.

F. Particle Size Determination

A simple, rapid method for preparing slides of the various spray-dried powders for microscopic examination was developed.

A thin film of a diluted solution of pressure sensitive adhesive, which dries to a clear film, was spread on a microscope slide using a blood-smear technique. The film was then dried to a permanently tacky condition and a small amount of the dried powder was blown across the tacky surface. The slide was gently tapped and the excess powder removed.

By means of the mirror and the Abbe condenser, it is possible to project into the plane of the object lying upon the stage, the image of the scale whose value has been ascertained. Both scale and object are magnified together and it, therefore, follows that no matter what may be the combination of the objective and ocular employed the value of the divisions of the scale image will remain unchanged, provided that the distance of the scale image will remain unchanged, provided that the distance of the scale from the condenser is not altered. This method was thoroughly tested out by Ives in 1903 "Journal of the Franklin Institute" 154, 73, and described in "Handbook of Chemical Microscopy," Chamot and Mason, Volume I, John Wiley & Sons under a chapter on "Microscopic Measurements."

4/ Duffie, J. A., Marshall, W. R., Jr., Chem. Eng. Prog. 49, 417, 480 (1953).

By this technique a large number of fields can be rapidly evaluated for particle size distribution. This is especially true if the projected scale image is calibrated by means of a stage micrometer to read in divisions of 10 microns.

A number of methods are known for determining the size of particles. Optical methods depend on light scattering or transmission and are used mainly for determining the sizes of particles in aerosols. Another method for determining the mean value for the diameters of the particles in materials is based on permeability or the rate of flow of a fluid or gas through a bed of particles.

The Fisher Sub-sieve Sizer manufactured by the Fisher Scientific Company embodies the principles of a particle size measuring apparatus assembled and used by Ernest L. Gooden and Charles N. Smith. 5/ This instrument has been designed for simplicity of the measurement of particle size through standardization of conditions.

Preliminary determinations on three samples of PABA indicated that this instrument would save considerable time in determinations of average particle size of powders.

A discussion of the various sizing methods has recently been published. 6/

G. Solvent Systems

Optimum solvent systems and operating conditions to provide the most efficient spray drying consistent with particle size and uniformity were empirically determined and 100-gram samples of PABA and saccharin were prepared using no antistatic agents and an antistatic agent designated as Cananac SN for each of the materials.

H. Powder Flowability

Two separate protective colloids were used under different conditions in an attempt to minimize crystal formation in the spray-dried materials. They are designated as PVP K-30 (polyvinylpyrrolidone) and Gelva V-7, a polyvinyl acetate. Selection of the proper solvent system and drying conditions made the use of these materials unnecessary.

5/ Gooden, Ernest L., and Smith, Charles M., Ind. Eng. Chem., Anal. Ed. 12, 479-482 (1940).

6/ Kaye, Brian, International Science and Technology 27, p. 40 (March 1964).

For a simple, rapid determination of the flow characteristic of the spray-dried powder, two 125 ml Erlenmeyer flasks were used. Equal quantities, on a volume basis, of the powders were placed in one of the flasks; another flask was inverted over the mouth of the powder containing flask and the two openings taped together. This apparatus is shown in use in Figures 27 and 28.

The taped together flasks were slowly inverted and the angle at which the most flowable powder ran into the lower flask was used as a criterion for the relative flowability of the other powders, i.e., the relative quantity of powder remaining in the upper flask for the other two when at the same angle gives a visible relationship as shown in Figures 27 and 28.

In the Statement of Work under "A, 2, c" was stated: "The flow characteristics of these powdered materials before processing shall be determined." Actually, very little comparison could be made between the original materials and the processed materials as resorcinol was supplied in rather large flakes whereas PABA was supplied in very fine, needlelike crystals which tend to mat together and do not flow. Saccharin, as supplied, has the appearance of aggregates of relatively large crystals which also tend to mat and not flow well. The powders shown in Figures 27 and 28 have all been spray dried. Untreated controls are not shown. No dried resorcinol is shown due to caking.

An attempt to control the uniformity and, perhaps, increase the density of the individual particle during spray drying was made by using very finely divided calcium aluminum silicate as a nucleating agent. ASP #602 with an average particle size of 0.8 microns supplied by the Minerals and Chemicals Corporation was used in the initial trials.

Five percent of ASP #602 when used in a solution of PABA using the dimpled spinning disc and the jet impingement technique provided complete drying and better uniformity of particle size. However, the overall particle size was larger than the desired 10 microns or less.

Using 5% of ASP #602 with either the centrifugal type or the pneumatic nozzle caused very rapid clogging in both cases.

I. Bulk Density

When a powder is permitted to fall under standardized conditions on a horizontal surface, the bulk density of the resulting material decreases with decreasing particle size. This technique has been reviewed by Rose, H. E., "The Measurement of Particle Size and Very Fine Powders," Constable, London, 1953. The powder is poured through a chute and allowed to fall into a cup of known volume. The excess powder is scraped off the top of the cup which is then weighed. The method is strictly empirical and must be calibrated for each type of powder used.

Rose has listed several disadvantages. Slight variations in technique affect the results: the different calibration curves required for each material; not all materials will flow through a chute in a satisfactory manner.

J. Air Classification of Powdered Materials

A simple, efficient apparatus was devised for the separation of particles in the desired size range and is shown in Figures 29 and 30.

The collected material was introduced through a hole in the top of the air classifier and the proper air flow determined empirically. A stream of air was introduced tangentially through the bottom of the collection system and the particles 10 microns or less in size were carried upward in the slowly moving air stream and collected on the undersurface of the filter paper as shown in Figure 30. A flowmeter was provided to allow accurate control of the air stream and thus achieved proper separation.

Subsequent microscopic examination of the particle size of the separated materials showed at least 90% or better of particles less than 10 microns in size.

CONCLUSIONS & RECOMMENDATIONS

The assembled apparatus proved satisfactory and sufficiently adaptable and flexible to allow for the large number of variations that were necessary to achieve the desired results. The final assembly of the apparatus was found to operate for long periods of time with very little attention and proved to be easily disassembled for cleaning which, in an investigation of this type, is of major importance. Operating instructions are included in the appendix.

A technique has been developed for spray drying PABA and saccharin to provide particles of 10 microns or less in diameter.

The technique when applied to spray drying resorcinol was effective but the hygroscopic nature of the very fine particles of resorcinol caused subsequent caking when exposed to the atmosphere.

An antistatic agent designated as Catanac SN markedly increased the flowability of both PABA and saccharin when introduced in the solvent system in the proportion of 1% of the materials to be spray dried.

Although a mean average particle diameter of less than 10 microns but not less than 0.75 microns as 90% of the total dried particles obtained, agglomeration of the discrete particles was not entirely prevented by addition of the antistatic agent that was used.

It is our recommendation that the spray dryer apparatus (with necessary modifications) be used to investigate an "encapsulating" procedure wherein active agents may be used to cover or "encapsulate" carrier particles of the desired particle size.

Such a procedure would consist, essentially, of spraying an active material into the dryer in a suitable solvent either cocurrent or countercurrent to an inert carrier material of the desired particle size and the subsequent collection of the coated particles.

An example of a carrier material that might be suitable is "Acrawax C" manufactured by Glyco Products of Williamsport, Pennsylvania.

The Glyco literature states that the material may be obtained in a particle size having an average diameter of five microns. Some of the powdered material on hand in the laboratory does not show any agglomeration of the particles when examined under the microscope and disperses readily in air currents.

With the different atomizing devices supplied with the dryer, the equipment can obviously be used for drying many other materials with a wide range of particle sizes.

APPENDIX

OPERATING INSTRUCTIONS

Prior to shipment of the complete spray dryer equipment to Edgewood Arsenal, the various components will be color coded or numbered to facilitate assembly. Reference to the illustrations in the technical report will assist in determining relative positions of the component parts.

After the equipment has been assembled and the appropriate electrical, exhaust, air and fluid connections have been made, the collecting receiver can be a one-quart or one-half gallon wide mouth Mason jar fastened into place below the cyclone separator as shown in Figure 16 and the blower turned on.

The heater switches are thrown into the "On" position and the system allowed to reach the desired temperature for operation.

If the centrifugal disc atomizer is used, it is installed through the top of the dryer shell as shown in the diagram and photographs. The solvent solution of the material to be dried is then fed in at the desired rate to provide the particle size required.

When using the pneumatic type nozzle, the nozzle is installed to feed from the side as shown in the detailed operating photograph, Figure 12. The air valve is then opened wide and air at 50 psi first turned on. The fluid flow rate is adjusted to the desired rate of feeding by observing the flowrater and use of the Fisher and Porter calibration curve, Figure 31.

If an electrostatic precipitator is to be used, it is connected between the blower outlet and the exhaust duct. At the end of a run the equipment may be first brushed out with an ordinary clean paint brush and then disassembled and thoroughly cleaned out with an appropriate solvent or cleansing method before reassembly.

PARTS LIST - SPRAY DRYING EQUIPMENT

A. Heater

2 - 120V, 750W Chromalox strip heaters
4 - 240V, 750W Chromalox strip heaters
12 - Strips #303 stainless steel strips (see drawing)
12 - 1/4" x 20 x 3/4" stove bolts
1 - stainless steel #1808 heater shell (drawing)
Wiring, #10 copper
4 - 240V switches
2 - 120V switches
1 - Sears Roebuck #9A 16967 protector bag

B. Drying Chamber

1 - 16" x 22" spun aluminum filter pot
1 - 60° stainless steel cone
1 - 10-3/4" Mirro aluminum ring salad mold
1 - Sears #98A 1515 adjustable elbow
4 - screws, clamp and dogs

C. Atomization Apparatus

1 - Grainger #2M037 motor
1 - 400-6 "Swagelok" steel union
1 - fabricated stainless steel shaft and turbine wheel
1 - 2" x 8" stainless steel tube
1 - 1/4" ball bearing
1 - nylon bushing for bearing
1 - 1/4" x 10" copper tubing
1 - #1/4 LN-60 centrifugal nozzle (Spraying Systems)
1 - #1A J pneumatic nozzle (Spraying Systems)
Air and fluid lines, air valve
Stainless steel container
1 - F&P Flowrator No. OIN-150-A
1 - # Bete #UP-1 nozzle

D. Powder Separation Section

1 - Multicone standard 61F size 1
1 - galvanized adaptor
1 - adaptor for collection jar

E. Blower and Miscellaneous

Sears #98A 1515 adjustable al. elbows
Sears #98A 1514 al. 3" ducting
1 - Grainger #2C647 blower
1 - Dwyer Flex-Tube Manometer #25
1 - pipe and wood fabricated stand
2 - Weston dial thermometers Model 2281
Various adaptors for ducting were fabricated from materials at hand
Electrical wiring, plugs and outlets as needed.

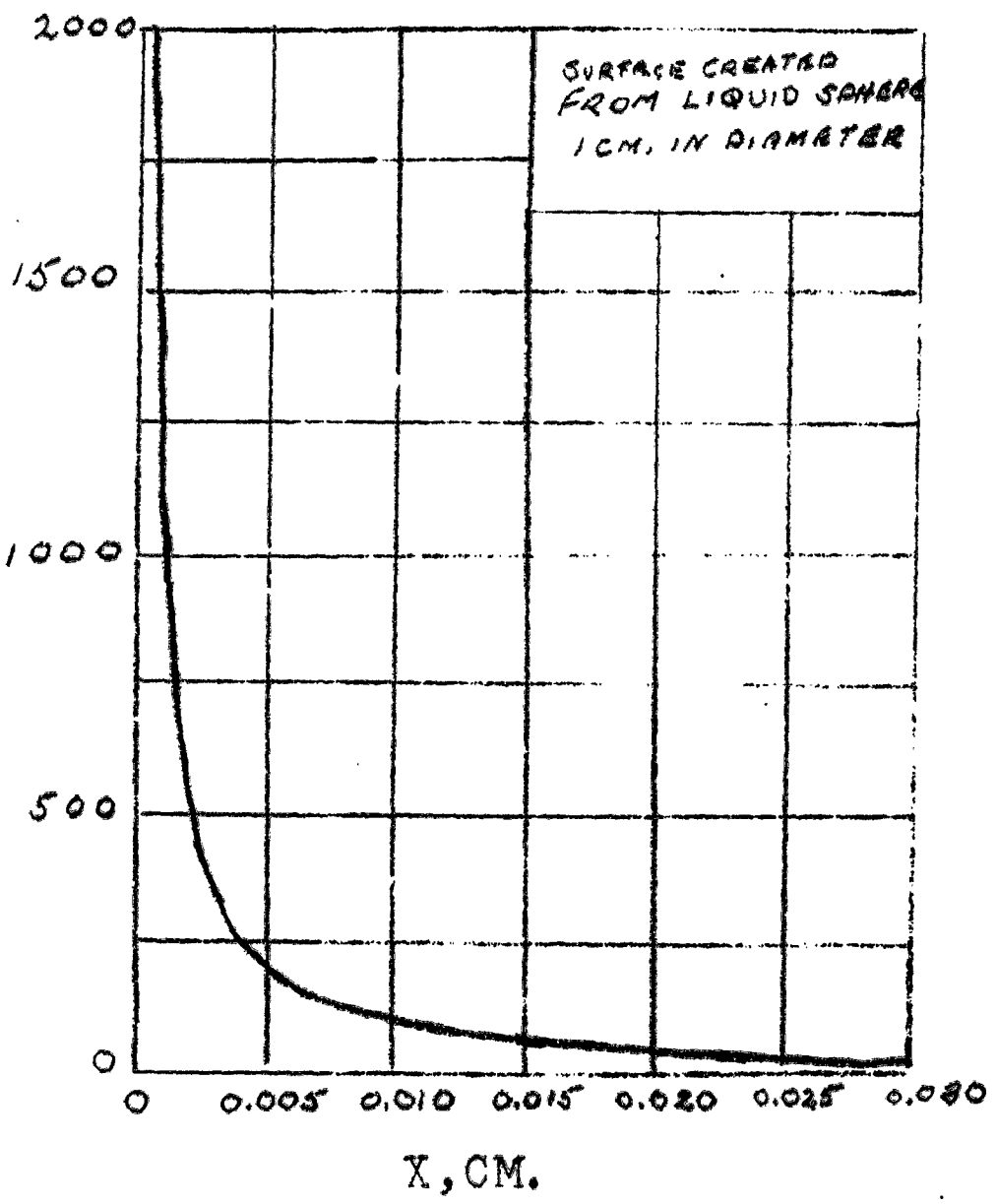

Figure 1. Curve Showing the Great Increase in Liquid Surface Area Produced When a Liquid Sphere 1 cm. in Diameter is Atomized to Form Drops of Uniform Diameter X.

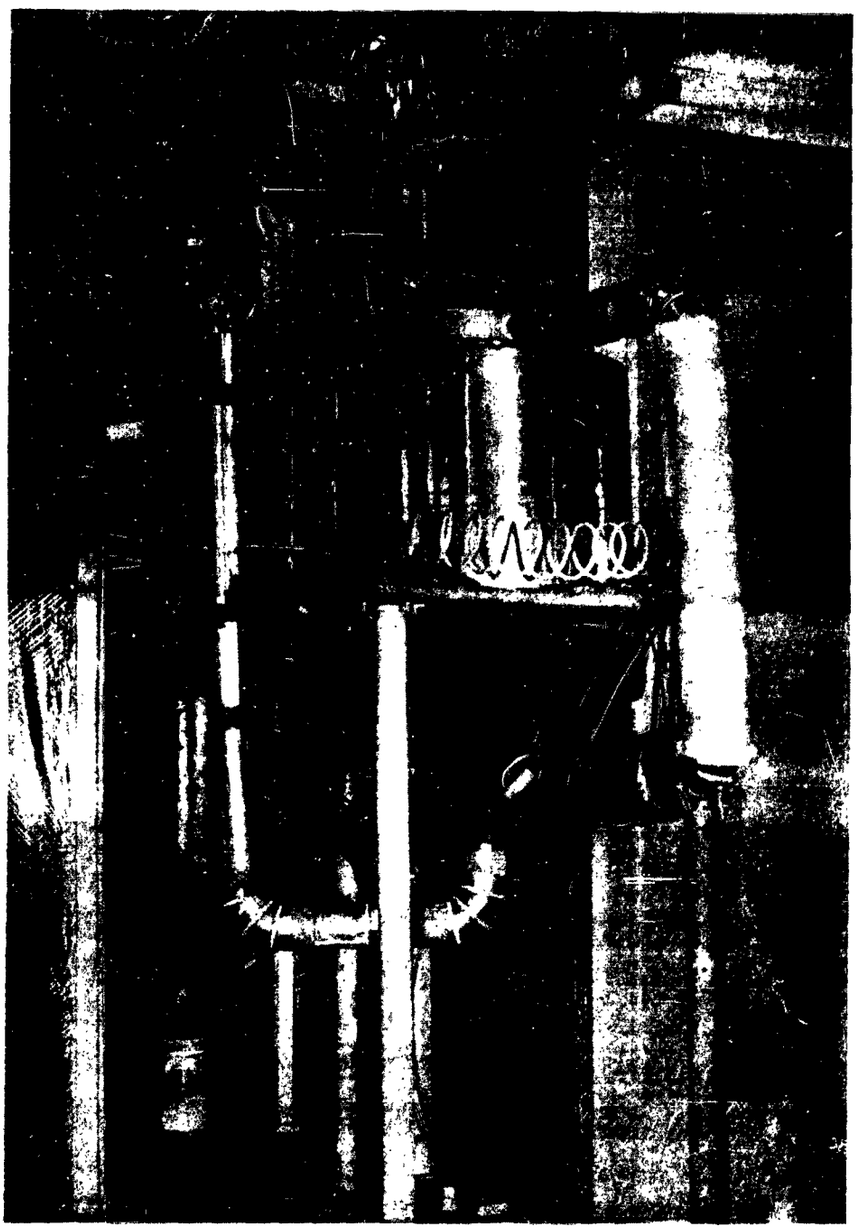

Figure 2. Completely Assembled Spray Drying Apparatus Ready for an Experimental Run.

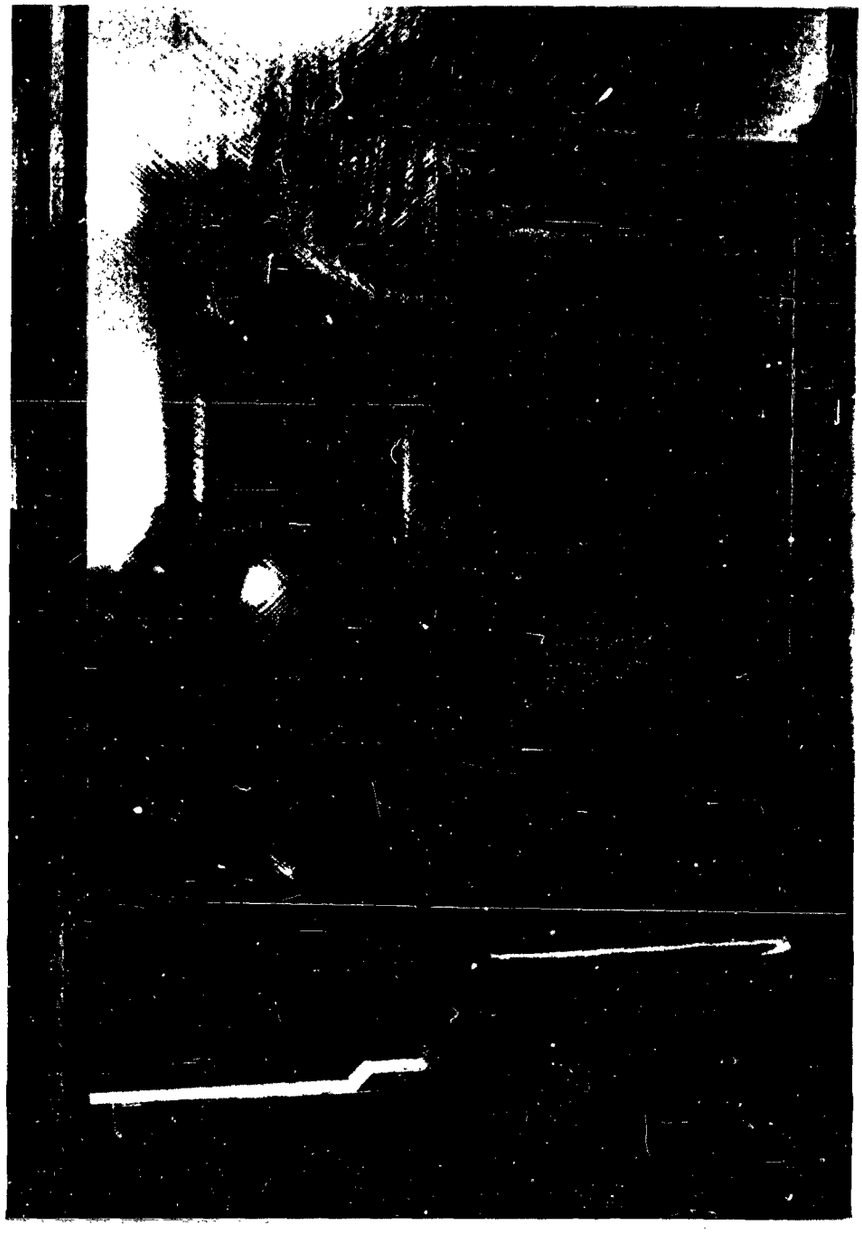

Figure 3. Totally Enclosed Room for Spray Drying Apparatus.

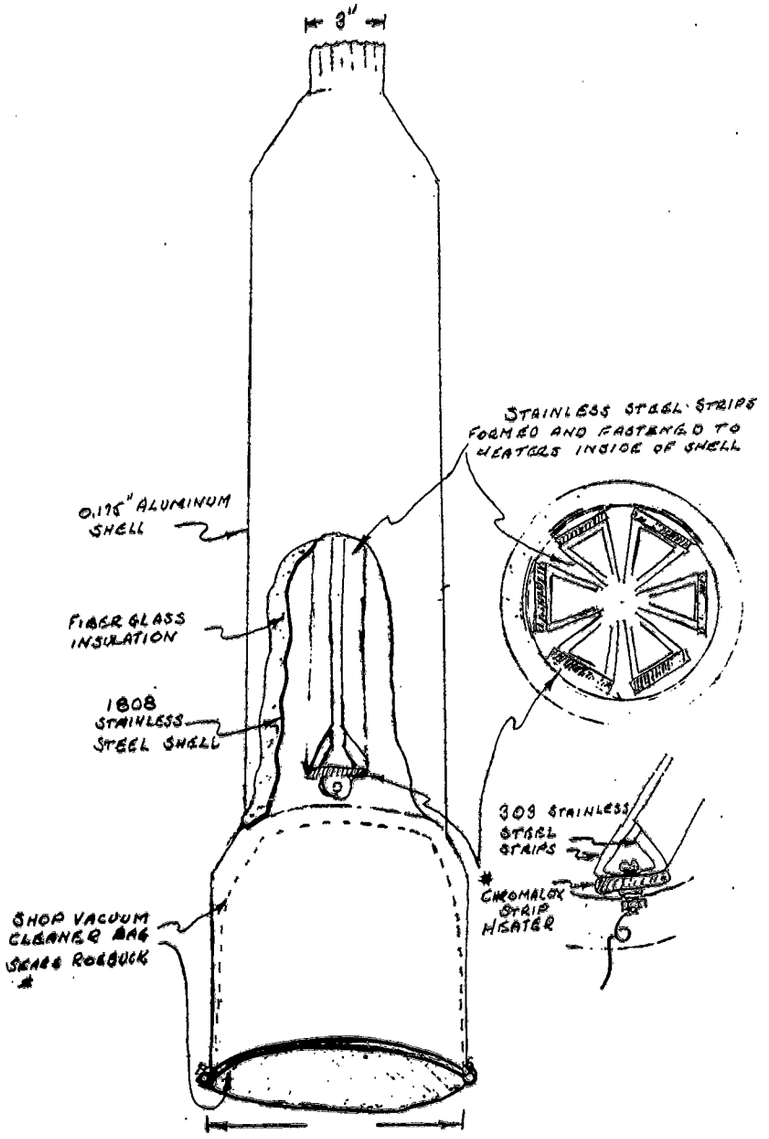

Figure 4. Stainless Steel Heater Shell.

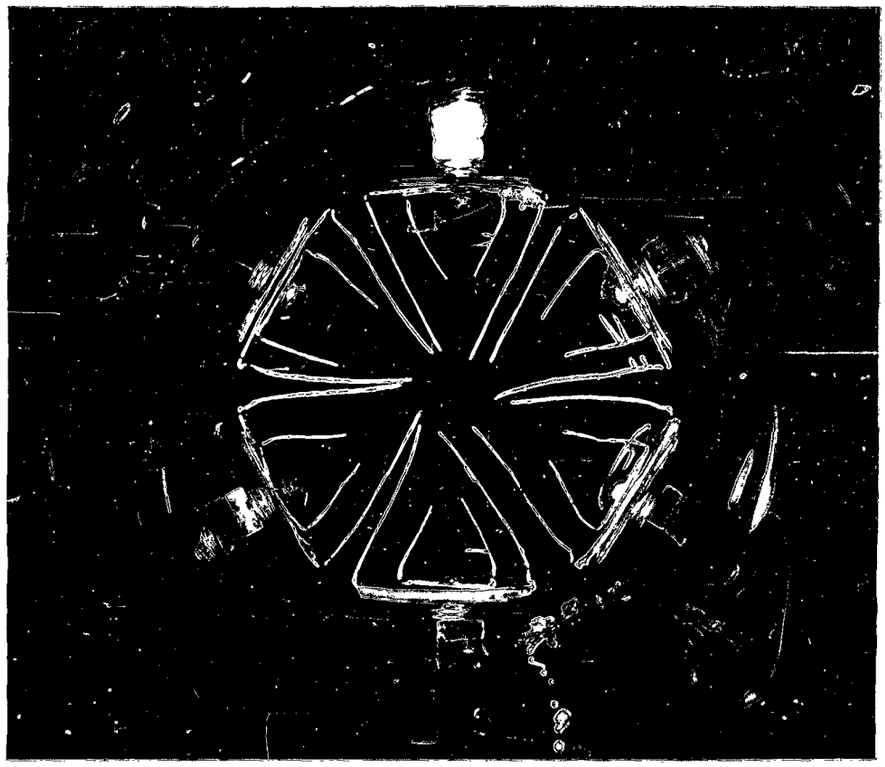

Figure 5. Heater Shell - Interior Cross Section Detail.

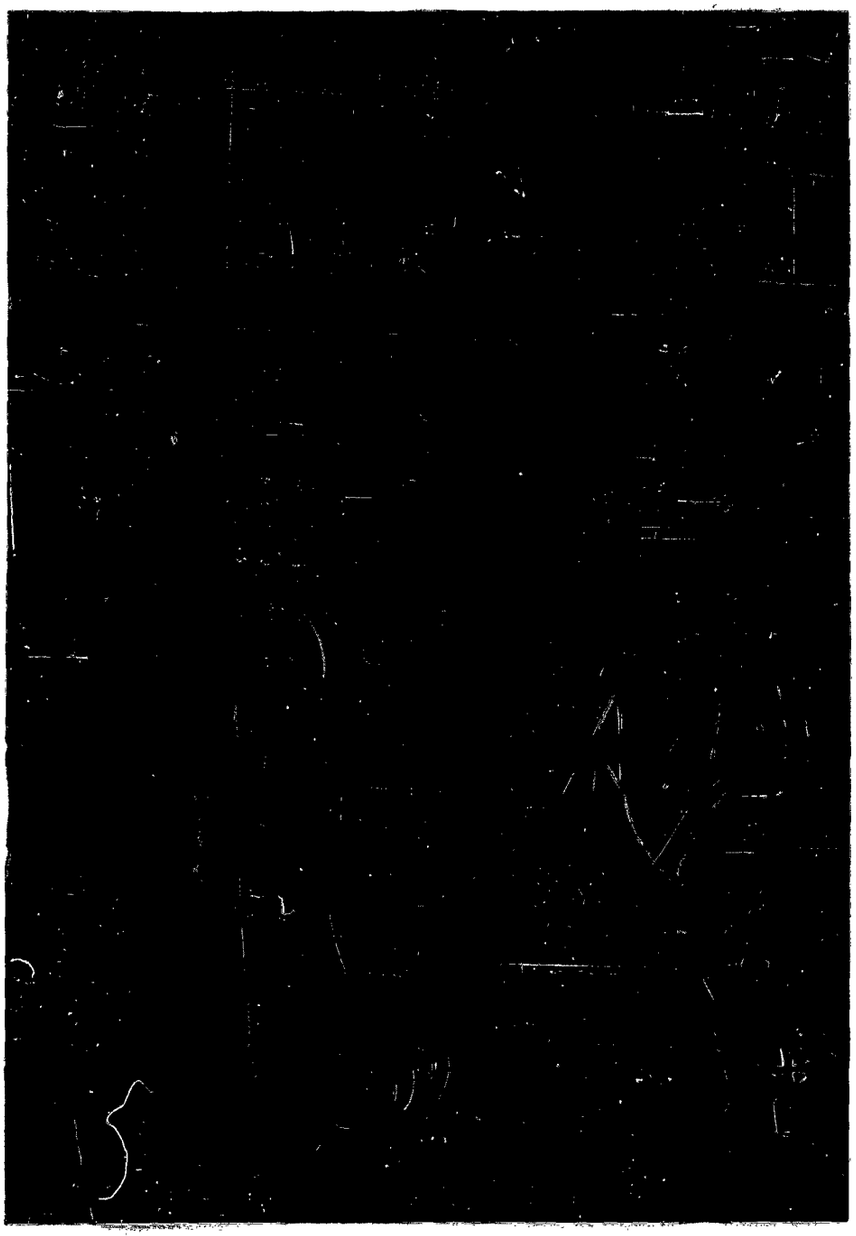

Figure 6. 60° Stainless Steel Cone Fitted to Bottom of Drying Chamber.

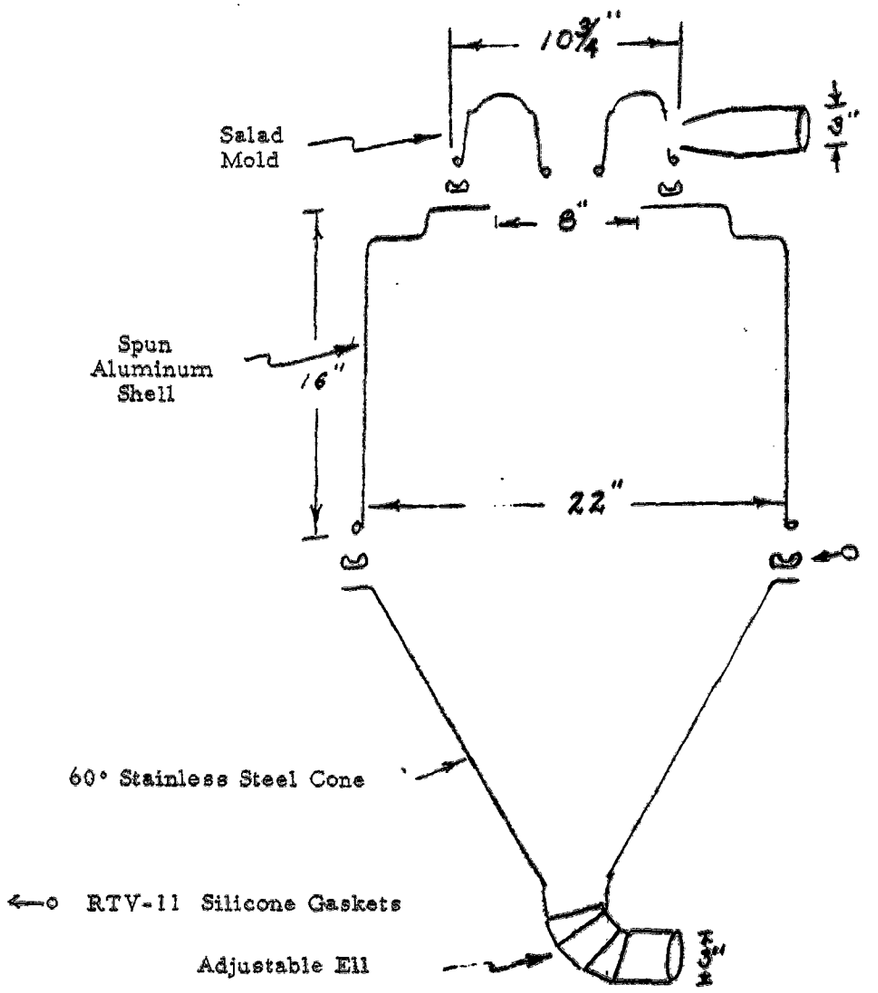

Figure 7. Exploded Cross Section of Drying Chamber.

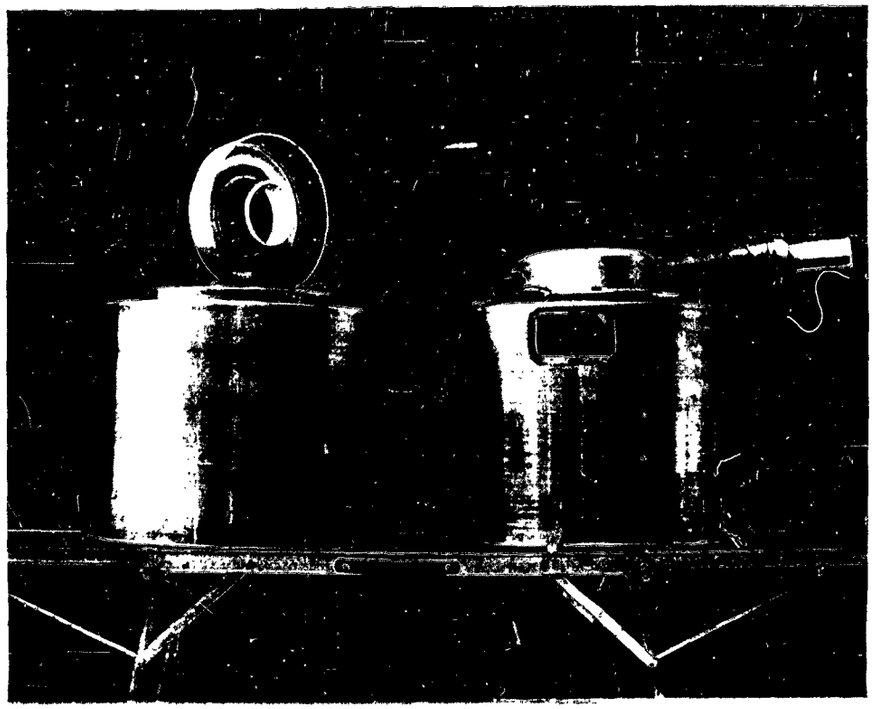

Figure 8. Drying Chamber - Spun Aluminum Shells.

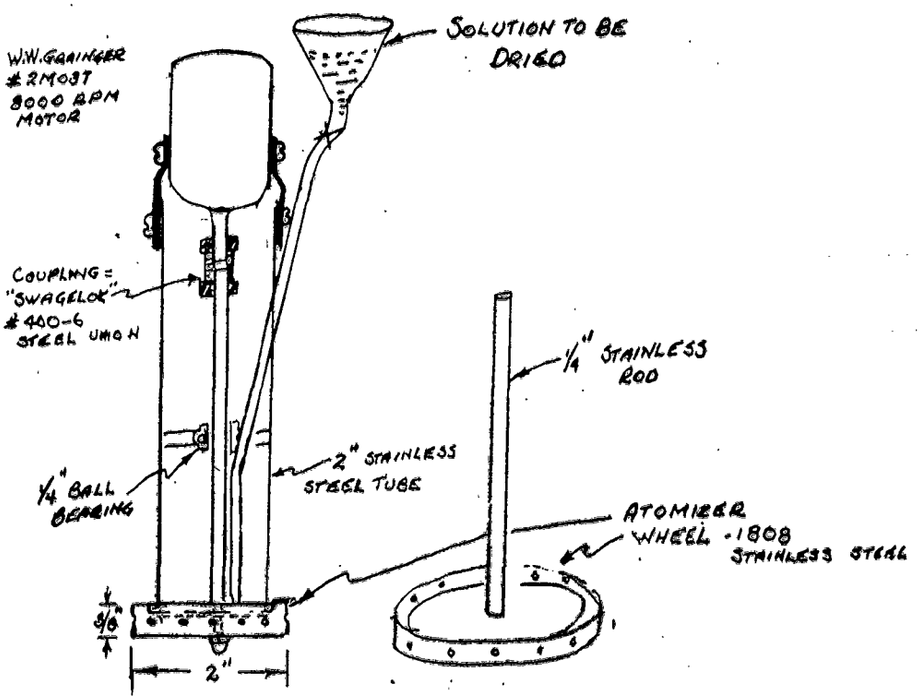

Figure 9 . Centrifugal or Spinning Disc Type Atomizer.

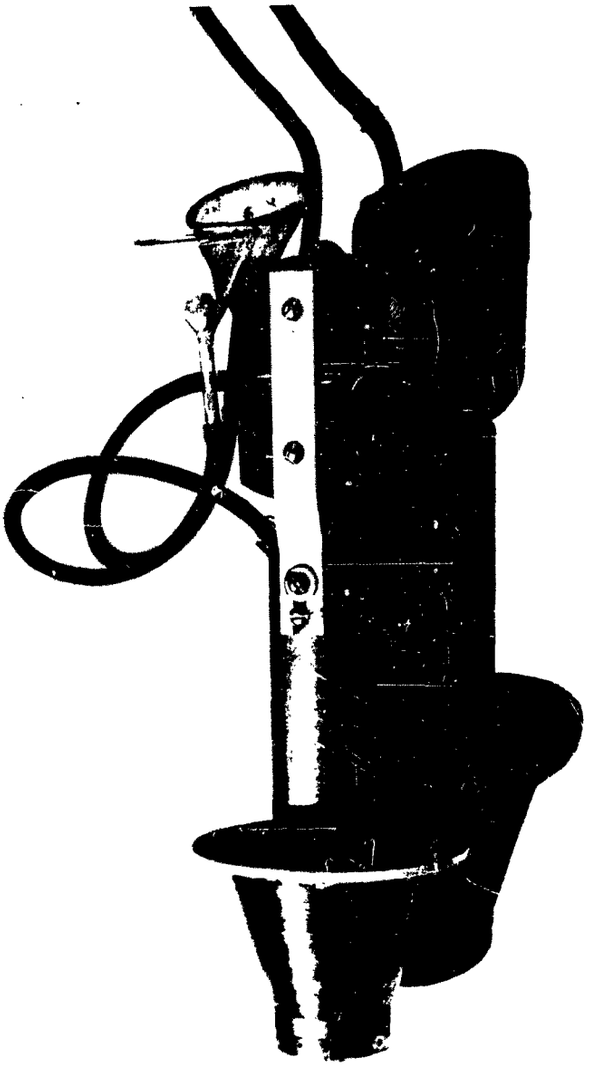

Figure 10. Centrifugal or Spinning Disc Type Atomizer Used in Initial Trials.

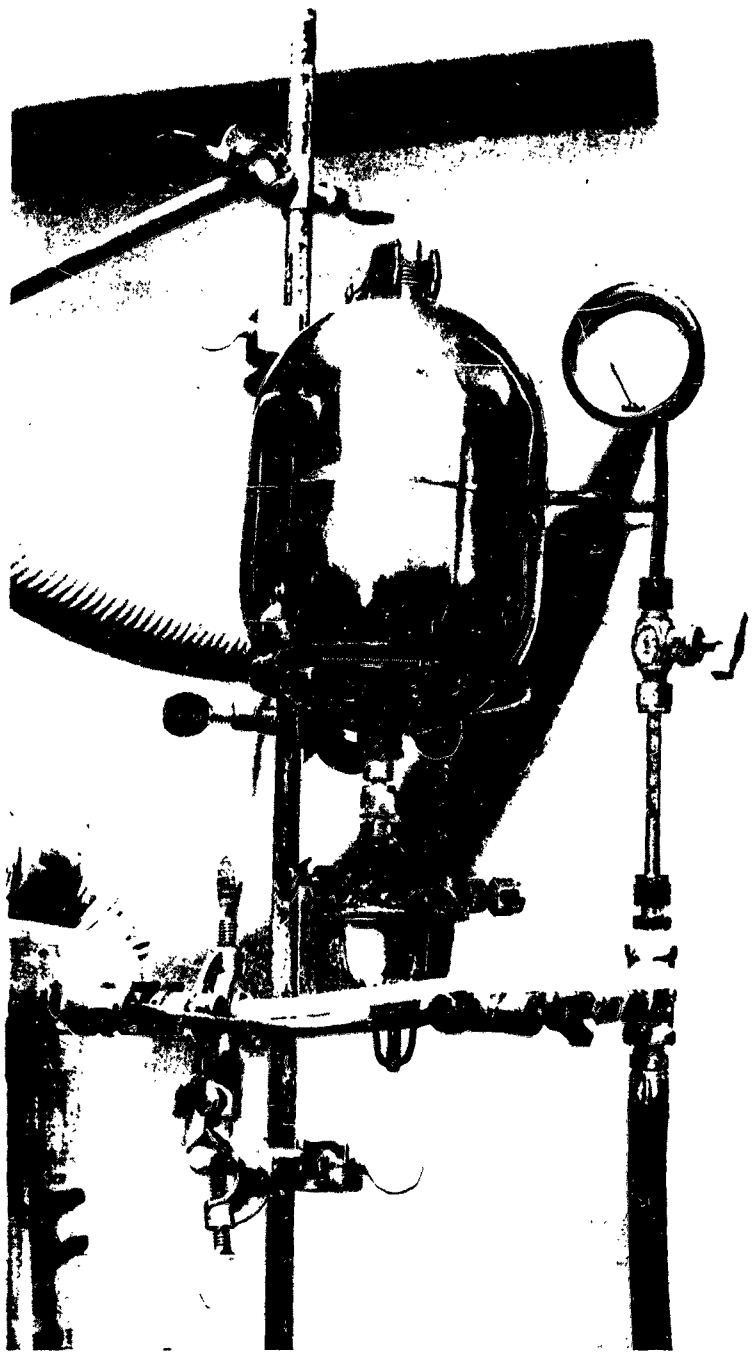

Figure 11. Stainless Steel Vessel with Fuel Filter.

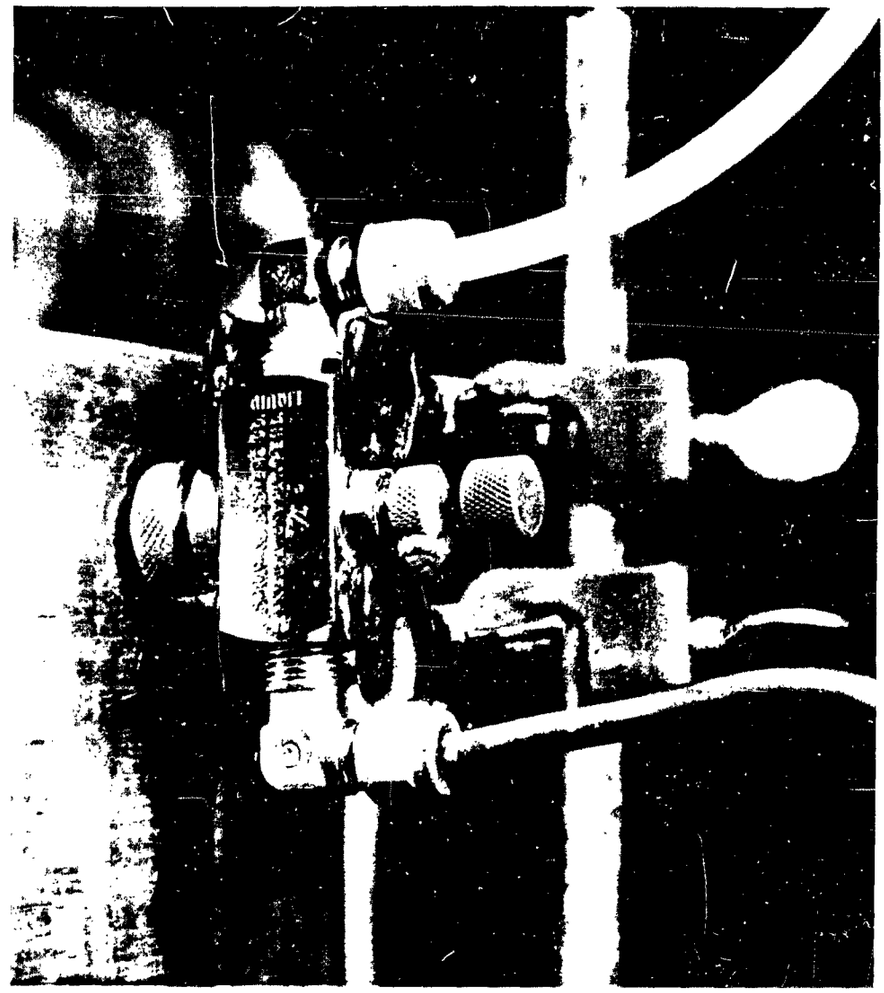

Figure 12. Pneumatic Atomizing Spray Nozzle-Feed Through Side of Drying Chamber.

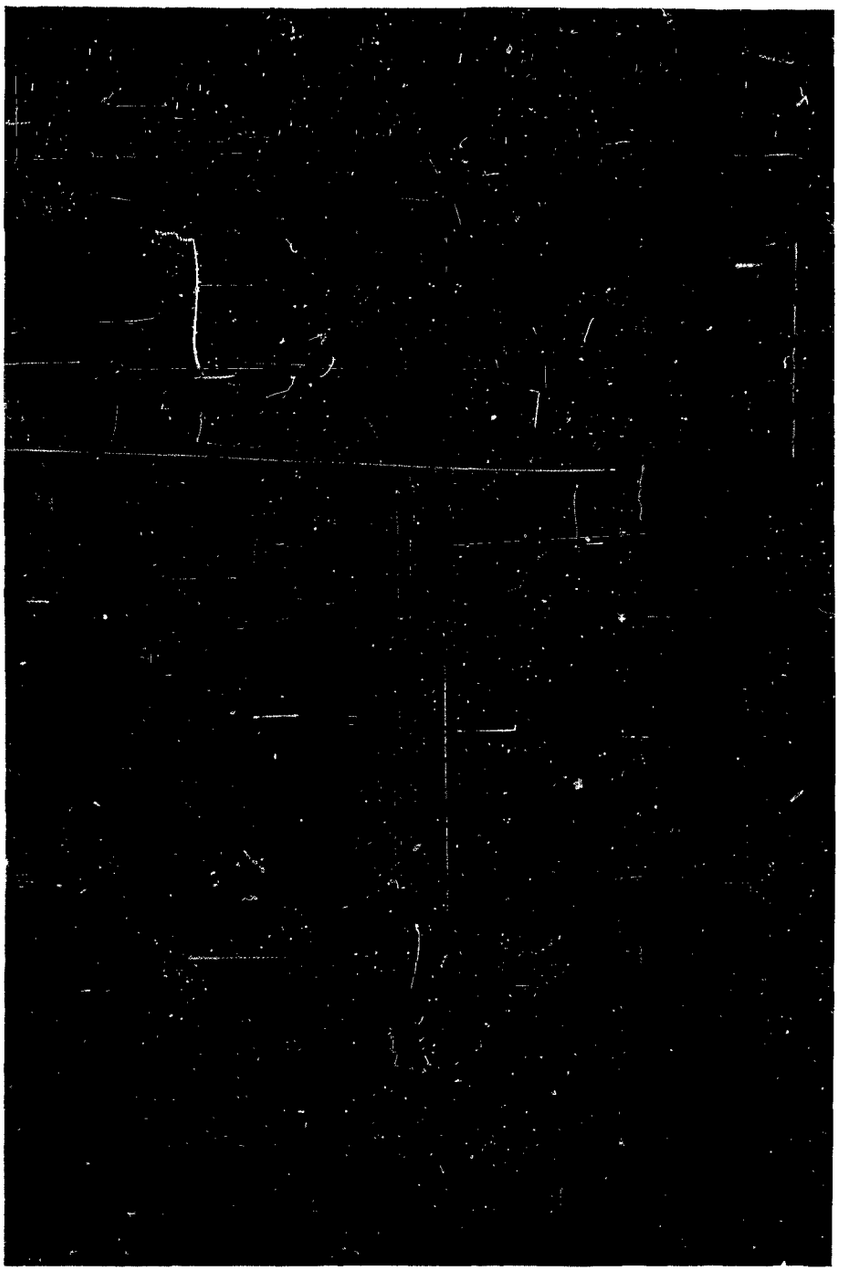

Figure 13. Flow Meter in Liquid Feed Line.

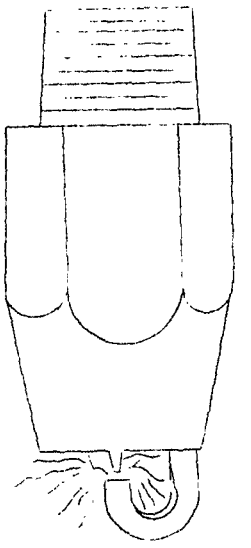

Figure 14a. Bete Impingement Nozzle

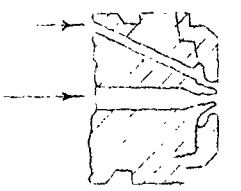

Figure 14b. Pneumatic Nozzle

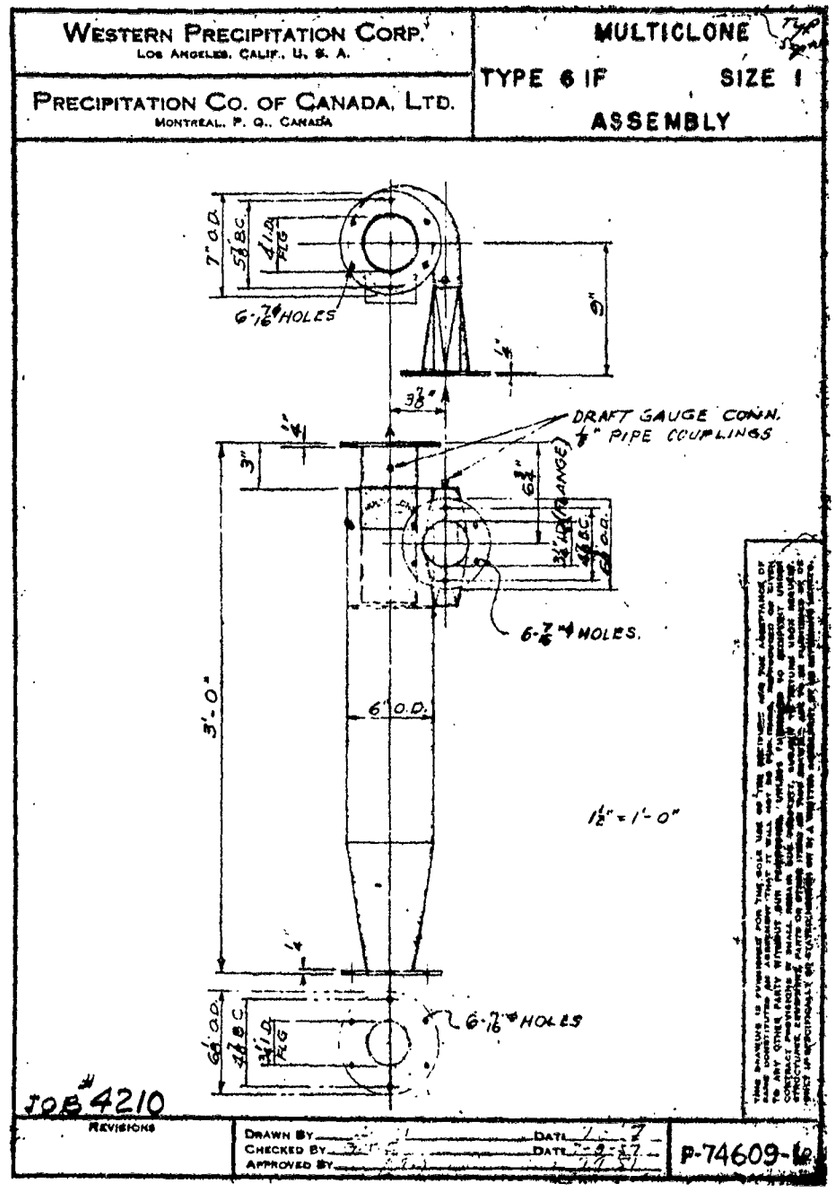

Figure 15. Powder Separation Section, Standard Multiclone, Cyclone Type Separator.

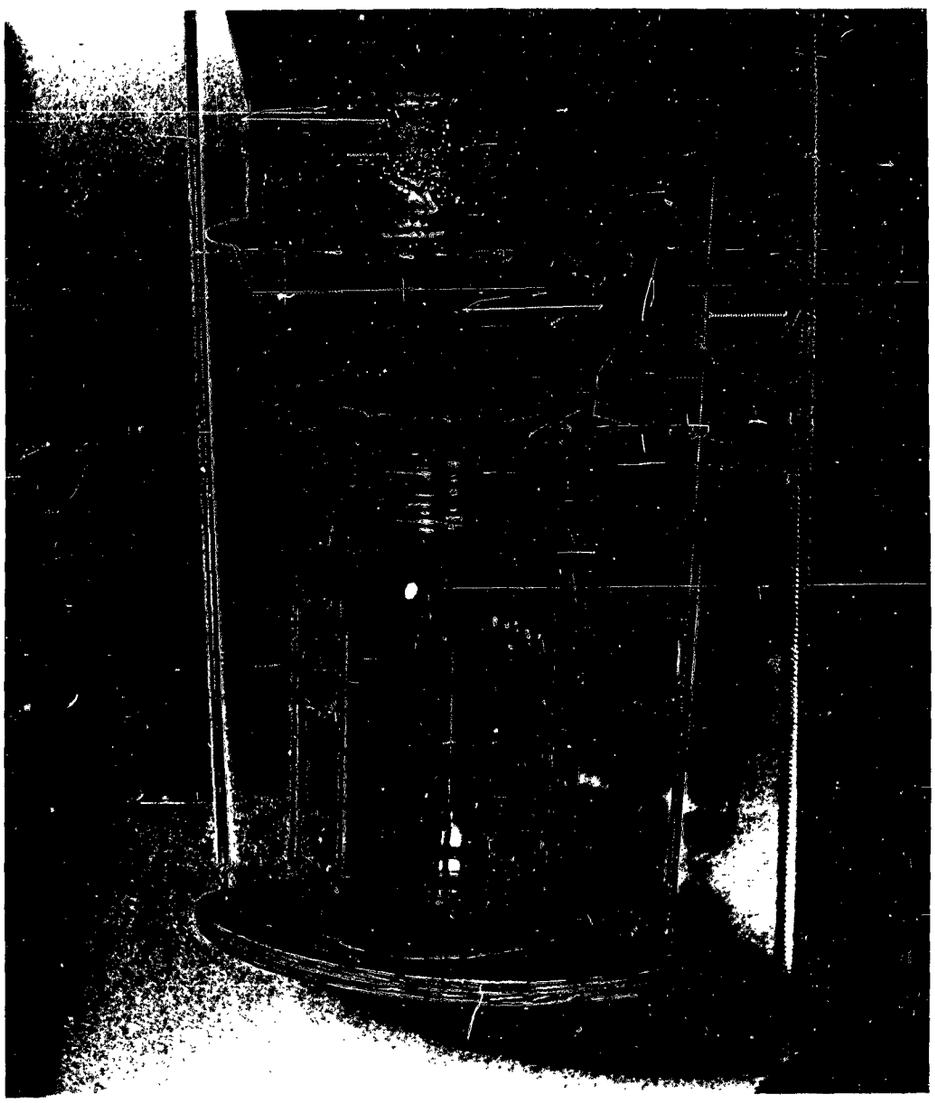

Figure 16. Powder Separation Section - One-Quart Collection Jar.

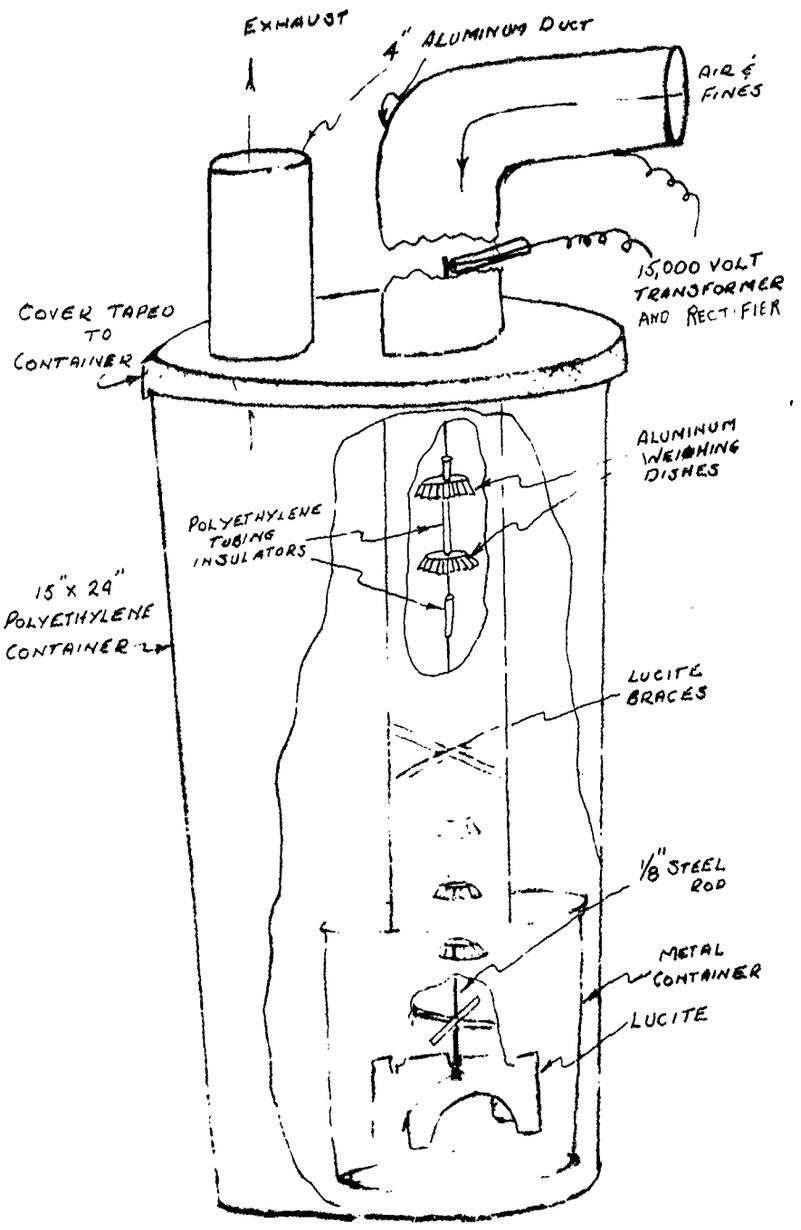

Figure 17. Electrostatic Precipitator.

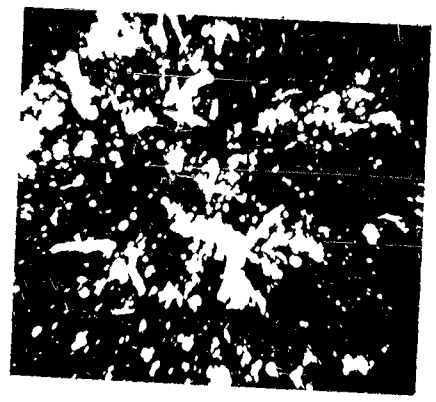

Figure 18. Photomicrograph of Spray Dried PABA + 1% Catanac SN Showing Typical Crystals When Dried from Alcohol. Solvent: Alcohol and Carbon Tetrachloride.

Figure 19. Photomicrograph of Spray Dried PABA + 1% Catanac SN Direct from Dryer. Solvent: Acetone and Carbon Tetrachloride.

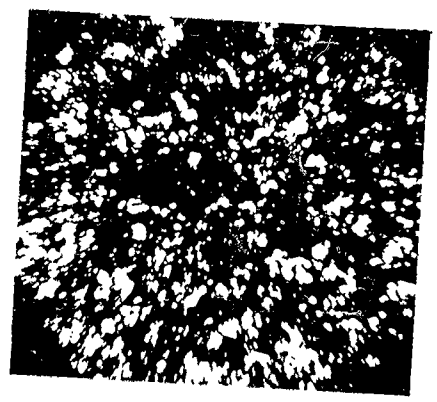

Figure 20. Photomicrograph of Spray Dried PABA Without Catanac SN. Solvent: Acetone and Carbon Tetrachloride.

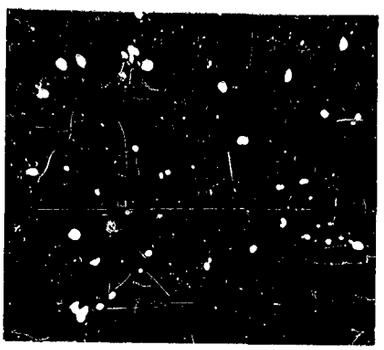

Figure 21. Photomicrograph of PABA (Fines) from Air Classifier. Solvent: Acetone and Carbon Tetrachloride.

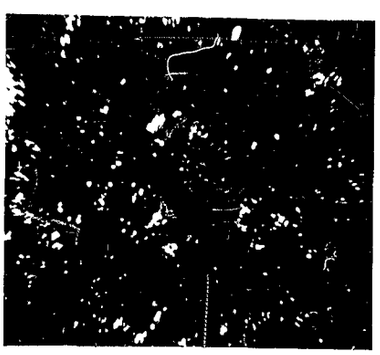

Figure 22. Photomicrograph of PABA from Electrostatic Precipitator. Solvent: Acetone and Carbon Tetrachloride.

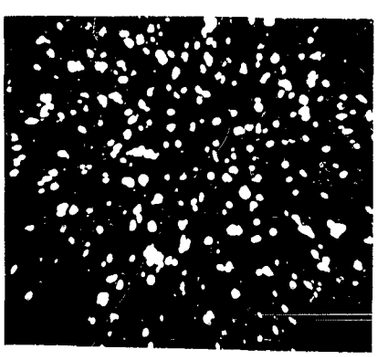

Figure 23. Photomicrograph of Coarse Material (Tailings) Left in Bottom of Air Classifier After Separation of Fines. Material: Saccharin + 1% Catanac SN. Solvent: Acetone and Carbon Tetrachloride.

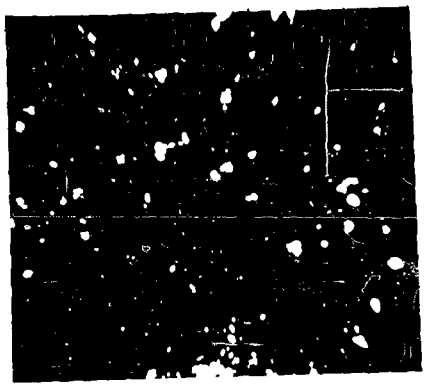

Figure 24. Photomicrograph of Saccharin + 1% Catanac SN. Solvent: Acetone and Carbon Tetrachloride.

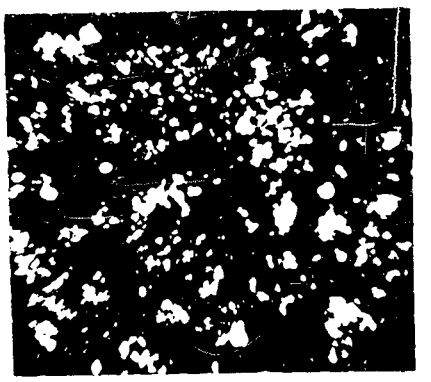

Figure 25. Photomicrograph of PABA Dried by Biochemical Process Company. Solvent: Alcohol.

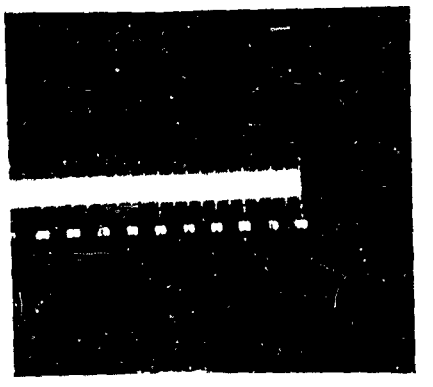

Figure 26. Micrometer Stage Scale - Each Division = 10 microns.

Figure 27. Apparatus for Determination of Relative Powder Flow--PABA.

Figure 28. Apparatus for Determination of Relative Powder Flow--Sucrose.

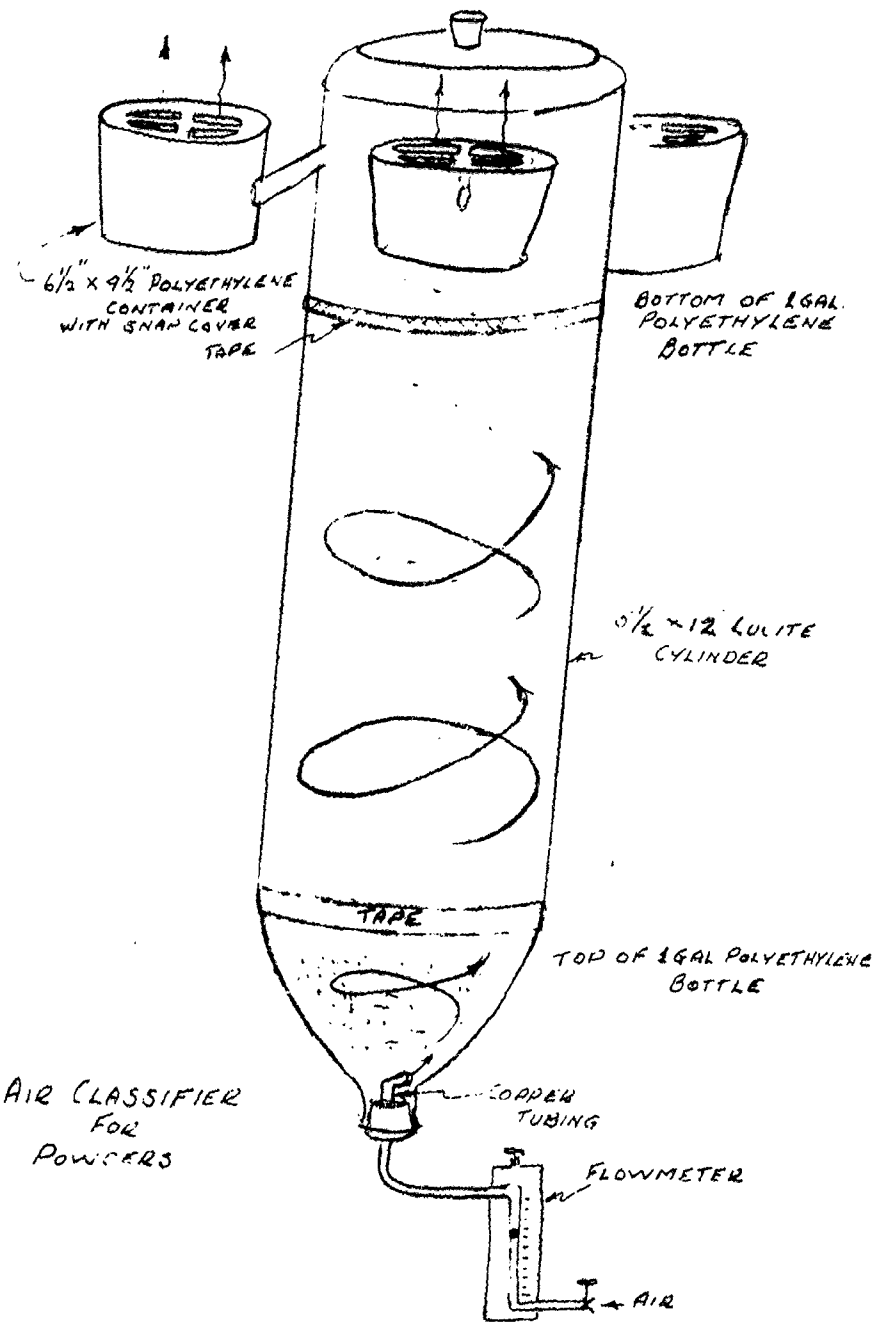

Figure 29. Air Classifier Apparatus for Separation of Powders.

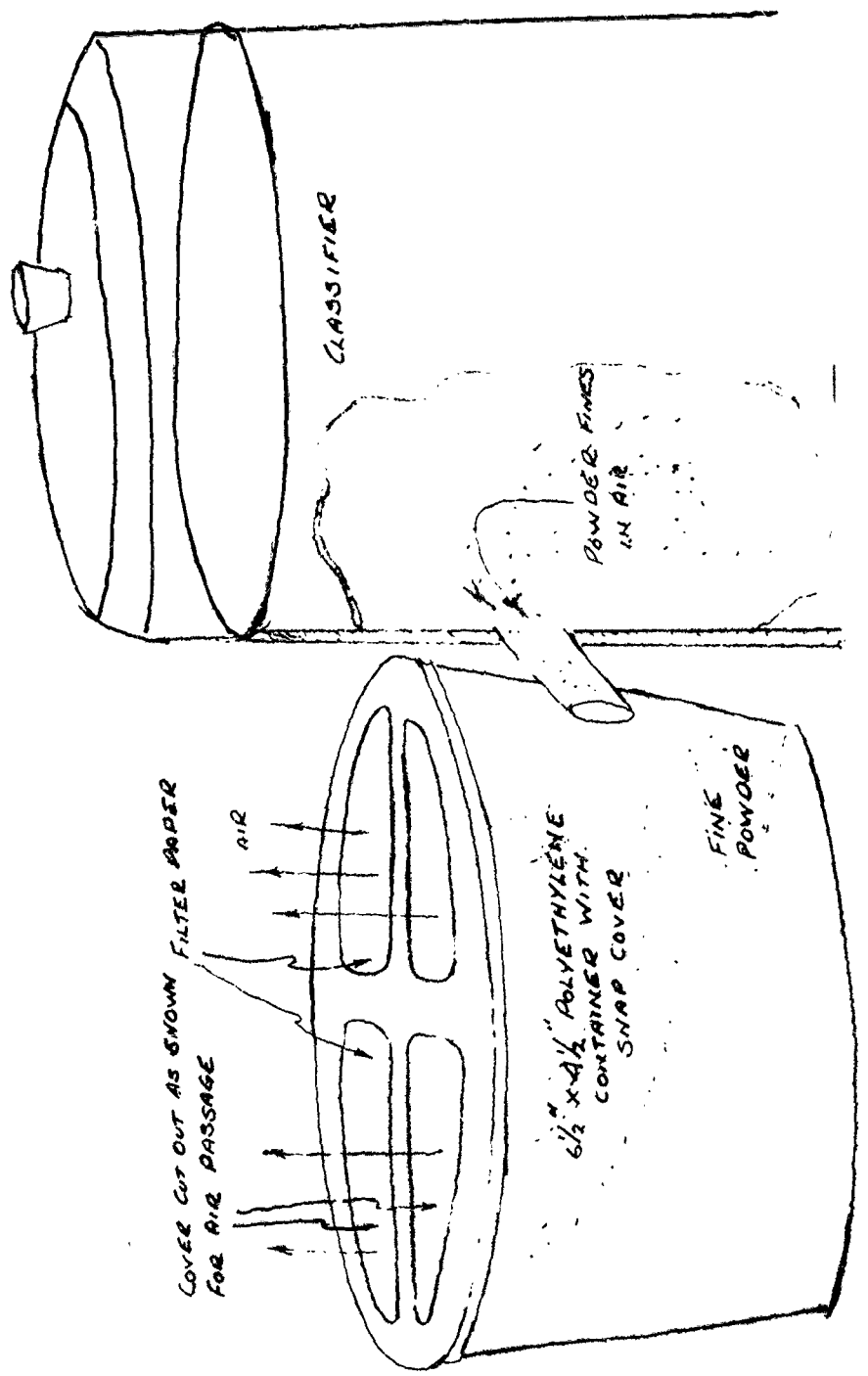

Figure 30. Exploded View of Air Classifier - Fine Powder Collection Section.

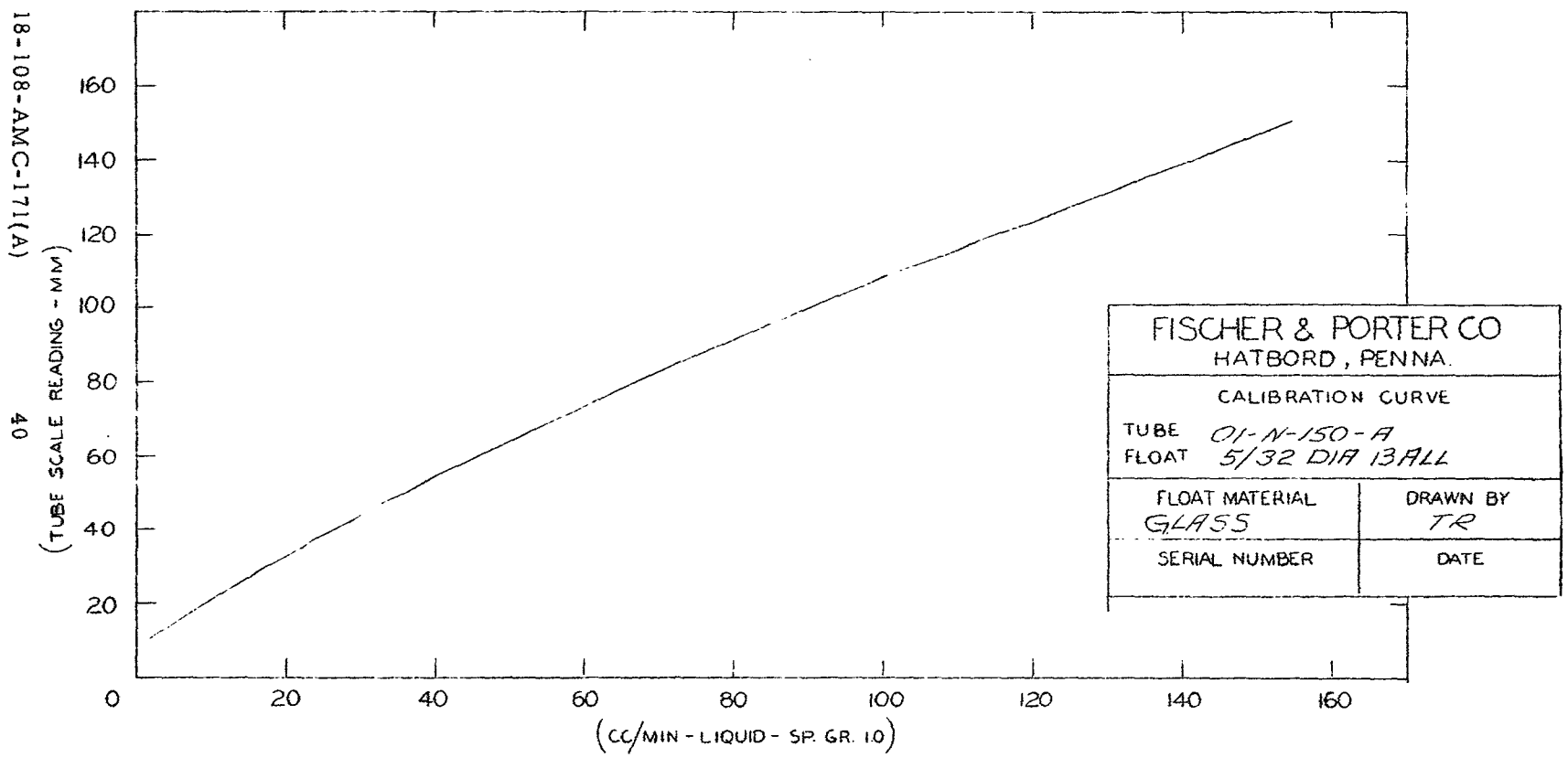

Figure 31. Fisher and Porter Calibration Curve for Determination of Fluid Flow Rate.

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achieved. Incorporation of an anti-static agent, Catanac SN, markedly increased the flowability of the powders. A spray-dryer was designed and constructed with sufficient adaptability and simplicity to permit evaluation of various atomization devices (spinning disk, centrifugal pressure nozzle, impingement and pneumatic nozzles), hot-gas spray mixing procedure, powder collection and separation equipment.

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