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By B. K. Shibler and D. A. Martin

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ILLUSTRATIONS

DESIGN AND CONSTRUCTION OF A LABORATORY-SCALE FLUIDIZED-BED REACTOR

by

B. K. Shibler[1] and D. A. Martin[2]

ABSTRACT

A highly flexible laboratory-scale fluidized-bed reactor was designed, constructed, and successfully operated continuously at temperatures up to 1,300° C during an investigation of thermal methods for decomposing gypsum. This report discusses briefly the many problems encountered in developing the 10-inch-diameter reactor and describes its design and construction in sufficient detail to enable other research organizations to build a comparable unit.

The reactor consists of a 10-inch-inside-diameter vertical tube which can be varied from 6 to 10 feet high; it is formed from 2- and 3-foot sections of silicon carbide pipe. The tube is surrounded by a 3-inch heating space formed by an outer 4.5-inch-thick circular wall of insulating firebrick. A cone-shaped fluidizing chamber is bolted to the bottom of the reactor. Fluidizing gas enters through the bottom of the cone, and ore is introduced by means of a screw feeder connected to an inlet located just above the gas port.

A silicon carbide overflow tube of 1.25-inch inside diameter extends upward into the reactor. The depth of the bed is controlled by substituting different lengths of overflow tube. Twin dust-recovery systems, each composed of a cyclone and Fiberglas filter, are attached to two gas outlets at the top of the reactor tube.

The reactor is designed for research on both exothermic and highly endothermic reactions. It can be fired externally by means of four natural gas burners that jet tangentially into the annular space at the base of the reactor. Alternatively, it can be fired internally, using a mixture of air or oxygen and natural gas as the fluidizing medium.

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A separate instrument panel contains flowmeters, manometers, an automatic program controller, and a multiple-point recorder for printing a record of the temperatures measured by thermocouples located at critical points in the reactor.

INTRODUCTION

Gypsum and anhydrite have been used as a source material for the production of sulfuric acid in England and continental Europe for several years.[3] Decomposition of the calcium sulfate usually is effected in rotary kilns with the aid of carbon reductants. The objective of research undertaken by the Bureau at Salt Lake City was to investigate the effectiveness of fluidized-bed roasting as a method of decomposing gypsum to obtain sulfur dioxide and a usable lime or a residue suitable for making cement. The results of this investigation will be presented in a Report of Investigations.

Fluidized-bed roasting techniques present a number of advantages over conventional fixed-bed or rotary kiln roasting methods. The large surface area provided for the fluidizing gas by the reacting particles promotes high reaction rates and efficient heat and mass transfer.[4] Uniform bed temperature, ease of temperature,control, and control of fludizing gas atmosphere are important advantages in fluidization.

Conversely, there are several disadvantages in fluidized-bed heating, calcining, or roasting. First, the attrition of particles in the fluidized bed may cause considerable dust, necessitating the use of dust-collecting equipment to clean the effluent gas stream; second, the rapidly moving particles may erode the containing walls of the reactor and lines of transport; and third, some of the fresh ore particles short circuit through the bed and receive an insufficient reaction time. These, of course, are common disadvantages that are not considered to be particularly serious in a number of metallurgical processes.[5]

An externally fired reactor with an inside diameter of 4 inches was used in the initial phases of investigation. Fusion and defluidization were difficult to control in this unit, and the 10-inch-diameter reactor, embodying highly flexible operating characteristics, subsequently was designed and constructed.

GENERAL CONSIDERATIONS

The prime considerations involved in designing a fluidized-bed reactor for experimental work are (1) the nature of the variety of materials to be processed, (2) the reactions that are to be sustained within the reactor, and (3) the capacity desired. These factors reasonably well define the temperatures, heat requirements, retention times, materials, construction, and size of the components of the reactor.

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[3] Fleck, Alexander. The British Sulfuric Acid Industry: A Retrospect and Research Prospect. Chem. Ind. (London), Dec. 6, 1952, pp. 1184-1192.
[4] Othmer, D. F. Fluidization. Reinhold Pub. Co., New York, N.Y., 1950, pp. 2-76.
[5] Counselman, T. B. How Fluidization Can Serve the Mineral Industries. Eng. Min. J., v. 156, No. 3A, March 1955, pp. 70-76.

A versatile reactor was planned because of the diverse nature of metallurgical investigations conducted by the Bureau of Mines. Further, it was believed that the highly endothermic decomposition of gypsum would be the most demanding process for which the reactor would ever be used. Thus, the reactor was designed to include this process within the upper limits of its operating capabilities.

The generalized equation for the gypsum process is

$$2CaSO_4 \cdot 2H_2O + C \rightarrow 2CaO + 2SO_2 + CO_2 + 4H_2O.$$

The temperature and retention time necessary for this reaction were established by tube furnace tests and experimentation in a 4-inch fluidized-bed reactor. Results indicated that temperatures in the range of 1,200° to 1,250° C and an average retention time of 1.5 to 2 hours would be necessary for effective decomposition of the gypsum.

Calculations made on the basis of the heats of formation of the reactants and products indicated that approximately 91 kcal would be necessary to effect the reaction for each gram mole of gypsum processed at 1,200° C. An additional 45 kcal would be required to heat the products and reactants to 1,200° C.

Estimates of heat requirements for this process were made for reactors of circular cross section, with diameters ranging from 6 to 20 inches. These estimates included reactor heat losses and were premised on a reaction zone 3 feet in height insulated with 8 inches of firebrick. Other factors, such as a bed temperature of 1,200° C and a feed rate of 0.7 pound of minus 10-mesh gypsum per min per sq ft of reactor cross-sectional area, were used in making the estimates. This feed rate, established in the 4-inch reactor, allowed sufficient retention time in a 2.5-foot fluidized bed to produce good decomposition of the gypsum at 1,200° C.

Calculations were made to ascertain the net heat available for the reaction that could be derived from internal firing using air or oxygen, mixed with natural gas as the fluidizing medium. Tests in the 4-inch reactor established that a gas velocity of 15 ft per min, measured at 24° C and 5 psig, was adequate to maintain a 2.5-foot bed of gypsum in a fluidized state without excessive elutriation at temperatures near 1,200° C. This provided about 1 ft per sec of gas entering the column. Calculations based on complete combustion of the fluidizing gas indicated that the heat requirement of the reactor could not be attained by simple internal firing with natural gas and air at the gas flow rate required for proper fluidization. Calculations also showed that the use of a mixture of oxygen and natural gas as the fluidizing medium would, at the necessary flow rate, supply approximately three times the necessary heat for the reaction. Thus, it appeared that the reactor should be designed for external as well as internal firing, as this would allow the use of the less costly air and natural gas mixture as the fluidizing medium for the gypsum process. The remainder of the heat requirement then could be supplied by external heating.

This design also offered other advantages. A reactor capable of being fired externally would allow a choice in fluidizing atmospheres. It also would facilitate the temperature control of exothermic reactions. Alternatively, air or other gaseous coolants could be circulated through the heat exchange space, or the annular heating space could be packed with a granular or fibrous insulation.

Several factors were considered in determining the reactor size. It was believed that a larger reactor would alleviate the problems of fusion and defluidization encountered with the smaller 4-inch fluidized-bed reactor. Heat loss also was a consideration. The heat loss is proportional to the surface area of the reaction zone, and the available heat from the combustion of a gas mixture at a given space velocity is proportional to the cross-sectional area. Thus, the percentage of heat loss is reduced proportionately as the radius of the reactor is increased because the increase in surface area is directly proportional to the increase in radius, but the cross-sectional area of the reactor increases in proportion to the square of the radius. Finally, the reactor should be sufficiently large to obtain relaible data for use in making economic evaluations and yet not require impractical quantities of ore for test operations.

REACTOR DESIGN AND MATERIALS OF CONSTRUCTION

To assure ready adaptability of the reactor to various research projects, the unit was designed to permit easy disassembly and modification and accurate control of temperatures, fluidizing gas, and ore feed rates. It was essential that the reactor height and bed depth could be changed as required and that the components in the interior of the unit would be accessible for inspection and cleaning. The reactor also had to be capable of containing highly refractory processes such as the reductive decomposition of gypsum, and the 1,200° to 1,300° C temperatures necessary for this reaction required adequate materials of construction and provisions for thermal expansion.

These factors all were considered and influenced the design of the reactor, shown in cross-section view in figure 1. (In this diagram the burner system and flame ports in the magnesite base support have been simplified for clarity.)

Reactor Tube

Cast tubes of circular cross-section silicon carbide, bonded with silica, were chosen for the reactor because of their inert nature and ability to withstand high temperatures. Two 3-foot and two 2-foot sections were procured. These had 10-inch inside diameters, 0.75-inch wall thicknesses, and lap-joint ends. Initially two 3-foot sections were assembled to form a 6-foot reactor. The shorter units were added later. The lap joints facilitated assembly and were sealed readily by calking with alumina fiber rope and a refractory cement.

In practice, the top of the reactor tube is closed with a mold-cast disk of crystalline silicon carbide that is pierced by two holes for effluent gaslines. The disk is shown in place on the reactor tube in figure 2. Several

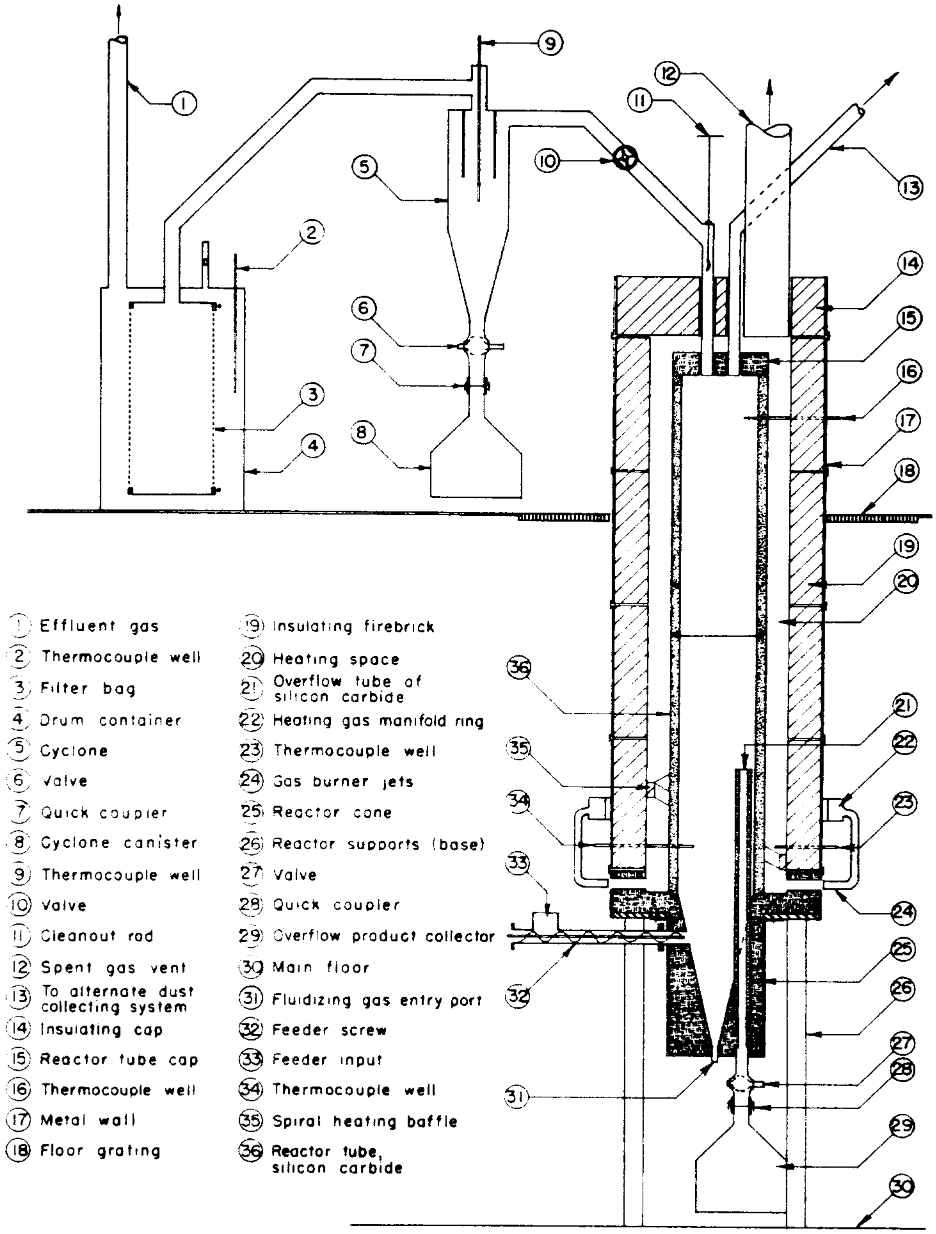

FIGURE 1. - Ten-Inch Fluidized-Bed Reactor.

holes one half of an inch in diameter were drilled through the reactor tube wall with a diamond drill bit for insertion of thermocouples and manometer connections.

Insulating Mantle

The reactor tube is insulated by means of Fiberglas and circular sections of firebrick held in place by a 16-gage sheet-iron shell. A section is shown in figure 3. Each section but one has a diameter of 28 inches and a height of 18.5 inches; the one exception is the insulating cap which has a height of 9 inches and which provides top closure for the external heating space. The cap has ports for the two effluent reactor gaslines and the burner gas exhaust.

The firebrick is retained in each section by a 2-inch lip of 12-gage type 316 stainless steel extending inward from the bottom edge of the shell. The general construction of each insulating section is shown by the diagram in figure 3. Each section contains a 1-inch layer of Fiberglas mat adjacent to the outer wall and 4.5 inches of cupola firebrick. This leaves a 3-inch space around the reactor tube for external firing. This annular space can be packed with a granular or fibrous insulation whenever internal firing is feasible.

FIGURE 2. - Reactor Tube.

A special feature of the bottom insulating section, which serves as a combustion chamber, is a spiral of brick extending outward into the heating space within 0.25 inch of the reaction tube. This spiral of brick was added to impart a swirling motion to the combustion gases and improve heat transfer to the fluidized-bed zone. In the first insulating section below the cap, three bricks extend outward and rest lightly against the reactor tube to center and support the tube within the mantle. This allows vertical expansion of the tube but prevents lateral movement within the mantle.

Each section is reinforced with a 1- by 0.25-inch steel band to which two lugs are welded for the purpose of installing or removing the tubular sections (fig. 2).

Reactor Supports

Thermal expansion requires that the reactor be secured at only one point. The fixed support is provided at the base of the reactor. The base support is a circular plate of type 316 stainless steel, 0.5 inch thick, and has a 28-inch outside diameter with a 10.375-inch-diameter opening in its center.

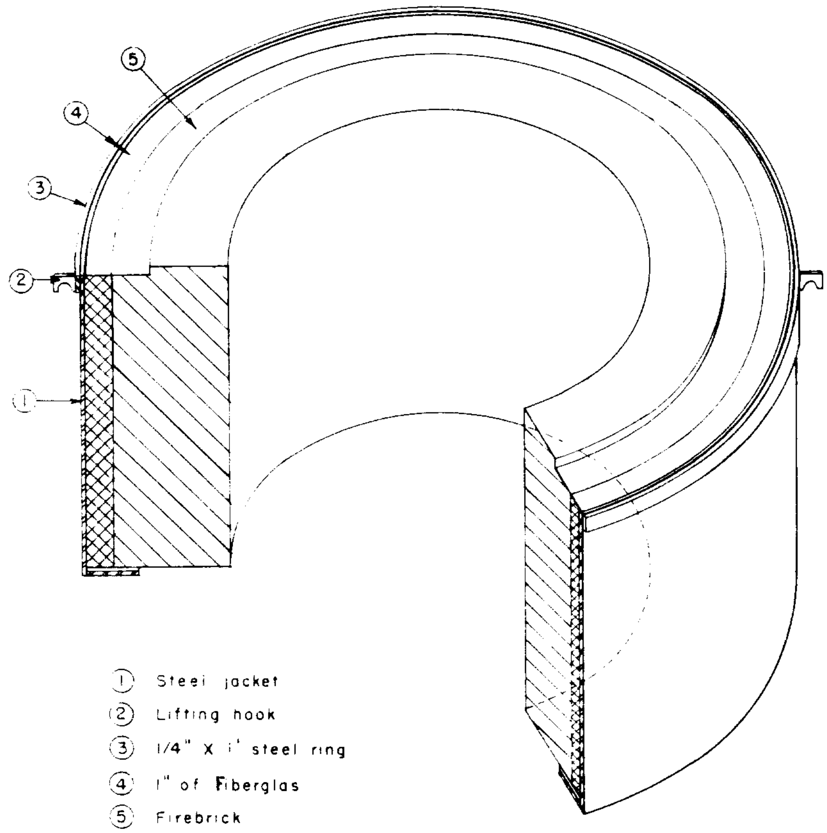

FIGURE 3. - Construction of Insulating Firebrick Section.

This supporting plate is insulated by a cylindrical block of dead burned granular magnesite cast on the upper surface of the plate and fired in place. The magnesite is retained by a 6-inch rim of 16-gage sheet iron welded to the outer edge of the plate.

The magnesite casting is contoured to accept the tubular reactor and provide transition to the 9-inch-diameter ore fluidized chamber later described. The magnesite block also supports the insulating mantle and, as indicated by the cutaway drawing which is figure 4, contains the flame ports for the four natural gas burners. These ports are cast to allow the burner gases to jet tangentially into the 3-inch external heating space. This unit rests on a 23.75-inch-square frame constructed from 2.5-inch angle iron with 42-inch long legs bolted to the floor. It is held in position by four brackets attached to the top of the angle iron frame.

The reactor is supported at the top in a manner that allows vertical expansion but prevents lateral movement. This was accomplished by projecting the reactor through a deck or mezzanine 100 inches above the floor. The reactor passes through a circular opening in this deck that is one-half of an inch larger than the diameter of the insulating mantle. The arrangement of the opening in the mezzanine floor is shown in figure 2.

Ore-Fluidizing Chamber

This section is the site of the screw-type feed inlet, fluidizing-gas port, and solids recovery system. It also serves as the bottom closure for the reactor tube. A photograph of the unit plus a drawing are presented in figures 5 and 6. The chamber consists of a gastight cylindrical container formed from 12-gage sheet iron. A flange of type 316 stainless steel is welded to the top outer edge of the cylinder. This 0.5-inch-thick, 1.75-inch-wide flange is drilled to receive the six 0.75-inch studs on the bottom of the magnesite-stainless steel support plate.

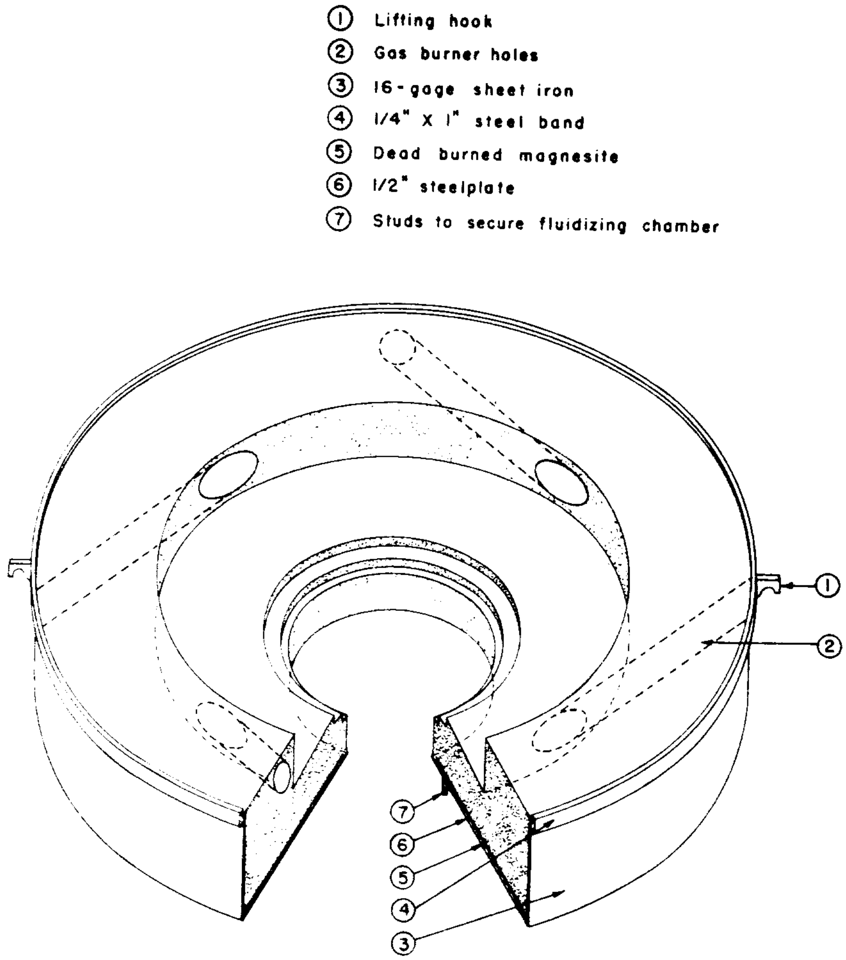

FIGURE 4. - Magnesite Casting on Base Support.

A molded fluidizing cone, composed of magnesite, is formed in the cylinder. Ore is injected into the cone through the 2.25-inch-diameter port 3 inches below the flange. Fluidizing gas enters the chamber through the apex of the cone via an 0.5-inch pipe, the size of which insures a gas velocity exceeding the settling velocity of the solids and the velocity of flame propagation in mixtures of natural gas and air.

The product overflow tube is set into the magnesite opposite the feed port. This tube is crystalline silicon carbide, with an inside diameter of 1.25 inches and an outside diameter of 2 inches. The base of the tube rests on a channel which was cast in the magnesite. This channel leads down to a pipe nipple welded to the base of the cylinder. Connection to the solids recovery canister is completed through a lubricated valve and a cam-type quick coupling.

Ore Feeder

Ore is injected into the fluidizing chamber at the bottom of the reactor by a horizontal conveying screw. The 2.25-inch entry port to the fluidized bed is centered 3 inches below the reaction support plate of the fluidizing chamber. Entry of ore near the bottom of the fluidized bed insures some retention time for all ore particles. Ore of very small particles size would be blown immediately out of the reactor by the fluidizing gas if introduced above the surface of the fluidized bed.

The ore feeder is a removable unit. It rests on four set screws which slide on two angle iron rails cantilevered out from the legs of the reactor. The set screws are used for proper horizontal alinement of this unit. The feeder, detached from the reactor is shown in figure 7.

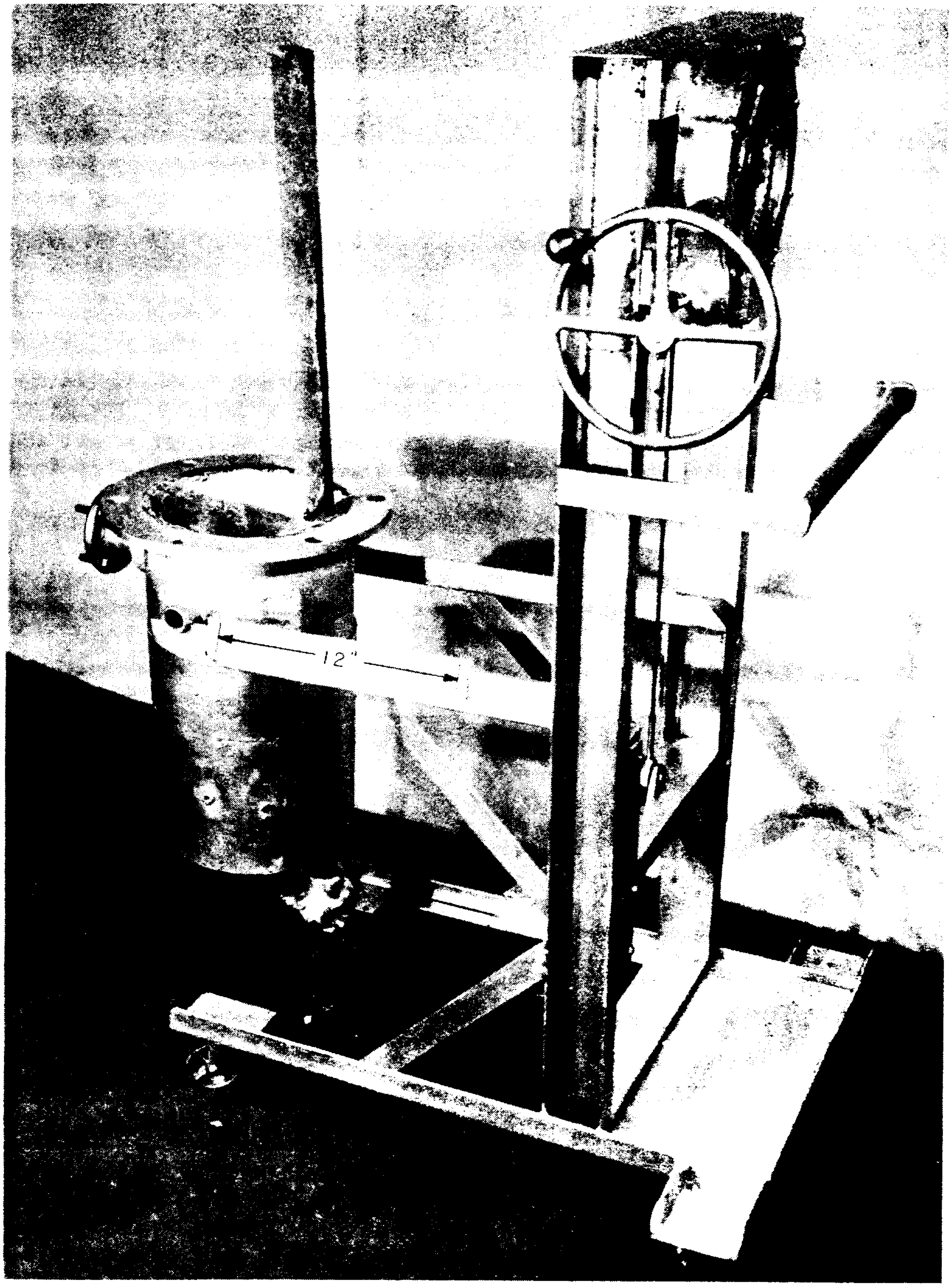

FIGURE 5. - Ore-Fluidizing Chamber.

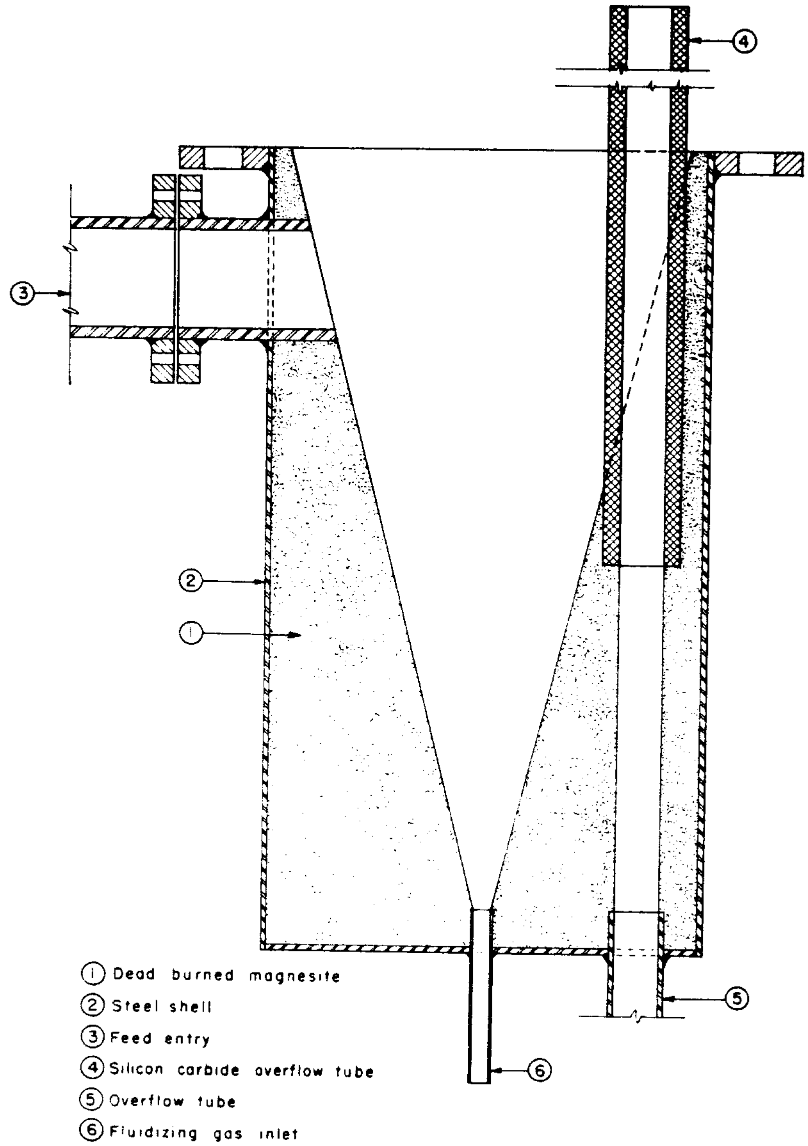

FIGURE 6. - Cross-Sectional Drawing of Fluidizing Cone.

The feeder is secured to the reactor at only one point. This is accomplished by means of a flange on the delivery end of the screw housing which attaches to a matching flange on the fluidizing chamber.

The 2-inch-diameter screw, 16.75 inches long, is housed in a seamless steel tube slightly larger than 2 inches in inside diameter. The screw, a conventional stoker type, has one bearing at its intake end. It ends one-half flight or 1 inch short of the inner face of the fluidizing cone. This provides a 1-inch layer of ore to shield the screw from direct heat radiation in the fluidized bed. A type 446 stainless steel rod, 1.5 inches long and 0.25 inch in diameter, is welded to the end of the screw to provide agitation in the entry port. This rod can be seen in figure 7.

The chain-driven screw is powered by an electric motor through a variable-speed hydraulic transmission. The chain gear is held in place on the screw shaft by means of a shear pin that prevents possible damage to the variable-speed transmission or electric motor if the screw should fuse during operation.

A hopper with a gastight lid is fixed to the deck or mezzanine floor above the feeder. The ore flows from the hopper by gravity through a steel tube of 2-inch inside diameter, a ball valve, and a flexible rubber connecting sleeve to the 18-inch sight glass on the screw feeder. An electrically powered vibrator is attached to the steel tube below the hopper to assure uniform flow of ore to the screw.

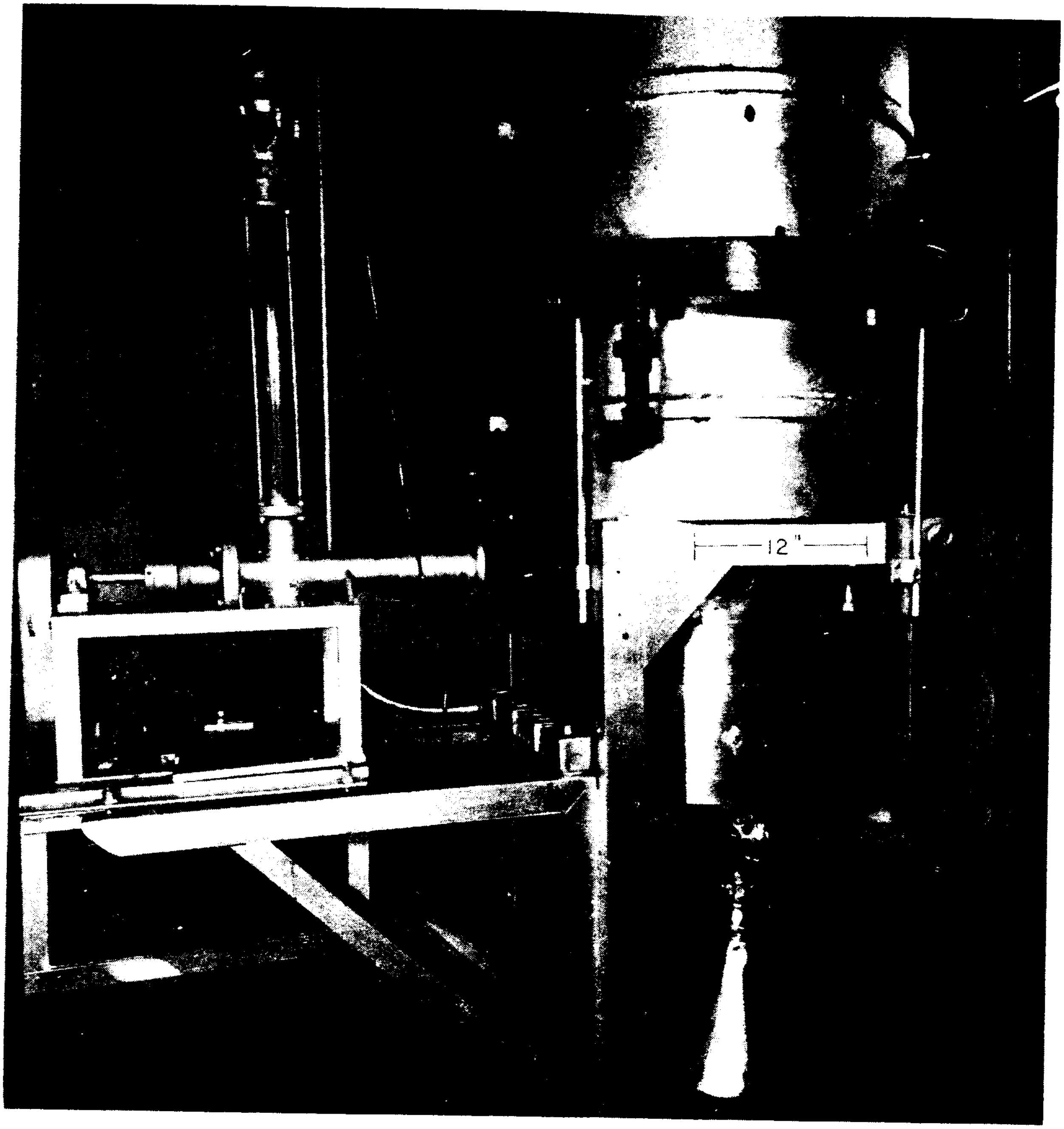

FIGURE 7. - Base of Reactor.

External Firing System

The external firing system capable of supplying up to 300,000 Btu/hr to the unit is shown in place on the reactor in figure 7. It consists of four natural gas burners which jet through the magnesite base support into the annular heating space surrounding the reactor tube. All four burners are supplied with natural gas and air from a manifold encircling the reactor 9 inches above the burners. The manifold was constructed from 12-gage sheet iron with a 3-inch-square cross section. The fuel mixtures are pumped to the manifold by an automatic-temperature-controlled proportioning pump.

A pilot flame is attached to each of the four burners. A thermocouple located in the flame of one pilot actuates an automatic shutoff valve on the fuel pump if the pilot flame is extinguished. This is a necessary safety feature because the pump intermittently supplies fuel to the reactor under the regulation of an "on-off" automatic controller. During the work it was revealed that the combustion chamber was too small when gas was burned with air at the maximum rate. The excessive turbulence created in the combustion chamber under this condition resulted in irregular external heating of the reactor tube. However, this inadequacy in the design and construction can be corrected readily. Probably it would be resolved if the diameter of the annular space between the reactor tube and the cast magnesite combustion chamber were increased and the face of the first course of bricks in the insulating mantle tapered from top to bottom to match the increased diameter.

Dust Recovery System

Duplicate dust recovery units, complete with separate ducts from the reactor tube, are installed at the top of the reactor. Each unit consists of a cyclone and Fiberglas filter. The dual system prevents the necessity of shutdown if one effluent line should plug during a test run. One recovery unit attached to the reactor is shown in figure 8.

The effluent gas ducts are set into the silicon carbide disk which closes the top of the reactor tube and sealed in place with a refractory cement. Each duct or pipe is 18 inches long and 1.5 inches in diameter. The pipe is type 446 stainless steel. A 5-inch section of pipe is welded to the vertical pipe at a 45° angle 3 inches from the upper end. The angular section is threaded for attachment to a fitting on a flexible steel tube which conducts the gas through a lubricated valve to the cyclone. The vertical end of the 18-inch pipe terminates with a pipe nipple serving as a packing gland for a mechanical probe. The probe was installed for cleaning the effluent line in the event of plugging.

The cyclones rest on portable frames of angle iron. The cyclones were constructed from 12-gage type 316 stainless steel. Their design is conventional, with a cylindrical top section 8 inches in diameter and 16 inches high, and with a conical bottom 12.5 inches deep. The internal cylindrical baffle which conducts the relatively dust-free gas upward and out of the cyclone is 4 inches in diameter and 9 inches long. Dust recovered in the cyclone drops through a valve to a canister that is fitted with a cam-type quick coupling to permit exchange during operation of the reactor.

The cyclones are backed by bag filters housed in 32-gallon steel drums. The single-filter element in each drum is a tubular section of woven glass fabric 11.5 inches in diameter and 26 inches in length. The reactor gas after being cleaned of its dust load can be released either to the atmosphere or piped to a recovery process.

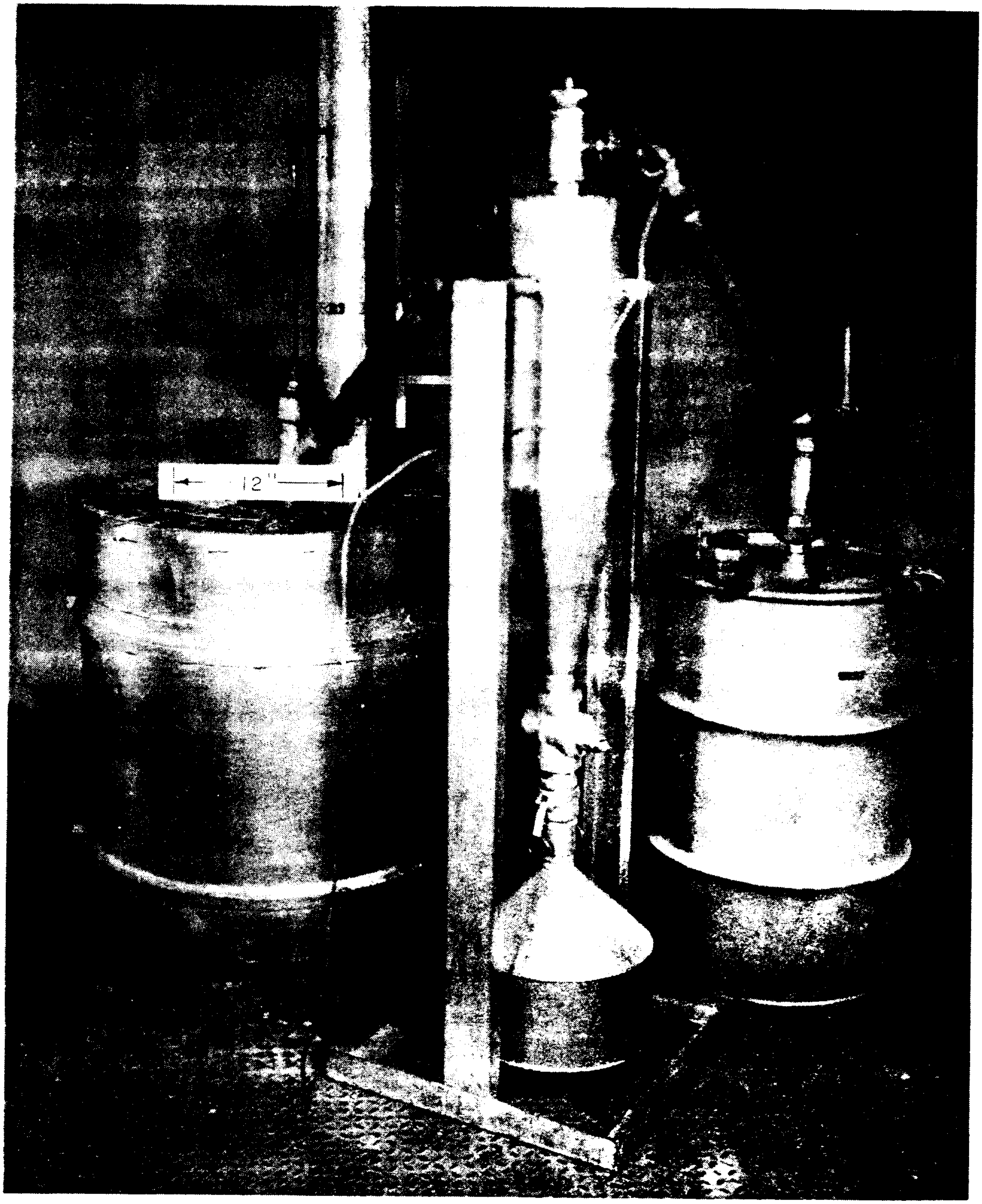

FIGURE 8. - Dust Recovery System.

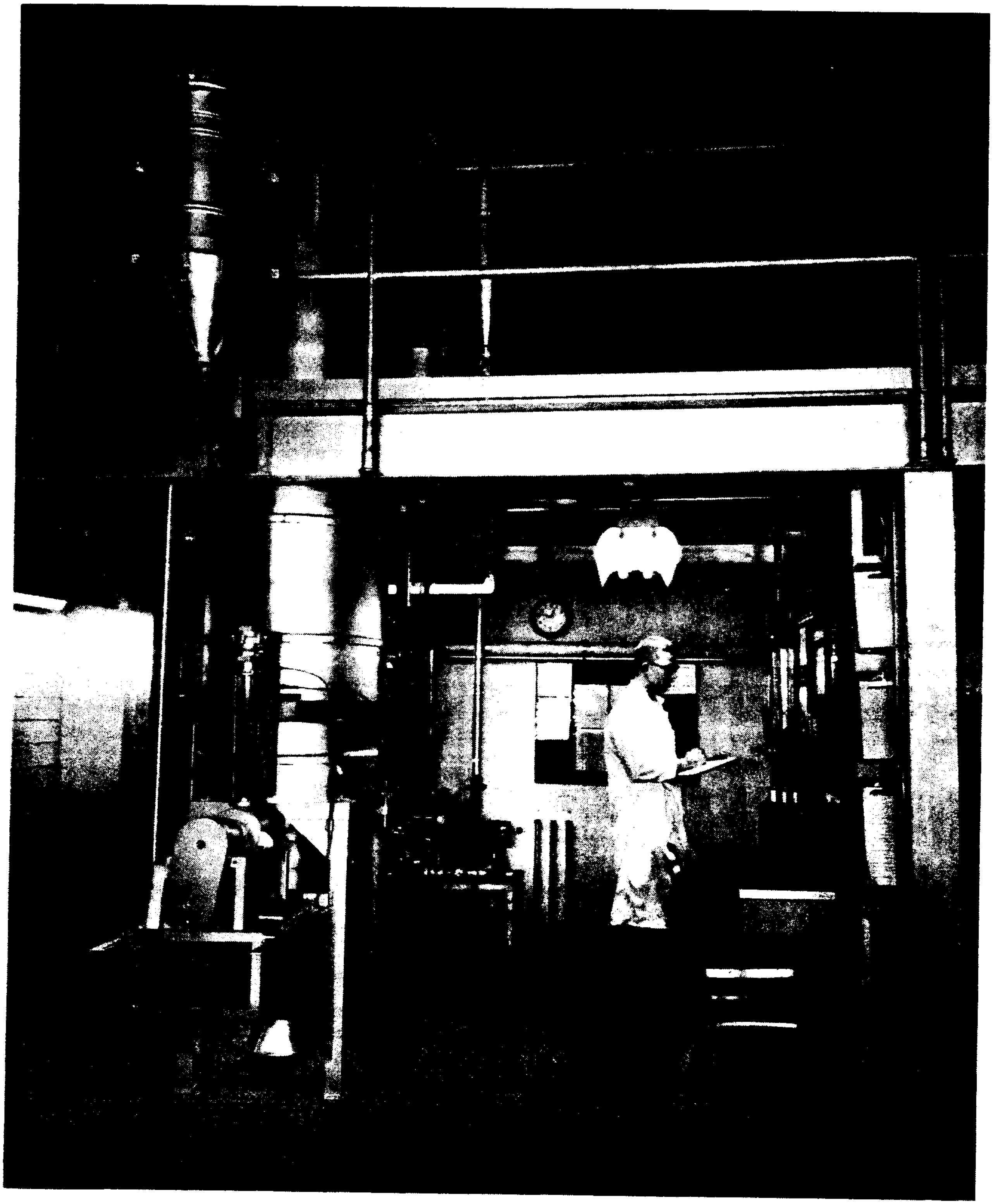

FIGURE 9. - Assembled Reactor and Controls.

Instrumentation and Operating Controls

Instruments and fluidizing gas controls are located on a panelboard near the reactor. This unit is shown in figure 9. A 12-point potentiometric temperature recorder and indicating pyrometer are provided for continuous recording or observation of temperatures during reactor operation. Temperatures are measured by thermocouples which can be placed in the equipment wherever desired as the reactor is assembled.

Three rotameter-type flowmeters are mounted on the panelboard for metering air, natural gas, or any gas being used as the fluidizing medium. There are thermometers and pressure gages at each flowmeter. Needle valves and pressure regulators for flow controls also are located here.

Feed rates are controlled by the variable-speed transmission assembly between the motor and screw on the screw feeder. The feeder is standardized by correlating screw revolutions per minute with the feed rate for any given material.

External firing of the reactor is regulated by an automatic controller operated in conjunction with a thermocouple. The thermocouple is placed on the wall of the reaction tube in the combustion area. The controller actuates an on-off switch on the fuel pump as previously stated in "External Firing System." This equipment combination can maintain a selected temperature or follow a program of temperature change determined by the operator.

Pressure differentials between various parts of the reactor and the atmosphere can be measured by four manometers, also located on the panelboard. The manometer lines are purged with air at the rate of 1 cu ft per min to prevent plugging by ore particles.

CONCLUSIONS

A versatile fluidized-bed reactor has been described. The unit can be used for internal and/or external firing on a wide variety of substances, using either air or controlled atmospheres. The construction of the reactor permits disassembly and cleaning with miminum difficulty. The design is recommended for laboratories engaged in experimental work under variable conditions. The unit may be inspected at Salt Lake City by anyone interested in additional construction details.