Analysis of Products

Infrared analyses of fractions of the paraffin-naphthene fractions of coal-hydrogenation light oil have confirmed previous analyses based on boiling range, refractive index, etc.

Further work was done on the separation and identification of the phenolic compounds in coal-hydrogenation oil.

The countercurrent distribution technique, which was developed for the separation and analysis of very small quantities of closely related compounds, such as antimalarial drugs and penicillin preparations, is being applied to the problem of determining the composition of complex tar-acid mixtures found in the heavy-oil product from the hydrogenation of coal (see fig. 39). In this procedure, a mixture of phenols is distributed between two immiscible solvents in successive countercurrent stages until separation into its components is achieved. In this way, it is possible to isolate individual or groups of closely related tar acids. Thus, for example, mixtures of C_8 tar acids, consisting of xylenols and ethylphenols, were separated. This technique is particularly valuable for mixtures, such as m- and p-cresol, that cannot be separated by other known physical methods.

Countercurrent distribution analysis has indicated that five compounds are present in the C_{10} tar-acid fraction of coal-hydrogenation oil. Further application on a larger scale has so far resulted in the concentration and isolation of one of the components in crystalline form.

As some of these components are present in substantial amounts in coal-hydrogenation oils, it is expected that the possibility for industrial-chemical utilization of tar acids from coal hydrogenation will be more clearly outlined by this research.

Separation of Constituents of Synthetic-Liquid Fuels

Installation of batch-distillation equipment has been completed (see figs. 40 and 41). Batches of 50 to 500 gallons of oil can be distilled in this equipment to provide large samples of gasoline Diesel oil or byproduct tar acids for practical tests and for precision separations to be conducted by the distillation group of the Organic Chemistry Section. The latter has been engaged in developing standards for vacuum distillation and in designing and erecting a number of precision stills. The service manifold for these stills is almost completed (fig. 42).

Analytical Control Laboratories

Analytical control work for the synthetic-liquid-fuels laboratory and pilot-plant operations is done in two standard-type laboratories (see fig. 43). Most of the gas analyses of the branch are made on the mass spectrometer. The number of such analyses frequently is 500 to 600 per month. To decrease the load on the existing mass spectrometer, a much smaller and less expensive mass spectrometer is under construction that can be used satisfactorily for analyses up to a molecular weight of 44. In this way, some time will be made available on the larger instrument for special research problem analyses.

Vortex Combustor Studies for Powdered-Coal Gasification

The construction of a vortex-type reactor and accessory equipment for studying the gasification of powdered coal by steam and oxygen was completed, and the unit was put into operation in March 1948.

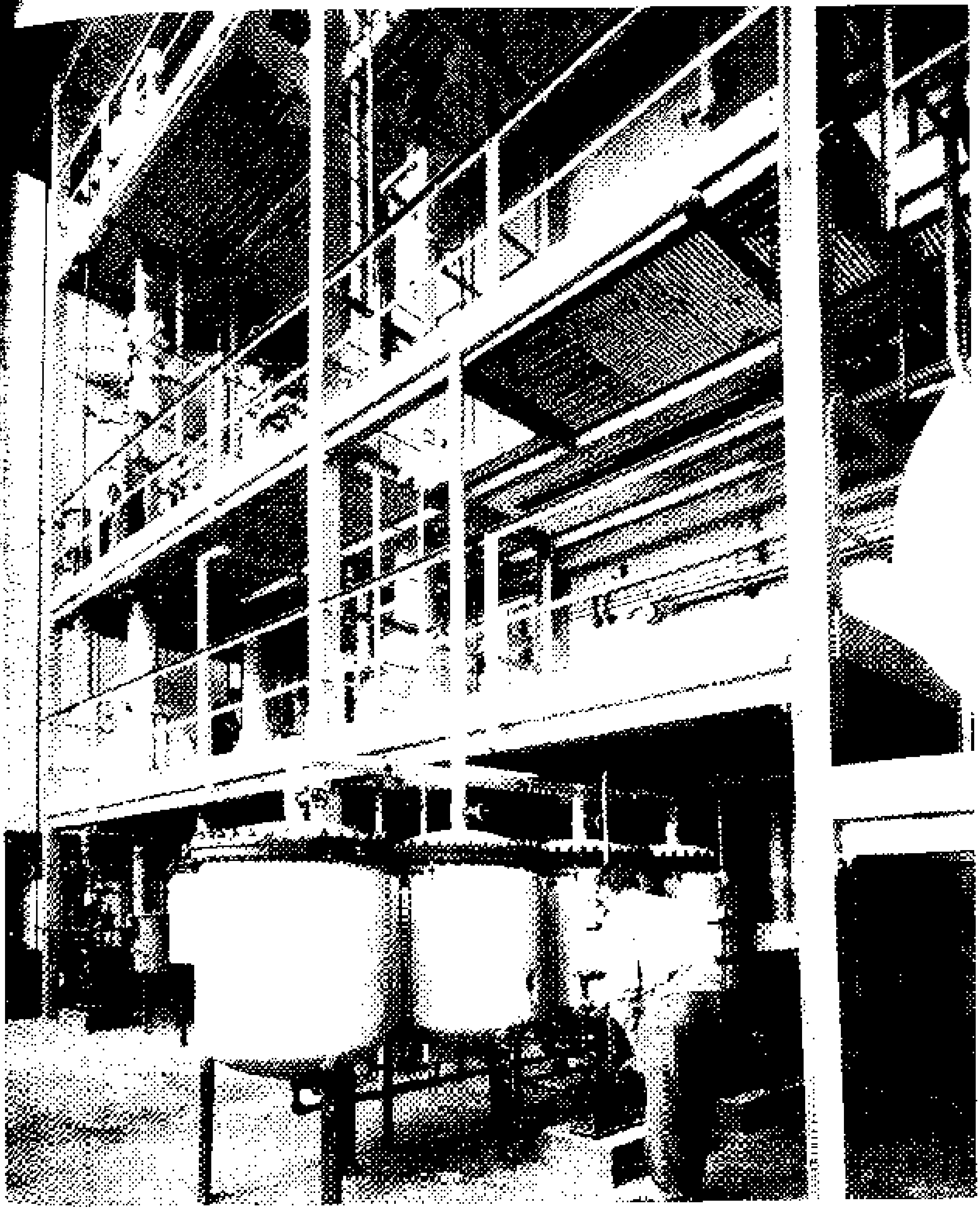

Figure 40. - Batch-distillation equipment (50- to 100-gallon).

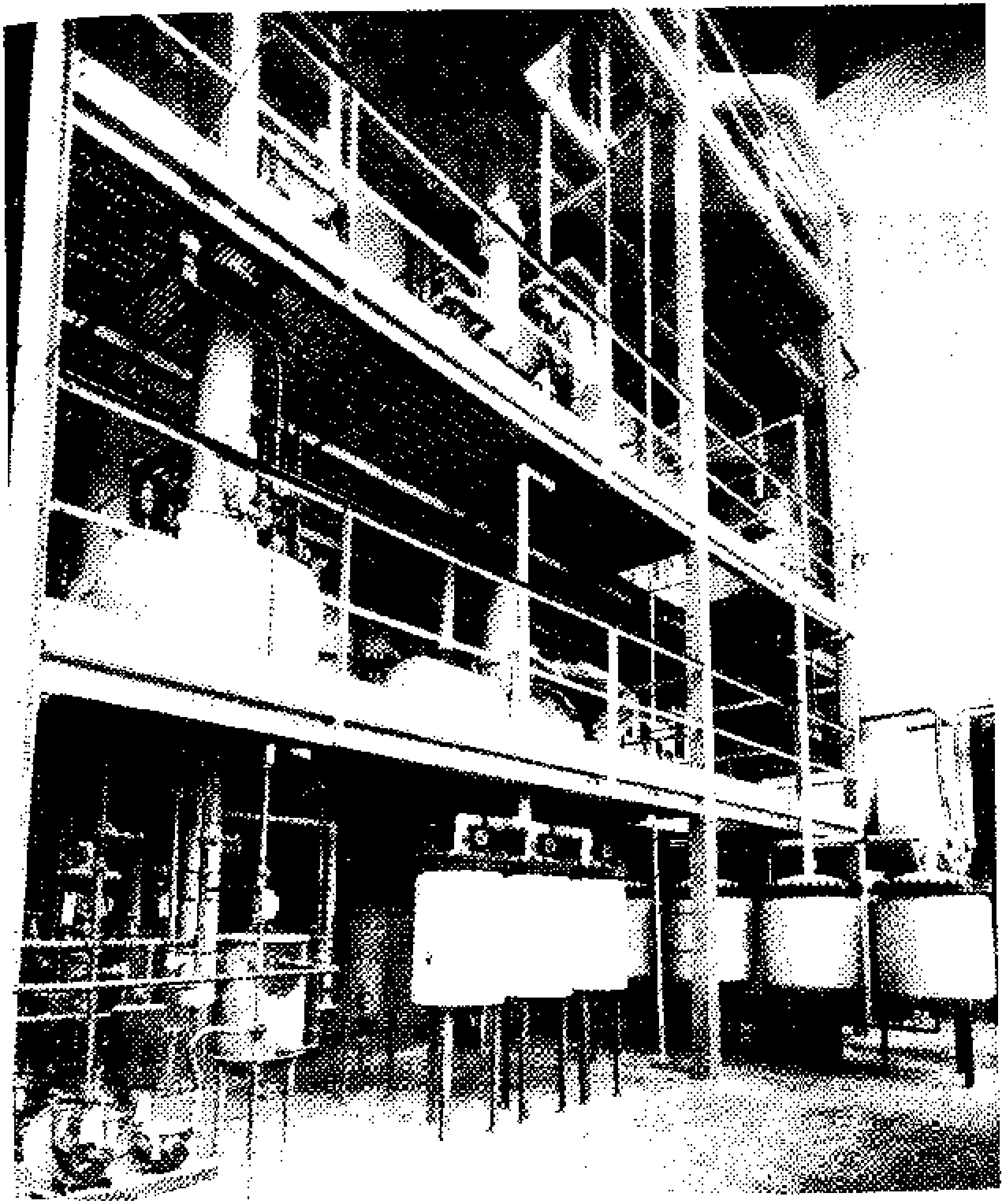

Figure 41. - Batch-distillation equipment (100- to 500-gallon).

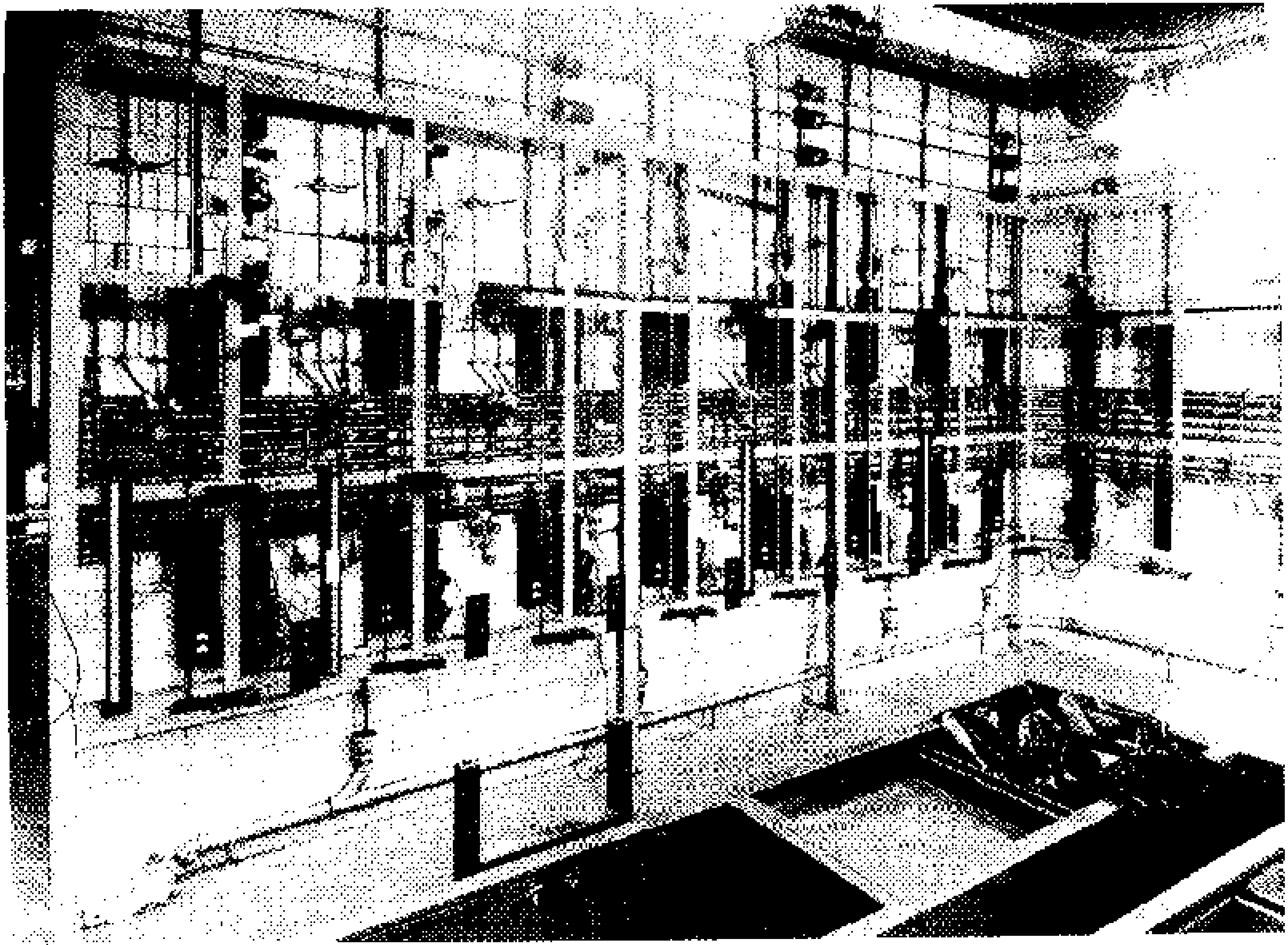

Figure 42. - Service manifold for precision-distillation columns.

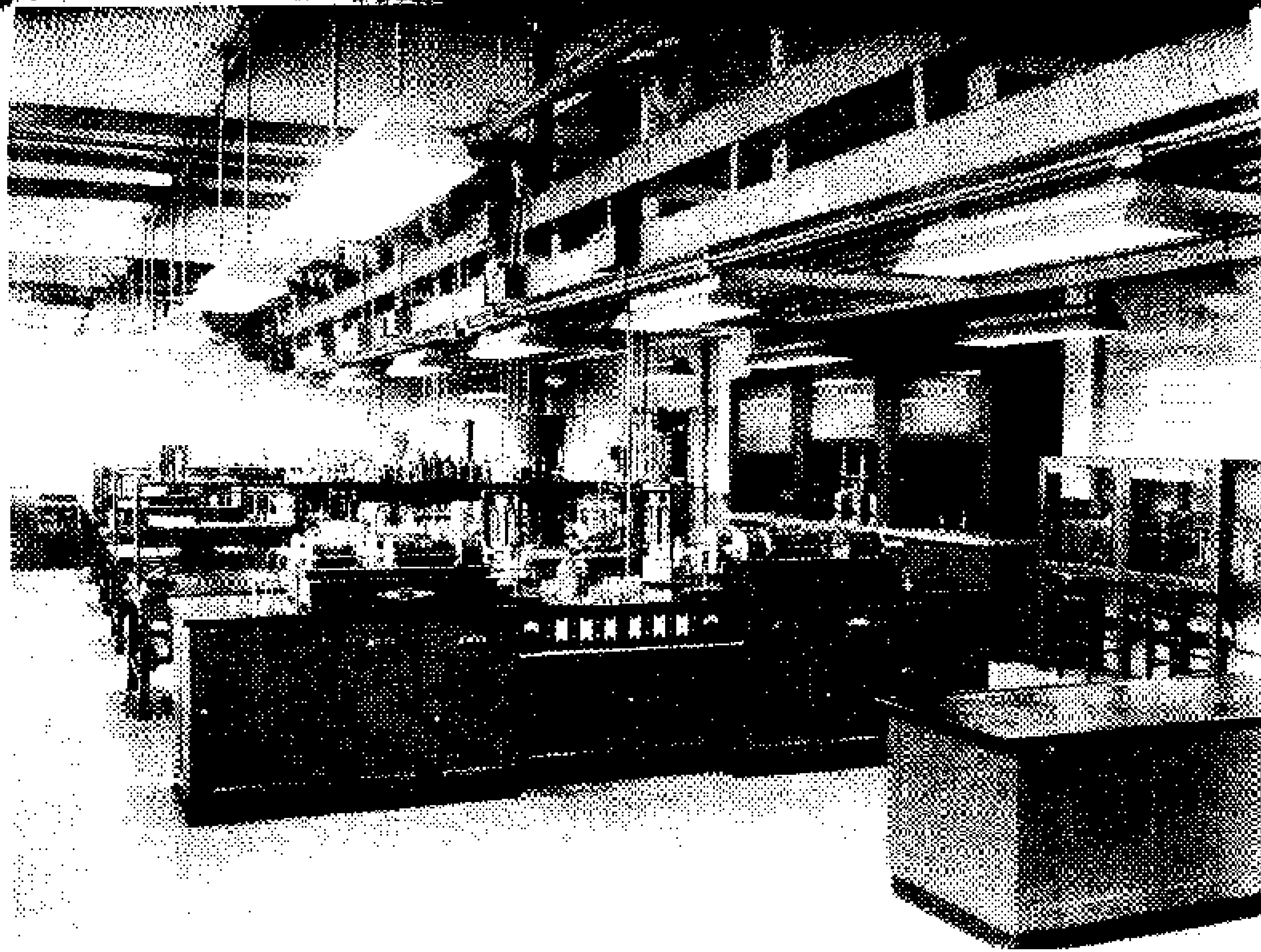

Figure 43. - Analytical control laboratory.

The vortex reactor is a cylindrical chamber having an internal diameter of 2 feet and a depth of 8 inches. Oxygen and steam enter tangentially at the periphery through refractory guide vanes. Coal is fed into the rotating gas stream from the top of the reactor and distributed uniformly around the circumference of the reactor by dropping from the inside edge of a conical distributor. Reaction products leave the chamber through a bottom opening 4 inches in diameter.

The hydrodynamics of the vortex reactor are such that, as a particle reacts and becomes smaller, it moves toward the center. This affords a means for operating at slagging temperatures in a portion of the unit without deposition of slag on the walls.

Experience gained in the initial stages of operation of the vortex has shown that it is desirable to introduce the coal horizontally so that it is traveling with the rotating gas stream. In the present design, the rotating gas stream must change the direction of motion of a coal particle from vertically downward to horizontal. In providing conditions to accomplish this, undesirable limitations are placed upon both design and operating variables. To overcome this difficulty, a method for introducing coal horizontally into the rotating gas stream has been developed and will be installed.

Because of coal-feeding difficulties, a systematic study of operating variables has not been possible. Test runs under uniform operating conditions have been made for periods up to 2 hours. In most of these tests, the temperature in the reactor exceeded the softening temperature of the ash, but in only one instance was slag found on the bottom of the reactor. This occurred when the exit-gas temperature was 2,800° F. These preliminary results indicate that difficulties with slagging in the reactor may not be severe. However, tests of much longer duration are necessary to establish this point.

Technical Reports and Foreign Document Work

The technical reports and foreign document group continued reviewing and abstracting current literature pertaining to synthetic liquid fuels and publishing the results in the form of a bimonthly abstract journal. Exhaustive literature and patent surveys on synthetic-liquid-fuel processes are in progress. That on coal hydrogenation is about 80 percent, and the one on the gas-synthesis processes is about 25 percent completed. Inquiries and service to industrial groups interested in foreign documents on synthetic liquid fuels consume an appreciable and continuously increasing fraction of the time of this group.

A more detailed review of the captured German documents on the Fischer-Tropsch and related documents is being made and all information useful for our research program abstracted. Detailed data on pilot-plant operations of 1,000 to 6,000 hours duration, using iron catalysts and laboratory experiments with different precipitated-iron catalysts pretreated in various ways, have been particularly helpful.

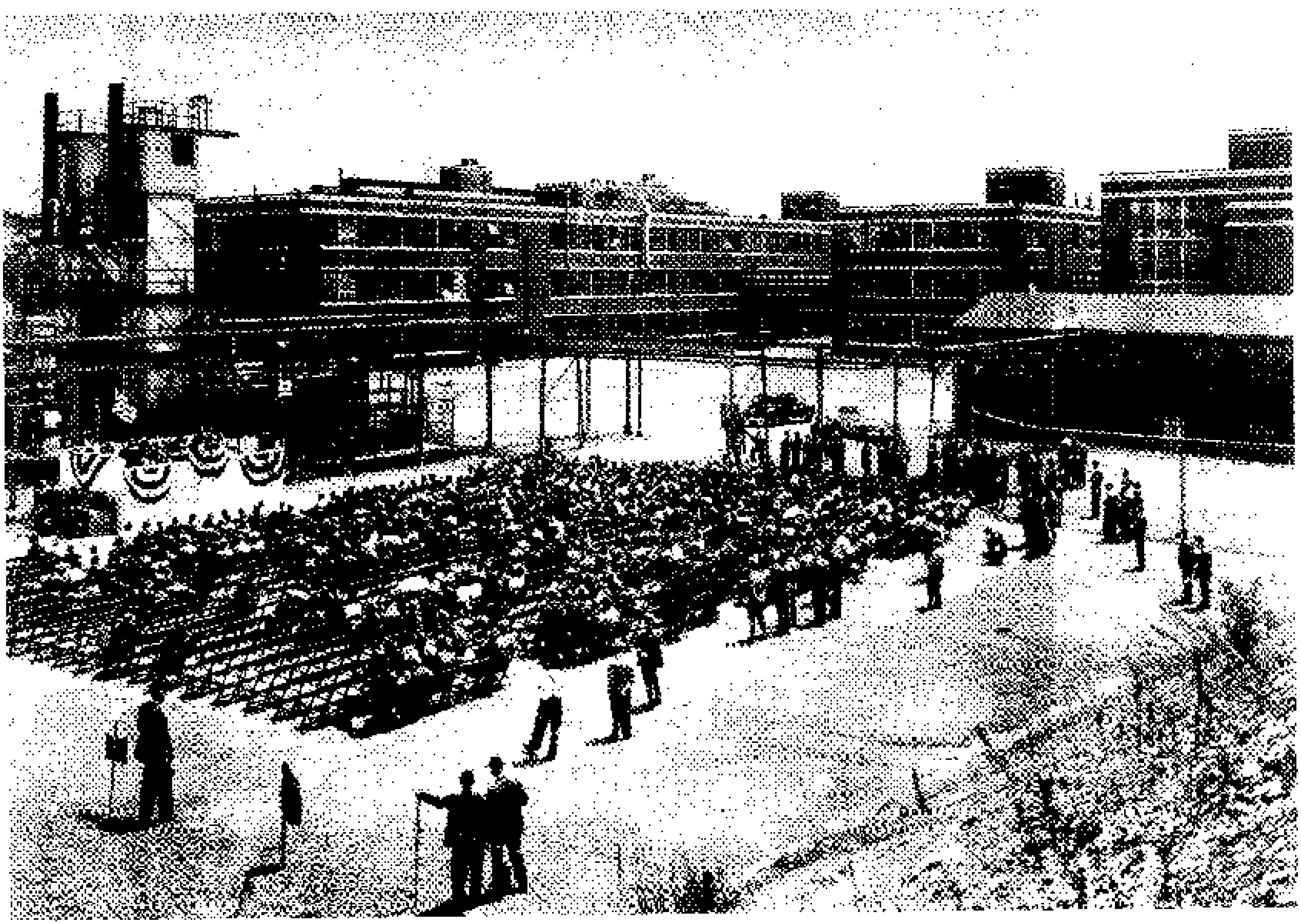

Figure 44. - Scene at Bruceton dedication, May 1948.

Dedication Ceremonies

Dedication ceremonies for the Synthetic Liquid Fuels Laboratories at Bruceton, Pa., were held on May 21, 1948 (see fig. 44).

Outline of Research Programs

(A) Coal Hydrogenation

(1) Laboratory research on the functions of oil vehicles or solvents and catalysts in the first stages of the conversion of coal to oil, covering a range of temperatures, pressures, and rank of coal.

(2) Study of new processes or improvements suggested by the results of (1). This study usually is conducted in bench-scale (relatively small) pilot plants.

(3) Pilot-plant operations (capacity 1 to 100 gallons per day):

(a) Problems of immediate importance to the proposed operation of a 200-barrel-per-day plant at Louisiana, Mo.

(b) New processes developed from results of (1) and (2).

(4) Separation and identification of products of coal hydrogenation.

(B) Gas Synthesis

(1) Reaction-mechanism studies.

(2) Measurement of physical properties of catalysts, namely, X-ray diffraction studies, magnetochemical constants, surface areas, pore volume, pore-size distribution, adsorption of reactants, adsorption of products.

(3) Catalyst activity and durability tests.

(4) Measurements of importance in plant design, such as heat transfer in packed columns and fluidized beds and influence of gas-to-liquid interface in the oil-catalyst slurry process.

(5) Study of new processes or improvements suggested by results of (1) to (4), in relatively small pilot plants.

(6) Pilot-plant operation (capacity, 1 to 100 gallons per day).

(7) Separation and identification of products of hydrogenation of carbon monoxide.