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12 Heavy-Water Production

Savannah River's pioneers in heavy-water research and production have good reason to characterize the history of heavy-water production at the two Du Pont plants—Savannah River and the Dana plant—as a historical saga.[1] As the following narrative shows, the drama of discovery, ardent experimentation, dangerous working conditions, critical need, and achievement all play a role in the history of heavy-water production at Savannah River.

Deuterium was discovered by Harold Urey and his co-workers at Columbia University in 1931. Courtesy of Argonne National Laboratory Archives, managed and operated by The University of Chicago for the U.S. Department of Energy under Contract No. W-31-109-ENG-38, negative 201-2016.

HEAVY WATER AND ISOTOPIC SEPARATION

H_2O, the water that we drink, is composed of two atoms of hydrogen (H) and one atom of oxygen (O). Hydrogen is the smallest and simplest of all the chemical elements and is assigned an atomic mass or weight of 1. Despite its size and simplicity, hydrogen has three forms, or isotopes, that act the same but have different masses. The most common form of hydrogen found in drinking water has an atomic mass of 1. However, in 1 of about 5000 hydrogen atoms, the atomic mass is 2. This heavy hydrogen atom is identified as deuterium, with the symbol D. Tritium, the third known isotope of hydrogen, has an atomic weight of 3 and a symbol of T.

When deuterium is combined with oxygen, the result is heavy water, chemically deuterium oxide or D_2O. Heavy water looks no different than plain water and we encounter it naturally in small quantities. Each time a person uses 52 gallons of water, an ounce of heavy water is present, and there is a pound of heavy water in every three tons of plain water.[2]

The fact that deuterium is so rare in nature made the recovery of large quantities of heavy water a difficult and expensive proposition. Because isotopes of an element have essentially the same chemical properties, common methods were not applicable for separating deuterium from regular water. Scientists instead looked to chemical differences between the isotopes and developed methods based on quantitative differences in reaction rates and reaction equilibrium constants to identify workable separation processes. In their favor was the fact that the physical and chemical differences between hydrogen and deuterium were relatively large, due to their twofold mass

ratio. Still, a great deal of research and development was needed to identify the process that would be both safe and economically feasible to produce heavy water on an industrial scale.

Educating the Public about Heavy Water

"Heavy water, which looks just like ordinary water, has an important part in the operation of certain types of reactors or atomic furnaces.

To understand, a look at the neutron is first necessary.

With the exception of simple hydrogen, all atoms occurring in nature are built of neutrons, protons, and electrons. Because it can transmute or change one element to another, a neutron has the leading role in the operation of reactors.

Neutrons, electrically neutral, serve as projectiles or bullets in piercing the hearts of atoms to cause abrupt internal changes. When a reactor has been loaded with fuel—uranium—and placed in operation, neutrons go flying out of the center of the atoms of uranium-235, flow though a "moderator" that surrounds the uranium slugs, enters other slugs of uranium, and wind up in the nuclei of atoms of uranium-238. This makes new heavier atoms of uranium. These atoms go through a series of internal changes to adjust themselves to the extra weight and finally become plutonium, which is useful in an atomic bomb. Reactors are also used to produce changes in the internal structure of stable atoms of many elements, which then emit radiation useful in medicine, industry, and agriculture.

Generally the more neutrons available in a reactor, the greater is the rate of splitting of the U-235 atoms, with consequent release of greater amounts of energy, and greater production of plutonium and other products.

Since U-235 is a scarce commodity, neutrons are precious. So the reactor is built in such a way that as few neutrons as possible are wasted. And materials are used that soak up as few of them as possible—the more busy neutrons there are, the cheaper and more efficient the reactor is in producing the materials desired.

Besides the fuel (uranium), the moderator is another main component of a reactor. The moderator brings us back to heavy water. Here's why:

When neutrons are emitted from uranium, they have very high velocities. It is necessary to have their velocities considerably reduced before they can be efficiently used. A bridle must be put upon them. If not, they bypass the U-235 and scatter about. This slowing down is accomplished by the moderator, which is placed around the uranium fuel. The neutrons go through many collisions with the atoms of the moderators. In each collision the neutrons reduce their velocity until finally they attain the proper velocity for greater efficiency. Two of the most used moderators in production reactors are graphite and heavy water. Graphite is used in the atomic energy installation at Hanford, Washington. Graphite is composed of carbon, the same substance that ordinary coal is made of. However, it is denser and purer than coal and has a different crystal structure.

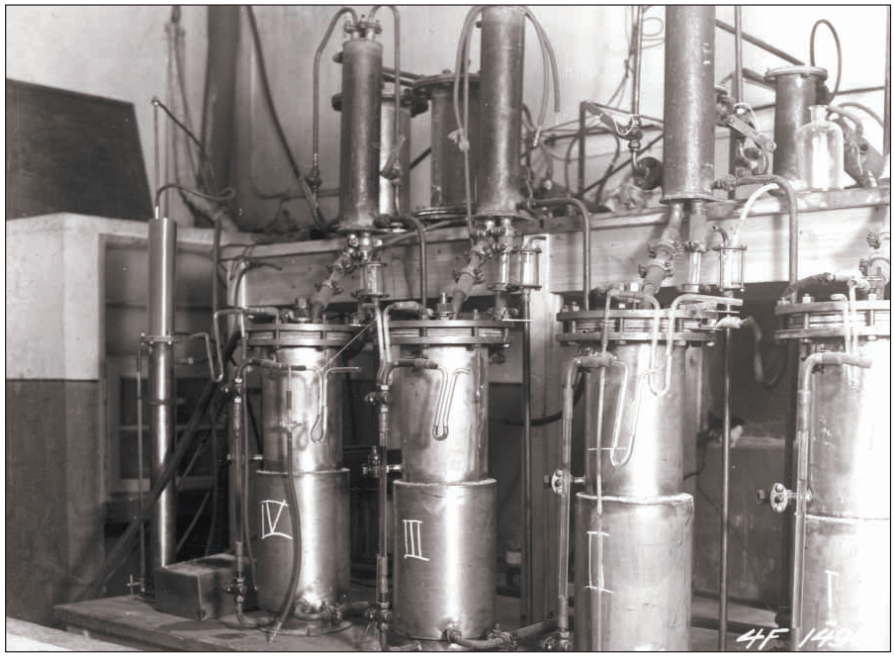
It takes over 6,000 gallons of water to make one gallon of heavy water.

Heavy water is more efficient than graphite as a moderator because it slows down neutrons more quickly and also absorbs fewer neutrons in the process, giving a greater supply of the particles."

Excerpted from: Atomic Energy Commission Press Release on Heavy Water (No. 83), *SRP News and Views*, April 16,1955.

PREVIOUS RESEARCH

Harold Urey and his co-workers at Columbia University discovered deuterium in 1931. American research and development into economical separation of deuterium from light water was conducted at Columbia University during the 1940s. The first rough separation was by fractional evaporation of liquid hydrogen. Later, G. N. Lewis, with his research assistant Ronald T. MacDonald of the University of California, used electrolysis



Electrolysis plant at Vemork under German control during World War II. In 1943, the high concentration plant's heavy water cells and stock were successfully demolished by saboteurs, actually ski commandos of Norway's Linge Company, to slow Germany's progress on its "uranium project." Courtesy of Norsk Industriarbeidermuseum, Vemork, negative UF-131.

The original heavy-water, high concentration cells at Vemork, Norway. Norsk Hydro plant in Vemork, Norway, above the Maane River gorge, was the world's largest electrolytic hydrogen plant in the 1930s. By mid-1934, plant apparatus was modified to produce heavy water that was concentrated to better than 99% purity. Courtesy of Norsk Industriarbeidermuseum, Vemork, negative UF-113.

to concentrate deuterium oxide from a large quantity of water.[3] Early heavy-water research was devoted to discovering more about the two isotopes and their compounds and to evaluating deuterium's possible use as a tracer in biochemical processes and chemical reactions. The demand for heavy water was small, but it was produced commercially as a sideline industry within manufacturing plants that were already producing hydrogen and oxygen electrolytically. The Norsk Hydro plant in Vemork, Norway, the largest electrolytic hydrogen plant in the world in the 1930s, was producing heavy water by 1934. Historical documents show that the Norwegian plant shipped heavy water to researchers around the world, including Harold Urey at Columbia.[4]

By 1940, interest in heavy water heightened as scientists began to research its potential as a neutron moderator. A research and development program established at Columbia University under Urey began to investigate how heavy water could be mass-produced. This research, under the auspices of the Office of Scientific Research and Development, was carried out at other American universities and in industrial research laboratories as well.[5] After obtaining funding, Urey set up a pilot plant in Louisiana that was operated by the Standard Oil Company. The pilot plant was charged with studying the liquid–gas phase exchange reaction under high pressure, while Urey worked on developing a catalyst.[6] Heavy-water research continued, but the effort moved from Louisiana to Canada. In a cooperative effort between the United States, Britain, and Canada, Harold Urey worked with Hans von Halban, who was to head a heavy-water research program for the British. The program was located at Trail, British Columbia, near the American–Canadian border. An existing hydro-powered, electrolytic-hydrogen plant for ammonia production was in operation at Trail, owned by Consolidated Mining and Smelting Company of Canada LTD (Cominco). Cominco converted a loop in their ammonia plant for heavy-water production. By mid-June the plant had produced enough deuterium for Enrico Fermi to make measurements which proved that deuterium was an excellent moderator.[7] Unfortunately, the conclusive tests occurred six months after CP-1's success at Stagg Field in Chicago. Once the graphite-moderated reactor went into operation, heavy-water research was "relegated to a secondary role as the moderator for the second generation of nuclear reactors."[8]

Heavy-water production continued at three heavy-water plants, called P-9 plants, operated by Du Pont during the war. Du Pont successfully produced 32 tons of heavy water at their wartime plants. The chemical firm used excess steam from existing Department of War facilities at Morgantown, West Virginia; Childersburg, Alabama; and Newport, Indiana, to operate the plants. An electrolysis unit for final concentration and purification that served all three plants was located at Morgantown.

The Du Pont heavy-water facilities operated for two years, producing collectively 2,400 pounds of 99.8% heavy water each month. Wartime production included vacuum distillation of water followed by electrolysis for final concentration, because these processes offered little risk of failure and the needed materials were available. "The Columbia group and the Du Pont engineers who designed the wartime plants believed that stainless steel would be the only satisfactory material of construction for the H_2S process; the quantities required would have been prohibitive. After contemplating the building of a small demonstration plant, they set the dual-temperature process aside."[9] At the close of the war, sufficient heavy water had been produced for Argonne's planned heavy-water-moderated test reactors, so the Du Pont units were closed down.

Between 1940 and 1945, five primary separation techniques for recovering heavy water had been developed: distillation of hydrogen, fractional diffusion of hydrogen gas, electrolysis of water, distillation of water, and gas–liquid exchange processes (both dual- and single-temperature processes). In 1942, a dual-temperature, single-stage chemical exchange method of concentrating deuterium in an H_2O/H_2S system was completed in glass on a bench scale at the SAM laboratories at Columbia University.[10] Jerome Spevack, working in the Columbia University laboratory program, patented the dual-temperature process at this time and suggested its use with the water–hydrogen sulfide system.[11] Work on the development of the dual-temperature process at Columbia was suspended after

Squaredancing, Towers, and Cascades

"The chemical exchange process of isotopic concentration with which this study is concerned is based on two facts (scientifically established phenomena).

The first is that chemical molecules perform a kind of square dance in which there is an exchange of partners. We think of the oxygen in water as having each hand held by a hydrogen atom, thus H-O-H, with the hydrogen atoms releasing the oxygen and then joining hands with other oxygens while the first oxygen also gets new partners. Since the oxygens prefer not to be alone, only one hydrogen is exchanged at the time, thus:

HOH H + OH HOH HO + H

Hydrogen Sulfide, H_2S, exhibits similar behavior, thus:

HSH H + SH HSH HS + H

If some of the hydrogens are deuterium, some of the water molecules can be represented H-O-D (HDO) or even D-O-D (D_2O)—heavy water.

Now, if we mix H_2O, which contains a low concentration of deuterium atoms, and H_2S which also contains the same concentration of deuterium atoms, the hydrogen and deuterium atoms are continually exchanging partners, a particular H or D being now with an O and later with an S, etc. If the selection of partners were truly random, the exchange phenomenon would not alter the concentration of deuterium in either the water or the hydrogen sulfide. If, however, the deuterium should happen to prefer oxygen to sulfur, then after the chemical exchange has gone on for a while, the water would be enriched in deuterium and the hydrogen sulfide would be impoverished in deuterium due to this preference. This actually occurs. A measure of this preference is called the equilibrium constant (K). The existence of this equilibrium constant is the second fact necessary for the chemical exchange process. If there were no preferences, then the equilibrium constant would be unity or 1.000. Where the equilibrium constant is substantially different from unity, as is the case for the H_20/H_2S system, deuterium can be concentrated in water by mixing water and hydrogen sulfide and keeping them in contact so as to promote the chemical exchange phenomenon.

When one of the substances is a liquid and the other is a gas, the chemical exchange process is conveniently carried out by the use of countercurrent flow in fractionating columns. A fractionating column or tower is a well known device for obtaining intimate mixing of fluids by the placing of obstructions called 'plates' in the path of flow of the fluids. In laboratories these columns are small glass units, but in full-scale plants they are metal and may vary in diameter from a few inches to many feet, while several stories (50 feet) in height is not unusual. Usually some of the gas is dissolved or mixed in the liquid or vice versa.

In a system of countercurrent flow of two phases, it is convenient, and on a plant scale it is economical, to use two or more fractionating columns of decreasing size, transferring a portion of the enriched phase from the first column to the smaller second column and so on for as many units as desired. Each of these units is called a 'stage.' This technique is not novel with chemical exchange process for isotopic concentration, but it is an old chemical engineering expedient to achieve economy in capital and operating costs. Such multistage apparatus may be referred to as a 'cascade,' and the transfer of fluids between stages can be called 'cascading.'"

Source: Hagley Museum and Library, Wilmington, Delaware, Records of the Atomic Energy Division, Hugh K. Clark, *A Study of the Evidence Available to E. I. du Pont de Nemours and Company Concerning the Validity of U.S. Patent No. 2,895,803 issued July 21, 1959 to Jerome Spevack, August 30, 1968*, Accession 1957, Series II, Box 6, Folder 19.

1943. While laboratory-scale tests and some corrosion work had been accomplished, a semiworks demonstration of the process, which became known as the GS process, did not progress beyond the planning and design stage.[12]

The selection of heavy water as a moderator for the new reactors for the AEC expansion program in 1950 reintroduced investigations into the three potential production processes that could increase the one part in 7000 of deuterium in plain water by a hundredfold. These were vacuum distillation of water, distillation of liquid hydrogen, and exchange of liquid water with hydrogen sulfide (H_2S) gas in a dual-temperature cycle. The drawbacks on each were studied and compared in the light of the impending events and the availability of materials.[13]

In 1949, the Girdler Corporation, a Louisville, Kentucky-based firm, was asked to study the feasibility of converting the old Du Pont P-9 facility at the Wabash River Ordnance Works (WROW) to a low-pressure dual-temperature production plant under the guidance of the AEC's New York Operations Office. Given that the WROW facility was designed for vacuum operation, it could not be converted into a high-pressure production system. It was estimated that the converted plant could produce 2.5 tons of heavy water per month at lower pressures. In a report (NYO-681), Girdler recommended that a new, more efficient, high-pressure facility could be constructed at the same cost as the conversion of the existing facilities.[14] Girdler also reviewed a report to the AEC (NYO-508) by Jerome S. Spevack, titled "Pilot Plant Testing of the Dual-Temperature Heavy Water Process."[15] Spevack, a member of Urey's group at Columbia University in 1942, had been hired as a consultant by the AEC in 1948 to prepare a report on the dual-temperature process for isotopic concentration. Spevack improved his 1942 design by increasing heat efficiency with interstage cascading.[16] The Girdler review stated that the process suggested by Spevack appeared technically feasible, and it recommended pilot-plant tests. Reuse of the federal property in Indiana and some of the original P-9 facilities would substantially reduce costs that had heretofore been considered prohibitive.[17] If the pilot-plant tests at WROW were successful, then the facility could be expanded.

While Girdler's studies were ongoing, the AEC also funded work with the hydrogen-distillation process, requesting that Hydrocarbon Research, Inc., design a plant based on this process. On March 1, 1950, the commission approved construction of the second pilot plant. In this design, hydrogen gas would be cooled to liquid temperatures and fractional distillation would be used to separate deuterium from the gas.[18]

When Du Pont contracted with the AEC to construct the Savannah River Plant, they evaluated the three methods and decided that the Girdler sulfide (GS) process showed clear advantages, as long as three drawbacks were addressed. These drawbacks were difficulty in process control, the toxicity of the gas, and its tremendous corrosive power. Du Pont's engineers began to work cooperatively with Girdler personnel in the design and later operation of the Dana Plant, which was situated at the WROW. Girdler's primary engineers on the project were R. M. Reed, E. A. Comley and N. Updegraff. In a critical meeting between the two firms in early November of 1950, Du Pont's Explosives Department was represented by V. R. Thayer, D. F. Babcock, W. P. Bebbington, J. B. Tinker, W. H. Holstein, A. J. Schwertfeger, and M. S. Bloomsberg. The Engineering Department sent S. I. Winde, J. M. Hoffman, A. E. Daking, and A. K. Shadduck for design; C. S. Robinson and R. T. Matthews for power; H. E. Houck for electrical; E. B. Showell and C. S. Moore for water treatment; and J. R. Boyer as a specialist on blower

seals.[19] The strength and numbers of the Du Pont team underscored how important the design and construction of the pilot plant was to the overall Savannah River Project.

Girdler had become a subcontractor to Du Pont on November 1, 1950. Its scope of work was to design and initially operate the pilot plant until December 15, 1950, when it would be turned over to Du Pont's operations staff. The main production facilities at Dana were by contract to include six GS units, each with a capacity to produce 40 tons of heavy water per year, a distillation (DW) plant, and an electrolytic (E) plant. This projected output was considered sufficient to put Savannah River's two proposed reactors in operation.

(Right) Aerial of Dana Plant and (Below) Safety Platform, 1952. Source: The Girdler Corporation, *Dana Plant Construction History, Du Pont Project 8987*. (Wilmington, Delaware: E. I. du Pont de Nemours & Co., 1952).

The ordnance works property, located west of Indiana State Highway No. 63 in the east central portion of the WROW reservation, had been the site of one of Du Pont's largest wartime heavy-water production sites.

At this point, Du Pont joined Girdler with primary responsibility for oversight of design and development activities, including operation of the pilot plant to demonstrate operability and process control. Du Pont's experience with hazardous materials gave optimism that dedicated safety procedures and equipment could handle gas toxicity, and an extensive corrosion research program was initiated within the Du Pont Engineering Research Laboratory to confirm and extend preliminary conclusions that conventional materials of construction could be used. Du Pont engineers worked with Girdler on process design and materials of construction. Girdler engineering personnel were very capable and cooperative, and this collaboration worked well. Construction of the pilot plant was nearing completion and about a dozen Du Pont people were transferred there to oversee operations. We were

there on duty in late November 1950, when the Savannah River Site was announced. That heavy-water facility soon became known as the Dana plant, named for the nearby village of Dana, Indiana.[20]

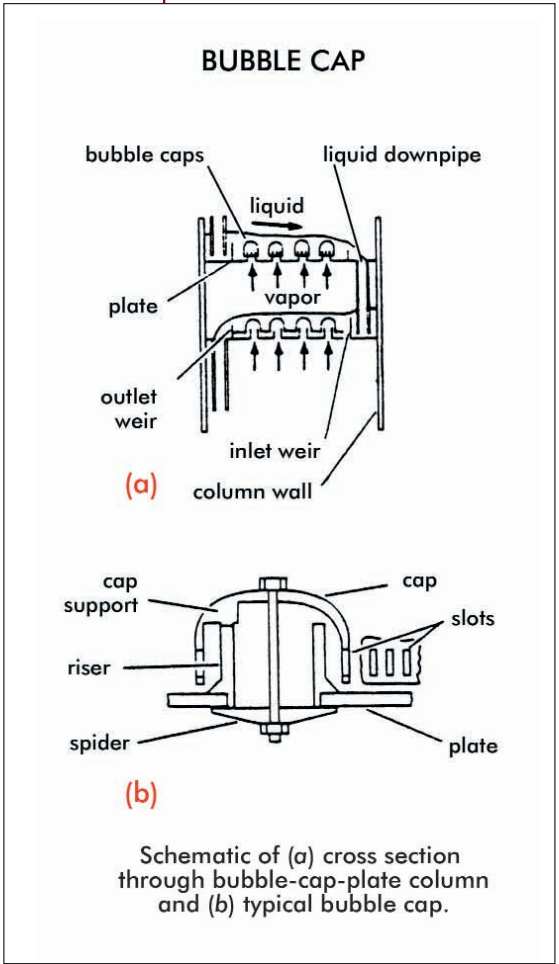
DANA AND THE GIRDLER SULFIDE (GS) PROCESS

Dana's design reflected the concept developed by Girdler under its original contract with the AEC. Water from the Wabash River was fed into six GS units (a total of 96 towers), each having five stages of cold–hot tower systems. The first stage of each unit contained four pairs of parallel hot–cold towers that were 120 feet high, 11 and 12 feet in diameter, and contained 70 separative trays. The second stage had one tower set with the same dimensions as the first stage, and the remaining three stages involved towers of equivalent height but decreasing diameters, 6 feet at the third stage and 2.5 feet at the fifth stage.[21] The gas and liquid streams from the first-stage tower pairs were combined for heating and cooling and then redistributed among the towers.

At the heart of the GS process is a pair of gas–liquid contacting towers, one of which operates at 30 to 35º C (the cold tower) and the other at 120 to 140º C (the hot tower). Ordinary water enters the system, flows downward through the cold tower and then upward through the hot tower, all the while flowing countercurrent to a stream of hydrogen sulfide gas. The towers are stacked with "bubble-cap trays" that force the gas to pass through the water. Deuterium is absorbed from the gas in the cold tower, and stripped from the liquid in the hot tower. Having completed a stage, the concentrated deuterium can be withdrawn from the base of the cold tower and from the top of the hot tower for use as the "product" as is, or can move on to a second stage to be further concentrated. The hydrogen sulfide gas, which is the transport medium for deuterium, circulates in a closed loop within the process.[22]

At Dana, a major problem with the process as envisioned—process control—was solved. For the system to work, very tight control of the ratio of flows of gas and liquid was needed. Bebbington states that the ratio had to be precisely that called for by process theory.[23] Deviation from that control led to poor productivity, and instrumentation at that time was not able to ensure operation of the optimum ratio. Dale F. Babcock, the senior member of Du Pont's task force at Dana, solved this dilemma. Babcock observed that if the concentration of deuterium at the middle plate of the cold tower is the same as at the middle of the hot tower, then the flow ratio was correct. This principle—the comparison of mid-column concentrations as a basis for process control—was used to guide the pilot planning, and the ratio was used regularly during production.[24]

More information about the GS process evolved as the research at Dana moved forward. A first stage was built, composed of two steel towers, each 3 feet in diameter and 110 feet in height with 70 bubble-cap or separative trays. Successful operation of the first unit occurred on October 26, 1950, slightly more than a month before the Savannah River



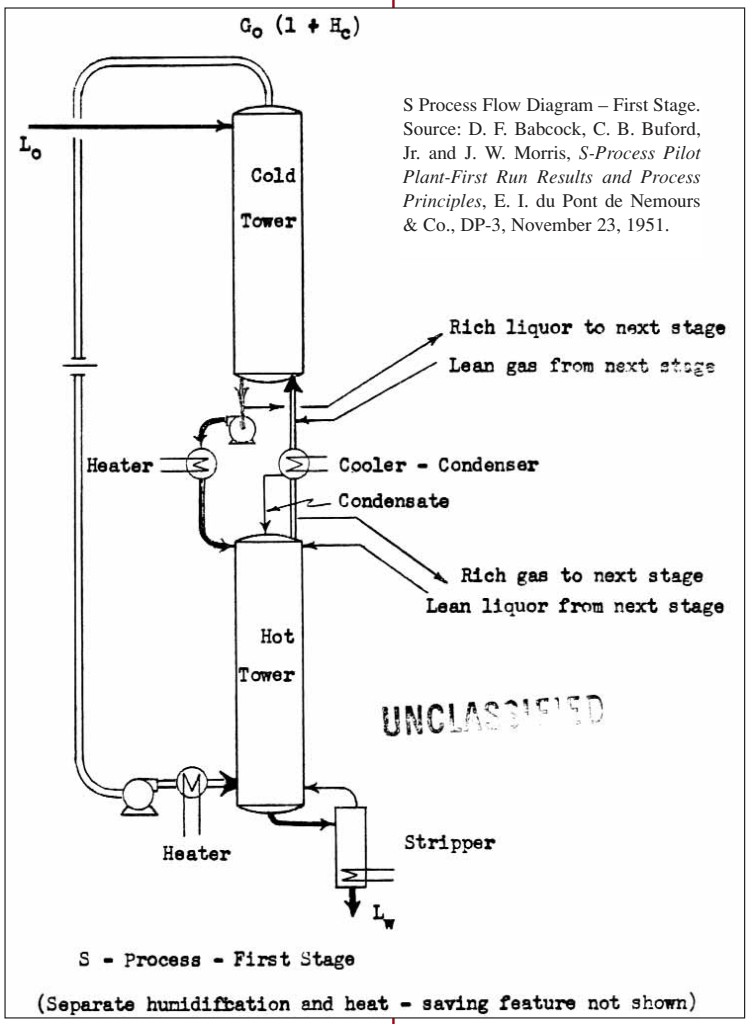
Schematic of (a) cross section through bubble-cap-plate column and (b) typical bubble cap.

Each GS tower was stacked with bubble cap trays that forced the gas to pass through the water. Savannah River's first stage GS towers (24 cold and 24 hot) had 70 trays in each cold tower and 60 in each hot tower. The cold towers were 11 feet in diameter; hot towers were one foot wider. Source (image): *McGraw-Hill Dictionary of Scientific and Technical Terms*, editor Sybil P. Parker, Fourth Edition, New York.

(Opposite Page) 400 Area 1. 412-D, negative 4-419-10; 2. Control Room, 412-D, April, 1957, negative 4121-3; 3. Concentrator Building, 420-D, negative 4-412-5; 4. Finishing Building and "E" Process Cylinder Loading, 421-D and 421-1D, negative 4-389-9; 5. Powerhouse, the largest built at Savannah River, 484-D, negative 698-28; 6. Flare Tower, 419-D, negative 1273-3; 7. Shops, Stores, and Change House, 717-D, negative 4-406-3; 8. Central Laboratory and Supervisor's Office, 772-D, negative 4-382-7.

announcement. At this point, Du Pont's engineers, Dale Babcock, C. B. Buford and J. W. Morris, referred to the GS process as the "S" process.[25] The first 300 hours of operation demonstrated the effectiveness of the Babcock ratio and determined that the bubble-cap trays were at least 45% effective in the two-column test system. "Taken as a whole, the pilot plant operations and data were judged adequate to justify the choice of the GS process for the production plant."[26]

Due to operational considerations and the possibility of the loss of heavy water through high-pressure leaks, it was decided that the GS process would be used to extract deuterium oxide from natural water and concentrate it to 15 to 20%. Vacuum distillation (the DW process used at Du Pont's wartime plants) would further concentrate the heavy water to 90% and a third process, batch electrolysis, would bring it to reactor-grade purity of 99.75%. Morris *et al*. point out that the bulk of the separative work and costs of the heavy-water production processes emanated from establishing and operating the GS process, which had never been attempted on an industrial scale. The other two processes were well known and had been demonstrated before their use at Dana and SRP.[27]

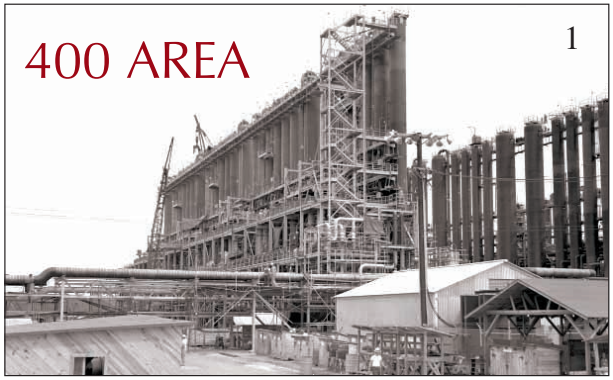


S Process Flow Diagram – First Stage. Source: D. F. Babcock, C. B. Buford, Jr. and J. W. Morris, *S-Process Pilot Plant-First Run Results and Process Principles*, E. I. du Pont de Nemours & Co., DP-3, November 23, 1951.

The first startup of a Dana GS unit in the winter of 1951–1952 was unsuccessful. The cold Indiana weather caused the hydrogen sulfide and water to form a solid hydrate, collapsing the interior trays. Moreover, some of the slotted bubble caps on the trays were broken because they had not been annealed. The corrosive power of the hydrogen sulfide gas also became "painfully evident" when internal roller bearings shattered on both the gas blower, which circulated gas through the towers, and its spare. The cause was hydrogen-sulfide stress-corrosion cracking. A great deal of research would be dedicated to learning about the effect of the gas on metal and the stress the process placed on it. Accordingly, changes were made in procedures and equipment to correct these challenges. By August, the lessons learned in the earlier attempt paid off, as the first of Dana's GS units went into operation.[28]

The experiences at Dana were compelling. Experimental data, coupled with operations information from each of the plants, led to research on construction materials and methods that could be applied to the programs at both plants. The corrosive power of the hydrogen-sulfide gas made this research and development program critical. Carbon steel was used for process vessels and for heat exchanger shells and piping. Tests were performed to find defects before use. The bubble-cap trays in the exchange towers were stainless steel. Carbon steel, "low alloy" steel, and stainless steel had to be heat treated to relieve stresses that were imbedded during fabrication. Bolts posed a special problem. If exposed to a

400 AREA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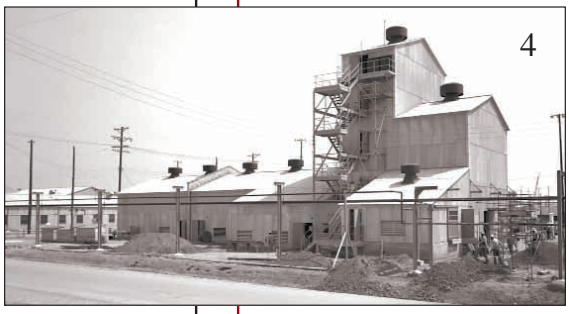
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hydrogen-sulfide leak, the bolt can absorb hydrogen. If it is stressed beyond a certain threshold, it will crack. To avoid this, all bolts were heat treated and installed to a predetermined stress level with torque wrenches. Metal parts in which stress or hardness was necessary were designed in isolation from the gas. Maintenance procedures were devised and minimum thickness holes, 1/8-inch in diameter, partially drilled through a pipe from the outside, were used to better gauge the effects of corrosion and give early warning of problems.[29]

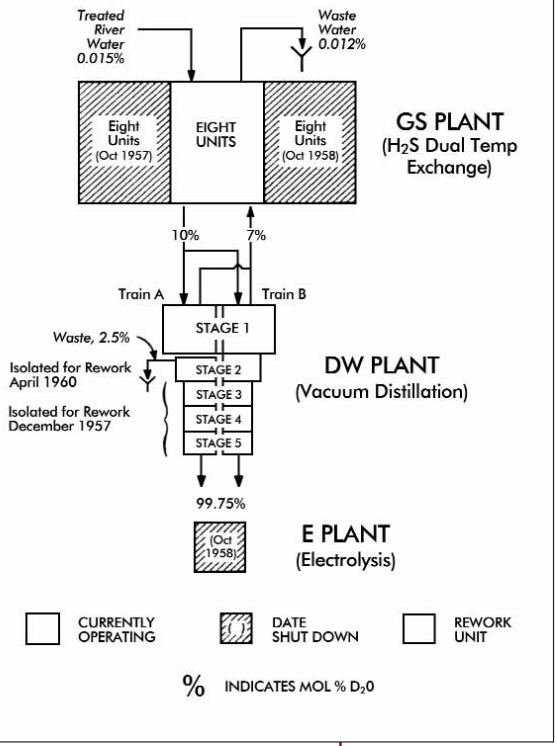


Diagram of D Area Processes, circa 1962. Source: W. P. Bebbington, J. F. Proctor, W. C. Scotten, and V. R. Thayer, "Production of Heavy Water in the United States" in *Proc. Third International Conference on the Peaceful Uses of Atomic Energy, Vol. 12 Nuclear Fuels–III Raw Materials* (New York: United Nations, 1965), 334.

SAVANNAH RIVER'S 400 AREA

The initial estimate of heavy water needed for SRP reactors was increased when the expansion program went from two to five reactors. The Dana Plant's heavy-water production capacity, designed to handle the needs of two heavy-water reactors, was not equal to the task. Also, the original estimate of 150 tons of heavy water per reactor was raised to 200 tons per reactor as the result of a Du Pont study. A second plant was immediately deemed necessary, and that plant became SRP's 400 Area.

SRP's 400 Area received top construction priority because, without heavy water, the reactors could not operate. Du Pont shifted gears, and responded to this critical change in scope by hiring the Lummus Company to design the SRP facilities. They shortened the Lummus Company's learning curve by hiring the Girdler Company as SRP consultants.[30] The facilities at Savannah River included a hydrogen-sulfide generating plant that produced 15 tons a day, an inert gas plant, water treatment facilities, a massive coal-burning powerhouse, and a river pumphouse.

The problems resulting from the operational complexity of Dana's staging led to design changes at Savannah River, principally in eliminating interrelated process flows. The 400-Area GS units were simpler in design and operated independently from each other. Twenty-four first-stage tower units were erected, each with its own second-stage towers that were larger than those at Dana. As a consequence, each second-stage tower at SRP had more than twice as many separative trays as the towers in the Dana design units.

SRP's DW plant, a multi-staged operation, started up in 1952. This second step in the concentration process at SRP included bubble-cap tray towers and was divided into five stages of enrichment and distillation until, in the fifth stage, the product was approximately 98% deuterium oxide. The E process, batch electrolysis, was the final purification step. The concentration of the deuterium oxide solution was raised to 99.8% through electrolysis in this last step. After removing impurities, the D_20 was distilled, an electrolyte was added, and the fluid was fed through a number of cells through which passed a current of 1000 amperes. After this step was completed, the heavy water was stored in aluminum drums, ready for immediate use or later sale.

Savannah River's 400 Area was in operation by May of 1953. In the spring of 1953, a small plant was constructed in the D Area to produce deuterium gas from heavy water by

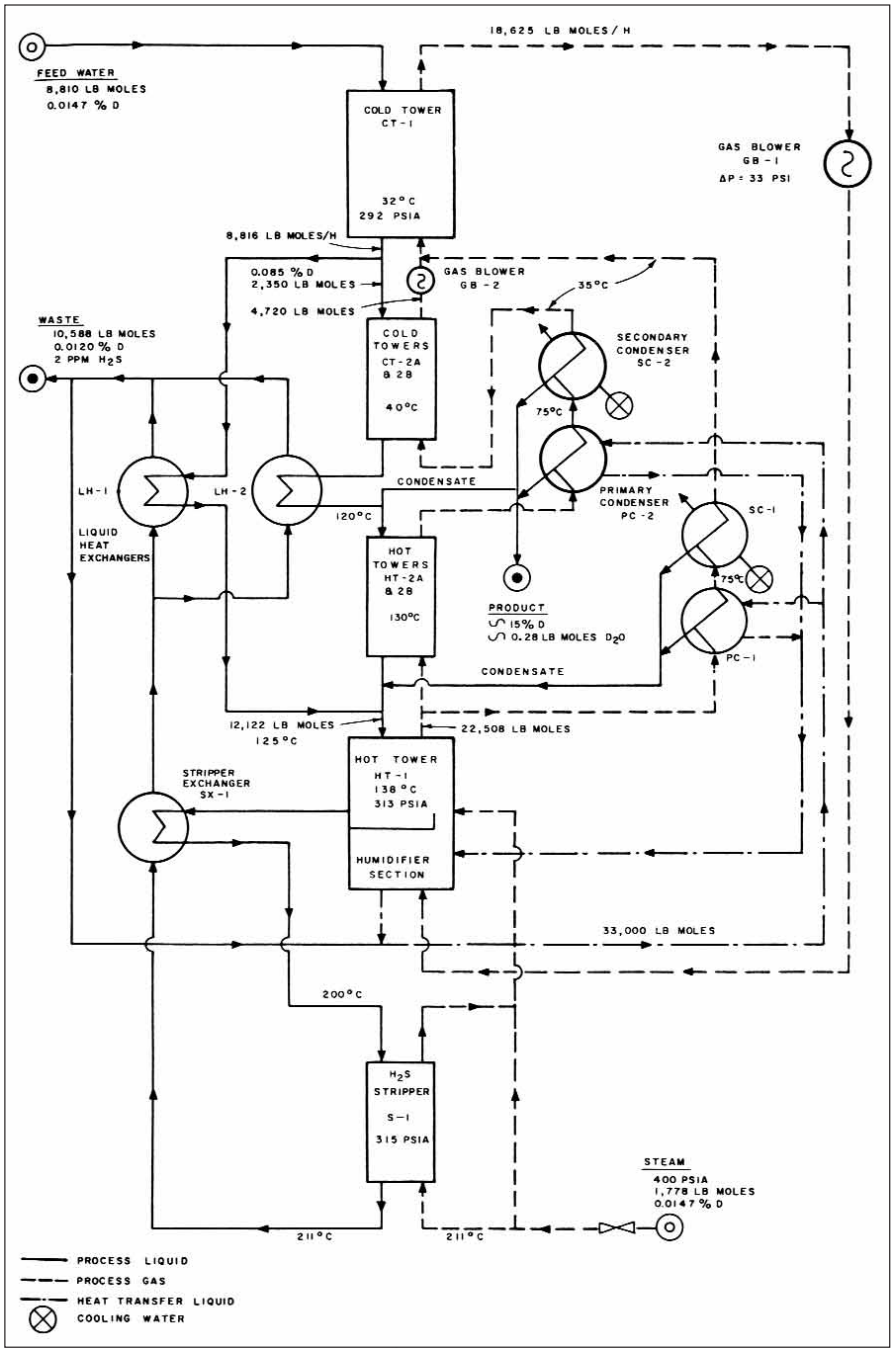
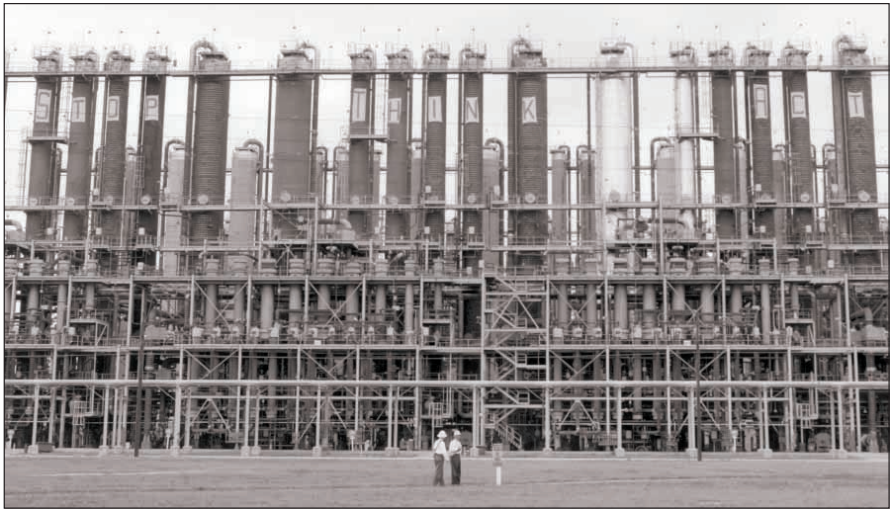


Diagram of the Savannah River GS Process. Source: W. P. Bebbington, J. F. Proctor, W. C. Scotten, and V. R. Thayer, "Production of Heavy Water in the United States" in *Proc. Third International Conference on the Peaceful Uses of Atomic Energy, Vol. 12 Nuclear Fuels–III Raw Materials* (New York: United Nations, 1965), 334.

electrolysis. Some of this deuterium was used at Savannah River in the Tritium Facility (tritium reservoirs were actually filled with a mixture of tritium and deuterium), and some was sent to the Oak Ridge Site to be converted to the lithium deuteride used in the secondary assemblies of thermonuclear weapons. A second, larger, deuterium plant was constructed in the D Area in 1954.[31] A 1962 estimate of the AEC's capital investment in D Area at Savannah River was quoted at $145 million with $110 million, or 75%, of that

total devoted to the piping, towers, heat exchangers, structures, instrumentation, and switch gear involved in the GS process. D Area's steam and electric power plant, the largest of all of Savannah River's power facilities, represented a $15 million investment. The vacuum distillation plant was estimated at $2.5 million, the electrolytic plant at $1.5 million, the H_2S plant at $1 million. The remainder was invested in water treatment, gas storage facilities and flare tower, and general plant facilities.[32]



DANGERS AND HAZARDS

The GS process used at Dana and at Savannah River's 400 Area involved the use of large quantities of hydrogen sulfide. Hydrogen sulfide is an extremely toxic, flammable gas that forms explosive mixtures with the air. Its odor, similar to that of rotten eggs, is noticeable even from small concentrations, and while some concentrations can be tolerated for a short period, contact can prove fatal. Moreover, after prolonged exposure, individuals lose their ability to smell it, thus exacerbating the danger. Leaks in piping, the distillation towers, or the heat exchangers and any other components within the GS units could cause such a release. To prevent and guard against any incidents, a number of safety measures were designed and implemented at both plants.

At Dana, a gas monitoring division was established in July 1951 and an elaborate communication system put in place. Monitors carried devices to detect leaks, and all personnel were well trained in first aid. Evacuation procedures, if needed, would be given from the monitor headquarters that kept tabs on the wind direction.[33] At SRP, monitoring instruments continuously sampled the air and sounded the alarm when the gas was identified.

> Each man carried a small dispenser of H_2S sensitive paper that would reveal presence of the gas even if he could not smell it. All personnel in the area were issued gas masks containing adsorbent carbon, and the workers in the units carried masks that had their own cylinders of compressed breathing air.

D Area experienced the site's only fatalities. Two men, working within a confined space on a GS unit support structure, unplugged a process drain valve and were unable to avoid the hot water and steam that was released. Access openings on the units were enlarged after this tragedy.

The need for vigilance was very real. D Area reported at least two serious releases of hydrogen sulfide due to the failure of a seal on a blower and a failure of a screwed joint in a pipe. Fortunately, neither incident caused any personnel injuries. Author W. P. Bebbington notes that the hydrogen sulfide plant was shut down during early days of operation when two D Area employees were overcome by gas. The plant was shut down until the equipment was fixed despite the urgent need for its product. Source: The Girdler Corporation *Dana Plant Construction History*. Subcontractor for Engineering Department, E. I. du Pont de Nemours & Co., Wilmington, Delaware. Du Pont Project 8987, December 1952, DPEG-17. William P. Bebbington, *History of Du Pont at the Savannah River Plant*, (Wilmington, Delaware: E. I. du Pont de Nemours & Co., 1990).

(Right) STOP THINK ACT, D Area safety signs mounted on GS Towers, 1956. Courtesy of SRS Archives, negative 3970-3.

(Below) Gas monitoring team at work with resuscitator, fully equipped safety carry-all in background.

> Perhaps the most important precaution was procedural. Workers in and near the GS units worked in pairs so that if one man was overcome, the other could summon help, put on his own mask and pull his "buddy" to safety. The buddies were required to stay several feet apart so that both would not enter an area of high concentration simultaneously and both had to have special training. An untrained person entering the GS area had to be accompanied by two trained people.[34]

During operation, when a GS unit needed inspection or maintenance, the hydrogen sulfide gas was returned to the hydrogen sulfide plant in 400 Area where it was compressed and stored in tanks. If an emergency demanded, the gas could be released to a 400-foot flare tower to be ignited by its pilot flame, burned to sulfur dioxide, and released to the atmosphere. The use of these measures gave the 400-Area workers a strong record of safe work performance with no serious injuries from exposure to hydrogen sulfide.[35]

WORKFORCE

From the outset, Dana had its own administrative offices. In December 1950, the AEC's Dana Area Office's three branches were headed by B. C. Samples. H. N. Hinchman worked in administration. C. W. Reilly was the chief of construction, C. E. Williams was responsible for safety, and F. J. Holtzner handled security. E. J. Grabowski and C. A. Konwinski were in the Technical Branch.[36]

The original lead for Girdler's staff was R. M. Reed, the technical director for the Dana Project. R. E. Alexander was project engineer, and the process engineer was E. R. Comley. On November 1, 1950, there were 389 employees on the Girdler payroll. Peak construction employment occurred in July 1951 with 5,111 employees. The force was reduced by over 2,000; subsequently, on the basis of new estimates, a new peak occurred of 5,458 occurred in February 1952. While some Girdler employees stayed to help orient Du Pont's incoming operations personnel, all of the subcontractor's employees left by mid-January 1951.[37]

From July 1951 to December, Du Pont's forces at Dana grew from 594 to 969 individuals. The total operating force at Dana consisted of 926, of whom 170 were supervisory personnel. The largest number of employees, 405 individuals, was in maintenance, underscoring the need for a strong inspection and proactive maintenance program at the GS facilities. Two hundred and thirty were directly involved with the operation of the process facilities, and 63 worked in technical.[38] Don A. Miller was Dana's manager initially. He was transferred over to SRP to manage the plant. R. Paige Kelly took over at Dana for Miller, with J. A. Monier as his assistant plant manager. Kelly remained as manager of the plant until it was closed in 1957. Some of the Dana staff would move over to Savannah River, providing a core group of experienced operating and

D Area Committee in November 1956 included Les Ahrens, GS department superintendent; J. H. Nuzum, area superintendent; S. P. Brown, Captain Herman Caldwell, H. R. Casebolt, L. R. Di Fillipo, H. V. Graybeal, W.H. Keeter, W. S. Marting, H. M. Moore, R. D. Pillsbury, Dr. George A. Poda, R. M. Radford, K. W. Brendell, M. A. Werner, and A. D. Williams. The committee was composed of representatives of various departments that were part of D Area operations. Source: *Savannah River Plant News*, November 9, 1956. (Not all D Area Committee members are shown.)

technical staff at the younger plant. J. W. (Bill) Morris served as technical superintendent at Dana until 1953 when he was assigned to the Savannah River Laboratory, and W. C. Scotten, who was part of Dana's Works Technical in 1951–1952, was transferred to SRP in 1952 to serve in Extraction Area Process Assistance.

At Savannah River, organizational charts show that the GS Department or 400 Area was one of six departments under Du Pont's Production Department. F. H. Endorf was head of the GS Department and J. H. Nuzum was area superintendent in 1954, with A. J. Sauerborn as area superintendent for finishing. A Heavy Water Technology Section, with W. P. Bebbington as superintendent, provided process, mechanical, and operational assistance to the GS Department that held responsibility for 400 Area operations in 1954.

Les G. Ahrens became superintendent of the GS Department in 1956 and J. H. Nuzum remained as the area superintendent. The early workforce of about 900 operations staff was drawn from a number of Du Pont's departments including Reactor and Reactor Materials Works Technical, Health Physics, Instruments, Service, Maintenance, Traffic and Transportation, and Power and Security. Ahrens remained superintendent until 1964 when Ken French, who was also responsible for the Heavy Water Components Test Reactor (HWCTR), was assigned to the GS Department as superintendent.

PRODUCTION

The required quantity of heavy water for each of Savannah River's reactor was 200 tons; the Process Development Pile, a test reactor in the 300 Area, required 110 tons. As the Dana Plant and D Area went into operation, estimates on production levels could be made. On the basis of production figures for Dana, then D Area between May 1952 and March 1953, it was projected that the quantity needed to charge the reactors would not be produced until 1955.[39] This production schedule worked well as the plants, sized to produce the necessary amount of heavy water to charge the reactors, produced sufficient heavy water for each reactor as it was constructed. The staged construction of the five reactors thus was linked to the heavy-water production rate that progressively increased in output.

HEAVY-WATER PRODUCTION, 1952–1953

Date	Dana Plant	D Area	Cumulative combined production (tons)
1952			
May	0.83		0.83
June	2.50		3.33
July	5.33		8.66
August	9.25	0.68	18.59
September	13.80	2.02	34.41
October	15.00	4.93	54.34
November	15.00	7.59	76.93
December	15.00	10.50	102.43

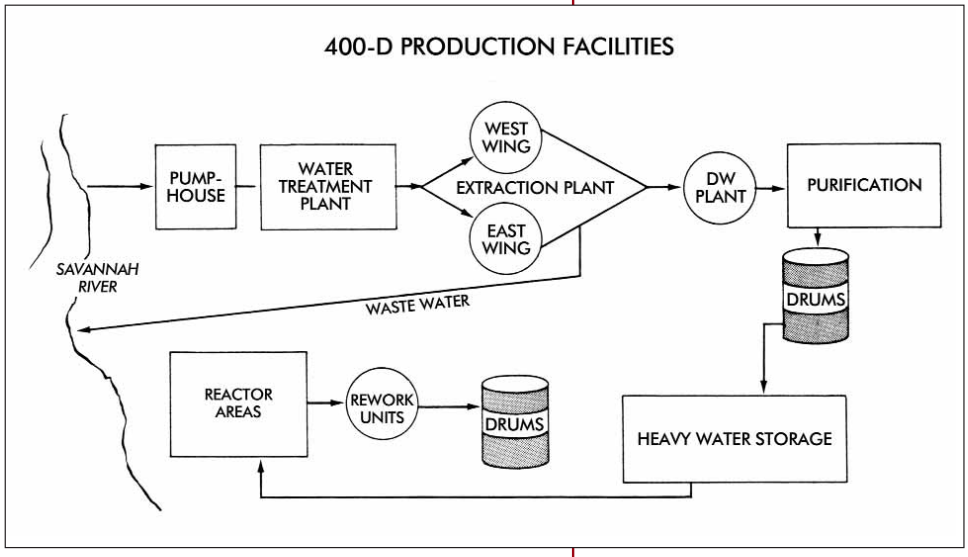
1953			
January	16.00	13.40	131.83
February	16.00	16.10	163.93
March	16.00	16.80	196.73

Source: Control Division, "Heavy Water Production and Production Costs Fiscal Year 1951 Through August of Fiscal Year 1956," DPW-4742-1, March 11, 1952. National Archives, Records of the Atomic Energy Division, A-1957, Series II, Box 45, Folder 14.

The need to continue heavy-water production lessened after the reactors were in operation and an adequate stock of heavy water had been stored for future use. Both plants were designed for a production rate of 240 tons of heavy water per year; they exceeded these original projections after three years in operation.[40] In 1956, the GS Department's net production of heavy water was 478.13 tons.[41]

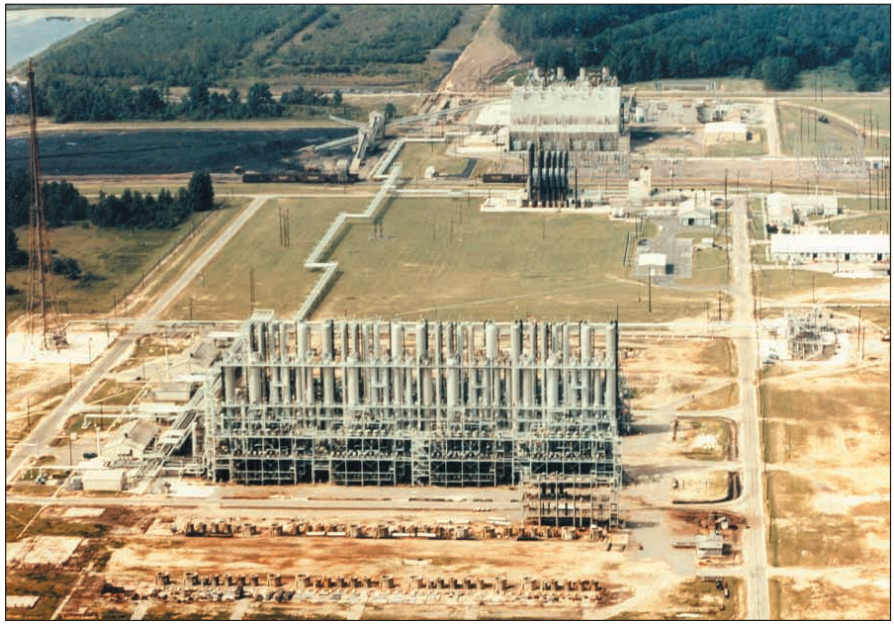
In 1957–1958, the Dana Plant and two thirds of Savannah River's D Area GS units (Facilities 411-D and 413-D) were shut down. The plants had proven successful, producing sufficient heavy water for SRP reactors including the test reactor and a stockpile for other uses. Dana's closure was predicated on needed repairs that would have proved costly and slightly lower productivity levels that stemmed from the GS unit's complicated design. The same winnowing occurred at SRP. The GS unit with unclad towers, suffering from more corrosion than its counterparts, was dismantled beginning on October 4, 1957. The E Plant was also shut down as the concentration of the final product could be handled in the DW Plant without a "significant loss in production."[42]

As a result, Dana's staff was reduced to a skeleton crew of 12; some members of the operations staff reported to Savannah River's GS Department. However, the closing of D Area facilities also reduced the staff at Savannah River. In 1957, D Area had 588 employees, after 145 positions had been cut in response to the closing of Building 411-D. More than 149 jobs were cut the next year when Building 413-D was closed, reducing the D Area workforce to 435.[43] In 1959, a workforce of 395 produced 185.33 tons of heavy water in the remaining eight extraction units in Building 412-D and 9 of the 12 towers in the DW plant. The other three DW towers were used as a rework unit for the reconcentration of degraded heavy water from the reactors containing tritium.[44] Du Pont was officially released from responsibility for the Dana Plant in July of 1959 after the purging of all equipment with nitrogen. The U.S. Army Corps of Engineers assumed responsibility for the plant at that time.



Flow Chart showing Processes in D Area, 1970s. Source: D Area Tour Booklet, not dated. Courtesy of SRS History Project.

Between 1959 and 1964, D Area would steadily lose more staff. In 1964, a force of 322 produced 345.26 tons of heavy water.[45] Total production of heavy water grew between 1959 and 1964 as the operating staff worked with economy and focus with the remaining facilities. The plant histories for this time period show rework assuming greater importance in D Area's operation, while the costs of maintaining older facilities, now closed down, grew steadily.



Aerial view of 400 Area after dismantlement of 411-D, only the tower's cylindrical foundations remain. 412-D was the last group of original GS towers to operate. Courtesy of SRS Archives.

Tons of Heavy Water produced at Dana and Savannah River and exported by December 1963

Canada	276.00
Germany	132.00
France	70.00
United Kingdom	66.00
Sweden	57.00
Japan	48.00
India	36.00
Euratom	28.00
Norway	25.00
Switzerland	23.00
Denmark	17.00
Australia	13.00
Italy	10.00
Israel	4.40
Netherlands	1.60
Belgium	0.25

Source: W. P. Bebbington, J. F. Proctor, W. C. Scotten and V. R. Thayer "Production of Heavy Water in the United States of America," *Chemical Engineering Progress 55* (9), September 1955; 335.

"ITS PRODUCTS GO FAR"

Heavy water was the Savannah River Plant's first direct contribution to the Atomic Energy Commission's vigorous campaign to develop atomic energy for peaceful purposes. When Commission Chairman Lewis L. Strauss visited the plant in March of 1955, he announced that the United States had agreed in principle to sell 10 tons of heavy water to the Italian government for use in Italy's first research reactor.[46] The market price in 1956 for heavy water was $28 a pound or $14,000 a drum. By 1957, Sweden, Canada, Switzerland, France, Australia, Norway, and the United Kingdom had received substantial shipments of Savannah River and Dana's heavy water for use in research reactors and incipient power reactor programs. Italy, Denmark, Japan, Israel, West Germany, New Zealand, Pakistan, and South Africa had pending commitments with the AEC for purchase of the plant's heavy water through the Commission's "Atoms for Peace" agreements.[47] Sales continued briskly through the 1970s. In 1970, a final shipment for a $27 million dollar sale of heavy water for use in Canada's Ontario Hydro Pickering Station rolled off-site in tractor trailers. Pickering Station was one of four reactors being built by the Ontario Hydro-Electric Commission near Toronto.[48] The national laboratories also received heavy water from SRP and Dana; Brookhaven National Laboratory received 100 tons of Dana's product in 1966 after the Indiana plant closed.[49]

Tests and preparations for 1969 shipment of heavy water valued at $9 million to Sweden, 1968. In 1969, the AEC negotiated sales of 450 tons of heavy water for an estimated value of $42 million dollars. Courtesy of SRS Archives, negatives (Above) DPSPF 12818-8 and (Below) DPSPF 12818-11.

HEAVY-WATER TECHNOLOGY AND EXPERTISE

While Savannah River's heavy water traveled far, so did its technology and its operations know-how. The AEC and Savannah River, under a bilateral agreement between the USA and Canada, worked cooperatively with Atomic Energy of Canada, Ltd., in the overall field of heavy-water production and heavy-water reactors. Canada's decision to found

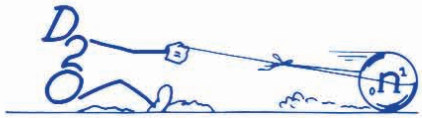
Mass Spectrometers and Heavy-Water Analysis

During World War II scientists involved with heavy-water production used the sole unique characteristic of heavy water, its "heaviness," to determine the concentration of their product. Using comparisons of liquid densities, they were able to control their experiments, and these comparisons allowed them to gauge their progress in concentration efforts.

The mass spectrometer, which was a research laboratory tool prior to 1950, became a common analytical instrument used by personnel at Dana and SRP. The development of the mass spectrometer, able to differentiate between the masses of water molecules and to measure directly the ratio of heavy to light molecules, was a boon to heavy-water producers. Mass spectrometers were installed in the GS control rooms and were used as the plant's major source of analytical control data in its heavy-water production.

"Why Heavy Water Is So Good." Cartoon figures coupled with serious facts bring home the positives about heavy water. Source: Savannah River Laboratory, *Nucleonics of Tomorrow in the Making Here Today* (Aiken, South Carolina: E. I. du Pont de Nemours and Company, not dated).

WHY HEAVY WATER IS SO GOOD



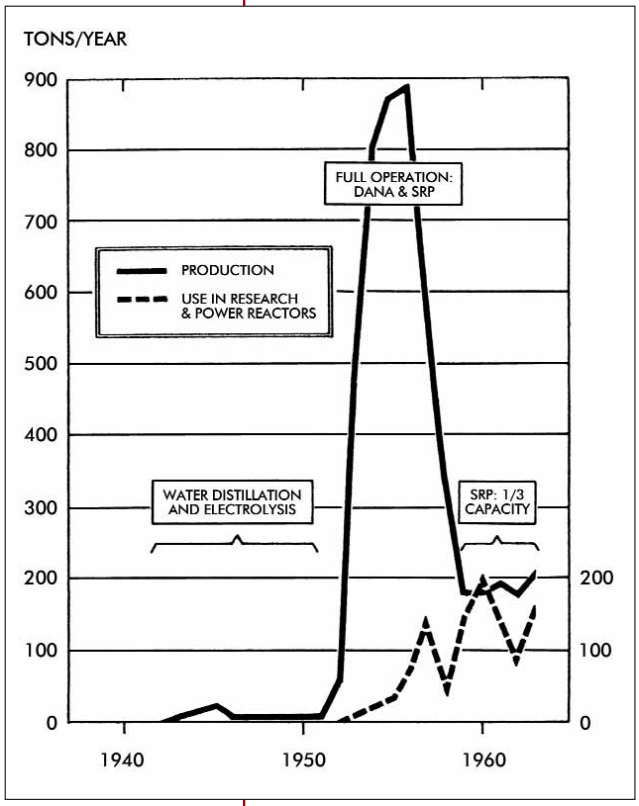
Heavy water slows fission neutrons to thermal energies without absorbing them. It does this important job more efficiently than any other substance. Its moderating ratio, $\Sigma_s \xi / \Sigma_a$, is 70 times that of graphite, the next best moderator. Being a liquid, it can serve as both coolant and moderator. For experimental work, it is wonderfully flexible. You don't have to drill holes in *this* moderator!

With heavy water, natural uranium can be used as fuel. Enriched uranium is not necessary. Power systems based on D_2O have important economic and politico-economic potentialities. They are being developed with vigor, particularly by the United States, Canada, Norway, and Sweden. Production systems based on D_2O show unsurpassed neutron economy.

The Savannah River Plant makes practically all of the free world's supply of heavy water. A sensation was created at Geneva in 1955 when the AEC offered to sell D_2O at one-third of the previous world price. Already, more than $23 million worth have been sold.

their commercial nuclear power industry on heavy-water-moderated reactors solidified earlier technological ties between the U.S. and Canada that had developed during World War II. Canadian researchers were able to learn from operations staff. They watched closely Savannah River's operation of the Heavy Water Components Test Reactor between 1962 and 1964, even sending a researcher to participate in the project launched under the auspices of the Atoms for Peace Program. Other countries interested in heavy-water technology, including India and Argentina, also benefited from the experience of Savannah River's heavy-water experts. The 400 Area hosted chemical engineers from India, providing them with first-hand knowledge of large-scale, heavy-water production.

The first heavy-water plant constructed outside of the USA was built at Glace Bay on Cape Breton Island, Canada, as a joint venture between Deuterium of Canada Ltd. (DCL) and the province of Nova Scotia. The plant was unable to operate as designed, and Du Pont was asked to consult in diagnosing the problems and in offering recommendations for future operation. Bebbington notes that the plant was rebuilt with modifications that were patented by a retired Du Pont heavy-water expert.[50] The La Glace facility was to have supplied heavy water for an Argentinean reactor. Due to delays at the Canadian facility, Argentina acquired heavy water from the AEC that had been taken from Savannah River's L Reactor, then on standby. Later Canadian heavy-water plants built by General Electric of Canada Ltd. and Ontario Hydroelectric also benefited from experience gained from the design and operation of heavy-water facilities at Dana and Savannah River. Du Pont engineers were involved in each Canadian plant, and Canadian representatives were sent to Savannah River to learn from D Area operations. With the closure of D Area, these Canadian facilities became the world's largest heavy-water production facilities.



Graph showing production and use of heavy water between 1945 and 1965. Source: W. P. Bebbington, J. F. Proctor, W. C. Scotten, and V. R. Thayer. "Production of Heavy Water in the United States" in *Proc. Third International Conference on the Peaceful Uses of Atomic Energy, Vol. 12 Nuclear Fuels–III Raw Materials* (New York: United Nations, 1965), 334.

SUMMARY

Between 1951 and 1982, the towers erected in Savannah River's D Area and at the Dana Plant in Indiana yielded about 7,500 tons of heavy water for the nation's atomic energy program. The Dana Plant operated from 1951 until 1957, when an adequate supply of heavy water was on hand. Two of Savannah River's three GS units were closed in 1957–1958 for the same reason, leaving one in successful operation through 1982. Morris *et al*. recollect the camaraderie that existed between the sister plants along with good-natured competition in safety and productivity. The heavy water they produced for our nation's production reactors and the heavy water that led to nuclear energy research around the world and SRP's first Atoms for Peace product, is a remarkable legacy.