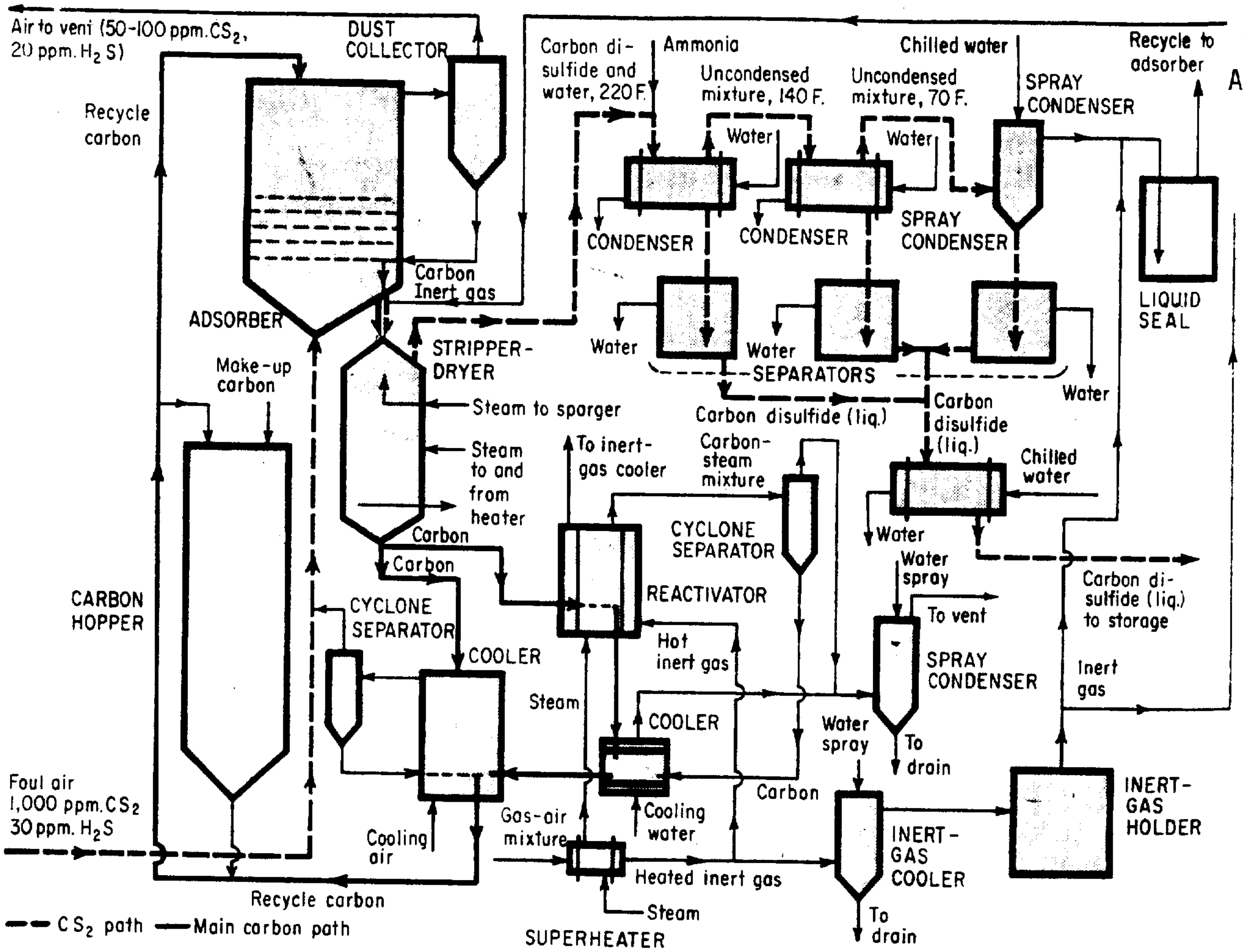

FLUIDIZED RECOVERY SYSTEM NABS CARBON DISULFIDE

Departing from tradition, a British viscose plant uses fluidized instead of fixed beds, finds CS_2 recovery can be economical even if the foul air stream contains only low levels of the disulfide.

The modern trend that has seen continuous fluidization of solids replace many fixed-bed processes has gained another convert. Courtaulds Ltd., British synthetic-fibers giant, is using a fluidized-bed adsorber to recover carbon disulfide from a foul air stream at its Holywell, Wales, viscose plant. Conventional CS_2 recovery at viscose plants is accomplished by parallel fixed beds that operate on alternate cycles, one being stripped and dried while the other is adsorbing.

Recovery is not practiced by the viscose makers in the U. S. because disulfide can be purchased for less than the recovery cost. But producers are eyeing the British process because tightening pollution laws may eventually make recovery necessary—and fluidized adsorption may be the best way to reduce discharge of disulfide.

In the viscose (rayon) process, CS_2 is a processing agent that converts cellulose to a xanthate form before a spinning solution is formed. Most of the disulfide is later released when the cellulose is regenerated in sulfuric acid, and additional quantities are removed from the fiber upon drying.

Courtaulds' $1.7-million operation, called Landmark, recovers 1.5 long tons/hr. from a 250,000-cfm. air stream that contains about 1,000 ppm. of CS_2. About 50% of the viscose plant's CS_2 input is regained from the stream, at 80-95% efficiency. Circulation rate of the activated-carbon adsorbent is about 50,000 lb./hr.

▶ **Process Description**—Before the foul air stream goes into the adsorber, it is subjected to spraying (not shown) by an alkaline ferric oxide suspension that removes about 90% of H_2S, which in the presence of oxygen would form elementary sulfur and clog the adsorbing surface. A 1,000-hp. fan then boosts the stream pressure to about 3 psi. ahead of the adsorber.

Carbon disulfide in the air is removed by five shallow beds of activated carbon in the 45-ft.-high, 38-ft.-dia. mild-steel adsorber. The carbon is a specially prepared coconut charcoal of between 6 and 14 mesh. Cleaned air, vented to the atmosphere through a dust collector, contains about 50 to 100 ppm. CS_2 and 20 ppm. H_2S.

▶ **Carbon Cycle**—Recycled carbon

is deposited on the top bed in the adsorber by a conveyor and distribution system. Transfer from bed to bed is regulated by downcomers that extend up 1½ to 2 in., determining bed height. After 45 min. of adsorption, the carbon has picked up about 7% of its weight of CS_2.

Discharge from the bottom bed to the stripper is through seal legs. Originally these were fitted with two rotary-seal valves, with inert gas introduced between them to prevent steam from passing upward into the adsorber. But severe valve wear led to their removal; now, inert gas alone suffices to keep the steam out, while the height of the carbon column provides sufficient head to cause the carbon to flow down into the stripper below.

Carbon is stripped and dried in two stages. In the top of the vessel, 300 F. steam strips out the CS_2; then a battery of 52 vertical steam-heated dryers completes the contaminant removal. Specially designed seal legs help maintain uniform carbon flow through the latter portion of the vessel.

The carbon stream leaving the dryer is split; 10% is reactivated, and the remainder is cooled to 160 F. in a bed fluidized with 6,000 cfm. of air. Reactivation is accomplished by fluidization at an unspecified temperature with superheated steam, in a jacketed vessel that is heated by combustion gases from a superheater. Product from this vessel cools first in a water-cooled unit, then passes into the air cooler where it mixes with the rest of the recycle carbon.

Fresh carbon make-up for losses and attrition is admitted to a storage vessel and added into the recycle stream as needed. The vessel provides sufficient storage volume to hold all of the circulating adsorbent if necessary.

▶ **Disulfide Recovery**—Because of the low CS_2 content of the feed stream to the process, loading on the carbon is low and hence a high ratio of stripping steam is needed. Exit gases leaving the stripper have a steam/CS_2 ratio of 7 to 1.

To protect the aluminum tubes in the condensing system, about 2 lb./hr. of ammonia is injected into the stream before it goes into the first two stages, where most of the steam is condensed. Temperature of the circulating cooling water is maintained at just about the temperature at which CS_2 condenses (117 F.).

The bulk of the CS_2 is removed in a third condenser, and final cleanup is obtained in a spray tower with 35 F. spray water. Condensate from all three condensers and the spray tower is separated by gravity, with the heavier disulfide drawn off the bottom. The disulfide is chilled again before it recycles to the viscose plant for reuse. The product contains less than 10 ppm. of nonvolatiles and 7 ppm. of H_2S, and can be reused directly without further purification.

▶ **Operating Experience** — In the year that the plant has been operating, Courtaulds has learned that adsorption efficiency gradually decreases from about 95 to 80% in six months. After prolonged operation, fines are produced that increase air flow resistance and hence raise carbon residence time in the adsorber.

Because the adsorption rate for CS_2 is greater than that for water at normal residence times, whereas water displaces CS_2 at equilibrium, longer residence times tend to allow the carbon to pick up more water, lowering efficiency. By drawing off fines separately from the adsorber dust-collector instead of returning them to the lower bed (see flow diagram), Courtaulds has partially offset the fines' detrimental effect. The amount must be made up with fresh carbon, of course.

Although this full-scale prototype plant operates at a level appropriate for a 200-million-lb./yr. viscose plant, Courtaulds expects to be able to use the method to compete with fixed-bed adsorption at any level of output and concentration required.—FCP

World Trade Flourishes For Nitrogen Goods

After a year of stagnation, worldwide import-export trade in nitrogen fertilizers and technical-grade nitrogen rebounded to a healthy 3.16 million metric tons of nitrogen content during the 1961-1962 market year. Representing a 13.7% increase, this occurred despite the startup of new production facilities (using locally supplied raw materials) in many countries that import fertilizers.

These facts are from a special report recently issued by British Sulphur Corp., London, which also suggests that new fertilizer-production capacity in underdeveloped countries will stimulate worldwide demand in the long run, even though the immediate level of imports to these countries may briefly fall off.

According to the report, over-all world production increased by 7.2%, to 14.24 million tons of nitrogen content. Consumption—which has been rising steadily by a million tons each year—totaled 14.06 million tons, an increase of 7.5%. Import-export trade represented just over 22% of this consumption; during 1960-1961, the corresponding figure was 21%.

Despite a decline in its relative world-trade importance, ammonium sulfate continued to account for the largest trade volume, 1.05 million long tons (an increase of 9% over the previous year). Urea exports expanded by 40% over the previous year, and they now account for one sixth of the world import-export total.

West Germany remains the world's leading exporter of nitrogen products, with total shipments containing 583,000 metric tons of nitrogen. Japan ousted Italy (351,400 tons) from second place by shipping 413,800 tons, an increase of 37% over the previous market year. Other major exporters: Netherlands, 247,800 tons; U. S., 226,900 tons; Norway, 223,900 tons; and Canada, 217,600 tons. Though Great Britain increased its exports by 88% to 63,300 tons, it nevertheless accounts for only 2% of the total world trade.