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ABSTRACT

A process has been developed by TVA to produce sulfur-coated urea, a controlled-release nitrogen fertilizer, by coating urea with sulfur. The product has been produced on a pilot-plant scale at rates of 1 ton or more per hour. Sulfur-coated urea has shown significant agronomic advantages over more conventional fertilizers when used with certain crops or in certain climatic or soil conditions.

In a continuous process, urea particles are preheated and their surfaces are polished in a rotating drum equipped with electric radiant heaters. Next, the urea enters a second rotary drum where multiple, thin, concentrically formed layers of sulfur are applied. The molten sulfur, which is filtered to remove solid impurities, can be applied using either pneumatic or high-pressure hydraulic atomizing spray nozzles.

When using pneumatic nozzles, heated air is used as the atomizing medium. Sulfur flow is metered to each of eight nozzles by an in-line orifice to ensure equalized flows. The nozzles are oriented

to spray sulfur directly onto the fastest moving portion of the cascading bed. The sulfur-coating temperature is maintained at about 165°F and wax is applied in another rotary drum to seal imperfections in the coating. Then the granules are quick cooled in a fluid bed cooler and lightly dusted with conditioner. By increasing the sulfur-coating temperature to about 200°F and decreasing the production rate to 1/2 ton per hour, a slow-release fertilizer having only a sulfur coating can be made. Total coating requirements are less for the wax-sealed product making the nitrogen content of a typical product about 40%. Sulfur-only products contain about 38% nitrogen. Important variables include: coating weight of sulfur, wax, and conditioner; type and temperature of wax; size and condition of the urea; type, size, number, position, and orientation of sulfur spray nozzles; pressure, volume, and temperature of atomizing air; rotational speed of the sulfur-coating drum; purity and temperature of sulfur; and cooling conditions of the product.

When sulfur is applied by hydraulic atomizing nozzles, the sulfur-coating drum is provided with lifting flights which discharge urea onto a collecting pan that forms the urea at a fixed position into a falling curtain. The sulfur is sprayed directly onto the curtain through eight nozzles at pressures ranging from 750 to 1500 psig. As compared with the pneumatic spray system, process variables are somewhat different in both relative importance and absolute value. Hydraulic

pressure replaces atomizing air conditions as a variable. Controlled-release sulfur-coated urea has been made both with and without sealants. Sulfur-coating temperatures are maintained from 165° to 185°F for both products. Hydraulic spraying offers some major advantages over pneumatic spraying including reduction of sulfur dust and mist in the sulfur-coating drum and doubling the production capacity of the plant without significant reductions in the product quality. A typical sulfur-coated, wax-sealed product contains 38% nitrogen, while the sulfur-only product contains 37% nitrogen.

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INTRODUCTION

For several years TVA has been encapsulating soluble fertilizer granules and prills with sulfur to impart controlled-release properties to them. These studies have been carried out on both bench and pilot scale (1, 2, 3). Most of the work has centered on the sulfur coating of urea, $CO(NH_2)_2$. In the pure state, urea contains 46.67% nitrogen, making it the most concentrated solid nitrogen fertilizer extensively produced. Further, urea can be manufactured economically and has demonstrated itself to be a good source of nitrogen for many plants, grasses, and trees. It is becoming the world's leading nitrogen fertilizer. Our research and development effort has shown it to be a very desirable base for a slow-release fertilizer.

When urea has been properly sulfur coated, agronomists have found that it possesses some significant advantages over more conventional fertilizers when used with certain crops or in certain climatic or soil conditions (4, 5, 6). In field tests, rice, sugarcane, pineapple, and forage crops are among those crops for which sulfur-coated urea

showed advantages as compared with uncoated urea or ammonium nitrate. A significant reduction in leaching losses has been observed in some tests (7). In other tests, luxury consumption of nitrogen has been greatly reduced or eliminated and burning of vegetation and damage to seedlings have been prevented. Exceptionally good results have been obtained when sulfur-coated urea was used in the fertilization of lawns and ornamental shrubs. In almost all cases these results can be obtained at a lower application cost because of a reduction in the number of fertilizer applications required.

Aside from the agronomic advantages, sulfur-coated urea has several physical advantages over conventional nitrogen fertilizers. It is resistant to caking in humid conditions. Uncoated urea must be protected from caking with itself by the incorporation of formaldehyde or the application on the solid granules of a parting agent, such as a kaolinite clay. Sometimes even these precautions are not effective enough to prevent caking. Sulfur-coated urea is compatible with other fertilizers such as superphosphates and may be used in a wider range of blend formulations than uncoated urea which is not compatible with superphosphate.

Cost estimates have indicated that sulfur-coated urea can be produced for about one-third more per unit of nitrogen than straight urea. This would be much cheaper than other controlled-release products now marketed--such as urea formaldehyde or isobutylene diurea (IBDU).

SULFUR-COATED UREA - PRODUCT DESCRIPTION

Conventional sulfur-coated urea consists of granules or prills of urea uniformly coated with a layer of sulfur. A seal coating of wax is applied to the sulfur coating to fill pores, cracks, or thin spots in the sulfur coating. For most pilot studies, TVA has used a microcrystalline wax with an oil content of about 10%. A coating of conditioner is applied to the waxed granules to absorb this oil and thus provide good flow properties to the final product. The amounts of sulfur, wax, and conditioner which are applied to the urea depend on the desired nitrogen dissolution rate. These amounts vary with the processing conditions, with the size and shape of the substrate urea, and with the type of wax and conditioner used. The typical product which has a nominal size range of minus 6 plus 9 Tyler standard sieve size consists of 82% urea, 1% clay conditioner (applied as a parting agent by the urea supplier), 13% sulfur, 2% wax, and 2% diatomaceous earth. In the past, the sulfur-coated urea has also contained 0.25% coal tar applied as a microbicide to reduce the soil microbial attack on the wax. However, recent agronomic tests have demonstrated the coal tar to be of very little value. It was therefore dropped in favor of a more simplified process and better handling characteristics of the final product.

Recently, TVA has been able to produce a slow-release urea coated only with sulfur (8, 9). This material, which also has a nominal size range of minus 6 plus 9 Tyler standard sieve size, typically consists of 80% urea, 1% preapplied parting agent, and 19% sulfur. Much

less agronomic data have been generated on this product than on the conventional product and its effectiveness as a slow-release nitrogen fertilizer is not fully known. However, early reports are very encouraging.

The comparative controlled-release characteristic of sulfur-coated urea is measured by determining the percentage of urea that will dissolve when a 50-gram sample of product is immersed in 250 grams of water and held at a constant temperature of 100°F for 7 days. The quality of the coating is determined by comparing this dissolution rate with the total coating of sulfur, wax, coal tar, and conditioner applied to the urea. Products with 20 to 30% dissolution in 7 days and 1% or less daily thereafter are usually sought. The dissolution rate can be varied by varying the coating weight. Soil and climate conditions and the growing season of the crop being fertilized are among other variables that might make higher or lower dissolution rates desirable.

As might be expected, the crystalline makeup and the uniformity of the sulfur coating are important, but are even more so for the sulfur-only material (10). The sulfur structure must have elasticity and the urea must be evenly coated. Operating conditions must be controlled to give this structure. Dr. G. H. McClellan fully discusses the subject of sulfur crystalline structure in Chapter OO.

RAW MATERIALS

Urea

The physical characteristics of the substrate material are very important in the production of any coated material and this is especially true of sulfur-coated urea. The urea should be spherical and have a smooth surface. If the granules or prills have sharp corners, surface blips, cracks, dimples, holes, or if they are agglomerated so that the granules have a very irregular joint, they are much more difficult to coat completely and effectively with sulfur. When the sulfur coating is sealed with wax, most imperfections in the granules can be overcome by using heavier coatings of sulfur and/or wax. Unfortunately, this results in a reduction in the nitrogen content of the product and increases the manufacturing and shipping cost per unit of nitrogen. When the coating is only sulfur, the imperfections are much harder to overcome. Wax can flow into dimples, cracks, holes, etc., sealing them, where sulfur cannot because it freezes on impact with the relatively cold substrate. For example, most prilled urea has a small dimple or hole in the surface of the sphere. Prills have been successfully coated with a conventional coating which includes wax, but efforts to make a sulfur-only product from prills have been discouraging. Product made from prilled urea with a total coating of 25% (20% sulfur, 3% wax, 2% conditioner) had a 7-day dissolution rate of 22%. Using the same prills as a raw material, the sulfur-only product with a sulfur

coating of 33% had a 7-day dissolution rate of 49% and data indicated that further increases in the sulfur coating would have little effect on the 7-day dissolution.

It appears that granules must have about the same thickness of coating on them regardless of their size to give the same dissolution rates. If this is a valid observation, then the amount of sulfur a substrate urea requires per unit of weight varies directly with the surface area of the urea substrate or inversely with the square of the average diameter of the granules provided all other variables are constant. It is thus obvious that as long as agronomic benefits and handling characteristics are equal, if the size of the urea particles that are coated increase, the coating needed will decrease, reducing the production costs per unit of nitrogen and increasing the nitrogen content in the final product.

The urea substrate should be hard. Soft granules or prills give less support to the sulfur coating making it more susceptible to fracture on handling.

The urea substrate may have a precoating on it to prevent caking during shipment and storage and still be coated with sulfur without adverse effects on the final product. In most pilot-plant sulfur-coating tests, the substrate material used was spray drum-granulated urea supplied with a 1% kaolinite coating (Barnet clay). Kaolinite or

some other parting agent, or a chemical caking inhibitor, such as formaldehyde, is needed in most cases to prevent the substrate urea from caking unless the sulfur coating is applied to very recently produced urea. It has been found that a 1% coating of Barnet clay actually may lower the sulfur requirements by as much as 2 percentage points. However, the economics of including an extra step in the process to precoat feed urea with kaolinite prior to sulfur coating would be extremely questionable at best. Increasing the preconditioner kaolinite to more than about 1% is detrimental. Urea preconditioned with formaldehyde can be coated with sulfur without any apparent effect on the quality of the final product. [Fleming has developed a process where finely divided powder, in particular carbon black, is applied as a precoat on the urea to substantially reduce the usage of sulfur in the final coating. No work has been done in the TVA pilot plant on this aspect of sulfur coating, and it is mentioned only for information to those who might wish to pursue it further (11)].

Sulfur

TVA uses bright sulfur having a carbon content of around 500 ppm and an ash content of around 55 ppm. It is mined by the Frasch process. The carbon content of the sulfur appears to be a very important factor. Most of the carbon is in the form of hydrocarbons which react with hot

sulfur to form a solid carbon-sulfur complex, carsul. Because this reaction is very slow and continuous at normal storage and usage temperatures, the carsul is difficult to keep removed from the sulfur and this will cause extreme problems if the sulfur is not handled properly or if the hydrocarbons are excessive in the sulfur.

Wax

A microcrystalline wax, manufactured by one of the major U.S. oil companies, is used to seal the sulfur coating in the conventional process. Recently, because of the energy crisis, there have been several changes in product lines of many of the major oil companies. Microcrystalline wax production has been curtailed by some companies and even eliminated by others. Thus, the availability and cost of microcrystalline wax is very uncertain, especially for new consumers. Therefore, TVA is continuing extensive small-scale work with sealants and has found other waxes and organic substitutes which appear to be almost comparable in effectiveness. At present, these latter products have been tested only on a bench scale, but pilot-plant tests are planned in the immediate future for the most promising ones.

Conditioner

The main function of the conditioner is to absorb the oils in the wax and thus remove the stickiness from the product. Therefore, it is needed only when an oily wax is used. Primarily, diatomaceous earth has been used as the conditioner because of its oil-absorbing capacity,

139 pounds per 100 pounds as reported by the supplier. Kaolinite with a much lower oil-absorbing capacity of 34 pounds per 100 pounds proved to be a poor substitute, requiring twice as much coating to provide a final product free of stickiness.

BASIC PROCESS

The basic process for producing a sulfur-coated urea with a wax sealant is shown in Figure 1. The first step of TVA's continuous process involves heating granular urea in a rotating drum equipped with an electric radiant heater. The heat prepares the surface of the granules to accept the subsequent coating of sulfur. Sulfur is applied in a rotary drum in which urea granules are sprayed with molten sulfur. The sulfur can be sprayed through nozzles which use heated, compressed air as the atomizing force, or it can be atomized by pumping the sulfur at high pressures through hydraulic atomizing nozzles. In either case, a uniform coating of sulfur is built up on the individual particles. The material is discharged to a wax-coating drum by means of a bucket elevator. Wax (2% of total product weight) is applied to seal the imperfections in the sulfur coating. The urea is then cooled to solidify the wax. A fluid bed cooler is used which minimizes handling problems with the sticky, wax-coated material while cooling it efficiently. The material is discharged from the cooler to the conditioning drum where

diatomaceous earth (2% of total product weight) is applied. The product is screened to remove any oversize material formed in the process. The amount of oversize removed is usually in the range of 0.1 to 0.5% of the total production and is in the form of agglomerates.

(Fig. 1)

When producing a controlled-release urea with only a sulfur coating, no subsequent processing is required, but some quick cooling, which is beneficial under certain operating conditions, is recommended.

DESCRIPTION OF PILOT-PLANT EQUIPMENT

The TVA pilot plant was originally designed for the production of 1 ton per hour of sulfur-coated urea, but it is now capable of producing 2 tons per hour. It consists of a urea storage hopper, urea feeder, urea preheater, sulfur feed system, sulfur-coating drum, wax-coating drum, fluidized bed cooler, conditioning drum, and auxiliary process equipment. The preheater is a rotary drum about 2 feet in diameter and 6-1/2 feet long. Heat is provided by twelve 440-volt resistance radiant-type heaters installed inside the rotating drum. The temperature of the urea is controlled by cycling the heaters on a timed basis between one-quarter and full-load output. The shell of the preheater is smooth inside and insulated outside.

The sulfur-coating drum is about 4 feet in diameter and 6 feet long. The internals of the drum and the associated sulfur piping are completely different depending on whether the sulfur is atomized

pneumatically or hydraulically. A schematic drawing of the sulfur-coating drum operation with the two types of sulfur spraying is shown in Figure 2.

(Fig. 2)

When the pneumatic spraying system is used, the sulfur-coating drum has a 3-3/8-inch retaining ring at the discharge end. It contains no flights and is insulated outside. It has a carryover shield to prevent occasional granules from falling on the steam-heated pipe and melting. The dripping melt causes agglomerates when it falls into the bed of granules. The sulfur is distributed through a steam-heated header equipped with eight air-atomized spray nozzles. The nozzles are positioned to spray the sulfur directly downward at the most active part of the rolling bed of granules. The atomizing air for the sulfur sprays is steam heated in a shell-and-tube heat exchanger. The pneumatic sulfur-spraying system is fully discussed later in this chapter.

When the hydraulic spraying system is used, the sulfur-coating drum has a 5-inch retaining ring at the discharge end. Twenty-four lifting flights are installed in the drum at 15-degree intervals. The flights are straight with flat surfaces 3 inches wide and 58 inches long. They are installed parallel to the axis of the drum and, to increase their lifting capacity, they are slanted 15 degrees forward from the perpendicular with the shell of the drum. As the drum rotates, the flights carry material out of the bed of granules and discharge it onto a

collecting pan. The pan extends the length of the drum and is installed parallel to the axis of the drum and level with the horizontal with respect to its length. The pan is 20-1/2 inches wide and is sloped counter to the direction of the rotation of the drum at an angle so that the granules will cascade down it. As the granules discharge from the pan, they strike a deflector plate which forms them into a continuous vertical curtain of free-falling granules on which the sulfur is sprayed. The slope on the collecting pan and its relative position with the bed of cascading granules can be varied. The sulfur is distributed through a steam-heated header equipped with eight hydraulic spray nozzles. Atomization of the sulfur is achieved when sulfur passes through the small orifice tips of these nozzles at high pressures.

The wax-coating drum is about 3 feet in diameter and 2 feet long. Wax is supplied to the wax-coating drum from a steam-heated tank with a small submerged diaphragm-type metering pump. It is distributed onto the bed of material in the wax-coating drum through a "drip tube" distributor. The material from the wax-coating drum feeds by gravity through a rotary airlock valve into the fluidized bed cooler.

The cooler has a bed area of 10 square feet. Air for fluidization and cooling is provided by a centrifugal fan located so that the air is forced through the cooler. The ductwork and damper arrangement allows close control of airflow into the cooler. The fluidized bed unit is very efficient, giving product temperatures that are within 5°F of the

cooling air temperature at the 1-ton-per-hour rate. Pressure taps and temperature gages are positioned to permit monitoring of air pressures and temperatures throughout the cooler and airflow system. Air from the cooler is vented through a stack. The cooled product flows by gravity through a rotary airlock valve to the conditioning drum.

The conditioning drum is about 3 feet in diameter, 3 feet long, and has a smooth shell. Diatomaceous earth conditioner is metered to the conditioning drum with a vibrating screw-type feeder.

The conditioned product is discharged from the conditioning drum onto a belt conveyor which feeds a 4-mesh scalping screen. Product from the screen is fed by conveyor to a bagging hopper. Bucket elevators are used to (1) fill the urea storage hopper and (2) elevate the product from the sulfur-coating drum to the wax-coating drum. All rotary equipment is driven with electric motors. All of these units have variable-speed drive except the conditioning drum. Instrumentation is provided to obtain process data throughout the system.

Good performance has been obtained from all of the pilot-plant processing equipment.

SPECIFIC PROCESSES AND VARIABLES

The process and the related variables are dependent on whether the plant is producing sulfur-coated urea with or without a sealant and whether the sulfur is being applied with the pneumatic or hydraulic sulfur-spraying system.

PNEUMATIC SULFUR-SPRAYING SYSTEM

Sulfur-Coated Urea With Sealant

Figure 3 shows a flow diagram of the pneumatic sulfur-spraying system. Better atomization occurs when the sulfur is applied with this system than with the hydraulic system, which will be discussed later. With the pneumatic system less sulfur is required, making possible a higher analysis sulfur-coated urea fertilizer.

(Fig. 3)

The sulfur is pumped out of a steam-heated, scale-mounted tank by means of a hydraulically operated diaphragm-type metering pump. Its head containing a stainless steel diaphragm and two ball check valves is remotely mounted in the weigh tank. The sulfur in the tank is around 300°F. It is sucked into the pump through an 80-mesh wire cloth having 60 square inches of area to filter out any large particles of carsul or other impurities which might cause pumping or spraying problems. The steam-jacketed pipeline from the pump to the spray nozzles is used as the fine temperature control, adjusting the temperature of the sulfur to 310°F for spraying. Sulfur is at its lowest viscosity (0.004 lb/ft-sec) at 310°F. The sulfur is pumped through two more filters before entering the sulfur header. A 100-mesh stainless steel cone filter having 9 square inches of filter area and a small 60-mesh filter having 1.8 square inches of filter area are installed immediately prior to the sulfur header. The larger unit is used to contain most of the particles which might

otherwise plug the orifices or nozzles. The small unit acts as a cleanup filter to retain anything formed in the pipeline between the large filter and the sulfur header or anything which might have passed the larger filter because of particle orientation or filter failure. Both of these filters need cleaning after about 3000 pounds of sulfur has passed through them. The inlet filter is self-cleaning because of the pulsation of the pump. However, the carsul which accumulates in the bottom of the weigh tank must be drained occasionally. Adequate filtration of the sulfur is a must if the process is to work. It has shown itself to be possibly the most important part of this process from an operational standpoint and it is even more important in the hydraulic spraying system.

The filtered sulfur is metered equally to each spray nozzle by an in-line sulfur flow control orifice prior to each atomizing nozzle. The development of this orifice control system to ensure uniform feed to the nozzles was a main feature of the successful design of the pneumatic coating system. The nozzles (see Fig. 4) require little or no hydraulic pressure because the heated atomizing air ($\approx 295°F$) acts as an asperating force and pressure drops in the nozzle are minimal. This coupled with the physical characteristics of sulfur and slight differences between nozzles caused starvation of some nozzles and excessive output on others when flows to the individual nozzles were not regulated. With identical orifices installed before each nozzle, they delivered the same sulfur flow, provided at least a 5- to 10-pound-

per-square-inch pressure drop occurs across the orifice. Unless the orifices are extremely small, approximately 0.018 to 0.028 inch in diameter, this pressure drop cannot be obtained. This small opening and the somewhat larger opening of 0.052 inch in diameter at the nozzle tip are the reasons for the extremely fine filtration requirements mentioned above. Besides providing some asperating force, the air which passes through the annular space surrounding the liquid nozzle acts as the force to atomize the sulfur as it expands on leaving the nozzle. The air to the wing tips of the nozzle serves only to shape the spray pattern of the atomized sulfur and has very little atomizing or asperating effect on the molten sulfur being sprayed.

(Fig. 4)

Variables which affect the production rate and the quality of the coated products include the following.

1. Coating weight of sulfur, wax, and conditioner (total coating weight). If all other variables are kept constant, the dissolution rate or slow-release characteristics of the final product are controlled entirely by how much sulfur and wax are applied to the urea. The graph in Figure 5 shows the standard quality of product that can be produced by the sulfur-coated urea pilot plant using a pneumatic sulfur-spraying system. The total coating requirement for a product with a desired dissolution rate can be estimated as well

as the nitrogen and sulfur contents. For example, a product having a 30% dissolution rate in 7 days can be produced with about a 13% total coating, thus, resulting in a product having a nitrogen content of 40% and a sulfur content of 8%. The graph is based on the use of excellent quality granular feed urea coated with a seal coating of 2% wax and 2% conditioner. The urea from which this graph was constructed had been coated with 1% of conditioner by the manufacturer to minimize caking.

(Fig. 5)

2. Size and condition of the urea. This has been previously mentioned but is reemphasized because of its extreme importance. The substrate material graphed in Figure 5 was spherical, smooth, and closely sized. Its Tyler screen size is given below.

+6 mesh = 0%
-6 +7 mesh = 9%
-7 +8 mesh = 70%
-8 +9 mesh = 19%
-9 mesh = 2%

3. Type and size of pneumatic sulfur spray nozzle. External atomizing nozzles are used as previously described and schematically pictured in Figure 4. The nozzles produce a finely atomized spray in the shape of an elongated oval.

Depending on the make of nozzles used, various alterations to the nozzles are needed to maximize the spraying efficiency and eliminate operational problems associated with startup and shutdown.

The nozzles should be sized for maximum atomization at the spray distance of 4-3/4 inches while discharging a sulfur spray with a density of about 2.5 pounds per square inch per hour at that distance.

4. Number of sulfur nozzles. Eight nozzles per ton of sulfur-coated urea per hour are considered as the optimum number. More than eight nozzles per ton are not desirable from the standpoints of ease in operations and simplicity in design. As the ratio of nozzles to production rate is reduced, the quality of the product is decreased (see Fig. 6). When the sulfur was put on with eight nozzles at the production rate of 1 ton per hour, a total coating of 13% (8% sulfur) gave a material with 7-day urea dissolution rate of 30%. When four nozzles were used at the same production rate, a total coating of 20% (15% sulfur) was required to give a material with the same 7-day urea dissolution rate of 30%. (Fig. 6)

5. Position and orientation of sulfur nozzles. For best results, the spray nozzle header is located parallel to the axis of the sulfur-coating drum. The nozzles in the header are 8 inches apart on centerline. The header is

located so each nozzle is spraying vertically downward onto the fastest moving section of the rolling bed from a distance of 4-3/4 inches. If the nozzles are placed farther away, the sulfur dust loading in the drum is increased and the quality of the product is decreased. Increasing the spray distance beyond about 8 inches from the bed is both impractical and hazardous as sulfur explosions may occur if concentrations of dust exceed 35 grams per cubic meter in air. Reducing the spray distance increases the sulfur spray density at the impact point and decreases the area of coverage by the spray nozzle. Both are detrimental to the quality of the coating.

6. Rotational speed of the sulfur-coating drum. Increasing the interaction of particles in the sulfur-coating drum and the speed by which they pass under the sulfur spray nozzles increases the quality of the sulfur coating. With the 4-foot-diameter sulfur-coating drum the best speed is 65 to 70% of critical speed (the speed at which centrifugal force holds the particles to the shell of the drum for its entire revolution). Beyond this speed, carryover of material becomes high and sulfur-urea buildup on the coating drum accelerates. Normally, there is some buildup on the shell of the sulfur-coating drum as a result of sulfur mist in the drum. In the

pilot-plant unit this buildup provides a rough surface on the drum which facilitates the rolling action of the granules in the drum. The errosive action of the granules rolling and bouncing around keeps the buildup in check. However, in a full-scale plant a cleaning device may be needed for the shell of the drum.

7. Atomizing air volume. The atomizing air provides the force to atomize the sulfur. The maximum available air pressure of 80 pounds per square inch gage is used. The volume of air used at this pressure is approximately 4.7 scfm per nozzle. This air is split so that the ratio of air through the wing tips of the nozzle to that which goes through the center opening is approximately 2.2 to 1.

8. Process temperatures. The process temperatures are critical. Without proper control of the temperatures the sulfur does not crystallize in the proper structure and the coating is less effective. However, because there is a seal coating of wax applied to the product in this process, some variation in the sulfur structure can be tolerated and the temperatures are somewhat less critical than in the sulfur-only process.

   Temperature of preheated urea. The urea should be preheated to a temperature between 145° and 150°F. If the urea is insufficiently preheated, the sulfur

coating has a crusty appearance and high dissolution rates result from an otherwise well-coated material. Much higher temperatures give trouble further along in the process.

Temperature of liquid sulfur. As previously mentioned, the best sulfur temperature for spraying is approximately 310°F. Tests have shown that as the temperature decreases, the effectiveness of the coating is reduced. Temperatures up to 317°F are acceptable but at temperatures of 318°F and above, the sulfur viscosity is so great that good atomization of the sulfur is virtually impossible.

Atomizing air. Air heated to the maximum temperature attainable in the pilot-plant system (between 290° and 300°F) is used for atomizing the sulfur with good results. Somewhat higher temperatures of around 315°F are probably more desirable.

Sulfur-coating drum temperature. As the material passes through the sulfur-coating drum, it increases in temperature because it receives the heat of fusion released by the solidifying sulfur. When a high oil microcrystalline wax is used, it is important that the wax be applied to granules with a temperature of 155° to 160°F. The urea must, however, be above the freezing point of the wax for the roll-on distribution system

to be effective. The wax must be able to flow onto the granules and into the imperfections before it freezes. The urea loses 5° to 10°F between the sulfur-coating drum and the wax-coating drum; therefore, the sulfur-coating drum exit temperature is controlled at about 165°F.

Temperature of liquid wax. When using the wax referred to above, a preheat temperature of 220°F is used. The wax temperature does not appear critical. When applied, the wax quickly takes on the temperature of the sulfur-coated urea granules because of their 50 times greater mass.

Cooling temperatures. The liquid wax applied to the sulfur-coated urea must be solidified before storage. Using the microcrystalline wax with a high oil content requires cooling the product to a maximum of 105°F. Unless this is done, the wax remains too sticky and must be coated with excessive amounts of conditioner to prevent poor handling characteristics. As previously mentioned, a fluid bed cooler does this cooling job quite adequately without chilled air, provided the bed is fluid at all times and the material is never allowed to come in contact with the fluidizing screen. It is important that the cooling air temperature be no lower than about 60°F. At temperatures cooler than this, the wax freezes too fast and agglomerates are formed.

Sulfur-Coated Urea Without Sealant

In order to make a slow-release urea fertilizer coated only with sulfur, the sulfur crystalline structure and uniformity must be closely controlled, for there is nothing to seal imperfections in the coating. However, several of the steps in the sealant process can be eliminated and some of the other processing equipment can be substantially reduced in size. Since there is no wax or conditioner added, all equipment associated with their application can be omitted. Cooling is still important, but a substantial reduction in cooling capacity can be made. Handling complications of the waxed product are eliminated, greatly simplifying the engineering design of the cooler and associated product-handling equipment. On the negative side, more preheating capacity is needed and the sulfur-coating drum throughput capacity is only half of that when a sealant is used because of the need for increased amounts of sulfur. A process diagram of the pilot plant when producing 1/2 ton per hour of urea coated only with sulfur is shown in Figure 7.

(Fig. 7)

Variables 2 through 7 mentioned in the preceding section are more important than when a sealant is used, but their specified values remain the same.

Variable 1--the total coating weight-- is substantially changed. The dissolution rate of the product still varies with the

amount of coating applied, but now the coating is only sulfur. About 50% more of it is required on an actual sulfur basis to give a specific dissolution rate. However, the total coating of the product is only increased by 4-1/2 percentage points (see Fig. 5). A total coating of 18% (17% sulfur, 1% preapplied clay conditioner) gives a 7-day dissolution rate of 30%. A total coating of only 13.5% is required to give a 7-day dissolution rate of 30% when wax sealant is used. The sealant coating of 2% wax and 2% conditioner has a raw material cost of about $19 per ton of nitrogen coated if it can be obtained. The additional coating of sulfur in the sulfur-only product has a raw material cost of about $9 per ton of nitrogen coated. Thus, the sulfur-only coating is very competitive even though it does have a lower nitrogen content in its final product, 38% as compared with 40% for sealant coating. (The above figures were all based on using minus 6- plus 10-mesh substrate urea, producing a product having a 7-day dissolution rate of 30%, and January 1974 costs of sulfur, wax, and conditioner f.o.b. Muscle Shoals, Alabama.)

The process temperatures (variable 8) are very different from those used when applying a sealant with the exception of the molten sulfur and its atomizing air which are still maintained at about 310° and 295°F, respectively. The most critical temperature is that of the substrate when the sulfur is applied. At no place in the sulfur-coating drum should the temperature of the substrate be allowed to

exceed 204°F. Yet, the closer the temperature is to this value, the better the quality of the final product. Therefore, the preheater exit temperature is regulated to give a sulfur-coating drum exit temperature of from 195° to 200°F, which is about as close as the temperature can be controlled in the pilot plant. At a production rate of 1/2 ton per hour in the pilot plant, the temperature of the material exiting the preheater or entering the sulfur-coating drum is approximately 175°F. Figure 8 shows the effect of the substrate temperature on the quality of the final product. This figure also shows that if the temperature of the material is allowed to exceed 204°F in the sulfur-coating drum, then quick cooling, such as can be attained in the fluid bed cooler, will prevent a complete degradation of the protective coating. It has also been shown that some quick cooling is helpful on product where the temperature does not exceed 204°F. Therefore, use of a small cooler is recommended.

(Fig. 8)

HYDRAULIC SULFUR-SPRAYING SYSTEM

Sulfur-Coated Urea With Sealant

Figure 9 is a flow diagram of the hydraulic spraying system used in the 1-ton-per-hour sulfur-coated urea pilot plant. Since nozzle openings are smaller (0.007-0.015) than the orifices used in the pneumatic system, the filtration of the sulfur in this system is even more important.

A 400-mesh screen is installed in the sulfur line from the weigh tank to remove carsul, ash, and other foreign materials. The sulfur then flows through a 2-micron filter to remove any small particles which might still be in the sulfur and which might combine to form larger particles farther downstream. Sulfur is pumped by a double-acting air-driven piston pump capable of developing pressures up to 3000 psig. Because of the high temperatures and incompatibility of sulfur and hydrocarbons, the pump is lubricated with a fluorocarbon lubricant. All of the piping downstream from the sulfur pump is stainless steel high-pressure piping which was tested at 4500 psig. A pulse eliminator tank is located downstream from the sulfur pump. Air trapped in the top of the tank provides a constant pressure on the sulfur spray nozzles and thus maintains a constant flow during changes in the stroke direction of the pump. A 200-mesh filter is installed in the pulse eliminator primarily to take out trash which might get into the line during maintenance of the pump. Pump pressure is regulated by the amount of air pressure applied to the piston air motor. The flow rate of sulfur is determined by the combination of this pressure and the size and number of spray nozzles installed in the sulfur header. The only way of reducing sulfur flow through a given spray nozzle or set of spray nozzles is to reduce the pressure in the hydraulic system.

(Fig. 9)

To provide for depressurization of the system, a recycle line is connected between the pump and the sulfur weigh tank. A pressure relief valve is installed on the feed side of the pump to prevent possible pressurization of this portion of the piping should a malfunction of the pump allow backflow from the pulse eliminator tank. Air is provided for purging of the sulfur line and for back cleaning the 400-mesh screen in the sulfur weigh tank. The sulfur pump and all pipe fittings and valves not steam jacketed are submerged in a molten sulfur bath to keep sulfur from freezing in them during operating periods.

The spray header itself is much simpler than the spray header of the pneumatic system. No in-line flow control orifice is needed since each nozzle at these high pressures acts as its own flow control orifice. Further, because no air is used, no pneumatic piping is needed. A high-pressure union allows the header to be rotated so that the angle of spray with reference to the bed can be easily changed. Also, the position of the nozzles with reference to the bed of urea can be altered as desired by use of externally mounted adjustable hangers. The entire header unit is steam jacketed.

Except for the absence of any pneumatic equipment, the simplified flow diagram for the hydraulic spraying system is the same that is shown in Figure 1 for the pneumatic spraying system. However, major changes as discussed previously were made to the internals of

the sulfur-coating drum and to its operation to provide a curtain of falling urea granules. The sulfur is sprayed onto this curtain. The resulting product is not so high in nitrogen content as that made using pneumatic nozzles (see Fig. 5), but the process is much more operational. A total coating of 18% gives a product with a 7-day dissolution rate of 30% and a nitrogen content of about 38%.

A primary reason for the installation of a hydraulic spraying system was to decrease sulfur mist and dust formation in the sulfur-coating drum. This has been accomplished. Based on an airflow through the sulfur-coating drum of 200 scfm, dust loading in the atmosphere of the sulfur-coating drum ranged from 0.068 to 0.163 gram per cubic meter per pound of sulfur sprayed when pneumatic nozzles were used. With the use of hydraulic nozzles, the dust loading has been reduced 88 to 98%. The dust loadings have ranged from 0.003 to 0.008 gram per cubic meter per pound of sulfur sprayed. Problems associated with the dust have been either substantially reduced or eliminated.

Safety. The minimum explosive concentration for sulfur is 35 grams per cubic meter of air. The heaviest dust loading measured in the sulfur-coating drum while using hydraulic atomizing nozzles was 3.2 grams per cubic meter of air, well below the explosive range.

Air pollution abatement. Dust loadings for best operating conditions were less than 0.006 gram per cubic meter of airflow per pound of sulfur sprayed. Further cleanup of

the discharged air, if required, appears to be quite easy, probably involving only a heated cyclone separator.

Sulfur-coating drum buildups. Such buildups appear to have been eliminated. A related problem, production of agglomerates, caused by sulfur particles coalescing on the hot header piping with subsequent dripping of molten sulfur into the bed of urea, has also been substantially reduced.

Variables which affect the hydraulic process are generally the same ones which affected the pneumatic process with some exceptions. However, the relative importance and optimum values of these variables are quite different in a number of cases.

1. Coating weights of sulfur, wax, and conditioner (total coating weight). Provided all of the other variables are kept constant, the dissolution rate or slow-release characteristics of the final product are still controlled by how much sulfur and wax are applied. However, slight changes in coating content have a pronounced effect on the final product when using the hydraulic process (see Fig. 5). When sulfur was applied by using the pneumatic spray process, a total coating reduction of 5 percentage points from 18.5 to 13.5% gave an increase in 7-day dissolution rate from 15 to 30%. With the hydraulic process, a reduction of only 2 percentage points, 20 to 18%, gave the same increase in 7-day dissolution rate, 15 to 30%.

2. Size and condition of the urea. There is increased mixing action in the sulfur-coating drum which is a result of the lifting flights and the falling curtain of urea. There is less particle segregation and thus more uniform coating between particles. Therefore, particle size and surface condition are not as critical with this system, although they are still extremely important.

3. Type and size of hydraulic sulfur spray nozzle. The hydraulic spray nozzles used are very simple. They have no vanes, cones, or other inserts which can give problems. Sulfur is forced by pressure through an elongated oval making a highly atomized fan pattern. The nozzle tip size is varied depending on the amount of sulfur to be sprayed and the desired atomizing pressure. Changes can be made quickly and easily. A final protective filter is inserted into each nozzle assembly prior to the nozzle tip (see Fig. 10). The final filter, the mesh size of which varies depending on the size of the orifice in the nozzle tip, normally removes very little material, and thus does not present a cleaning problem.

(Fig. 10)

4. Hydraulic pressure. The pilot plant has the capability of spraying at pressures up to 3000 psig. Theoretically, nozzle atomization efficiency increases with pressure thus making the highest pressure attainable and the smallest nozzle available the most desirable for application of the sulfur.

However, filtration problems associated with the smallest nozzles combined with the practical number of nozzles that can be used in the process make a nozzle with a tip opening of 0.011 or 0.013 the most desirable. These nozzles are operated in the range between 750 to 1500 psig. Tests showed that increases in pressure up to 3000 psig had very little effect on the final product using these nozzles. The lower operating pressures are much more desirable from an operational standpoint.

5. Position of the sulfur nozzles. For best results, the nozzles are oriented to spray sulfur perpendicularly onto the falling curtain of urea at a point 2 inches above the bottom of the curtain. The distance between the nozzles and the curtain is 5-1/2 inches. The nozzles in the header are 7-3/4 inches apart.

6. Number of sulfur nozzles. The number of sulfur nozzles should be minimized. Four nozzles per ton of production appear to give optimum results.

7. Rotational speed of the sulfur-coating drum. The rotational speed of the sulfur-coating drum still affects the quality of the coating. However, it has much less effect than with the pneumatic spraying system. The density of the falling curtain of urea and the mixing action in the drum are

increased by higher drum speeds; however, the speed at which the granules pass in front of the nozzles (which is extremely important) is not affected by changes in drum speed in the hydraulic process.

8. Process temperatures. The process temperature control is less critical in the hydraulic process than in the pneumatic system.

Temperature of preheated urea. Data show that the urea needs to be preheated only to approximately 100°F for sulfur-coating purposes. However, material must be 160°F when coated with the microcrystalline wax. Therefore, for the best product the urea should be preheated to at least 130°F.

Liquid sulfur temperature. Best temperature for the liquid sulfur is still 310°F.

Sulfur-coating drum temperature. The sulfur-coating drum temperature should not be allowed to exceed approximately 200°F. Best results were obtained when the exit temperature of the sulfur-coated urea was maintained between 160° and 185°F.

Wax-coating temperature. Wax-coating temperatures do not appear to be so critical in the hydraulic process. Good results are obtained when the wax-coating temperature is between 155° and 180°F.

Other temperatures and conditions not associated directly with the application of the sulfur and wax (e.g., cooling) remain the same as in the pneumatic spraying process.

Sulfur-Coated Urea Without a Sealant

When making a slow-release fertilizer coated only with sulfur using the hydraulic spraying system, coating conditions are much different from those used with the pneumatic nozzle system. The basic flow diagram is the same with the exception, of course, that no atomizing air is used (see Fig. 7). Operating variables 2, 3, 4, 5, and 7, discussed in the previous hydraulic spraying system section, are the same when producing a sulfur-only product. However, the use of eight instead of four nozzles per ton of production (Item 6) will give better results. Figure 5 shows the quality of product which can be produced at 1 ton per hour. Increasing the production rate up to 2 tons per hour requires only an increase of 2 percentage points of sulfur to maintain the same 7-day dissolution rate. As discussed previously, the same equipment when used in the pneumatic spraying system will only produce about 1/2 ton per hour of sulfur-only product or one-fourth the rate obtainable with the hydraulic process. The best product with a 30% 7-day dissolution rate made in the hydraulic system (at 1 ton/hr) is not so high in nitrogen content as the best product made with pneumatic nozzles (at 1/2 ton/hr). However, at equal production rates of 1 ton per hour or higher, the hydraulically produced material is much superior.

Process temperatures in the hydraulic system are much lower than those in the pneumatic system. The urea should be preheated to at least 135°F for best results, but the temperature in the sulfur-coating drum should not be allowed to exceed 175°F. At operating temperatures on either side of these values, the product quality falls off rapidly. Quick cooling of the final product to about 130°F is necessary to prevent some degradation of the product.

SULFUR-COATED UREA IN THE FUTURE

Research work on the production and use of coated urea for controlled release of nitrogen is being continued. TVA hopes to be able to reduce the ratio of nozzles to production rate and further decrease the size of the sulfur-coating equipment. A thorough agronomic evaluation of the material coated only with sulfur is being conducted which will fully indicate its potentials as a slow-release fertilizer.

TVA plans to initiate demonstration-scale production of sulfur-coated urea in about 1975-76. Production rate for this unit will be about 10 tons per hour.

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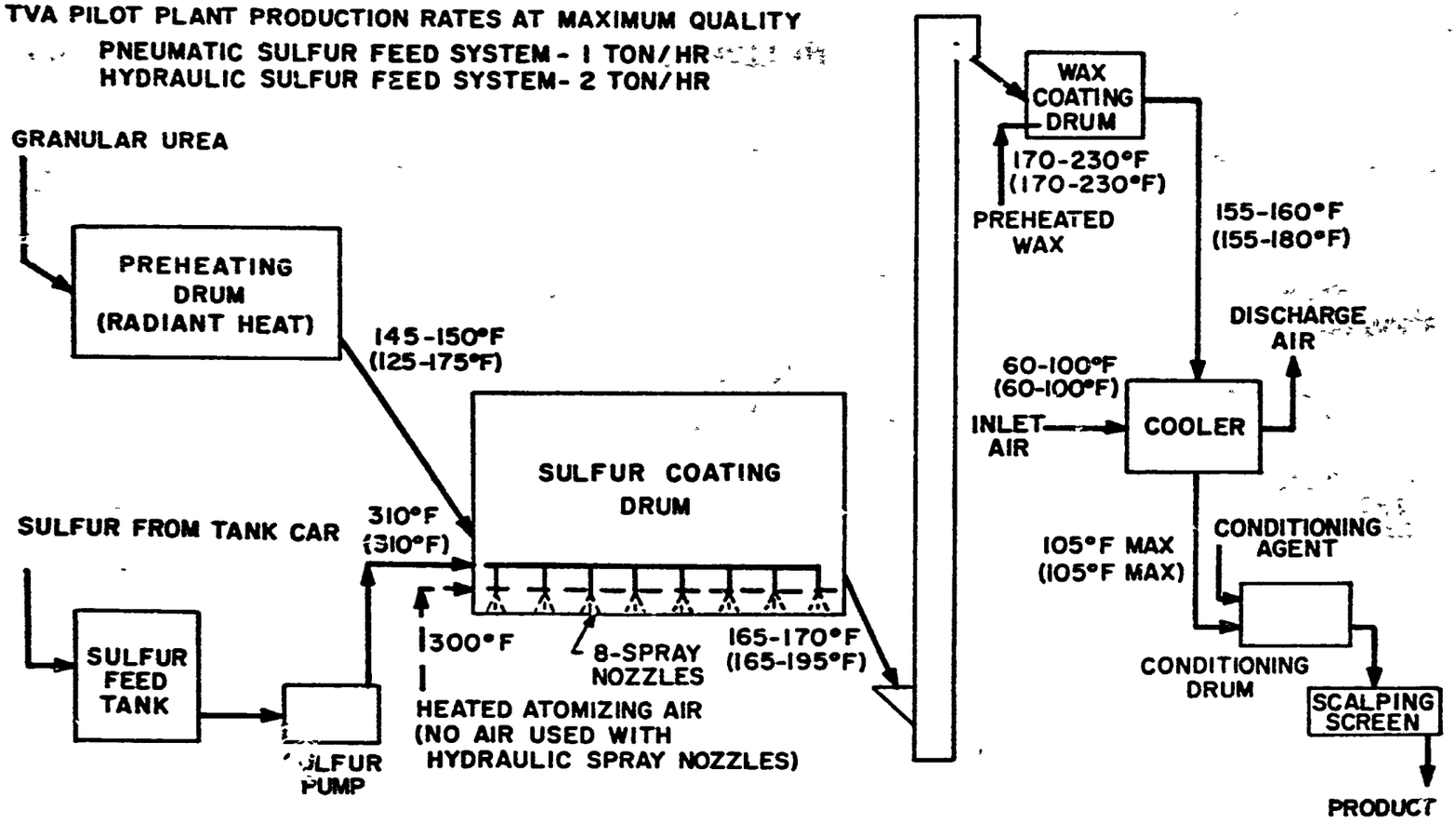
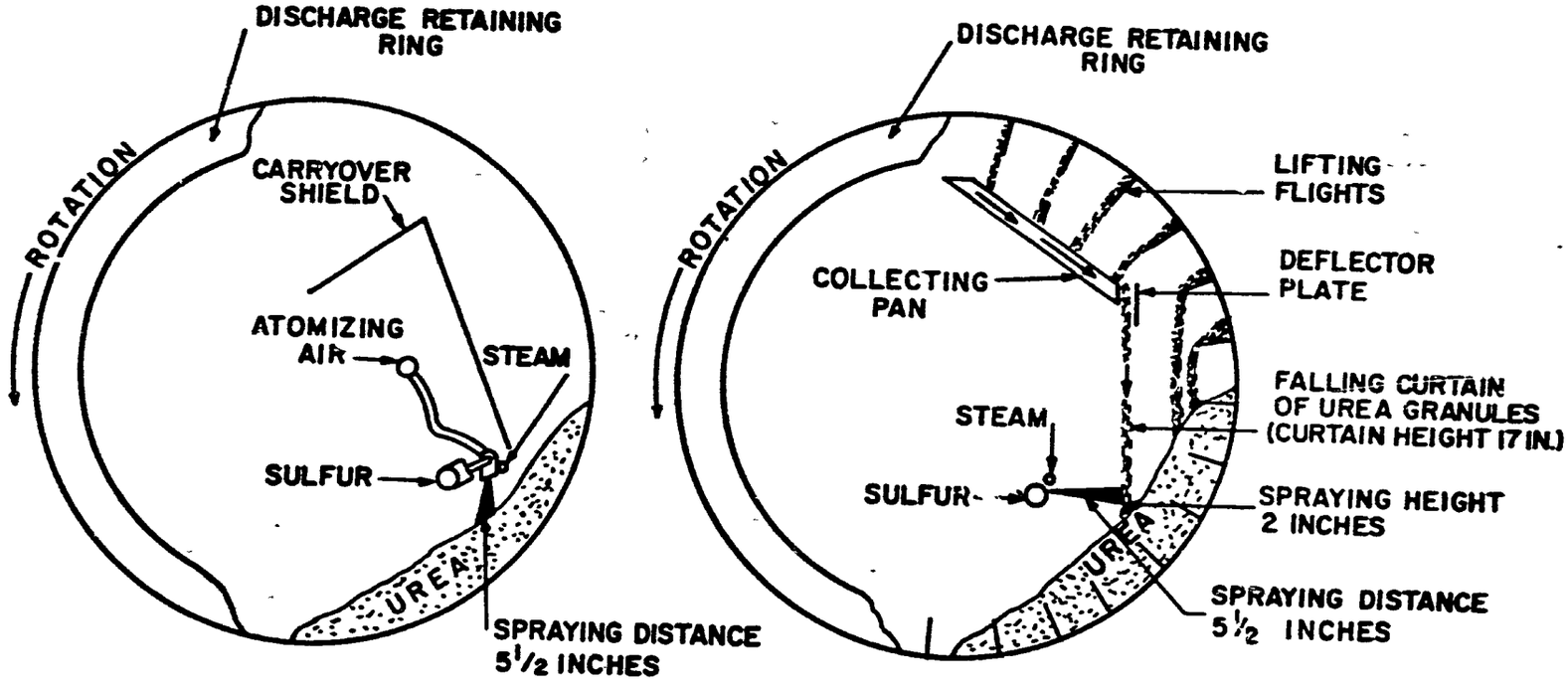


TEMPERATURE CONDITIONS FOR PROCESS WITH A HYDRAULIC SULFUR FEED SYSTEM ARE SHOWN IN PARENTHESIS

FIGURE 1. FLOWSHEET OF PROCESS FOR SULFUR-COATING UREA WITH ADDITION OF A WAX SEALANT USING EITHER PNEUMATIC OR HYDRAULIC SULFUR SPRAYING SYSTEM



USING PNEUMATIC SULFUR SPRAYING

USING HYDRAULIC SULFUR SPRAYING

FIGURE 2. SULFUR COATING DRUM OPERATION

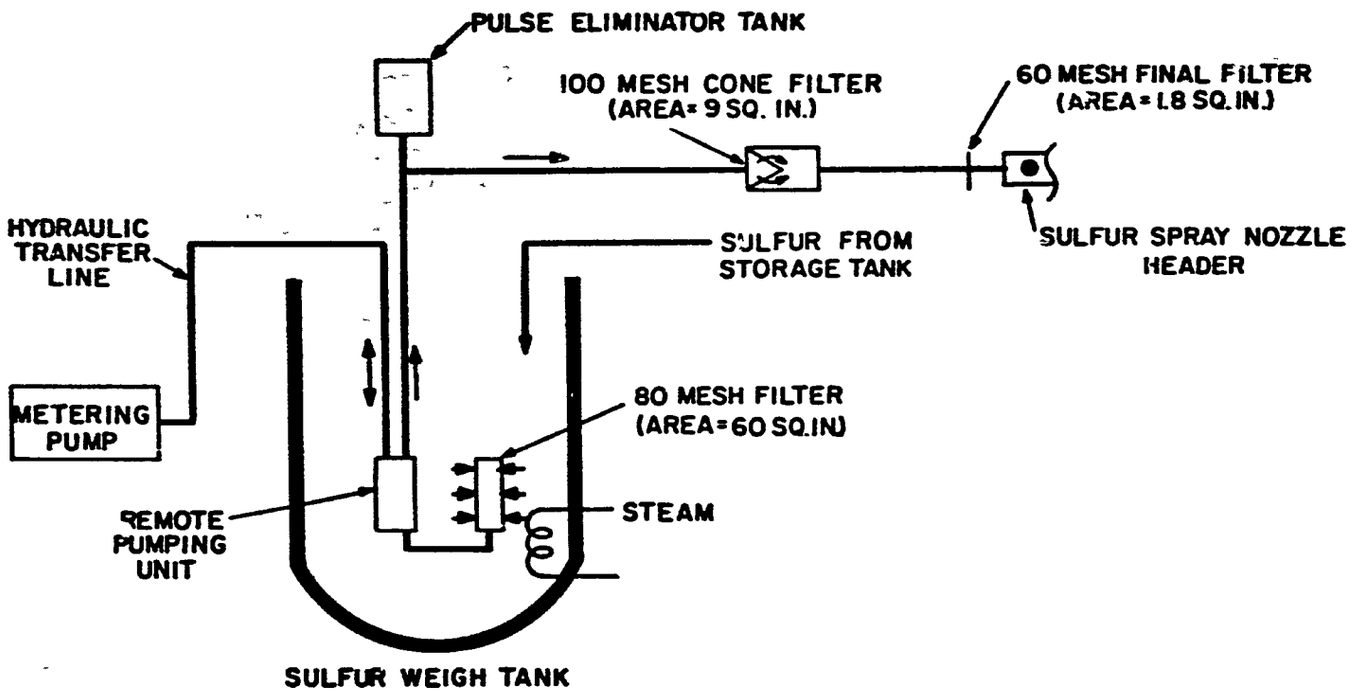


FIGURE 3. FLOW DIAGRAM OF SYSTEM FOR SUPPLYING SULFUR TO PNEUMATIC SPRAY NOZZLES

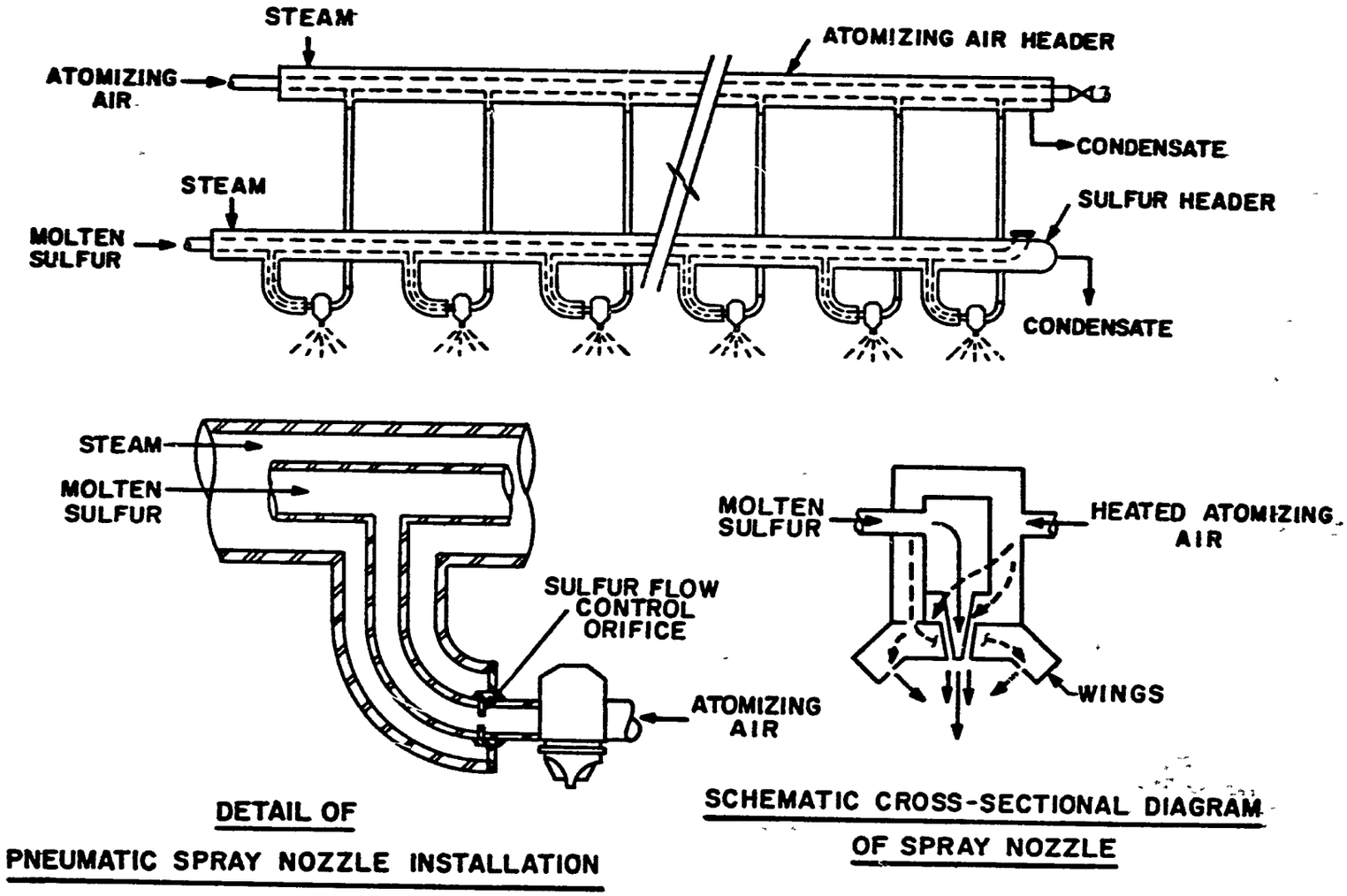


FIGURE 4. SULFUR SPRAY HEADER SYSTEM AND SPRAY NOZZLE DETAILS

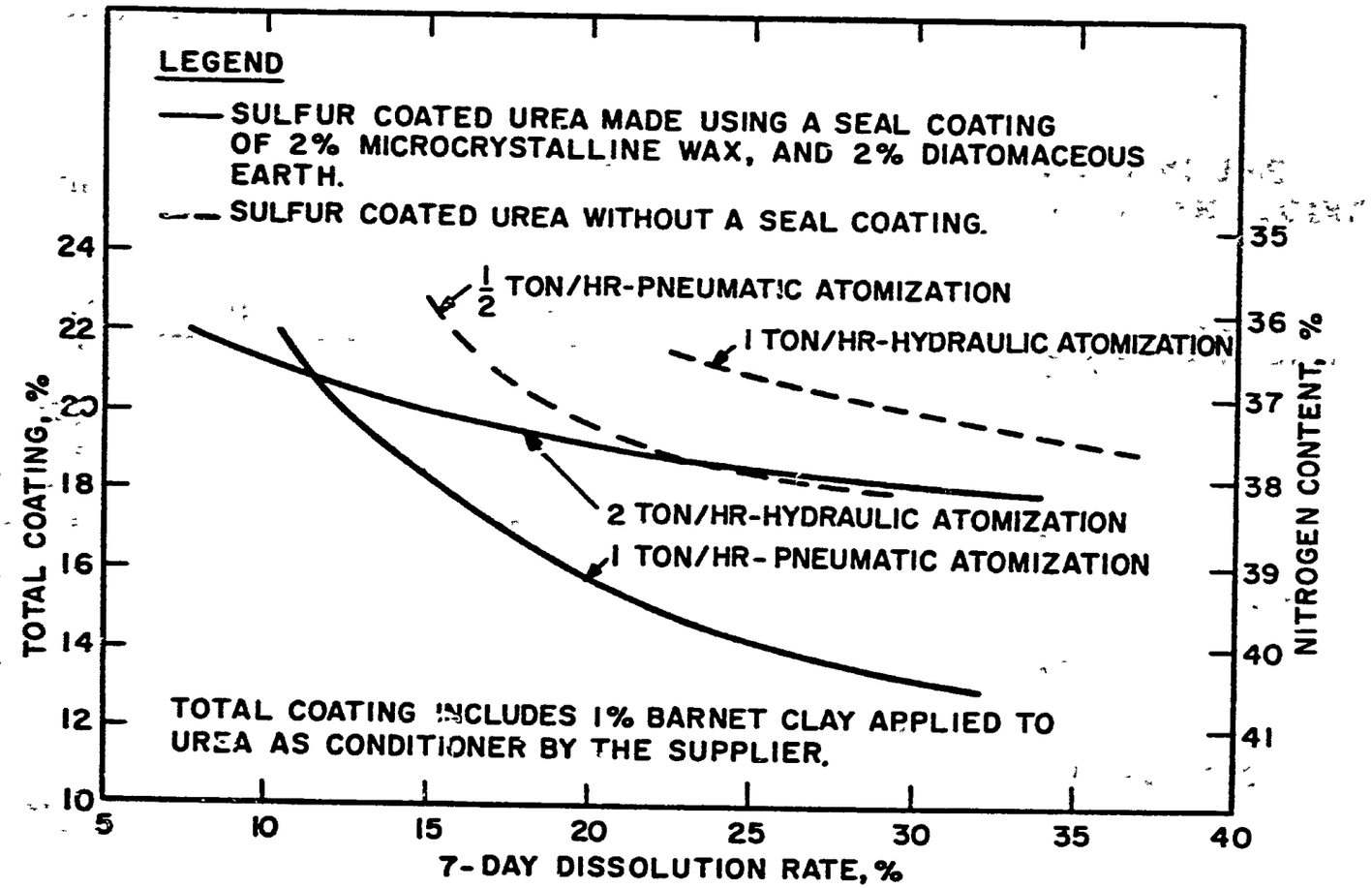


FIGURE 5. SULFUR COATED UREA QUALITY USING OPTIMUM PRODUCTION RATES AND OPERATING CONDITIONS FOR PNEUMATIC AND HYDRAULIC SPRAYING SYSTEMS.

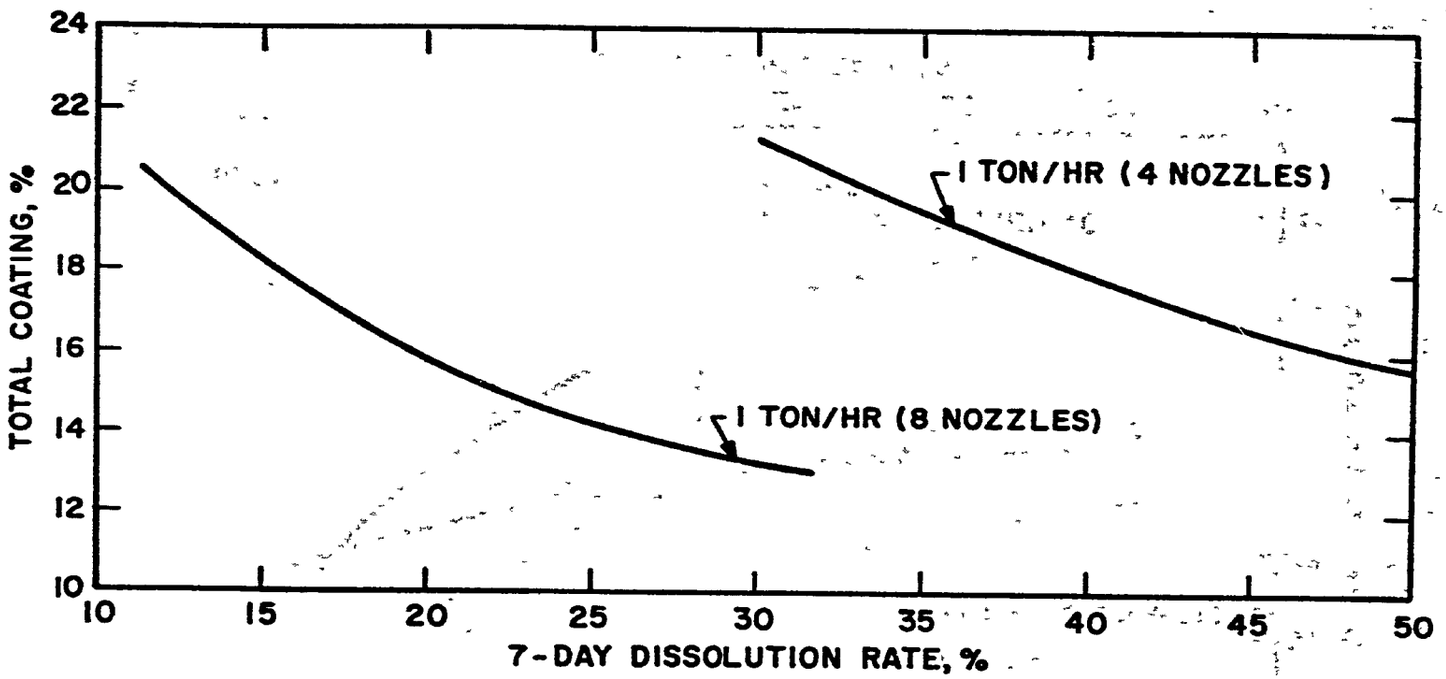


FIGURE 6. EFFECT OF EIGHT VERSUS FOUR SPRAY NOZZLES ON THE TOTAL COATING REQUIREMENT FOR SULFUR-COATED UREA AT THE PRODUCTION RATE OF I TON/HR.

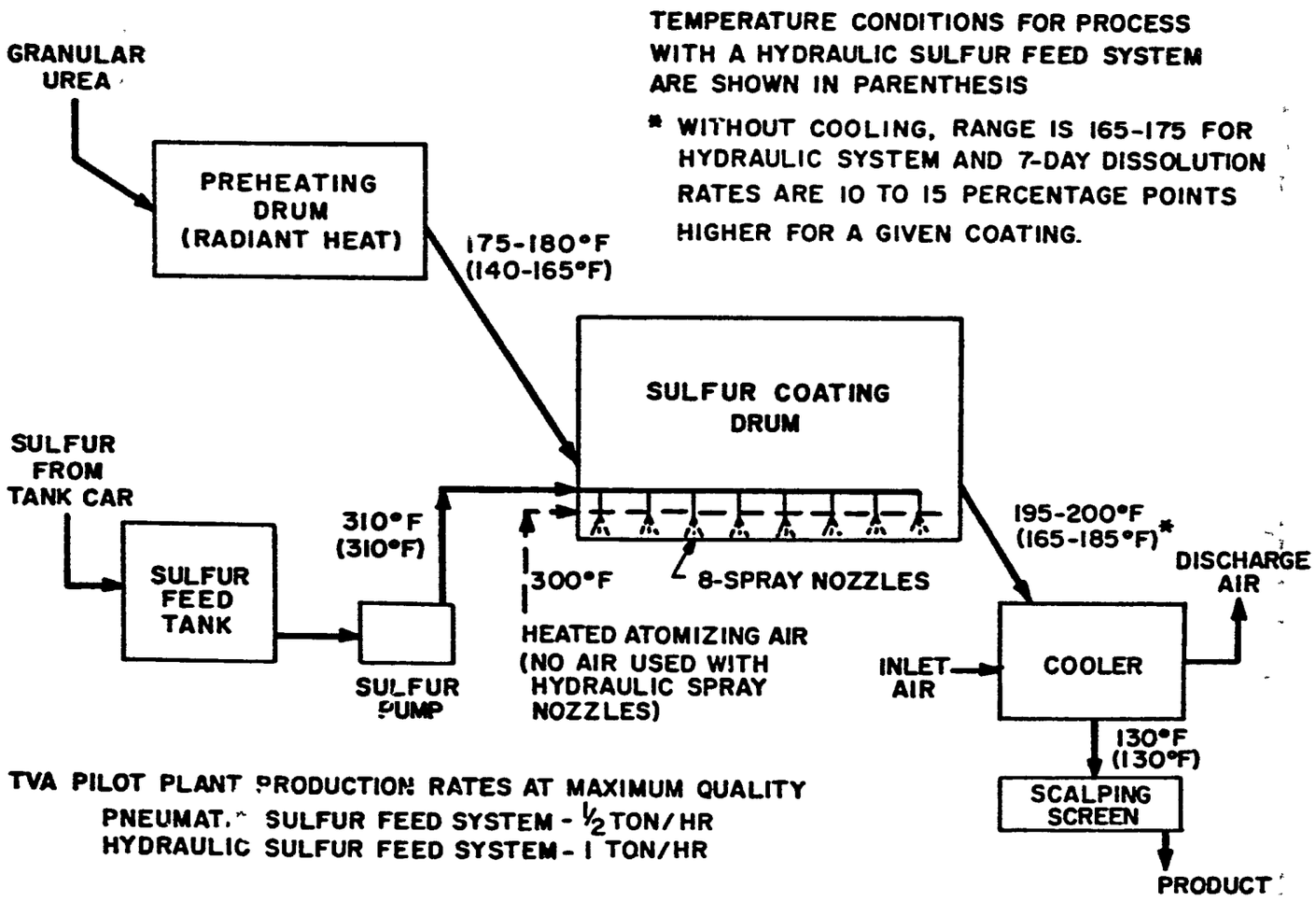


FIGURE 7. FLOWSHEET OF PROCESS FOR SULFUR-COATING UREA WITHOUT SEALANT USING EITHER THE PNEUMATIC OR HYDRAULIC SULFUR SPRAYING SYSTEM

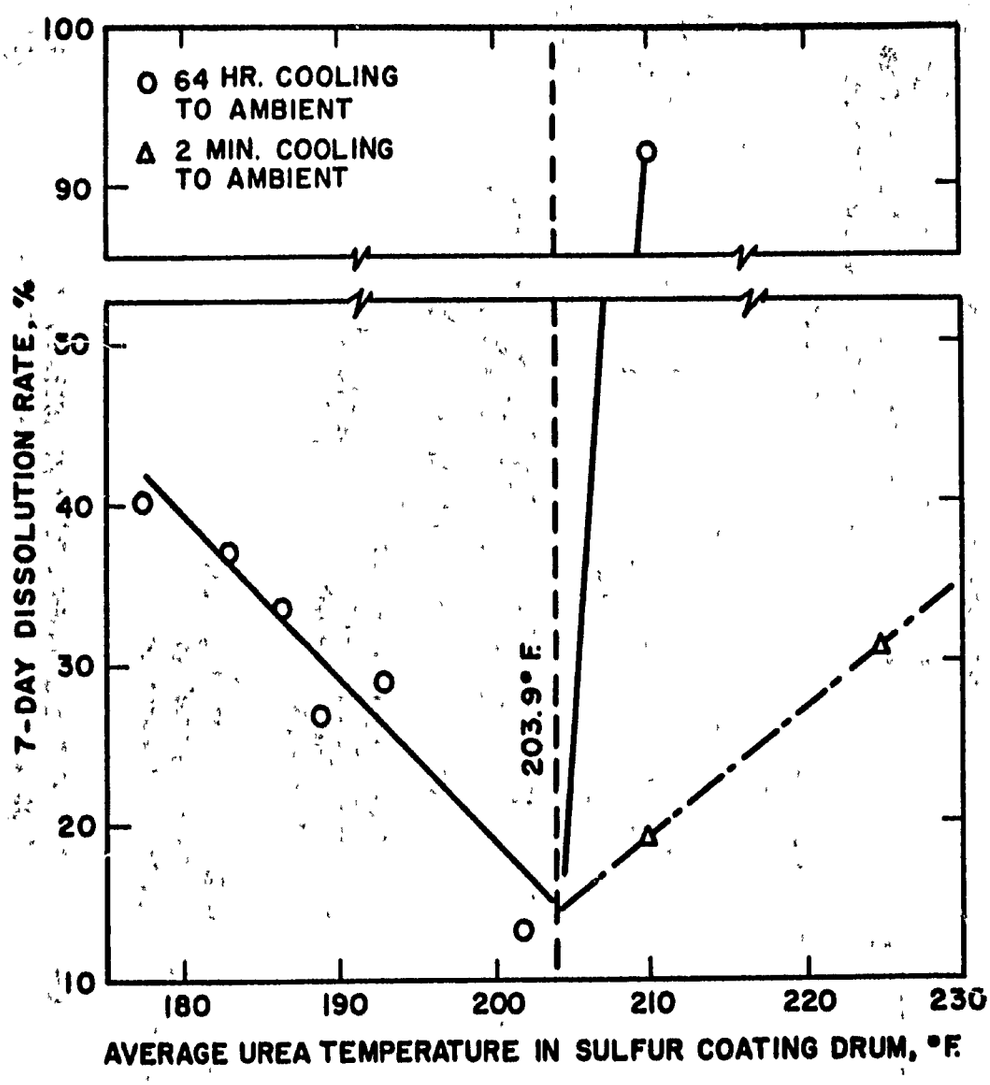
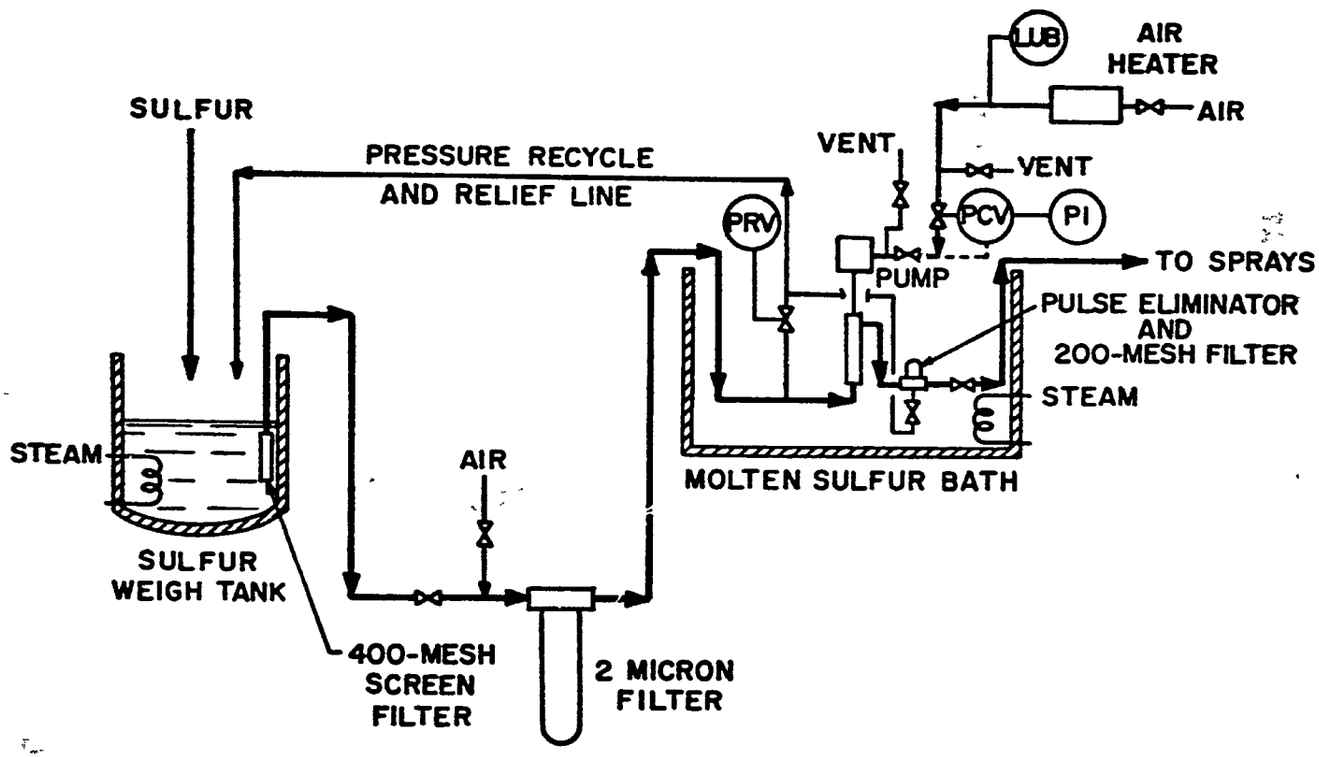


FIGURE 8. EFFECTS OF UREA TEMPERATURE IN SULFUR-COATING DRUM ON DISSOLUTION OF SULFUR-COATED UREA WITH NO SEAL COATING (USING THE PNEUMATIC SULFUR SPRAYING PROCESS)



NOTE: ALL SULFUR LINES ARE STEAM JACKETED OR SUBMERGED IN MOLTEN SULFUR.

FIGURE 9. FLOW DIAGRAM OF SYSTEM FOR SUPPLYING SULFUR TO HYDRAULIC SPRAY NOZZLES

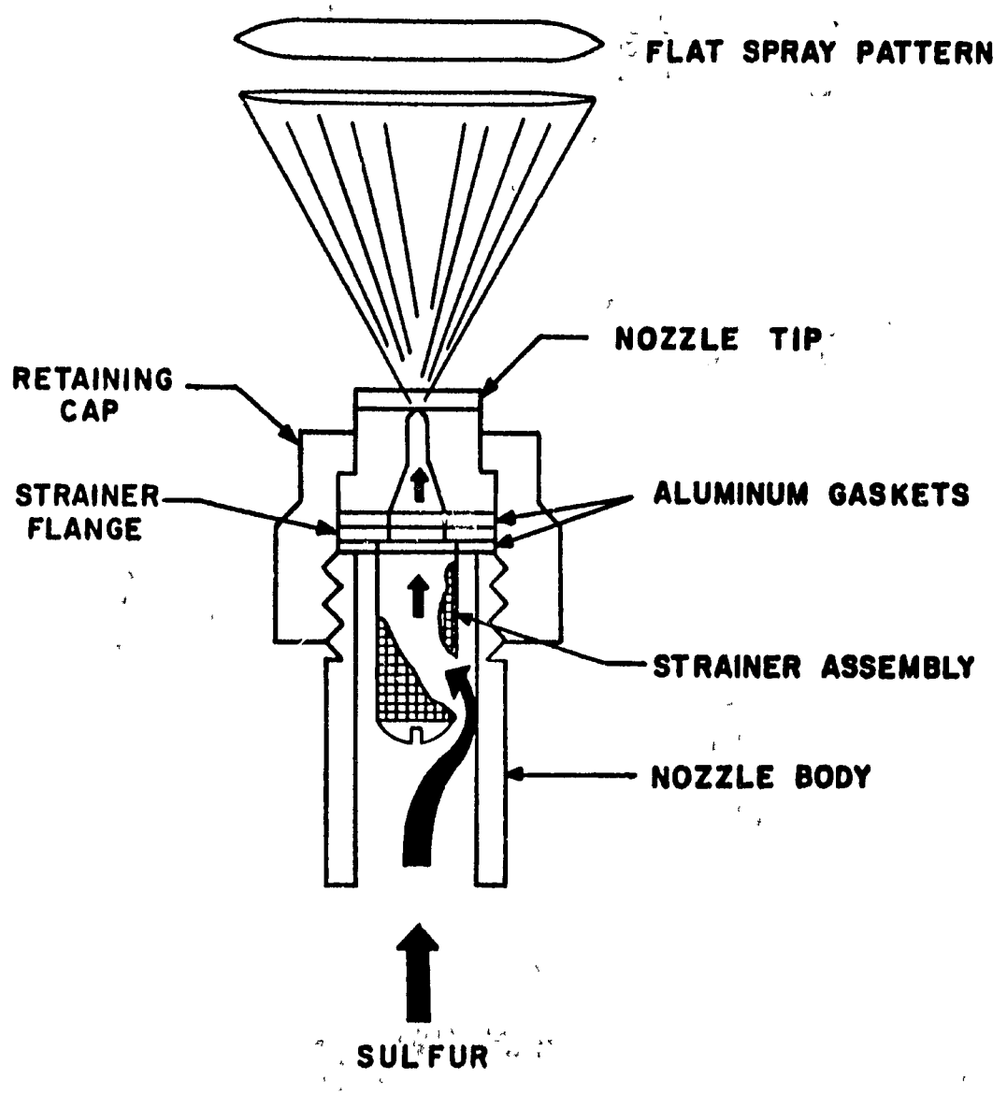


FIGURE 10. SCHEMATIC DIAGRAM OF HYDRAULIC SPRAY NOZZLE ASSEMBLY