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SULPHUR GRANULATION WITH KALTENBACH-THÜRING FLUID DRUM GRANULATION (FDG) PROCESS

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RESUME

Les procédés produisant du soufre sous forme sphérique sont devenus très importants à mesure que la consommation de soufre élémentaire augmentait. Comme le soufre est produit très loin des utilisateurs, il doit être transporté sur de longues distances et le soufre granulé est un mode très sûr de manutention et de transport.

Le procédé FDG Kaltenbach-Thüring a été essayé dans notre atelier pilote pour la granulation du soufre, après emploi convaincant pour la granulation de l'urée, du nitrate d'ammonium, du nitrate de calcium, du sulfate d'ammonium, du sulfonitrate d'ammonium, du superphosphate, etc. dans des unités grandeur nature.

La technologie FDG convenant bien à la granulation du soufre, Kaltenbach-Thüring a transformé son atelier pilote et commencé le développement. Les premiers résultats ont été (comme prévu) très encourageants et la qualité du soufre granulé ainsi produit correspond à des spécifications de produit haut de gamme (selon la définition SUDIC).

Le premier chapitre de cet exposé présente tous les résultats concernant les essais en pilote. Les analyses de qualité étaient semblables aux meilleures caractéristiques de ce qui a été publié jusqu'ici.

Les résultats des essais pilote permettent de proposer une application industrielle jusqu'à 1000 t/j.

Différentes options sont présentées et en particulier la simplification conséquente du procédé due à la disponibilité possible de fines de source extérieure ayant une granulométrie moyenne comprise entre 1,0 et 1,8 mm.

Pour une unité productrice de soufre grandeur nature, les consommations spécifiques d'énergie, d'utilités et de main-d'oeuvre sont intéressantes :

. *Electricité*	*9,6 kWh/t*
. *Vapeur*	*7,0 kg/t*
. *Eau de refroidissement*	*530 kg/t*
. *Personnel opératoire*	*Un homme par équipe*

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Air pollution regulatory bodies are becoming more concerned about SO_2 emission and are requiring higher recoveries. Most of the sulphur on the market is a by-product of oil and gas industry. Sulphur is removed in a hot molten state.

Sulphur is mainly used in a liquid form. Since both producer and consumer handle sulphur as liquid why bother with solid sulphur. Transportation is the reason.

When transported as a liquid provision must be made to reheat the sulphur at its destination. When consumer is connected to producer by truck, rail or barge it is most economical to ship liquid. When sulphur is shipped overseas the situation changes.

Shipping liquid requires vessels specially equipped and dedicated only to liquid sulphur. These ships cannot have different cargo on return trips. Shipping solid can be done with regular cargo ships. The cost advantage of this compensates the extra cost involved in converting liquid to a transportable solid and then remelting solid to liquid.

Continental or domestic sulphur is usually transported as liquid while transoceanic or offshore sulphur is shipped as solid sulphur.

Problem with solid sulphur is dust:

Dust is generated at every step of handling. Dust is an environmental contaminant, combining with moisture in soil or open water to create acid. Dust represent also a non-recoverable product loss. Accumulations of dust are highly flammable and can be ignited at relatively low temperature. Cigarettes, vehicule exhaust pipes, friction point of rotating equipment, all can start fires.

Sulphur has low electrical conductivity. This means that moving sulphur creates static electricity which can and has led to spontaneous explosion of accumulated dust sulphur. Dust during handling obscures visibility and is a serious eye and respiratory irritant.

In 1972 environmental and explosion hazard problems with crushed bulk have led to an embargo against its handling by the offshore terminal in Vancouver. The slating process was immediately adopted by all of the major export production plants in Canada. The slating process was an interim solution because dust was not eliminated.

In 1975 the Sulphur Development Institute of Canada (SUDIC) published the "PREMIUM PRODUCT SPECIFICATIONS" for the solid sulphur end product.

These specifications are listed on **Table 1**.

Product quality is a central issue when deciding upon a sulphur granulating process. Other critical considerations are process safety, environmental, capital and operating costs.

Critical characteristics of solid sulphur:

Minimizing generation of sulphur dust from the solid product during handling, storage and transport is the most important factor.

Various shapes of solid sulphur are manufactured today by different existing processes:

- air and water prilling
- drum granulation
- pastillators.

We will examine how the forming processes respect the characteristics specified by SUDIC and their positive-negative aspects.

Friability is probably the most important of all the factors that affect product quality. Shape, compaction, size, size distribution of solid product, the manner in which solids are formed and in which they age are only important as they relate to friability.

The first SUDIC specification states that premium product must be generally spherical. Spherical forms provide enhanced flowability when compares to the irregular shaped forms with flat surfaces and sharp edges. Non spherical sulphur forms suffer badly from impact, crushing and abrasion each time the product is moved and from rail car vibration during transport.

Compaction or crushed hardness values of air or water prills do not reach the high hardness results of granules.

TABLE 1

PREMIUM PRODUCT SPECIFICATIONS FOR SOLID SULPHUR FORMS
(published by Sulphur Development Institute of Canada (SUDIC) in 1975)

Shape:	Generally spherical
Size :	Between 2 mm and 4 mm diameter 75% between +/- 1 mm of average diameter
Size distribution:	Not more than 5% retained on a 4.75 mm (# 4) screen Less than 2 % passing a 1.19 mm (# 16 U.S.) screen Less than 0.1% passing a 300 microns (50 mesh U.S.) screen
Moisture content:	Less than 0.5% moisture (bulk average) as produced
Friability rating on performance:	Less than 1% of fines to 0.3 mm (-50 mesh material) generated under standard Stress Level I friability test (I) Less than 2% fines generated under standard Stress Level II test (II)
Bulk density:	Not less than 1044 kg/m^3 (65 lb/ft3) loose Not less than 1204 kg/m^3 (75 lb/ft3) packed
Compaction:	Fines, generated after direct shear test under static loading of 1.65 bar (24 psi) (approximately 15 meters (50 feet) stockpile): not to exceed 0.2% fines Fines generated after direct shear test under dynamic loading at 24 psi: not to exceed 0.75% fines
Angle of repose:	Not less than 25
Chemical purity:	As required by present specifications

(I) Standard stress level I test: refers to S4-77 SUDIC friability test in a 10 inches tumbler (450 revolutions at a speed of 19 + or - 1 RPM).

(II) Standard stress level II test : (40 revolutions at a speed of 31 + or - 1 RPM)

Prills are spherical products but they suffer from structural weakness due to the formation of a shell surrounding the remaining molten sulphur. As solid sulphur has a specific gravity less the molten one, an inward collapse of the outer shell occurs resulting in cratering and dimpling.

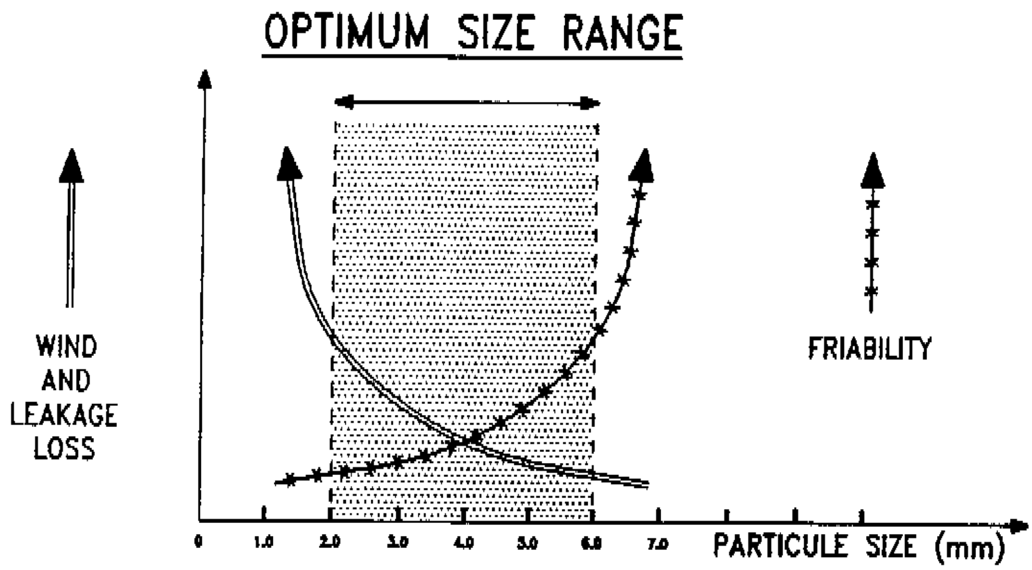
On the other hand the hardness of granules comes from the layering effect of successive sprayings. Crystallization occurs in each of their layer. Granules are more dense and hard than prills. Furthermore there is no cavity which can result in cracks as in the case of prills .

Size of solid product is an important factor. Small material is airborne under moderate wind conditions. Large product has a greater break-down by impact during handling. According to curves drawn of **Figure 1** the optimum size range is 2 of 6 mm.

Size distribution, high density and angle of repose are specified in premium product. There are factors for storage and transportation systems.

Moisture content is also a very important factor. Dry product do not create corrosion on handling and transportation equipment. Wet sulphur is more difficult to melt and requires more energy just to steam the trapped water. Each 1% of water content requires 20% more energy. A third comment might be pointed out: wet sulphur releases trapped water which is very acidic and a strong ground contaminant.

FIGURE 1



Internal cavities of prill is linked with the surface. Rain can penetrate cavities which takes a long time to drain off. Air prilled product can be a wet product after storage or transport.

Fluid drum granulator process:

The Fluid Drum Granulator (FDG) process is a combination of:

- one granulating drum consisting on a cylindrical horizontal drum turning around its axis and fitted with lifters
- one fluidized bed installed inside the granulator and fed with atmospheric air
- spraying headers fitted with nozzles.

This equipment is drawn on **Figure** 2.

Seed or granules to be fattened have to undergo a double operation in the granulator : size increase and crystallization by cooling. This is obtained in a cycle performed as follows:

- lifters bring the seeds or granules to be fattened to the upper part of the drum from where they fall on the surface of the fluidized bed
- on the fluidized bed product is cooled
- surface slope allows the product to flow down along the table from where it fall as a curtain into the lower part of the drum

- during the fall, cool product is sprayed with liquid sulphur, then with water
- lifters lift the granules coated with a new sulphur layer to be further crystallized.

Same cycle is then repeated as many times as necessary to reach the end product size.

<u>Characteristics of sulphur fluid drum granulation process:</u>

First of all resultant products are granules. As mentioned here above they are spherical, hard, dense and dry.

As part of the KALTENBACH-THÜRING process, water is sprayed over granules just wetted with a new sulphur layer. The reason for that is first to cool down as quickly as possible the liquid sulphur. Water vaporizes and exchanges heat at the right place.

Second, on the fluidized bed wetted granules are dried and cooled. Blown air helps the evaporation of water. In this way air is most efficiently used in the drum. Air flow rate is minimized.

By spraying water in the FDG the process is safe due to high humidity of air.

Sulphur fluid drum granulation unit is composed of three main items of equipment:

- a fluid drum granulator
- a screen
- a scrubber

Flow sheet is drawn on **Figure 3**.

The granulator is the core of the process. In a sulphur fluid drum granulator seeds are coming from:

- recycled undersize granules passing through the screen
- break-down of granules in the drum
- dust capted by the scrubber and recycled with discharged scrubber water

Mean size of the end product, production rate and efficiency of fattening process define the number of seeds and the residence time in granulator drum.

To control the internal load of the plant (that is to say the recycle ratio) a part of the end product is recycled with undersize granules. Output rate has to be equal to the sprayed molten sulphur rate.

Fattening ratio of a FDG unit is large. Recycle ratio is in the range of 20%. That means that major part of product coming in the FDG is small particules.

Fluidized bed is built of two successive tables. The first one handles smaller granules than the second final bed.

An elevator lifts the product coming out from the FDG to a screen fitted with just one deck. The screen separates the undersized granules.

Ambient air blown in the granulator is scrubbed in a wet venturi scrubber before being exhausted to atmosphere. Recovered dust sulphur is recycled in the FDG with discharged water.

Table 2 hereunder lists the final product quality which can be obtained in a KALTENBACH-THÜRING Fluid Drum Granulation Unit.

Table 3 summarizes the overall size of equipment as well as the consumption and effluents figures for a 1000 mtpd unit.

A general layout is shown on **Figures 4 and 5**.

<u>Conclusion</u>:

The KT FDG process in its application to sulphur granulation permits the product attains a quality superior in all aspects to the SUDIC premium product specifications.

KT sulphur granules are spherical, dense, hard, without internal hollow. They have a smooth surface and a low moisture content. Their friability factor is excellent and KT product can be considered as an industry granular sulphur.

The control of a low temperature all along the granulator minimizes degradation of granular sulphur as it ages. Major part of crystallized sulphur is already rhombic inside the FDG.

The plant operates safely. Process air flow is minimum. Its moisture content is high.

Capital cost is reduced by the use of small size equipment. Simplicity of the plant and the use of small size equipment are a direct result of the higher air efficiency.

Operating cost is reduced for the same reasons which are amplified by the small recycle rate.

The KALTENBACH-THÜRING Fluid Drum Granulation process takes care of the environmental aspects by a low sulphur emission as well as a low flow of gaseous effluent. There is no liquid effluent.

TABLE 2

PHYSICAL PROPERTIES OF FINAL PRODUCT
Compared to premium product specifications according to Sulphur Development Institute of Canada (SUDIC)

FINAL PRODUCT	SUDIC RECOMMENDATIONS
Mean size (d50) = 3.4 mm	between 2 and 4 mm
Size distribution	
. 1%max bigger than 4.75 mm	less than 5%
. 95% min between 4.4 and 2.4 mm	more than 75% with ± 1 mm
. 0.5% max smaller than to 2 mm	not defined
. 0.3% max smaller than 1.19 mm	less than 2%
. 0.1% max smaller than 300 microns	less than 0.1%
Moisture content	
0.15% (depends on raw material)	less than 0.5%
Friability performance	
.Standard stress level I Friability test (10 inches tumbler-S4-77 test)	
Fines : not measurable	less than 1%
.Standard stress level II friability test (28 inches tumbler - S5-77)	
Fines : 0.1%	less than 2%
Bulk density	
. loose : 1210 kg/m^3	not less than 1040 kg/m^3
. packed : 1230 kg/m^3	not less than 1200 kg/m^3
Angle of repose	not less than 25°.
= 30°.	
Crushing strength	not defined
Granules between — kg	
2 and 2.5 mm — 1.85	
2.5 and 3.0 mm — 2.20	
3.0 and 4.0 mm — 2.50	
4.0 and 5.0 mm — 3.20	
Compaction	
. Fines generated after direct shear test under STATIC loading at 24 psi less than 0.1%	not to exceed 0.2%
. Fines generated after direct shear test under DYNAMIC loading at 24 psi less than 0.5%	not to exceed 0.75% fines

TABLE 3

FOR A PRODUCTION OF 100 MTPD

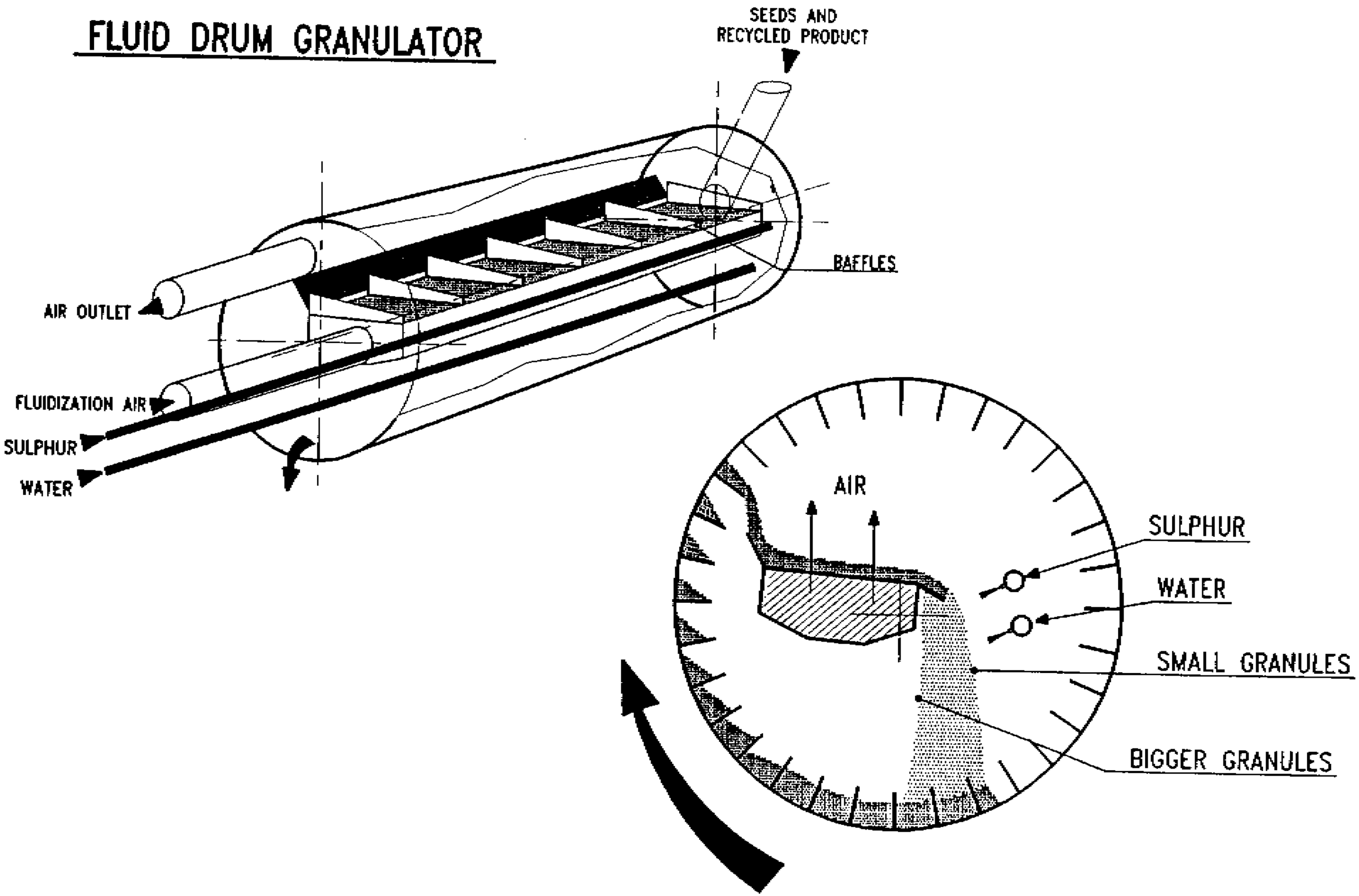
Granulator	:	Diameter Length	: :	2.20 meters 6 meters
Fluidized bed	:	Surface	:	2.8 m^2
Blown air	:	420 kg (365 m^3) per ton of sulphur		
Sprayed water	:	36 kg per ton of sulphur		
Exhaust air	:	460 kg (500 m^3) per ton of sulphur		
Effluents	:	Gaseous Liquid	: :	40 mg per standard cubic meter of air none

Energy consumptions

Steam	:	less than 1 kg per ton of sulphur (just used for sulphur jacketed line)
Electricity	:	3 kWh per ton of sulphur
Operator staff	:	one operator per shift
Ground overall dimensions	:	7 x 15 meters

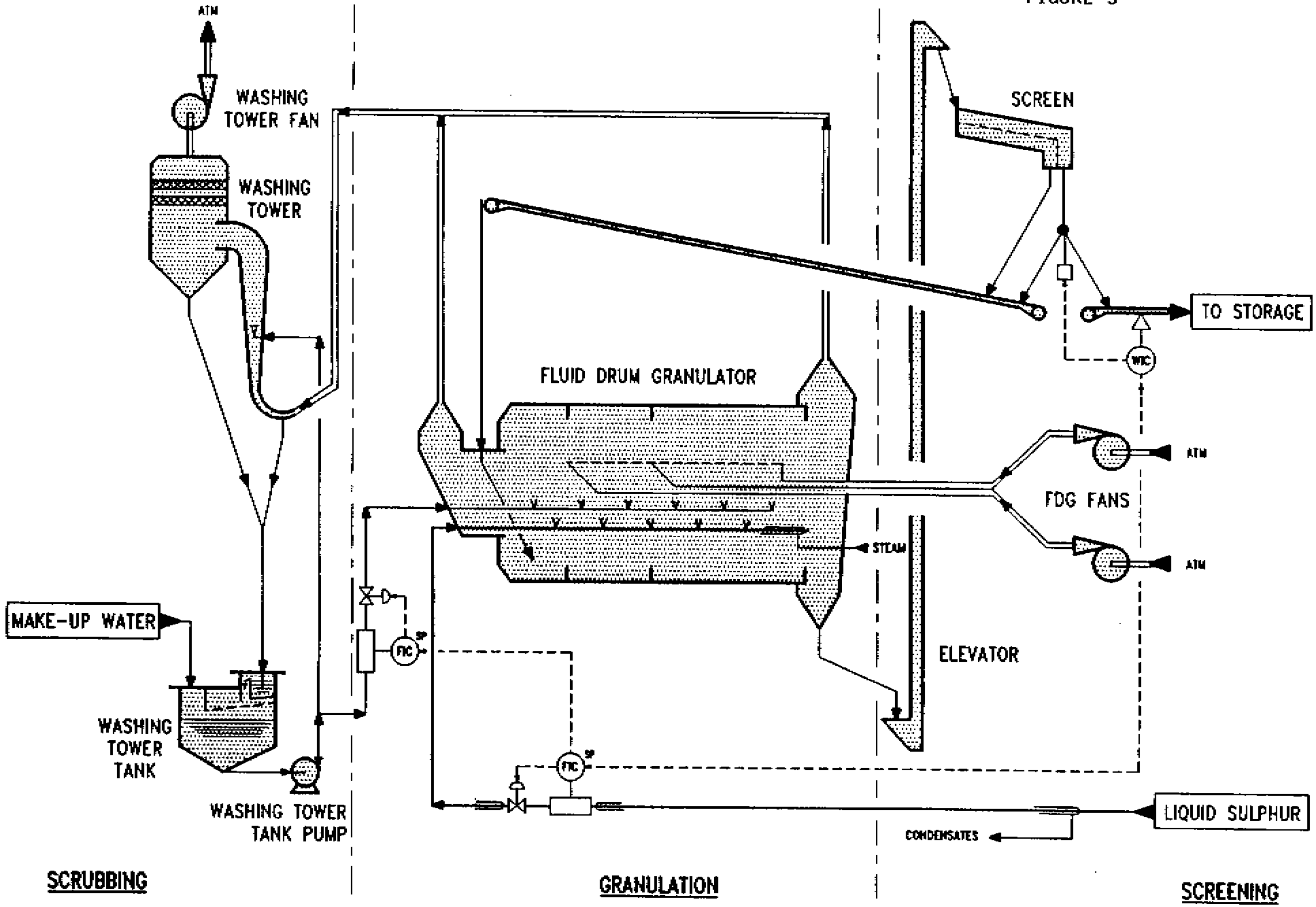
FIGURE 2

FLUID DRUM GRANULATOR



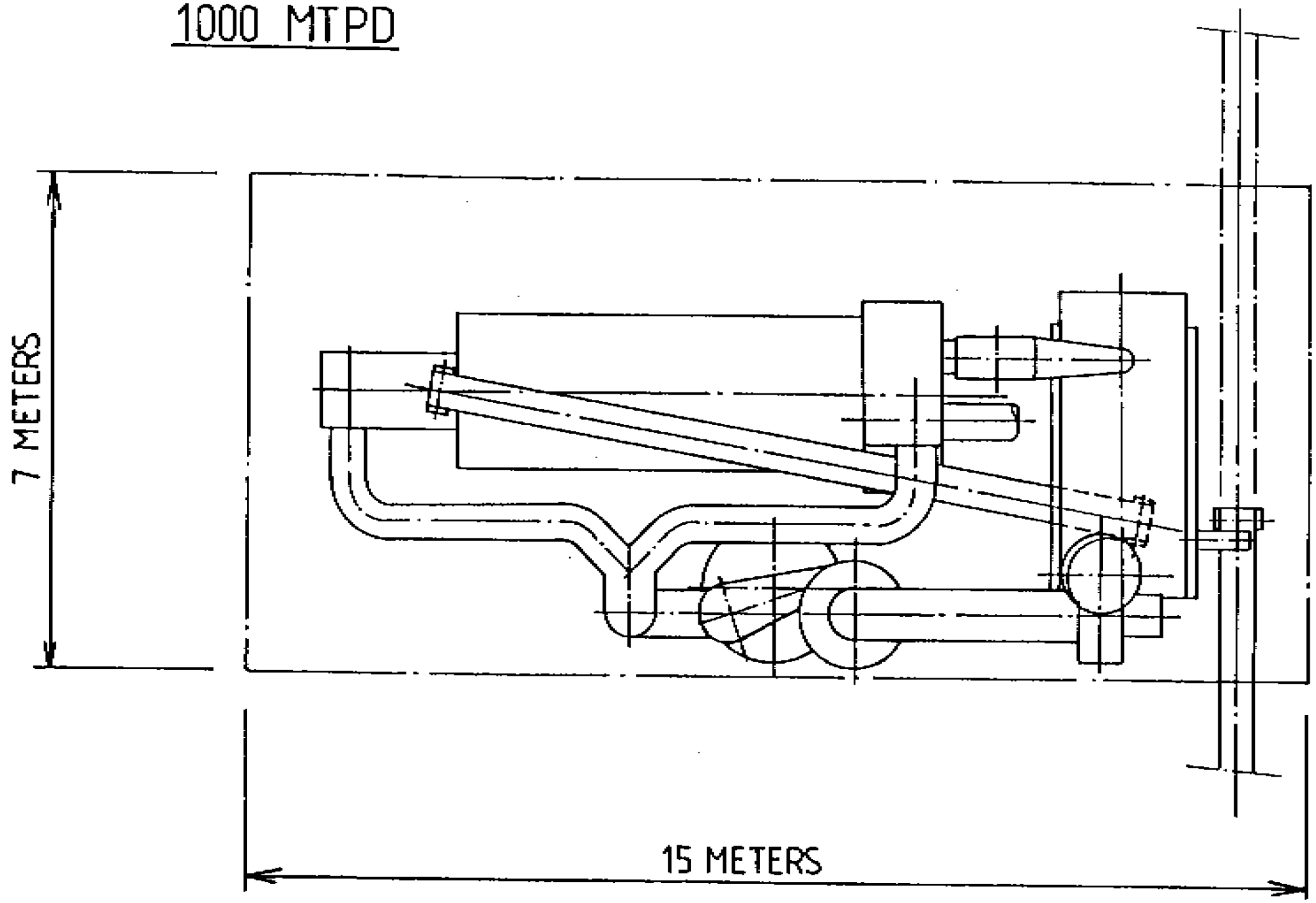
SULPHUR FLUID DRUM GRANULATION

FIGURE 3



SULPHUR GRANULATION WITH FLUID DRUM GRANULATOR 1000 MTPD

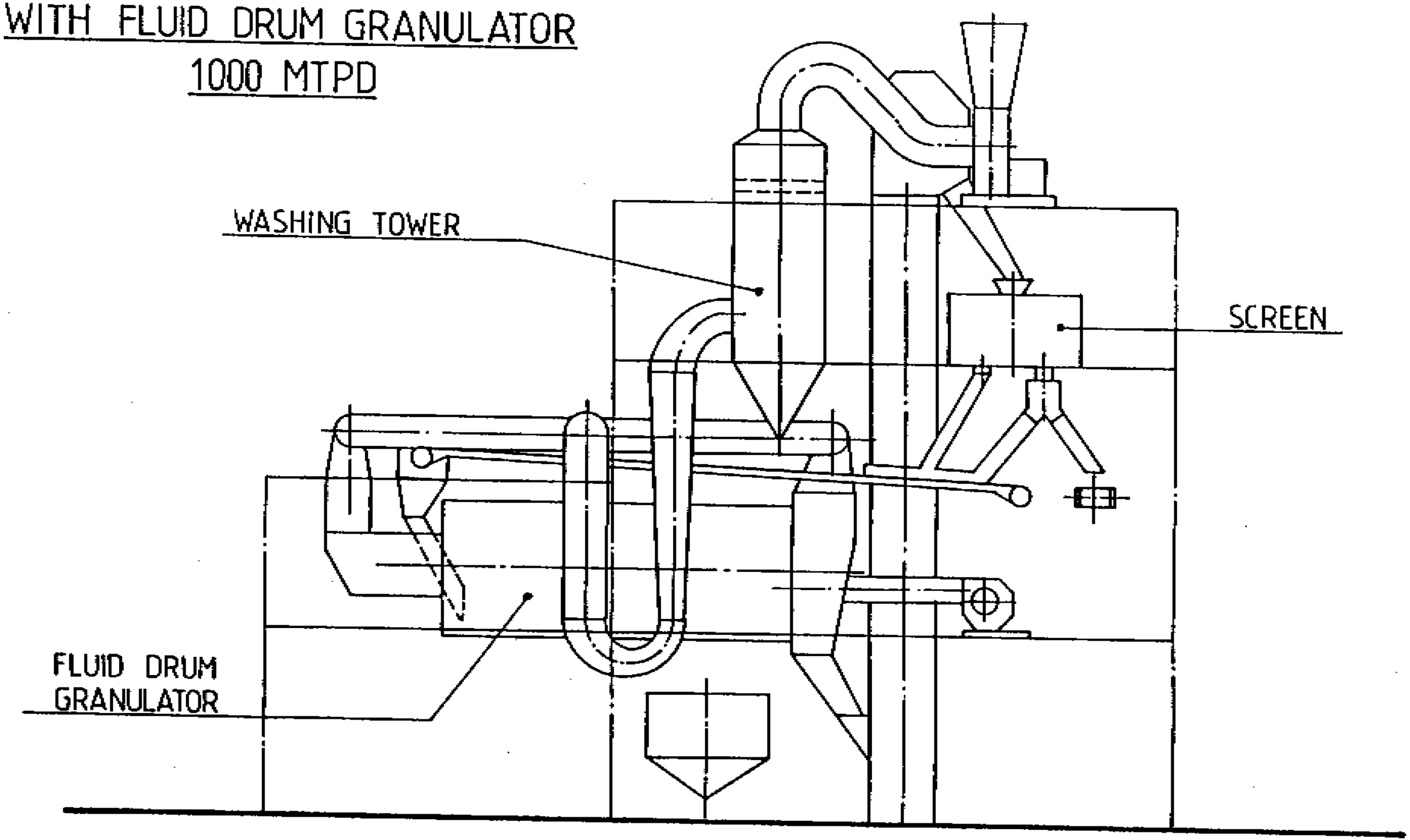
FIGURE 4



PLAN VIEW

SULPHUR GRANULATION WITH FLUID DRUM GRANULATOR 1000 MTPD

FIGURE 5



LONGITUDINAL ELEVATION