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NOTES ON THE FLASH ROASTER - PETERSEN TOWER SULPHURIC ACID PLANT AT ABERDEEN.

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INTRODUCTION.

In 1949 when Scottish Agricultural Industries were faced with provision of sulphuric acid plant to replace obsolete and worn out chamber plants, our first preference was for a sulphur-burning contact plant. At that time, however, our Government would not permit us to use sulphur. As an alternative we chose the type of plant which we have erected. The raw material - pyrites - is burnt in a flash roaster and conversion is by a Petersen Tower system. At the time of the design and erection of the plant, choice of materials of construction was limited by availability and in several quite major issues we had to choose not what appeared to us to be the best possible solution but best compromise between availability and suitability.

DESCRIPTION OF PLANT

The acid plant consists of a pyrites-burning flash-roasting section, supplied and erected by Messrs. Simon-Carves Ltd., and a Petersen section designed by Dr. Petersen of Wiesbaden and erected by ourselves.

Flash-Roasting Section

The rated capacity of the flash roasting plant is 80 tons of pyrites per day. It may best be described in five main parts - the pyrites drying section, the pyrites-grinding section, the roasting section, the heat recovery and the gas cleaning section.

Pyrites is stored under cover and is fed from stock pile by means of a mechanical shovel to a skip hoist which, in turn, feeds a wet pyrites storage bunker. The pyrites is dried in an oil-fired rotary louvre dryer, from which it is conveyed to a dried pyrites storage bunker. This feeds an air-swept ball mill. The dried and ground pyrites is collected from the air stream by a cyclone delivering to the roaster feed bunker. The primary air for the roaster carries the pyrites from the pyrites feeder into the roaster through an injection head along with some excess primary air and recirculation gas. The SO_2 gas generated is first cooled by passing through a waste heat boiler and then cleaned by Buell cyclones and electrostatic precipitators before being passed into the Petersen section.

Petersen Section.

The Petersen section consists of six brick-built towers over which, with the exception of the RT, acid is circulated. These are as follows:-

(i) Denitration Tower (or DT) - ring-packed
(ii) Production Tower (or PT) - granite-packed
(iii) Stabilising Tower (or ST) - ring-packed
(iv) Regeneration Tower (or RT) - void
(v) Nitration Tower 1 (or NT1) - granite-packed
(vi) Nitration Tower 2 (or NT2) - granite-packed

The functions of these towers might briefly be described as follows:-

Tower	Function
Denitration Tower	Denitration of acid; production of product acid; liberation of oxides of nitrogen in the gaseous phase; production of nitre free acid for use on NT2.
Production Tower	Absorption and oxidation of SO_2; liberation of nitrogen oxides
Stabilising Tower	Absorption and oxidation of remaining SO_2.
Regeneration Tower	A void tower which provides space and time for re-oxidation of the nitre gases from NO to a mixture of NO and NO_2.
Nitration Towers 1 and 2	These towers absorb nitre from the gaseous phase in acid recycled from PT and DT respectively.

The acid circulation system may be described as being in two rings. In the first acid circulates over the Denitration and second Nitration Towers, and in the second over the first Nitration and Production Towers. Acid ex NT2 is circulated over the Stabilising Tower. These circuits are not wholly separate and a certain mixing occurs between the flows. Acid leaving the first three towers is passed through extensive glass coolers before being returned to circulation.

Both sections of the plant are fully instrumented, the instruments being grouped in a control-room in which remote control devices permit of making many of the process control adjustments.

Production

Before the erection of the flash roaster was completed, some 6,500 tons 100% H_2SO_4 was made in the Petersen section from sulphur burned in a Glens Falls burner. A total of some 44,000 tons 100% H_2SO_4 has been made from pyrites since the start-up of the flash roaster in September 1953 to the end of April 1955. It was necessary to shut the plant down for a time to reconstruct the glass acid coolers, which in the initial stages of operation gave a great deal of trouble. Apart from this shut-down, the plant has been operational for 82% of the time since production started on sulphur at the end of January 1953.

Flash Roaster Section

Pyrites Feed

A wet pyrites storage hopper holding around 100 tons is provided. It is circular in section, nine feet in diameter and has a cylindrical section nineteen feet high with a cone shaped bottom ten feet deep. Wear has been observed only at the bottom of the cone where movement of the pyrites has abraded the plates but elsewhere a static layer of material has apparently afforded protection.

A six foot diameter table feeder extracts the wet pyrites to the dryer. It has suffered considerable wear. The original table wore out in six months and the renewed table has been fitted with wearing plates for ease of maintenance. The depth of the pyrites layer is regulated by adjustable skirts on the hopper discharge. Pyrites is ploughed from the feeder table to the dryer by a rubber covered plough. This particular arrangement of hopper storage of wet pyrites and the feeder has proved satisfactory in operation.

Data on Pyrites Used

Type	% Sulphur (dry)	% Moisture	Screen Analysis (B.S. Screens)				
			- 60	- 100	- 200	- 300	- 300
Boliden	50.0	2.8	52-35%	13-21%	25-26%	21-25%	
Rio Tinto	46.9-47.9	6.5	30-35	14-16	12-14	6-9	25-35
Reocin	49.5	4.6	0.5-5	3-12	17-26	6-13	54-60
Waite Amulet	48.6	4.5	0.4-12	2.5	16-23	14-16	55-65

Dryer, Burner and Combustion Chamber

Crude oil is burned through a rotamiser burner in a brick-lined combustion chamber 2' in diameter and 7'6" long. A radio-viser flame failure device is fitted and temperature is controlled by a Kent recorder and controller. A damper is provided on the inlet side of the fan for controlling air conditions in the furnace, and primary air is also controlled on the rotamiser burner assembly.

The dryer is of the rotary louvre type 4'6" diameter x 20' long. On the whole it has run satisfactorily. Our troubles were those generally to be expected when working with extremely fine and abrasive pyrites. Wear on the rollers, runner band and driving pinion of the dryer was heavy. These had frequently to be readjusted to compensate for roller wear. The dryer and seals, which were rather light in construction and did not have sufficient overlap, showed a tendency to leak.

The main difficulty in dryer operation was that of cleansing the gases exhausted to atmosphere. These carried over extremely finely divided pyrites dust. A cyclone was only partially successful in removing dust and a mild steel water scrubbing tower has been installed. The scrubber is three feet in diameter and twelve feet high and is packed for seven feet with 3" Nori rings. It is washed with 500/600 g.p.h. of water which, after passing through a settling tank, is allowed to run to waste. This tower proved successful as a scrubber but tended to choke and by causing back pressure and reducing the dryer fan throughput limited the dryer capacity. An expansion dust collecting chamber was later introduced between the cyclone and scrubbing tower. This reduced choking and erosion of the tower.

The dried pyrites is carried to the dry pyrites storage bunker by two screw conveyors and a rubber belt bucket elevator, all of which have given satisfactory service and have shown little signs of wear.

Dryer Performance Data

The unit is handling around 3¾ - 4½ tons per hour, the material being dried from an initial moisture content of from 4% to 7% down to a final moisture content of 0.05% to 0.25%. Gas is admitted to the dryer at around 300°C. and leaves at around 100°C.

Ball Mill

Pyrites is ground in a Hardinge air-swept ball mill. Feed to the mill is by a constant weight feeder and the air circulation through the mill carries the ground material through a separator to a cyclone. This feeds through a rotary air lock to the roaster feed storage bunker. The air from the cyclone is returned to the ball mill by a fan and the circuit is vented to the flash roaster primary air-line. At present the entire feed of pyrites has to pass through the mill and when handling the finer flotation concentrates this has led to the production of an excessive proportion of superfines. It is proposed to instal screening plant to bypass the fines in the dried pyrites direct to the roaster feed storage bunker and to grind only the coarser material. This should lead to considerable economy in grinding.

Considerable wear has been found in the ball mill driving gear on the inlet end bearing and also on the inlet and scroll liners. The heaviest wear occurred on the air-swept ducting, separator and cyclone, all of which, due to erosion, have required extensive renewal and repair. Similar conditions have obtained on the flash roaster primary air ducting.

Operation of the mill was reasonably trouble-free. When the ducting deteriorated, however, there was difficulty in maintaining a sufficient air-flow to control the size of the product satisfactorily. The air from the circuit is purged at around ½" w.g. to the roaster primary air circuit and it supplies around 50% of this primary air. It is interesting to note that sufficient pyrites dust is carried over to the roaster to warrant a reduction in feed when the ball mill is working. Arrangements have been made for an SO_2 purge to permit of operating on an oxygen deficient gas in case of fire in the circuit, but so far it has not been necessary to use this as only one minor fire occurred shortly after the roaster start-up. This was in the purge pipe to primary air line and was easily extinguished.

Performance Data

Realistic figures on pyrites grinding output are difficult to arrive at as production was restricted for some time by the poor condition of the air circuit ducting. The highest figures recorded over a seven day period were as follows:-

Rio Tinto	4.6 tons per hour
Reocin	5.6 " " "
Waite Amulet	4.8 " " "

Ball load about 2.5 tons of 2" balls
Ball usage approximately 0.9 lb./ton pyrites ground

	B.S. Screens				
	+ 60	+ 100	+ 200	+ 300	- 300
Boliden	0.3	1.0	19.3	14.8	64.6
Rio Tinto	0.4	2.9	11.7	13.1	71.9
Reocin	0.8	1.0	10.4	11.4	76.6
Waite Amulet	0.5	1.0	8.0	7.8	82.7

Roaster

The flash roaster consists of a firebrick-lined steel chamber further enclosed by a light steel shell forming a cooling jacket. Its shape may be described as a conical bottomed vertical cylinder of around 25' diameter surmounted by a dome shaped top. The overall height is around forty feet. The pyrites, along with primary air for combustion is introduced through an injection head in the top of the chamber. Preheated primary air is drawn from the roaster cooling jacket by the primary air fan and is split into two streams. Pyrites is fed into one stream and just sufficient air to carry the pyrites to the injection head is allowed to flow in this branch. This is in order to reduce wear on the ducting by the pyrites laden air by restricting its velocity to a minimum. Excess air to the quantity required for combustion is carried in the other branch and the two streams mix just before entering the injection head. Recirculated gas from the boiler is also added at the injection head and the whole stream of gas and pyrites passes into the roaster with a swirling motion imparted by a charger fitted with ten turbine-like blades. Secondary air, to supply further oxygen, is drawn into the bottom of the roaster chamber by the reduced pressure in the system.

Compressed air soot blowers, operated intermittently at 100 p.s.i., prevent cinder accumulation in the bottom cone.

It is worthy of note that the roaster is built in the open. The domed top is covered by light steel plating which, while not being entirely waterproof, provides adequate protection against the weather.

The flash roaster has required comparatively little maintenance. The injection head required partial replacement and the charger was renewed three times in one year. The reason for one replacement of the charger was that when the primary air line became eroded at the bend above the injection head, the air stream was deflected and partly burned pyrites built up in the head. Distribution of pyrites into the roaster was thereby affected and some may have reached the channel. The throat and mixing tube of the injector have also been replaced. The main body of the casing seems in good condition. Replacement of the injection head is straightforward and can be carried out in four to six hours.

When starting up, the flash roaster is heated by an oil burner consuming approximately 45 gallons/hour. The rate of heating is as under:-

5°C./hour up to 300°C.
10°C./hour 300 - 600°C.
20°C./hour 600 - 900°C.

Thereafter, temperature is maintained at 900°C. for at least 24 hours.

If the plant has been shut down for some time, the heating period must be sufficiently long to dry out all ducting, etc. otherwise a rapid build-up of dust on the sticky iron oxide-sulphate skin, which remains after cleaning, occurs.

One unpleasant incident occurred during heating up when unvaporised oil droplets from the burner passed to the cinder hopper. As these were evaporated by the rising temperature on further firing, the vapour flashed and caused an explosion. Little damage was done but the oil burner is now continuously checked by the operators for efficient combustion, and a smaller burner is used when firing at lower temperatures.

No sintering has been observed in the roaster and very little trouble with cinder slides has occurred. This may be due to our having chosen operating temperatures below the maximum recommended and to our use of a comparatively low burner gas strength.

Serious cinder build-up in the channel between roaster and boiler has occurred on a number of occasions and had to be removed by raking. Once build-up has started, soot blowing of the channel may not prove effective as the air from the lances tends to tunnel through the obstruction without removing it. On one occasion the build-up is thought to have been due to a fault in the injection head. On other occasions it may have been due to a high percentage of superfines in the pyrites feed, or have been initiated by a period of low load and consequent low gas velocity. It is difficult to relate build-up to the use of any particular type of pyrites.

Roaster Performance Data

On present full feed the indicated air and gas flows are:-

Primary air	2,200 c.f.m.
Secondary air	2,400 c.f.m.
Recirculation gas	3,200 c.f.m.

This is somewhat below theoretical requirements but the cause of the difference could be either instrument calibration errors or false air from the cinder hopper etc. bypassing the secondary air flowmeter orifice plate.

These figures are for the current maximum plant throughput which is at present limited by partial choking of the Production Tower to 105 tons monohydrate per day. The roaster works without difficulty at 65% load but it is thought that the lower gas velocities lead to heavier dust deposition in the roaster channel and draught pipes.

Roaster temperatures are as follows:-

Top	940° C.
Middle	957° C.
Bottom	978° C.
Channel	932° C.

Temperature control, for a given feed of pyrites, is effected by varying the air flows. At present with our maximum gas flow restricted we control by the pyrites feed rate.

Sulphur in cinders

Figures obtained using different types of pyrites are given below:-

Type of Pyrites	Total Sulphur Content of	
	Cinders ex Roaster % S (dry basis)	Cinders ex whole unit % S (dry basis)
Boliden	1.28 - 1.64	0.63 - 0.94
Rio Tinto	0.71 - 1.21	0.60 - 1.04
Reocin	0.84 - 1.43	1.19 - 1.32
Waite Amulet	0.53 - 0.73	0.46 - 1.01

These figures are representative of the whole year's running and include the periods when the ball mill air circuit was in poor condition, with resultant variation in pyrites particle size.

Gas Strength

The SO_2 content of the roaster gas is recorded at the point of entrance to the Denitration Tower. The values obtained at this point do not significantly differ from those given by tests made at the roaster channel. In other words, there is little dilution.

Normal working percentages of SO_2 have varied from around 6.5 - 7.5% SO_2 to 9.5% SO_2.

Waste Heat Boiler and Turbo-Generator

The SO_2 gas leaves the roaster tangentially through a channel at the top, passes downwards through the boiler, and thence to the gas cleaning section. The boiler is of the water tube type with five tube sections, the top four originally being generating tubes and the bottom one a super-heater. During December 1954, this was altered so that the fourth bank down became the super-heater and the bottom bank became a generating bank. This change was made to increase the degree of super-heat of the steam fed to the turbine. The boiler operates at 325 p.s.i. and on full load is rated at 10,500 lbs. per hour steam at approximately 700°F. super-heat. This is equivalent to a steam production of 1.2 to 1.4 tons steam per ton of pyrites burnt.

Boiler operation has been quite satisfactory after the settling down period. On inspection, after fourteen months' service, some sagging was found in the top bank of tubes and it was necessary to replace the centre two which had sagged around 1¼". The water side of the boilers required little cleaning, but the gas side was very difficult to clean. The boiler is essentially a gas cooler, and the water tubes are quite closely spaced at 5½" centres. The tubes are a nominal 2" inside diameter and are protected by cast iron sleeves, around ¾" thick, against erosion and corrosion. With such close spacing, intensive soot blowing and rodding could give no assurance of cleanliness and it was decided to wash down with cold water. In January 1955, the plant was shut down and cold water was immediately injected over the boiler tubes. This appeared to be completely effective in cleaning scale.

Power Generation

It was decided to use the 325 p.s.i. steam from the boiler for power generation and for this purpose a Turbo-Generator was chosen. The reason for this choice in preference to a Turbo-Alternator, was that it is a simpler, cheaper and more robust unit. It has the disadvantage that it may be used only when a mains supply is available for exciting purposes but it has the advantage that it may be switched on without careful phase synchronisation with the mains supply. The plant used is a Turbine running at 7,500 r.p.m. driving, through reduction gearing, an induction generator rated at 310 k.w. The turbine operates against a back pressure of 30 p.s.i. and pass-out steam is fed into the works low pressure system. As an alternative the steam can be bypassed through a reducing valve enabling the works low pressure system to be fed from the acid plant when the turbine is shut down. In addition 100 p.s.i. steam can be bled off from the turbine for feeding to the works high pressure system and this is also controlled by a reducing valve. The induction generator runs in parallel with the mains supply with a reverse current relay to trip the load in the event of steam failure. The rated output is 310 k.w. with a steam supply of 9,750 lb./hour at 325 p.s.i. and 650°F.

Energy Distribution

An energy distribution diagram has been prepared showing the generation, utilisation and loss of energy in the system. This may be of general interest and is included as Appendix 1.

Buell Cyclones

Gases leaving the waste heat boiler pass through three Buell cyclones arranged in parallel. Cyclone operation has been satisfactory and trouble free. Internal examination after one year's running showed little sign of wear. Cinders are discharged through flap valves into the cinder conveyor.

Electrostatic Precipitators

Recirculation gas to the roaster is taken from the exhaust manifold of the Buell cyclones, and the main gas flow passes through the booster fan to the electrostatic precipitators which consist of two sections in parallel, each section having an inlet and an outlet zone. The inlet zones of the two sections are normally controlled together as are also the outlet zones, but if necessary individual zones can be isolated. The positive electrodes in this set are 1" x ⅛" strips in place of the usual wires.

The precipitators have operated well but require opening for cleaning after around three months' use in addition to the routine hand and automatic rapping of the various zones during normal operation. The points of heaviest deposition are the inlet louvres and a certain amount of "sausage" formation has been found on the electrode strips. If left for periods longer than three months, fine cinder starts to pass to the Denitration Tower and while not apparent from the colour of the acid, it causes a build-up on the blades of the wet gas fan.

The electrical gear has given no trouble and no electrodes have had to be renewed.

Cinder Conveyors and Mixer

Cinder is collected from the following points:-

From the roaster cone
" " boiler hopper
" " Buell cyclones
" " electrostatic precipitators

The cinder from the roaster hopper is fed by screw conveyor, and from the other collectors through flap valves, to the main cinder conveyors which are of the push pull type, actuated by hydraulic cylinder. The cinder is then elevated by a chain and bucket conveyor to a hopper which feeds a cinder mixer where water is added to the cinders to reduce dust nuisance before they are discharged by rubber belt conveyor to the cinder store. Very heavy wear occurs in the cinder mixer and the rubber lining had to be completely renewed after one year's service. Replacement of paddles is at the rate of one per day and the cost of each paddle is over £1.

Fans and Ducting

A considerable amount of vibration has been experienced from impellers out of balance due to local erosion and as gas circulation is vital to operation of the plant, we would recommend that especial care be taken with the grouting and holding down of fans in any similar installation.

A very successful feature has been the fitting of hydraulically operated dampers to the main fan ductings, controlled from the central control room by a hand pump. Gas flows can thus be altered from the control room and the effect throughout the whole system simultaneously observed on the instruments.

PETERSEN SECTION

The logical start to this section would be a description of the towers but, as the performance of the whole plant has been affected by and even controlled by the behaviour of the glass acid coolers, it is proposed first to give some account of our experience with these units.

Acid Coolers (See Appendix Figures 1 and 2)

It might be appropriate to mention the reasons for our choice of glass coolers and this is best done by quoting from the paper given by my colleague, Mr. J.P.A. Macdonald, on construction details of this plant, at the Technical Meetings of the Association held at Cambridge in 1953.

"(a) Provision has to be made for cooling 375 g.p.m. (100 cu.m./hour) of nitrous acid at 90°C. and 45 g.p.m. (12 cu.m./hour) of denitrated acid at 120°C. This is normally done in cast iron coolers for the former and lead coolers for the latter. At the time of construction vertical cast iron could not be obtained in this country and lead was expensive. Moreover neither material used in their respective coolers give entirely satisfactory performance and so there seemed to be a case for examining other fields.

"(b) After consideration of all the likely alternatives, the decision was made in favour of Pyrex heat resisting glass (boron silicate) for all coolers. The apparent advantages of this material were that it would be immune from chemical attack from nitrous or denitrated acid, and that it would not be affected externally by slight leakage from above. Moreover, it would not suffer from the fall off in heat transfer as is caused by external rust films on cast iron.

"(c) The joints were made in fluon (polytetrafluorethylene) sheathed asbestos with Bakelite flanges and stainless steel bolts. The cooler units were of 3 inch (76 mm.) internal diameter formed in 'U' bends with ten foot (3.1 m.) legs. The coolers are in banks of four pipes wide by seventeen pipes high with external water cooling. The number of banks required is ten and the total length of glass is 2.7 miles (4.3 km.)".

The original form of construction for these coolers is shown in Fig. (1). As was mentioned in Mr. Macdonald's paper, considerable breakage of the glass piping was experienced due to the stresses set up in the bends by any slight inaccuracies of installation. These breakages caused such serious interruption to plant operation that it was decided to re-design and reconstruct the coolers as shown in Fig. (2). Shorter straight lengths of pipe were used joined at the end with short bends. Stress, and hence breakage, has been largely eliminated but the problem of leakage from the joints grew with the greatly increased number of joints. Different jointing materials have been tried and at present the most satisfactory are as follows:-

Joint - Butyl rubber protected with fluon
Insert - Either asbestos protected with alkathene, or bakelite
Flanges- Special bakelite

Reasonably satisfactory service is given by these joints but leaks do still occur and acid dropping on the joints below causes further deterioration.

Cooling water is circulated over the acid coolers, collected in a lead lined pond and pumped over cooling towers to a high level from which it is redistributed over the coolers. (The pond was originally asphalt lined, but this proved useless). The pH of this water is regulated by ammonia dosage controlled by pH meter. If, however, severe contamination takes place, the water is allowed to flow to waste. Water usage thus varies with the incidence of leaks but is currently around 1,000 gallons per ton H_2SO_4.

Though we expect to solve our present jointing difficulties we would not, if we were building a new plant at the present time, use glass for a similar duty.

Towers

The Petersen towers are built on a 3 foot thick reinforced concrete deck, 17 feet above ground level. The general structure of the towers consists of a 9" wall of acid resisting Nori brick lined externally with alkathene and covered by a 4½" protective layer of the same brick. The towers are girdled by steel bands round vertical timber stringers. The tops are domed and lead saucers are provided at the base. Acid circulation tanks and pumps are sited below the concrete deck.

The brick and alkathene construction of towers has been satisfactory on the Stabilising Tower, Regeneration Tower, Nitration Towers (1) and (2), i.e. the "Cold" towers. The outsides of these towers are dry and in good condition. On the Denitration and Production Towers, i.e. the "Hot" towers, the outer skin of brickwork is cracked in places, and when the towers are cold (during a shut-down) there is some leakage of acid. This disappears on heating up with consequent tightening by expansion of cracks in the brickwork.

In considering this deterioration of the brickwork, it must be borne in mind that during the actual life of 2½ years, shutdowns have necessitated the cooling and reheating of the towers more than 100 times. This is more often than one would expect during a normal life of 25 years.

There has been no sign of deterioration in the brick domed tower tops.

Space Data

The tower space provided is 0.96 cu. ft./lb.S/24 hours (this is equivalent to a ratio of 51 kgm. H_2SO_4/cu.m./day.) Of this 0.96 cu.f.t. some 68.5% is packed and the remainder void.

Tower Packing

The ring packing used in the Denitration and Stabilising Towers remains in good condition. The two Nitration Towers and the Production Tower were packed with granite from a local source - Corennie - after exhaustive laboratory tests. While this has proved satisfactory in the Nitration Towers, there are signs of some deterioration in the Production Tower. The pressure drop across this tower has risen from the original 1" - 2" to 10" w.g.

Fan and Ducting

The wet gas fan of multi-blade centrifugal type is in stainless steel and has given satisfactory service. Slight build-up has occurred when the electrostatic precipitators were in need of cleaning. Fine dust surprisingly passed through to the fan and this build-up on the impeller has caused sufficient drop in efficiency to require shut-down for cleaning. Ducting between the towers in mild steel, brick lined, has been satisfactory but the 15 lb. lead duct between the DT and PT has been heavily attacked at the bottom end in the acid condensation area. It is worth noting that the Ammonia Oxidation Plant discharges into this duct.

Acid Circulation

(a) Pumps

The pumps are vertical spindle glandless type fitted with impellers either suitable for 12½ H.P. or for 20 H.P. giving pumping rates of 75 g.p.m. or 150 g.p.m. Casings of the pumps are in cast iron. The impellers and impeller housings are of R.55 alloy. Originally the impeller housings were of cast nickel iron, but this was not found satisfactory. The pumps gave satisfactory service, the main trouble being due to wear between the impeller housing and the bottom casing where the cast iron corroded. This was corrected by machining off the worn face and fitting a packing ring in R.55 alloy. There appears to be no serious fall off in performance after approximately two years' running and, in relation to the number of hours run by each pump, maintenance has not been expensive.

(b) Reaction Water

The pumps draw acid from the circulation tanks at a level normally 4 - 5 feet below the impellers. The P.V.C. suction pipe is in the centre of a rigid P.V.C. rose piece which has the form of a cylinder one foot in diameter and four feet high. Acid enters the rose piece through 5,000 holes ⅛" (3.2 mm.) in diameter (maximum flow 160 g.p.m.) The reaction water by which the process is controlled is added inside the rose pieces of the pumps feeding the Denitration, Production and Stabilising Towers, so that acid and water mix properly before entering the pumping line.

(c) Acid Lines

The acid pumping lines are all in cast iron with the exception of a lead bend on the suction side of each pump, this latter being to facilitate installation and removal of the pump. There is no evidence of any deterioration in these pumping lines.

(d) Valves and Cocks

The standard valve used in the acid circulation system is a simple cast iron cock. This is liable to seize up if left long without being moved. An experimental sulphate resisting cock has been tried and found more satisfactory. Gate valves have been used on the acid coolers and in one position in the acid circulation, and have proved quite satisfactory. They have the advantage that finer adjustment of the feed rate is possible than by cocks.

(e) Douches

The acid distribution over the towers has been found to be critical at full production, particularly on the Production Tower. Redesigned douches were installed on the PT last summer in conjunction with constant head overflow arrangements, to ensure a constant distribution pattern. The douches are made in cast nickel iron and this has generally been satisfactory although recently it has been discovered that the hole size has in some cases increased appreciably, e.g. from 0.196" up to 0.209". Choking of the douches is fairly frequent. Changing of the douches on the Production, Stabilising and Nitration Towers is easily effected in approximately fifteen minutes without unduly interfering with the running of the plant but in the case of Denitration Tower, it is necessary to shut the plant down to change the douche. (It should be possible to modify the DT douche arrangement and have a spare douche mounted on a turret ready to swing into position with practically no interruption to the acid feed).

<u>Plant Operation</u>

In operation, the tail gas from the plant is uniformly low in SO_2 or SO_3 content, the tail gas total acidities ranging normally from 0.1 - 0.3 grains SO_3 per cubic foot (0.23/0.68 gm./cu.m.). Over a series of over four hundred tests, the maximum, minimum and average acidities found were 0.49, 0.03 and 0.25 grains SO3 per cubic foot respectively (1.12, 0.07 and 0.57 gm./cu.m.). Over the same period sulphur efficiencies have been from 97% to 99% on the Petersen section and from 94% to 96% overall. Since the first objective of a sulphuric acid manufacturer, high sulphur efficiency, is so readily obtained on a Petersen plant, the main objective of the plant control personnel becomes the control of nitre recovery and efficiency. Under steady operating conditions we have found nitre usage to be around 2% $NaNO_3$ on sulphur burnt (0.88 kgm. HNO_3/tonne H_2SO_4). Numerous shut-downs have caused expensive nitre losses, however, as on the average the equivalent of 2,000 lb. $NaNO_3$ was lost at each shut-down.

To maintain nitre consumption at a low level, it is necessary to ensure that:-

(1) tower feeds are at the correct rates and are being properly distributed by the douches.
(2) nitre distribution in the gas phase of the system is correct.
(3) acid temperatures are correct.

The first condition is essential for proper contact between gas and acid. Normally the feeds on all the towers except the Denitration Tower are kept constant and this feed is adjusted with the make and with the nitre distribution.

The nitre distribution in the gas phase can be calculated from the nitrosities of the acids entering and leaving the towers. The efficiency of nitre recovery in the last two towers depends on the oxidation ratio of the oxides of nitrogen which in turn is dependent both on the nitre concentration and on the oxygen concentration in the Regeneration Tower.

The acid temperature affects the rates both of SO_2 absorption and of H_2SO_4 formation and also, more importantly, the condition of nitrous oxides as the gas temperature varies with the acid temperature in the last three towers. The inlet temperature of acid to the towers should be as follows:-

NT 2 - below 35°C.
Other towers - 40/50°C.

In practice control of plant under steady operating conditions is best achieved by a study of the nitre distribution in the gas phase of the towers. Ideal conditions for normal operation at full capacity have been calculated as shown in the graph attached (Appendix 3). It will be seen that in the Regeneration Tower a concentration equivalent to 1% NO by volume is essential.

Nitre distribution in the gas phase of the towers is controlled by altering the circulating acid strength by the addition of reaction water, and by the feed rate of nitrous acid to the Denitration Tower. It is also affected by -

SO_2 concentration in the system
PT and ST acid feed rates
Acid temperatures
O_2 concentration

These latter items are, however, kept as constant as circumstances permit.

One further factor affecting the nitre distribution is the amount of SO_2 being fed to the plant, i.e. the rate of operation.

Under low feed rates quite different conditions are obtained as the example shows (See Appendix 4). The plant was working at around 65% load at the time.

In this case most of the SO_2 is oxidised in the Denitration Tower and the reaction is completed in the first half of the Production Tower.

Oxides of nitrogen are therefore absorbed in the Production and Stabilising Towers and the concentration in the Regeneration Tower is consequently as low as 0.15%. The concentration remains constant over the first Nitration Tower and the remaining oxides of nitrogen are absorbed by the nitre free acid circulated over the second Nitration Tower. During the three day period that the plant operated under these conditions, nitre loss was negligible and the ammonia oxidation plant was shut down.

Operational Difficulties

The interruptions occasioned by glass cooler failure with consequent disturbance of plant conditions have caused many deviations from the desired conditions for efficient nitre control. Typical causes are as follows:-

(1) Variable conditions when starting up;
(2) Reduced acid feeds on DT, PT (and hence on NT1) and ST occasioned by cooler trouble;
(3) Uneven acid distribution in the towers caused by deterioration and choking of douches;
(4) Interruptions to tower feed caused by exchange of douches;
(5) Low nitre concentration in circulating acid.

(1) Starting up Conditions

Plant stoppages of more than a few hours' duration are usually accompanied by loss of nitre. This loss occurs during the restarting of the plant and is thought to be due mainly to low acid temperatures and to insufficient oxidation from NO to NO_2. Conditions change quite rapidly during the starting up period, however, and it is difficult to tell when conditions of under or over-oxidation obtain until they are sufficiently steady to permit of calculating the nitre distribution.

(2) Reduced Tower Feeds due to Cooler Troubles

Owing to cooler troubles, the acid feeds to DT, PT (and hence NT1) and ST had, at different times, to be reduced.

Low acid feeds make it impossible to maintain proper nitre distribution in the towers. Due to insufficient contact between gas and acid, SO_2 comes through into NT1 where some of the NO2 present is reduced. The results of too low a nitre concentration in the DT, PT and ST and the presence of SO_2 in NT1 are under-oxidation of oxides of nitrogen, poor absorption and consequent nitre losses.

(3) Faulty Acid Distribution from Douches

Uneven acid distribution on the tower surface has been caused both by choking of douches and by erosion of the douche holes, particularly in the first three towers. Channelling occurs and SO_2 passes to NT1. The gauges used to indicate acid pressure in the douches have been somewhat unreliable and have occasionally failed to indicate a change of as much as 50% in pressure and thus did not show changing conditions sufficiently quickly.

(4) Exchanging Douches

The plant has to be stopped when the DT douche is being changed since, as has been mentioned earlier, we have no provision for switching over directly to a spare douche. Changing the DT douche is at present often delayed until some other repairs can be done at the same time. Normal time for the change is 15 minutes, the flash roaster having to be off for approximately 1 hour.

All other douches can be changed while the plant is running but some slight disturbance to nitre distribution follows.

(5) Low Nitre Concentration in Circulating Acid

Too low a nitre concentration in the circulating acid or too high a strength of this acid causes low nitre concentration in the gas phase. Conditions in the towers automatically change and maximum nitre concentration drops. Provided the SO_2 feed is not too high (70% of full load say) stable conditions are attained after some time and then nitre losses are not excessive.

On full feed, however, HNO_3 must be added immediately to restore proper nitre concentration and to avoid heavy losses.

Normally, if high acid strength is responsible, the addition of more reaction water will liberate nitre to the gas phase but this is limited by the condition that the acid strengths must be maintained sufficiently high to avoid attack on the cast iron pumping lines.

Normal Running Conditions

	Volume at N.T.P. M^3.p.m.	Volume at N.T.P. c.f.m.	Temperature	Volume at Op. Temp. c.f.m.
Primary and Secondary air from flash roaster	198	7000	10°C	7,260
Recirculation gas	52	1825	360°C	4,230
Gas in roaster channel	250	8825	920°C	38,500
Inlet to DT	192	6785	330°C	15,000
Exit Gas	165	5820	40°C	6,630

Pressure across Plant:

	Normal Conditions Ins.W.G.
Boiler Outlet	- 2.5
Recirculation fan in	- 7.5
Recirculation fan out	- 2.0
Booster fan in	- 5.5
Booster fan out / Dust Precipitator in	- 0.4
Dust Precipitator out / DT inlet	- 1.0
DT Outlet / PT Inlet	- 2.5

PT Outlet	- 4.3
ST Outlet	- 6.0
RT Outlet	- 6.2
NT1 Outlet)	- 7.8
Wet Gas Fan in)	
Wet Gas Fan out)	
NT2 Inlet)	- 1.2

Acid Temperatures:

DT Outlet	125 - 135°C.	40°C. ex Cooler
PT Outlet	65 - 90°C.	40-50°C. ex Cooler
ST Outlet	45 - 50°C.	35°C. ex Cooler

NT1 Outlet) 1°C. from Inlet temp. under good
NT2 Outlet) conditions (no SO_2 coming through)

Acid Strengths and Nitrosities (ex Tower):

	DT	PT	ST	NT1	NT2
Acid Strengths	138-140	136-140	135-139	136-140	138-140°Tw.
Nitrosities	-	110	140	120	15-20 lbs. $NaNO_3$/ ton acid

Nitre Distribution in Gas Phase

It might be interesting to consider the nitre distribution under actual operating conditions and the series of diagrams attached shows the reaction to various conditions (in each case the dotted line shows normal distribution).

Diagrams A to E represent the case history of a disturbance and show the stages in bringing the plant from poor operating conditions with loss of nitre back to satisfactory operating conditions.

(a) Losing Nitre: All SO_2 is oxidised in PT, and ST acid absorbs nitre from the gas. Nitre concentration in RT is low and therefore oxidation is slow. NT1 is absorbing no more nitre than is NT2.

(b) By reducing DT feed from 54 to 50 g.p.m. the rate of oxidation of SO_2 has been slowed down. SO_2 in ST reduces NO_2 and less N_2O_3 is absorbed in ST than before. The concentration of nitrous oxides in RT is high. Good oxidation in RT and good absorption in NT1, the latter being four times the absorption of NT2. Under ideal conditions NT1 should absorb 90% and NT2 10% of the nitrogen oxides.

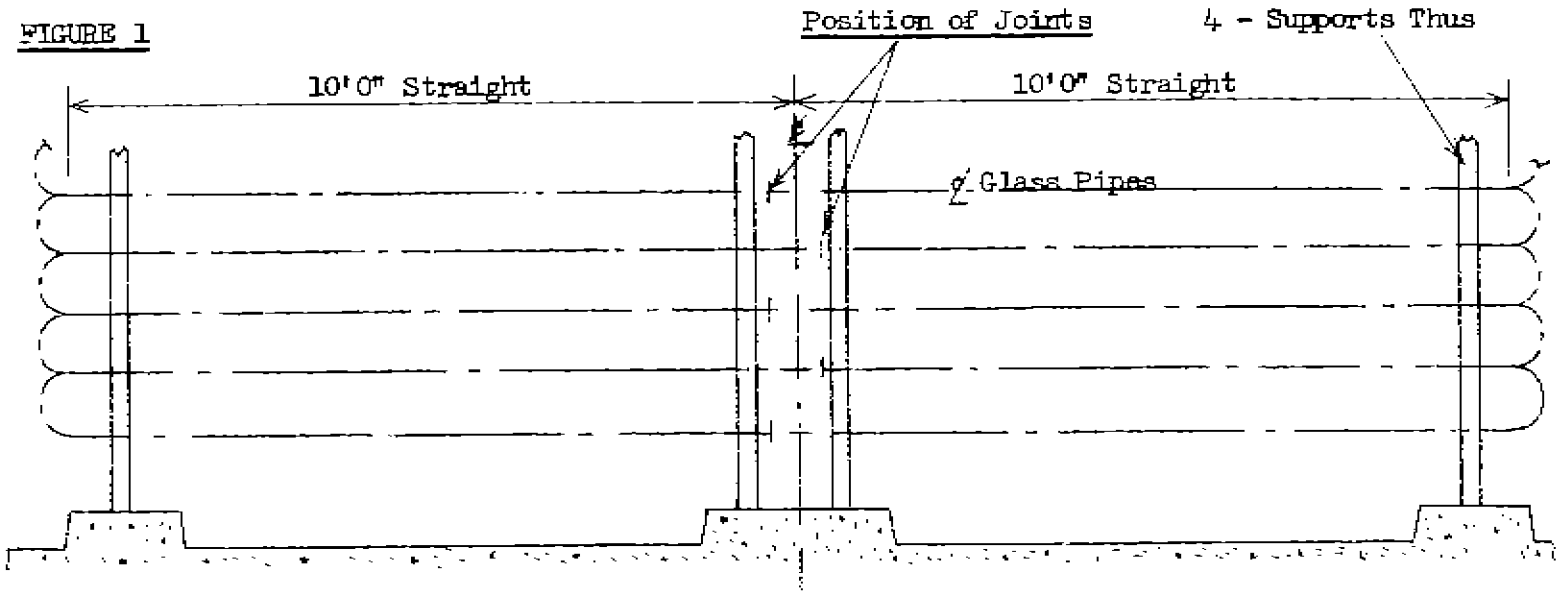
(c) The acid circulation system is such that nitre distribution is very stable. This applies also with bad conditions. With the same flows, concentration and temperatures as four hours earlier, nitre concentration in RT drops and absorption in NT1 grows worse. This could have been remedied by reducing acid strengths in PT and ST, thus expelling more nitre from the acid.

(d) Troubles with the reaction water pipe made the reduction of acid strengths impossible and the nitre concentration in RT has become still less.

(e) The reaction water required was now available. Nitre concentrations in all towers were rising and NT1 starts to absorb more nitre. Good conditions have almost been reached.

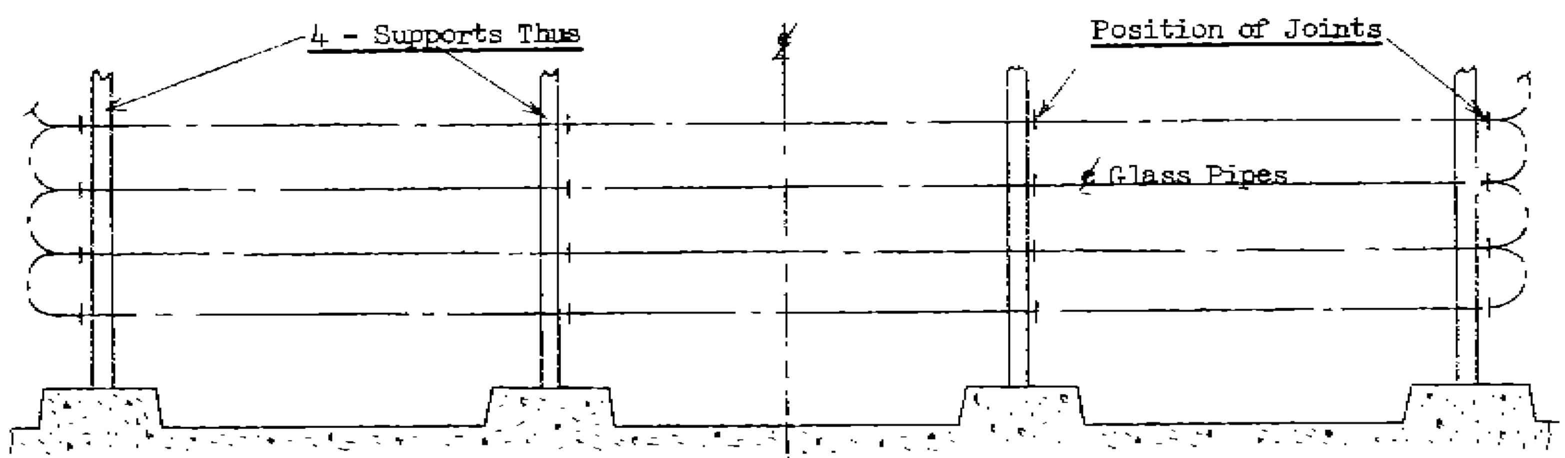
(f) This diagram shows good conditions when the plant is working at low throughput.

FIGURE 1



FIRST ARRANGEMENT

FIGURE 2



SECOND ARRANGEMENT

ARRANGEMENT OF ACID COOLERS

(g) Conditions here are unusual and were occasioned by cooler trouble resulting in an enforced reduction in the DT feed. The interesting point is the extremely good nitre absorption in NT1.

Another cause of nitre loss which might be mentioned could be described as SO_2 flushes coupled with very low O_2 content, i.e. large sudden increases in the quantity of SO_2 being fed to the plant. These occurred on several occasions when burning sulphur, and once when burning pyrites. As a measure of the rate of nitre loss which can result, a dark red nitrous emission lasting for six minutes was estimated at containing between 5 and 10% of nitre. Over a period of two hours, the nitre loss was on this occasion estimated as 1000 lb. $NaNO_3$.

Ammonia Oxidation Plant

Losses of nitre in the system are normally compensated for by nitrous oxide from a conventional ammonia oxidation plant. To cope with abnormal conditions, additions of nitric acid have been made to the circulating acid.

INSTRUMENTATION

It was decided to have fairly comprehensive instrumentation on both flash roaster and Petersen Sections of the plant and we have endeavoured to group as many as possible of the instruments in a control-room serving the plant as a whole. It has been possible to put all the flash roaster instruments on a panel 9' x 13' and the acid making instruments on a panel 9' x 9'.

Flash Roaster Section

The instrumentation of this section has been largely satisfactory. What trouble we have had has been due mainly to choking of pipes with dust and to the use of insufficient protection.

Flame Failure Device and Dryer Temperature Control on Pyrites Dryer Furnace

These function well apart from slight sticking of the oil control valve.

Air and Gas Flow Recorders

Primary air, secondary air and recirculation gas are measured. These instruments operate reliably but there is some doubt as to their calibration as it is thought that the readings tend to be low.

Temperature Recorder and Indicator

Temperature measure is by electrical resistance thermometer. Temperatures are recorded at six points and indicated at twelve. The points are distributed over the roaster section, the principal ones being the three roaster points, gas channel, boiler, recirculation gas, precipitator inlets and outlets and the steam leaving the superheater. The only trouble with this installation has been breakage of the ceramic sheaths at some points and it is proposed to replace these by stainless steel sheaths.

SO_2 Recorder

Blockage of the sampling lines with dust has been the only trouble so far experienced.

Gas Pressure Indicators

Blockage of pipes is again the difficulty and frequent cleaning is necessary.

Steam Flow Recorders

These measure the rate of steam flow from the boiler and from the turbine and after some modification have given reliable service.

Petersen Section

Flow Meters

Each main pumping line to the tower sprays is provided with an orifice type flowmeter. The differential pressure from the orifice plate is passed to a pneumatic transmitter which transfers the differential into air pressure. A local dial gauge is provided over each pump to assist adjustment of the control valve and the measurement is repeated on an edgewise instrument mounted on the control panel. This type of instrument has not been altogether satisfactory.

Pressure Gauges

As distribution of acid from a spray nozzle in the tower is a function of both flow and pressure, it is necessary to know both to permit of proper control and we measure pressure immediately behind the spray nozzle by the air reaction principle. The air pressure required to maintain a small discharge into the acid line is shown on an edgewise indicator on the panel arranged as a pair with the corresponding flow gauge. Like the flowmeter this instrument has not been too reliable though it is helpful in guiding us in the operation of the plant.

Depth Gauges

As the pumping cisterns and storage tanks are enclosed, we have to provide a ready means of showing the depth of acid in each. This is done by air reaction gauges with bells at the bottom of each cistern. This arrangement has worked very well.

Thermometers

Electric resistance thermometers are provided to indicate the temperature at important points in both acid and gas streams. The instruments are of the direct reading type with selector switches. These have worked well.

Reaction Water

We have three taper tube water flow gauges with control valves to each for the regulation of reaction water to the acid circuits feeding the denitration, production and stabilising towers.

Colour Tube

Despite fairly elaborate instrumentation, control of the plant is still dependent to some extent on the colour of the exit gases. These are only visible during daylight hours so we have a colour tube mounted in the control room. The sample of gas is taken from the outlet of the wet gas fan and returned to the inlet. The tube is constructed in welded polythene with windows at each end and with a light bulb. This colour tube has been most helpful to us.

CONCLUSIONS

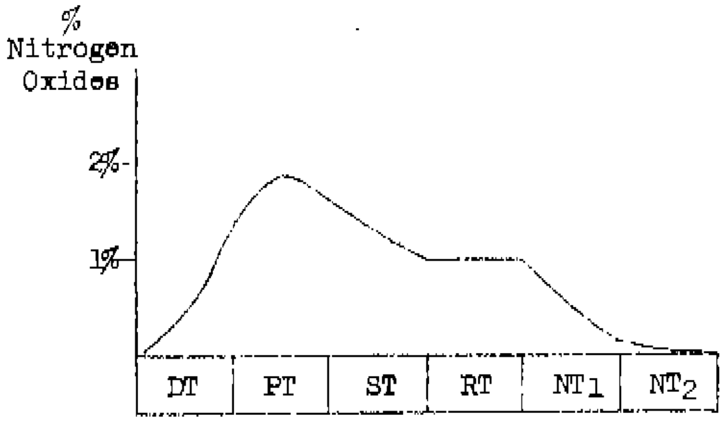
Our experience with our acid plant has been mixed. On the flash roaster side of the plant, the steam raising, power generation and gas dedusting, once initial teething troubles were overcome, have been smooth in operation. However, the pyrites grinding and separating equipment requires costly maintenance and is expensive in power consumption and we intend to try to modify this section of the plant by the incorporation of a screen to remove oversize and so obviate much of the use of the ball mill circuit. We may also decide to improve the cinder wetting arrangements. Otherwise, we are well pleased with this section of the plant.

The Petersen section of the plant has operated well when cooler troubles have not caused disturbances. Unfortunately cooler troubles have been so serious as to disturb the working of the plant to such an extent that we think it fair to say that no type of sulphuric acid conversion system other than a tower system would have been capable of operation. Under the circumstances we have been well pleased with the Petersen section of the plant.

ACKNOWLEDGMENTS

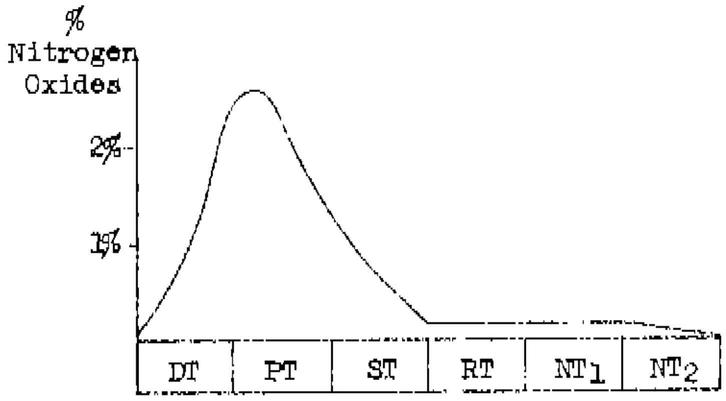
In conclusion I wish to express my thanks to the Directors of Scottish Agricultural Industries for permission to present this paper and to those of my colleagues who have assisted in its preparation.

III



% Nitrogen Oxides
2%
1%
DT
PT
ST
RT
NT_1
NT_2

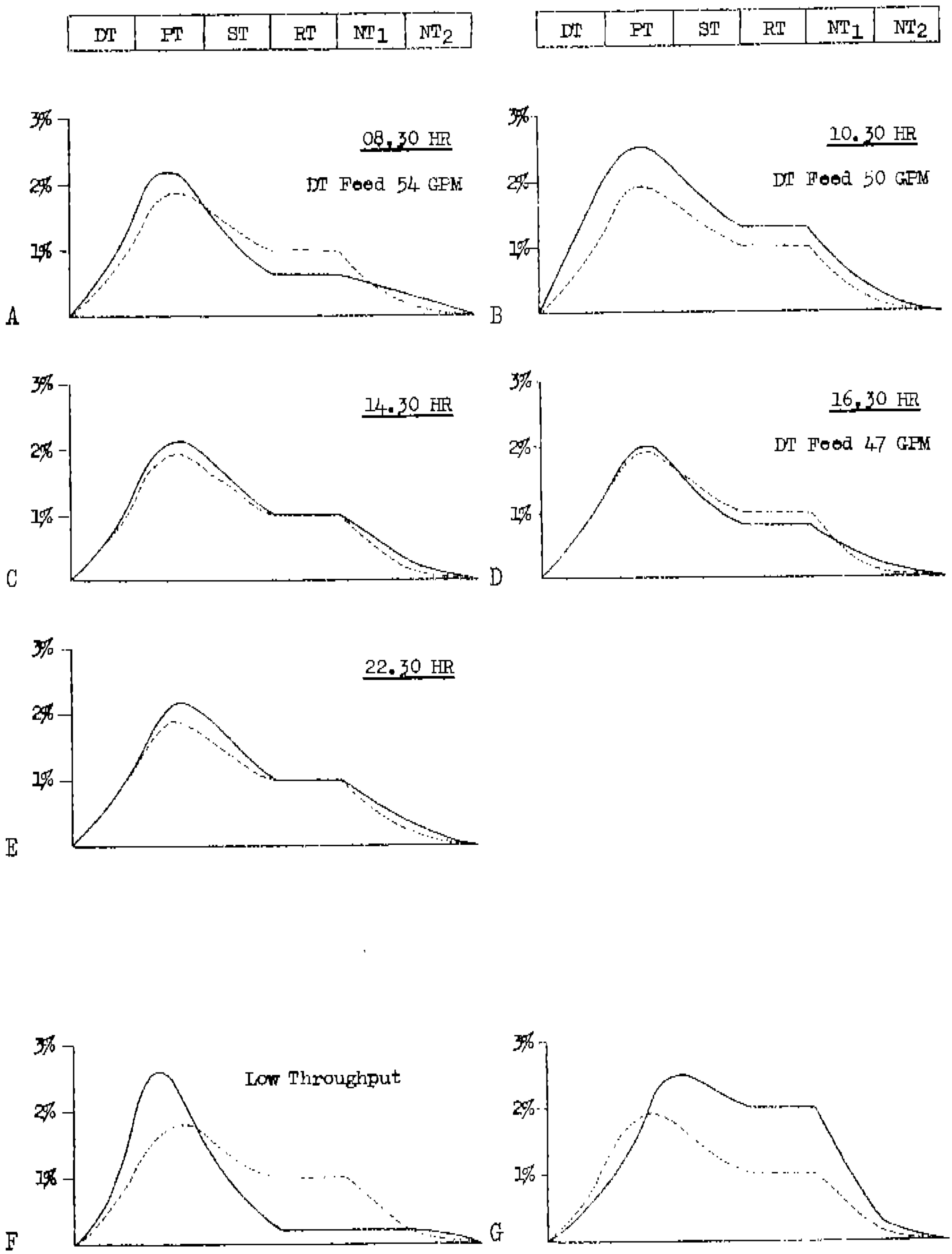
IV



% Nitrogen Oxides
2%
1%
DT
PT
ST
RT
NT_1
NT_2

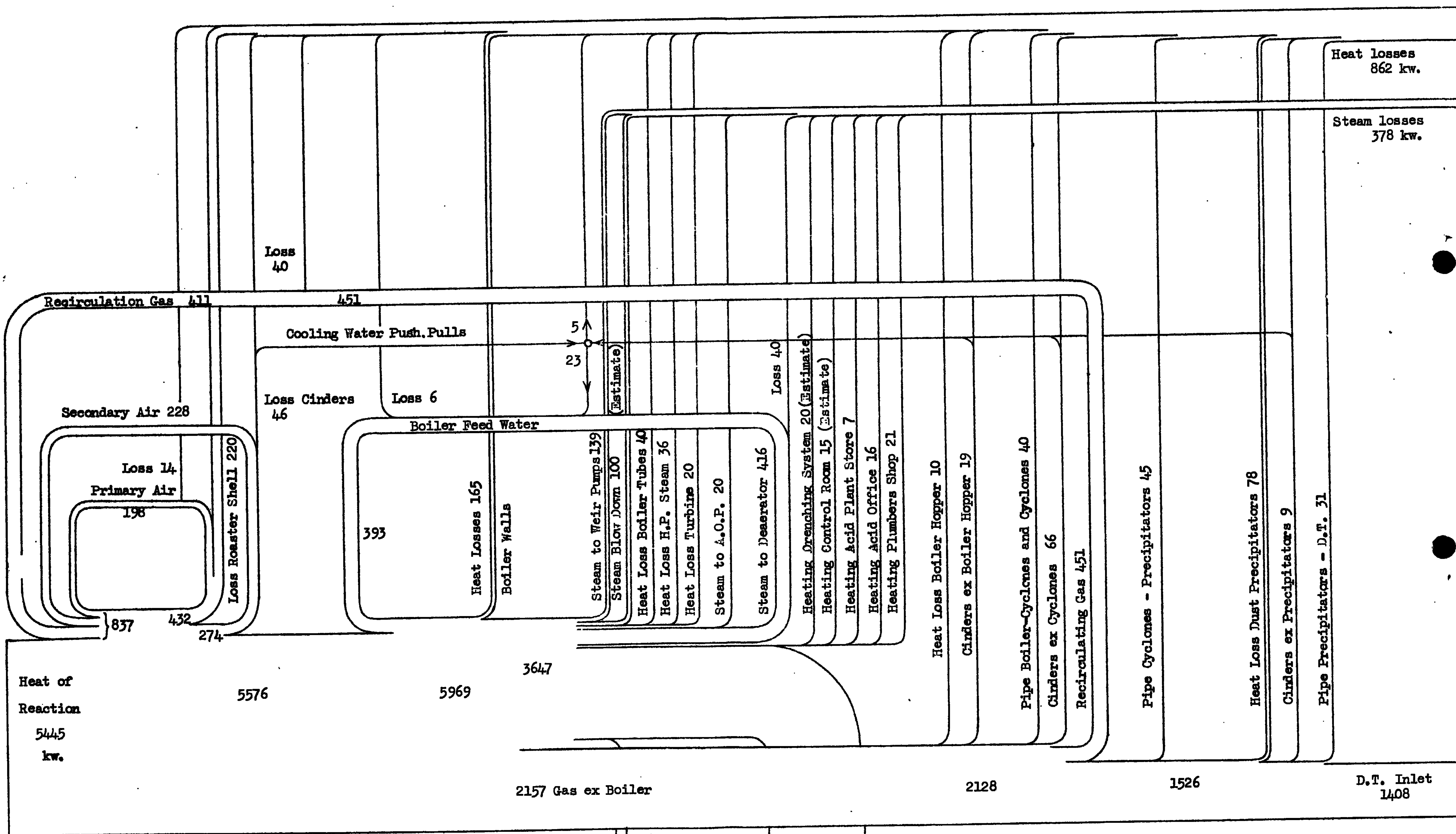
V

NITRE DISTRIBUTION IN TOWERS



ABERDEEN ACID PLANT

ENERGY DISTRIBUTION IN FLASH ROASTER SECTION



FULL LOAD: 80 = Pyrites 48% S/Day

ZERO ENERGY LEVEL = $10^{\circ}C$

FIGURES REPRESENT KILOWATTS

1 kw = 3415 Btu/hr.