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MEASUREMENT OF FLUORINE EVOLUTION DURING SUPERPHOSPHATE MANUFACTURE

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During the decomposition of phosphate rock with sulphuric acid to form superphosphate, part of the fluorine contained in the phosphate rock is given off as silicon tetrafluoride. To avoid damage through atmospheric pollution, the SiF_4 must be washed out of the reaction gases as completely as possible. In order to control the efficiency of the wash towers and to study the conditions which influence the evolution of fluorine under operation conditions, we required a method of measuring the fluorine content of the gaseous effluents.

A. Analytical methods for determining the amount of fluorine given off

1) Indirect Method

The evolution of fluorine may be measured indirectly from the F : P_2O_5 ratio in the rock and in the superphosphate. This measures the total fluorine less during the whole of the reaction process, that is, it includes fluorine loss during grinding, transport and storage of the superphosphate.

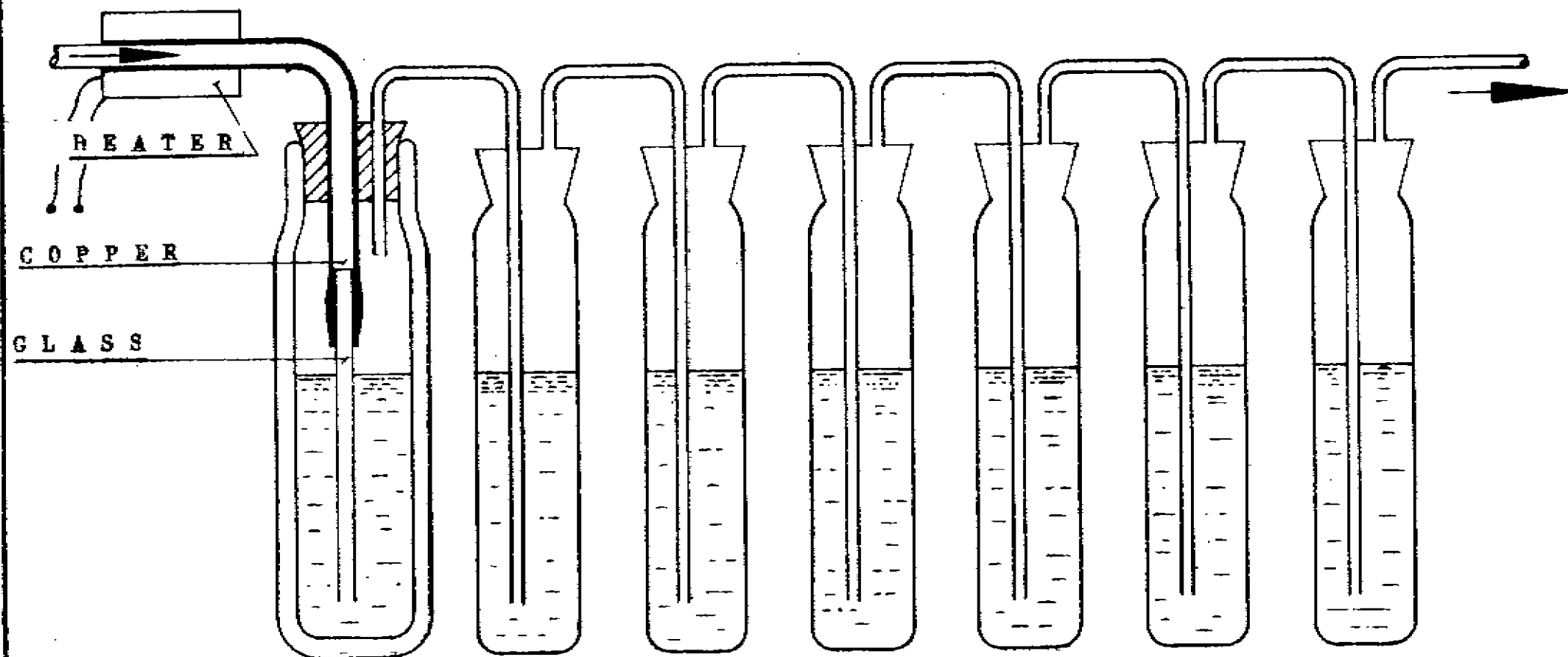
This indirect method may be used to obtain data on the evolution of fluorine from various types of rock and under various reaction conditions, as the papers by Delomenie (1) and Tessier (2) have shown.

2) Direct Method

Another way of determining the amount of fluorine evolved is to analyse the actual fluorine compounds formed.

A suitable laboratory method was found by A.L. Whynes and T.P. Dee (3). The gases given off during experimental decomposition in the laboratory are carried away by a flow of air, sodium hydroxide reacts with carbon dioxide and fluorine compounds, also with part of the water, while magnesium perchlorate reacts with the rest of the water.

APPARATUS FOR TESTING WASTE GASES FROM SUPERPHOSPHATE SLURRY



CONTENT : SULPHURIC ACID
 80°C 20°C

ALCOHOL

SODIUM HYDROXIDE

ABSORPTION OF H_2O

SiF_4

CO_2

Fluorine and carbon dioxide are determined in the sodium hydroxide solution.

3) Gas Analysis under Operating Conditions

During investigation of the evolution of fluorine at the reaction stage in the plant, a need was felt for a rapid, easy and accurate method of analysing the reaction gases. We also thought it important - particularly in view of ever-increasing industrialization and the consequent tightening of the laws governing industry - to know the composition of the gases released to atmosphere. There was, of course, only one method worth considering, one which would analyse a part of the gas flow; thus, a method was developed permitting quantitative, and separate, determination of water, silicon tetrafluoride and carbon dioxide.

B. Description of the New Analytical Method

1) General Principles

- a) The gas sample is taken from the gas stream in a tube; if the tube is warmed, condensation of water can be avoided.
- b) The water vapour is absorbed in hot concentrated sulphuric acid.
- c) SiF_4 is taken up in methyl or ethyl alcohol and titrated with an alkali.
- d) Carbon dioxide is combined with sodium hydroxide in an aqueous solution and titrated with an acid.
- e) The sample of gas is drawn off by means of an aspirator and the remaining volume of gas is measured.

2) Description of Analytical Method

The apparatus is represented in the attached diagram in simplified form.

a) Removal of gas sample

A copper tube is used for removal of the gas sample, extending 20 cm into the gas stream. It is heated by means of a 200 watt heating mantle, as sold commercially for electric soldering irons. Using a tension regulator over a resistance or a control transformer, the temperature is maintained at about 20°C above the temperature of the gases. An electric bandage heater may be used instead of a mantle. Heating of the sample tube prevents the condensation of water vapour and hence premature formation of fluosilicic acid and silicic acid. Preliminary trials showed that when the tube was not heated, about 90% of the silicon tetrafluoride was retained. Copper was chosen for its high conductivity.

b) Water determination

Absorption of the water vapour was by means of concentrated sulphuric acid heated to 80°C . A commercial $\frac{1}{2}$ -litre Thermos flask was used as the wash bottle. It was closed with a rubber stopper through which the copper tube and a glass tube were passed to take the gas flow. One end of a glass tube is

immersed in the sulphuric acid while the other end is inserted into the copper tube whose end is about 5cm above the surface of the sulphuric acid. The seal is made by covering the join with a sleeve.

Instead of the Thermos flask, a wash bottle in Jena glass can be used and the temperature maintained at 80° by a bandage heater.

After passing through the warmed sulphuric acid, the gas is passed through a normal gas wash bottle with concentrated sulphuric acid at room temperature. The gas is dehydrated by passing through the hot sulphuric acid in the first bottle and all the fluorine is converted back to SiF₄.

The water content is found by weighing before and after the determination. The amount found is of the order of 2-4 g H₂O per determination.

c) Absorption and determination of the SiF₄

The silicon tetrafluoride is absorbed in alcohol. Methyl or ethyl alcohol take up to 33 or 36% by weight respectively of SiF₄ without separating out silicic acid. Two normal gas wash bottles are used. The amount of SiF₄ absorbed is found by titrating with alkali.

d) Absorption and determination of CO₂

Carbon dioxide is absorbed in 20% caustic solution. Three intensive gas wash bottles with internal coils are connected in series, and have been found satisfactory. The amount of CO₂ is determined by titrating with acid. No attempts have so far been made to determine the CO₂ content gravimetrically. It would first be necessary to absorb the alcohol vapour carried over from the earlier bottles.

e) Aspirator

The aspirator consists of a Winchester bottle full of water with water flowing out through a siphon. The volume of water flowing out from the time the gas flow begins until the time the sampling tube is removed from the effluent stream corresponds to the volume of gas remaining. 5-8 litres of residual gas was found to be a suitable amount. Finally, 2 litres of air are drawn through to cleanse the gas wash bottles. Any error arising from the water and CO₂ content of the air may be ignored.

The determination takes from 20 to 50 minutes and the accuracy of the analyses is adequate.

3) Calculation of the Gas Analyses

The volume of residual gas is calculated to standard volume with due regard to barometric pressure, slight vacuum in the aspirator and water vapour content of residual gas. The water vapour content of the residual gas is governed by the temperature of the residual gas and the slight vacuum in the aspirator. From the standard volume and the calculated normal volumes for water, silicon tetrafluoride and carbon dioxide, the percentage volumes for each component are worked out.

C. Examples of application : Measurements taken during Operation of the Moritz-Standaert Don

This method of analysis was used to study the gas given off during operation of the continuous Moritz-Standaert den. In the following circumstances, the readings should show to what extent the evolution, or recovery, as the case may be, of fluorine depends on the various factors:

- 1) 77% pebble phosphate reacted with 76% sulphuric acid.
- 2) 68% Morocco phosphate with 76% sulphuric acid, various production rates.
- 3) Morocco/pebble phosphate mixture with 76% sulphuric acid, reduced ventilation.
- 4) Pebble/Morocco mixture with 78% sulphuric acid.
- 5) Constantine phosphate with 78% sulphuric acid.
- 6) Composition of effluent after passing through absorption tower.

Tables I to 6 in the Appendix give data on the reaction conditions, the results of the analyses and the deductions made from them.

Following data are included:-

- type of phosphate rock, F and CO₂ content
- concentration of sulphuric acid
- production rate in tons of superphosphate/hour
- time from start-up of plant when sample was taken
- analysis of gas by volume: % air (by volume)
- % water vapour (" ")
- % CO₂ (" ")
- % SiF₄ (" ")
- fluorine evolved according to gas analysis, %
- fluorine evolved according to gas analysis, kg F/ton rock
- air temperature, °C
- effluent temperature, °C
- dew-point temperature of H₂O vapour, °C
- No. of m³ effluent/ton rock
- No. of m³ air/ton rock
- No. of m³ air/hour
- kg H₂O/ton rock
- fluorine evolved, determined indirectly, %
- fluorine evolved, determined indirectly, kg F/ton rock
- recovery of fluorine in absorption tower

The recognisable influence of the various factors is discussed in the following description of each reaction period.

1) 77% Pebble Phosphate, Table I

77% pebble phosphate, 3.8% F and 3.1% CO₂, was reacted with 76% sulphuric acid. The table gives the results of 5 runs. The SiF₄ content of the effluent was only 0.4-0.7% vol., and that of CO₂, 2.8-4.2%.

From the F : CO₂ ratio in the effluent and in the rock, we calculated the amount of fluorine driven off and from this calculated the fluorine evolution. When the plant is newly started up, fluorine evolution is significantly lower. This may be attributed to the fact that the plant has not yet warmed up.

The amount of air drawn through the plant is significantly more at the beginning than later, because the effluent is still cold. In addition, the reaction area of the den which is empty at first offers less resistance to the passage of air. Moreover, until the plant is warmed through less water is vaporized so that more air can be drawn in instead; at the start the amount of water vaporized or entrained is only 70% of the average amount.

The gas analyses give an average of 24.2% for fluorine evolution, the indirect analysis 25.6%. The agreement can be regarded as satisfactory when it is considered that the gas analysis reflects conditions at an instant in time, while indirect determination from the samples of rock gives an average of the total.

The amount of fluorine collected in the absorption tower was 90% of that evolved. This means that 10% was lost in the effluent.

2) Morocco Rock 68%, Table 2

68% Morocco rock, 4.12% F and 5.8% CO₂, was reacted with 76% sulphuric acid. The SiF₄ content of the effluent sometimes rose to 1%. Fluorine evolution at 30.6% was significantly higher than with pebble phosphate.

The ambient temperature was 14° lower than in the previous run, but the temperatures of the gaseous effluent were practically the same for both runs. This shows that Morocco rock reacts more readily.

In a second run, production was reduced from 21 to 14 tons/hour. This increased the amount of air drawn through the system per ton of rock by 61%.

There was an increase of 4% in fluorine evolution, to 31.7%. The amount of water vaporized per ton of rock rose by 38%. The increase in fluorine and water evolution may be explained by the longer retention time of the reaction mixture in the mixer and by increased entrainment in the relatively greater volume of air.

The indirect method gave 29.6% F removal. Recovery in the absorption tower was only 89%.

3) Influence of the Volume of Air used to ventilate the Den, Table 3

A mixture of Morocco rock and pebble phosphate, 3.95% F

and 4.4% CO_2 , was reacted with 76% sulphuric acid. Capacity was 21 tons superphosphate per hour. Fluorine evolution was 31.5%, the same as for pure Morocco rock.

For a short time the air-stream drawn through the den was reduced by a throttle valve in the effluent duct to the extent that atmosphere around the plant became almost unbearably unpleasant because of the reaction gases. The CO_2 content of the effluent rose by 64% and the SiF_4 content by 29%. Fluorine evolution fell by 21% and the removed or entrained water by 35%. As the effluent was moving more slowly, it was cooled more in the effluent duct. Its moisture content rose to a point where its dew point temperature was above the effluent temperature. Condensation then occurred and this resulted in deposition of SiF_4 .

Throttling down of the effluent stream can easily occur in normal working as a result of blockages in the effluent ducts and in the absorption tower. This test showed that when this happens, not only is the surrounding area made unpleasant by the reaction gases, but that the amount of fluorine evolved or entrained is reduced and hence the recovery in the absorption tower is lower.

4) Morocco Rock/Pebble Phosphate Mixture, Table 4

A mixture of Morocco rock and pebble phosphate was reacted with 78% sulphuric acid. This SiF_4 concentration was between 0.6 and 0.8% on the two days the tests were run. Here, too, it was noticed that fluorine evolution was less shortly after start-up. Fluorine evolved, at 28.2%, was on average higher than with pure pebble phosphate, but somewhat lower than with pure Morocco rock at the same rate of tons/hour. The values determined by the two methods agree exceptionally well. Recovery in the absorption tower reached 95%.

5) 65% Constantine Phosphate, Table 5

Constantine rock, 3.70% F and 6.65 % CO_2 , relatively low in fluorine and high in carbonate, was reacted with 78% sulphuric acid. Fluorine evolved was 32.4%, a little higher than with Morocco rock at the same rate of production. The amount of fluorine evolved was, however, 8% less because of the lower fluorine content of the rock. There was good agreement with the values obtained by the indirect method. Recovery in the absorption tower was 97 %.

6) Analysis of Gaseous Effluent after passing through Absorption Tower, Table 6

Some observations on the gaseous effluent after the absorption tower are given below. They were carried out during reaction of the rocks mentioned under 1) and 4) above. In calculating fluorine losses, expressed as a percentage of the fluorine evolved, average values from Tables 1 and 4 were used.

TABLE 6

| Test Measurement | Table 1 | | Table 4 | | |
|--------------------------------------|---------|-------|---------|-------|-------|
| | 1 | 2 | 1 | 2 | 3 |
| air (% by volume) | 74.5 | 89.4 | 81.3 | 85.8 | 73.7 |
| H ₂ O " | 22.0 | 7.5 | 14.5 | 10.6 | 21.7 |
| CO ₂ " | 3.5 | 3.0 | 3.8 | 3.5 | 4.6 |
| SiF ₄ " | 0.022 | 0.050 | 0.009 | 0.034 | 0.023 |
| F loss in % | 3.9 | 11.0 | 2.2 | 8.1 | 5.7 |
| gr. F/no. m ³ of effluent | 0.8 | 1.7 | 0.3 | 1.1 | 0.8 |

D. Discussion of Tests and Conclusions

Table 7 summarizes the main results given in Tables 1 to 5. These tests have enabled us to give some of the answers as to the causes of more or less fluorine being evolved. We were able to confirm that fluorine evolution increases with a larger carbonate content of the phosphate rock. In the reaction with Morocco rock, fluorine evolution was 26% higher than with pebble phosphate; 36% more was evolved from Constantine rock. Since Morocco rock contains more fluorine than Constantine and pebble, fluorine recovery was at about the same level for Morocco and Constantine and 37% above pebble. Unfortunately, the test runs could not be carried out under completely similar conditions. For example, the amount of Constantine rock available was too small to give a full run over a long enough time. Air temperatures, which were influenced by weather conditions, fluctuated widely. The variation in acid concentration, from 76 to 78%, used in the test runs was too small to give clear results in view of the fluctuations in the other reaction conditions. According to tests carried out by A.L. Whynes and T.P. Doe (3), an increase in the sulphuric acid concentration from 76 to 78% induces a relative increase in fluorine evolution of 2.5%, equivalent to about 0.5% absolute. This could only have been appreciated under constant conditions.

A reduction in output to 65% brought a slight increase in fluorine evolution because the amount of air per unit of phosphate rock, and thus also of entrained water vapour, was increased. Throttling down the effluent stream was unfavourable as was to be expected. The water vapour was not removed quickly enough and the effluent became supersaturated with water vapour because of increased cooling. Part of the silicon tetrafluoride was deposited in the effluent ducts.

SUMMARY

A method is described for the study of gaseous effluents during operation of superphosphate plants.

The contents of air, water vapour, carbon dioxide and

silicon tetrafluoride were determined. Making use of these values, it is possible to calculate fluorine and water vapour evolution or entrainment, in relation to the fluorine and carbon dioxide content of the phosphate rock.

The application of the method of analysis is demonstrated on the basis of various series of tests.

REFERENCES

- 1) H. Delomenie ISMA Technical Meetings 1955
- 2) M. Tessier " 1957
- 3) A.L. Whynes & T.P. Dee " 1953

TABLE I

Reaction of 77% Pebble Phosphate

Rock : 77% pebble phosphate; F 3.8%; CO₂ 3.2%

Acid : 76% sulphuric

| | 1 | 2 | 3 | 4 | 5 | Average |
|---|------|------|------|------|------|---------|
| Tons/hour superphosphate | 21 | 21 | 21 | 21 | 21 | |
| Time from start-up in hrs | 1 | 2 | 3 | 5 | 7 | |
| Gas analysis | | | | | | |
| air % (by volume) | 78.9 | 62.3 | 64.1 | 65.9 | 57.0 | |
| water % " | 17.9 | 33.4 | 31.8 | 29.3 | 39.6 | |
| CO ₂ % " | 2.8 | 3.6 | 3.5 | 4.2 | 2.8 | |
| SiF ₄ % " | 0.34 | 0.69 | 0.58 | 0.66 | 0.57 | |
| % F removed according to gas analysis | 18.2 | 27.8 | 28.8 | 23.5 | 29.0 | 24.2 |
| F removed according to gas analysis, in kg/ton rock | | | | | | 9.2 |
| Air temperature °C | 23 | 25 | 26 | 26 | 25 | 25 |
| Effluent temperature °C | 82 | 79 | 74 | 84 | 79 | 80 |
| Dew-point temperature °C | 57 | 72 | 71 | 69 | 76 | 69 |
| No. of m ³ effluent/ton rock | 578 | 450 | 468 | 391 | 578 | 492 |
| No. of m ³ air/ton rock | 456 | 281 | 298 | 257 | 330 | 325 |
| No. of m ³ air/hour | 5620 | 3450 | 3660 | 3160 | 4060 | 4000 |
| kg H ₂ O/ton rock | 80 | 115 | 113 | 88 | 176 | 113 |
| Fluorine removal, determined indirectly (%) | | | | | | 25.6 |
| F removal, determined indirectly (kg F/ton rock) | | | | | | 9.7 |
| F yield in absorption tower (kg F/ton rock) | | | | | | 8.8 |

TABLE 3

Influence of Volume of AirRock : Morocco/pebble mixture; F 3.95%; CO₂ 4.40%

Acid : 76% sulphuric

| | 1 | 2 | Average 1-2 | 3*) |
|---|------|------|----------------|------|
| Tone/hr superphosphate | 21 | 21 | 21 | 21 |
| Gas analysis | | | | |
| air % (by volume) | 59.2 | 61.8 | | 55.0 |
| water % " | 35.5 | 33.1 | | 36.8 |
| CO ₂ % " | 4.6 | 4.4 | | 7.4 |
| SIF ₄ % " | 0.72 | 0.75 | | 0.95 |
| % F removed according to gas analysis | 30.1 | 33.0 | 31.5 | 25.0 |
| F removed according to gas analysis in kg/ton rock | | | 12.5 | 9.8 |
| Air temperature °C | 19 | 21 | 20 | 25 |
| Effluent temperature °C | 80 | 75 | 78 | 71 |
| Dew-point temperature °C | 73 | 72 | 73 | 74 |
| No. of m ³ effluent ton/rock | 484 | 505 | 495 | 300 |
| No. of m ³ air/ton rock | 287 | 312 | 300 | 156 |
| No. of m ³ air/hour | 3530 | 3950 | 3740 | 1920 |
| kg H ₂ O/ton rock | 135 | 132 | 134 | 87 |

*) Effluent flow throttled down.

TABLE 4

Rock : Morocco/Pebble mixture; F 4.05%; CO₂ 5.50%

Acid : 78% sulphuric

| | I | | | | | Aver. 1-4 | II | | | Aver. 1-3 |
|---|------|------|------|------|------|--------------|------|------|------|--------------|
| | 1 | 2 | 3 | 4 | 1 | | 2 | 3 | | |
| Tons/hour superphosphate | 21 | 21 | 21 | 21 | 21 | 21 | 21 | 21 | 21 | 21 |
| Time from start-up in hrs. | 1.5 | 2.5 | 4.5 | 6.5 | | | 1.5 | 3 | 4 | |
| Gas analysis air % (by volume) | 77.8 | 75.5 | 68.8 | 65.8 | | | 68.5 | 67.0 | 75.0 | |
| water % " | 17.1 | 19.8 | 25.8 | 26.9 | | | 25.2 | 26.2 | 18.5 | |
| CO ₂ % " | 4.6 | 4.2 | 4.9 | 6.5 | | | 5.7 | 5.7 | 5.8 | |
| SiF ₄ % " | 0.55 | 0.55 | 0.57 | 0.79 | | | 0.54 | 0.81 | 0.68 | |
| F removed according to gas analysis | 27.7 | 31.0 | 27.6 | 28.4 | 28.7 | 22.2 | 33.6 | 27.1 | 27.6 | |
| F removed according to gas analysis, in kg/ton rock | | | | | 11.6 | | | | | 11.2 |
| Air temperature °C | 7 | 8 | 10 | 10 | 9 | 8 | 10 | 11 | 10 | |
| Effluent temperature °C | 60 | 66 | 68 | 70 | 66 | 70 | 60 | 70 | 67 | |
| Dew-point temperature °C | 57 | 60 | 65 | 67 | 63 | 66 | 67 | 59 | 64 | |
| No. of m ³ effluent/ton rock | 605 | 662 | 567 | 427 | 565 | 488 | 488 | 480 | 488 | |
| No. of m ³ air/ton rock | 481 | 500 | 390 | 280 | 412 | 336 | 327 | 360 | 341 | |
| No. of m ³ air/hour | 5820 | 6180 | 4820 | 3460 | 5173 | 4150 | 4050 | 4460 | 4220 | |
| Kg H ₂ O/ton rock | 82 | 103 | 115 | 91 | 98 | 97 | 101 | 70 | 89 | |
| Fluorine removal determined indirectly (%) | | | | | 28.6 | | | | | 27.6 |
| F removal, determined indirectly (kg F/ton rock) | | | | | 11.6 | | | | | 11.2 |
| F yield in absorption tower (kg F/ton rock) | | | | | 10.9 | | | | | 10.9 |

TABLE 5

Rock : 65% Constantine; F 3.7%; CO₂ 6.85%
 Acid : 78% sulphuric

| | I | II | | Average |
|---|------|------|------|---------|
| | 1 | 1 | 2 | |
| Tons/hour superphosphate | 17 | 10 | 10 | |
| Time from start-up in hrs. | 6 | 2.5 | 6 | |
| Gas analysis | | | | |
| air % (by volume) | 85.0 | 83.3 | 81.9 | |
| water % " | 8.6 | 11.3 | 11.2 | |
| CO ₂ % " | 5.9 | 4.9 | 6.2 | |
| SiF ₄ % " | 0.59 | 0.48 | 0.64 | |
| % F removed according to gas analysis | 32.6 | 31.4 | 33.3 | 32.4 |
| F removed according to gas analysis | | | | 12.0 |
| Air temperature °C | 5 | 4 | 8 | 6 |
| Effluent temperature °C | 71 | 68 | 66 | 67 |
| Dew-point temperature °C | 43 | 48 | 48 | 46 |
| No. of m ³ effluent/ton rock | 593 | 708 | 558 | 629 |
| No. of m ³ air/ton rock | 508 | 590 | 456 | 516 |
| No. of m ³ air/hour | 5080 | 3490 | 2690 | 3740 |
| Kg H ₂ O/ton rock | 40 | 63 | 49 | 51 |
| Fluorine removal determined indirectly (%) | | | | 32.8 |
| F removal determined indirectly (kg F/ton rock) | | | | 12.1 |
| F yield in absorption tower (kg F/ton rock) | | | | 11.7 |

SUMMARY OF TABLE 1 - 5

| | 1 | 2 | | 3 | | 4 | | 5 |
|---|---------------|----------------|------|---------------------------------|------|---------------------------------|------|---------------|
| Phosphate Rock | Pebble 77% | Morocco 68% | | Morocco 68% Pebble 77% | | Morocco 68% Pebble 77% | | Conat. 65% |
| % F | 3.8 | 4.1 | | 3.9 | | 4.1 | | 3.7 |
| % CO ₂ | 3.2 | 5.8 | | 4.4 | | 5.5 | | 6.8 |
| Acid strength | 76 | 76 | | 76 | | 78 | | 78 |
| Tons/hr super | 21 | 21 | 14 | 21 | | 21 | | 10 |
| m ³ air/ton rock | 325 | 260 | 419 | 300 | 156* | 412 | 341 | 517 |
| m ³ air/hour | 4000 | 3200 | 3440 | 3740 | 1920 | 5173 | 4220 | 3740 |
| m ³ effluent/ton rock | 492 | 429 | 642 | 494 | 300 | 565 | 483 | 529 |
| Air temperature °C | 25 | 11 | 10 | 20 | 25 | 9 | 10 | 6 |
| Effluent temperature °C | 80 | 80 | 79 | 78 | 71 | 66 | 67 | 67 |
| Dew-point temperature °C | 69 | 71 | 69 | 73 | 74 | 63 | 64 | 45 |
| kg H ₂ O/ton rock entrained | 113 | 108 | 149 | 134 | 87 | 98 | 89 | 51 |
| SiF ₄ % (by vol) in effluent | 0.59 | 0.89 | 0.63 | 0.73 | 0.95 | 0.61 | 0.68 | 0.57 |
| F removal % | 24.2 | 30.6 | 31.7 | 31.5 | 25.2 | 28.7 | 27.6 | 32.4 |
| F removal kg F/ton rock | 9.2 | 12.6 | 13.1 | 12.5 | 9.9 | 11.6 | 11.2 | 12.0 |
| F removal determined indirectly % | 25.6 | 29.6 | | | | 28.6 | 27.6 | 32.8 |

* Effluent flow throttled down