## SEPARATION-OF BERYLLIUM FROM ALUMINIUM BY DIFFERENTIAL DESULPHATISATION

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### ABSTRACT

A study of the dissociation pressures of aluminium and beryllium sulphates indicates the feasibility of selective desulphatisation of anhydrous aluminium sulphate from a mixture of the two metal sulphates. The results of the experiments conducted, based on this theoretical consideration are given in this paper. The investigation shows that it is possible to effect the separation of the two metal sulphates.

### INTRODUCTION

" Chemically speaking, beryllium is quite closely related to aluminium and its complete separation from this element is difficult."[1] An investigation was undertaken with a view to develop a suitable method for the separation of beryllium from aluminium sulphate by differential desulphatisation.

The decomposition of a sulphate or its formation from its oxide, at any temperature is determined by its dissociation pressure at that temperature. If the partial pressure of sulphur trioxide superimposed on the system is greater than the equilibrium partial pressure $P_{SO_3}$, the oxide will get sulphatised and if less, decomposition of the sulphate will be favoured. The stability of a sulphate

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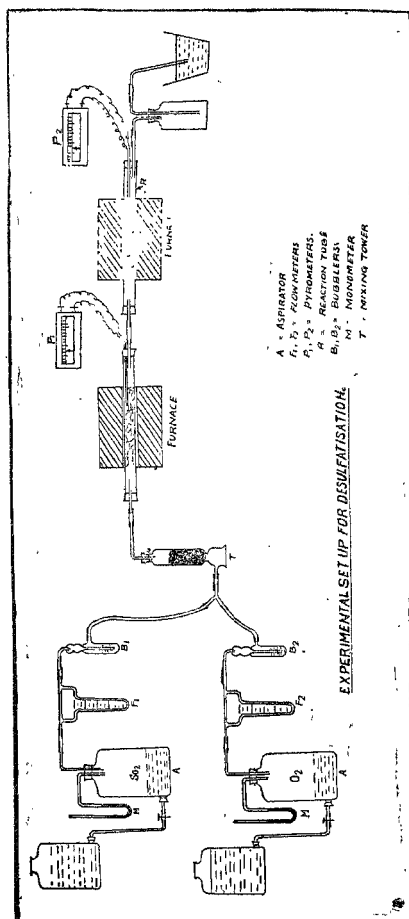
FIG. I

is therefore dependent on the temperature and partial pressure of sulphur trioxide to which it is subjected. It is therefore seen that if two sulphates have different dissociation pressures for any specific temperature, it would be possible by imposing a pressure of sulphur trioxide intermediate between the dissociation pressures of the two sulphates, to decompose one of the sulphates, leaving the other in the sulphate condition.

A study of the dissociation pressures of aluminium and beryllium sulphates indicates the feasibility of selective desulphatisation of anhydrous aluminium sulphate from a mixture of the two metal sulphates[2, 3]. For example, at 750°C, the dissociation pressures of anhydrous beryllium sulphate and anhydrous aluminium sulphate are 365 mm. and 900 mm. respectively, indicating the possibility of selective desulphatisation of aluminium sulphate at 750°C in an atmosphere of sulphur trioxide. If the mixture could then be cooled before the reaction reversed appreciably and then leached with dilute sulphuric acid, the unaltered beryllium sulphate would be dissolved, thus removing it from the mixture. The above principle was made use of in the following experiments for the quantitative separation of aluminium from beryllium by differential desulphatisation.

### EXPERIMENTAL METHODS AND RESULTS

*Materials*:—The aluminium sulphate used was of Analar quality and the beryllium sulphate chemically pure.

*Experimental procedure*:—The desulphatisation experiments were carried out in a silica tube furnace and the details are shown in Fig. I.

Sulphur dioxide was stored in the aspirator which contained water, over which was kept a layer of liquid paraffin, in order to prevent direct contact between gas and water. The gas was displaced from the aspirator and any moisture contained in the gas was removed by bubbling it through concentrated sulphuric acid. The rate of flow was measured by means of a calibrated flow meter included in the circuit.

Similarly oxygen was stored in another aspirator containing water and was displaced from the aspirator. Any moisture content in the oxygen was removed by bubbling it through concentrated sulphuric acid and the rate of flow was measured by means of another calibrated flow meter.

The gases were then mixed and passed into a catalyst furnace packed with catalyst for the conversion of $SO_2$ and $O_2$ to $SO_3$. The catalyst tube furnace was wound with nichrome and the temperature inside was measured by means of a calibrated pyrometer. The sulphur gases from the catalyst furnace were next led into desulphatising furnace for carrying out differential desulphatisation.

The desulphatisation experiments were conducted in a $30''$ long $\times 1''$ diameter (internal) nichrome wound silica tube furnace. The temperature of the furnace was controlled by a variable resistance and the temperature measurements were made using the chromel-alumel thermocouple. Silica boat containing

weighed quantity of the sulphates was placed inside the desulphatising furnace and sulphur dioxide and oxygen in diverse proportions were passed at different temperatures. Three methods were adopted for freezing the equilibrium at the end of the experiment. The final mixture was allowed to cool in air or allowed to cool in the furnace itself in an atmosphere of sulphur gases or quenched in water. The products were then leached with dilute sulphurid acid (2N).

The residue and extract were analysed for aluminium and beryllium by standard methods.[4] Experiments were conducted to study the influence of (1) temperature, (2) concentration of $SO_2$ in the gases, (3) method of freezing the equilibrium and (4) effect of catalyst on the degree of separation of aluminium from beryllium in the mixture.

## RESULTS

Preliminary experimental results shown in Table I indicate that decomposition of hydrated aluminium sulphate is a maximum at 850°C and that of hydrated beryllium sulphate a minimum, but the separation was not complete in mixtures of the hydrated sulphates. It is postulated here that this was so because hydrated aluminium sulphate which melts under its water of crystallisation,[6] on heating bulges out and envelops the particles of beryllium and aluminium sulphates, preventing the direct contact of sulphur gases with some of the beryllium and

### TABLE I

$SO_2 : O_2 :: 2 : 1$ (by volume); $P_{SO_3}$ calculated[5] : 0.16 atmosphere. Temperature of vanadium pentoxide catalyst furnace : 460°C. Duration of desulphatisation : 1 hour and 30 minutes. About 1.5 gm. each of hydrated aluminium and beryllium sulphates are accurately weighed in separate boats.

| Expt. No. | Desulphatisation Furnace Temperature °C | % Hydrated aluminium sulphate decomposed | % Hydrated beryllium sulphate decomposed |
|---|---|---|---|
| 1. | 700 | nil | nil |
| 2. | 700 | nil | nil |
| 3. | 780 | nil | nil |
| 4. | 780 | nil | nil |
| 5. | 800 | nil | nil |
| 6. | 800 | nil | nil |
| 7. | 850 | 96.0 | 10.1 |
| 8. | 850 | 96.3 | 10.4 |
| 9. | 850 | 95.1 | 10.5 |
| 10. | 850 | 96.2 | 10.9 |

aluminium sulphate particles and the escape of the sulphur gases due to decomsition of hydrated aluminium sulphate. This condition considerably altered the $P_{SO_3}$ superimposed over the sulphates and consequently the separation was incomplete.

Subsequent desulphatisation experiments were hence carried out with mixtures of anhydrous aluminium and beryllium sulphates. The results are given in Tables II, III and IV. From an examination of the results presented in the Tables, it is inferred that (1) nearly 85% of aluminium sulphate can be decomposed by cooling the boat in the furnace itself in an atmosphere of sulphur gases; (2) the method of freezing the equilibrium and time taken to freeze the equilibrium has definite influence on the decomposition of beryllium sulphate and (3) the type of catalyst used for the conversion of sulphur dioxide and oxygen to sulphur trioxide has no appreciable influence on the degree of separation. It may be noted that under identical experimental conditions, the dissociation figures are slightly varying. This may be due to the reversal of the reaction to a small extent owing to slight differences in cooling rates.

TABLE II

Temperature of vanadium pentoxide catalyst furnace: 460°C. Duration of each test: 1 hour and 30 minutes. About 1.5 gm. each of anhydrous aluminium and beryllium sulphates are accurately weighed and mixed.

| Expt. No. | $SO_2 : O_2$ ratio by volume | Desulphatisation furnace tempe- rature °C | % Aluminium sulphate decomposed | % Beryllium sulphate decomposed | Freezing method |
|---|---|---|---|---|---|
| 11. | 1.5 : 1 | 750 | nil | nil | Air cooled |
| 12. | 2 : 1 | 780 | nil | nil | ,, |
| 13. | 1.5 : 1 | 800 | nil | nil | ,, |
| 14. | 1.5 : 1 | 800 | nil | nil | ,, |
| 15. | 2 : 1 | 800 | nil | nil | ,, |
| 16. | 2 : 1 | 850 | 89.8 | 14.9 | ,, |
| 17. | 2 : 1 | 850 | 88.2 | 14.1 | ,, |
| 18. | 2 : 1 | 850 | 88.9 | 14.7 | ,, |
| 19. | 2 : 1 | 880 | 82.0 | 24.5 | ,, |
| 20. | 2 : 1 | 880 | 81.1 | 24.3 | ,, |
| 21. | 2 : 1 | 850 | 88.1 | 3.1 | Water quenched |
| 22. | 2 : 1 | 850 | 85.4 | 1.55 | Furnace cooled up to 700°C |
| 23. | 2 : 1 | 850 | 86.0 | 1.01 | ,, |

Table III

Temperature of platinised asbestos catalyst furnace: 460°C. $SO_2 : O_2$ ratio (by volume) :: 2 : 1. Duration of each test : 1 hour and 30 minutes. About 1.5 gm. each of anhydrous aluminium and beryllium sulphates are accurately weighed and mixed.

| Expt. No. | Desulphatisation furnace temperature °C | % Aluminium sulphate decomposed | % Beryllium sulphate decomposed | Freezing method |
|---|---|---|---|---|
| 24 | 780 | nil | nil | Air cooled |
| 25 | 850 | 91.2 | 15.3 | ,, |
| 26 | 850 | 88.2 | 4.2 | Water quenched |
| 27 | 850 | 84.4 | 0.88 | Furnace cooled up to 700°C. |
| 28 | 850 | 85.1 | 0.91 | ,, |
| 29 | 850 | 85.3 | 0.76 | ,, |

Table IV

Temperature of platinised asbestos catalyst furnace: 460°C. Temperature of desulphatisation furnace: 850°C. $SO_2 : O_2$ ratio (by volume) :: 2:1. About 1.5 gm. each of anhydrous aluminium and beryllium sulphates are accurately weighed and mixed.

| Expt. No. | Duration of test hours | % Aluminium sulphate decomposed | % Beryllium sulphate decomposed | Freezing method |
|---|---|---|---|---|
| 30 | $1\frac{1}{2}$ | 83.7 | nil | Furnace cooled up to 700°C |
| 31 | $1\frac{1}{2}$ | 83.6 | 4.51 | ,, |
| 32 | 3 | 83.0 | 3.6 | ,, |
| 33 | 3 | 82.4 | 3.7 | ,, |
| 34 | $1\frac{1}{2}$ | 87.3 | 4.1 | Water quenched |
| 35 | $1\frac{1}{2}$ | 87.6 | 6.5 | ,, |

1. Hampel, A.   ..   "*Rare Metals Handbook*," Reinhold Publishing Corporation, 1954, 42.

2. Kubaschewski, O. and Evans, E. LL.   "*Metallurgical Thermochemistry*," Pergamon Press, 1956.

3. Willard, H. H. and Fowler, R. O.   "*J. Amer. Chem. Soc.*, 1932, 54, 496-516.

4. Scott.   ..   "*Standard Methods of Chemical Analysis*," V Edition, 1, Van Nostrand Co. Inc.

5. Kelly, K. K.   ..   "Thermodynamic Properties of Sulphur and its Inorganic Compounds," *U. S. Bureau of Mines Bulletin*, 1937, 406.

6. Mellor.   ..   "*Mellor's Inorganic and Theoretical Chemistry*," Longmans, Green & Co., 1924, 5, 334.