#### Hüseyin Gülensoy

limestone and clay in different compositions exist in commercial samples together with boron mineral, it is impossible to determine CaO percentage directly with the method given in the following.

Method: 1,000 g crystalline sample, which is ground in an agate mortar, is taken into a 400 ml beaker. On it 100 ml n 10 Titriplex III solution is added. The beaker content is stirred by a magnetic mixer with a medium speed for one hour. Dissolution is complete. In a Florance flask the beaker content is completed to 250 ml by distilled water, from this 25 ml. is taken into an erlenmayer flask. After diluting with 100-150 ml of distilled water, 2-3 g NH<sub>4</sub>Cl, 5 ml. concentrated ammonia and one Ind. puffer pill are added. The green-coloured solution is titrated with n/10 (F=1) Mg + solution until the red turning point.

If the factor of 0,1 N Titriplex III solution is 1,00 and if S ml of Mg<sup>++</sup> solution is spent for this back titration, in the sample the CaO % will be equal to  $(10-S) \times 5$ , 6.

In this kind of studies Murexid, Calcon acid, etc. can also be used as indicator, if the necessary conditions are established.

In a similar way CaO determinations can easily be done for Pandermite, Colemanite, Gypsum, Calcite, Aragonite and marble minerals, too.

The percentage of CaO in pure Colemanite crystal having the composition  $2CaO \cdot 3B_2O_3 \cdot 5H_2O$  is 27,27 %

The percantage of CaO in pure Pandermite crystal having the composition  $4CaO \cdot 3B_2O_3 \cdot 7H_2O$  is 30,53%

In both pure crystals by the determination methods discussed above, the following results were obtained.

	In Colemanite	In Pandermite	
% CaO		30,21	
	27,49	30,12	
	27,09	30,62	

Determination of BO,, CaO and As.O, in Boron minerals Depending ...

## II. BO3 DETERMINATION

# a°. In pure Crystals.

100 ml of sample taken from 250 ml solution obtained from studies similar to (I) is put into a 600 ml beaker. 30 drops of phenolphatalein indicator solution % 1 in alcohol is added. The colourless solution is titrated with 0,5 N (F=1) KOH solution until light pink colour is reached. The amount spent is shown as  $(S_1)$  ml.

At this moment the titration is stopped and 5 g mannite is added to the sample. The solution becomes colourless and it is again titrated with the same KOH solution until the colour turns to pink. The spent amount is  $(S_2)$  ml.

The first titration is to increase the pH of the solution to 8 which has been lowered to 5 during the dissolution of mineral, the second is to neutralize H ions which have been passed into the solution by the addition of mannite

Thus; %  $B_{2}O_{3}=S_{2}\times 2,5\times 1.741$ 

The results of the  $B_2O_3$  determinations in Pandermite, Ulexite and Colemanite by the discussed method are given in the following.

	B <sub>2</sub> O <sub>3</sub> In original form	B <sub>2</sub> O <sub>3</sub> By determination
In Colemanite mineral	% 50,85	% 50,81
		50,67
		50,58
In Pandermite mineral	% 52,30	% 52,22
		52,17
		<b>52</b> ,08
In Ulexite mineral	% 42,95	% 42,75
		42,67
		42,81

### b°. In commercial samples.

The  $B_2O_3$ , in commercial samples, is also determined by the same method discussed in (a). Approximately 1 g of finely ground sample is dissolved in 100 ml 0.1 N Titriplex III solution, and then the beaker content is completed to 250 ml by distilled water. 100 ml of solution sample is taken, 30 drops of phenolphatalein indicator solution % 1 in alchol is added and then titrated with 0.5 N KOH until the colour turns to light pink. The amount spent is  $(S_1)$  ml.

Then 5 g mannite is added into the same solution and titrated with 0.5 N KOH until the colour turns to pink again. The amount spent is  $(S_2)$  ml.

Thus

1 ml. 0,5 N (F=1) KOH solution will correspond to 17,41 mg of  $B_2O_3$ .

For these studies a commercial colemanite sample having the following complete analysis results, were taken as basis.

$B_2O_3$	% 41,15	Na <sub>2</sub> O	% 0,27
SiO <sub>2</sub>	6,54	K,O	0,40
Al <sub>2</sub> O <sub>3</sub>	1,06	As O-	0,23
Fe <sub>2</sub> O <sub>3</sub>	3,98	SO3	0,31
CaO	24,26	Fire loss	19,52
MgO	2,20		

For this mineral ore, at the end of the analysis by the method discussed above, the following results were found.

The titration of  $B_2O_3$  can also be made in two steps. For this, 100 ml. sample taken from 250 ml dissolution sample, is titrated until the colour turns to light pink by the presence of 30 drops of phenolphatalein ind. The amount spent is  $(S_1)$  ml. Then 100 ml sample in again taken and 30 drops of phenolphatalein ind. solution is added and titrated until the colour turns to pink again. The amount spent is (S) ml.

The amount spent for  $B_1O_3$ :  $S_2=S-S_1$  ml.

22

Determination of B<sub>2</sub>O<sub>1</sub>, CaO and As<sub>2</sub>O<sub>2</sub> in Boron minerals Depending...

#### **III.** As<sub>2</sub>O<sub>3</sub> DETERMINATION

2,0000 mg mineral ore sample is taken and dissolved in a beaker as described before by addition of 200 ml. 0,1 N Titriplex III solution. The undissolved part is filtered and the decantate is completed to 250 ml by distilled water. 100 ml sample, taken from this solution, is titrated with N 20 I<sub>2</sub> solution after adding 2-3 g of NaHCO<sub>3</sub>. Starch solution is used as indicator.

5c As<sub>2</sub>O<sub>3</sub> will be equal to 0,31×S where the (S) is the spent amount.

As the result of these kind of studies percantages of  $As_2O_3$  were found 0,228 %, 0,221 % respectively. This percentage was 0,23 in the commercial colemanite sample whose complete composition is given on IIb.

In the analysis given on IIb the  $As_2O_3$  determination had been made by spectrophotometer after dissolving the sample in sulphuric acid. The same result was once more obtained by iodine titration after sperating  $AsCl_3$  by distillation.

### CONCLUSION

In pure crystals of Ca - hydroborate mineral such as Colemanite, Pandermite, etc.  $B_2O_3$  and CaO determinations can easily be done by using 0,1 N (sometimes 0,2 N) Titriplex solution as solvent. It is obvious that  $B_2O_3$  and MgO determinations, in Kurnakovite and Boracite minerals, can also be done in the same way.

After a dissolving process in this way, percentages of  $B_2O_3$  and  $As_2O_3$  in the commercial boron mineral ore samples can be obtained easily and in a short time compared to other methods.

In determination of  $B_2O_2$  at which Titriplex III is used as a solvent, the solvent at the same time attaches the metal ions to itself which can come from the ore and be harmful to the titration.

## Literature

- H. Gülensoy and H. Savcı. «The solubilities of some calcium compounds and minerals in EDTA».
- Bull. of the Min. Research and Explo. Ins. of Turkey. 86, 77-94, (1976).
- E.D. Glover. Petrol. 31, 622 6, (1961) Chem Abst. 3104 d, (1962)
- W.E. Hill and D.R. Evans. Kansas Bull. No: 175, (3), 1 22. (1965) Chem. Abst. 64, 13923 c, (1966).