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Quantitative Spectrum Analysis (Part V) : Determination of Rubidium Chloride contained in an Artificial Carnallite

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Quantitative Spectrum Analysis. Part V. Determination of Rubidium Chloride contained in an Artificial Carnallite.

By

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I. Introduction.

As is generally known rubidium occurs widely distributed though in small amounts all over the earth's crust, but its quantitative determination by means of emission-spectra has not yet been made in our country. The present writer tried to determine the amount of rubidium chloride contained in an artificial carnallite which was prepared from bittern obtained from the In-land-Sea district.

II. Sample electrodes.

For the quantitative determination, the solid sample or its very concentrated solution was transformed into solid conductive electrodes in the usual way. For example, 40 c.c. of 70% aqueous solution of carnallite and 10 c c. of concentrated hydrochloric acid were mixed in a mortar. To this solution 20 gms. of dehydrated zinc oxide were added little by little and after mixing thoroughly, the mixture was pressed into a mould and dehydrated in an air-oven. Six tablets were thus prepared, each weighing 7.5 gms. A pair of the tablets were employed as electrodes for exciting sparks.

Several electrodes containing varying amounts of rubidium chloride were similarly prepared with the artificial carnallite and pure rubidium chloride of Kahlbaum make.

III. The apparatus used and the method of experiment.

To excite sparks between a pair of electrodes, a transformer giving 10 K. V. in the secondary was used. Self-inductance of a magnitude of 5500 cm. was always inserted in the secondary circuit, and no capacity was inserted. The light from an uncondensed spark between the electrodes was focused, by means of a lens, on the slit of a constant-deviation-spectrograph. The dispersion of the spectrograph was 25 Å per mm. at $\lambda=4200$ Å.

The rubidium lines at $\lambda=4215.6$ Å and 4201.8 Å etc. were detected; of these lines, the line at $\lambda=4201.8$ Å was the most sensitive.

The method of analysis was to draw a density-concentration curve in connection with the line at $\lambda=4201.8$ Å and to determine graphically the concentration of the rubidium chloride from the density of the rubidium line in the spectrogram of the unknown sample.

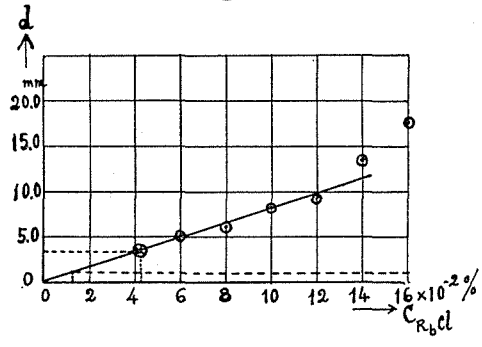
The so-called "method of comparison" was also tried, and the two results were compared.

IV. Sensitivity of spectrographic analysis.

The sensitivity of the spectrographic analysis of rubidium chloride contained in artificial carnallite was determined in the same way as was described in the preceding paper.[1] The relation between the concentration of rubidium chloride contained in artificial carnallite and the intensity of the rubidium line at $\lambda=4201.8$ Å is shown in the following table and the figure, d representing the galvanometer deflection measuring the intensity of the line.

Conc. of RbCl %	$d_{mm}\lambda=4201.8$Å
x	3.5
6.10^{-2}	5.0
8.10^{-2}	6.1
10.10^{-2}	8.2
12.10^{-2}	9.1
14.10^{-2}	13.5
16.10^{-2}	17.6

Fig. 1.



1. These Memoirs **14**, 51—2, 1931.

The corresponding microphotometer records are shown in PL. I. a and b.

The sensitivity was thus found to be $1.2 \cdot 10^{-2}$% under the conditions of the experiment.

V. Examples of analysis.

In the above table, the concentration of rubidium chloride initially contained in the sample of artificial carnallite taken for experiment is denoted by x, and the height of the curve corresponding to the density of the rubidium line was found by measurement to be 3.5 mm. That height of the curve corresponds to the concentration of $4.2 \cdot 10^{-2}$% of rubidium chloride, as is shown in the figure. Another sample of an artificial carnallite was also taken and the analysis was performed according to the methods described in III. The concentration of rubidium chloride contained in that sample was found to be $1.5 \cdot 10^{-2}$%. (See PL. I. c)

The results of the experiments showing how the method of comparison may be employed for the analysis, turned out as follows: (See PL. II. a and b)

Let S denote the concentration of rubidium chloride in the sample taken.

Ex. S=0.060 % RbCl

Conc. of RbCl %	$d_{mm}\lambda = 4201.8$ Å	
0.040	19.6	> 7.6
S	27.2	
0.080	34.4	> 7.2

by proportion we get, $40 \times \frac{7.6}{14.8} = 20.6$

$\therefore$ S=0.040+0.0206=0.0606

$$\Delta = 100 \times \frac{6}{600} = 1.0\ \%$$

The result of analysis of the same sample by the graphical method of applying the density-concentration-relation is shown in Table II and Fig. 2, the microphotomer records being given in PL. II. c.

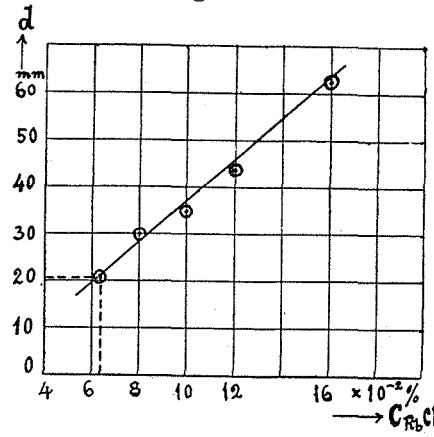
Another sample was similarly analysed and the content of rubidium chloride was found to be 0.172 % as against the calculated value of 0.180 %, while by the graphical method from the density-concentration-

Table II.

Conc. of RbCl %	$d_{mm}\lambda=4201.8$Å
x	20.5
0.080	30.2
0.100	34.9
0.120	43.4
0.160	66.0

x=0.064 %

Fig. 2.



relation it was found to be 0.191 % as is shown in No. 3 column 5 in the following table.

Table III.

No.	Calculated value	Comp. method	△ %	d-c-relation	△ %
0	——	——	——	0.042	——
1	——	——	——	0.015	——
2	0.060	0.0606	+1.0	0.064	+6.6
3	0.180	0.172	−4.4	0.191	+5.6

It is thus seen that both method of analysis may be adopted for determining the amount of rubidium chloride in an artificial carnallite, with results which agree with each other and also with the calculated value fairly well.

Sammary

1) Solid sample electrodes were prepared from an artificial carnallite, zinc oxide and hydrochloric acid; and the amount of rubidium chloride in the carnallite was spectrographically determined with a sensitivity of $1.2\cdot10^{-2}$%.

2) Two samples of artificial carnallite prepared from bittern produced in the In-land-Sea district were thus analysed and found to contain 0.042 % and 0.015 % rubidium chloride.

3) Both the " method of comparison " and the graphical method were found to be applicable for determination of rubidium chloride in artificial carnallite with fair exactness, errors being always within the permissible limits.

Plate I

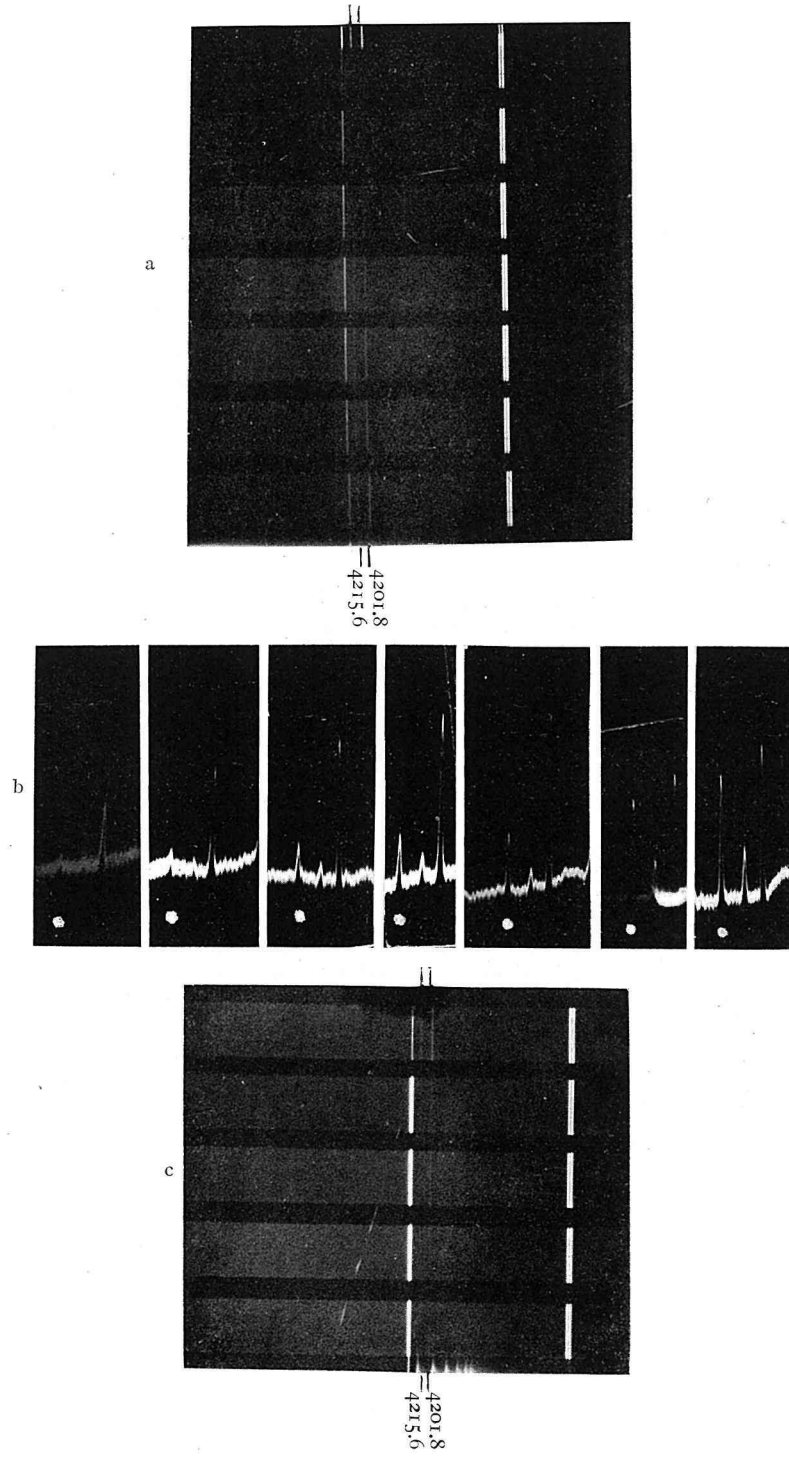
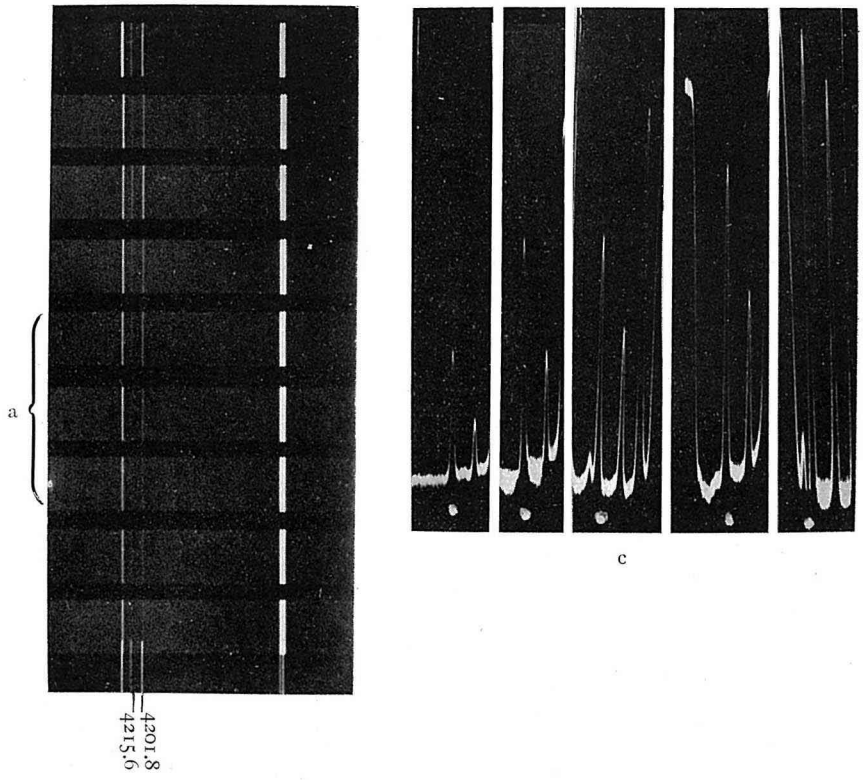



Plate II