

## Micro-colorimetric Determination of Chloride Ion by a Spot Test

by

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With a water sample of which the concentration of chloride ion ranges from 1 to 100 mg/l and the volume is above 20 cc, MOHR's titrimetric method modified by Y. MIYAKE [1] is ordinarily used. Also, with a water sample of which the chloride concentration ranges from 0.1 to 10 mg/l and the volume is above 20 cc, the colorimetric method using mercuric thiocyanate [2] is very convenient.

The method of chloride determination in one drop (ca. 0.02 cc) of a water sample of which the chloride concentration ranges from 10 to 500 mg/l is presented in this report.

### 1. General procedure

A water sample and a series of standard solutions, each containing between 10 and 500 mg of chloride ion per one liter, are taken and 0.02 cc of each is dropped on a white glass plate through a micro-pipet. Then, the appropriate one of the following two methods is selected corresponding to the chloride concentration in the sample. 1) When the chloride concentration in the sample is below 200 mg/l, about 0.01 cc of 0.3 per cent silver nitrate solution is added to a spot of each sample and also of the standard solution at the same time. Afterwards, these spots are irradiated under ultraviolet rays for a few minutes. By comparing the violet coloration of the silver chloride precipitate dispersing in the spot of the sample water with that produced in one of the series of standard solutions, the chloride content can be determined. 2) When the chloride concentration in a sample exceeds 30 mg/l, the determination can be made by comparison of the yellow colors appearing in a sample and in one of the standard series after the addition of about 2 mg of solid silver chromate to each spot. The color may perfectly appear 5 or 10 minutes after the addition of the reagent.

### 2. Application

The method described above was applied to a determination of chloride ion in rain water. A test determination was made with a solution containing one mg chloride ion per one liter, which is nearly equivalent to an average concentration of chloride ion in rain water.

At first, 5 or 10 cc of a water sample is evaporated down to 0.1 cc. Then, from the residual solution is taken through a micro-pipet a drop approximately

equal to 0.02 cc. The succeeding procedure is carried out in the same way as mentioned in the previous section. The result of the comparison of the conventional titrimetric method with the present micro-colorimetric one is shown in Table 1.

Table 1. Comparison of a titrimetric method with a micro-colorimetric method.

Method	Volume of sample, cc	Evaporated to, cc	Determined concn. mg/l
titrimetric	50	—	1.2
micro-colorimetric	10	0.1	1.1
	9	0.1	1.2
	8	0.1	1.0
	7	0.1	1.0
	6	0.1	1.2
	5	0.1	1.2
	2.5	0.05	1.1

### 3. Disturbing elements

The existence of an organic substance disturbs the determination when it is carried out by the first method. Disturbing elements against MOHR's method must also be eliminated for the second method.

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### References

- [1] MIYAKE, Y., 1954: "Suishitsu-bunseki" (in Japanese), Tokyo, p. 71.
- [2] UTSUMI, S., 1952: New colorimetric determination by use of thiocyanates. I-II. New colorimetric determination of chloride using mercuric thiocyanate and ferric alum (1-2). (in Japanese), J. Chem. Soc. Japan. 73, p. 835.