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A Method of Concentrating Solutions by Means of a Paper Strip for Solution-Spectrochemical Analysis

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Abstract

A simple method of quantitatively concentrating the solution of more than several millilitres to less than 0.1 ml. was studied. A strip of filter paper for qualitative chromatography which is cut into a definie figure is hung at the solution dish and the sample solution is soaked down gradually from the top of the strip. When the solution reached to the lower part of the strip, the solute in the solution that soaked down is deposited by infrared heat irradiation. Then, the deposited solute is eluted with the solvent which is soaked down in the same method as used in developement. More than 99 percent of the solute in the solution can be recovered in the first a few drops that drop from the lower end of the strip.

I. Introduction

Various concentration techniques are used in the analytical chemical procedures, and they often raise the sensitivity of chemical analysis. The usual concentration techniques contain evaporation of the solvent, liquid-liquid extraction of the solute, ion-exchange technique and so on. These methods are useful and can be applied to the concentration of the several ml. of solution, but they cannot be quantitatively used for smaller amounts of solutions of less than 1 ml..

The technique mentioned below is of concentrating the solutions from more than 1ml. to the volume of 0.12–0.04 ml. quantitatively. The micromethods of chemical analysis are enhanced by this technique. Especially, the technique is useful for concentration of very dilute solutions for spectrochemical analysis using the soak up electrode, and it is also useful for the samples of paper chromatography.

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II. Procedures

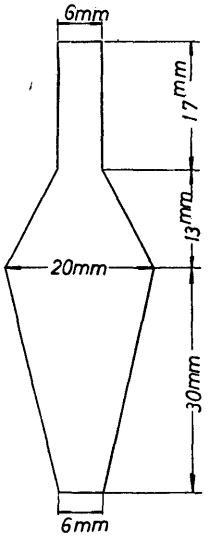

Fig. 1.

The procedures consist of two steps. At first, the sample solution in a small dish which is to be concentrated, is soaked down by a strip of filter paper for qualitative chromatography which is cut into a definite figure (Fig. 1). A smaller area of the strip is convenient for efficient elution, but some larger area is needed to quicken the vaporization of the solvent in the sample solution. The whole apparatus is shown in Fig. 2. The paper strip is hung at the edge of the solution dish (*A*) of 1 ml. capacity. The solution is soaked down through the paper strip and when it has reached to the lower part of the strip, hot ray from the infrared heating lamp irradiates the part (*c*) of the strip. The upper part of the strip is hidden behind the shield of the aluminum foil (*D*) of 0.1 mm. thickness, 15 cm. width and 10 cm. length. The shielding foil is set on between the two vertical poles stood at the both sides of the stand that supports the sample dish. During the sample solution soaks down and the hot ray heats the lower part of the strip, the sample solution loses its solvent and only solute deposits at the lowest part of the strip. Thus the major part of the solute in the sample solution can be collected within the narrow band at the lowest end of the strip.

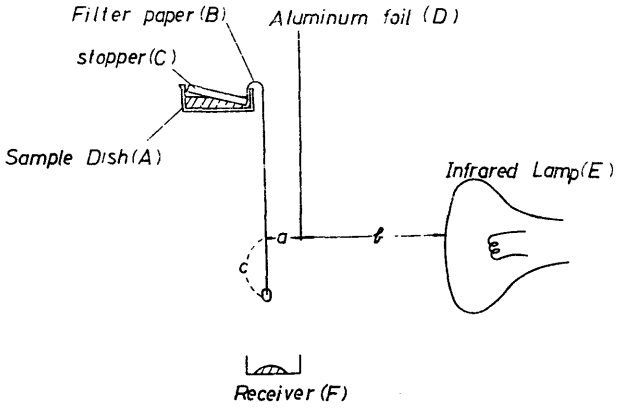

Fig. 2.

Next, the deposited solute is eluted with the solvent which soaks down gradually from the top of the paper strip by using just the same apparatus as developed the sample solution (Fig. 2). The solvent that soakes down through the paper strip, elutes the adsorbed solute from the upper and middle part

of the strip to the lowest end of the paper. Then, the solvent successively extracts the major part of the solute which has previously deposited at the lowest end of the strip. When the eluant which soaked down is accumulated, it drops down from the lowest end of the strip into the receiver. The rate of dropping must be controlled, as it relates to the efficiency of concentration. In the early experiments, the solvent was charged by dropping it from the micropipette, but the same apparatus used in developing the solution was found to be convenient to control the rate of the solvent, later. In this apparatus the eluant that drops from the lowest end of the strip can be easily controlled by the strength of the infrared irradiation from the front side.

The reservoir made of polyethylene has the capacity of about 1ml., but larger quantities of solutions can be concentrated by successive adding the solution. The sizes of concentrated drops are depended upon the shape of the paper strip chiefly its lower edge. The strip shown in Fig. 1 delivers drops of about 0.04 ml. each.

The time to develop and the area of deposition are defined by the geometrical conditions of irradiation (Fig. 2). A part of the ray emitted from the infrared lamp (E) is shielded by a sheet of aluminum foil (D) and it heats the lower part of the strip. The larger the distance ($a+b$) between the strip and the infrared lamp, the longer the time required for evaporating solution. Fig. 3 shows this relation,

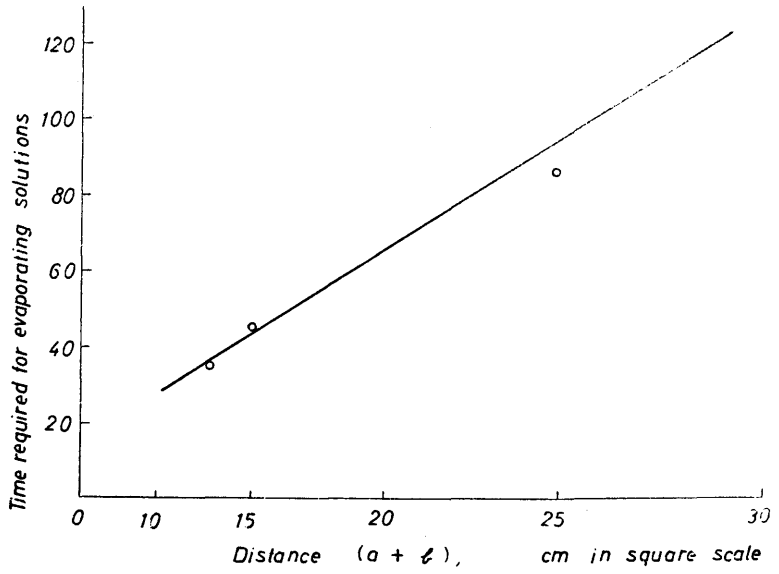

Fig. 3.

in which the time taking for evaporating is proportional to the square of the distance $(a+b)^2$. In case of putting the distance $(a+b)$ constant, the larger the distance (a), the shorter the time for drying the solution because of the larger irradiation area by the infrared lamp. On the other hand, increase of the length

(*c*) results worse recovery owing to make the area increase where the solute adheres more tightly than the upper parts.

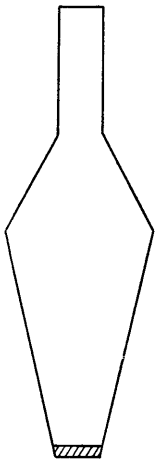

Fig. 4.

In practice, the area near the lowest end of the strip where the solute deposits is required to be smaller, and the distance (*a*) and (*c*) are defined as 10 mm. and 7 mm. respectively. Thus the solute deposits within the width of about 1 - 2 mm. as is shown in Fig. 4.

III. Results and Discussions

The ferric chloride solutions were tested to concentrate by this technique, and the results are shown in Table 1. In these experiments, the 0.1 N HCl was used as effluent and the elution time was 30 sec. per one drop. The 2 mg—25 μg/ml. of ferric chloride solutions were concentrated and iron in the concentrated solutions were determined by the colorimetric determination of thiocyanate method. These results are very good and more than 99 % of iron are recovered in the first two drops. As is shown in Table 1, the smaller iron concentrations seem to promote

Table 1. Concentrating the iron chloride solutions

Fe taken	Fe recovered in the concentrated drop			
	1 st	2 nd	3 rd	recoveries
(μg)	(μg)	(μg)	(μg)	(%)
2000	1620	340	0	99
2000	1700	280	2	99
1600	1450	150	0	100
250	250	0	0	100
125	125	0	0	100
50	50	0	0	100
24	25	0	0	100

Elution time; 30 sec./drop, Effluent; 0.1 N HCl

Table 2. Effects of elution time on concentrating the very dilute solutions. (Determination by using 10μg/ml. of Co solutions containing Co-60.)

Co taken	Elution rate	Recovered in drops (cpm)						recoveries
(cpm)	(min./drop)	1-2	3-4	5-6	7-8	9-10	11-12	(%)
94100	15	89643	4513	215	46	–	–	100
93814	10	91776	1700	137	66	–	–	100
93814	5	62946	20261	1454	625	451	261	91.4
93814	1	61051	19611	3044	1258	1303	510	91.7

Effluent; 0.1 N HCl, Natural background; 376 cpm

recovering in the first drop, but it may be apparent phenomena comes from analytical errors due to larger dilution —— a drop of solution to the cuvette volume and consequent lack of colorimetric sensitivity.

For removing these causes, the radioactive tracer, cobalt-60 was used and results are shown in Table 2, from which it was known that the recovery was effected by elution rate. The slower dropping rates makes the concentration efficient. But if the rate of dropping of the concentrated drops from the lowest end is too fast, the deposits which left on the strip become larger. With 10 min. elution, 10 μg/ml. of various substances containing the radioactive tracers were concentrated, and the results in Table 3 were obtained.

As is clear from mentioned above, the technique can be used simply and useful in all analytical procedures and makes the sensitivities of the analytical methods higher, although it contains some problems to be further improved.

The solvent of 0.1 N HCl was used for all over the experiments, but some efficient solvent have to be searched in case by case.

On the other hand, some considerations are needed on development of the sample solutions. For example, slightly burning of the filter paper by over irradiation lowers the recovery, and by long time standing the developed strip the recovery lowers further. In developing the sample solution, the shadow part of the aluminum foil must be also heated a certain degree, and a part of solvent in the solution that soaks down must be removed by evaporation. Unless this vaporization in the upper part is not proceeded, the developing solution reaches so rapidly to the lowest end of the strip and the drops of eluant do not stop there for a long time, and consequently efficient concentration is not proceeded. For this reason a thin aluminum foil was used instead of the thick metallic plate.

Figures of the strips were also examined. After examining many shapes of developing paper, we found out that such one as in Fig. 1 satisfies the micro-concentating conditions. This strip consists of the top-inlet portion of the narrow band and of the wide portion of rohmb which is cut at the lower portion as is shown in Fig. 1.

Although the technique has some problems that have to be improved, the authors report it for its usefulness in various applications.

Table 3. Recovered percentages of the ions, containing radioactive ion, concentrated into four drops.

No. of experiments / Ions	1	2	3	average
$^{45}Ca^{2+}$	98	97	96	97
$^{60}Co^{2+}$	99.1	99.0	99.6	99.3
$^{65}Zn^{2+}$	95	94	97	95
$^{32}PO^{3-}$	98	99	98	98

Elution rate; 10 min./drop Effluent; 0.1 N HCl, Ion concentration, 10μg/ml.