Determination of Micro Amounts of Elements
Using the Ring Oven Method
with Preliminary Chromatographic Separation

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In a previously published paper¹ one of us (M. B. Č.) together with Weisz² described a method for the determination of some elements using the Weisz ring oven with preliminary chromatographic separation. This method consisted in chromatographing the mixture of the elements on filter paper and burning the cut zones. The ash obtained is then dissolved in mineral acid, and the solution obtained is diluted with water to a volume of 1 ml. The concentration of the elements in the solutions obtained is determined using the ring oven method in the usual manner.

Continuing our investigations we have simplified this method to avoid both the burning of the filter papers with the separated elements, and the preparation of solutions. We separated the elements by chromatographing them on narrow strips of filter papers (5 mm). It is necessary to take four strips for one determination. By means of a capillary pipette (about 2 µl) varying quantities (1–10 drops) of the solution of the mixture of the elements to be estimated (about 0.1 mg/ml of each ion) were put on each strip. On the strip with the greatest numbers of drops the elements were detected by means of suitable reagents, in order to find the locations of the elements on the other strips, i.e. to find the places where the strips have to be cut. The cut out parts from the strip with the separated elements were rolled spirally and put in a glass tube with a tapering end, pressed slightly to protrude a little out of the narrow end of the tube and give contact with the round filter paper lying on the ring oven. The element was then washed into a ring by means of a capillary pipette, adding as much of the solvent as the rolled strip could absorb.

* Weisz² also used the ring oven method to make possible the detection of very small amounts of elements on developed chromatograms.
The elements in the ring zones were transformed first into the corresponding sulphides* and then to silver sulphide. The ring zones obtained were compared in the usual manner with the ring zones of a silver sulphide standard scale. The method described was checked by determining the following elements:

1. Cu (from the solution: Cu + Pb),
2. Pb (from the solution: Cu + Pb),
3. Co (from the solution: Co + Zn),
4. Zn (from the solution: Co + Zn).

The results obtained were in the range of errors obtained by the determination of the individual elements by means of ring oven, i.e. in a range of ± 5%.

Paper chromatography is an excellent method for the separation of small amounts of elements, but it is not very suitable for their exact determination. The ring oven method is a very good one for the determination of small amounts of elements, but elements with similar properties cannot be determined in mixtures. Because the method described combines the good properties of paper chromatography and the ring oven method, it offers the possibility for the determination of small amounts of elements in mixtures of very varying composition.

**Experimental Part**

For the preparation of the solutions examined, Cu(NO₃)₂, Pb(NO₃)₂, CoSO₄ and ZnSO₄ were used. The chromatography was performed on strips of Whatman No. 1 filter paper 5 mm × 20 cm. The strips were cut by means of a special knife made from two razor blades.

The developing was carried out in a glass cylinder by the ascending method. The solvent mixture for Cu and Pb was prepared by mixing 90 ml of ethanol and 10 ml of 5 N HCl. The time of development was about 4 hours. The elements were detected by gaseous H₂S. The R₁-values were: Pb — 0.0; Cu — 0.4.

The solvent mixture for the separation of Co and Zn was prepared by mixing 50 ml of n-butanol and 50 ml of 5 N HCl. The time of development was about 10 hours. Co was detected by spraying the strip with a 1% alcoholic solution of salicylaldoxime and Zn was detected with a 0.05% chloroformic dithizone solution. After spraying, the strips were exposed to gaseous ammonia. The R₁-values were: Co — 0.42; Zn — 0.98.

* Before precipitating lead sulphide, the round filter paper should be exposed to gaseous ammonia.