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Detection of some noble metals by heat coloration on thin layers with direct flame

Hitherto, little work has been carried out on the thin-layer chromatography (TLC) of such noble metals as platinum, palladium, etc. The present studies were undertaken to separate platinum, palladium, gold and copper by TLC with a singular coloration method. The method was suggested considering a long known fact¹ that colloidal solutions of platinum or gold have been easily prepared when the surface of the metal salt solution came in contact with the outer flame of a Bunsen burner. Prior to the experiments with mixtures, detection limits of the individual metals were established using the microsyringe (for gas chromatography) to apply a minute amount of each metal solution onto the thin-layer plate. As a result, 10 γ of Au, 5 γ of Pt, 1 γ of Cu and 2.5 γ of Pd were detected. By this chromatography method, every process of the experiment was simple and rapid.

Experimental

Silica Gel H (Merck, according to STAHL) was used as adsorbent. The powder was treated with HCl (1:1) to remove mainly iron and was washed thoroughly with distilled water using centrifugation repeatedly. The adsorbent was moderately dried in the air and thereafter the suction filter was applied. Finally, it was washed with benzene, and the powder was then dried in an oven at 120° for 2 h.

The thin-layer plates were prepared as follows: 2–2.5 volumes of water were added to one volume of the powder freed from impurities and mixed thoroughly until a slurry was obtained. The slurry was spread onto the glass plates (3.5 × 10 cm) using the applicator to give a layer 0.7 mm thick. (The thickness was not so critical for the experiment.)

After exposing the plates to air for 15 min, they were dried at 100–110° for 1 h. Sample solutions used were chlorauric acid, copper nitrate, chloroplatinic acid and palladium dichloride, containing 10 mg of the individual metallic ion in 1 ml. A minute amount of the solution containing four components was applied with the aid of a capillary tube to a point on the layer surface 1.2–1.5 cm from the lower edge of the plates. The plates containing the spotted samples were left exposed to the air for 15–20 min; the development was then allowed to take place. A small desiccator (base diameter, 11 cm) was used as the chromatographic tank in which the solvent had been placed to a depth of 5 mm. The thin-layer plates were put in the tank, resting against the wall, so that the lower edges were immersed in the solvent. The development was stopped when the solvent front had moved 7–9 cm from the starting point. The development time was 15–20 min.

Of several solvents tried, pure acetone (water content, 0.3–0.4%) gave the most satisfactory result. Dioxane–acetone (1:1) and dioxane–acetone–conc. HCl (200:100:1) were second to acetone for the separation of four components, although slight tailing could be seen in the case of the latter two systems.

The plates removed from the tank after development were then dried in the air. Finally, the whole surface of the thin layer was heated by contacting the oxidizing flame of the aerated Bunsen burner as uniformly (to prevent the glass plates from breaking) as possible for 1–1.5 min.

TABLE I

R_F VALUES AND SPOT COLORS

<i>Metal (spot color)</i>	<i>R_F values</i>		
	<i>Acetone</i>	<i>Dioxane-acetone- conc. HCl (200:100:1)</i>	<i>Dioxane- acetone (1:1)</i>
Au (purplish pink)	0.94	0.84	0.99
Pt (brown)	0.68	0.40	0.49
Pd (brownish black)	0.81	0.29	0.44
Cu (yellowish brown) ^a	0	0.79	0.09

^a Mixtures probably of Cu₂O and CuO.*Results and discussion*

All spots were colored by the heating. The colors of the individual spots and *R_F* values corresponding to solvents are given in Table I. Of course, before any separation experiment, the colors of the individual components caused by the heating, must be established. In this method, in addition to differences in the colors and locations of the spots, the palladium ion is very quickly reduced and, therefore, also facilitates the identification of those components.

*Faculty of Science,
Kumamoto University (Japan)*

DAISEI YAMAMOTO
HIROKO UNO

1 W. OSTWARD, P. WOLSKI AND A. KUHN, *Kleines Praktikum der Kolloidchemie*, Verlag von Theodor Steinkopff, Dresden and Leipzig, 1923, p. 4.

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