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Thin layer chromatography of platinum and palladium-ammine complexes : a thesis ...

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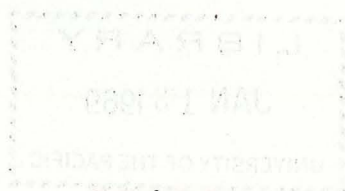
THIN LAYER CHROMATOGRAPHY OF
PLATINUM AND PALLADIUM-AMMINE COMPLEXES

A Thesis

Presented to

the Faculty of the Graduate School
of the University of the Pacific

In Partial Fulfillment
of the Requirements for the Degree
Master of Science
in the Department of Chemistry



by

Edgar Roberto Montezuma

Stockton, 1968

This thesis, written and submitted by

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is approved for recommendation to the
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Dated Sept. 23, 1968

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INTRODUCTION

Recent work in this laboratory has centered around Platinum metals and their coordination compounds. Specifically, Thin-Layer Chromatography of Platinum metal ions has been reported in one of the recent dissertations (1). The purpose of the present work was to carry out a systematic study of Thin-Layer Chromatography on the following complexes:

Tetrammine Platinum (II) Chloride	$[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$
Tetrammine Palladium (II) Chloride	$[\text{Pd}(\text{NH}_3)_4]\text{Cl}_2$
Hexammine Platinum (IV) Chloride	$[\text{Pt}(\text{NH}_3)_6]\text{Cl}_4$
Hexammine Palladium (IV) Chloride	$[\text{Pd}(\text{NH}_3)_6]\text{Cl}_4$

After the complexes had been synthesized, the main problem lay in finding a suitable adsorbent and solvent system for separating a mixture of all four complexes as well as an appropriate locating reagent, so that once the separation had been accomplished, complexes could be identified.

The Thin-Layer Chromatography of inorganic complexes has not been studied extensively. Nevertheless, a few related studies were found useful for suggestions regarding complexing and locating agents, and general reactions, predominately those from Seiler's group (2). Feigl's classic book on spot tests (3), and the B.D.H. manual (4) were

both used for suggestions regarding locating agents. A paper by Ayres and co-workers (5) concerning separation of colored platinum metal complexes and an interesting study by Hranisavljevic-Jakovljevic and Pejkovic-Tadic (6) on the separation by T-L-C of Metal Dithizonates were very useful.

Thin-Layer Chromatography bears some resemblance to paper chromatography; however it has shown considerable advantages, especially for the separation of lipophilic substances: excellent sharpness of separation; high sensitivity; great speed of development; more concentrated spots than can usually be obtained on paper; and the possibility of using more drastic reagents. On the other hand, R_F values are not so reproducible as they are in paper separations. The variations are probably attributable to the difficulty of getting an absolutely uniform and reproducible thickness in the thin-layer plates (7).

EXPERIMENTAL

PREPARATION OF COMPOUNDS

The preparation of $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$, $[\text{Pd}(\text{NH}_3)_4]\text{Cl}_2$, $[\text{Pt}(\text{NH}_3)_6]\text{Cl}_4$ and $[\text{Pd}(\text{NH}_3)_6]\text{Cl}_4$ reported here were modifications of preparations described in the literature (8).

Tetrammine Platinum (II) Chloride (Reiset's First Chloride), $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2 \cdot \text{H}_2\text{O}$, was prepared by the reaction of an aqueous solution of commercial platinum chloride (10% of PtCl_2 in 50 ml. H_2O), then 1.07 g. of solid $\text{N}_2\text{H}_4 \cdot 2\text{HCl}$ was added in small portions, so that the solution effervesced each time due to evolution of N_2 . Within five minutes of adding the last of the hydrazine salt, the deep red solution was heated on a steam bath until no further gas evolved; it was then filtered to remove the small quantity of platinum black which may deposit out. The filtrate (H_2PtCl_4) was boiled and treated with an excess of concentrated ammonia. With continuous stirring, ignoring the appearance of a green precipitate, the latter redissolved and the solution became colorless. It was evaporated until only a faint odor of NH_3 remained. Then, it was neutralized to litmus, 1 ml. of concentrated HCl added, and the mixture treated with 10 times its volume of 1:1 alcohol-acetone. It was allowed to stand for an hour; the white precipitate was removed, washed a few

times with small portions of alcohol-acetone, and rinsed with pure acetone on a suction filter. The resulting Reiset's chloride was dried in air (9).

Properties: Colorless tetragonal crystals. Solubility (20° C.) about 20g./100g. H₂O; more soluble in hot water, insoluble in alcohol, ether and acetone. Melting point: 250°C. Forms the hydrate $[\text{Pt}(\text{NH}_3)_4] \text{Cl}_2 \cdot \text{H}_2\text{O}$ on recrystallization or concentration of an aqueous solution.

Tetrammine Palladium (II) Chloride, $[\text{Pd}(\text{NH}_3)_4] \text{Cl}_2$, was prepared by treating a solution of PdCl₂ with NH₄Cl in order to precipitate as (NH₄)₂ PtCl₆ any slight Pt impurity which may be present. The filtrate was boiled with an excess of NH₃, filtered again and acidified with HCl. A yellow precipitate of pure $[\text{Pd}(\text{NH}_3)_2] \text{Cl}_2$ was formed. Then, after passing gaseous ammonia through an aqueous solution of the diammine compound at room temperature, the yellow precipitate redissolved. The solution was allowed to stand for two hours until a light yellow precipitate was formed; the precipitate was then removed, washed a few times with small portions of methanol, and rinsed on a suction filter. The resulting Tetrammine Palladium (II) Chloride (10) was dried in a desecator.

Properties: Light-yellow crystals, soluble in water, insoluble in organic solvents and decomposing at 180° C.

Hexammine Platinum (IV) Chloride, $[\text{Pt}(\text{NH}_3)_6]\text{Cl}_4$ was prepared by making a dilute, weakly acidic (HCl) solution of H_2PtCl_6 and oxidizing it with H_2O_2 . It was then reacted with an excess of NH_4Cl (at least three parts by weight of NH_4Cl to one of Pt) and slowly evaporated to dryness on a steam bath. The salt crust, mixed with the resulting residue, was broken up with a glass rod. The solids were heated on a steam bath, with continuous stirring, until the powdery mass no longer gave off the odor of HCl. The dry residue was then carefully moistened with some distilled water, taken up in cold saturated NH_4Cl solution and filtered (11). The precipitate $(\text{NH}_4)_2\text{PtCl}_6$ was reacted with $(\text{NH}_4)_2\text{CO}_3$ in 100 ml. of liquid ammonia in a round flask, provided with a stirring rod, and cooled with a dry ice bath. The reaction took place in 18 hours. The ammonia was allowed to evaporate leaving a white product. This residue was extracted with dilute NH_4OH . The remaining residue was treated with 6N HCl to form $[\text{Pt}(\text{NH}_3)_6]\text{Cl}_4$ and $[\text{Pt}(\text{NH}_3)_5\text{Cl}]\text{Cl}_3$. The separation of Hexammine Platinum (IV) Chloride was performed by fractional crystallization. The product was dried at 100°C . under vacuum (12).

Properties: White powder. Dissolves very slowly in hot water, insoluble in cold water and organic solvents. Decomposes above 340°C .

Hexammine Palladium (IV) Chloride, $[\text{Pd}(\text{NH}_3)_6]\text{Cl}_4$, was prepared by using the same technique described for Pt(IV) but

the main reaction took place in 5 hours.

Properties: Yellow powder. Insoluble in cold water and organic solvents. Soluble in hot water. Decomposes above 340° C.

APPARATUS

The instruments and apparatus used in this study included the following items:

A Warring Blender was used to mix the adsorbent (Silica Gel) with water.

The Desaga-Brinkman T.L.C. spreader, with 275 μ gate, was used for coating the 100x200 mm. standard glass plates. Desaga developing tanks with ground-glass covers were used for plate development.

Both Desaga 10 μ l and Research Specialities 2 μ l pipettes were used for plate spotting.

Drying, after developing the plates, was accomplished by means of an electrical air blast dryer.

An Sprayon Product Inc. "Jet Pak" unit of Sprayon was used for spraying locating agents.

Drying after spraying was carried out by a drying lamp.

MATERIALS

Adsorbent: E. Merck A.G. Silica Gel H was used.

Solids: The platinum and palladium metals used for preparing the complexes were obtained from K, and K. Laboratories as their chlorides $PtCl_2$ and $PdCl_2$.

Locating reagent: B. and A. Diphenylthiocarbazone (Dithizone) as 0.05% chloroform solution.

Solvents: Methanol and acetone were obtained from B.K.H. Division of Van Waters and Rogers. The absolute alcohol was obtained from Commercial Solvents Corporation. Chloroform, and Dioxane were obtained from Mathenson, Coleman and Bell. 2-Aminoethanol was obtained from Eastman Organic Chemicals. Hydrochloric acid (38.0%) was from E.I. Du Pont. Ammonium Hydroxide Technical was obtained from J.T. Baker Chemical. Fresh Demineralized water was also used.

PROCEDURE

Glass plates were thoroughly scrubbed with scouring powder and brushed under running tap water; then they were rinsed with distilled water and dried on a drying rack at room temperature. Ten 10 x 20 cm. glass plates were placed on the aligning tray, with a drop of water under each to prevent them from sliding. The left and right-hand ends of the row of plates were completed with two 5 x 20 cm. plates (end-plates) (13).

Twenty five grams of Silica Gel H were mixed with fifty milliliters of distilled water in the blender for sixty seconds. The mixture was immediately transferred to the spreader and the plates were coated with the slurry; they were allowed to dry overnight at room temperature. The edge of the plates was scraped in order to eliminate the so-called "edge-effects" (14). Using the Desaga Labeling Template and a stylus, a line was scratched on the Layer 100 mm. from the point where the spots were to be applied. One microliter spots of the 0.1% aqueous solutions of the compounds and the mixture were then applied by means of a micropipette, keeping the spot size down to 20 mm². Developing tanks were fitted with filter paper to cover the back of the tank. The solvent system was placed in the tank and the tank allowed to come to equilibrium for at least an hour. The spotted thin-layer plates were then developed in the tank.

When the solvent front reached the scratched line, the plates were withdrawn from the tank and dried with the air-blast dryer. The locating agent was then sprayed on (15). The plates were then dried, by means of the drying lamp. The chromatograms were recorded by using a Xerox Copier(16).

PRELIMINARY RESULTS

Preliminary studies were carried out to determine which adsorbent and solvent gave the best results and were the most convenient to use.

In Thin-Layer Chromatography, a system of at least three components is used — adsorbent, solvent and the compound being chromatographed. The behavior of the compound is dependent upon both the adsorbent and the solvent (17). According to Stahl (18), it is now possible to predict, within certain limits, which adsorbents and which solvents will be suitable for the chromatography of a given substance. Thus, the best preliminary estimate is that for separating polar substances is that it would be best to use a polar solvent in conjunction with a less active adsorbent.

A number of lists have been published in which adsorbents are ranked in order of activity, (19) and solvents according to their "eluting power" (20).

Because of adsorbents, the choice is usually narrowed down to what is commercially available or has been found to function before, it was decided to try Silica Gel H as the layered adsorbent (21).

Since an inactive plate was being used to separate polar compounds, the more polar solvent in any eluotropic series

would give the greatest chance of success. The more polar solvents there consist of: dioxane, acetone, alcohols of decreasing carbon chain length, and water. So an attempt at separation was made by using each of these solvents in turn, and the chromatograms which resulted are shown in the eluotropic series Figures: Fig. 1 (n-propanol), Fig. 2 (ethanol), Fig. 3 (methanol), Fig. 4 (acetone), Fig. 5 (dioxane), and Fig. 6 (water).

The immediate objective was not to find a solvent that would accomplish the separation at all, but rather to find one that would move the compounds from the spotting points, and showing some differentiation in degree of transport. Parenthetically, the search for a good solvent was paralleled by a search for locating agents; (these results are discussed later). The results, with most of the solvents used, showed poor separation and poor differentiation between spots, but with water as solvent the results appeared promising, so it was decided that water should serve as the major component in a solvent system. Thus other solvents or reagents would be added to it to enhance both differentiation between spots and separation of the mixture into its components.

Since the layered adsorbent had been selected, it was necessary to fix the activity of the layer using a standard method of preparation. The use of a high activity plate, as suggested by Takitani (22), was not appropriate for this

study because it was very difficult to control all of the variables involved in order to produce thin-layer plates of uniform activity for the reproducibility of R_F values.

It was decided to use plates that were less active.

Seiler (23) suggested the use of inactive plates when separating polar substances with polar solvent systems, as in this case. In order to promote both low activity and uniformity, then, it became standard practice to allow the plates to air dry over-night, exposed to the humidity of the laboratory atmosphere. Under these conditions the changes in thin-layer activity due to daily changes in relative humidity were practically negligible.

VARIATION OF WATER

The first aqueous solvent systems were carried out in order to determine whether the compounds were affected by the presence of acid or basic solvent.

When water --- aqueous ammonia (99:1) was used as the solvent system no differentiation or separation resulted at all, as shown in Figure 7.

Results with water --- HCl (99:1) showed the degree of transport of the spots were increased, as shown in Figure 8. It will be seen that all R_F values were the same and that the equimolecular mixture of all four of the compounds were not separated, but travelled as one spot. The immediate objective, then, was to find a new component for the solvent system that would depress to some extent the R_F values of the spots. It was decided that the addition of a basic compound to the water-HCl system might cause some or all of the ammine compounds to form less polar complexes, thus depressing their R_F values. The choice of ethanolamine as a complexing agent was made, on the basis of convenience, to cause changes in solutes, as suggested by Thoma (21). Accordingly, a water-HCl-Ethanolamine series was undertaken, with the following volume-to-volume ratios:

Water	98	96	94	92	90
HCl conc.	1	2	3	4	5
Ethanolamine tech.	1	2	3	4	5

The results of the series are shown in Figures 9 through 13. The series as a whole showed that the presence of ethanolamine did decrease the R_F values, as expected. Moreover, there was good differentiation and even some separation of the equimolecular mixture, but the presence of tails on three of the spots offered some disadvantage.

It was of passing interest to note that as the concentration of ethanolamine increased, R_F values also increased and the Platinum (IV) spot became less visible. Also it was noted that the pH of the solvent system had to be kept as neutral because in both acid or basic media a second solvent front was present, and all the spots were located on it.

If "tails" could be eliminated from the spots, the solvent system reported for Figure 10 would present a possibility of detection and separation of the mixture of the compounds in a very well defined area.

According to Lederer (24) the tailing of spots can be attributed to an equilibrium between the ionic form in which the compounds have been applied, and complex formation with the solvent employed. It is usually avoided or minimized by

the incorporation of a strong acid in the developing solution, but the present solvent system already has a strong acid, so this suggestion was eliminated. Another suggestion to eliminate tailing from the spots caused by a fast solvent flow or adsorption effects was hinted by Bock (25), MacFarren (26), and Munier (27): a) The incorporation of a compound into the solvent system which would reduce the velocity of the solvent flow and b) by mixing the adsorbent with an ionic salt such as Sodium Chloride; in many cases this procedure has been found not only to reduce tailing but to improve separation by the radical alteration of R_F values within a given solvent system.

To reduce the velocity of the solvent flow, it was decided to use an alcohol as the new compound to associate with water of the solvent system, by means of a hydrogen bonding. As a matter of convenience, Methanol was chosen for this purpose.

METHANOL VARIATIONS IN THE SOLVENT SYSTEM

In order to obtain reproducible results, it was advisable to standardize the mixture water-HCl by preparing a 0.5N HCl solution. A series of runs was thus instituted using 0.5N HCl - Methanol - Ethanolamine with the following ratios (V/v):

HCl 0.5N	90	80	70	60	50	40	30	20	10	0
Methanol	10	20	30	40	50	60	70	80	90	100
Ethanolamine	2.0	2.0	1.75	1.5	1.5	1.5	1	1	1	1

The results of the series are shown on Figures 14 through 23.

The use of Methanol in the solvent system was a success. The best results of the Methanol variation in the solvent system series corresponded to 10 ml., 20 ml., and 30 ml. of the Methanol ratio. In Table I these results are summarized.

To this point in the study, duplication of experiments was not a common practice, but as better and better solvent system proportions were found, duplications of runs became standard procedure, not only to check on accuracy of results, but also to continue the search for good locating agents.

TABLE I
 VARIATION OF R_F VALUES
 WITH METHANOL CONCENTRATIONS

HCl 0.5N		R_F values of Platinum and Palladium			
Methanol		Ammine Compounds			
Ethanolamine					
(ml/ml/ml)	Pt (II)	Pd(II)	Pt(IV)	Pd(IV)	
100/0/2	0.7	0.6	0.85	0.68	
90/10/2	0.71	0.52	0.79	0.6	
80/20/2	0.62	0.5	0.74	0.53	
70/30/1.75	0.61	0.54	0.75	0.58	
60/40/1.5	0.45	0.44	0.61	0.48	
50/50/1.5	0.47	0.52	0.65	0.55	
40/60/1.5	0.40	0.47	0.63	0.51	
30/70/1	0.32	0.45	0.58	0.48	
20/80/1	0.20	0.33	0.46	0.33	
10/90/1	0.12	0.22	0.48	0.26	
1/100/1	0.10	0.18	-----	0.20	

*Note: The R_F values were measured at
 the top of each spot.

LOCATING AGENTS

During the preliminary studies and in the attempts to find the best solvent system, it became necessary to institute a search for locating agents that would impart a characteristic color to each of the different platinum and palladium-complexes. A number of suggestions from books and articles (5) were followed. Of special interest was the article of Waldi (15), but it soon became clear that none of these results could be expected within this study. Some complexing agents were tried as locating agents, with no success. Success was achieved by using Diphenylthiocarbazone, usually abbreviated to "dithizone", as suggested by Nagal (28), by preparing 0.05% solution in chloroform and spraying it on the developed plates.

After chromatography, the plates were freed from solvent by blowing with warm air and then sprayed with Dithizone solution. The surface of the plate became purple, and three of the spots immediately stood out, showing bright colors.

Pd (II) was greenish-red, Pt (IV) was yellow, and Pd (IV) was greenish-blue. Pt (II) was not detectable, but when the plate was dried by means of an infra red drying lamp for a period of 5 minutes at 120° C., Pt(II) became visible as a tan colored spot and the purple color of the plate vanished.

This resulted in the establishment of a locating reagent for the separation of Platinum-and Palladium Ammine compounds.

Since the layered adsorbent had been found and the solvent system for separating the mixture of the four Pt-and Pd- Ammine Complexes, as well as the appropriate locating reagent had been determined, the successful separation and identification of Tetrammine Pt (II) Chloride, Tetrammine Pd (II) Chloride, Hexammine Pt (IV) Chloride and Hexammine Pd (IV) Chloride now seemed assured.

FINAL RESULTS

AND

CONCLUSIONS

Of those solvent systems tested, the best one for the separation of the studied compounds was the 0.5N HCl - Methanol - Ethanolamine (80:20:2) (v:v:v). In order to investigate the possibility of a new solvent system, which could be substituted for methanol, it seemed appropriate to carry out some tests using dioxane, acetone, and ethanol instead of methanol, but in the same proportions. The results with acetone were a success. Therefore, a new solvent system was found:

HCl 0.5N - Acetone - Ethanolamine (80:20:2)

The final chromatograms, presented in Figure 24 (Methanol System), Figure 25 (Acetone System) showed: Tetrammine Pt (II) Chloride, Tetrammine Pd (II) Chloride, Hexammine Pt (IV) Chloride, and Hexammine Pd (IV) Chloride could be separated and identified in either of the two chromatograms.

The migration sequence found was the same for the two systems: Pt (IV) > Pd (II) > Pt (IV) > Pt (II). The developing time variation was: Acetone system < Methanol system.

All of the final results are summarized in Table II.

Merck Silica Gel H was used as the Layer Adsorbend and thin-layer plates were air dried in the laboratory atmosphere, producing plates of a low activity.

TABLE II
FINAL RESULTS

Adsorbent: Silica Gel H

Locating reagent: Dithizone

Solvent System	Developing Time	R_F Values			
		Pt(II)	Pd(II)	Pt(IV)	Pd(IV)
HCl 0.5N 80 ml.					
Methanol 20 ml.	25 min.	0.62	0.41	0.82	0.48
Ethanolamine 2 ml.					
HCl 0.5N 80 ml.					
Acetone 20 ml.	45 min.	0.69	0.52	0.83	0.60
Ethanolamine 2 ml.					

A series of experiments, using different solvents, fixed water as the major solvent component. Further attempts showed that HCl added to the water, increased R_F values, and that ethanolamine added to water decreased them. It was found that a combination of all three components gave good differentiation of R_F values, but with tailing effects. In order to eliminate the tailing, methanol or acetone was added to the solvent system, using various volume-to-volume ratios, showed that maximum resolution of the mixture resulted with an 80:20:2 ratio of: HCl 0.5N -- Methanol -- Ethanolamine, as

well as an Acetone system offering two solvent systems. The Methanol system showed a better separation between spots than the Acetone system.

Dithizone was used as a locating reagent on a 0.05% chloroform solution. Colors obtained with the Pt and Pd complexes were well characterised after drying the plates.

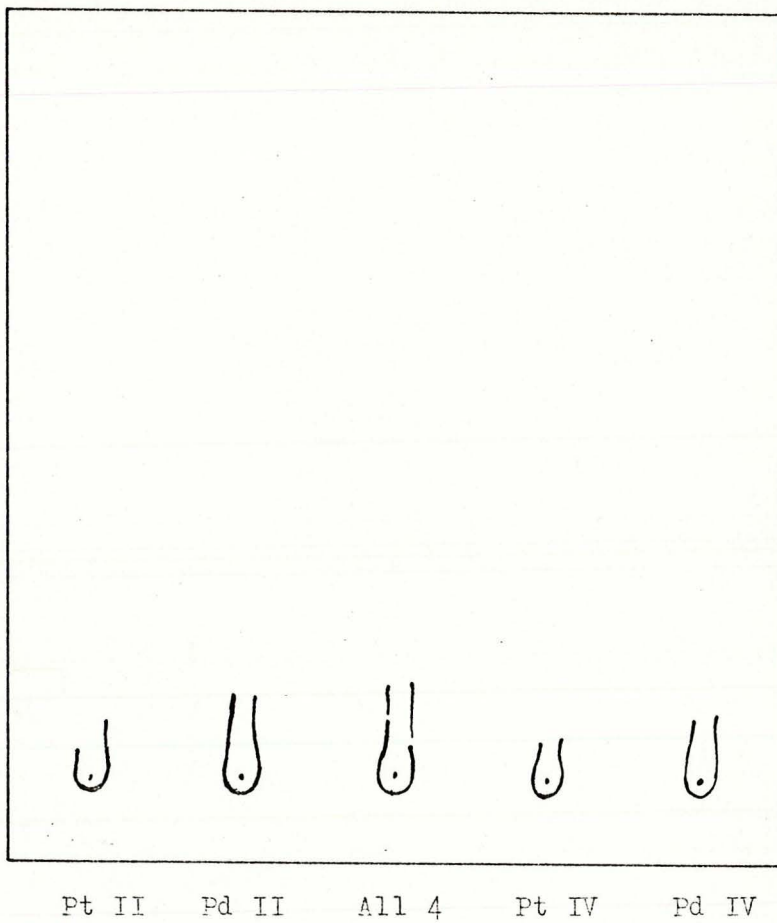
The sequences of R_F values is:



The separation and identification of $[\text{Pt}(\text{NH}_3)_4] \text{Cl}_2$, $[\text{Pd}(\text{NH}_3)_4] \text{Cl}_2$, $[\text{Pt}(\text{NH}_3)_6] \text{Cl}_4$, and $[\text{Pd}(\text{NH}_3)_6] \text{Cl}_4$ by Thin-Layer Chromatography was studied, each prepared as 0.1% aqueous solutions. The mixture to be separated from equal aliquots of these solutions.

ELUOTROPIC SERIES I

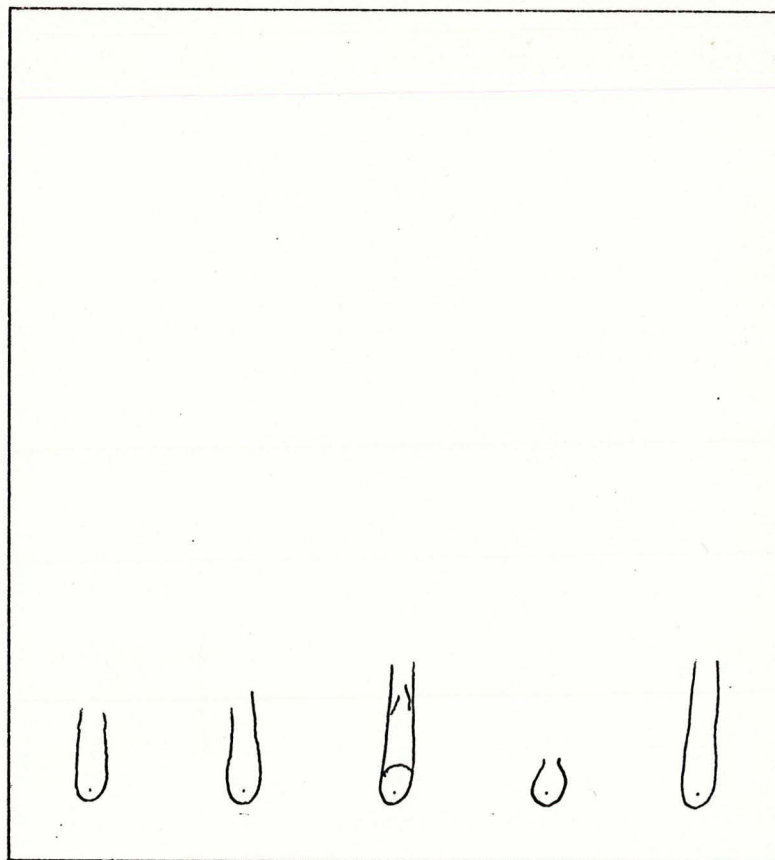
FIGURE I



Layer: Silica Gel H
Solvent: n-Propanol
Loading: 1 ul Aqueous Solution
Locating Agent: Dithizone
Time: 1 Hour

ELUOTROPIC SERIES II

FIGURE 2



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

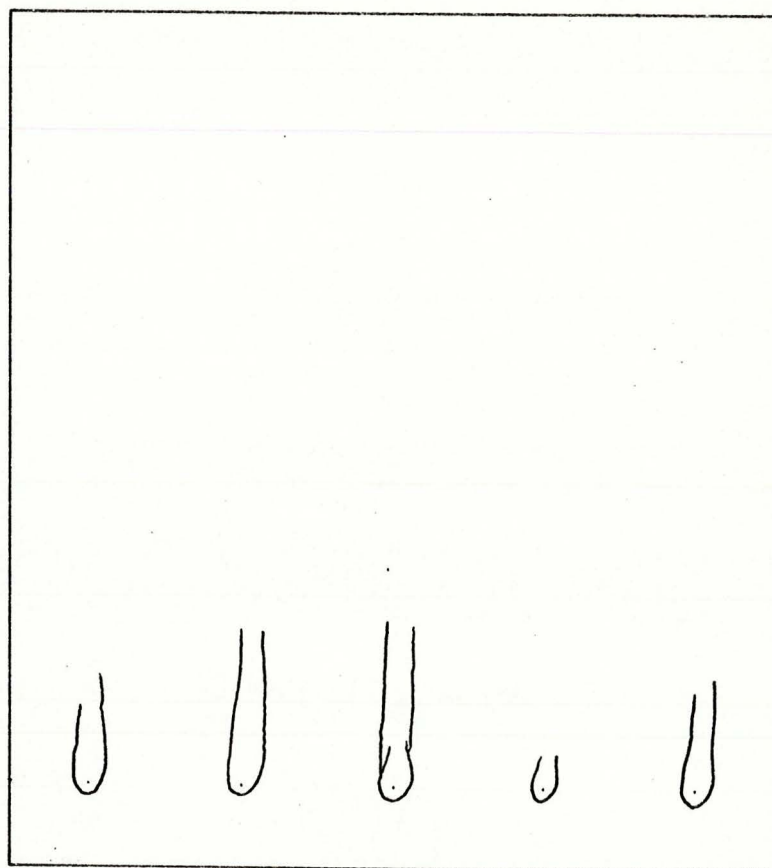
Solvent: Ethanol

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 32 Minutes

FIGURE 3



Pt II

Pd II

All 4

Pt IV

Pd IV

Layer: Silica Gel H

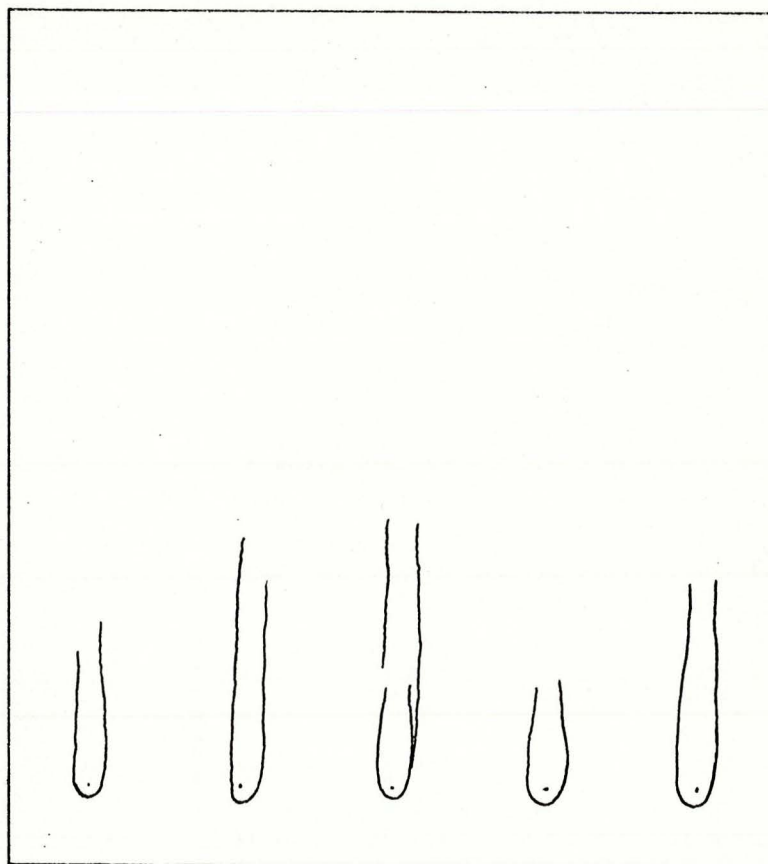
Solvent: Methanol

Loading: 1- μ l Aqu. : Solution

Locating Agent: Dithizone

Time: 14 Minutes

FIGURE 4



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

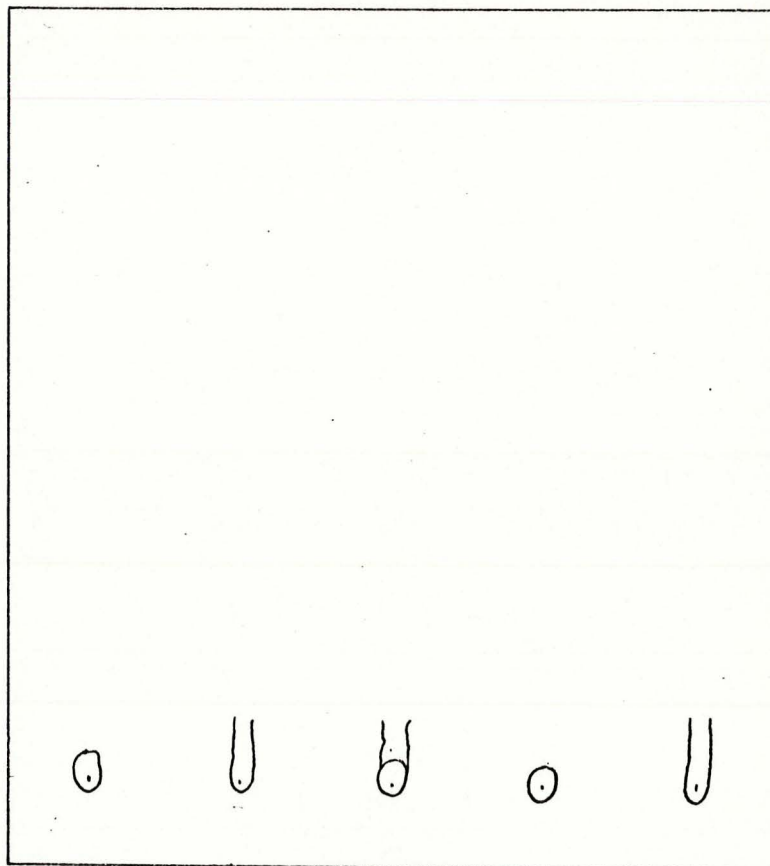
Solvent: Acetone

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 12 Minutes

FIGURE 5



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

Solvent: Dioxane

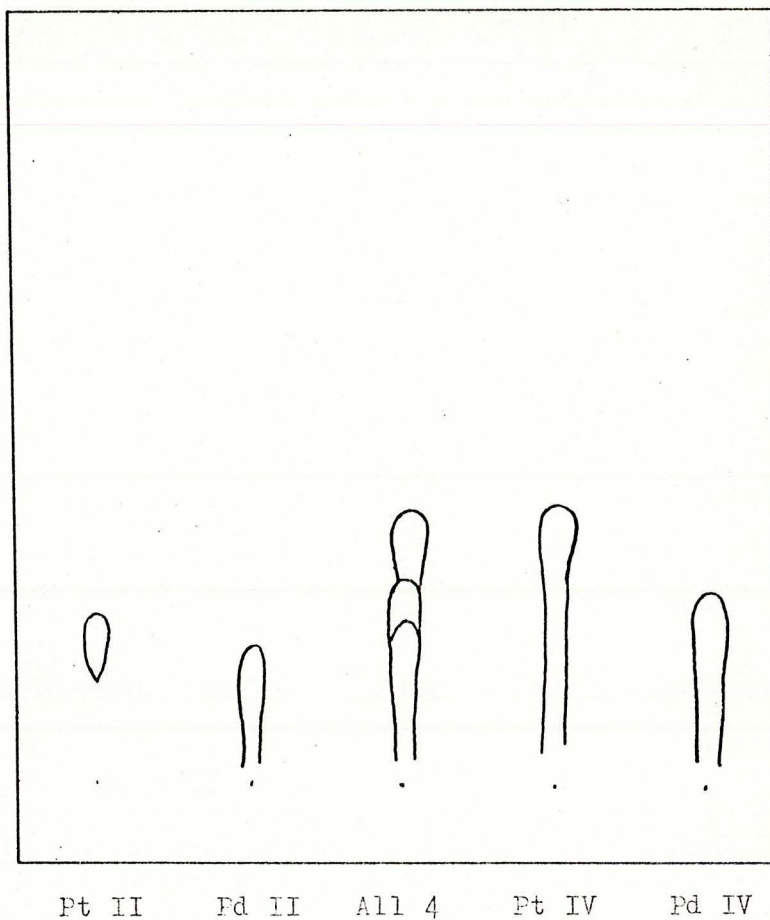
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 24 Minutes

ELUOTROPIC SERIES VI

FIGURE 6



Layer: Silica Gel H

Solvent: Water

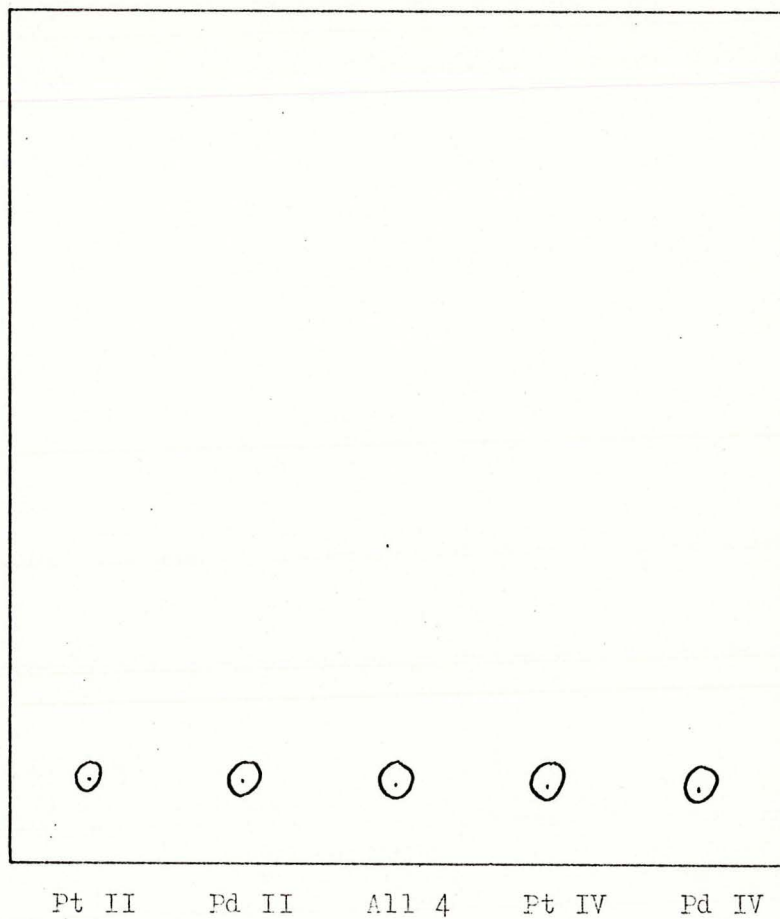
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 10 Minutes

VARIATION OF WATER I

FIGURE 7



Layer: Silica Gel H

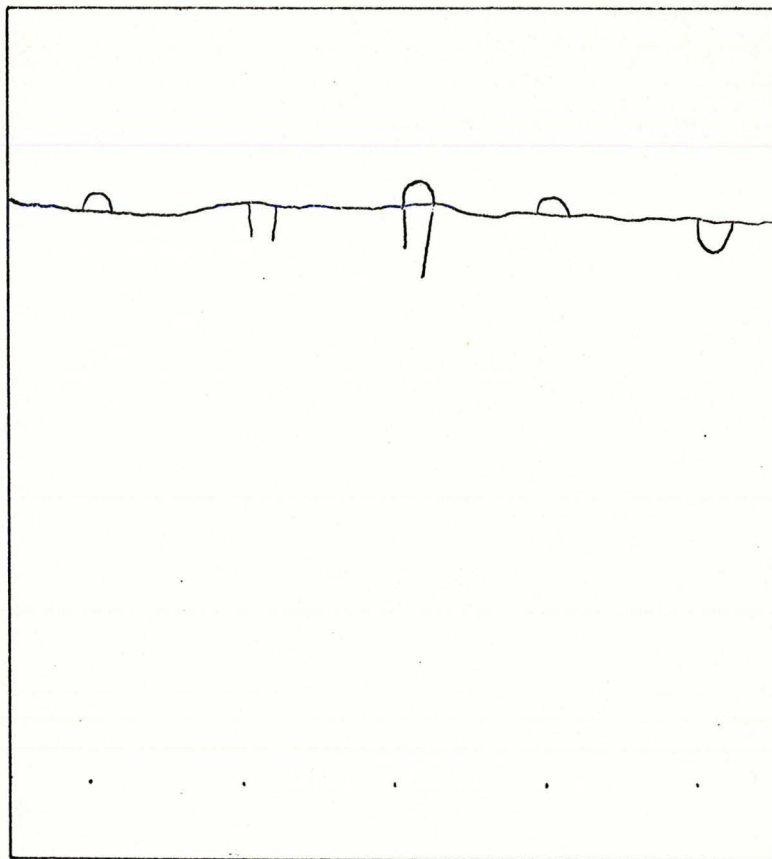
Solvent System: Water-NH₄OH tech. (99:1 v/v)

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 12 Minutes

FIGURE 8



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

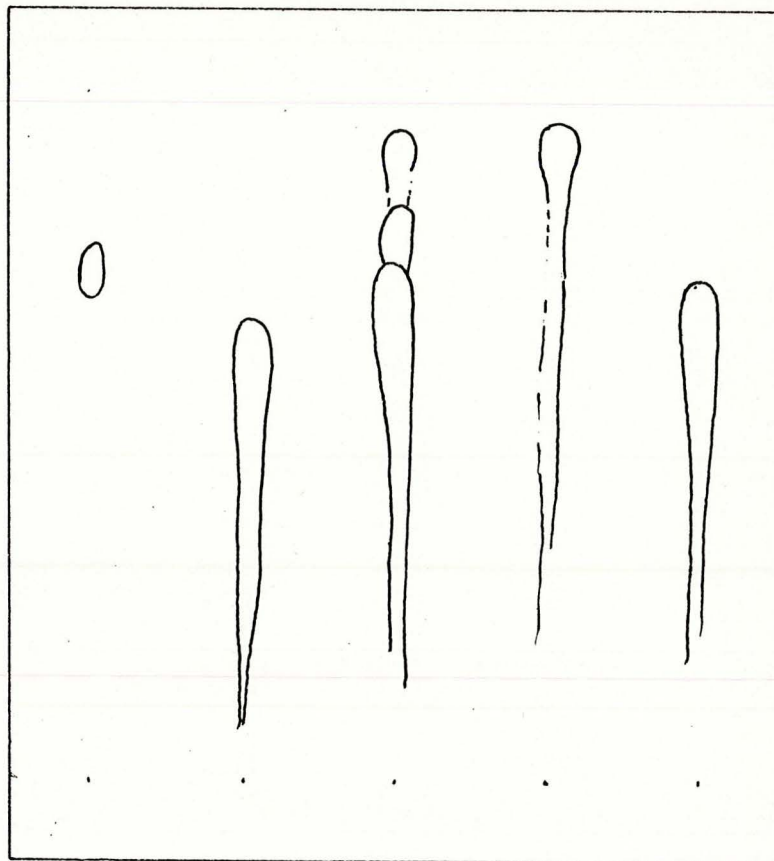
Solvent System: Water-HCl conc. (99:1 v/v)

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 12 Minutes

FIGURE 9



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

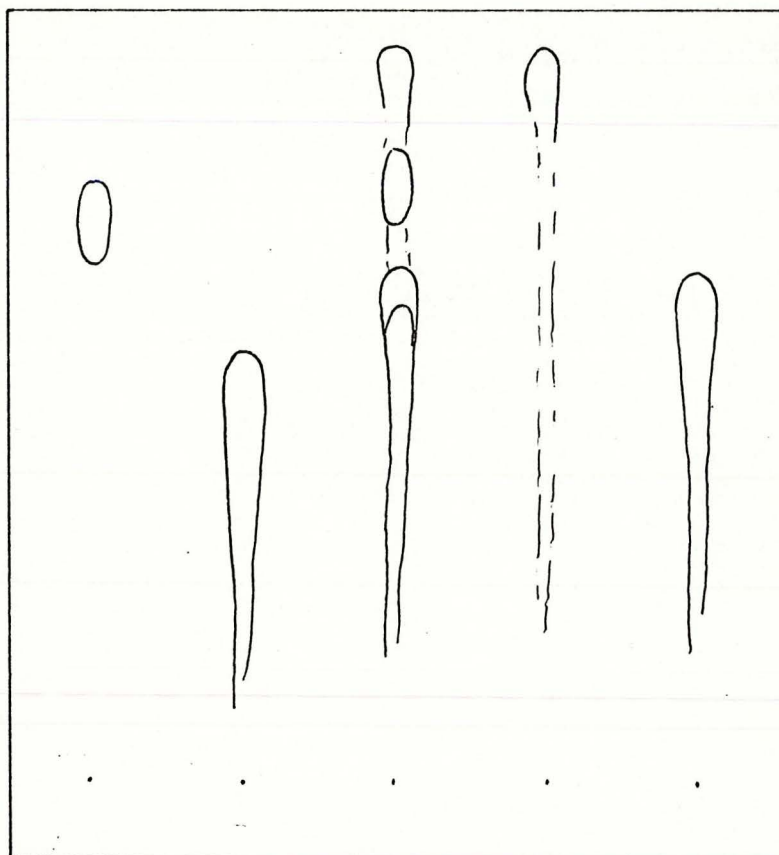
Solvent System: Water-HCl conc.-Ethanolamine (98:1:1)

Loading: 1 ul Aqu. ; Solution

Locating Agent: Dithizone

Time: 10 Minutes

FIGURE 10



Pt II

Pd II

All 4

Pt IV

Pd IV

Layer: Silica Gel H

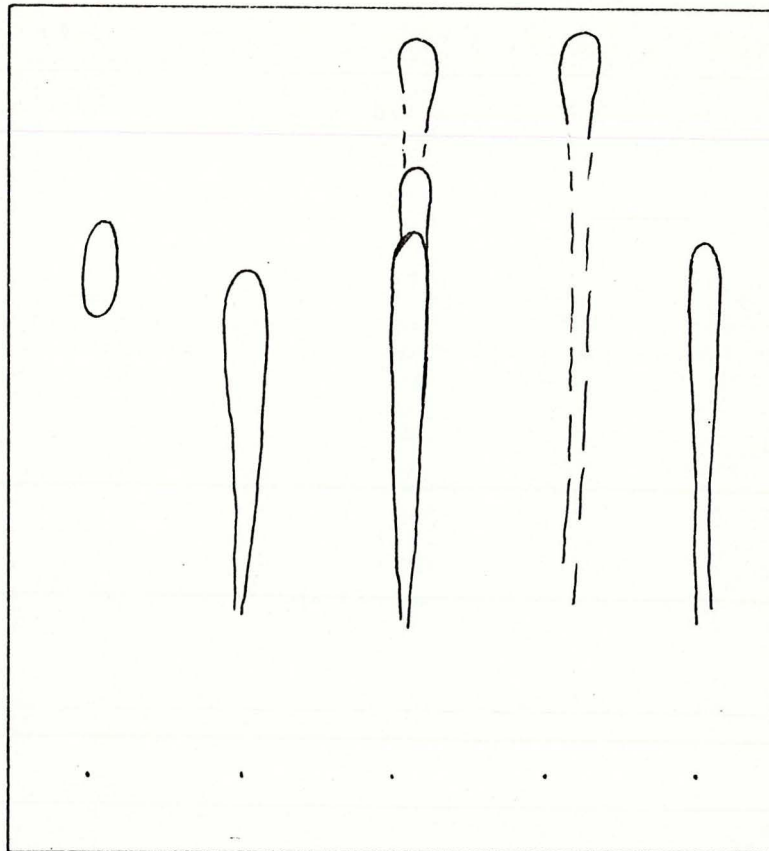
Solvent System: Water-HCl conc.-Ethanolamine (96:2:2)

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 14 Minutes

FIGURE 11



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

Solvent System: Water-HCl conc.-Ethanamine (94:3:3)

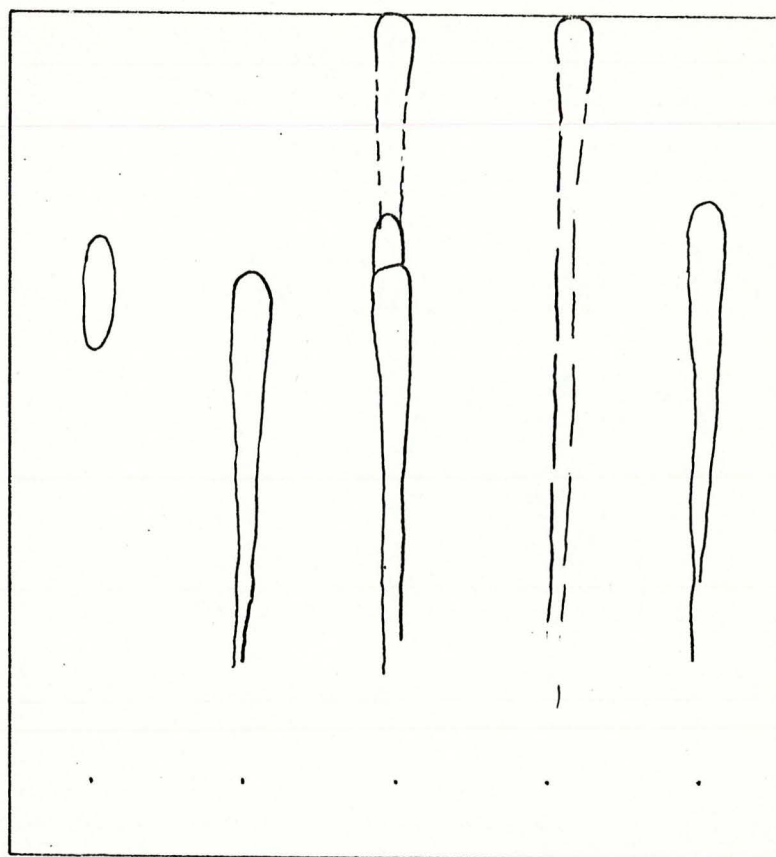
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 18 Minutes

VARIATION OF WATER VI

FIGURE 12



Pt II

Pd II

All 4

Pt II

Pd II

Layer: Silica Gel H

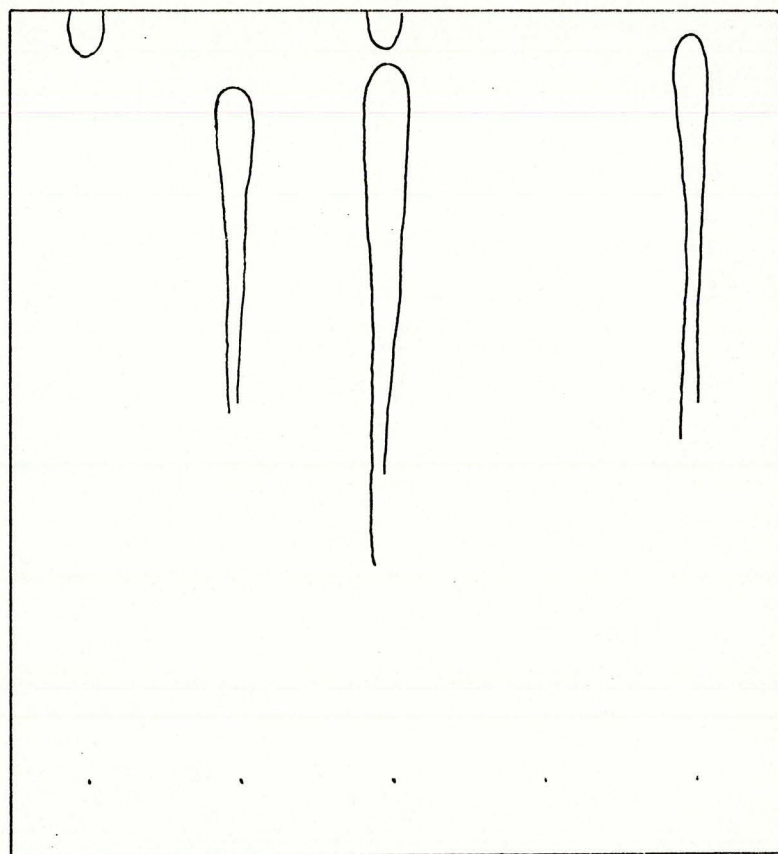
Solvent System: Water-HCl conc.-Ethanolamine (92:4:4)

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 20 Minutes

FIGURE 13



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

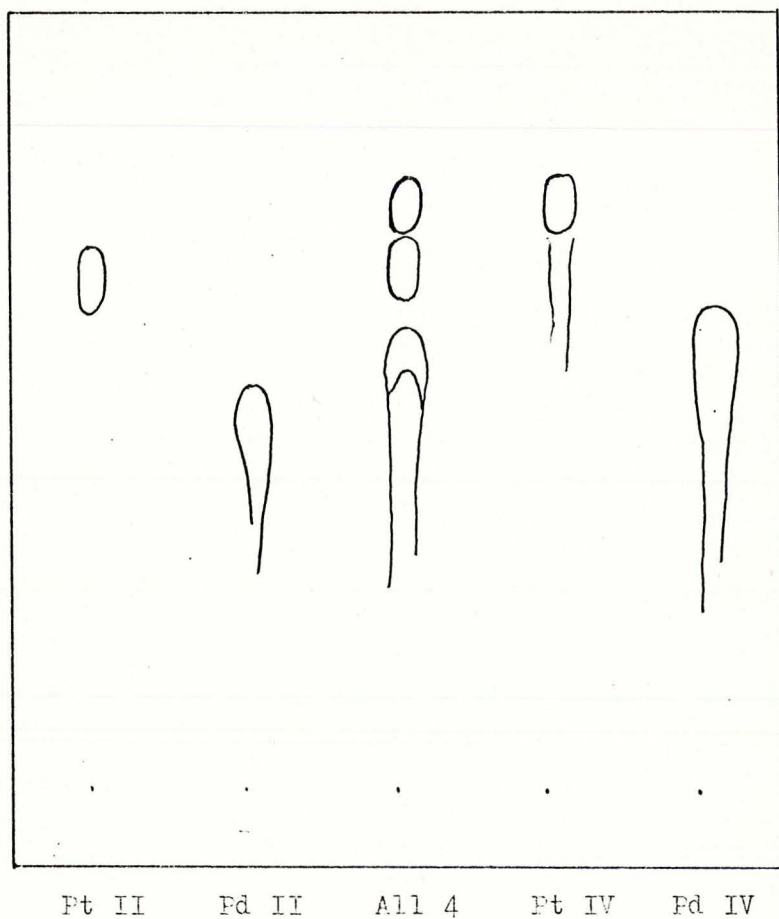
Solvent System: Water-HCl conc.-Ethanolamine (90:5:5)

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 28 Minutes

FIGURE 14



Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine(90:10:2)

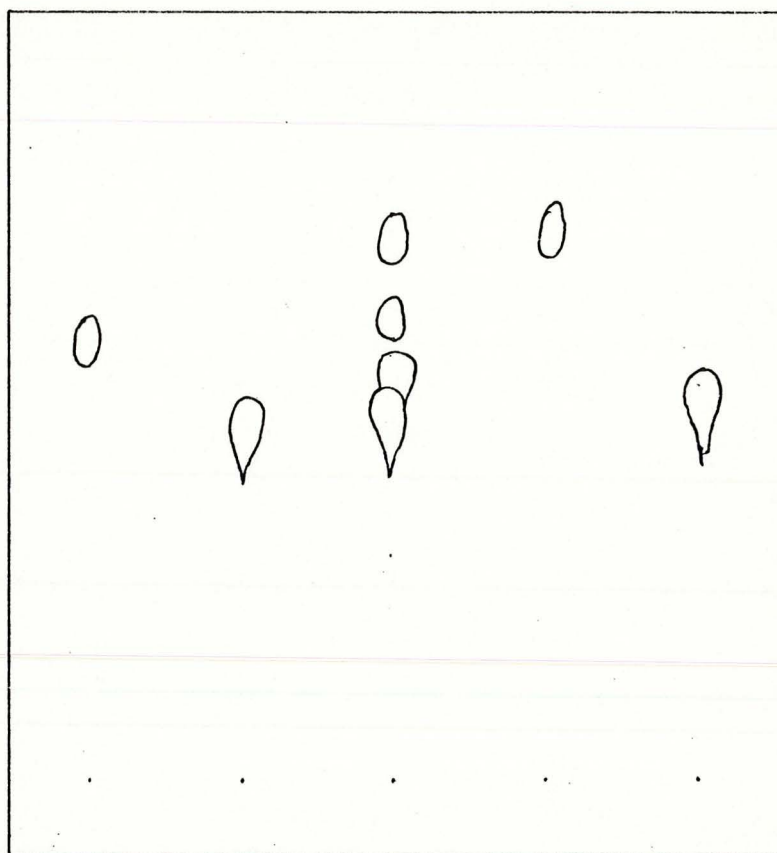
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 24 Minutes

pH 7

FIGURE 15



Pt II

Pd II

All 4

Pt IV

Pd IV

Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine (80:20:2)

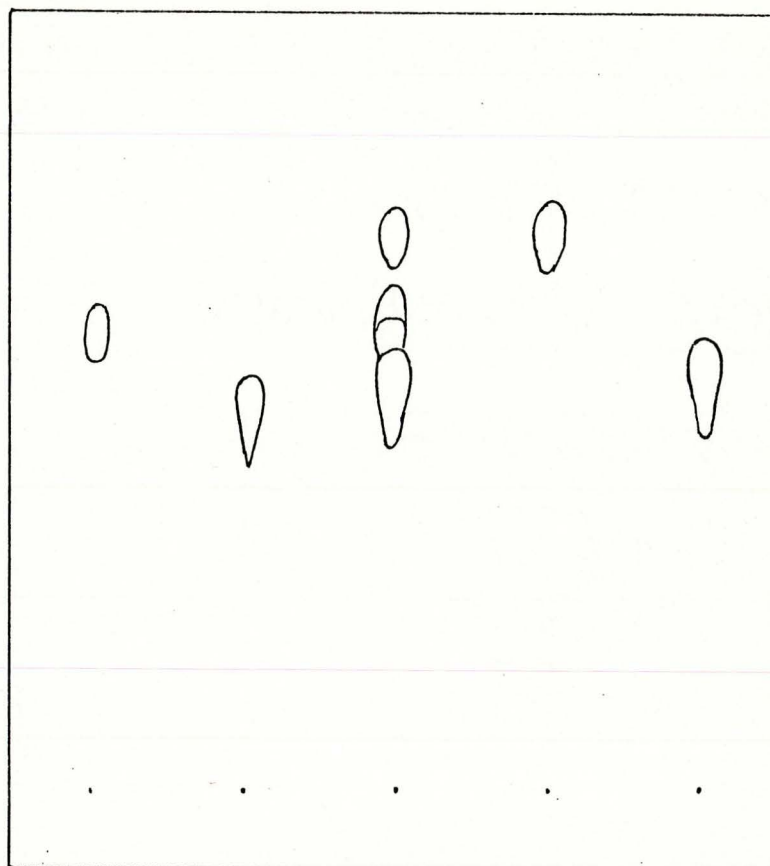
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 35 Minutes

pH = 7

FIGURE 16



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine (70:30:1.75)

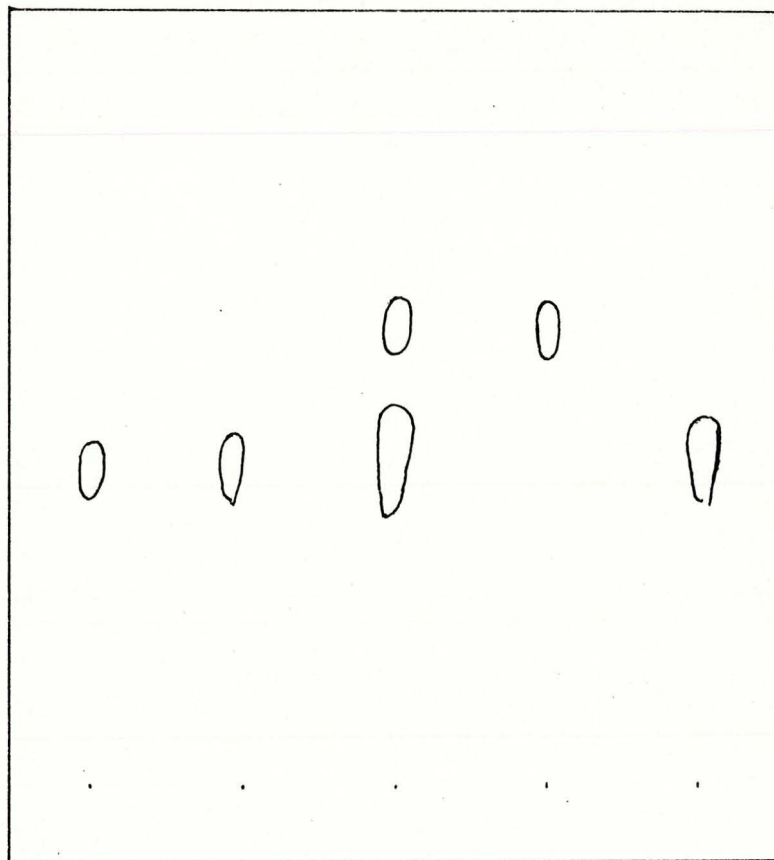
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 43 Minutes

pH = 7

FIGURE 17



Pt II

Pd II

All 4

PtIV

Pd IV

Layer: Silica Gel H

Solvent System: 0.5M HCl-Methanol-Ethanolamine (60:40:1.5)

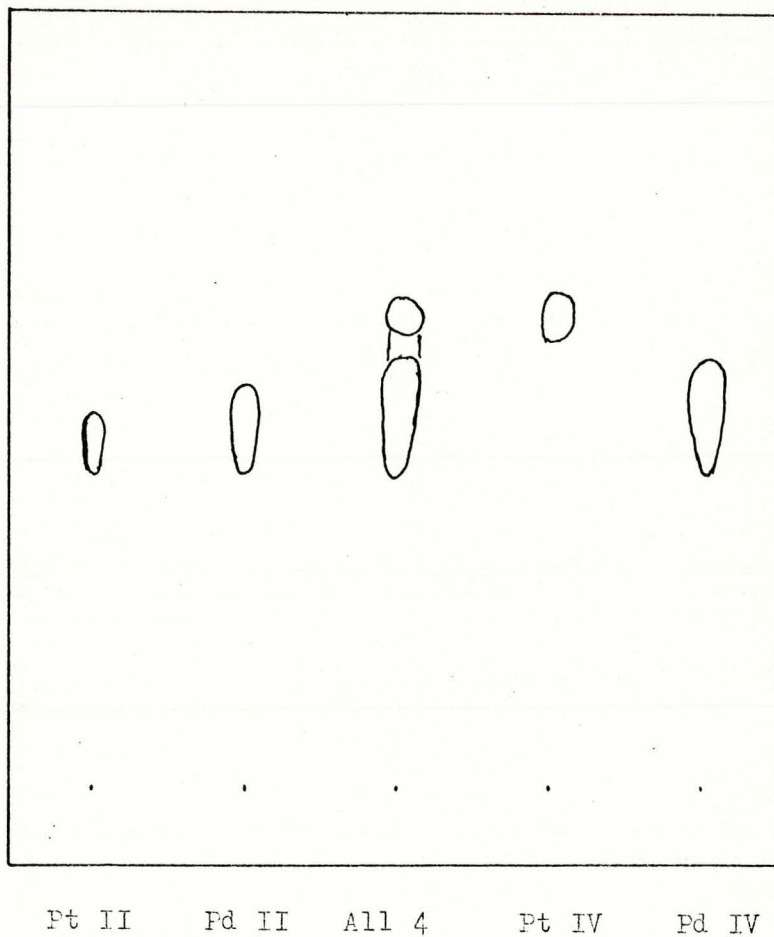
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 48 Minutes

pH = 7

FIGURE 18



Layer: Silica Gel H

Solvent System: 0.5M HCl-Methanol-Ethanolamine (50:50:1.5)

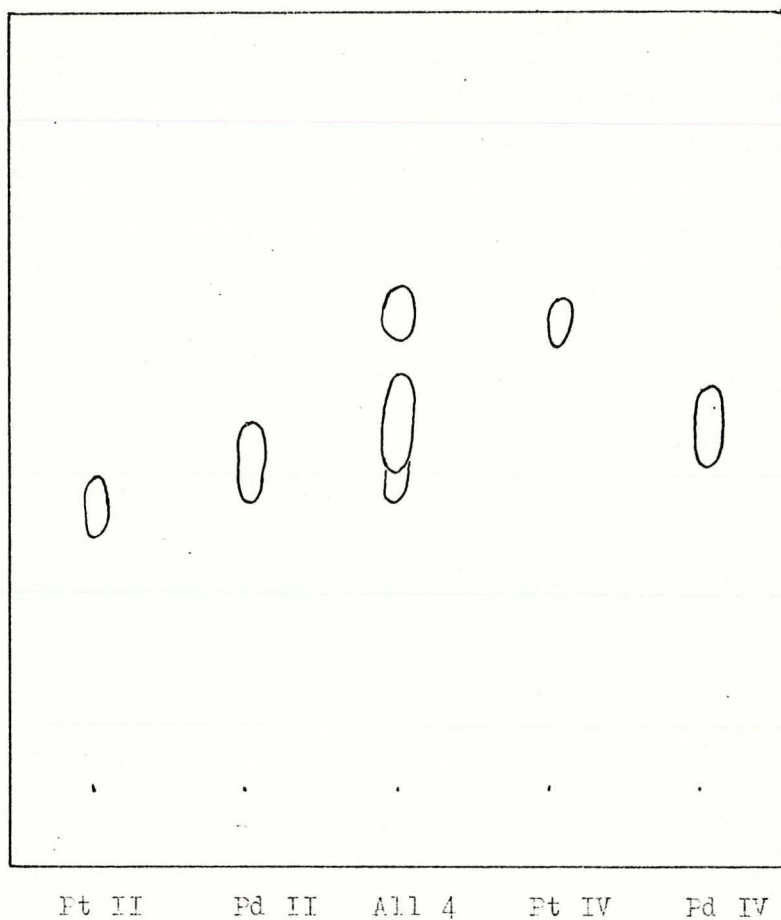
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 53 Minutes

pH = 7

FIGURE 19



Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine (40:60:1.5)

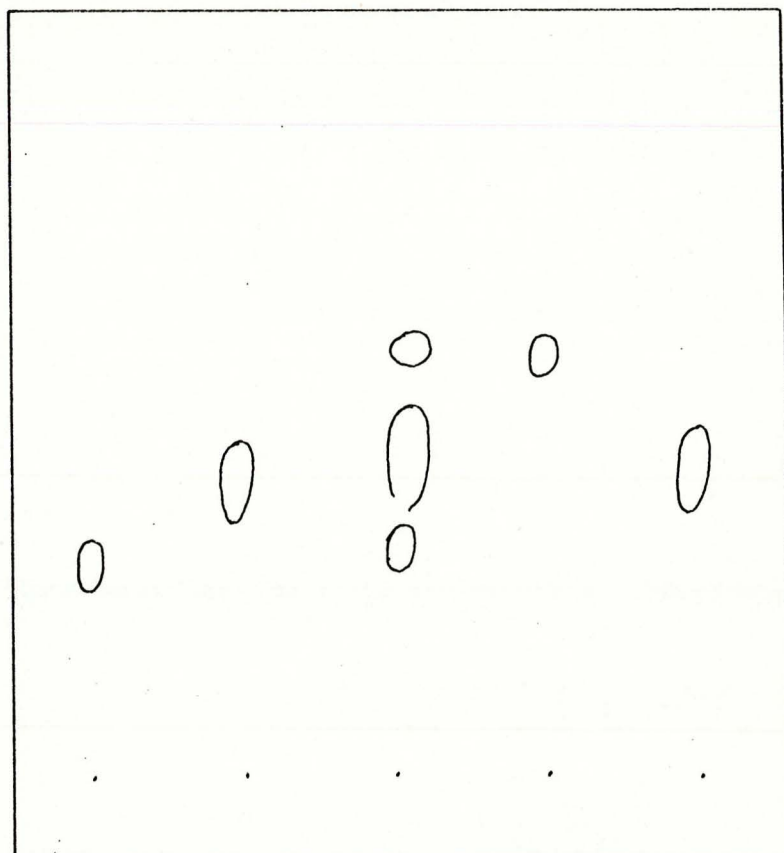
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 52 Minutes

pH = 7

FIGURE 20



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine (30:70:1)

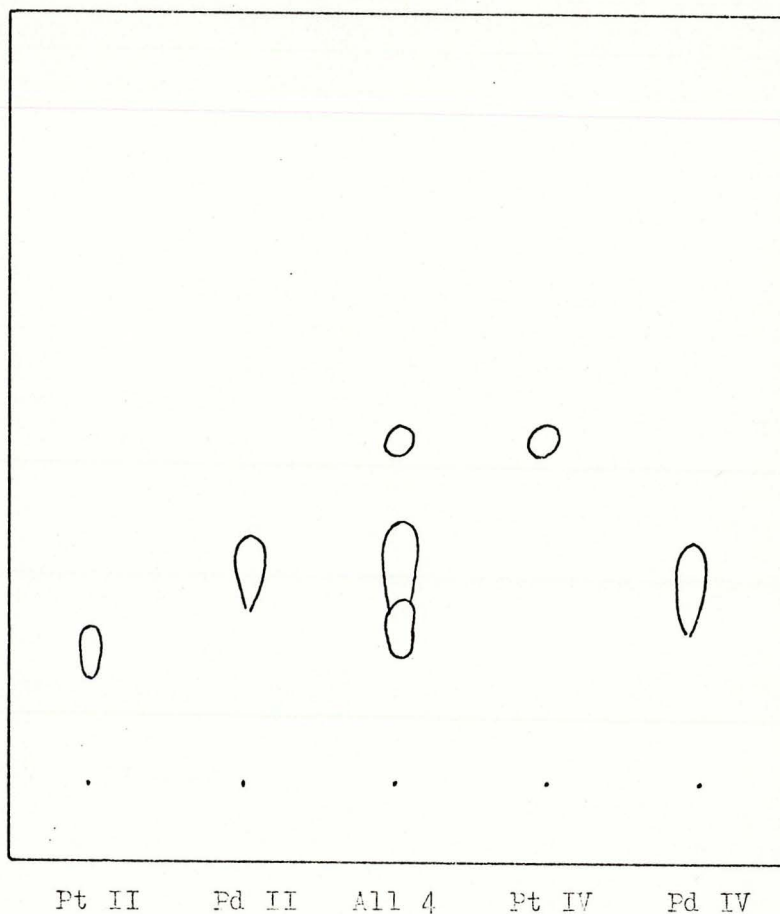
Loading: 1 ul Aqu. Solution

Time: 48 Minutes

pH = 7

Locating Agent: Dithizone

FIGURE 21



Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine (20:80:1)

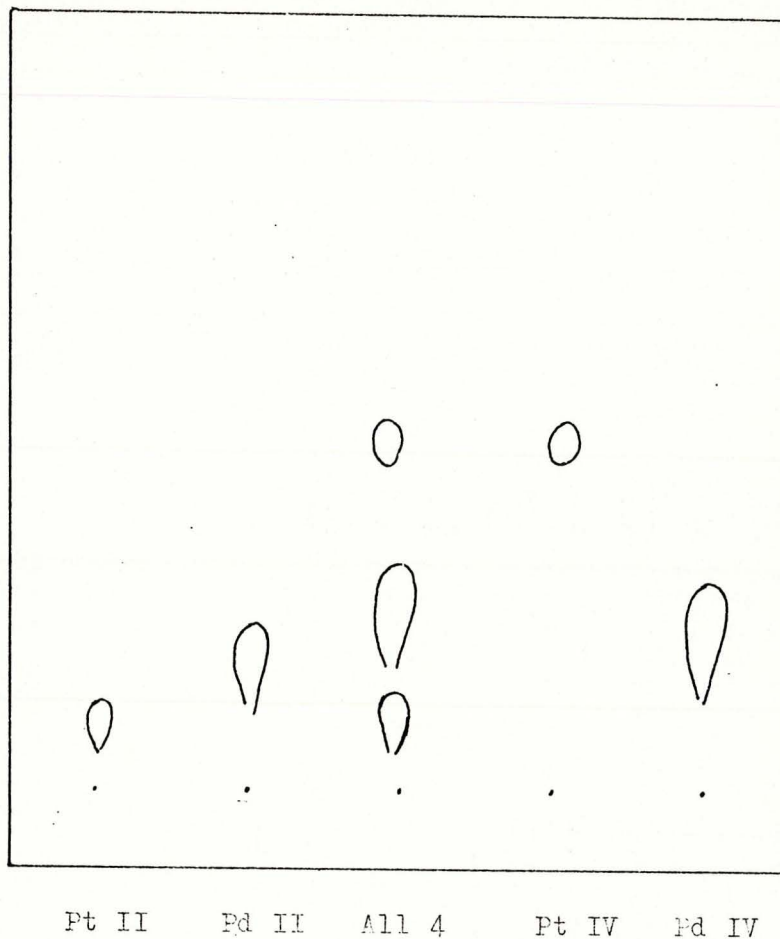
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 43 Minutes

pH = 7

FIGURE 22



Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine (10:90:1)

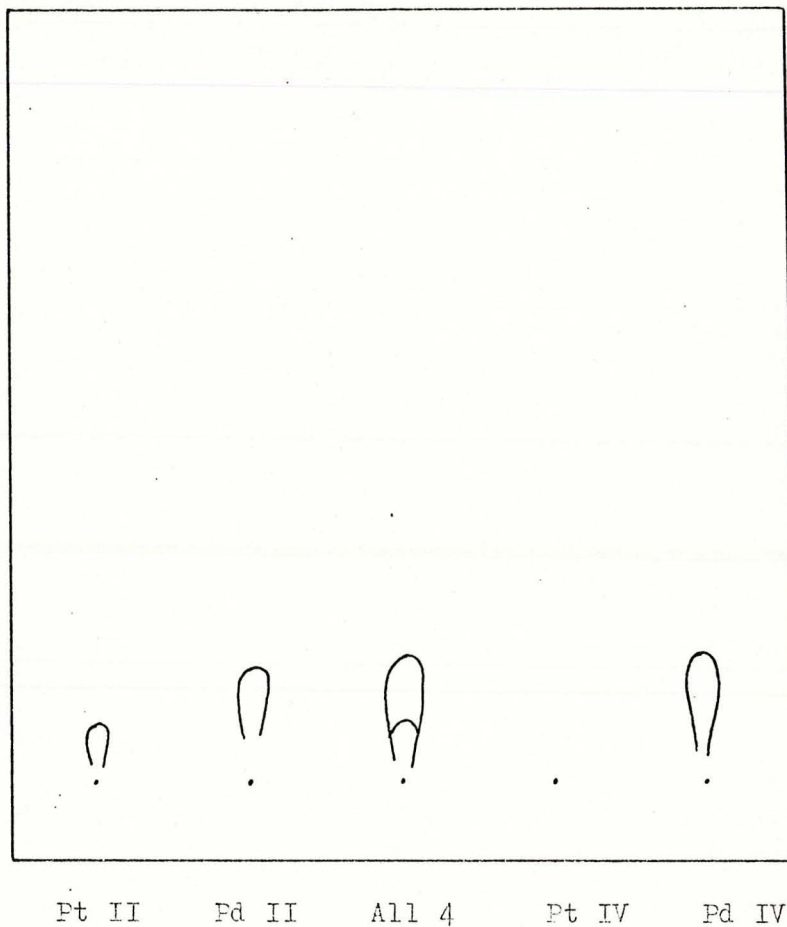
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 45 Minutes

pH = 7

FIGURE 23



Layer: Silica Gel H

Solvent System: HCl conc.-Methanol-Ethanolamine(0.1:100:1)

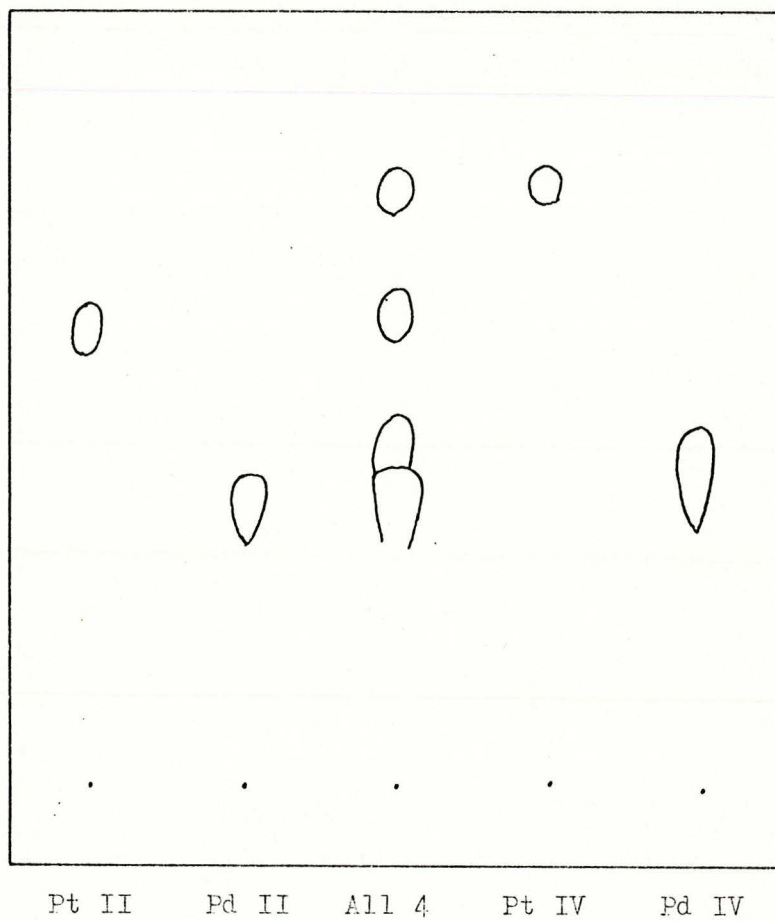
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 35 Minutes

pH = 7

FIGURE 24



Layer: Silica Gel H

Solvent System: 0.5N HCl-Methanol-Ethanolamine(80:20:2)

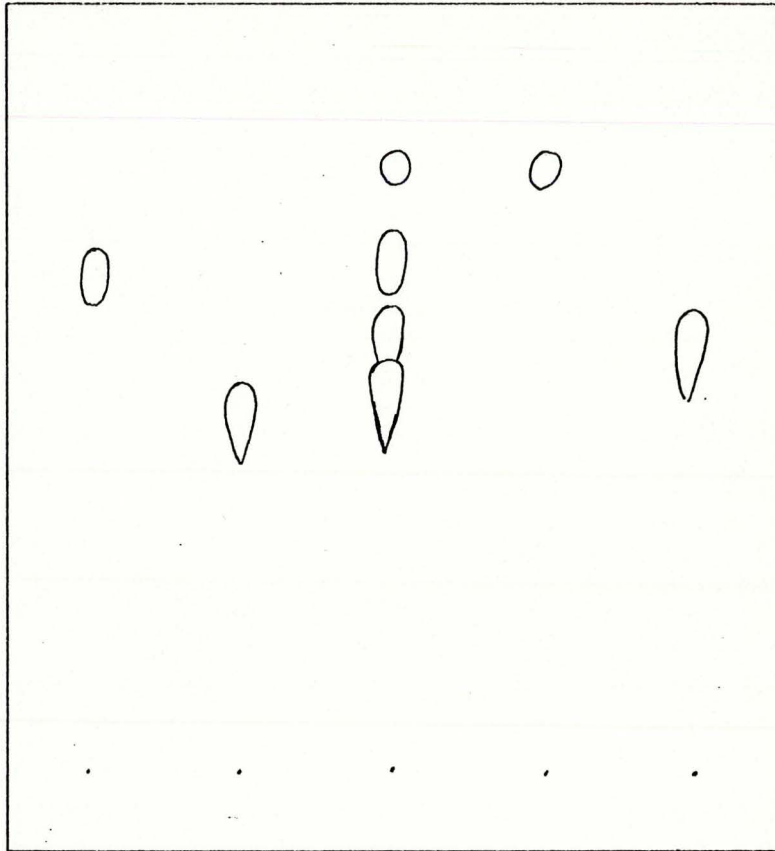
Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 35 Minutes

pH = 7

FIGURE 25



Pt II Pd II All 4 Pt IV Pd IV

Layer: Silica Gel H

Solvent System: 0.5N HCl-Acetone-Ethanolamine(80:20:2)

Loading: 1 ul Aqu. Solution

Locating Agent: Dithizone

Time: 45 Minutes

pH = 7

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