Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

Inorganic Analysis by Combined Thin-Layer Chromatography and Ring-Oven Technique Incorporating Fluorescent Support

K. N. Johri[a]; H. C. Mehra[a]
[a] DEPARTMENT OF CHEMISTRY, UNIVERSITY OF DELHI, DELHI, INDIA

To cite this Article Johri, K. N. and Mehra, H. C.(1971) 'Inorganic Analysis by Combined Thin-Layer Chromatography and Ring-Oven Technique Incorporating Fluorescent Support', Separation Science and Technology, 6: 5, 741 — 745
To link to this Article: DOI: 10.1080/00372367108057968
URL: http://dx.doi.org/10.1080/00372367108057968

SEPARATION SCIENCE, 6(5), pp. 741–745, October, 1971

NOTE

Inorganic Analysis by Combined Thin-Layer Chromatography and Ring-Oven Technique Incorporating Fluorescent Support

K. N. JOHRI and H. C. MEHRA

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF DELHI
DELHI-7, INDIA

Summary

Efficient microinorganic analysis of Ge(IV), As(III), and Sb(III); and Ti(IV), Fe(III), and V(V) on silica gel G and cellulose powder supports has been made by thin-layer chromatography in combination with ring-oven technique. A new sensitive reagent, tropolone, has been incorporated as a spray reagent.

The growing importance of thin-layer chromatography (TLC) in inorganic analysis has been well established in the recent review by Volynets and Ermakov (*1*). Furthermore, various microanalytical techniques have been combined with TLC for the determination (*1*, *2*) of chromatographed species. Of these, the combination of TLC and ring-oven techniques (*3*–*7*) in the separation and determination of metal ions is prominent.

In the present work the separation of (a) Ti(IV), V(V), and Fe(III); and (b) Ge(IV), As(III), and Sb(III) on cellulose powder and silica gel G supports, respectively, is reported. The use of the new spray reagent tropolone has been found useful in producing characteristically colored complexes with metal ions for the location of Ti(IV), V(V), and Fe(III) on the developed plates. In the case of Ge(IV), As(III), and Sb(III), quercetin (*7*) and potassium thiocarbonate (*5*) have been used as visualizers. For the determination of these metal ions, subsequent separations are made on the fluorescent support and visualized by UV light and then estimated by ring oven incorporating suitable developing reagents.

Standard Samples. Solutions of the metals Ge(IV), As(III), Sb(III), Ti(IV), V(V), and Fe(III), 1 mg/ml, were prepared by dissolving analytical grade reagents.

Weisz Ring Oven. Procured from National Appliance Co., Portland, Oregon.

SEPARATION PROCEDURE FOR Ge(IV), As(III), AND Sb(III)

To the glass plates coated with a thin layer of silica gel G (E. Merck), 1–5 μl each of Ge(IV), As(III), and Sb(III) salt solutions was applied separately with a microsyringe along the base line as a reference. At the other end of the base line, 1–5 μl each of the sample solutions was applied at the same spot to produce a mixed sample of known composition. Each plate was developed within 45 min by the ascending technique, using the solvent system isobutyl alcohol–2 N HCl (11:3, v/v), thereby affording a clean separation of the constituent metal ions. The plates were then removed from the jars, air dried, and the resulting spots due to Sb(III) and As(III) were visualized with sprays of 0.05 M PTC while Ge(IV) was visualised with 0.01 M/quercetin (Fluka), so as to standardize the conditions for their separation with a particular solvent system.

With the above solvent system Ge(IV) did not move. The order of movement found was Sb(III) > As(III) > Ge(IV).

SEPARATION PROCEDURE FOR Ti(IV), Fe(III) AND V(V)

In this case glass plates coated with cellulose powder (CAMAG) and the solvent system acetone–3 N HCl (4:1, v/v) were employed. The development time recorded was 15 min. The position of the chromatographed species was located with sprays of 0.01 M/tropolone (Aldrich). The order of migration was Fe > V > Ti.

Limit of identification of the metal ions, R_F values, and the characteristic colors of the spots are given in Table 1.

TABLE 1

Results of TLC Separation

Ion	Visualizer	Color	$R_F \times 100$	Limit of identification (μg)
Ge(IV)	Quercetin in alcohol	Yellow	0	0.28
As(III)	0.05 *M* aq PTC	Yellow	50.00	3.82
Sb(III)	0.05 *M* aq PTC	Orange	95.00	3.16
Fe(III)	0.01 *M* aq Tropolone	Black	78.00	0.08
V(V)	0.01 *M* aq Tropolone	Bluish-black	54.00	0.12
Ti(IV)	0.01 *M* aq Tropolone	Yellow	28.00	0.22

DETERMINATION OF THE CHROMATOGRAPHED CONSTITUENTS BY RING COLORIMETRY (9)

The foregoing separation procedures were repeated on silica gel G 2% fluorescent indicator (M. Woelm) under the same conditions for the evaluation of Ge(IV), As(III), and Sb(III). Visualization of the separated ions on the adsorbent layer was by examination of the plate under a UV (254 nm) lamp. The dark spots representing the different ions with a fluorescent background were then marked out. The marked out portions of the adsorbent layer were collected by means of a Mottier gadget (*8*) and then transferred to filter paper set on the Weisz ring oven.

Similar separation procedures were adopted with cellulose powder DSF support for the estimation of Ti(IV), Fe(III), and V(V), and visualization of the chromatographed species was carried out under UV light. The marked out portions were transferred to the ring oven for evaluation.

A circle of filter paper (Whatman No. 41), 55 mm in diameter, was placed on the oven set at 100–110°. The collected material containing the individual constituent was placed at the center of the filter paper and washed into the ring by means of a self-filling capillary pipet with distilled water. The resulting rings were developed with the reagents listed in Table 2.

The required standard scale was similarly prepared with 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10 drops of the respective metal ions solution taken separately. The rings for the various ions were matched with the rings of the standard scale in order to compute the amounts present in each case.

TABLE 2

Representative Results of Determination

Ion	Developing agent	Color of the ring	Metal ion (mg/ml) Taken	Metal ion (mg/ml) Found	Error (%)
Ge(IV)	0.03% Phenyl fluorone in alcohol	Pink	1.65	1.60	−3.0
			2.00	2.10	+5.0
As(III)	0.05 *M* aq PTC, 1% $AgNO_3$	Black	1.85	1.80	−2.4
			1.30	1.25	−3.9
Sb(III)	0.05 *M* aq PTC	Orange	2.15	2.25	+4.6
			1.55	1.50	−3.2
Ti(IV)	5% aq Disodium chromotropic acid	Reddish-brown	0.90	0.87	−3.3
			1.32	1.38	+3.7
V(V)	1% 3,3′-Dimethyl naphthidine in acetic acid	Red-violet	0.76	0.72	−5.2
			1.12	1.06	−5.3
Fe(III) + ascorbic acid	1% DMG, NH_4OH	Red	0.58	0.56	−3.4
			0.92	0.88	−4.3

In the case of As_2S_3 ring, where the color is light yellow, efficient comparison for direct evaluation was made by using Silver Scale (*9*) method.

DISCUSSION

Scrutiny of the results of TLC separations given in Table 1 shows that with tropolone as the sensitive spraying reagent even minute amounts of metal ions can be visualized. There is a need for the development of determination procedures for fresh chromatograms under similar conditions but using fluorescent support that can then be visualized nondestructively by UV light. This nonassayed technique has advantage when compared with the usual visualization procedure. Furthermore, inspection of the results in Table 2 for the determination of chromatographed species indicates they are sufficiently accurate and the errors lie within permissible limits of semiquantitative analysis.

REFERENCES

1. M. P. Volynets and A. N. Ermakov, *Usp. Khim.*, *39*(5), 934 (1970).
2. E. J. Shellard, *Quantitative Paper and Thin-Layer Chromatography,* Academic, London, 1968.
3. L. J. Ottendorfer, *Anal. Chim. Acta, 33*, 115 (1965).
4. H. Scherz, E. Bancher, and K. Kaindl, *Mikrochim. Acta, 1965*, 255.
5. K. N. Johri, H. C. Mehra, and N. K. Kaushik, *Chromatographia, 1970*, 347.
6. K. N. Johri and H. C. Mehra, *Microchem. J.*, *15*, 642 (1970).
7. K. N. Johri and H. C. Mehra, *Mikrochim. Acta, 1971*, 317.
8. M. Mottier, *Mitt. Geb. Lebensmittelunters Hyg.*, *49*, 454 (1958).
9. H. Weisz, *Microanalysis by Ring Oven Technique,* 2nd ed., Pergamon, London, 1970.

Received by editor February 11, 1971