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Review

Planar layer chromatography in the analysis of inorganic pollutants

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ABSTRACT

This review covers salient features of planar layer liquid chromatography as applied to the analysis of inorganic pollutants present in a variety of complex matrices. The discussion is limited to classical planar chromatography (paper and thin-layer chromatography) as used in the analysis of real and synthetic environmental samples containing inorganic pollutants. The matrices from which inorganic pollutants have been isolated and analysed include: biological, food, geological, industrial, pharmaceutical, soil, water and wastewater. Eighty-nine references have been cited from the literature of the last twenty years. Over-pressured layer chromatography has not been utilized so far for the analysis of inorganic species present in natural environmental samples. *In situ* densitometry and spectrophotometry have been commonly used for the quantification of inorganic ions present in environmental samples.

CONTENTS

1. INTRODUCTION

Planar layer chromatography (PLC) is a liquid chromatographic technique in which the mobile phase migrates through a porous support (stationary phase) either by capillary forces or under the influence of forced flow. Depending on the mode of transport of the mobile phase, PLC can be classified as conventional (classical) PLC and forced-flow PLC (FFPLC). Many of the procedural steps (sam-

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ple spotting, development of chromatograms, detection of spots, direct scanning, etc.) and principles are identical in both techniques. High-performance thin-layer chromatography (HPTLC), an instrumentalized version of TLC with a perfectly uniform surface of thin layers, is capable of providing faster separations, reduced zone diffusion, better separation efficiency and higher sensitivity. FFPLC includes all techniques where the mobile phase migrates by a forced solvent flow, which can be achieved either by application of external pressure (over-pressured layer chromatography, OPLC) [1–3] or by centrifugal force (rotational planar chromatography, RPC) [4–6]. Electroosmosis is applied to force the mobile phase in high-speed TLC (HSTLC) [7–8]. OPLC, developed by Tyihak *et al.* [1] combines the advantages of classical TLC, HPTLC and a high-performance liquid chromatography (HPLC). Several reviews [9–12] have appeared on OPLC with emphasis on instrumentation, chamber types, development modes and applicability. More recently, Kaiser and Rieder [13,14] developed high-pressure planar liquid chromatography (HPPLC), a circular version of OPLC for achieving a better separation efficiency at higher operating pressure. According to the literature, OPLC has applications in solving various analytical problems relating to agriculture, biochemistry, food stuffs and forensic medicine. As a result, it has several applications to the analysis of a large number of organic substances, *e.g.*, dyes, amino acids, antibiotics, antioxidants, aromatic hydrocarbons, polyamines, alkaloids, aliphatic aldehydes, amines and their derivatives, doping agents, essential oils, lipids, peptides, fatty acids, steroids, flavonoid glycosides, ginsenosides, iridoid glycosides and anthraquinone aglycones [15].

It is surprising that in spite of the availability of several attractive features of OPLC, it has not been used in the analysis of inorganic samples. As far as we are aware, no work has been reported on the use of OPLC in analysing environmental samples containing inorganic pollutants.

This review is therefore restricted to outlining the current state-of-art procedures for classical layer chromatography [paper chromatography (PC) and TLC] as applied to the analysis of synthetic and real environmental samples for inorganic pollutants.

2. SAMPLE PREPARATION

A sample volume (1–10 μl) containing a sufficient amount of analyte (0.1–10 μg) is generally applied with the aid of calibrated microcapillary, syringe or micropipette (Drummond microcaps) about 2 cm above the lower edge of a precoated or laboratory-made chromatoplate or paper strip. The one-dimensional ascending technique has usually been used for the development of chromatograms in a closed chamber (cylindrical or rectangular), allowing the mobile phase (solvent) to migrate 8–10 cm from the starting line on the plate or paper strip. Multiple, two-dimensional, centrifugal and gradient development techniques have also occasionally been used.

3. DETECTION

Chemical, physical, enzymatic and radiochemical procedures have been usually used to locate the analytes on chromatoplates. Chromogenic and fluorogenic spray reagents that are capable of forming coloured products with the separated species are sprayed on the chromatoplates to detect the solute. Typical chromogenic reagents used are dithizone, dithiosemicarbazone, aluminon, dimethyl glyoxime, 1-(2-pyridylazo)-2-naphthol (PAN), arsanazo III, *m*-nitrochlorophosphonazo, alizarin and tribromochlorophosphonazo. Among the physical methods, observation under UV light has been the most preferred procedure. Selenium in food samples [16] has been detected as the 2,3-diaminonaphthalene–Se complex, which produces pink fluorescence under UV light (366 nm). An enzymatic method [17] has been used to detect $CuSO_4$, $HgCl_2$, $CdSO_4$ and $AgNO_3$ in fresh water after their separation on TLC plates. Sometimes labelled species of very low-level inorganic materials can be detected using a radioactivity detector [18,19].

4. IDENTIFICATION AND QUANTIFICATION

Compound identification in planar liquid chromatography is based on the R_F value, which is a measure of the ratio of the distance travelled by the analyte from the point of origin to the distance travelled by the solvent. Visual observation, zone elution and scanning densitometric methods have been

used for quantitative analysis. *In situ* densitometry has found some novel applications in the determination of trace amounts of inorganics present in complex matrices [16,20–29].

In spite of the extensive application of planar chromatography to analyses for inorganic and organic compounds in standard sample solutions, its use in the analysis of real environmental samples [30–46] containing inorganic polluting agents is limited. Extraction of the analyte from the sample matrix, clean-up of the extract and concentration of the analyte usually precede TLC or PC if the concentration of the analyte in a complex sample (food, biological, human, plant or environmental) is low. However, if a compound is present in sufficient purity and concentration, the sample can be applied directly.

The following sections provide a brief description of planar chromatography as used in the analysis of environmental samples.

5. APPLICATIONS

Applications have been divided into sections depending on the nature of the sample to be analysed, *viz.*, biological, food stuffs, geological, industrial, pharmaceutical, soil, water and industrial wastewater and miscellaneous.

5.1. Biological samples

TLC in combination with densitometry has been applied to determine selenium in biological tissues [28]. Selenium is removed from the matrix, complexed with diaminonaphthalene and the complex is extracted into cyclohexane and analysed on CRP-Whatman HP plates. TLC separation of the selenium-containing complex permits the complete elimination of interfering fluorescent compounds. The recovery of selenium achieved with this method is 85–90%.

An accurate and highly sensitive HPTLC method for the determination of selenium in water and biological matrices has been developed by Funk *et al.* [29]. Selenium-containing biological samples (blood and serum) and environmental materials (drinking and surface water) were oxidized by a wet chemical digestion procedure, derivatized with 2,3,1-naphthoselenodiazole, extracted on an Extrelut column using cyclohexane, dried, dissolved in chloroform and analysed. The chromatographic separation was carried out on purified silica gel HPTLC plates (Merck) using chloroform containing 0.01% of butylhydroxytoluene (antioxidant) as the mobile phase. The quantitative evaluation was completed in the fluorescence mode with λ_{ex} = 365 nm and fluorescence intensity measurement at λ_{fl} = 560 nm. The proposed method includes the following advantages: comparable to spectrophotometry, polarography, gas chromatography, neutron activation analysis, X-ray fluorescence and atomic absorption spectrometric methods; high sensitivity (detection limit 250 fg of Se per spot); novel digestion procedure (wet chemical digestion) for oxidation of organic matrices; accurate quantitative preparation of biological matrix; the sample preparation step [oxidation of selenium to Se(VI) and subsequent reduction to Se(IV)] is less susceptible to systematic errors; and interfering metal ions (Fe^{3+}, Cu^{2+}) can be easily masked by addition of suitable chelate-forming reagent.

Scanning densitometry and TLC have been used for the simultaneous determination of traces of Cu^{2+}, Fe^{2+}, and Fe^{3+} in serum [23] as their complexes with 2-[(5-bromo-2-pyridinyl)azo]-5-(diethylamino)phenol. The complexes, after extraction from serum samples, were separated on TLC plates coated with silica gel G and sodium carboxymethylcellulose using butyl acetate–acetone (7:3) as the eluent. The absorbances of the separated metallic complexes were measured by densitometry.

Silica gel KSK layers (free from iron) with *n*-butanol–water (84:14) as the eluent were used for the detection of magnesium chlorate isolated from biological objects [47]. This method provides a detection limit of $\geqslant$5 ng per 100 g of biological sample.

A TLC method for the detection of metal–EDTA complexes in human faeces [48] has been reported. An anion-exchange column packed with carboxymethylcellulose was used to clean up and preconcentrate the sample.

Spectrophotometry in combination with TLC has been used to determine Cd^{2+}, Hg^{2+} and Pb^{2+} in blood and urine samples [49]. Metals were separated on silica gel layers using various organic solvents as eluents prior to their quantification.

A chromatographic system [silica gel layers, di-

chloromethane–methanol (9:1) and benzene eluents] has been identified as a sensitive method [50] for the detection of heavy metals (Zn^{2+}, Hg^{2+}, Ag^{+}, Cu^{2+}, Bi^{3+}, Co^{2+}, Ni^{2+} and Pb^{2+}) as dithizonates in biological samples (urine, blood and excrement). The sensitivity of the proposed method was 10^{-3} g/l.

A TLC method for the detection of Pb^{2+}, Cd^{2+}, Hg^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Cu^{2+} and Mn^{2+} present in autopsy tissues [51] has been described. The metals were separated on silica gel layers developed with carbon tetrachloride–chloroform (5:2), xylene, benzene and toluene.

A successful method for the quantitative separation of inorganic mercury and methylmercury from animal, fish and plant tissues has been developed [52]. Mercury species were determined by TLC after extraction from tissues. To achieve the highest efficiency of extraction the fish tissue was initially treated with ethanolic KOH solution whereas the plant tissue was treated with a mixture containing HNO_3 and $HClO_4$.

5.2. Food stuffs

An analytical procedure involving extraction, TLC and densitometry has been developed for the determination of bromate ion in breads and fluor dough [22] after its separation on silica gel plates developed with *n*-butanol–*n*-propanol–water (1:3:1). The limit of detection of BrO_3^- with tolidine–HCl was 0.1 μg/g in bread. Bromate was extracted from food stuffs (bread) and purified using an alumina column.

$[Fe(CN)_6]^{3-}$ and $[Fe(CN)_6]^{4-}$ present in juice and wine samples have been detected on paper chromatograms [53]. A method has been reported [54] for the separation and identification of Fe^{3+}, Mn^{2+} and Co^{2+} present in human milk. The metals were extracted with isobutyl methyl ketone–amyl acetate (2:1), chromatographed and identified on cellulose plates. Similarly, Li^{+} and Bi^{3+} down to 0.25 and 1.5 μg, respectively, were identified and detected in spiked human milk samples [55]. The sample milk was spiked with Li_2CO_3 or $Bi(NO_3)_3$, ashed, dissolved in an appropriate solvent, applied to cellulose-coated plates developed with methanol–10 *M* HCl (6:4) and Li^{+} or Bi^{3+} were detected using an appropriate chromogenic reagent. Cheese and milk samples have been analysed for polyphosphoric acids [56]. The acids were extracted from cheese or milk samples with 25% trichloroacetic acid and separated on polyamide plates using *n*-butanol–formic acid (1:1) as developer. The separated polyphosphoric acids were identified and determined. Two-dimensional TLC and ion-exchange column chromatography have also been used for the separation and determination of ortho- and polyphosphates in soft drinks [57].

Pb^{2+} and Zn^{2+} in model food systems have been determined by TLC on Silufol plates after complexation with sodium diethyldithiocarbamate [58]. The plates were developed with benzene–chloroform (1:1) or xylene–dichloromethane (2:1). Diphenylthiocarbazone in CCl_4 was used as the detection reagent.

A TLC method for the identification of selenium in foods was developed by Moreno-Dominguez *et al.* [16] using thin layers of MN-300 cellulose powder (activated at 110°C for 30 min before use). A spectrofluorimetric procedure was used for the determination of selenium in different foods of animal and vegetable origin and blood samples. The procedure involves the digestion of the food sample, formation and extraction of selenium–2,3-diaminonaphthalene (DNA) complex with cyclohexane, fluorimetric determination and confirmation of the presence of selenium by TLC. After carrying out the fluorimetric determination, the cyclohexane phase containing the selenium–2,3-diaminonaphthalene complex was concentrated nearly to dryness and the residue was dissolved in 0.5 ml of cyclohexane. This solution was spotted on the chromatoplate and chromatographed along with a standard selenium sample using ethanol–25% ammonia solution (70:30) as the mobile phase. The selenium–2,3-diaminonaphthalene complex produces pink fluorescence on exposure of chromatogram to UV radiation (360 nm).

Silicon in edible oils has been separated [59] on silica gel layers using light petroleum–diethyl ether (98:2) as the mobile phase. Rhodamine B was used as the detection reagent.

5.3. Geological samples

A novel method for the analysis of rocks for rare earth elements was developed by Ryabukhin *et al.*

[60]. Rare earth metals, after preconcentration by circular TLC on Fixion 50-X8, were determined by neutron activation analysis. The determination limits ranged from 0.05 to 10 μg/g for 10–30-mg samples. Another attractive method for the simultaneous determination of light rare earths in monazite sand by densitometry on thin-layer chromatograms using diisopropyl ether–diethyl ether–bis(2-ethylhexyl) phosphate–nitric acid (8:8:0.4:0.07) as eluent was reported by Chinese workers [20]. The R_F values for lanthanum, cerium, praseodymium, neodymium, samarium and yttrium on silica gel H mixed with 1% carboxymethylcellulose binder containing 4% ammonium nitrate layers were 0.13, 0.39, 0.55, 0.69, 0.90 and 0.98, respectively, showing good separation possibilities. The densitometric calibration graphs were linear in the range 0.015–0.60 μg of individual rare earth metals. The limits of detection for lanthanum, cerium, praseodymium, neodymium and samarium were in the range 9–12 ng.

TLC has been used for the determination and separation of rare earth metals in ores, rocks and irradiated nuclear fuels [61] using diethyl ether–bis (2-ethylhexyl) phosphate–nitric acid (100:1:3.5) as the mobile phase. Another TLC method [62] includes the use of silica gel as stationary phase and diisopropyl ether–tetrahydrofuran–tributyl phosphate–nitric acid (10:6:1:1) as the mobile phase for the determination of lanthanum, cerium, praseodymium, neodymium and samarium in monazite sand.

Paper electrophoresis has been used for the separation and determination of Al^{3+}, Ti^{4+} and Fe^{3+} in bauxite [63] using lactic acid as carrier electrolyte. The bands were eluted and the metals were determined spectrophotometrically at 510 nm (Al^{3+}), 400 nm (Ti^{4+}) and 510 nm (Fe^{3+}).

A simple paper chromatographic procedure for the determination of microgram amounts of germanium and gallium [64] in different raw mineral materials has been reported. A TLC method [65] involving the use of alumina layers and mixed aqueous–organic solvent systems as mobile phase has been used for the analysis of minerals consisting of Mo^{6+}, Au^{3+}, Sb^{3+}, Hg^{2+}, Cd^{2+}, Bi^{3+}, Mn^{2+}, Pb^{2+}, UO_2^{2+}, Cr^{3+}, Ti^{4+}, etc.

5.4. Industrial samples

TLC coupled with densitometry has been applied to the determination of Co^{2+} in white wine samples [25] in the concentration range 2.5–4.5 μg l^{-1}. The process involves the fixation of Co^{2+} as the 1-(2-pyridylazo)-2-naphthol complex on a membrane filter followed by direct determination of the reflection absorbance of the complex by densitometry.

A method involving circular TLC and spectrophotometry for the determination of 0.01–1.0% lanthanum and yttrium in molybdenum-based alloys has been described [66]. Cellulose layers treated with 0.2 *M* trioctylamine in toluene were used as the stationary phase. Hydrochloric acid at various concentrations was used as the developer. TLC has been used for the determination of Cu in Al alloys following the sampling of the investigated material by anodic dissolution [67]. The chromatograms were developed with acetone–HCl–H_2O (70:15:15) and 1-(2-pyridylazo)-2-naphthol reagent was used for detection.

The radiochemical purity of $Na^{131}I$ solution has been evaluated [68] by TLC on silica gel layers developed with acetone. Perchlorates in explosive residue have also been detected on paper strips and TLC plates [69].

A combination of paper electrophoresis, TLC and densitometry has been proposed for the determination of anionic species [21]. Diphenylamine solution (0.2% in H_2SO_4) with which most of the anions produce blue products was used for detection. Anions were chromatographed on Silufol 254 plates and developed with propanol–ammonia solution (2:1). The method was successfully applied to the densitometric determination of NO_3^- and $Fe(CN)_6^{3-}$ in molasses.

A TLC method has been reported for the rapid detection of copper, iron and manganese ions in cotton materials [70]. The separated metal ions on microcellulose plates developed with acetone–HCl–H_2O (8:1:2) were detected with rubeanic acid followed by exposure to ammonia vapour. TLC in combination with spectrophotometry has also been used for the determination of traces of manganese in textile materials.

5.5. Pharmaceutical products

Spectrophotometry in combination with TLC has been used for the determination of Fe^{2+} in pharmaceuticals [71]. Fe^{2+} was separated from oth-

er cations present in pharmaceutical formulations on microcrystalline cellulose plates using propanol–4 *M* HCl–HNO_3–acetic acid–chloroform (30:5:5:5:10) as developer. 1,10-Phenanthroline was used as the colour reagent. A TLC system consisting of silica gel G as stationary phase and chloroform–acetone–concentrated HNO_3 (5:4:1) as eluent has been used for the identification of mercury salts (chloride, nitrate, cyanide, sulphate and sulphide) in homeopathic drugs [72].

Buchbauer and Vasold [73] developed a TLC method for the separation and identification of inorganic impurities (cationic and anionic) in drugs of the Austrian Pharmacopoeia. Aqueous solutions (10%) of drugs were spotted on cellulose-coated micro-plates and developed with methanol–HCl (8:1) for cations or with methanol–*n*-butanol–water (2:1:1) for anions. Inorganic impurities as coloured spots were detected by spraying the developed plates with sixteen spray reagents.

Aqueous–organic solvent systems have been used as mobile phases with silica gel layers for the determination of radioactive impurities in pharmaceuticals [74] and the radiochemical purity of $Na_2{}^{51}CrO_4$ injections [75]. A photodensitometric method [76] for the determination of manganese in pharmaceuticals, vitamins, etc., as a manganese–1-(2-pyridylazo)-2-naphthol complex with preliminary TLC separation on silica gel layers using pyridine–methyl isobutyl ketone–choroform (20:4:1) as mobile phase has been reported.

5.6. Soil, water and industrial wastewater samples

A rapid TLC method for the analysis of industrial and wastewaters for total heavy metal content (Fe^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+} and Cu^{2+}) was developed by Volynets et al. [77]. Heavy metals as coloured diethyldithiocarbamates were concentrated during the chromatographic process on plates in the shape of irregular strips as shown in Fig. 1, coated with Silufol, and determined semi-quantitatively directly on the plates on the basis of their colour intensities. The method was successfully applied to the semi-quantitative determination (mg l^{-1}) of nickel and copper in electroplating wastewater.

An interesting method involving the use of TLC in combination with spectrophotometry has been reported [27] for the determination of boron at ng

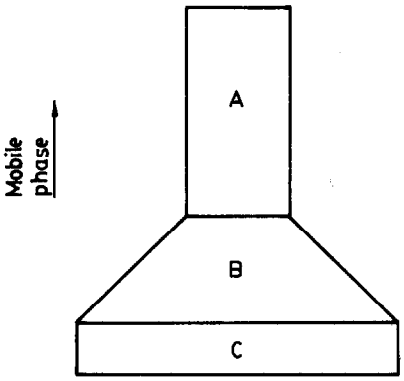

Fig. 1. Irregular strips used for concentrating diethyldithiocarbamates of heavy metals during the chromatographic process. A = Separation zone; B = concentration zone; C = sample application zone.

levels in soil and water samples. A water sample (25 ml) was concentrated by evaporation to 0.5 ml and acidified to pH 1. Aliquots (5–25 μl) of the sample were applied on MN 300 cellulose layers. The plates were developed with butanone–water–ethylene glycol (85:13:2). Boron (R_F = 0.43) was detected with azomethine (1 g in 100 ml of 1% ascorbic acid) spray reagent. After 1 h the sample zone was scanned at 400 nm. The calibration graphs were linear for 50–450 ng of boron.

A combined spectrophotometric–TLC method for microgram determination and recovery of Hg^{2+} from river and industrial wastewater was developed by Ajmal *et al.* [78]. Hg^{2+} (R_F = 0.85) was separated from Pb^{2+}, Ni^{2+}, Hg^{+} and Cu^{2+} on silica gel layers impregnated with 2% oxalic acid using ethyl acetate–acetone–formic acid–water (8:7:4:1) as the mobile phase. Hg^{2+} was detected with dithizone; the area corresponding to Hg^{2+} was scraped off the working plate, and the complex was extracted with carbon tetrachloride and determined spectrophotometrically. The calibration graph obtained under optimum experimental conditions was linear over the concentration range 5–15 μg per 10 ml of Hg^{2+}.

HPTLC and *in situ* densitometry have been successfully used to detect, separate and determine inorganic mercury and some organomercury species at the nanogram level as dithizonates in tap and sea waters [79]. Detection and semi-quantitative determination of Pb^{2+}, Zn^{2+}, Cd^{2+}, Hg^{2+} and Cu^{2+} in

industrial wastewater has been accomplished on silica gel and cellulose plates [80].

An attempt has been made to use a chelating cellulose sorbent with azopyrocatechol groups for the separation and determination of heavy metal ions (Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cr^{3+}, Cr^{6+}, Fe^{2+}, V^{4+} and V^{5+}) by TLC [81]. The metals were determined according to the colour reaction in the sorbent zone. Heavy metals react with the sorbent phase to produce coloured products at pH 1–6. The detection limit of the elements in the zone is in the range 0.05–2.0 μg. A mixture of 1-butanol, acetone, glacial acetic acid, 5% ammonia solution and water (7:5:3:3:2) was used to develop the chromatogram. A brief outline of the procedure as used for the determination of heavy metals is given below.

Sample solution containing 20–400 mg/l of the ions being determined is adjusted to pH 1 by adding HCl and an aliquot (2–5 μl) is applied to the chromatoplate. The coloured zone that appears is dried and the plate is developed with the chosen mobile phase keeping the ascent to 10 cm (development time 15–45 min). After complete drying of the developed plate, the content of the elements in the zone is evaluated by comparing the obtained chromatogram with the standards.

The method was applied to the analysis of waste waters from an electroplating process before their treatment to determine the contents of heavy metal ions in the range 20–400 mg/l. The results were comparable to those obtained by atomic emission spectrometry.

Reversed-phase paper chromatography using triphenylphosphine oxide as the stationary phase and organic complexing agents (sodium malonate, acetate and succinate) as the mobile phase has been applied to the separation and determination of As^{3+}, Sb^{3+} and Bi^{3+} in water and alloy samples [82].

A simple and portable bacterial enzymatic paper chromatographic procedure has been developed for the determination of Cu^{2+} and Hg_2^{2+} in water samples [83].

Hg^{2+}, Zn^{2+}, Cu^{2+} and Pb^{2+} present in water and aquatic plants have been detected on silica gel layers [84]. Aquatic plants were mineralized in concentrated H_2SO_4, HNO_3 and H_2O_2 and extracted in water. The extract was treated with chloroform containing dithizone and the resulting extract was chromatographed on silica gel plates. The detection limit of metals was in the range 0.5–5.0 μg.

A simple PC and micro-TLC method for the separation and detection of some heavy metals ($HgCl_2$, $CuSO_4$, $CdSO_4$, $AgNO_3$, etc.) in fresh water has been reported [17]. Precipitation and evaporation techniques were adopted to concentrate the samples. The preconcentrated samples were dissolved in citric acid and separated by PC and micro-TLC using 0.1 *M* NaCl solvent. The separated heavy metals were detected by horse liver acetone powder succinate dehydrogenase inhibition using 2-(4-iodophenyl)-3-(4-nitrophenyl)-5-phenyltetrazolium chloride–sodium succinate–N-methylphenazonium methosulphate mixture as the chromogenic reagent. PC was found to be most suitable for the separation of $CuSO_4$–$HgCl_2$ and $CdSO_4$–$HgCl_2$ mixtures whereas micro-TLC was suited to the separation of $CuSO_4$–$CdSO_4$ mixture.

5.7. *Miscellaneous*

TLC has found an interesting application in the analysis of cosmic dust particles containing Fe^{3+} and Co^{2+} [85]. Thin-layer plates coated with silica gel KSK mixed with starch (5%, w/w) were spotted with solutions containing 15–20 μg of material and developed with acetone or acetone–3 *M* HCl (99:1). The semi-quantitative determination of Fe^{3+} and Co^{2+} on the basis of spot size and colour intensity or by reflectance densitometry was carried out.

A method involving qualitative TLC followed by quantitative PC has been developed to determine Pb^{2+} in airborne dust [86]. TLC was performed on silica gel layers using dioxane–1.5 *M* HCl–butanol–acetylacetone (50:20:50:0.5) and dioxane–1.5 *M* HCl–acetylacetone (50:20:0.5) as mobile phases.

Rare earth elements (REEs) and other fission products in freshly irradiated nuclear fuels have been determined after their enrichment and separation by TLC [87,88]. REEs were quantitatively separated from all other fission products by two-dimensional TLC using diisopropyl ether–tetrahydrofuran–HNO_3 (100:80:4) and diethyl ether–bis (2-ethylhexyl) phosphate–HNO_3 (100:4:2) as developer. After separation, REEs were determined by γ-spectroscopy.

NO_3^- in feeds has been determined after its separation on alumina G plates [89] developed with

0.05 *M* NaOH–acetone (3:17). After TLC, the nitrate-containing zone was scraped off and treated with 3,4-xylenol in sulphuric acid medium. The reaction product was extracted with light petroleum. The organic layer was subsequently shaken with aqueous alkali. The aqueous layer was separated and the absorbance of this layer was measured at 430 nm.

6. CONCLUSIONS

From the above survey it is clear that planar layer chromatography (specially TLC) has been a dependable and useful analytical tool for the determination of inorganic pollutants after their separation. However, these studies have been mostly restricted to heavy metals and therefore efforts are required to utilize TLC by coupling it with sensitive instrumental techniques for the determination and identification of other inorganic species in environmental samples. In addition, forced-flow planar liquid chromatographic techniques should be developed for utilization in the analysis of inorganic species present in both environmental and non-environmental samples. OPLC, a real planar version of HPLC, has special advantages as a planar system with great prospects for the future.

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