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

Thirty-Five Years of Thin-Layer Chromatography in the Analysis of Inorganic Anions

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To cite this Article Mohammad, Ali and Tiwari, Sharad(1995) 'Thirty-Five Years of Thin-Layer Chromatography in the Analysis of Inorganic Anions', Separation Science and Technology, 30: 19, 3577 — 3614

To link to this Article: DOI: 10.1080/01496399508014147

URL: http://dx.doi.org/10.1080/01496399508014147

SEPARATION SCIENCE AND TECHNOLOGY, **30**(19), pp. 3577–3614, 1995

REVIEW

Thirty-Five Years of Thin-Layer Chromatography in the Analysis of Inorganic Anions

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ABSTRACT

An exhaustive review focusing on types of stationary phases, mobile phases, detection reagents and techniques involved in the identification, separation, and determination of inorganic anions in various samples is presented. Results on thin-layer chromatographic studies of anions covering the period 1959 to December 1994 have been collected from all available sources such as research papers, review articles, *Analytical Abstracts*, *Current Contents*, and *Chemical Abstracts*. Care has been taken to provide as much information as possible in a condensed form without omissions. This is the first review to provide all necessary information in respect of TLC separation of inorganic anions. A necessity is felt for developing forced flow planar chromatographic techniques for the analysis of anionic species.

INTRODUCTION

The work on thin-layer chromatography (TLC) of inorganics published up to the end of 1972 has been admirably reviewed by Brinkman et al. (1), and that appearing during 1972–1980 has been presented by Kuroda and Volynets (2). The latest work on TLC of inorganics and organometallics covering the period 1978–1988 has been described by Mohammad and Varshney in a chapter of the *Handbook of Thin-Layer Chromatography* edited by Sherma and Fried (3).

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According to the literature, less emphasis has been given on the use of TLC in the analysis of anionic species. In spite of a number of interesting review articles or books published in the recent past (4–26), a review covering all aspects of TLC of anions has not yet appeared. The aim of this article is to present a complete report of work carried out exclusively on anions using TLC procedures since 1959, when the publication on TLC of inorganic compounds started to appear in *Chemical Abstracts*, till the end of 1994. To our knowledge, it is the first review to provide an up-to-date picture of anion TLC.

RESULTS

The results are encapsulated in Tables 1–5. The stationary phases, mobile phases, and detection reagents used in TLC analysis of inorganic anions are listed in Tables 1–3. Table 4 presents complete information about chromatographic systems as used by workers for analytical studies of various anions. (A list of abbreviations appears on pages 3607–3608.) Research papers on TLC of anions from some selected journals are shown

TABLE 1
Stationary Phases Used in TLC of Inorganic Anions

Code	Stationary phases
S_1	Aluminum oxide D-5, silica gel D-O, aluminum oxide S
S_2	Aluminum oxide (act. grade IV, 100–150 mesh)
S_3	Aluminum oxide G-kieselguhr G (1:1 w/w)
S_4	Aluminum oxide G (Merck)
S_5	Aluminum oxide S (Hopkin and Williams), DS-5 (Camag), silica gel S (Hopkin and Williams)
S_6	Aluminum oxide G
S_7	Alumina
S_8	Alumina containing 5% gypsum
S_9	Alumina (MN-Polygram Alox N)
S_{10}	Alumina (CHD), alumina + silica gel G (1:1, 1:2, and 2:1)
S_{11}	Cellulose (precoated plates; Merck)
S_{12}	Cellulose (Avicel SF, tech. grade, precoated uniplates; F.M.C.)
S_{13}	Cellulose (precoated uniplates, Avicel; Anal. Tech.)
S_{14}	Cellulose MN 300 HR
S_{15}	Cellulose
S_{16}	Cellulose MN 300
S_{17}	Cellulose MN 300 + 1 wt% sodium CM-Cellulose
S_{18}	Cellulose (Whatman) + 5.5 wt% starch
S_{19}	Cellulose (Polygram Cel MN 300)

TABLE 1 Continued

Code	Stationary phases
S_{20}	Cellulose (>300 mesh; Toyo Roshi)
S_{21}	Cellulose (Avicel; F.M.C.)
S_{22}	PEI-cellulose (precoated plates)
S_{23}	Cellulose (for column chromatography; S and S)
S_{24}	Cellulose (Fertigfolic F 1440, S and S)
S_{25}	Cellulose CC-41 (Whatman)
S_{26}	Cellulose powder 142 dg and 140 dg + 2 wt% maize starch
S_{27}	Cellulose powder 142 dg-cellulose 144 (2:1 w/w)
S_{28}	Cellulose, cellulose impregnated with polyethyleneimine
S_{29}	Cellulose (144, 142 dg; S and S)
S_{30}	Cellulose (144; S and S)
S_{31}	Cellulose (Polygram Cel MN 300)
S_{32}	Cellulose (Toyo Roshi) suspended in aq. sodium polyacrylate (A_1) or in cellulose acetate in DMF (A_2), silica gel (Wakogel B-O) suspended in sodium polyacrylate (A_3), or in cellulose acetate in DMF (A_4)
S_{33}	Cellulose (Avicel, tech. grade; F.M.C.)
S_{34}	Cellulose MN 300 HR, silica gel MN-N-HR, starch
S_{35}	Cellulose-DEAE in the formate form, cellulose microcrystalline (Avicel SF; F.M.C.)
S_{36}	Cellulose (MN 300) impregnated with polyethyleneimine
S_{37}	Cellulose microcrystalline
S_{38}	Cellulose microcrystalline incorporating a fluorescent indicator
S_{39}	ECTEOLA-cellulose (S and S)
S_{40}	DEAE-cellulose MN 300 Polygram.
S_{41}	Cellulose (ready thin layers on Al foil; Carlo Erba), silica gel (ready thin layers on Al foil; Carlo Erba), silica gel (Baker flex 1B-F)
S_{42}	Cellulose (precoated plates; Merck, Darmstadt)
S_{43}	Cellulose (Merck 5716, ready-made plates)
S_{44}	Corn starch (Servo Mihol)
S_{45}	Baker-flex DEAE cellulose sheets, PEI-F cellulose sheets, Eastman Kodak silica gel sheets 6060, Baker-flex silica gel 1B sheets
S_{46}	Cellulose commercial
S_{47}	Dowex 1-X10, 1-X2, Bio-Rex 5, zirconium hydroxide
S_{48}	Dowex 2-X8 (Cl^- and Ac^-), Lonex 25-SB (MN) (Ac^-)
S_{49}	Hydrous II antimony V oxide.
S_{50}	Indium oxide plates (Cavadura 5, Blazers)
S_{51}	Keratin (unmodified, esterified, or deaminates)
S_{52}	Kieselguhr G (Merck)
S_{53}	Lucefol-quick layers
S_{54}	Maize starch + 10 wt% gypsum
S_{55}	Maize starch
S_{56}	Polyamide (Woelm), silica gel G, silica gel MN
S_{57}	Polyamide plates (Woelm)
S_{58}	Silica gel G, kieselguhr (Merck), lanthanum oxide (Aver Remy)
S_{59}	Silica gel MN S-HR
S_{60}	Silica gel

(continued)

TABLE 1 Continued

Code	Stationary phases
S_{61}	Silica gel MN G-HR
S_{62}	Silica gel H
S_{63}	Silica gel (Merck) impregnated with 3% $AgNO_3$ (10 mL/4 g silica gel)
S_{64}	Silica gel G
S_{65}	Silica gel G (Res. Specialties)
S_{66}	Silica gel G, Silufol UV 254:Fertigtolien (Kavalier CSSR)
S_{67}	Silica gel (Wakogel B-O) + 10 wt% starch
S_{68}	Domestic silica gels (250–300 mesh)
S_{69}	Silica gel H (Merck)
S_{70}	Silica gel + 7 wt% starch
S_{71}	Silica gel G (Merck)
S_{72}	Silica gel G (Merck) + 10 wt% Dowex 50W-X8 (200–400 mesh, K^+)–4 wt% CH_3COOK, silica gel KSK (3–25 μm) + 10 wt% Dowex 50W-X8 (200–400 mesh, Na^+)–5 wt% starch
S_{73}	Silica gel (Merck) + 5 wt% starch + 5 wt% Na_2CO_3, silica gel (Merck) + 5 wt% starch
S_{74}	Silica gel (Merck) + 5 wt% starch
S_{75}	Silica gel G + 10 wt% Dowex 50W-X8 (200–400 mesh, K^+) + 2 wt% potassium acetate
S_{76}	Silica gel (Wakogel B-10)
S_{77}	Sephadex G-200, LH 20 (Pharmacia Uppsala)
S_{78}	Silica gel foils (Ready made)
S_{79}	Silica gel (Wakogel B-O), instant TLC sheets (Gelman type SA)
S_{80}	Silica gel sheets
S_{81}	Sephadex G-25, G 200, LH 20
S_{82}	Silica gel/cellulose microcrystalline
S_{83}	Silica gel G and D
S_{84}	Silufol UV 254 layers
S_{85}	Chromagram silica gel sheets (Eastman Kodak 6061 polyvinyl alcohol binder)
S_{86}	Silica gel G_{254} (Merck, Darmstadt, GFR)
S_{87}	Silufor UV 254 (silica gel adsorbent with starch binder and a fluorescent indicator)
S_{88}	Silica gel G and D, 50:50 silica gel G–Silpearl, commercially prepared films UV 254 (containing a mixture of Silpearl and silica gel)
S_{89}	Silica gel KSS-4
S_{90}	Silica gel H 60, cellulose microcrystalline
S_{91}	Silufol 254 type commercial plates
S_{92}	Hydrated stannic oxide
S_{93}	Silica gel + 5 wt% starch
S_{94}	Silica gel G, alumina G (Merck)
S_{95}	Silufol 254
S_{96}	Silica gel impregnated with methanolic solution of fluorescein
S_{97}	Silica gel G and Zr(IV) molybdate
S_{98}	Silica gel + antimonic acid
S_{99}	Silica gel G, silica gel impregnated with 0.1–5.0% aq. $CuSO_4$ solution

TABLE 1 Continued

Code	Stationary phases
S_{100}	Silica gel G impregnated with 0.1% aq. $CuSO_4$, $ZnSO_4$, $NiCl_2$, $CoCl_2$, and $Co(NH_3)_6Cl_3$
S_{101}	Silica gel G, alumina, cellulose microcrystalline, alumina + cellulose (1:1, 1:2, 2:1), alumina + silica gel (1:1, 1:2, 2:1)
S_{102}	Silica gel impregnated with 1% aq. $CuSO_4$ solution, silica gel G, alumina, cellulose microcrystalline, kaolin, kieselguhr G, alumina + cellulose (1:1), kieselguhr + cellulose (1:2, 2:1)

TABLE 2
Mobile Phases Used in TLC of Inorganic Anions

Code	Mobile phases
M_1	Organic layer of butanol-1–pyridine–water–NH_3 (8:4:8:1)
M_2	Water, acetone, water–acetone (1:1), acetone–conc. H_2SO_4–water (45:4:1)
M_3	Butanol-1-acetone-conc. NH_3–water (X:10:2:1)
M_4	0.2 M KNO_3
M_5	Butanol-1–water–pyridine (2:2:1), butanol-1–water–pyridine–NH_3–acetone (8:12:4:1:8), butanol-1–water–pyridine–NH_3 (8:8:4:1)
M_6	0.05 N NaOH-acetone (3:17)
M_7	Water and NaOH (0.5 N and 1 N)
M_8	Methanol–28% NH_3 (10:10), water–28% NH_3 (10:1)
M_9	Aq. solutions of salts, acid, bases, and buffer solutions; K_2SO_4, K_3PO_4, and NaF solutions; 1 N KNO_3, 0.1 N Na_2CO_3
M_{10}	EMK, FA, acetone, methanol, FA + EMK/acetone/methanol/ethanol/*n*-propanol/isopropanol/*n*-butanol/butane-2-ol/50% picric acid solution in methanol (1:9, 4:6, 1:1, 6:4, 9:1 v/v)
M_{11}	Acetone-water (9:1)
M_{12}	Ethanol–pyridine–water–NH_3 (15:5:4:1)
M_{13}	Ethanol–pyridine–water–conc. NH_3 (15:5:4:1); mixtures of butanol-1, HCl, HF, HBr, TOPO
M_{14}	Acetone–water mixtures
M_{15}	Ethanol–isobutanol–propanol-2–water–trichloroacetic acid–NH_3 (35:15:20:30:5 g:0.4); methanol–isobutanol–water–FA–NH_3 (50:10:31:0.3:9)
M_{16}	Water–ethanol–isobutanol–propanol-2–22% NH_3–TCA (30:15:15:20:0.4:5 g); 22% NH_3–methanol–isobutanol–water–FA (9:50:10:31:0.3)
M_{17}	Butanol-1–20% TCA–acetone–25% NH_3 (120:60:40:3); propanol-1–20% TCA–25% NH_3 (120:80:3); isobutanol–20% TCA–acetone–25% NH_3 (20:60:40:3)
M_{18}	5 g TCA–0.3 mL conc. NH_3 dissolved in 100 mL ethanol–water or 100 mL propanol-2–water

(continued)

TABLE 2 Continued

Code	Mobile phases
M_{19}	Propanol-2–TCA–20% aq. tetraethylammonium hydroxide–water (75:5 g:1.6:25) used after 12 h; propanol-2–isobutanol–NH_3 (40:20:1 mL) set aside for 12 h
M_{20}	Dioxane–water–TCA–NH_3 (65:27.5:5 g:0.25)
M_{21}	Methanol–TCA–96% acetic acid–water (60:15:1:4), TCA (3 g)–25% NH_3 (30 mL) made up to 1 L with water
M_{22}	Methanol–(100 g TCA with water to 500 mL + 22.7 mL NH_3)–[96% acetic acid–water (1:4)]–water (60:10.3:5:8); propanol-2–isobutanol–water–conc. NH_3 (40:20:39:1)
M_{23}	28% NH_3, TCA, glacial acetic acid, water, isopropanol, and isobutanol
M_{24}	0.5–1.5 M solutions of NaCl and NH_4Cl
M_{25}	DMF–butanone–water–NH_3 (20:20:99:1)
M_{26}	Methanol–dioxane–[(propanol-2–water (7:1)]–[96% TCA–water (1:4)]–(125 g TCA + 32 mL 25% NH_3 to 1 L with water) (30:15:15:4:20)
M_{27}	Acetone–TFA–water–conc. NH_3 (140:6:52.8:0.6, 136:6:56.8:0.6, 132:6:62.8:0.6, 126:6:66.8:0.6, 120:6:72.8:0.6)
M_{28}	Methanol–dioxane–[propanol-2 + water (7:1)]–[glacial acetic acid + water (1:4)]–(125 g TCA + 32 mL 25% NH_3 diluted to 1 L with water) (30:15:15:4:20)
	Methanol–[propanol-2 + water (7:1)]–(125 g TCA + 32 mL 25% NH_3 to 1 L with water)–[glacial acetic acid + water (1:4)] (75:20:25:6)
	Dioxane–[160 g TCA + 82 mL 25% NH_3 to 1 L with water (7:3)]
	Methanol–[propanol-2 + water (7:1)]–(75 g TCA + 80 mL 25% NH_3 to 1 L with water)–[glacial acetic acid + water (1:4)] (67.5:22:6:50:6)
M_{29}	Methanol–dioxane–[propanol-2 + water (7:1)]–[glacial acetic acid + water (1:4)]–(125 g TCA + 32 mL 25% NH_3 to 1 L with water) (9:3:2:1:5)
M_{30}	0.05–3.0 M LiCl
M_{31}	Alcohols and/or dioxane–10% TCA–98% acetic acid–25% NH_3–water mixtures
M_{32}	Propanol-2–dioxane–10% TCA–98% acetic acid–NH_3–water (30:40:27:1:0.3:1.7)
M_{33}	Methanol–[propanol-2–water (7:1)]–(125 g TCA + 32 mL 25% NH_3 + water to 1 L)–[96% GAA + water (1:4)] (75:20:25:6)
M_{34}	Acidic: Acetone–acetic acid–water (35:11:9); Methanol–TCA solution 1–19% acetic acid (60:10:3.5); Ethanol–water–TCA–28% NH_3 (80:20:5 g:0.2); Propanol-2–water–TCA–conc. NH_3 (75:25:5 g:0.25); Propanol-2–water–89% FA–NH_3 (75:20:5:0.1, pH to 1.4); Propanol-2–*tert*-butanol–water–TCA (6:8:6:1 g); Propanol-2–dioxane–TCA solution 2–96% acetic acid–water (26.25:30:15.6:0.6:16.15); Propanol-2–ethylene glycol monomethyl ether–TCA–25% NH_3–water (80:40:5 g:0.3:40); *tert*-Butanol–water–picric acid (20:5:1 g); Dioxane–water–TCA–29% NH_3 (60:35:5 g:0.25); Acetone–TCA solution 3–water (13:5:2)
	Basic: Methanol–28% NH_3–10% TCA–water (30:9:2:18); Methanol–butanol-1–water–NH_3 (25:5:11:9, to pH 11.4 with FA); Ethanol–propanol-1–water–NH_3 (30:30:39:1); Ethanol–isobutanol–water–25% NH_3 (30:30:39:1); Propanol-1–water–28% NH_3 (3:1:1); Propanol-2–isobutanol–water–conc. NH_3 (40:20:39:1); Pyridine–water–NH_3 (13:6:1)

TABLE 2 Continued

Code	Mobile phases
M_{35}	Acetone–ethyl acetate–water (3:1:1, 6:1:3); Acetone–water (4:1); Acetone–ethyl acetoacetate–water (6:3:1); Ethanol–water–1.5 N NH_3 (6:2:1); Methanol–butanol–water (2:1:1); Methanol–FA (100 mL:10 drops); Butanol-2–4 N HCl (4:1); Butanol–propanol-2–1.5 N NH_3 (1:2:3); *Methanol–ethylenediamine (100 mL:10 drops)*
M_{36}	Methanol, ethanol, propanol-2, isobutanol, *tert*-butanol, acetone, butanone, FA, TCA, potassium acetate, 0.88 M NH_3, water, pH
M_{37}	Aqueous FA solutions
M_{38}	Water–ethanol–isobutanol–propanol-2–22% NH_3–TCA (30:35:15:20:0.4:5 g); Dioxane–[160 g TCA + 8 mL 25% NH_3 to 1 L water (7:3)], methanol–[propanol-2–water (7:1), 125 g TCA + 32 mL 25% NH_3 to 1 L with water]–[GAA–water (1:4)] (75:20:25:6); Gradient elution with ethanol–water mixtures containing variable amounts of TCA and NH_3, methanol, propanol-2, 10% TCA, 98% acetic acid, 25% NH_3 (60:14:25:1:0.6); Methanol, 100 g TCA with water to 500 mL, then add 22.7 mL NH_3–[96% acetic acid–water (1:4)], water (60:10.3:5:8)
M_{39}	2.0 M LiCl
M_{40}	Acetone, water, NH_3, ether, chloroform, dioxane, pyridine, and alcohols in various ratios
M_{41}	Acetone, water, ether, dioxane, HNO_3, TCA, GAA, ethylene glycol, and alcohols in various ratios
M_{42}	27 mobile phases
M_{43}	Propanol-2–water–acetic acid (20:5:2)
M_{44}	6 M HCl
M_{45}	Butanol–pyridine–1 N NH_3 (2:1:2)
M_{46}	Basic and polar developing solvent systems
M_{47}	Abel and MD-4 acid mobile solvents
M_{48}	28% NH_3–acetone–*n*-butanol (60:130:30)
M_{49}	28% NH_3–acetone–*n*-butanol (60:130:30); 28% NH_3–acetone (2:3); Dioxane–water (3:2); Acetone–acetic acid–water (20:1:20)
M_{50}	TCA–propanol-2–water–0.1 M EDTA–25% NH_3 (5 g:80:39:1:0.3)
M_{51}	3 M NH_4NO_3; 3 M Ammonium hexafluorophosphate; 6 M NH_4NO_3; Acetone–heptane–0.29 M aq. tetrabutylammonium chloride (12:2:0.5); Acetone–heptane–0.072 M aq. tetrabutylammonium chloride (9:3:0.5)
M_{52}	Only one solvent system used
M_{53}	Methanol–dioxane–88% isopropanol–20% acetic acid–9% TCA (50:10:15:5:25)
M_{54}	Butanol (or propanol)–methanol–water–TCA (35:35:25:5 g)
M_{55}	Isopropanol–13.5% TCA–25% NH_3 water (140:40:0.6)
M_{56}	1.5×10^{-2} M sodium salicylate; 25% Ethanol
M_{57}	1 M $NaNO_3$; 1 M $LiNO_3$; 2 M NH_4NO_3; 3 M NaCl; 2 M NaCl
M_{58}	pH 5.0 buffer [20 mL pyridine + 13.4 mL glacial acetic acid (+ 0.25 M NaCl for oligophosphates) to 1 L with water]
M_{59}	Six aqueous organic acids
M_{60}	Methanol–TCA–dimethylamine (55:3:52); Water–ethylene glycol (11:1)
M_{61}	HCl–KCl buffer (pH 2.5, 10.2); 0.2 M KCl (pH 5.0 with HCl)
M_{62}	Propanol-2–water–20% NH_3 (75:25:0.3) + added TFA

(continued)

TABLE 2 Continued

Code	Mobile phases
M_{63}	Acetone–3 N NH_3 (7:3); 3 N NH_3, water
M_{64}	Acetone–3 N NH_3 (1:1)
M_{65}	Acetone–1% ammonium acetate (9:1); Ethyl acetate–GAA (19:1); Methanol–2 N HCl–acetone (7:2:1); Acetone–conc. NH_3 (3:1); Methanol–water (1:1), Methanol–water–acetone (5:3:2) + drop NH_3; Methanol–GAA (19:1); Methanol–2 N HCl (19:1); Methanol–2 N HCl–acetone (13:3:2); Methanol–conc. HNO_3 (19:1); Methanol–2 N HCl (4:1)
M_{66}	Butanol–FA (1:1)
M_{67}	Acetone–6 N NH_3 (2:6, 2:5, 2:4, 2:3, 2:2, 2:1); 1 N NH_3; 1 N NH_3–acetone (1:1)
M_{68}	Ethyl acetate–ether saturated with water–TBP (25:25:1); Methanol–propanol-1–water–conc. NH_3–10% TCA (50:30:15:8:1.5)
M_{69}	Acetone–propanol-2–conc. NH_3–water (13:4:2:2)
M_{70}	Butanol-1–propanol-1–di-*n*-butylamine (9:9:2)
M_{71}	Butanone–ethanol–NH_3 (5:5:2); Butanone-1–ethanol–water (2:2:1)
M_{72}	Water saturated with isobutanol; 10–15% aq. ammonium acetate; water saturated with isobutanol–40% ammonium acetate (4:1)
M_{73}	Acetone–butanol-1–conc. NH_3–water (13:4:2:1)
M_{74}	Methanol–conc. NH_3–10% TCA–water (10:3:1:6); Propanol-2–THF–conc. NH_3 (5:3:2)
M_{75}	Methanol–water (3:1)
M_{76}	Butanol–acetone–NH_3–pyridine (12:5:1:2); Butanol–pyridine–NH_3 (2:2:1)
M_{77}	Ethanol–butanol–1.6 N NH_3 (75:75:4–12); Ethanol–butanol-1–1.6 N NH_3–1 M ammonium acetate (15:15:2:0–8)
M_{78}	Methanol–propanol-1–concn. NH_3–water (10:10:1:2); Methanol–dioxane–conc. NH_3–water (3:6:1:1)
M_{79}	Methanol–NH_3–10% TCA–water (10:3:1:6)
M_{80}	Methanol–conc. NH_3–TCA–water (3:3:1:13)
M_{81}	Methanol–conc. NH_3–10% TCA–water (10:3:1:6)
M_{82}	Anhydrous alcohol–benzene (1:10)
M_{83}	0.1 or 0.2 M solutions of sodium salt of anion in 30 or 50% ethanol–water, after application of solution of crystal violet at a starting point 5 cm above the lower edge of the plate
M_{84}	Solution of sodium salts of various anions of molarity: complex of Co(III), 0.2; Ni, 0.1; and Cu and Pt, 0.5
M_{85}	*tert*-Butanol–acetone–water (9:35:6); Ethanol–propanol-1–GAA–acetone–water (37.5:37.5:5:1:20)
M_{86}	Acetone–water (10:1); Methanol–butanol-1–water (3:1:1); Acetone–benzene (1:1)
M_{87}	Acetone–water (10:1); Methanol–butanol-1–water (3:1:1); Butanol-1 saturated with 2 N HNO_3
M_{88}	Butanol-1–ethanol–water (2:2:1)
M_{89}	*tert*-Butanol–acetone–water (18:70:12)
M_{90}	Acetone, butanone, 28% NH_3, 14% NH_3, 2% NH_3
M_{91}	0.01 N HNO_3; 3 N $LiNO_3$; 3 N CH_3COOLi; 0.01 N HNO_3–ethanol (1:1)
M_{92}	Acetone–FA–water (70:10:10); Butanol–ethanol–water (20:60:20); Distilled water; Propanol–$CHCl_3$–benzylamine (60:30:10)

TABLE 2 Continued

Code	Mobile phases
M_{93}	Dioxane–methanol–conc. NH_3–water (6:3:1:1); Heptanol-2–methanol–water (85:10:5); Methanol; 99% Ethanol; 5% (v/v) water in butanol-1; Octanol-1 saturated with water; propanol-1–methanol (1:1); Ethanol–*n*-butanol–aq. ammonia (75:75:4), (75:75:8), (75:75:12); Ethanol–*n*-butanol–aq. NH_3–1 M CH_3COONH_4 (75:75:10:0), (75:75:10:10), (75:75:10:20), (75:75:10:40)
M_{94}	Acetone; Acetone–water (83:17)
M_{95}	0.01 M HCl; 4M LiCl; Ethanol–0.01 M HCl (1:1)
M_{96}	Acetone, dioxane mixed with NaOH or NH_4OH solutions
M_{97}	Isopropanol–ethanol–water (9:4:3)
M_{98}	Ethanol–dioxane–water–NH_4OH (30:60:50:25)
M_{99}	Methanol solutions containing 10 g water/100 mL, 0–3.7 M HCl or 0–3.8 M in HNO_3
M_{100}	1 M KNO_3
M_{101}	Acetone–butanol–25% NH_3–water (65:20:10:5)
M_{102}	Ethanol–pyridine–water–NH_3 (60:20:16:4)
M_{103}	Acetone–C_6H_6–water (16:5:2)
M_{104}	Butanol–C_5H_5N–water–25% NH_4OH (80:40:80:5); Ethanol–water (70:40)
M_{105}	Acetone–FA–water (7:1:1)
M_{106}	Various alcohol–aq. ammonia systems
M_{107}	Various alcohol–NH_4OH systems
M_{108}	*n*-Propanol–conc. NH_4OH (2:1), *n*-Propanol–pyridine–water (5:3:3)
M_{109}	Ammonia buffer (pH 10) system
M_{110}	Methanol–benzene–EtOAC–EtCoMe–NH_3 (6:6:6:2:1)
M_{111}	Acetone–C_6H_6 (1:1)
M_{112}	Butanol–acetone–water (45:45:10)
M_{113}	Acetone–water (10:1); Methanol–butanol–water (3:1:1); Butanol standardized with 2 N HNO_3.
M_{114}	Butanol–propanol–water (1:3:1)
M_{115}	Polyhydric alcohols; Formamide; DMF; Methyl amine; Pyridine; Water; Methanol; Ethanol; Other alcohols; Ketones, Esters; Water–acetone (10:90, 20:80); Dioxane; 25% aq. ammonia; Water–methanol (5:95, 10:90); Water–ethanol (5:95, 10:90); Water–isopropanol (10:90, 20:80); Water–*tert*-butanol (20:80, 30:70); Water–$Me_2COCHCH_2COMe$ (20:80, 30:70); Water–pyridine (5:95, 10:90); Aq. ammonia–methanol (20:80, 40:60); Aq. ammonia–ethanol (20:80, 40:60); Aq. ammonia–isopropanol (20:80, 30:70); Aq. ammonia–*tert*-butanol (40:60, 60:40); Aq. ammonia–acetone (20:80, 30:70); Aq. ammonia–dioxane (40:60, 60:40); Aq. ammonia–DMF (20:80, 40:60, 60:40); Aq. ammonia–$(CH_2OH)_2$ (20:80, 40:60); Aq. ammonia–butanol (60:40, 80:20)
M_{116}	Acetone–butanol–10% NH_4OH–water (65:20:10:5)
M_{117}	Propanol–NH_4OH (2:1)
M_{118}	THF–acetic acid–water (10:1:1)
M_{119}	Acetone–butanol–conc. NH_3–water (3:4:2:1); Butanol–ethanol–conc. NH_3–water (2:2:1:1)
M_{120}	Pyridine–water–*n*-butanol–NH_4OH (20:40:40:5)
M_{121}	Toluene–acetic acid (10:2)
M_{122}	Aq. organic acid systems

(continued)

TABLE 2 Continued

Code	Mobile phases
M_{123}	HCl–acetone (1:9, 9:1); NaCl–acetone (1:9, 9:1); HBr–acetone (1:9, 9:1); NH_4OH–acetone (1:9, 9:1); FA–acetone (1:9, 9:1)
M_{124}	Propan-2-ol; butan-2-ol; and *t*-butanol; 10% solutions of DPA or DEAH in methanol; DMA and TEA; acetone; EMK; isobutyl methyl ketone and acetophenone; phenol; FA (22 M); FA (22 M) mixed with alcohols, amines, ketones in the ratio 1:9, 1:1, 9:1; DMA, TEA, *s*-butylamine; 10% methanolic solution of DPA; 10% methanolic solution of DEAH; isobutyl methyl ketone; EMK; acetophenone, propan-2-ol; butane-2-ol; *t*-butanol and phenol; 10% DPA or DEAH in methanol; or TEA or DMA–acetone or EMK or acetophenone–FA (5:35:60); 10% DPA or DEAH in methanol or TEA–isopropanol or isobutanol–FA (5:35:60); DMA–isopropanol or isobutanol–FA (5:35:60)
M_{125}	FA–acetone (1:9); DMSO–acetone (1:8, 3:6, 6:3); FA–DMSO–acetone (1:1:8, 3:1:6, 5:1:4); $H_2SO_4/HClO_4/HCl$–DMSO–acetone (1:1:8)
M_{126}	Distilled water
M_{127}	Acetone–chloroform (3:1); Acetone–0.1 M HCl (4:1)
M_{128}	0.1 M HCl–acetone (1:9); 1.0 M FA; 1.0 M sodium formate; Double distilled water

TABLE 3
Detection Reagents Used in TLC of Inorganic Anions

Code	Detection reagents
D_1	Aq. saturated $AgNO_3$ solution: AsO_2^-, $AsO_4^- \cdot H_2PO_4^-$, CrO_4^{2-}, I^-, Br^-, Cl^-, S^{2-}, $S_2O_3^{2-}$, CrO_4^{2-}
D_2	$FeCl_3$ (10%) solution containing 10 mL HCl (2 M): I^-, $H_2PO_4^-$, ClO_3^-
D_3	Sodium nitroprusside (20%) aq. solution: S^{2-}
D_4	KI (10%) solution containing 10 mL HCl (2 M): IO_4^-, IO_3^-, BrO_3^-, ClO_3^-, NO_2^-
D_5	$FeSO_4$ (10%) aq. solution containing 20 mL H_2SO_4 (2 M): SCN^-, CH_3COO^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$
D_6	Aq. saturated solution of $K_2Cr_2O_7$ containing 10 mL H_2SO_4 (2 M): AsO_2^-, SO_3^{2-}.
D_7	Mixture of 100 mL H_2O–saturated BuOH, 0.93 g PhNH, and 1.66 g *o*-phthalic acid and heated for 20 min at 130°C. Specific color for each anion has been reported.
D_8	Ammonical solution of $AgNO_3$ and fluorscein: I^-, Br^-, Cl^-
D_9	5% $AgNO_3$ followed by exposure to sunlight: I^-
D_{10}	5% KI followed by 6 N HCl: IO_3^-, IO_4^-, Te(IV)
D_{11}	10% $K_4[Fe(CN)_6]$ in 5 N HCl: MoO_4^{2-}, TeO_4^-
D_{12}	(KI solution + starch)–HCl solution: IO_3^-
D_{13}	$AgNO_3$ solution: I^-, RuO_4^{2-} gives dark spot itself
D_{14}	Autoradiography was also used
D_{15}	KI in 1 N HCl: IO_3^-, IO_4^-, BrO_3^-, CrO_4^{2-}
D_{16}	1% $FeCl_3$ solution in 2 M HCl: SCN^-, MoO_4^{2-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$

TABLE 3 Continued

Code	Detection reagents
D_{17}	Saturated solution of $AgNO_3$ in methanol: I^-, Br^-, Cl^-, IO_3^-, IO_4^-, VO_3^-, CH_3COO^-, $S_2O_3^{2-}$, CrO_4^{2-}, $Cr_2O_7^{2-}$
D_{18}	30 mL of 2% Ph_2NH solution mixed with 20 mL of 4 M H_2SO_4: NO_2^-, NO_3^-, IO_3^-, IO_4^-, BrO_3^-, WO_4^{2-}, VO_3^-, MnO_4^-
D_{19}	2% alcoholic solution of pyrogallol: MoO_4^{2-}
D_{20}	1% $BaCO_3$ solution followed by spraying with 2% phenolphthalein solution in methanol: SO_4^{2-}
D_{21}	Halide ions are detected by spraying with a 1% $AgNO_3$ solution, which prior to its use is mixed (ratio 9:1) with a solution of 0.5% of fluorescein + 0.5% of DCF containing a few drops of 4 N NH_3
D_{22}	0.04% bromocresol green in ethanol, made just basic with 0.1 N NaOH: Anions (acids)
D_{23}	0.1% bromocresol purple in ethanol made just basic with dil. NH_4OH: Anions (halides)
D_{24}	Mix equal volumes of 3.3% $AgNO_3$ in water and 0.3% sodium fluoresceinate in water. Examine in UV light: Anions
D_{25}	1% $(NH_4)_2MoO_4$ + 1% $ZnCl_2$ in 10% HCl: PO_4^{3-}
D_{26}	For visualization of the halides the plates were sprayed with an ethanolic solution of DCF (0.1%, w/v), and then oversprayed with $AgNO_3$ under UV light. The F^- appeared as a yellow spot and other halides as dark spots
D_{27}	Mixture of 5 g $(NH_4)_2MoO_4$, 420 mL H_2O, 50 mL 0.1 N HCl, and 25 mL 60% $HClO_4$. Plates were irradiated for 10–15 min with UV light with peak wavelength of 365 nm. Orthophosphate appeared as yellow spots, all other showed blue spots
D_{28}	Autoradiogram 200 mm by 50 mm detection. Kodak royal medical x-ray film. Time of exposure 24 to 48 h. 23°C
D_{29}	Molybdate solution (300 g $NaHSO_3$, 10 g Na_2SO_3, and 2 g metal dissolved in 1000 mL H_2O)
D_{30}	$(NH_4)_2MoO_4$ + $SnCl_2$
D_{31}	Detection in UV light, obtaining blue or white spots on a white background
D_{32}	Detection by molybdophosphate reaction followed by reduction
D_{33}	Detection by spraying with Hanes and Isherwood reagent [C. S. Hanes and F. A. Isherwood, *Nature*, *164*, 1107 (1949)]
D_{34}	$(NH_4)_2MoO_4$ solution
D_{35}	$AgNO_3$, DCF: BO_3^-, F^-, Cl^-, Br^-, I^-, $HAsO_4^-$, AsO_2^-, SO_3^{2-}, NO_2^-, $S_2O_3^{2-}$, CN^-, S^{2-}
D_{36}	$FeCl_3$: $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, SCN^-
D_{37}	$AgNO_3$: PO_4^{3-}, $S_2O_3^{2-}$, $Cr_2O_7^{2-}$
D_{38}	10% KI in 2 N HCl: BrO_2^-, IO_3^-, BrO_3^-, ClO_3^-, $S_2O_8^-$
D_{39}	DCF, $AgNO_3$, Kojic acid, *o*-coumaric acid: $C_2O_4^{2-}$, CO_3^{2-}
D_{40}	Kojic acid, *o*-coumaric acid, DCF, Laurents acid, heated DCF, Laurents acid, heat: CO_3^{2-}, SO_4^{2-}, NO_3^-
D_{41}	Molybdate–$HClO_4$ reagent, heated at 70°C, exposed to UV light. Blue spots of phosphates
D_{42}	1 volume 3.3% aq. $AgNO_3$ and 1 volume 0.3% Na salt of fluorescein and exposed to daylight or UV light: Sulfate yellow spots on a pink background

(continued)

TABLE 3 Continued

Code	Detection reagents
D_{43}	Alizarin–zirconium lake and $AgNO_3$, IO_3^-, and BrO_3^- (brown), ClO_3^- appeared as a light tan color in about 15 min. Using DCF the spots of $HAsO_4^{2-}$, SO_3^{2-}, and NO_2^- were tan, pink, and red, respectively. $AgNO_3$ gave pink color to AsO_2^-. Under UV light the AsO_2^- spot was yellow and the SO_3^{2-} spot became lighter in color. $C_2O_4^{2-}$ showed a red band. SO_4^{2-} (black band) and NO_3^- (light tan) were best detected by spraying with DCF and Laurents acid followed by heating for few minutes at 110°C. $AgNO_3$ and DCF gave visible pink to $S_2O_5^{2-}$ which later turned to reddish brown. $S_2O_8^{2-}$ formed a distinct visible brown on a yellow background with KI. A visible dark gray and dark brown detected the CN^- and S^{2-} with DCF, $AgNO_3$
D_{44}	1% aq. solution of $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ (ammonium heptamolybdate tetrahydrate) + 2% ascorbic acid in 10% TCA
D_{45}	254 nm UV indicator
	1% $AgNO_3$ solution: NO_3^- (brown), ClO_4^- (beige), S^{2-} (intense chestnut, disappears under UV), SCN^- (pale rose, violet gray under UV), CN^- (light brown, brown under UV), $Fe(CN)_6^{4-}$ (rose violet under UV), $Fe(CN)_6^{3-}$ (yellow, disappears under UV)
	Benzidine solution: NO_2^- (yellow), BrO_3^- (violet), ClO_3 (bright blue)
D_{46}	2540 Å UV light: Cl^- (purple), Br^- (gray), I^- (black), ClO_3^- (rose), BrO_3^- (deep red), IO_3^- (red)
D_{47}	1% $(NH_4)_2MoO_4$ in 0.6 M HCl, followed by 1% tin(II) chloride in 10% HCl: phosphate
D_{48}	1% $(NH_4)_2MoO_4$ in 0.125 M H_2SO_4, followed by saturated oxalic acid and 1% tin(II) chloride in 10% HCl: silicate
D_{49}	Methylene blue: $Re(IV)Cl_6^{2-}$, ReO_4^-
D_{50}	KI in 1 N to 10 N HCl: Halation of free iodine (dark brown)
D_{51}	Fluorescent morin–aluminum complex for detection. (Dissolve 5 g each of $AlCl_3$ and morin in a mixture of 10 mL of 30% acetic acid, 20 mL of 98% ethanol, and 20 mL of water. Viewed under UV light at 365 nm)
D_{52}	1% aq. solution of palladium chloride in HCl
D_{53}	Detection of anions by comparing the colors with a color table within 20 min. 13 spraying reagents were used
D_{54}	Spraying with a solution of Na_2MoO_4, NH_4NO_3, and HNO_3
D_{55}	Spraying with $(NH_4)_2MoO_4$ in HNO_3 followed by 1,2,4-aminonapthosulfonic acid in $NaSO_3$ solution
D_{56}	A coloring agent containing $SnCl_2$ was sprayed on the TLC plate
D_{57}	Indirect fluorometric detection
D_{58}	Radioactivity techniques: Cl^-, Br^-, I^-, AsO_3^{3-}, AsO_4^{3-}
D_{59}	10% $(NH_4)_2MoO_4$ followed by 10% $SnCl_2$ in 1 M HCl: phosphates
D_{60}	0.1% bromocresol purple in ethanol: OAc^-, HCO_2^-
D_{61}	5% KI in 1 M HCl: ClO_3^-, BrO_3^-, IO_3^-
D_{62}	10% $FeCl_3$: SCN^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$
D_{63}	1 M NH_3 solution saturated with H_2S
D_{64}	0.4% $(NH_4)_2MoO_4$ in 8% HNO_3 followed by 1% $SnCl_2$ in 10% HCl
D_{65}	1% aq. $FeSO_4$
D_{66}	1% aq. $Fe(NO_3)_2$

TABLE 3 Continued

Code	Detection reagents
D_{67}	5% $(NH_4)_2MoO_4$ solution in 50% HNO_3
D_{68}	2% $AgNO_3$: Cl^-, Br^-, I^-
D_{69}	1% bromocresol purple and NH_4OH: F^-
D_{70}	1% KI in 0.1 N HCl: ClO_3^-, BrO_3^-, IO_3^-
D_{71}	0.05% methylene blue: ClO_4^- (violet spot against blue background)
D_{72}	0.1% solution of congo red: BO_3^{3-}
D_{73}	0.2 N solution of $AgNO_3$: S^{2-}, CrO_4^{2-}, AsO_3^{3-}, AsO_4^{3-} (variation of spots from yellow to brown)
D_{74}	Chevalier indicator (40 mg methyl red, 80 mg bromomethyl blue, 80 mg PPL, 2 mL 0.1 N Na_2CO_3, 50 mL 95% ethanol, 50 mL H_2O): NO_2^-, SO_4^{2-}
D_{75}	Plates were sprayed with a reagent containing 5 mL 60% $HClO_4$, 10 mL HCl, and 25 mL 4% $(NH_4)_2MoO_4$ in 100 mL solution followed by spraying with a solution of 0.5% 2,4-diaminophenol–HCl and 5% Na_2SO_3. Polyphosphoric acids were indicated by blue spots on a yellow background
D_{76}	Detection by exposure to light.
D_{77}	Anions were identified partly in daylight and partly in UV light by spraying with $Fe(SCN)_3$, fluorescein–$CuSO_4$ or $AgNO_3$–fluorescein
D_{78}	Anion identification by R_F values and detection by spraying with following reagents. 0.1 N $AgNO_3$: Br^- (white), I^- (yellow). 2% $FeCl_3$: SCN^- (red), $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$ (blue), 2% $Pb(NO_3)_2$: ClO^- (yellow)
D_{79}	Spots sprayed with bromocresol purple and detected by UV light: ClO^-, ClO_2^-, ClO_3^-, ClO_4^-
D_{80}	$BaCl_2$ solution acidified with acetic acid and with sodium rhodizonate solution: SO_4^{2-}, SO_3^{2-} (pale yellow spots on a pink colored background)
D_{81}	0.1 mol/L $AgNO_3$ solution: polythionates and thiosulfate (dark brown spots)
D_{82}	Color change of complex by using strong base and acid confirms the presence of boron
D_{83}	Aq. solution of K_2S: Co(III) complex
D_{84}	Alcoholic solution of rubeanic acid: Cu(II), Ni(III) complexes
D_{85}	Rubeanic acid acidified with HCl and plate must be heated to 100°C: Pt(II) complex
D_{86}	0.1 N NH_3 solution of $AgNO_3$ or 0.1% solution of bromocresol green in 50% ethanol: halides and sulfate
D_{87}	Sorbent impregnated with Luminophor-L 36 and observed in UV light: polythionates
D_{88}	$AgNO_3$: S^{2-}. Fuchsine: SO_3^{2-}. $Cu(OAC)_2^-$–benzidine–methanol: CN^-
D_{89}	Detection by spraying with 5% $CuSO_4$ solution. NO_2^- appears as a green spot, and after heating OAc^- appears as a blue spot, and after extended heating, the $S_2O_3^{2-}$ appears as a dark brown, nearly black spot, and S^{2-} as a lighter shade. After very strong heating, the hypophosphite produces a reddish brown color. Spraying with 2% $FeCl_3$ solution revealed NO_2^- and OAc^- in the cold or with mild heating as a greenish blue color, and for a short time, $S_2O_3^{2-}$ and S^{2-} as a brownish red color which did not reappear even after strong heating
D_{90}	8% (w/v) $AgNO_3$ in acetone containing 10% (v/v) water
D_{91}	Scintillation counting when ^{35}S-labeled materials were used

(continued)

TABLE 3 Continued

Code	Detection reagents
D_{92}	GM counter for nonradioactive samples
D_{93}	5% aq. $FeCl_3$: I^-
D_{94}	10% HCl and 5% aq. KI solution: IO_3^-, IO_4^-
D_{95}	1% starch solution: I_2
D_{96}	1% aq. $KMnO_4$ solution: SO_3^{2-}, SO_4^{2-}
D_{97}	$AgNO_3$ + H_2S followed by removal of excess $AgNO_3$ with CO_3^{2-}-free H_2O
D_{98}	Aq. bromocresol green, adjusted to the transition point with NH_4OH, and appeared as pale spots on a blue green background: polythionates
D_{99}	Ammonical $AgNO_3$: sulfates
D_{100}	10% aq. KSCN and 10% $SnCl_2$ in HCl (1:1) (prepared fresh daily): SeO_3^{2} (red orange spot), MoO_4^{2-} (red), ReO_4^- (yellow)
D_{101}	Spot-test reagents
D_{102}	Bromocresol purple: halides
D_{103}	α-Naphthylamine-sulfanilic acid (1:1): NO_2^-
D_{104}	Fluorescein solution and a mixture of acetic acid and 30% H_2O_2: Br^- (bright red spot appears on a yellow background)
D_{105}	Radiometric detection
D_{106}	0.2% Ph_2NH in H_2SO_4: All anions reported appear as blue spots
D_{107}	$Cu(OAC)_2$, benzidine acetate, and KBr
D_{108}	Ammonium ferric sulfate: thiocyanate and selenocyanate
D_{109}	Tolidine-HCl: BrO_3^-
D_{110}	Benzidine in 2 N acetic acid: $Fe(CN)_6^{4-}$
D_{111}	Aq. $FeCl_3$ solution: $Fe(CN)_6^{3-}$
D_{112}	Detection at 250 nm with a dual wavelength densitometer
D_{113}	Alizarin, $AgNO_3$, and $MnSO_4$ in H_2SO_4; KI in 2 M HCl; and $FeSO_4$ + $FeCl_3$ were used for detection
D_{114}	Marshall's reagent [sulfanilic acid/(naphthyl) ethylenediamine]: NO_2^- (intense red purple zone).
D_{115}	Aq. 1% $K_4Fe(CN)_6^{4-}$: $C_2O_4^{2-}$
D_{116}	Chromatographic zones highly colored (Fe complexes) or fluorescent under UV light: terbium complexes

TABLE 4
Chromatographic Systems Used for Analytical Studies

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
S_1	M_1	AsO_2^-, AsO_4^{3-}, $H_2PO_4^-$, CrO_4^{2-}, $S_2O_3^{2-}$, Cl^-, Br^-, I^-, IO_4^-, IO_3^-, BrO_3^-, NO_2^-, ClO_3^-, $Fe(CN)_6^{3-}$, $[Fe(CN)_6]^{4-}$, CH_3COO^-, CNS^-, SO_3^{2-}	D_1-D_6	Saturated chamber. Run 1.5 cm in 1-2 min. Circular development	27, 28

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
S_2	M_2	IO_3^-, IO_4^-, XeO_3	—	Run 18–20 cm. The method is used to separate carrier-free amounts of $^{131m}XeO_3$ from parent $K^{131}IO_4$ and also to obtain $^{133}XeO_3$ from neutron-irradiated sodium perxenate	29
S_3	M_3	ClO_4^-, ClO_3^-, ClO_2^-, ClO^-, BrO_3^-, IO_3^-, BrO_2^-, BrO^-	D_7	Run 10 cm in 40–50 min. ClO^- and BrO^- decompose in NH_3	30
S_4	M_4	Cl^-, Br^-, I^-	D_8	Run 16 cm. Slurry prepared with dil. HNO_3	31
S_5	M_5	CrO_4^{2-}, Cl^-, Br^-, I^-, BrO_3^-, ClO_3^-, $Fe(CN)_6^{4-}$, $Fe(CN)_6^{3-}$, SCN^-, AsO_3^{2-}, SO_3^{2-}	—	Temp. 30–32°C. Circular development. The solution is diluted until its color after TLC matches that of a standard: accuracy ±5%	32
S_6	M_6	NO_3^-	—	Saturated chamber. Run 15 cm, The method is applied to the determination of nitrate in feeds. The sample solution is applied as a continuous band. After TLC, the nitrate-containing zone is scraped off and reacted with 3,4-xylenol in sulfuric acid medium. The reaction product is extracted with petroleum ether and subsequently the organic layer is shaken with aq. alkali. After separation of the layers, the absorbance of the aq. layer is determined at 430 nm	33
S_7	M_7	I^-, IO_3^-, IO_4^-, Te(IV)	D_9, D_{10}	Run 10 cm	34
S_8	M_7	MoO_4^{2-}, TeO_4^-, IO_3^-, I^-, RuO_4^{2-}	D_{11}, D_{14}	Ascending technique. Run 10 cm	35
S_9	M_9	IO_3^-, BrO_3^-, CrO_4^{2-}, IO_4^-, $Cr_2O_7^{2-}$	D_{15}	Developed for 30 min. Effect of pH and salt concentration is discussed and the adsorption is compared with the tendency for the formation of ion pairs between anions and Al^{3+} as observed in paper electrophoresis	36
S_{10}	M_{10}	$Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, SCN^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, I^-, Br^-, Cl^-, PO_4^{3-}, VO_3^-, WO_4^{2-}, IO_3^-, IO_4^-, NO_2^-, NO_3^-, BrO_3^-, MoO_4^{2-}, SO_4^{2-}, $S_2O_3^{2-}$, CH_3COO^-	D_{16}–D_{20}	Ascending technique. Run 10 cm. Plates activated at 100 ± 5°C. Mutual separation of I^-, Br^- Cl^-, NO_2^-, and NO_3^- in the presence of transition metals has been studied	37
S_{11}	M_{11}	Cl^-, Br^-, I^-	D_{21}	Accommodated plate. Hellendahl jar. Run 3 cm in 10–20 min. Number of thin-layer separations	38

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				have been reported which can be used to introduce chromatography in the teaching laboratory. Mostly cations have been chromatographed along with a few anions	
S_{12}	M_{12}	F^-, PO_4^{3-}, SO_4^{2-}, SO_3^{2-}, Cl^-, NO_3^-	—	TLC is used as a method of water analysis. Cations were also chromatographed	39
S_{13}	M_{13}	PO_4^{3-}, SO_4^{2-}, CO_3^{2-}	D_{22}–D_{25}	Saturated chamber. Run 10 cm in approx. 30 min. Cations were also chromatographed. Marked differences in R_F values upon temp. change	40
S_{14}	M_{14}	F^-, Cl^-, Br^-, I^-	D_{26}	Run 14 ± 0.5 cm in 30–40 min. Separation of halides from cations has been studied; it is due to cation exchange on the cellulose. F^- appears as a yellow spot, while other halides appear as dark spots. Na^+ and NH_4^+ appear as diffused yellow spots, while K^+ appears as a light brown spot	41
S_{15}	M_{15}	Linear, octa-, trimeta-, tetrameta-, and cyclic phosphates	—	Run 1.5–2 h	42
S_{16}	M_{16}	PO_4^{3-}, $P_2O_7^{4-}$, $P_3O_{10}^{5-}$, $P_4O_{13}^{6-}$, $P_5O_{16}^{7-}$, $P_6O_{19}^{8-}$, $P_7O_{22}^{9-}$, $P_8O_{25}^{10-}$, $P_3O_9^{3-}$, $P_4O_{13}^{4-}$	—	Run 14 cm in 1.5–2 h. Saturated chamber. An extensive study is made of the influence of the composition of the mobile phase on the efficiency of the separation	43
S_{17}	M_{17}	Ortho-, pyro-, and hexametaphosphates. All samples show presence of traces of other phosphates	—	Run 15 cm in 100 min. TLC and subsequent semiquantitative determinations serve as guides to the use of optimum sample size for quantitative analysis on ion-exchange columns. The method is applied to the analysis of tetracycline preparations	44
S_{18}	M_{18}	Ortho-, pyro-, tri-, and tetra-phosphates	—	Run 14 cm in 25–34 min or 39–65 min. R_F values of phosphates as a function of layer thickness (mg/cm^2), time of run (min), and water content have been plotted	45
S_{19}	M_{19}	Ortho-, pyro-, tripoly-, trimeta-, and tetrametaphosphates, hypophosphite, phosphite, hypophosphate,	D_{27}	Saturated chamber. Run 6–7 cm in 1 h. Temp. 19.5–20.5°C	46

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
		commercial sodium metaphosphate glass, commercial sodium hexametaphosphate glass, sodium polyphosphate glass (n = 20–30)			
S_{15}	M_{20}	Ortho- and pyrophosphates	D_{28}	Run 13.5 cm in 1 h. Temp. 23°C. Kodak royal medical x-ray film for detection, time of exposure 24 to 48 h	47
S_{15}	M_{21}	Mono-, di-, and triphosphates	D_{29}	Run in 90 min. Separation of phosphates is successful. Short time of run makes it superior to PC	48
S_{20}	M_{22}	Ortho-, pyro-, trimeta-, tetrameta-, pentameta-, hexameta-, heptameta-, octameta-, tripoly-, tetrapoly-, pentapoly-, and hexapolyphosphates	D_{30}	Saturated chamber. Run 10 cm. Temp. 5°C	49
S_{21}	M_{23}	Ortho-, pyro-, tripoly-, trimeta-, tetrameta-, pentameta-, hexameta-, heptameta-, octameta-, nonameta-, and decametaphosphates	D_{30}	Saturated chamber. Run 10 cm. Temp. 5°C	50
S_{22}	M_{24}	Ortho-, pyro-, tripoly-, trimeta-, tetrameta-, and hexametaphosphates	D_{30}	Saturated chamber. Run 10 cm. Temp. 5°C	51
S_{23}	M_{25}	Diamido- and triamidophosphates	D_{31}	Plates accommodated for 45 min. Run 17 cm in 20–30 min (isothermal) or 20–70 min (gradient). Chromatography carried out under isothermal conditions (20–70°C) and at a temperature gradient (0.5–2.7% cm)	52
S_{24}	M_{26}	Na_3PO_4, $Na_4P_2O_7$, $Na_5P_3O_{10}$	D_{29}	Saturated chamber. Run 10 cm in 100 min. Semiquantitative determination of the phosphates is carried out by comparing color intensity and size of the spots with those of standard substances	53
S_{25}	M_{27}	Ortho-, pyro-, tripoly-, trimeta-, and tetrametaphosphates	D_{29}	Run 15 cm in approx. 55 min. For quantitative determinations the appropriate part of the cellulose layer is scraped off and treated with 0.1 N H_2SO_4. After filtration and addition of molybdate reagent, photometry	54

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				is carried out at 822 nm. TLC is applied in a study on the use of phosphates as melting agents in processed cheese	
S_{26}	M_{28}	Mono-, di-, tri-, tetra-, penta-, hexa-, hepta-, octa-, trimeta-, tetrameta-, pentameta-, hexameta-, and heptametaphosphates	D_{32}	Run 16 cm in approx. 60 min. Two-dimensional TLC. M_2 and M_4 yield successful separation of monohexa-, trimeta-, and heptametaphosphates	55
S_{27}	M_{29}	Mono-, hexa-, tri-, and tetrametaphosphates	D_{32}	Run 16 cm in approx. 75 min. TLC separation of phosphates. Phosphate-containing zones are removed from the thin layer. The organic material is destroyed by treatment with $HClO_4$ and the phosphates are hydrolyzed. Color development is done using molybdate and hydrazine sulfate, and photometry is carried out at 825 nm	56
S_{28}	M_{30}	Ortho-, poly-, tripoly-, trimeta-, tetrameta-, and hexametaphosphates	D_{33}	Run 10 cm in 30 min. Temp. 22–25°C. The method can be used to estimate the relative chain-length of unknown polyphosphates. The TLC system described in this paper has been used to separate polyphosphates formed by caries-conducive streptococcus SL-1 in J. M. Tanzer and M. I. Krichevsky, *Biochem. Biophys. Acta*, *215*, 368 (1970)	57
S_{29}	M_{31}	Polycondensed phosphates	D_{34}	Saturated chamber. Run 12–14 cm in 1.5 or 5 h. TLC and TLEP of phosphates. Polyzonal analysis is helpful for the elucidation of phase formation of eluent components	58
S_{30}	M_{32}	Mono-, tri-, and tridecaphosphates	D_{34}	Saturated chamber. Run in 4 h (ascending on normal plate) or in up to 12 h (ascending on wedge-shaped plate). TLC and TLEP of phosphates	59
S_{15}	—	Mono-, di-, and triphosphates	D_{34}	TLEP separation of phosphates at 400 V. TLC separation of polyphosphates at right angles to the prior electrophoresis	60
S_{31}	M_{33}	Mono, di, tri, trimeta, and tetrameta long-chain phosphates	—	Run in 60 min (ascending) or 90 min (radial or circular). Separation of food colors,	61

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				phenolic compounds, catechnol amine and serotonin metabolites; narcotics also studied. Techniques are useful for separating large amounts of a substance in the presence of small amounts of second substance. R_F values are larger than those with ascending chromatography. Circular R_F values are equal to the square root of the corresponding ascending R_F values	
S_{32}	M_{34}	Ortho-, pyro-, tripoly-, trimeta-, and tetrametaphosphates	D_{30}	Run 10 cm in 15–80 min or in 1–6.5 h. Closed chamber. When acidic developing solvents were used, the order of R_F values was ortho- > poly- > metaphosphate, and when basic solvents were used the order of R_F values was meta- > ortho- > polyphosphate	62
S_{33}	M_{35}	BO_3^-, F^-, Cl^-, Br^-, I^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, SCN^-, BrO_2^-, IO_3^-, BrO_3^-, ClO_3^-, PO_4^{3-}, $S_2O_3^{2-}$, $Cr_2O_7^{2-}$, $HAsO_4^{2-}$, AsO_2^-, SO_3^{2-}, NO_2^-, $C_2O_4^{2-}$, CO_3^{2-}, SO_4^{2-}, NO_3^-, $S_2O_5^{2-}$, $S_2O_8^{2-}$, CN^-, S^{2-}	D_{35}–D_{40}	Run in 20–70 min. Microcrystalline cellulose can be successfully used to separate a large number of inorganic anions. The method has been applied to both thin-layer and column chromatography. The TLC plates gave a remarkably hard surface which stands up in a variety of solvents	63
S_{34}	M_{36}	Metaphosphimates, imidodi-, mono-, diamidotrimeta-, and imidotrimetaphosphates; sulfate, thiosulfate, trithionate, tetra- and pentathionates	D_{41}, D_{42}	Saturated chamber. Run 15 cm. Comparison of TLC and PC for the separation of inorganic compounds. Layer thickness 250 μm on 20 × 20 cm (for P-species) or 10 × 20 cm (for S-species) glass plates	64
S_{35}	M_{37}	F^-, Cl^-, Br^-, I^-	D_{43}	Ascending technique. Run 15 cm. 0.25 mm thick layers	65
S_{15}	M_{38}	Mono-, di-, tri-, tetra-, penta-, hexa-, hepta-, octa-, and polyorthophosphates	—	Relationship between the chromatographic behavior and the structural units of polyorthophosphates has been examined	66
S_{36}	M_{39}	Ortho (Pi), pyro (PPi), and tripoly (PPPi) phosphates	D_{44}	Work on the hydrolysis of various phosphates, particularly PPi and PPPi, as catalyzed by an inorganic redox system. A simple, rapid technique for the	67

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				separation and identification of Pi, PPi, and PPPi has been proposed	
S_{37}	M_{40}	NO_2^-, NO_3^-, ClO_3^-, ClO_4^-, BrO_3^-, S^{2-}, SCN^-, CN^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{45}	TLC separation and identification. Layer thickness = 500 μm. Run in 1.5–3 h	68
S_{38}	M_{41}	$C_2O_4^{2-}$, tartrate, F^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, IO_3^-, AsO_4^{3-}, AsO_3^{3-}, PO_4^{3-}, I^-, SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$	—	TLC separation and identification. In each case the influence of various solvent mixtures as well as the limiting concentrations on chromatographic development was studied	69
S_{38}	M_{42}	Cl^-, Br^-, I^-, ClO_3^-, BrO_3^-, IO_3^-	D_{46}	Ascending technique. TLC separation. The addition of TEA to the cellulose resulted in an increase in development and a decrease in separation efficiency. The optimum concn. for separation was 0.5–1% anion	70
S_{39}	M_{43}	PO_4^{3-}, SiO_4^{2-}	D_{47}, D_{48}	Run 10 cm in 1 h. Layer thickness 0.6 mm. SiO_3^- and phosphates are mutually noninterfering. Polymers of H_2SiO_3 are not detected. The detection limit of SiO_3 is 0.006 μg; of SiO_2 0.012 μg/cm^2	71
S_{40}	M_{44}	ReO_4^-, $Re(IV)Cl_6^{2-}$	D_{49}	Ascending technique. Studies on valancy states of rhenium	72
S_{41}	M_{45}	BrO_3^-, IO_3^-, IO_4^-, BrO_4^-	D_{50}	Saturated chamber. Run 9 cm in 2 h. TLC and electrophoresis of halates	73
S_{15}	M_{46}	ClO_4^-, ClO_3^-, ClO_2^-, BrO_3^-, IO_3^-, IO_4^-, SO_3^{2-}, SO_4^{2-}, PO_4^{3-}, BO_3^{3-}, $B_4O_7^{2-}$, NO_3^-, NO_2^-, SiO_3^{2-}, F^-, Cl^-, Br^-, I^-	—	TLC separation of oxyanions and halogens. Halogen ions readily solvated by a proton-donating solvent in the mobile phase, gave smaller R_F values owing to the dipole–dipole interactions	74
S_{15}	—	Various phosphates	—	Influence of cations on the separation of phosphates. Cu, Zn, Ca, Mg, Al, and Fe(III) ions distort the chromatogram for separation of P oxo-anions by TLC. The possibility of the analysis of samples containing large amount of Al(III) and Fe(III) was studied	75
S_{15}	M_{47}	PO_4^{3-}, $P_2O_7^{4-}$, $P_3O_{10}^{5-}$	—	Interference of Mo in the detection of phosphates by PC and TLC was studied. Mo(V) ⩾ 0.09 and Mo(VI) ⩾ 1.2 mg/mL interfere in the analysis of solutions containing ~3 mg P/mL.	76

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
S_{42}	M_{48}	ClO_2^-, ClO_3^-, ClO_4^-, BrO_3^-, IO_3^-, IO_4^-, SO_4^{2-}, PO_4^{3-}, BO_3^{3-}, $B_4O_7^{2-}$, SiO_3^{2-}, NO_2^-, NO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$	D_{51}	Saturated chamber. Run 10 cm. 15 inorganic oxyanions, e.g., PO_4^{3-}, $B_4O_7^{2-}$, and NO_2^-; 5 hexose phosphate, 6 hexose sulfates, adenosine 5-mono-, di- and triphosphates; and 2 steroid sulfates were separated by TLC using various solvents	77
S_{43}	M_{49}	F^-, Cl^-, Br^-, IO_3^-, IO_4^-, BrO_3^-, ClO_2^-, ClO_3^-, ClO_4^-, SO_3^{2-}, SO_4^{2-}, S^{2-}, $Si_2O_3^{2-}$, SCN^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, CrO_4^{2-}, $Cr_2O_7^{2-}$, NO_2^-, NO_3^-, PO_4^{3-}, WO_4^{2-}, MoO_4^{2-}, VO_3^-, VO_4^{3-}, AsO_2^-, AsO_4^{3-}, SeO_3^{2-}, SeO_4^{2-}	D_{51}	Ascending technique. Run 10 cm. TLC of inorganic compounds. The R_F values become higher as the electronegativity of the halogen decreases or as the ionic radius increases	78
S_{44}	M_{50}	Mono- di-, tri-, tetra-, penta-, hexa-, cyclotri-, and cyclotetraphosphates	D_{33}	Run 10 cm (circular) or 15 cm (ascending) in 6–8 h. Complex mixtures of condensed phosphates were separated. Circular TLC provides better separations	79
S_{45}	M_{51}	Polyhedral borane anions	D_{52}	Ascending technique	80
S_{15}	M_{52}	26 anions	D_{53}	Anion separation by TLC on plates the size of microscope slides	81
S_{15}	M_{53}	Monofluorophosphate	D_{54}	Na_2PO_3F is detected in extracts of toothpastes by TLC. The sensitivity was ≥ 5 μg/spot. Spot measured by planimetry, Method is suitable for quality control of toothpastes containing Na_2PO_3F	82
S_{46}	M_{54}	Polyphosphates, condensed phosphates	D_{55}	Run 10–13 cm in 2–3 h. Detection of polyphosphates in fish products with TLC. Detection limit was 1 mg/mL for poly- and triphosphates and 4 mg/mL for hexametaphosphate	83
S_{15}	M_{55}	Polyphosphate	D_{56}	Determination of polyphosphate in meat products by TLC. Recovery rate was 93.3–98.1%. Ham samples contained 0.42–0.63 mg polyphosphate and 0.80–1.18 mg phosphate/g. Light scanning at 780 nm was used to quantitate polyphosphate	84
S_{15}	M_{56}	NO_2^-, I^-, S^{2-}, IO_3^-, S^-, cetrimide	D_{57}	Nonabsorbing and nonfluorescing anions. Anions in the low nanogram level can be recognized visually by using a hand-held UV lamp of excitation	85

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
S_{47}	M_{57}	I^-, Br^-, Cl^-, IO_3^-, SCN^-, BrO_3^-, ClO_3^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, AsO_3^-, AsO_4^{3-}, $P_2O_7^{4-}$, PO_4^{3-}, $H_2PO_2^-$, tripoly P	D_{58}–D_{62}	TLC on ion-exchange resins. Several separations of anions from cations are reported. TLC on flexible plastic supports; realization of new analytical applications	86–93
S_{48}	M_{58}	Oligo- and amidophosphates	—	Run in 70 min. Room temperature for oligo and 5°C for amidophosphates. The procedure reportedly couples the advantages of TLC with those of ion-exchange techniques	94
S_{49}	M_{59}	17 anions	—	Qualitative separations	95
S_{50}	M_{60}	$H_2PO_4^-$, SO_4^{2-}	D_{14}	Run 0.6–1.0 cm in 110–120 s. Thin-film sorptography allows the separation of minute amounts (10^{-7}–10^{-14} g) of substances	96
S_{51}	M_{61}	$H_2PO_4^-$, $H_2P_2O_7^{2-}$, $H_2PO_3^-$, $H_2PO_2^-$, SeO_3^{2-}, AsO_2^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, IO_3^-, BrO_3^-, $Cr_2O_7^{2-}$, SCN^-, I^-, Br^-	D_{42}, D_{61}, D_{63}–D_{66}	After their preparation, all layers are allowed to stand for 4 h in the buffer solution subsequently used as the mobile phase. After equilibration, the plates are rinsed with water and dried at room temperature	97
S_{52}	M_{62}	Polyphosphates	D_{67}	Photodensitometric determination of the phosphates is superior to photometric determination. The method is applied to the analysis of food additives after fractionation by TLC	98
S_{53}	—	Ortho-, pyro-, tripoly-, and tetrapolyphosphates	—	Determination of the content of condensed phosphates in liquid commercial fertilizers of the polyphosphate type. Quick development time as compared to PC. High reproducibility of results has been reported	99
S_{54}	M_{63}	F^-, Cl^-, Br^-, I^-, ClO_3^-, BrO_3^-, IO_3^-, ClO_4^-, IO_4^-	D_{68}–D_{71}	Run 15 cm in 90 min. Ascending TLC; about 10^{-9} mole of the ions was detectable	100
S_{55}	M_{64}	SCN^-, NO_3^-, NO_2^-, N_3^-, $S_2O_3^{2-}$, S^{2-}, SO_4^{2-}, CrO_4^{2-}, BO_3^{3-}, AsO_4^{3-}, PO_4^{3-}, AsO_3^{3-}, CN^-	D_{72}–D_{74}	Run 15 cm in 90 min. TLC separation of anions	101
S_{56}	M_{65}	ONC^-, N_3^-, tricinate	—	Saturated chamber. Run 10 cm in 15–55 min. Temp. 20°C. TLC is used for the analysis of the detonators lead azide, mercury fulminate, and lead tricinate	102
S_{57}	M_{66}	Phosphates	D_{75}	Identification and determination of phosphates in dairy products by	103

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				TLC. Recoveries were 82.7 to 95.9%	
S_{58}	M_{67}	I^-, Br^-, Cl^-, NO_2^-, NO_3^-, IO_3^-, MnO_4^-, SCN^-, SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, SeO_3^{2-}, TeO_3^{2-}, MoO_4^{2-}, $Cr_2O_7^{2-}$, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, PO_4^{3-}	—	Radioinorganic TLC	104
S_{59}	M_{68}	Cl^-, PO_4^{3-}, SO_4^{2-}	—	Separation of reaction products of neutron-irradiated NH_4Cl. TLC, radiochemistry	105
S_{60}	M_{69}	I^-, IO_3^-, IO_4^-	D_{76}	Saturated chamber. Development immediately after application of the spots; plates protected from light. Separation of various oxidation stages of iodine by TLC and electrophoresis	106
S_{61}	M_{70}	F^-, Cl^-, Br^-, I^-, SCN^-, N_3^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{77}	Saturated chamber. Run 10 cm in 55–95 min. Separation of halides and pseudohalides by TLC	107
S_{62}	M_{71}	Cl^-, Br^-, I^-, CNS^-, ClO^-, CN^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{78}	Run 5.5 cm. Temp. 24 ± 1°C. Study of anions from Group I of Treadwells scheme by TLC and spot tests	108
S_{63}	M_{72}	I^-, Br^-, Cl^-, PO_4^{3-}	—	Plates accommodated to water vapor. Horizontal and ascending development on 2.5 mm-wide thin-layer strips. Quantitative analysis of I^-, Br^-, Cl^-, and PO_4^{3-} by TLPC. Sequence of anion containing bands: I^- band $<$ Br^- band $<$ Cl^- band $<$ PO_4^{3-} band	109–111
S_{64}	M_{73}	F^-, Cl^-, Br^-, I^-	—	Run in 30–40 min	112
S_{59}	M_{74}	Cl^-, ClO_2^-, ClO_3^-, ClO_4^-	—	Run in 50–60 or 70–80 min. The methods are used to study the chemical fate of Na pyrophosphate and Na salts of chloro(oxy) acids upon neutron irradiation and the influence of the presence of metal ions	113
S_{65}	M_{75}	I^-, IO_3^-	—	Run 12 cm in 40 min	114
S_{66}	M_{76}	ClO^-, ClO_2^-, ClO_3^-, ClO_4^-	D_{79}	Separation and identification by TLC and PC. Attempts to separate the anions by PC were not successful	115
S_{67}	M_{77}	$S_2O_3^{2-}$, $S_3O_6^{2-}$, $S_4O_6^{2-}$, $S_5O_6^{2-}$, $S_6O_6^{2-}$	D_{80}, D_{81}	Saturated chamber. Run in 2–2.7 h, ascending technique. The method is applied to semiquantitative determination of low valence sulfur compounds such as polythionates by TLC	116

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				and colorimetry with methylene blue. Layer thickness ~300 μm, on 200 × 200 × 3 mm glass plates	
S_{59}	M_{78}	SO_3^{2-}, SO_4^{2-}, $S_2O_3^{2-}$, $S_2O_8^{2-}$, $S_2O_6^{2-}$, $S_3O_6^{2-}$, $S_4O_6^{2-}$, $S_5O_6^{2-}$	—	Run in 45 min. TLC separation of sulfates and polythionates	117
S_{68}	M_{79}	Phosphite, phosphate, pyrophosphate, hypophosphate	—	Run 14 cm in 30–240 min. Separation of various phosphoric acid ions by TLC	118
S_{69}	M_{80}	Ortho-, tripoly-, and polymetaphosphates; Na and K salts	—	Run 10 cm in 45 min. Elongated spots are obtained with all phosphates. A reliable conclusion regarding the presence or absence of a single component in a two- or three-component mixture is sometimes possible	119
S_{70}	M_{81}	$H_2P_2O_7$, H_2PO_4, H_2PO_3, H_2PO_2	—	Run in 50–60 min	120
S_{71}	M_{82}	Boron	D_{82}	Saturated chamber. Run in 10 min. Colorimetric determination and TLC identification of boron as boric acid in caviar in 0–5 μg range. Aq. extract of caviar is made alkaline and evaporated to dryness. The residue is dissolved in curcumin solution treated with acetic acid–H_2SO_4 (1:1) and diluted with alcohol	121
S_{69}	M_{83}	NO_2^-, NO_3^-, $CHCl_2COO^-$, CH_2ClCOO^-, CH_3COO^-, ClO_3^-, ClO_4^-, Cl^-, Br^-, I^-, CO_3^{2-}, SO_4^{2-}, SO_3^{2-}	—	Run 12–13 cm. Temp. 17–18°C. The R_F values are considered to give a relative measure of the degree of hydration of the various anions. No special detection procedure for spot, as it was sufficiently colored. Layer thickness ~0.25 mm on 20 cm glass plates. Object of this study was to clarify the effect of hydration of anions on ion-pair formation by means of chromatography of the dyestuff	122
S_{69}	M_{84}	Cl^-, Br^-, I^-, NO_2^-, NO_3^-, $HCOO^-$, CH_3COO^-, OCN^-, SCN^-, $SeCN^-$, SO_4^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, H_2O	D_{83}–D_{85}	Temp. 20–23°C. TLC of inorganic salts. Behavior of square planar complexes in comparison with that of octahedral complexes. Layer thickness ~0.25 mm on 20 cm glass plates	123
S_{72}	M_{85}	Cl^-, Br^-, I^-, S^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, $S_2O_8^{2-}$, $S_3O_6^{2-}$, $S_4O_6^{2-}$, $S_5O_6^{2-}$, $S_6O_6^{2-}$,	D_{86}, D_{87}, D_{59}	S- and BN-chambers. Run in 30–40 min. Separation of anions by TLC	124

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
		$S_7O_6^{2-}$, PO_3^-, PO_4^{3-}, $P_2O_7^{4-}$			
S_{73}	M_{86}	I^-, Br^-, Cl^-, F^-, CN^-, NO_2^-, NO_3^-, IO_3^-, SCN^-, ClO_3^-, BrO_3^-, BO_2^-, S^{2-}, SO_3^{2-}, SO_4^{2-}, $S_2O_3^{2-}$, CrO_4^{2-}, $C_2O_4^{2-}$, PO_4^{3-}, AsO_4^{3-}, AsO_3^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{88}	Run 10 cm in 10 or 35 min. CN^- is separated as $Hg(CN)_2$ ($R_F = 0.65$) from all other anions ($R_F = 0.00$) in the system	125
S_{74}	M_{87}	I^-, Br^-, Cl^-, F^-, OH^-, NO_2^-, NO_3^- IO_3^-, SCN^-, ClO_3^-, BrO_3^-, BO_2^-, SO_4^{2-}, $S_2O_3^{2-}$, CrO_4^{2-}, $C_2O_4^{2-}$, AsO_3^{3-}, AsO_4^{3-}, PO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	—	Plate accommodated for 30 min. Run 13 cm in 20, 65, or 80 min	126
S_{62}	M_{88}	NO_2^-, CH_3COO^-, $H_2PO_2^-$, $S_2O_3^{2-}$, S^{2-}	D_{89}	Run 5 cm in 30 min. Temp. 21°C	127
S_{75}	M_{89}	I^-, Br^-, Cl^-, SO_3^{2-}, $S_2O_3^{2-}$, S^{2-}, $S_2O_6^{2-}$, $S_3O_6^{2-}$, $S_4O_6^{2-}$, $S_5O_6^{2-}$, $S_6O_6^{2-}$, $S_7O_6^{2-}$, $S_8O_6^{2-}$	D_{86}, D_{87}	Combined C- and BN-cells and glass tanks with ground caps. Run 10 cm in 30–40 min. Electrophoresis and TLC of organic base polythionates	128
S_{76}	M_{90}	I^-, Br^-, Cl^-, F^-, IO_3^-, IO_4^-, ClO_3^-, BrO_3^-, AsO_4^{3-}, CrO_4^{2-}, $S_2O_3^{2-}$	—	Sandwich chamber. Run 10 cm in 24–27 min	129
S_{77}	M_{91}	I^-, Br^-, Cl^-, $CNSe^-$, SCN^-, IO_4^-, BrO_3^-, ClO_4^-, IO_3^-, BrO_3^-, ClO_3^-	—	Adsorption of inorganic anions	130
S_{78}	M_{92}	CN^-, SCN^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{16}	Foils not activated. TLC separation and detection of pseudodohalides on ready-made foils	131
S_{79}	M_{93}	SO_4^{2-}, $S_2O_6^{2-}$, $S_2O_8^{2-}$, $S_3O_6^{2-}$, $S_4O_6^{2-}$, $S_5O_6^{2-}$, $S_6O_6^{2-}$, SCN^-	D_{14}, D_{90}, D_{91}	S-chamber, run 10 cm. Gelman ITLC chambers, run 13 cm. Ascending technique, run 2–2.7 h. $H_2S_2O_6$ is separated from H_2SO_4 by TLC on extremely thin layers (≤ 90 μm)	132, 133
S_{80}	M_{94}	I^-, IO_3^-, IO_4^-, SO_3^{2-}, SO_4^{2-}, I_2	D_{92}–D_{96}	Ascending technique. Run 7 cm. Application of TLC to radiochemical inspection of iodide and sulfite	134
S_{81}	M_{95}	I^-, SCN^-, ReO_4^-, $Cr_2O_7^{2-}$, $Co(NH_3)_6^{3+}$, $HAuCl_4$	—	R_F calculated by *ds*/*dps*, where *ds* and *dps* are the distances traveled by the sample and Dextran Blue, respectively	135
S_{82}	M_{96}	I^-, Br^-, Cl^-, ClO^-, ClO_3^-, ClO_4^-, IO_3^-, IO_4^-, BrO_3^-, TeO_3^{2-}, SeO_3^{2-}, CrO_4^{2-}, $Cr_2O_7^{2-}$, S^{2-}, $S_2O_3^{2-}$, $S_4O_6^{2-}$, SO_4^{2-}, $H_2PO_2^-$,	—	TLC separation of anions. The simplicity of the eluents ensures good reproducibility	136

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
		NO_3^-, NO_2^-, AsO_3^{3-}, CN^-, SCN^-			
S_{83}	M_{97}	CN^-, OCN^-	D_{97}	Identification and separation of ions by TLC and PC. Ascending technique. R_F: $CN^- = 0.09$, $OCN^- = 0.29$	137
S_{84}	M_{98}	S^{2-}, $S_2O_3^{2-}$, SO_3^{2-}, $S_2O_8^{2-}$, SO_4^{2-}, $S_2O_5^{2-}$	D_{98}, D_{99}	The relative mobilities decreased in the order $SO_3^{2-} > S_2O_8^{2-} > S_2O_5^{2-}$. TLC of sulfates and polythionates	138
S_{85}	M_{99}	SeO_3^{2-}, MoO_4^{2-}, ReO_4^-	D_{100}	Ascending TLC. Run 10 ± 0.5 cm. Layer thickness 250 μm. Anions determined colorimetrically. Influence of solvent composition on the R_F values of anions has been reported	139
S_{86}	M_{100}	VO_3^-, IO_4^-, IO_3^-, I^-, Br^-, Cl^-, BrO_3^-, ClO_3^-, BrO_4^-, NO_2^-, SCN^-, AsO_2^-, ReO_4^-, SeO_3^{2-}, TeO_4^{2-}, TeO_3^{2-}, MoO_4^{2-}, CrO_4^{2-}, $S_2O_3^{2-}$, PO_4^{3-}, AsO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{101}	Ascending technique. Run 15 cm. Wide-mouthed glass jars closed with rubber stoppers. PC on Whatman No. 1 strips impregnated with various hydrous oxides	140
S_{87}	M_{101}	F^-, Cl^-, Br^-, I^-	D_{102}	TLC identification, determination, and detection limits of halides on activated commercially available films. The R_F values and detection limits were F^- 6, 1 μg; Cl^- 25, 2 μg; Br^- 50, 8 μg; I^- 72, 2 μg, respectively	141
S_{88}	M_{102}	NO_3^-, NO_2^-	D_{103}	TLC identification and semiquantitative determination of nitrite and ammonium ions	142
S_{89}	M_{103}	Bisarenechromium iodides	—	TLC separation. Silica gel is characterized by the following parameters. Specific pore volume, 0.61–0.74 cm^3/g; specific surface, 550–650 m^2/g; medium pore radius, 6–20 Å; pH, 3.0	143
S_{90}	M_{104}	25 organic and inorganic anions	—	TLC separation procedure reported. Sensitivities of anions with various detection reagents are given	144
—	M_{105}	$Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, SCN^-, CN^-	D_{16}	TLC separation and detection limits of pseudohalides. The R_F values and detection limits were $Fe(CN)_6^{3-}$ 0.50, 0.1 μg; $Fe(CN)_6^{4-}$ 0.90, 0.1 μg; SCN^- 0.95, 0.08 μg; CN^- 0.65, 20 μg; respectively	145

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
S_{60}	—	Br^- residues	D_{104}	Quantitative TLC determination of Br^- residues in crops after soil treatment by methyl bromide. The calibration curve is linear for 10–90 ppm Br^-	146
S_{60}	M_{106}	I^-, IO_3^-, IO_4^-	D_{105}	Ascending TLC. Effect of alcohol type, alcohol–aq. NH_3 volume ratio, and NH_3 concentration on the separation efficiency and rate were followed	147
S_{60}	M_{107}	PO_4^{3-}, MoO_4^{2-}, TeO_4^-, RuO_4^{2-}, TeO_3^{2-}, I^-	D_{105}	Ascending TLC. Correlation between the R_F values and dielectric constants of the eluent system can be expressed by $R_F = k(\log \epsilon) + \log q$, where k and q are constants characteristic of the migrating anion	148
S_{91}	M_{108}	NO_2^-, NO_3^-, ClO^-, ClO_3^-, ClO_4^-, IO_4^-, BrO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, $S_2O_8^{2-}$, $S_2O_5^-$, MnO_4^-, $Mo_7O_{24}^{6-}$, WO_4^{2-}, SeO_3^{2-}, SeO_4^{2-}, VO_3^-, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{106}	TLC and paper electrophoresis of anions. Detection limit were ~0.1 μg. The relative standard deviations were 6 and 8% for detecting NO_3^- and $Fe(CN)_6^{3-}$ in molasses. The calibration curve were linear for 1–5 μg anion	149
S_{92}	M_{109}	Few anions	—	Analytical applications of hydrated stannic oxide as an ion-exchanger were investigated by TLC. Binder-free thin layers of hydrated stannic oxide are useful for some binary separations of anions	150
S_{60}	M_{110}	Cl^-, Br^-, NO_3^-, SCN^-, I^-	—	—	151
S_{93}	M_{111}	CN^-	D_{107}	TLC detection of CN^- as $Hg(CN)_2$ among 22 anions was studied; 25 cations also studied. Thin-layer glass sticks (2.5 mm diam × 13 cm) were used	152
S_{64}	M_{112}	SCN^-, selenocyanate	D_{108}	Microdetermination of anions by densitometry. Ascending technique; run 7 cm. Detection limits were 0.2 μg SCN^- and 0.01 μg selenocyanate	153
S_{60}	M_{113}	I^-, Br^-, Cl^-, SCN^-, ClO_3^-, BrO_3^-, IO_3^-, NO_2^-, $S_2O_3^{2-}$, SO_4^{2-}, CrO_4^{2-}, PO_4^{3-}, AsO_3^{3-}, AsO_4^{4-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	—	Thin-layer sticks were used. Sticks were allowed to dry in air and then immersed in a color reagent	154
S_{60}	M_{114}	BrO_3^-	D_{109}	Detection of bromate in bread after TLC separation. Recoveries of 100 or 1000 μg BrO_3^- from flour, dough, and bread were 72–92%.	155

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				Limit of detection was 0.1 μg/g. No bromate was detected in commercial flour (40 samples) and bread (72 samples)	
S_{94}	M_{115}	$Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{110}, D_{111}	Quantitative separation and recovery of anions by TLC. Determination of the ions in bleach and fixer solutions. R_F values are not influenced by the presence of 500-fold excess of halides, halogen-containing anions, nitrate, nitrite, carbonates, sulfates, sulfites, thiosulfate, CN^-, SCN^-, and phosphates (which are usually found in polluted water) in the spotting solution	156
S_{60}	M_{116}	Br^-	D_{102}	TLC procedure is reported for the detection of Br^- in 0.5 g tissue samples (mouse whole blood, plasma, and liver) in the presence of Cl^- using as indicator bromocresol purple. The peak area can be used to detect Br^- concentrations, and the sensitivity is improved 50-fold over direct spot spraying. Results are linear for 0.02–0.50 μmol Br^-. R_F values of Br^- and Cl^- vary with concentration	157
S_{95}	M_{117}	NO_3^-	D_{106}	Determination of NO_3^- by TLC. Densitometric evaluation	158
S_{60}	M_{118}	Phosphate (P), silicate (Si)	D_{112}	Determination of P and Si as their molybdate complexes by TLC. R_F values are 0.64 and 0.53 for P and Si, respectively. Calibration curve was linear for 12.5–125 ng P or Si. P and Si were detected in serum after treating with TCA	159
S_{62}	M_{119}	Cl^-, Br^-, I^-, IO_3^-, ClO_3^-, BrO_3^-, SCN^-, MnO_4^-, NO_3^-, OAc^-, CrO_4^{2-}, AsO_3^{3-}, AsO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6$	D_{113}	Separation and identification by TLC	160
S_{60}	—	NO_3^-	D_{101}	Sensitive and selective reaction for nitrate application in TLC. Allows rapid detection of NO_3^- down to 5 ng. Method is used for the detection of NO_3^- in food and water samples. 55 cations or	161

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				anions do not interfere in detection	
S_{60}	M_{120}	NO_2^-	D_{114}	TLC densitometric determination of nitrite in saliva. As low as 1.67 ng/µL could be detected by application of 50 ng saturated and 30 µL aliquots of saliva. Sensitivity, precision, and accuracy appear to be adequate for use of the method in clinical analysis	162
S_{96}	M_{121}	Br^-, I^-	—	TLC detection of bromine in the presence of I^-. Br^- forms red-colored spot on the plate	163
S_{97}	—	I^-, Br^-, F^-, IO_3^-, BrO_3^-, NO_2^-, NO_3^-, PO_4^{3-}	—	TLC and PC studies for the separation of anions	164
S_{98}	M_{122}	I^-, IO_3^-, IO_4^-, BrO_3^-, SCN^-, Br^-, CrO_4^{2-}, AsO_4^{3-}, PO_4^{3-}, $S_2O_3^{2-}$	—	Rapid TLC microgram determination of ferricyanide (1–40 µg) and dichromate (2–10 µg) ions was achieved	165
S_{99}	M_{123}	I^-, Br^-, NO_2^-, NO_3^-, SCN^-, VO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, MoO_4^{2-}, WO_4^{2-}, PO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{16}–D_{19}	Ascending technique, run 10 cm. TLC separation and identification of anions. Detection limits were 1 µg for VO_3^-, NO_2^-, NO_3^-, SCN^-, and $Fe(CN)_6^{4-}$; 0.5 µg for $Fe(CN)_6^{3-}$, CrO_4^{2-}, and $Cr_2O_7^{2-}$; 0.1 µg for MoO_4^{2-} and WO_4^{2-}; 10 µg for PO_4^{3-} and I^-; and 100 µg for Br^-	166
S_{64}	M_{124}	I^-, Br^-, Cl^-, IO_3^-, IO_4^-, BrO_3^-, CH_3COO^-, SCN^-, NO_2^-, VO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, $S_2O_3^{2-}$, MoO_4^{2-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{16}–D_{19}	Ascending technique, run 10 cm. TLC separation of common anions with 69 mobile phases studied. Significant aspect of this study is the separation of iodate from large excesses of I^-, Br^-, Cl^-, and vice versa	167
S_{100}	M_{125}	I^-, Br^-, IO_3^-, BrO_3^-, NO_2^-, NO_3^-, VO_3^-, MnO_4^-, SCN^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, WO_4^{2-}, MoO_4^{2-}, $C_2O_4^{2-}$, PO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{115}, D_{16}–D_{19}	Ascending technique, run 10 cm. Layer thickness 0.25 mm. TLC microgram separation and determination of anions. Effect of pH on R_F values examined. Detection limits of anions have been reported	168
S_{64}	M_{126}	I^-, IO_3^-, IO_4^-, BrO_3^-, MoO_4^{2-}, $Fe(CN)_6^{4-}$	D_{16}–D_{18}	Ascending technique, run 10 cm. Layer thickness 0.25 mm. Effect of cations on anions separation studied. TLC parameters such as ΔR_F, separation factor (α), capacity factor (K'), and	169

(continued)

TABLE 4 Continued

Stationary phases	Mobile phases	Anions studied	Detection reagents	Conditions/remarks	Refs.
				resolution (*Rs*) have been calculated. Detection limits (microgram) were IO_4^-, 4.15; IO_3^-, 4.08; BrO_3^-, 0.76; MoO_4^{2-}, 6.61; I^-, 7.65; $Fe(CN)_6^{3-}$, 5.24	
S_{101}	M_{126}	I^-, Br^-, Cl^-, NO_2^-, NO_3^-, MnO_4^-, SCN^-, IO_3^-, IO_4^-, BrO_3^-, VO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, WO_4^{2-}, PO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{16}–D_{19}	Ascending technique, run 10 cm. Layer thickness 0.25 mm. TLC identification and separation of anions on mixed bed systems. NO_2^- in artificial seawater has been identified. Effect of $CaCl_2$, $MgCl_2$, and $NaHCO_3$ on ternary separations has been examined. Detection limits (microgram) were MnO_4^-, 0.75; VO_3^-, 0.70; SCN^-, 7.63; IO_3^-, 8.71; IO_4^-, 8.30; $Fe(CN)_6^{4-}$, 0.70	170
S_{60}	M_{127}	I^-, Br^-, Cl^-, ClO_3^-, ClO_4^-, NO_2^-, NO_3^-, SCN^-, SO_4^{2-}, $H_2PO_4^{2-}$	D_{116}	Radial or ascending technique. Anions in the form of metal diantipyrilmethane (DAM) complexes and salts of protonated DAM were separated	171
S_{102}	M_{128}	I^-, Br^-, Cl^-, NO_2^-, NO_3^-, SCN^-, IO_3^-, IO_4^-, BrO_3^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, WO_4^{2-}, MoO_4^{2-}, $Mo_7O_{24}^{6-}$, PO_4^{3-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$	D_{16}–D_{19}	Ascending technique, run 10 cm. Layer thickness 0.25 mm. TLC of anions and their quantitative determination by volumetry and spot area measurement technique. Detection limit of few anions also reported	172

TABLE 5
Number of Papers Appearing in Different Journals on TLC of Anions During 1959–1994

No.	Name of journal	No. of publications
1	*Journal of Chromatography*	24
2	*Fresenius' Zeitschrift fuer Analytische Chemie*	10
3	*Analytical Chemistry*	6
4	*Bulletin de la Societe Chimique de France*	6
5	*Helvetica Chimica Acta*	5
6	*Seifen, Oele, Fette, Wachse*	5
7	*Bunseki Kagaku*	5
8	*Mikrochimica Acta*	5
9	*Microchemical Journal*	5
10	*Bulletin of the Chemical Society of Japan*	4
11	*Chromatographia*	3
12	*Journal of Radioanalytical Chemistry*	2

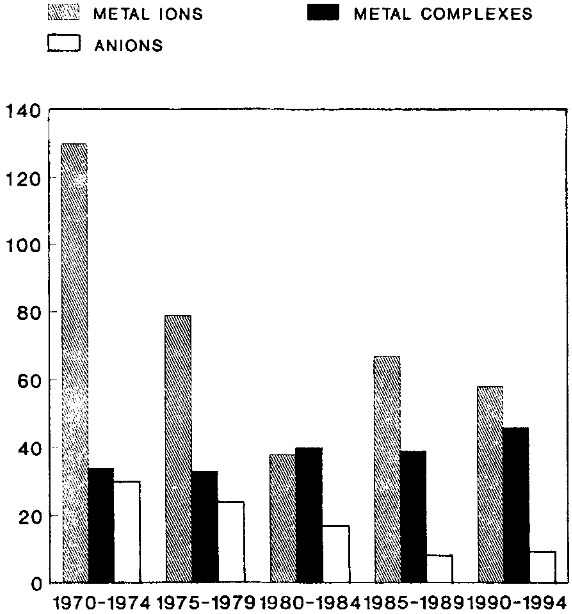

FIG. 1 TLC publications on metal ions, metal complexes, and anions (1970–1994).

in Table 5. Work done on TLC of anions, metal ions, and metal complexes during 1970–1994 is compared in Fig. 1.

ACKNOWLEDGMENT

One of the authors (S.T.) expresses his gratitude to the Council of Scientific and Industrial Research (CSIR), New Delhi, for providing financial support in the form of a Senior Research Fellowship.

ABBREVIATIONS

TCA	trichloroacetic acid
TFA	trifluoroacetic acid
GAA	glacial acetic acid
FA	formic acid
THF	tetrahydrofuran
EMK	ethyl methyl ketone
DMF	dimethylformamide
DCF	dichlorofluorescein
PC	paper chromatography

DMSO	dimethylsulfoxide
TLPC	thin-layer precipitation chromatography
TLEP	thin-layer electrophoresis
TLC	thin-layer chromatography
DPA	diphenylamine
TEA	triethanolamine
DMA	dimethylamine
DEAH	diethylamine hydrochloride
PPL	phenolphthalein

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Received by editor March 20, 1995