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# Separation of Alkali-Metal Ions from Each Other and from Other Metal Ions on Ion-Exchange Papers

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## NOTE

# Separation of Alkali-Metal Ions from Each Other and from Other Metal Ions on Ion-Exchange Papers

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## Summary

Cation-exchange paper chromatography with mixed solvents provides for the mutual separation of the alkali-metal ions. Development with a complexing solution on anion-exchange paper provides for the separation of the alkali metals as a group from other monovalent and multivalent metal ions.

In light of the recent comprehensive report (*1*) on the column ion-exchange separation of the alkali-metal ions, we have studied the use of ion-exchange papers for such separations. Wash liquids composed of aqueous acids such as HCl or aqueous salts such as $NH_4Cl$ yielded very poor separations. We turned next to aqueous–nonaqueous wash liquids of the type that had provided selective separations of other metal ions on ion-exchange papers (*2*). Reported below are two selective paper-chromatographic systems that permit the mutual separation of the alkali metals. These systems are more convenient than those recently reported that involve papers impregnated with inorganic ion exchangers (*3–6*), or thin-layer media (*7*) because the ion-exchange papers are ready for use as received from the commercial source.

## EXPERIMENTAL

Amberlite SA-2 and Machery–Nagel sulfonic acid cation-exchange papers were employed. The SA-2 paper consists of cellu-

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lose interspersed with Amberlite IR-120 cation-exchange resin, with a capacity of 2.0 meq/g. The MN paper consists of chemically modified cellulose with a capacity of 0.5 meq/g. Both are supplied in the Na–salt form and were converted to the hydrogen form before use (*8*). Some comparative runs were also made on Whatman No. 1 pure cellulose chromatography paper.

Development was by the descending technique over a distance of 35 cm in equipment already described (*9*). Individual spots of each ion and synthetic mixtures were tested in each system. The test solutions were 1.0 *M* in Na(I) and Li(I), 0.10 *M* in K(I), Rb(I), and Cs(I), and 0.050 *M* in the other ions. They were aqueous solutions of the nitrates of each metal, except for a few cases (*10*).

Two mixed solvents were employed for separation of the alkali metals from each other: solvent I was water–acetone–concentrated HCl, 20:8:1.8 (v/v); solvent II was prepared by diluting 33.3 ml of concentrated HCl to 100 ml with 80% (v/v) methanol.

Sodium and lithium ions were detected under UV light after spraying with zinc uranyl acetate (*11*). Potassium, rubidium, and cesium were detected as yellow spots on a tan background after spraying with a cobalt–lead acetate solution (*11,12*). The other ions were detected with sprays already described (*13*). For detection after development, it was necessary to spot 30λ of the alkali-metal solutions on SA-2 paper. In all other systems, initial zones of 10λ were employed.

The $R_F$ values of the front and rear limits of the developed zones reported below are the average of at least duplicate runs.

## RESULTS AND DISCUSSION

### Separation of Alkali Metal Ions from Each Other

Table 1 presents the results of migrations of the alkali metals with solvents I and II on SA-2, MN, and Whatman No. 1 papers. With solvent I, Li and Na were separated from each other and from the mixed zone of K plus Rb plus Cs. The actual $R_F$ values obtained when a synthetic mixture was developed were Li, 0.67–0.60; Na, 0.43–0.26; K + Rb + Cs, 0.22–0.12. With solvent II, Li, Rb, Cs, and K were separated from each other, but sodium was mixed with potassium. $R_F$ values actually obtained with a synthetic mixture excluding sodium were Li, 0.64–0.57; Cs, 0.42–0.36; Rb, 0.30–0.26;

TABLE 1

$R_F \times 100$ Values for the Alkali Metals in Various Systems

| Type of paper | | SA-2 | MN | Whatman No. 1 |
|---|---|---|---|---|
| Solvent I | Li | 66–59 | 87–78 | 96–86 |
| | Na | 40–30 | 92–82 | 93–86 |
| | K | 22–17 | 82–77 | 89–79 |
| | Rb | 19–14 | 83–77 | 88–80 |
| | Cs | 17–12 | 86–79 | 88–80 |
| Solvent II | Li | 62–56 | 80–70 | 83–73 |
| | Na | 25–14 | 55–40 | 58–42 |
| | K | 25–19 | 45–26 | 44–26 |
| | Rb | 30–25 | 49–36 | 49–38 |
| | Cs | 40–34 | 53–44 | 64–52 |

K, 0.26–0.20. Therefore, by choosing either solvent I or II, any pair of ions can be separated.

It is interesting that in solvent I the $R_F$ sequence varied inversely with the atomic number of the alkali metals but that this was not true in solvent II. This illustrates again that the use of aqueous–nonaqueous solvents in ion-exchange chromatography beneficially affects separation factors in many systems by superimposing partition chromatography and ion-exchange chromatography.

That the separations obtained on SA-2 paper are largely due to the ion-exchange resin rather than the cellulose backbone is shown by the results on Whatman paper (Table 1). No separations were obtained with solvent I, and the sequence of zones was different and the separations poorer with solvent II on the cellulose paper. $R_F$ values on the MN ion-exchange paper were usually slightly lower but very similar to those on Whatman paper. The separations reported for SA-2 paper could not be repeated on MN paper. The similarity between the MN and Whatman papers is undoubtedly due to the very low ion-exchange capacity of the MN paper, which in effect represents only a slight dilution of the cellulose matrix.

### Separation of Alkali Metals as A Group from Other Metal Ions

Because the alkali metals can be estimated in the presence of each other by techniques such as flame photometry, it is useful to have a chromatographic procedure for their separation from complex mixtures with other ions. Electrophoretic (*14*) and column (*15*)

TABLE 2

$R_F \times 100$ Values for Metal Ions on SB-2 Anion-Exchange Paper with 0.0125 *M* Nitrilotriacetic Acid, 0.10 *M* $NH_3$, and 0.075 *M* KCN as Wash Liquid

| | | |
|---|---|---|
| Ions that remained at the origin: | Ag(I), Pb(II), Cu(II), Cd(II), Co(II), Ni(II), Fe(III), Hg(I), Hg(II), Sn(IV), Pt(IV), Au(III), U(VI), Zn(II), Cr(III) | |
| Ions with $R_F$ limits of 100–95: | Li(I), Na(I), K(I), Rb(I), Cs(I) | |
| Other ions with their $R_F$ limits: | As(III), 80–73 | |
| | Mg(II), 95–48 (diffuse zone) | |
| | Al(III), 31–0 | Comets from the origin |
| | Ce(IV), 5–0 | |
| | Mn(II), 50–0 | |
| | Sb(III), 14–0 | |
| | Tl(I), 70–0 | |

methods have been described for such separations, but a simple paper chromatographic system has now been developed. Twenty-two representative ions plus the alkali metals were developed on SB-2 ion-exchange paper (containing Amberlite IRA-400 quaternary ammonium anion-exchange resin, 1.6 meq/g capacity) with 0.0125 *M* nitrilotriacetic acid, 0.10 *M* $NH_3$, and 0.075 *M* KCN as the wash liquid. Paper electrophoretic studies (*14*) had shown that a similar solution formed negatively charged complexes (which should be sorbed by anion-exchange resin) with many multivalent metal ions, Ag(I), Tl(I), and Hg(I). Results in Table 2 indicate that in fact the alkali metals, which traveled with the solvent front, can be separated from virtually any other metal ion in this chromatographic system. The low affinity of the alkali-metal ions would allow them to be quantitatively eluted and determined.

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