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### Separation of metal-EDTA complexes by thin-layer chromatography

The metal-EDTA complexes have many-sided uses. It is desirable to develop a suitable and rapid method of separation of these complexes. The present studies were undertaken to separate the compounds by thin-layer chromatography.

#### Experimental

Silica Gel H free from binder gypsum (Merck) was used as adsorbent. The powder was treated with concentrated HCl to remove metallic impurities and was freed from acid and soluble salts by washing with distilled water until no chloride ions were left in the filtrate. Finally a few washings were made with alcohol. The adsorbent was then dried in an oven at 120° for 4 h. The thin-layer plates were prepared by the method described by STAHL<sup>1</sup>: 30 g silica gel were shaken vigorously in an erlenmeyer flask with 60 ml of distilled water for about 2 min. The slurry was spread onto five dry glass plates (20 × 20 cm) using the applicator to give a thickness of 0.25 mm. The plates were dried in two stages, first with an alternately cold and warm air stream until the plates had a mat appearance, and then by heating in the oven for 4 h at 120°.

TABLE I

$R_F$  VALUES OF SOME METAL-EDTA COMPLEXES

Composition of solvent (%)						$R_F$ values					
Water	Glycol mono- methyl ether	Ethyl methyl ketone	Butyl alcohol	Acce- tone	Ammonia ( $D = 0.91$ )	Co	Cu	Ni	Mn	Cr	Fe
45	20	—	25	10	0.25	0.83	0.79	0.66	0.59	0.45	0.27
40	20	20	—	20	0.15	0.86	0.79	0.69	0.71	0.44	0.17
40	30	15	10	5	0.10	0.78	0.69	0.65	0.58	0.47	0.26

The chromatographic tank, containing 100 ml solvent, was provided with a wick for homogeneous chamber saturation. After an equilibration time of 16 h a few microliters of a 1% metal-EDTA solution were applied to the chromatographic plate. The solutions were prepared by dissolving the solid products, obtained according to the method of SAWYER AND MCKINNIE<sup>2</sup>, in distilled water. By their specific and intense color even 5 to 10  $\mu$ g of Co(III)-, Cr(III)-, Cu(II)-, Ni(II)- and Fe(III)-EDTA could be identified as colored spots without auxiliary means. The position of Mn(II)-EDTA could be located after spraying with a 20% ammonium peroxydisulfate solution.

#### Results and discussion

The choice and composition of the solvents together with the corresponding  $R_F$  value of each metal-EDTA complex are given in Table I.

Despite the variation in the composition of the solvent, the sequence of the

$R_F$  values, which show the relative migration rates of the complexes, is almost constant. This was to be expected owing to the fact that the metal ions are masked by the electron donors of the EDTA molecule, which suppress their specific properties. Moreover, these complexes are almost identical with regard to their chemical structure and molecular weight, with the result that the composition and the pH of the solvent should be chosen very judiciously. This has already been mentioned by SYKORA AND EYBL<sup>3</sup>, who separated these complexes by paper chromatography.

The choice of the solvent is limited by the following considerations:

(1) The pH of the solvent should be within very narrow limits because one has to take into account that the stability of the compounds varies with pH. This could be the reason why TSUNODA, TAKEUCHI AND YOSHINO<sup>4</sup> observed tailing effects for  $\text{CoY}^{2-}$  and  $\text{MnY}^{2-}$  when acetic acid was present in the developing solvent. It has already been reported<sup>5</sup> that the  $\text{MY}^{n-4}$  complexes of the divalent elements are quantitatively formed above pH 5, while at pH 8 Fe(III)-EDTA is decomposed in favor of the hydroxide.

(2) Because these complexes are almost insoluble in the commonly used organic solvents, there must be a certain percentage of water present in the mixed developing solvent. It would be interesting to find out if a correlation exists between the solubility of these compounds in the solvents and their respective  $R_F$  values.

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Faculty of Agricultural Sciences,  
Coupure, Ghent (Belgium)

J. VANDERDEELEN

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