

After the solvent has evaporated (approx. 10 min) sulphates form black spots. Fig. 1 shows a chromatogram thus treated. The spots from left to right are $N/10$ H_2SO_4 , $N/10$ HNO_3 , $N/10$ H_3PO_4 and a mixture of all three.

Phosphoric acid also produces a darkening and nitric acid a fluorescent spot in ultraviolet light. Both can, however, be distinguished by their R_F values from sulphate.

This reaction, simple as it seems, is, however, possible only under controlled conditions, and we shall enumerate the factors that may interfere.

The chromatogram may not be developed for more than 8 hours. Chromatograms that were left overnight, gave only a uniform darkening of the whole sheet. Likewise, chromatograms that were allowed to dry overnight also showed uniform darkening. In both cases the prolonged contact with HCl seems to produce considerable decomposition of the paper.

On the other hand the reaction works well, irrespective of whether an alkali sulphate, an alkaline earth sulphate, or free sulphuric acid, is chromatographed. In the case of the salts the cation stays in the region of the R_F 0-0.1 and thus cannot interfere with a reaction that seems to be that of free H_2SO_4 .

The sensitivity was tested by making successive dilutions of sulphuric acid and running them on the same sheet. A dark spot is formed with as little as 20 γ but when the paper is held under ultraviolet light even 5 γ may still be detected as a fluorescent spot.

The fluorescence has been observed to fade with time, while the dark colour appears to be quite stable.

We believe that this reaction may be reliable in testing for sulphates.

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Bemerkung zur Mikro-Jodbestimmung nach Bowden, Maclagan und Wilkinson

Nach dem von BOWDEN *et al.*¹ angegebenen Verfahren (Reaktion mit Cer(IV)Sulfat und arseniger Säure) lassen sich z.B. 0.01 Mikrogramm KJ und 0.1 Mikrogramm Thyroxin auf dem Papierchromatogramm erkennen und zwar als weisse Flecken auf hellgelbem Grund. Allerdings lassen sich Flecken, die von den angegebenen Substanzmengen herrühren, wegen des geringen Kontrastes nur schlecht photographieren.

Dem kann abgeholfen werden, indem man das Chromatogramm in Phenoldämpfe bringt, wodurch der gelbe Hintergrund hell- bis dunkelbraun wird, was die Flecken ausgezeichnet hervor- treten lässt.

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