

A Note on the Identification of Paracetamol, Aspirin and Salicylamide in Certain Analgesic Preparations by TLC

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The three major ingredients in certain modern analgesic preparations are separated by thin layer chromatography and identified by selective spray reagents.

Paracetamol, aspirin and salicylamide are the three major ingredients in modern analgesic preparations and are easily separated and identified by thin layer chromatography.

Reagents

1. *Silica gel sheets*: Kodak 6061—activated at 105°C for 30 minutes.
2. *Developing solvent*: Chloroform/*iso*-propanol (95:5).
3. *Ethanolic ammonia solution*: 5 ml of ammonia solution (S.G. 0.880) + 15 ml of Industrial Methylated Spirits (74° O.P.).
4. *Phosphomolybdic acid solution*: Dissolve 2 g of dodeca-molybdophosphoric acid in acetone and make to 100 ml.
(Store the solution in a refrigerator and discard when the solution has a greenish colour).
5. *Ethanolic ferric chloride solution*: 5 ml of a 5 per cent. ferric chloride solution diluted to 25 ml with Industrial Methylated Spirits (74° O.P.).

Method

Extract the active ingredients of the preparation (tablet, powder etc.) with chloroform and adjust the volume of the extract so that it contains between 1 and 5 milligramme of each ingredient in a millilitre. Apply 2 microlitres of this extract in 0.5 microlitre quantities to an activated silica gel sheet. Similarly apply as separate spots, 2 microlitres of 0.1 per cent. solutions of aspirin, paracetamol and salicylamide.

Develop the chromatogram with the chloroform/*iso*-propanol solvent until the solvent front has run 10 cm. Remove the sheet and allow the solvent to evaporate.

- (i) Examine under ultra-violet light of 254 nm wavelength. Salicylamide shows as a pale blue fluorescent spot near the solvent front; any free salicylic acid which may be present will show as a medium blue fluorescent spot near the origin.
- (ii) Spray the sheet with ethanolic ammonia solution and while still wet place it in a tank filled with an atmosphere of ammonia. Allow to

stand for 10 minutes (to allow any aspirin to hydrolyse to salicylic acid).

Remove the sheet and dry in a current of air until free from ammonia.

(iii) Spray with phosphomolybdic acid solution and return the sheet to the ammonia atmosphere, until the yellow colour of the reagent is discharged. Paracetamol shows as a dark blue spot. Dry the sheet in a current of air until free from ammonia.

(iv) Spray with ethanolic ferric chloride solution. Salicylamide and aspirin show as mauve coloured spots.

If stage (ii) is omitted only salicylamide and any free salicylic acid will give mauve coloured spots.

TABLE I

R_f VALUES OF MAJOR INGREDIENTS OF ANALGESIC PREPARATIONS

Ingredient						R _f Value (× 100)
(Salicylic acid)	..	..	..	..	..	12
Aspirin	..	..	..	..	..	12
Paracetamol	..	..	..	..	..	40
Salicylamide	..	..	..	..	..	78