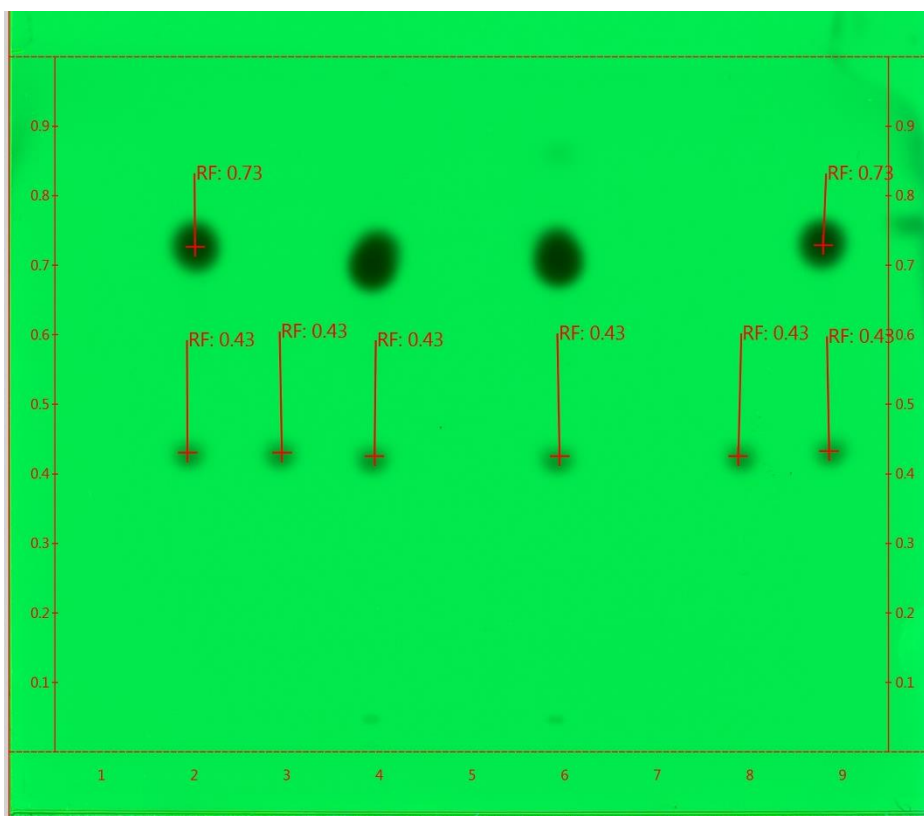




## Paracetamol, Codeine Phosphate and Caffeine Tablets – BP 2022

These chromatograms are provided for information only as an aid to analysts and are intended as guidance for the interpretation and application of BP monographs.

Typical chromatogram for the Identification D test for Paracetamol, Codeine Phosphate and Caffeine Tablets by Thin Layer Chromatography as published in BP 2022.

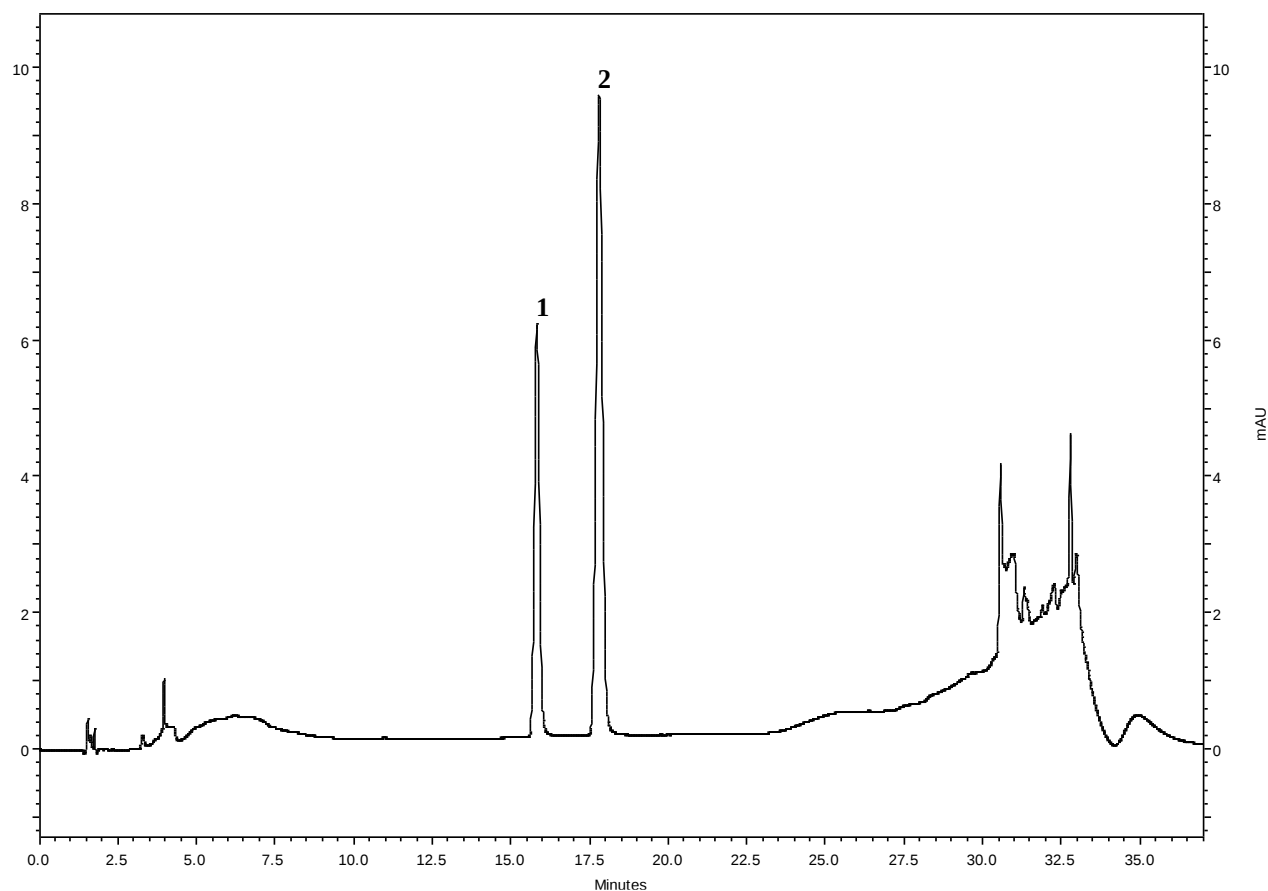


- |         |                                       |
|---------|---------------------------------------|
| 1; 5; 7 | Blank                                 |
| 2; 9    | System suitability                    |
| 3; 8    | 0.65 % w/v caffeine standard solution |
| 4; 6    | Tablet 0.65 % w/v sample solutions    |

|                       |                                                                         |
|-----------------------|-------------------------------------------------------------------------|
| TLC plate             | TLC silica gel 60 F254 plate (20 cm × 20 cm)                            |
| Plate preconditioning | N/A                                                                     |
| Diluent               | Methanol                                                                |
| Mobile Phase          | Acetic acid : ethanol : water : ethyl acetate (5 : 5 : 5 : 50, v/v/v/v) |
| Mobile Phase volume   | 65 mL                                                                   |
| Band application      | 3 mm band size with a spotting volume of 1 µL                           |
| Chamber saturation    | Minimum 60 minutes at room temperature                                  |
| Development           | 150 mm                                                                  |
| Development time      | 75 minutes                                                              |
| Drying time           | 10 minutes in air                                                       |
| Derivatisation        | N/A                                                                     |
| Visualisation         | Developed plate examined under UV light (254 nm)                        |



Typical chromatogram for solution (3) in the Related Substances test for Paracetamol, Codeine Phosphate and Caffeine Tablets as published in BP 2022.



Peak ID: 1: Codeine; 2: 4'-chloroacetanilide

Column : Kinetex, C18 (150 mm x 4.6 mm, 2.6  $\mu$ m)  
Method Ref. : Related Substances for the Paracetamol, Codeine Phosphate and Caffeine Tablets monograph from BP 2022  
Mobile Phase A : 5 mM sodium octanesulfonate, adjusted to pH 2.2 with phosphoric acid.  
Mobile Phase B : Methanol R1  
Gradient :

| Time (minutes) | Mobile phase A (% v/v) | Mobile phase B (% v/v) |
|----------------|------------------------|------------------------|
| 0 – 2.5        | 80 → 70                | 20 → 30                |
| 2.5 – 20       | 70                     | 30                     |
| 20 – 30        | 70 → 20                | 30 → 80                |
| 30 – 32        | 20 → 80                | 80 → 20                |
| 32 – 37        | 80                     | 20                     |

Diluent : 0.23 % w/v sodium chloride in a mixture of mobile phase B and mobile phase A (30: 70 v/v).  
Flow rate : 0.8 mL/min  
Column Temp : 35 °C  
Injection Volume : 20  $\mu$ L  
Detection : 246 nm