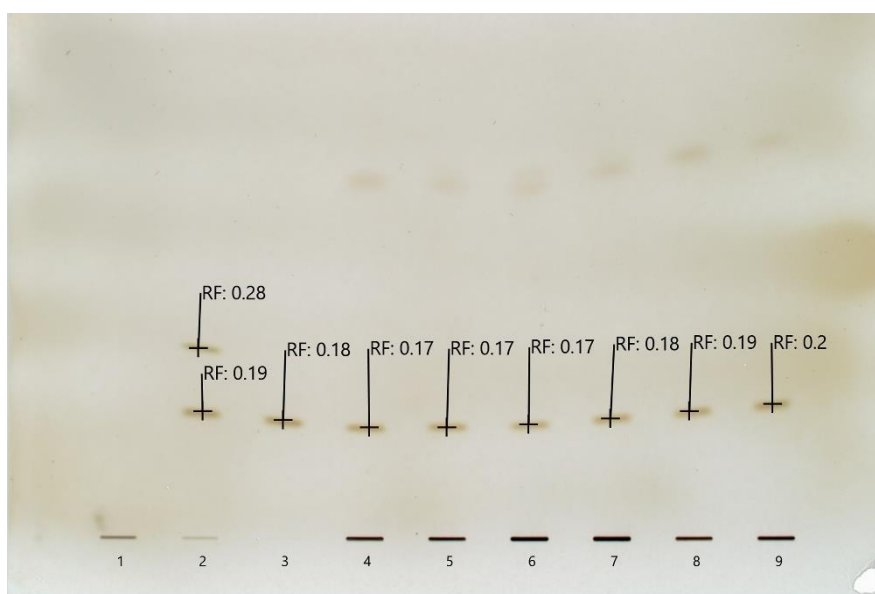




Digoxin Tablets – BP 2024

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 test for Digoxin Tablets as published in BP 2024.

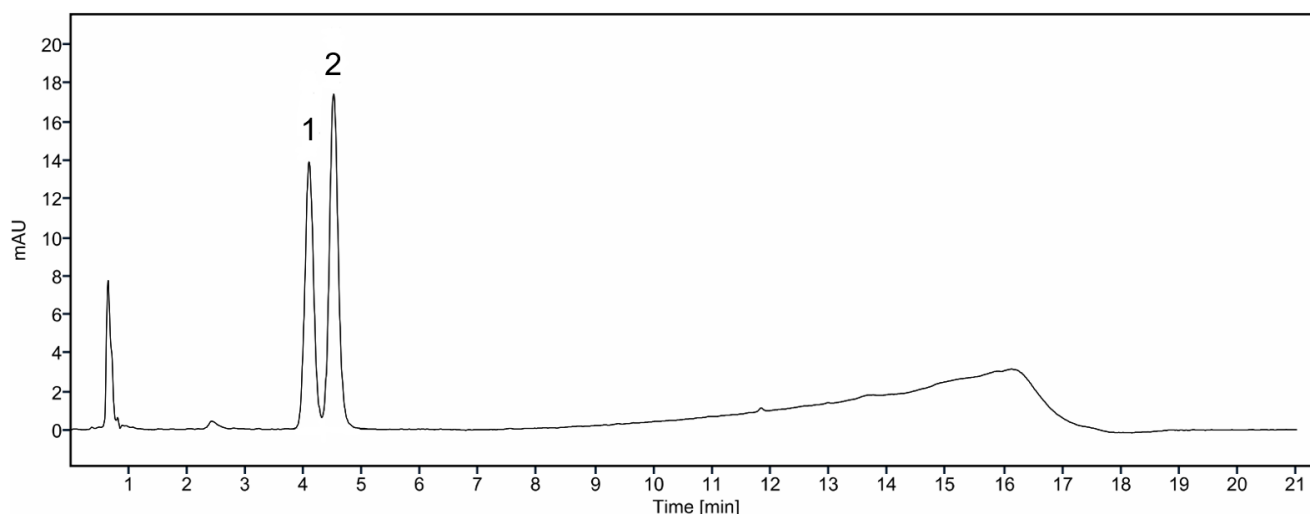


- | | |
|-----|---|
| 1 | Blank |
| 2 | Solution (3): 0.0025% w/v each of digoxin and digitoxin |
| 3 | Solution (2): 0.0025% w/v of digoxin |
| 4-9 | 0.0025% w/v solutions of digoxin tablets samples |

TLC plate	TLC silica gel 60 (20 x 20 cm)
Plate preconditioning	N/A
Diluent	A mixture of equal volumes of dichloromethane and methanol
Mobile Phase	Methanol: cyclohexane: dichloromethane (15:40:90 v/v/v)

Band application	8 mm band size with a spotting volume of 25 µL
Chamber saturation time	Minimum of 60 minutes at ambient temperature
Development	150 mm
Development time	35 minutes
Drying time	5 minutes in a stream of cold air. The development of the plate was repeated and dried again for 5 minutes in a stream of cold air
Derivatisation	Manually sprayed with a mixture of 1 volume of sulfuric acid and 9 volumes of ethanol (96%) then heated at 130°C for 15 minutes
Visualisation	White light

Typical chromatogram for solution (3) from the Related Substances and Assay tests for Digoxin Tablets as published in BP 2024.

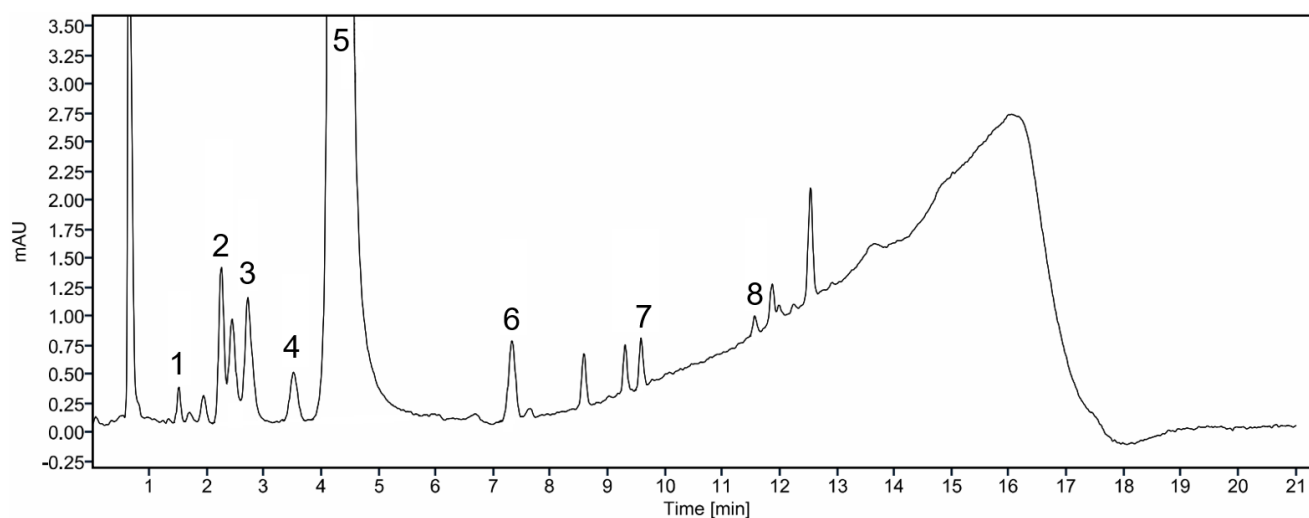


Peak ID: 1: Impurity H. 2: Digoxin.

Column	Symmetry C18 (150 mm x 3.9 mm, 5 µm)
Method Ref.	Related Substances from the Digoxin Tablets monograph from BP 2024
Mobile Phase A	Acetonitrile: water (10:90, v/v)
Mobile Phase B	Water: acetonitrile (10:90, v/v)
Diluent	Mobile phase B: Mobile phase A (22:78, v/v)

Flow rate	1.5 mL/min		
Column Temp	25°C		
Injection Volume	50 µL		
Detection	220 nm		
Gradient			
Time (minutes)	Mobile phase A (% v/v)	Mobile phase B (% v/v)	Comment
0 – 5	78	22	isocratic
5 – 15	78 → 30	22 → 70	linear gradient
15 – 16	30 → 78	70 → 22	linear gradient
16 – 21	78	22	re-equilibration

Typical chromatogram for solution (4) from the Related Substances test for Digoxin Tablets as published in BP 2024.



Peak ID: 1: Impurity C. 2: Impurity E. 3: Impurity F. 4: Impurity G. 5: Digoxin. 6: Impurity K. 7: Impurity B. 8: Impurity A.

Column	Symmetry C18 (150 mm x 3.9 mm, 5 µm)
Method Ref.	Related Substances from the Digoxin Tablets monograph from BP 2024
Mobile Phase A	Acetonitrile: water (10:90, v/v)

Mobile Phase B	Water: acetonitrile (10:90, v/v)		
Diluent	Mobile phase B: Mobile phase A (22:78, v/v)		
Flow rate	1.5 mL/min		
Column Temp	25°C		
Injection Volume	50 µL		
Detection	220 nm		
Gradient			
Time (minutes)	Mobile phase A (% v/v)	Mobile phase B (% v/v)	Comment
0 – 5	78	22	isocratic
5 – 15	78 → 30	22 → 70	linear gradient
15 – 16	30 → 78	70 → 22	linear gradient
16 – 21	78	22	re-equilibration