

BDG SYNTHESIS

Certificate of Analysis

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

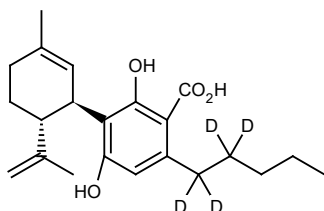
Neil Beare

Neil Beare, PhD, Director
5 September 2022

Name: Cannabidiolic Acid-d₄

CAS Number: 1244-58-2 (unlabelled)

Structure:



Molecular Weight: C₂₂H₂₆D₄O₄ = 362.50

Lot Number: BDG 18021

Appearance: Off-white, brittle foam

Purity By HPLC: 99.3 %

Isotopic Purity: Under 0.5 % d₀

Re-test Date: 5 September 2027

Storage and Handling: Temperature: Freeze (-20°C) for prolonged storage; may be handled and shipped at ambient temperature.

Humidity: not believed to be hygroscopic; may be handled in normal laboratory atmosphere.

Light: protect from strong sunlight.

Caution: only experienced laboratory personnel should handle the material.

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Isotopic Labelling: signals at the sites of deuteration are greatly diminished, compared with what would be expected for unlabelled material, indicating clean deuteration.

Residual Solvents: no residual solvents are observed.

Impurities: a trace of an unidentified impurity is seen in the baseline.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Isotopic Labelling: signals at the site of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found m/z 361.2322. $C_{22}H_{25}D_4O_4$ [M-H]⁻ requires m/z 361.2317. The deviation of 1.4 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC

A sharp, symmetrical peak is observed (99.3 %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

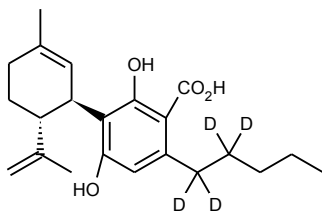
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials often contain higher levels of residual solvents and/or water, and we urge you to use the corrected purity where needed rather than the raw HPLC purity. This compound is intended for use as an analytical reference material and it is not for human administration. Structures are shown with relative stereochemistry unless otherwise specified.

The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

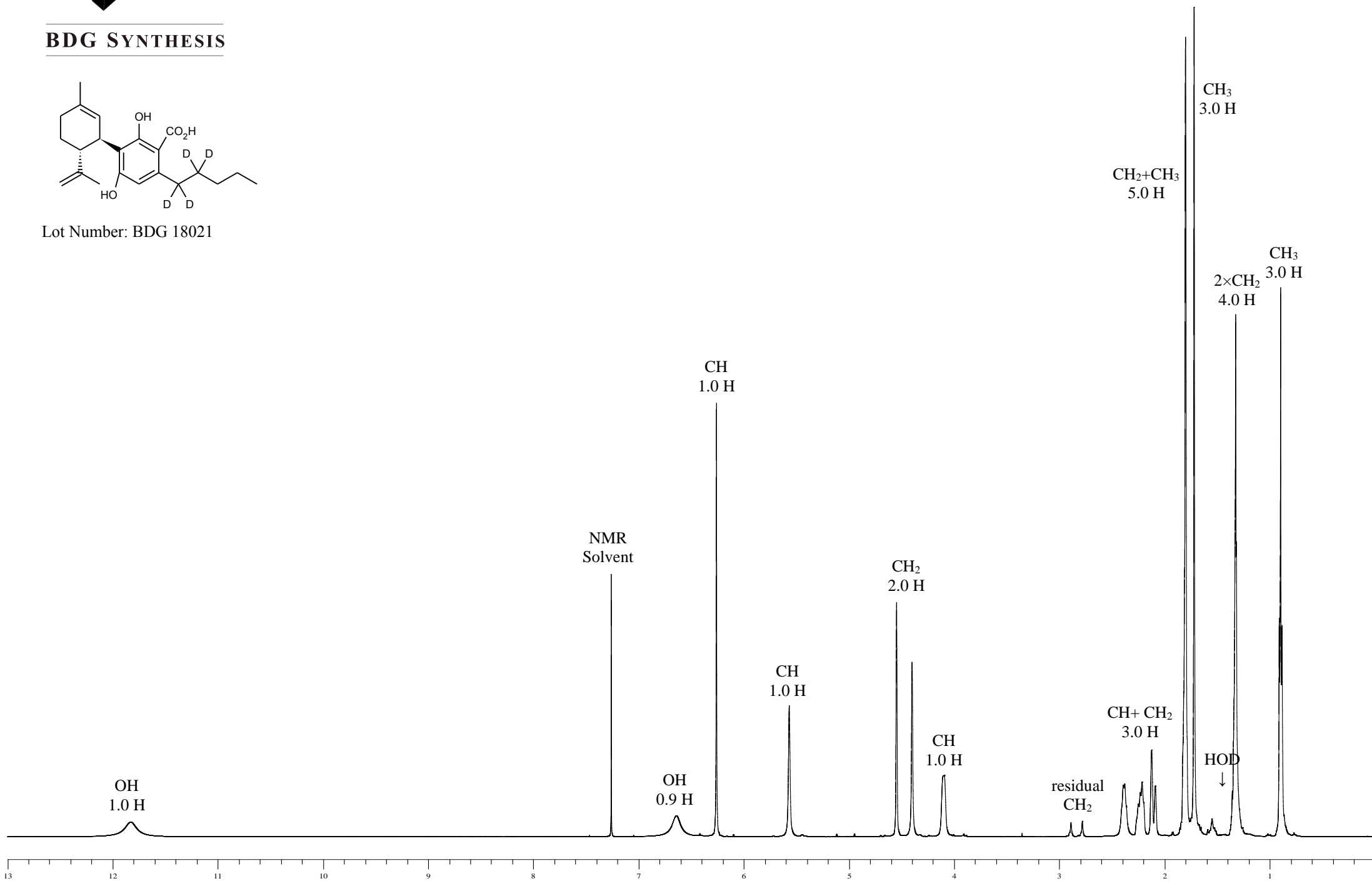


Proton NMR Spectrum of Cannabidiolic Acid-d₄ in CDCl₃

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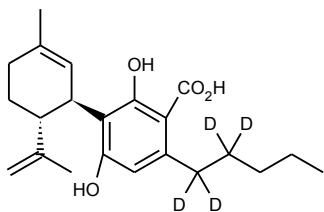
Lot Number: BDG 18021



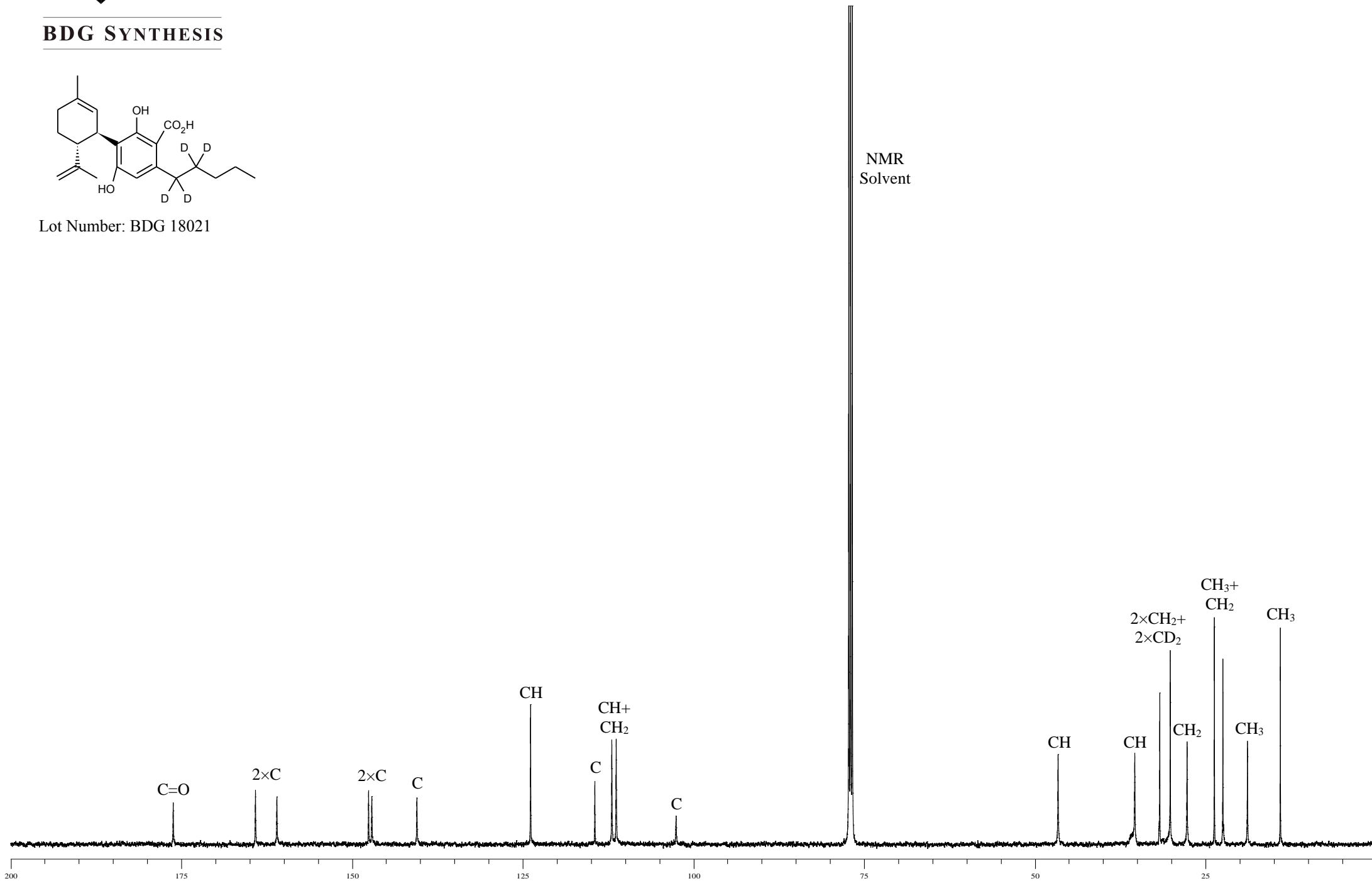


Carbon-13 NMR Spectrum of Cannabidiolic Acid-d₄ in CDCl₃

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Lot Number: BDG 18021



Analysis of BDG 18021, Cannabidiolic Acid-d₄

Date: 5th September 2022

Technician: Jennifer Hanley

Column: Phenomenex Luna C18(2), 5 µm, 250 x 4.6 mm

Guard: Phenomenex Security Guard C18, 4 x 3 mm

Mobile phase A: 22.5:77.5:0.05, Water : Acetonitrile : Formic Acid

Gradient (A): Isocratic, T₀ = 100, T₃₀ = 100,

Flow rate: 1.0 mL/min

Column temperature: 40 °C

Sample solvent: 22.5:77.5:0.05, Water : Acetonitrile : Formic Acid

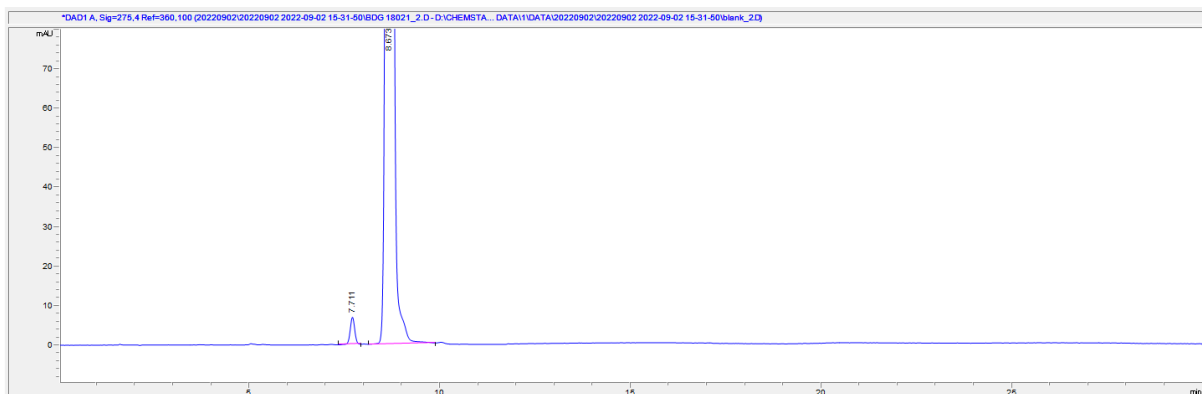
Sample concentration: ~1mg/mL

Injection volume: 5 µL

Detection: UV at 275 nm

Note: The sample appeared completely soluble in the sample solvent and was filtered (PTFE, 0.22 µm) before injection.

Sample with solvent blank subtraction:



#	Time	Type	Area	Height	Width	Area%	Symmetry
1	7.711	BB	56.8	6.9	0.1281	0.750	1.009
2	8.673	BB	7516.6	789.3	0.1471	99.250	0.827