



BDG SYNTHESIS

Certificate of Analysis

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

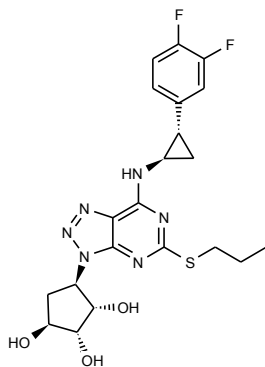
Neil Beare

Neil Beare, PhD, Director
11 July 2017

Name: Deshydroxyethylticagrelor

CAS Number: 220347-05-7

Structure:



Molecular Weight: $C_{21}H_{24}F_2N_6O_3S = 478.52$

Lot Number: BDG 12838.2

Appearance: White, crystalline solid

Corrected Purity: 99.1 % (HPLC) - 0.3 % (unidentified hydrocarbon) = 98.8 %

Re-test Date: 11 July 2022

Storage and Handling:

| | |
|--------------|---|
| Temperature: | refrigerate 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 the literature where available. All signals are duplicated indicating two conformers of the product exist in solution.

Residual Solvents: a small amount of unidentified hydrocarbon (0.3 % w/w) is observed.

Impurities: no significant impurities are evident in the spectrum.

Carbon-13 NMR Spectrum

Identity: The signals are consistent with the proposed structure and in accord with the literature where available. All signals are duplicated indicating two conformers of the product exist in solution.

High-resolution Mass Spectrum (ESI+)

Found m/z 479.1673. $C_{21}H_{25}F_2N_6O_3S$ $[M+H]^+$ requires m/z 479.1677. The deviation of 0.8 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.1 %). 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.

Elemental Analysis

| | | |
|--------------------------|-----------|----------------------------|
| | Found: | C 53.30, H 5.33, N 16.80 % |
| $C_{21}H_{24}F_2N_6O_3S$ | Requires: | C 52.71, H 5.06, N 17.56 % |

The elemental analyses fall slightly outside generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for due to sample size limitations).

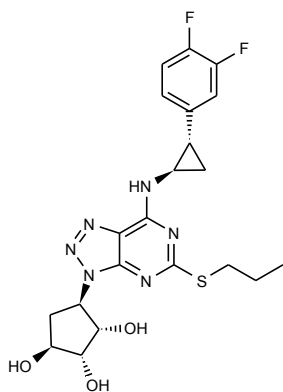
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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.

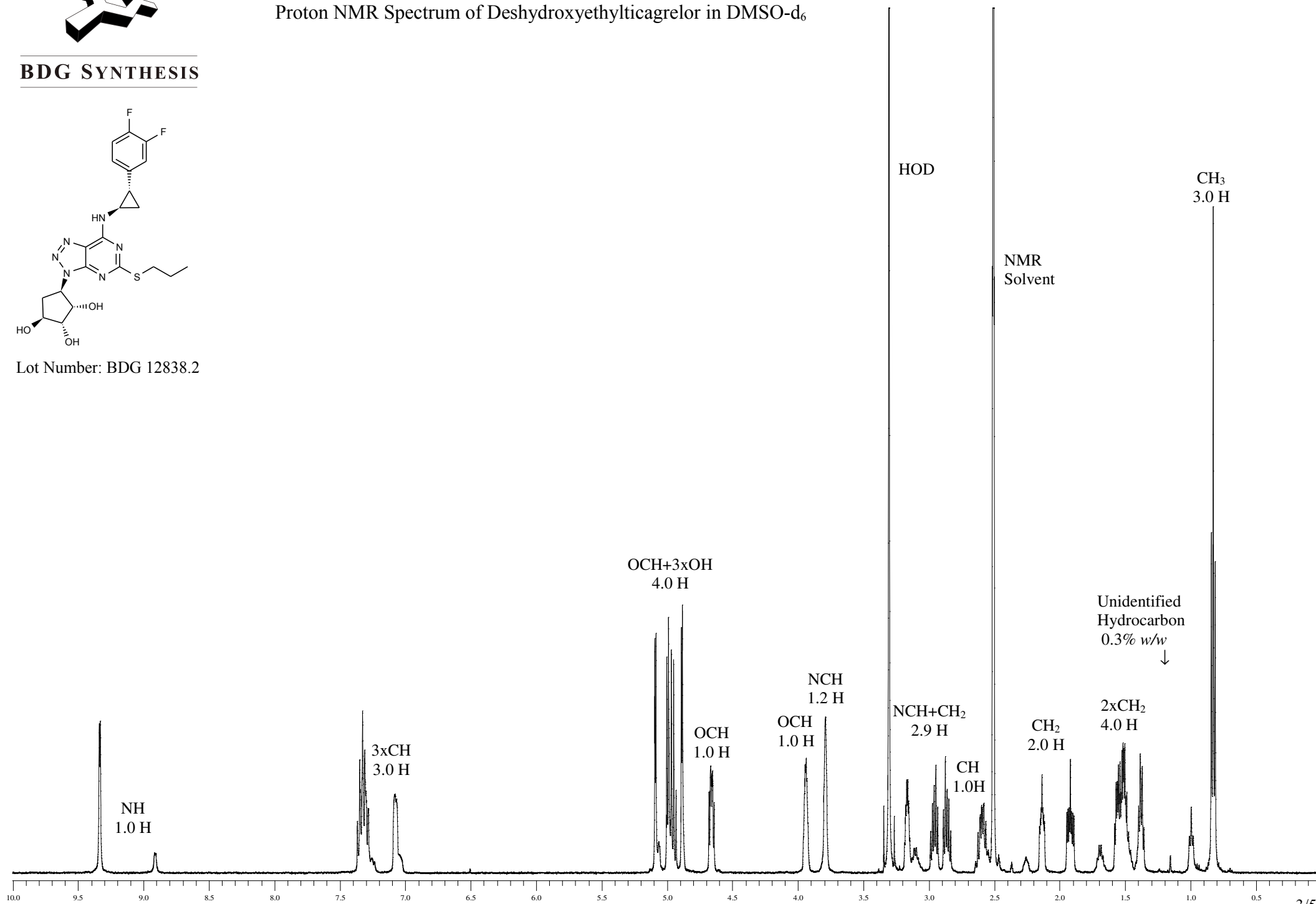


Proton NMR Spectrum of Deshydroxyethylticagrelor in DMSO-d₆

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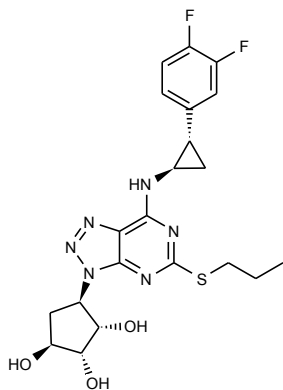
Lot Number: BDG 12838.2



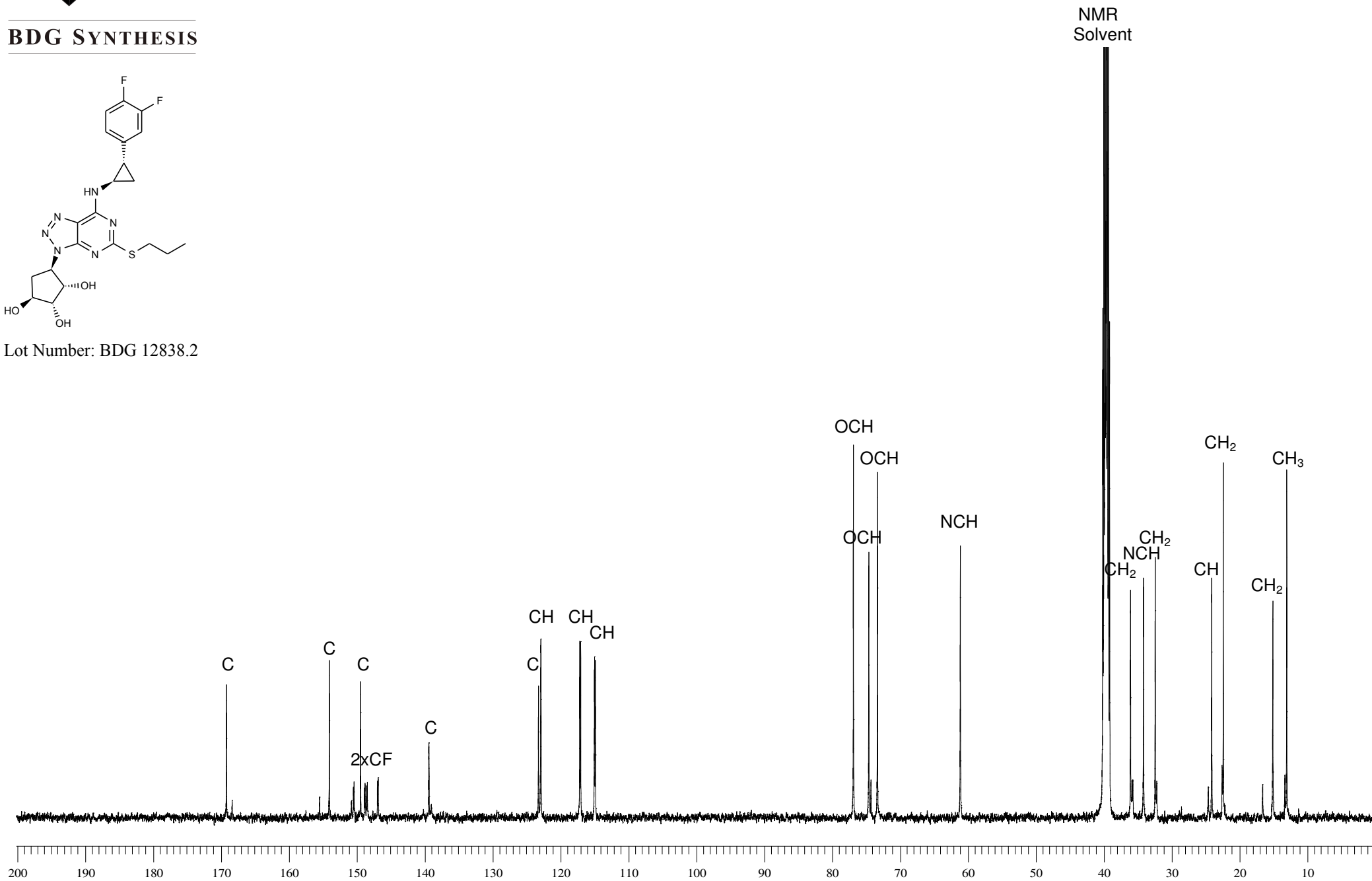


Carbon-13 NMR Spectrum of Deshydroxyethylticagrelor in DMSO-d₆

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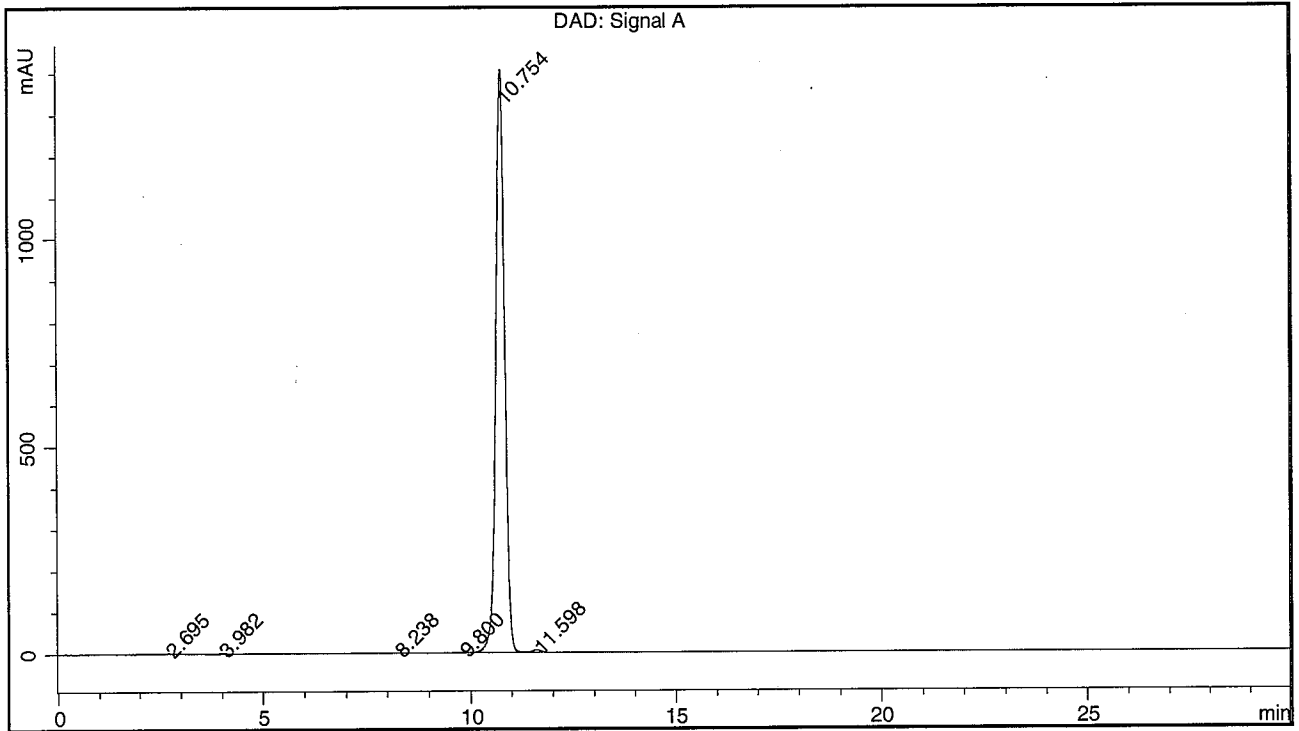
Lot Number: BDG 12838.2



BDG - Analysis of Deshydroxyethylticagrelor

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 50:50 20 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
 Flow Rate : 1.0 mL/min
 Sample Solvent : 50:50 Water : Acetonitrile
 Column Temperature : 20 C
 Injection Volume : 10 uL
 Detection : UV at 300 nm

| | | | |
|--------------------|------------------------------|----------------------|----------------|
| Sample Name | BDG 12838.2 | Instrument | AnalyticalLC01 |
| Acquisition | 11/07/2017, 13:43:00 | Method (rev.) | LC10585a (6) |
| Sequence | BDG_11Jul2017a - Reprocessed | Vial Position | 4 |
| Operator | solvation010\cerityadmin | Injection | 1 of 2 |



Area Percent Report

| Peak# | RT | Peak Height | Peak Area | Width | Area % |
|-------|-----------|-------------|------------|------------|----------|
| 1 | 2.70 min | 2.5507 | 12.2468 | 0.0740 min | 0.058 % |
| 2 | 3.98 min | 2.7080 | 29.7391 | 0.1501 min | 0.140 % |
| 3 | 8.24 min | 1.1557 | 15.0597 | 0.1780 min | 0.071 % |
| 4 | 9.80 min | 0.4402 | 6.0825 | 0.1932 min | 0.029 % |
| 5 | 10.75 min | 1409.0848 | 21063.4296 | 0.2304 min | 99.078 % |
| 6 | 11.60 min | 6.2364 | 132.8707 | 0.3046 min | 0.625 % |