

# **Certificate of Analysis**

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

Barry Dent

Barry R. Dent, PhD, Director 7 September 2013

Name: Ticagrelor

**CAS Number:** 274693-27-5

**Structure:** 

**Molecular Weight:**  $C_{23}H_{28}F_2N_6O_4S = 522.57$ 

Lot Number: BDG 13855.2

**Appearance:** White, crystalline solid

Purity By HPLC: 99.5 %

**Re-test Date:** 7 September 2014

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.

Version 1 (1d605) 1/5

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### **Identity and Purity**

### **Proton NMR Spectrum**

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

Residual Solvents: no residual solvents are 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 literature where available. All signals are duplicated indicating two conformers of the product exist in solution.

#### **High-resolution Mass Spectrum (ESI+)**

Found m/z 523.1938.  $C_{23}H_{29}F_2N_6O_4S$  [M+H]<sup>+</sup> requires m/z 523.1939. The deviation of 0.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

#### **HPLC**

A sharp, symmetrical peak is observed (99.5 %). 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 52.97, H 5.41, N 16.19 % C<sub>23</sub>H<sub>28</sub>F<sub>2</sub>N<sub>6</sub>O<sub>4</sub>S Requires: C 52.86, H 5.40, N 16.08 %

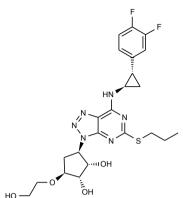
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

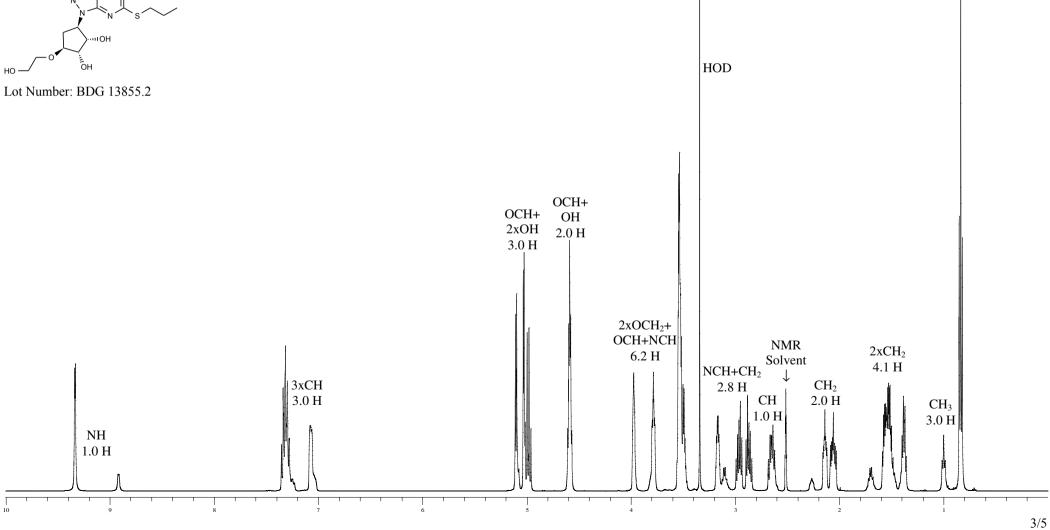
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

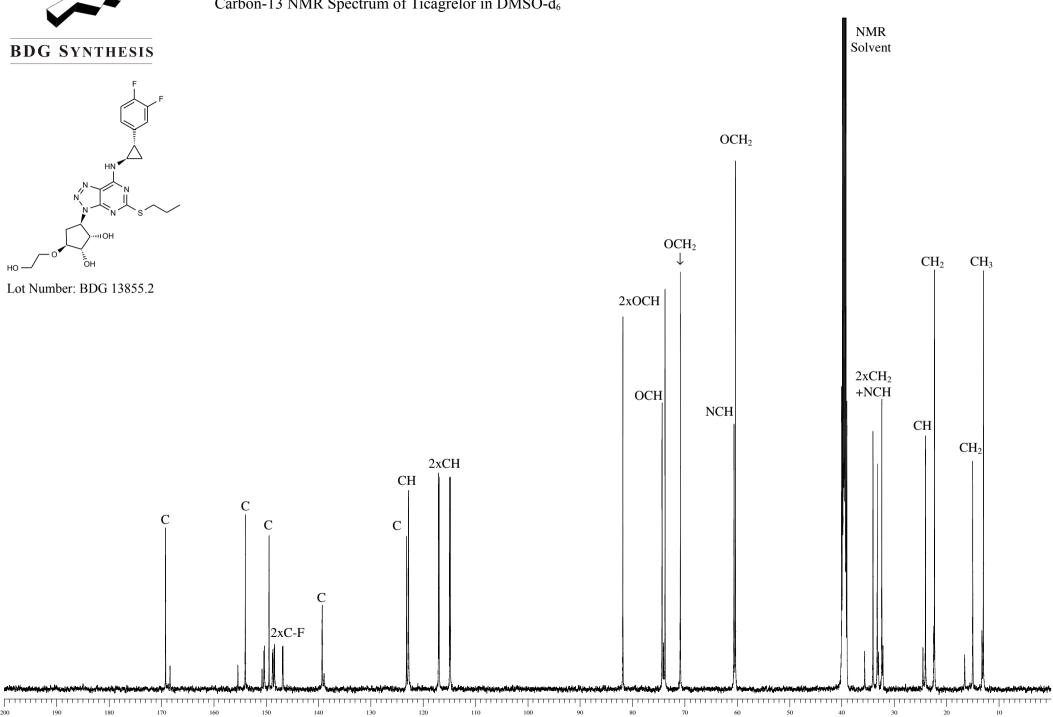


## **BDG SYNTHESIS**





# Carbon-13 NMR Spectrum of Ticagrelor in DMSO-d<sub>6</sub>

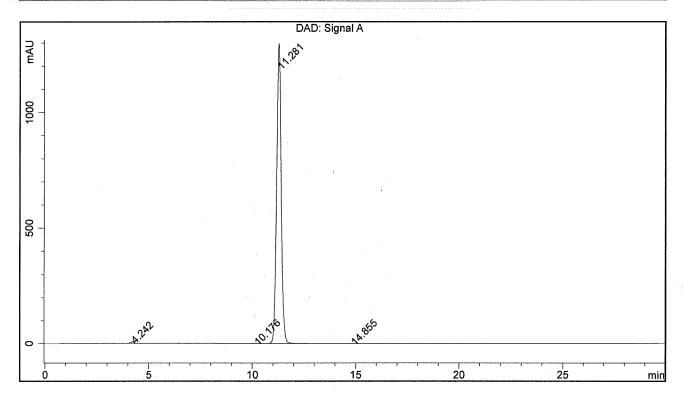


## BDG - Analysis of Ticagrelor

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 20mM Potassium diHydrogen Phosphate pH=3.0: Acetonitrile
Flow Rate: 1.0 mL/min
Sample Solvent: 50:50 Water: Acetonitrile
Column Temperature: 20C

Injection Volume : 10 uL Detection : UV at 300 nm

Sample Name	BDG 13855.2	Instrument	AnalyticalLC01
Acquisition	07/09/2013, 14:06:23	Method (rev.)	LC10584b ( 2)
Sequence	BDG_07Sep2013a	Vial Position	1
Operator	solvation010\cerityadmin	Injection	2 of 2



### **Area Percent Report**

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.24 min	7.6985	79.7770	0.1449 min	0.394 %
2	10.18 min	0.5192	11,1698	0.3310 min	0.055 %
3	11.28 min	1296.5996	20177.4207	0.2376 min	99.529 %
4	14.86 min	0.2141	4.4921	0.2608 min	0.022 %