

## BDG SYNTHESIS

### Certificate of Analysis

This material is a research-grade material obtained from the research department of a third-party manufacturer. The quantity available is limited, 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 research-grade materials. Research 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.

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

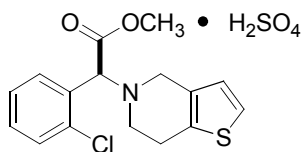
*Barry Dent*

Barry R. Dent, PhD, Director  
5 November 2008

**Name:** Clopidogrel Hydrogen Sulfate

**CAS Number:** 135046-48-9

**Structure:**



**Molecular Weight:**  $C_{16}H_{16}ClNO_2S \cdot H_2SO_4 = 419.90$

**Lot Number:** BDG 3412.1

**Appearance:** White, crystalline solid

**Purity by HPLC:** 99.4 %

**Expiry Date:** 5 November 2009

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

#### Storage and Handling:

Temperature: ambient laboratory temperature; may be refrigerated.

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:****Source of Material**

The material was a research-grade sample from a third-party manufacturer; any documentation supplied was reviewed and assessed as appropriate for the sample.

**Proton NMR Spectrum**

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

Residual solvents: no residual solvents are observed.

Impurities: traces of unidentified impurities are seen in the baseline.

**Carbon-13 NMR Spectrum**

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

**High-resolution mass spectrum (FAB+):** found  $m/z$  322.0661.  $C_{16}H_{17}^{35}ClNO_2S$  [free base,  $M+H$ ]<sup>+</sup> requires  $m/z$  322.0669. The deviation of 2.5 ppm is within normally accepted limits for the establishment of identity by HRMS.

**HPLC:** A broad, slightly tailing peak is observed (99.4 area %). 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.

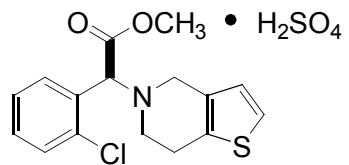
**Elemental Analysis:** Found: C 46.00, H 4.28, N 3.20 %

$C_{16}H_{16}ClNO_2S \cdot H_2SO_4$  requires: C 45.77, H 4.32, N 3.34 %

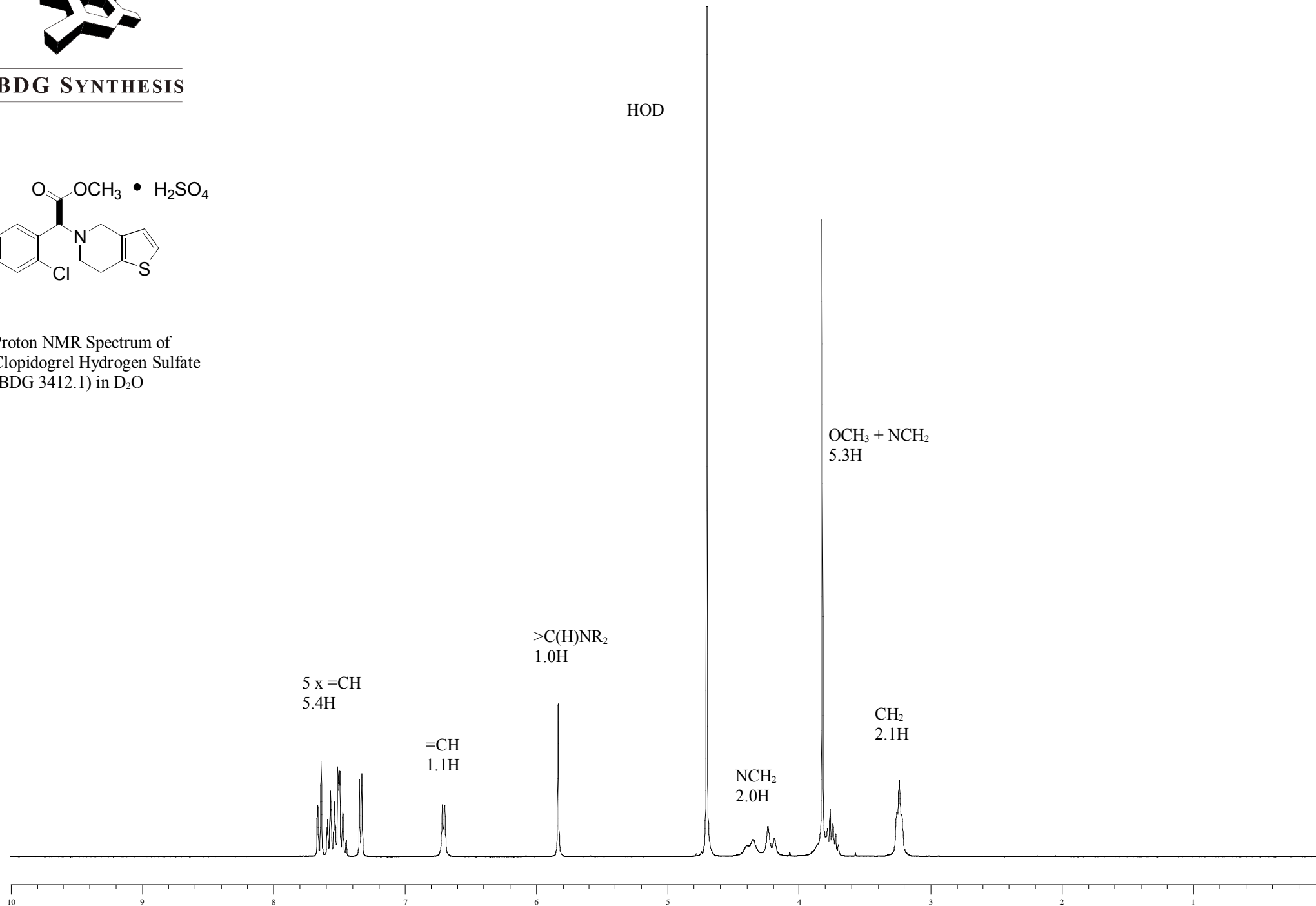
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).



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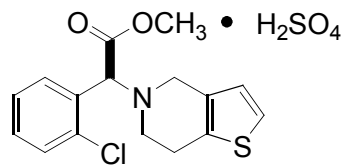


Proton NMR Spectrum of Clopidogrel Hydrogen Sulfate (BDG 3412.1) in D<sub>2</sub>O

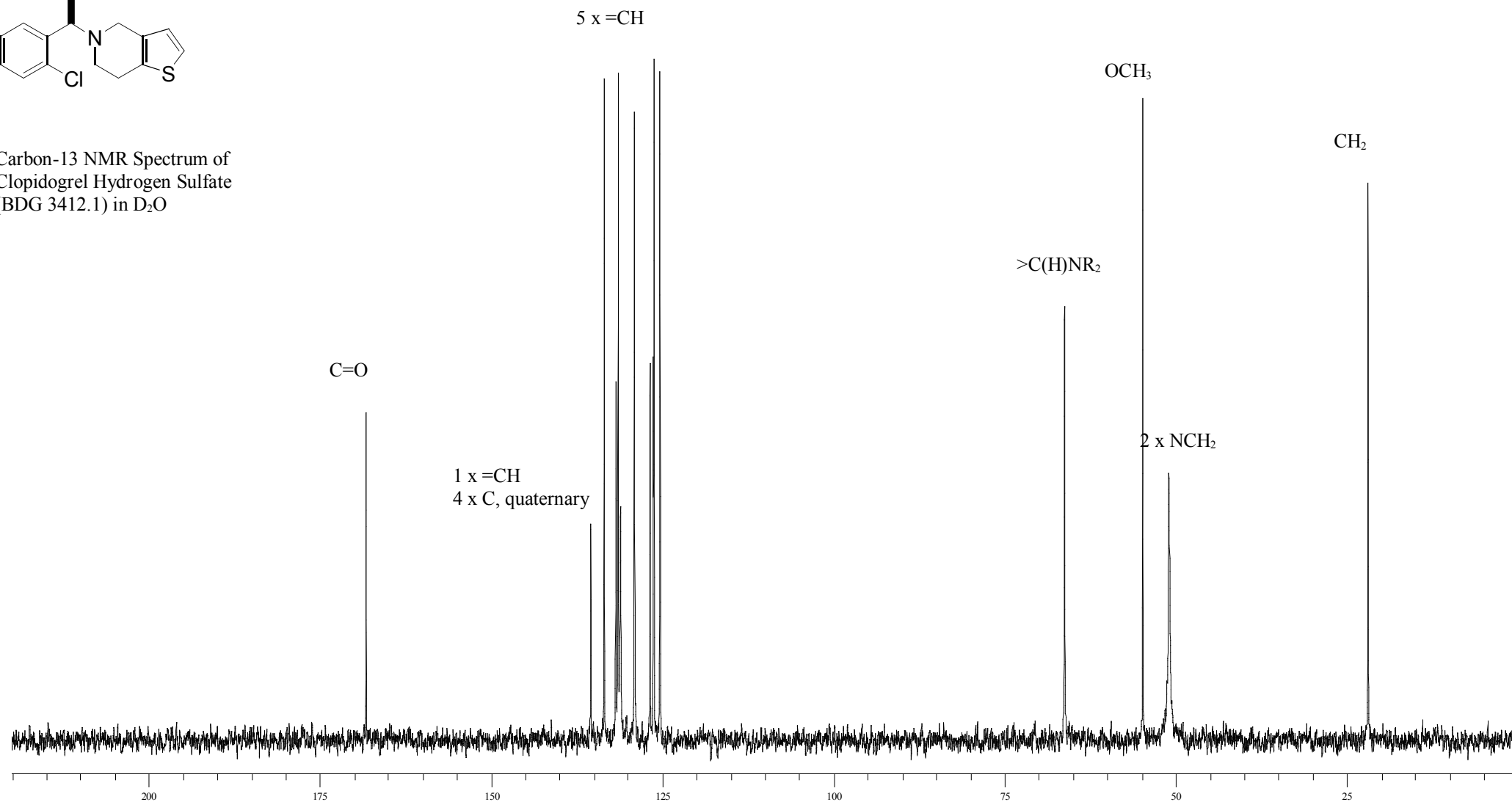




# BDG SYNTHESIS



Carbon-13 NMR Spectrum of Clopidogrel Hydrogen Sulfate (BDG 3412.1) in D<sub>2</sub>O



## BDG - Analysis of Clopidogrel Hydrogen Sulphate

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

Guard : Phenomenex Security Guard C18 RP 4 x 3 mm

Mobile Phase : 35/65 25mM Potassium diHydrogen Phosphate pH 3.0: Acetonitrile

Flow Rate : 1.0 mL/min

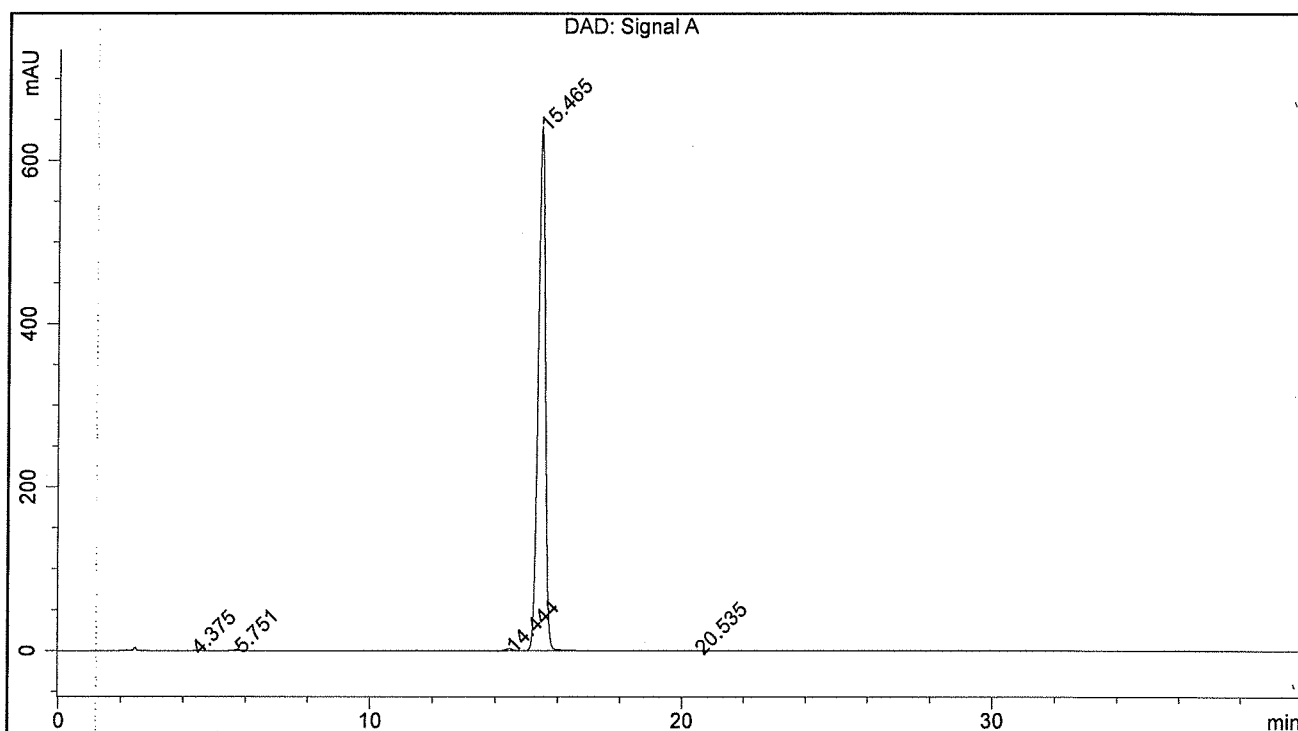
Sample Solvent : Mobile Phase

Column Temperature : 20C

Injection Volume : 10 uL

Detection : UV at 240 nm

Sample Name	BDG 3412.1	Instrument	AnalyticalLC01
Acquisition	05/11/2008, 13:04:57	Method (rev.)	LC10223c ( 5)
Sequence	BDG_05Nov2008d - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	1 of 1



## Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.38 min	0.1244	0.6208	0.0742 min	0.006 %
2	5.75 min	1.6530	15.9725	0.1387 min	0.152 %
3	14.44 min	2.5354	38.7705	0.2364 min	0.369 %
4	15.46 min	640.7683	10451.4763	0.2543 min	99.433 %
5	20.53 min	0.1951	4.1922	0.2926 min	0.040 %