BDG SYNTHESIS

Certificate of Analysis

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

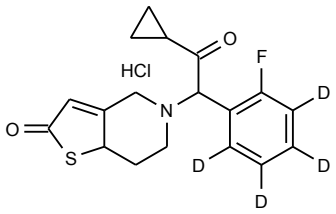
Barry Dent

Barry R. Dent, PhD, Director
11 October 2012

Name: **2-Oxoprasugrel-d_4 HCl**

CAS Number: 150322-38-6 (unlabelled free base)

Structure:



Molecular Weight: $C_{18}H_{14}D_4FNO_2S \cdot HCl = 371.89$

Lot Number: BDG 13205.2

Appearance: Off-white, crystalline solid

Corrected Purity: 98.6 % (HPLC) - 1.3 % (diethyl ether) - 3.7 % (water) = 93.6 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 11 October 2017

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 literature where available. The spectrum is complicated, as expected, by the presence of diastereoisomers and the 2-hydroxy form of the product.
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: a small amount of diethyl ether (1.3 % *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 literature where available. The spectrum is complicated, as expected, by the presence of diastereoisomers and the 2-hydroxy form of the product.
Isotopic Labelling: signals at the sites 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* 336.1365. $C_{18}H_{15}D_4FNO_2S$ $[M+H]^+$ requires *m/z* 336.1372. The deviation of 2.1 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

Three sharp, symmetrical peaks are observed (98.6 % total). The first-eluting peak (11.7 area %) is assigned as the 2-hydroxy form of the product by LCMS experiments. 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 55.80, H 4.21, D 2.25, N 3.51 %
$C_{18}H_{14}D_4FNO_2S{\cdot}HCl{\cdot}0.8H_2O$	Requires:	C 55.96, H 4.33, D 2.09, N 3.63 %, H_2O 3.73 %
$C_{18}H_{14}D_4FNO_2S{\cdot}HCl$	Requires:	C 58.13, H 4.07, D 2.17, N 3.77 %

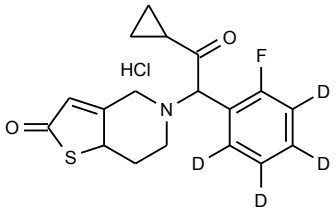
The elemental analyses fall somewhat outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the “best-fit” hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend that the “best-fit” water content be used when determining corrected purity.

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

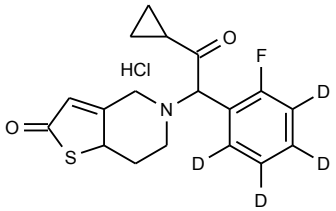
Proton NMR Spectrum of 2-Oxoprasugrel-d_4 HCl in DMSO-d_6



Lot Number: BDG 13205.2

BDG SYNTHESIS

Carbon-13 NMR Spectrum of 2-Oxoprasugrel-d_4 HCl in DMSO-d_6



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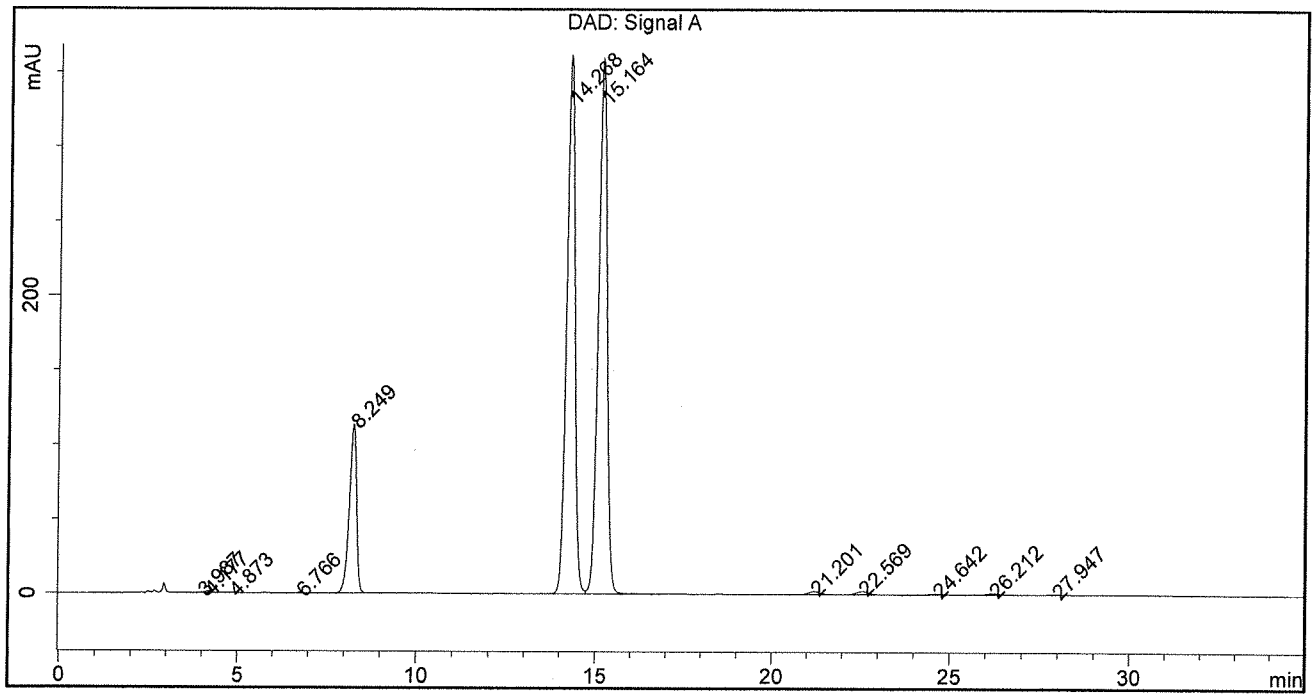
BDG - Analysis of 2-Oxoprasugrel-d4 HCl

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 KH2PO4 pH 3.0 : Acetonitrile
Column Temperature : 20C . . . . . Flow Rate : 1.0 mL/min . . . . . Detection : UV at 220 nm
Sample Solvent : 1:1 Water : Acetonitrile . . . . . Injection Volume : 10 uL

Sample Name	BDG 13205.2	**Instrument**	AnalyticalLC01
Acquisition	11/10/2012, 08:33:54	**Method (rev.)**	LC10538a (9)
Sequence	BDG_11Oct2012b - Reprocessed	**Vial Position**	37
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.99 min	0.4772	3.2651	0.0978 min	0.024 %
2	4.18 min	1.0993	10.0676	0.1288 min	0.073 %
3	4.87 min	0.4027	2.8215	0.1097 min	0.021 %
4	6.77 min	0.5257	5.0296	0.1396 min	0.037 %
5	8.25 min	113.4114	1601.5593	0.2188 min	11.672 %
6	14.27 min	361.8070	5814.8252	0.2475 min	42.378 %
7	15.16 min	359.0933	6110.6755	0.2625 min	44.535 %
8	21.20 min	2.1123	50.0146	0.3482 min	0.365 %
9	22.57 min	2.1655	54.7410	0.3469 min	0.399 %
10	24.64 min	0.8131	22.3226	0.3363 min	0.163 %
11	26.21 min	0.9240	35.9915	0.4704 min	0.262 %
12	27.95 min	0.3002	9.8785	0.3959 min	0.072 %