BDG SYNTHESIS

Certificate of Analysis

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

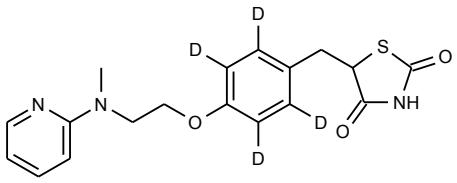
Barry Dent

Barry R. Dent, PhD, Director
26 March 2012

Name: **Rosiglitazone-d_4**

CAS Number: 122320-73-4 (unlabelled)

Structure:



Molecular Weight: $C_{18}H_{15}D_4N_3O_3S$ = 361.45

Lot Number: BDG 4462

Appearance: Pale yellow, crystalline solid

Purity By HPLC: 96.2 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 26 March 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.

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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.
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: 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.
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 (FAB+)

Found *m/z* 362.1469. $C_{18}H_{16}D_4N_3O_3S$ $[M+H]^+$ requires *m/z* 362.1476. The deviation of 2.2 ppm is within normally accepted limits for the establishment of identity by HRMS. Peaks for M-1 and M-2 are observed but the peak for M-4 (d_0) is present at background level.

HPLC

A sharp, symmetrical peak is observed (96.2 %). 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 59.35, H 4.21, D 2.24, N 11.54 %
$C_{18}H_{15}D_4N_3O_3S$	Requires:	C 59.81, H 4.18, D 2.23, N 11.63 %

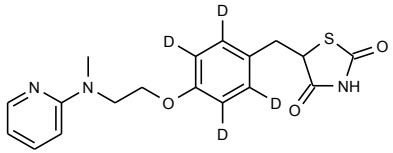
The elemental analyses fall within generally accepted limits (+/- 0.4 %) for establishing the molecular formula given, except the result for carbon. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because 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

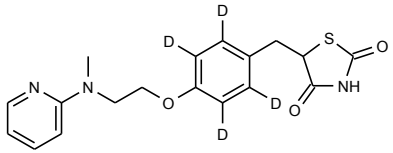
Proton NMR Spectrum of Rosiglitazone-d_4 in DMSO-d_6



Lot Number: BDG 4462

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Rosiglitazone-d_4 in DMSO-d_6



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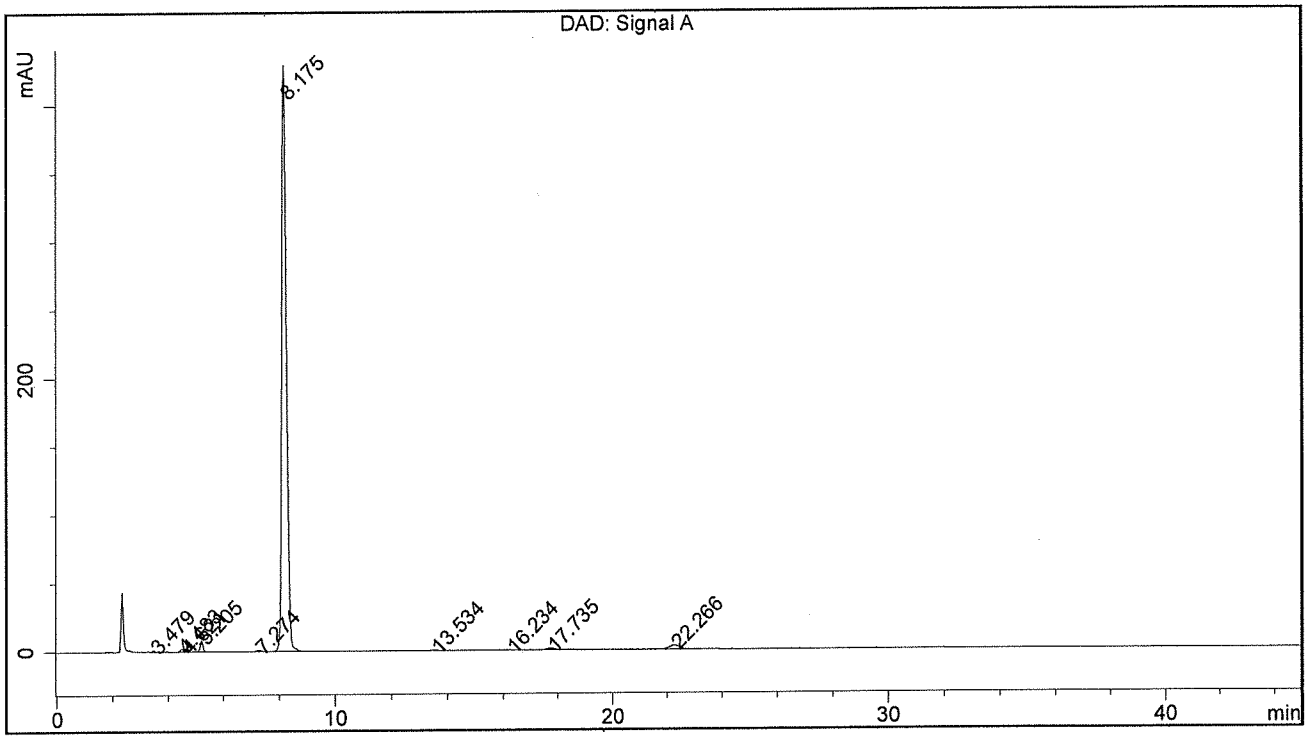
BDG - Analysis of Rosiglitazone-d4

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex SecurityGuard C18 4 x 3mm
Mobile Phase : 60:40 10 mM Ammonium Acetate pH=8.0 : Acetonitrile
Sample Solvent : Mobile Phase
Column Temperature : 50C
Injection Volume : 10 uL
Detection : UV at 247 nm

Sample Name	BDG 4462	**Instrument**	AnalyticalLC01
Acquisition	26/03/2012, 19:23:30	**Method (rev.)**	LC10498a (3)
Sequence	BDG_26Mar2012b - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.48 min	1.2136	5.7718	0.0735 min	0.109 %
2	4.48 min	1.9253	14.2432	0.1123 min	0.268 %
3	4.62 min	1.2548	7.8161	0.0927 min	0.147 %
4	5.21 min	7.9005	56.4224	0.1113 min	1.063 %
5	7.27 min	1.1059	10.1818	0.1394 min	0.192 %
6	8.18 min	429.5172	5106.3927	0.1847 min	96.169 %
7	13.53 min	0.4584	7.3233	0.2264 min	0.138 %
8	16.23 min	0.2824	5.4058	0.2373 min	0.102 %
9	17.74 min	1.1579	22.6456	0.2802 min	0.426 %
10	22.27 min	2.9197	73.6194	0.3742 min	1.386 %