BDG SYNTHESIS

Certificate of Analysis

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

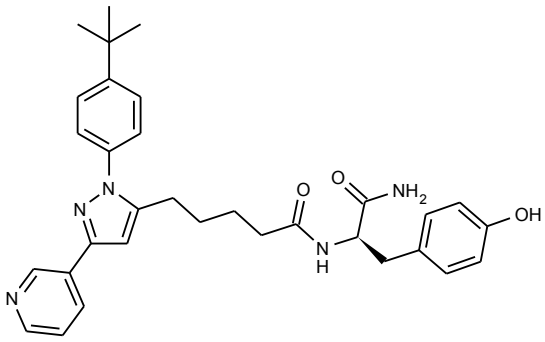
Barry Dent

Barry R. Dent, PhD, Director
30 April 2010

Name: **Pyrazole 10**

CAS Number: 373607-06-8

Structure:



Molecular Weight: $C_{32}H_{37}N_5O_3$ = 539.67

Lot Number: BDG 12018.3

Appearance: White, crystalline solid

Corrected Purity: 99.4 % (HPLC) - 1.5 % (unidentified hydrocarbon) - 1.8 % (water) = 96.1 %

Re-test Date: 30 April 2011

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

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Residual Solvents: a small amount of unidentified hydrocarbon (1.5 % *w/w*) is observed. This was calculated as 1.5% *w/w* hexane.
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.
Impurities: traces of unidentified impurities are seen in the baseline.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 540.2980. $C_{32}H_{38}N_5O_3$ $[M+H]^+$ requires *m/z* 540.2969. The deviation of 2.0 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.4 %). 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 70.44, H 7.17, N 12.60 %
$C_{32}H_{37}N_5O_3 \cdot 0.4H_2O$	Requires:	C 70.28, H 6.97, N 12.81 %
$C_{32}H_{37}N_5O_3$	Requires:	C 71.22, H 6.91, N 12.98 %

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.

Karl-Fischer Analysis

	Found:	H_2O 1.8 %
$C_{32}H_{37}N_5O_3 \cdot 0.4H_2O$	Requires:	H_2O 1.3 %

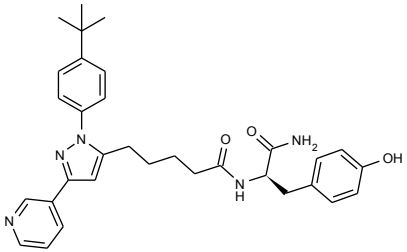
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

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

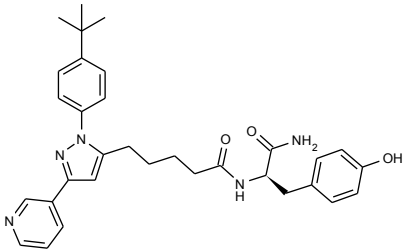
Proton NMR Spectrum of Pyrazole 10 in DMSO-d_6



Lot Number: BDG 12018.3

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Pyrazole 10 in DMSO-d_6

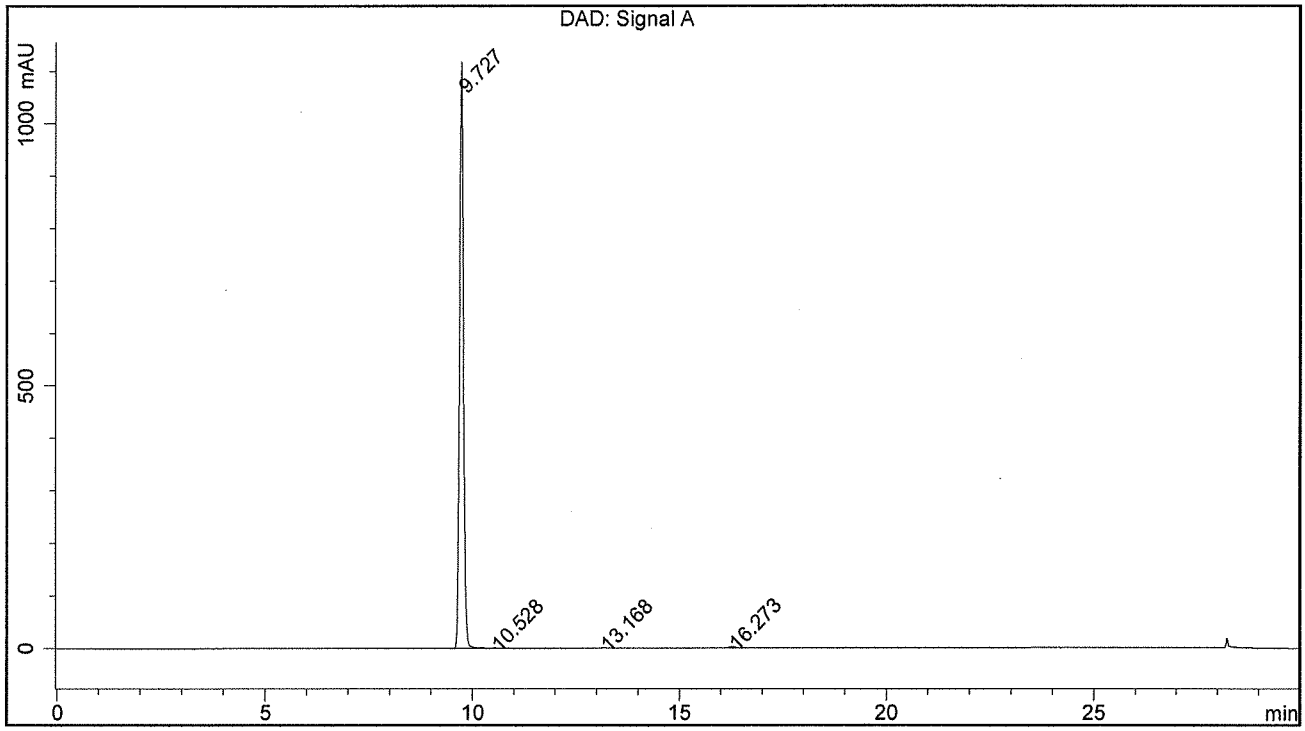


Lot Number: BDG 12018.3

BDG - Analysis of Pyrazole 10

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A : 60:40 Water : Acetonitrile
Mobile Phase B : Acetonitrile
Gradient (A:B) : T0=100:0, T20=0:100, T24=0:100, T27=100:0, T30=100:0
Flow Rate : 1.0 mL/min
Column Temperature : 20C
Detection : UV 266 nm
Sample Solvent : Initial Mobile Phase
Injection Volume : 10 uL

Sample Name	BDG 12018.3	**Instrument**	AnalyticalLC01
Acquisition	30/04/2010, 17:43:25	**Method (rev.)**	LC10378a (7)
Sequence	BDG_30Apr2010c - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	9.73 min	1118.7897	7363.6253	0.1008 min	99.445 %
2	10.53 min	0.5428	8.7054	0.2091 min	0.118 %
3	13.17 min	0.8474	7.0632	0.1252 min	0.095 %
4	16.27 min	2.6344	25.3552	0.1463 min	0.342 %