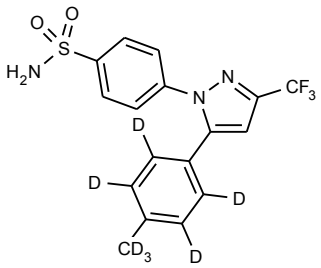
BDG SYNTHESIS

Certificate of Analysis

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

Neil Beare, PhD, Director
26 May 2016

Name:	**Celecoxib-d_7**
CAS Number:	169590-42-5 (unlabelled)
Structure:	
Molecular Weight:	$C_{17}H_7D_7F_3N_3O_2S$ = 388.42
Lot Number:	BDG 11293.9
Appearance:	White powder
Corrected Purity:	99.5 % (HPLC) - 0.2 % (dichloromethane) = 99.3 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	26 May 2021

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. The material is susceptible to static.

Mailing:
BDG Synthesis 2014 Limited
PO Box 38627, Wellington Mail Centre,
Wellington 5045, New Zealand.

Shipping:
BDG Synthesis 2014 Limited
Gracefield Research Centre, Building F,
69 Gracefield Rd, Lower Hutt 5010, New Zealand.

Phone: + 64 4 569 0520
Fax: + 64 4 569 0521

info@bdg.co.nz
www.bdg.co.nz

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 the spectrum of unlabelled material, indicating near complete deuteration. A small amount of H/D exchange is observed.
Residual Solvents: a small amount of dichloromethane (0.2 % *w/w*) is observed.
Impurities: a trace of an unidentified impurity is 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 the spectrum of unlabelled material, indicating near complete deuteration.

High-resolution Mass Spectrum (TOF MS ES+)

Found *m/z* 389.1272. $C_{17}H_8D_7F_3N_3O_2S$ $[M+H]^+$ requires *m/z* 389.1276. The deviation of 1.0 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 %). A signal for d_6 material was observed.

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 52.72, H 1.78, D 3.56, N 10.93 %
$C_{17}H_7D_7F_3N_3O_2S$	Requires:	C 52.57, H 1.82, D 3.63, N 10.82 %

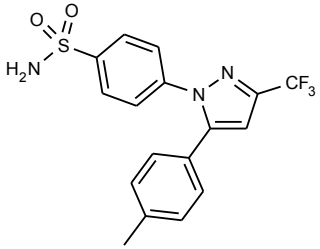
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

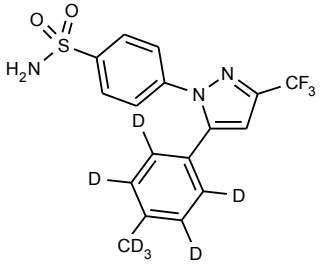
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

Proton NMR Spectrum of Celecoxib (top) and Celecoxib-d_7 (bottom) in Methanol-d_4

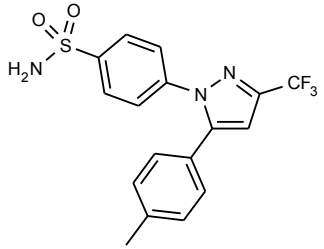




Lot Number: BDG 11293.9

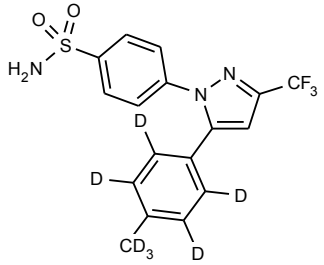
BDG SYNTHESIS

Carbon-13 NMR Spectrum of Celecoxib (top) and Celecoxib-d_7 (bottom) in Methanol-d_4



8xCH

CH_3



4xCH

NMR Solvent

Lot Number: BDG 11293.9

CH

5xC

←C

4xCD

CF_3

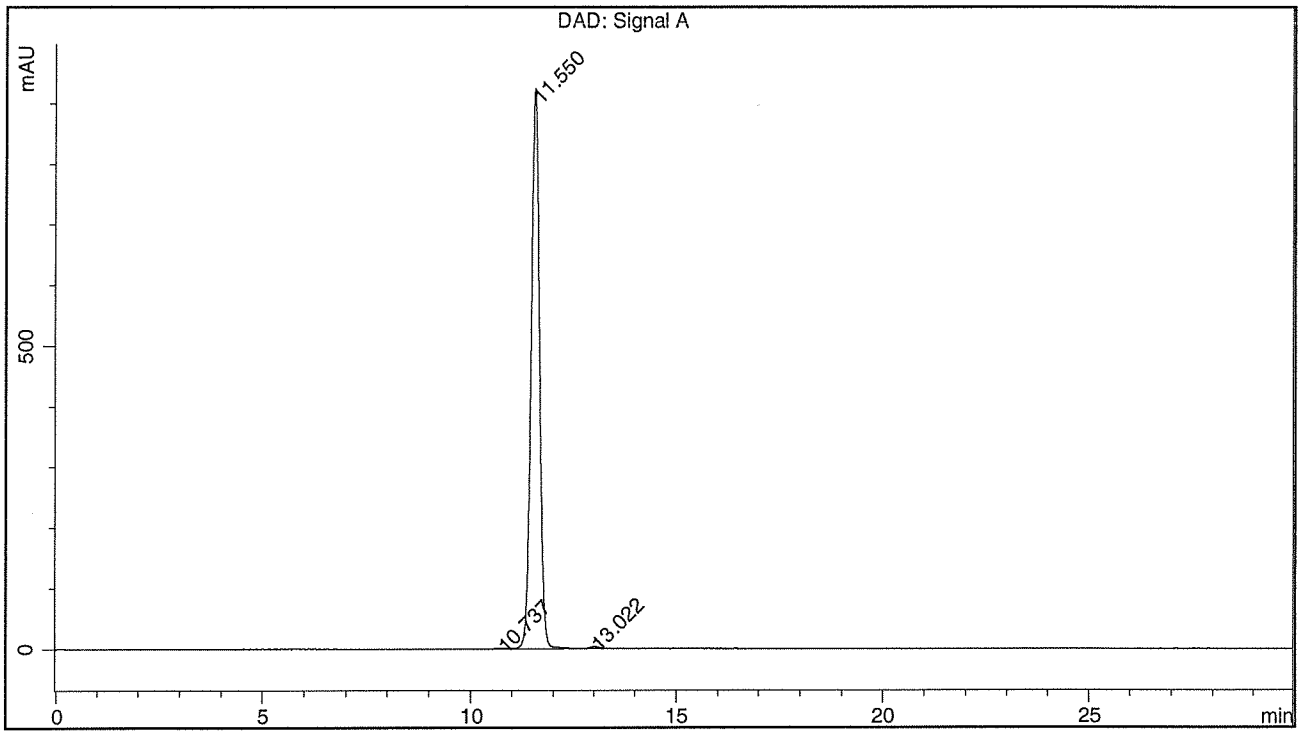
CD_3

200 175 150 125 100 75 50 25

BDG - Analysis of Celecoxib-d7

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 40:60 10 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
Flow Rate : 1.0 mL/min . . . . . Sample Solvent : Mobile Phase
Column Temperature : 20 C . . . . . Injection Volume : 10 uL . . . . . Detection : UV at 250 nm

Sample Name	BDG 11293.9	Instrument	AnalyticalLC01
Acquisition	26/05/2016, 10:00:27	Method (rev.)	LC10390b (4)
Sequence	BDG_26May2016a	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	10.74 min	0.2694	4.6136	0.2598 min	0.035 %
2	11.55 min	924.5138	13229.7441	0.2229 min	99.522 %
3	13.02 min	3.3227	58.9230	0.2669 min	0.443 %