

## BDG SYNTHESIS

### Certificate of Analysis

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

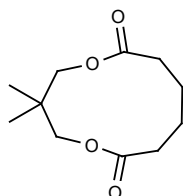
*Neil Beare*

Neil Beare, PhD, Director  
26 February 2019

**Name:** 3,3-Dimethyl-1,5-dioxacycloundecane-6,11-dione

**CAS Number:** 94113-47-0

**Structure:**



**Molecular Weight:**  $C_{11}H_{18}O_4 = 214.26$

**Lot Number:** BDG 17454.3

**Appearance:** White powder

**Purity By HPLC:** 99.6 %

**Re-test Date:** 26 February 2020

**Storage and Handling:** Temperature: freeze ( $-20^{\circ}C$ ) 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.

Residual Solvents: no residual solvents are 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.

### High-resolution Mass Spectrum (ESI+)

Found  $m/z$  237.1105.  $C_{11}H_{18}O_4Na$   $[M+Na]^+$  requires  $m/z$  237.1103. The deviation of 0.8 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A somewhat broadened, symmetrical peak is observed (99.6 %). 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 61.79, H 7.85 % |
| $C_{11}H_{18}O_4$ | Requires: | C 61.66, H 8.47 % |

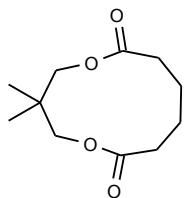
The elemental analyses fall within generally accepted limits (+/- 0.4 %) for establishing the molecular formula given, except the result for hydrogen. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because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

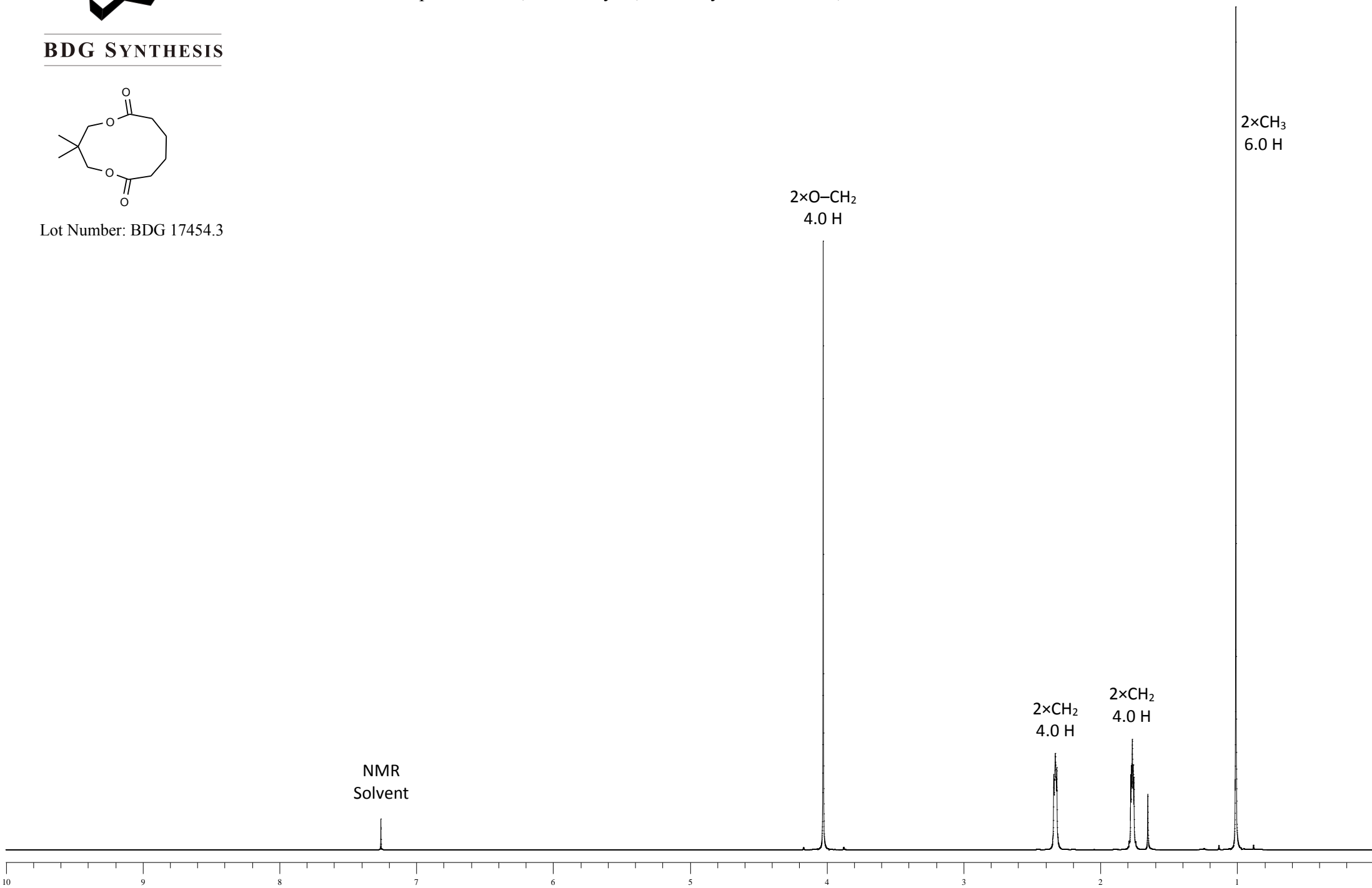


**BDG SYNTHESIS**



Lot Number: BDG 17454.3

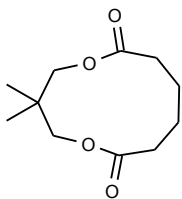
Proton NMR Spectrum of 3,3-Dimethyl-1,5-dioxacycloundecane-6,11-dione in CDCl<sub>3</sub>



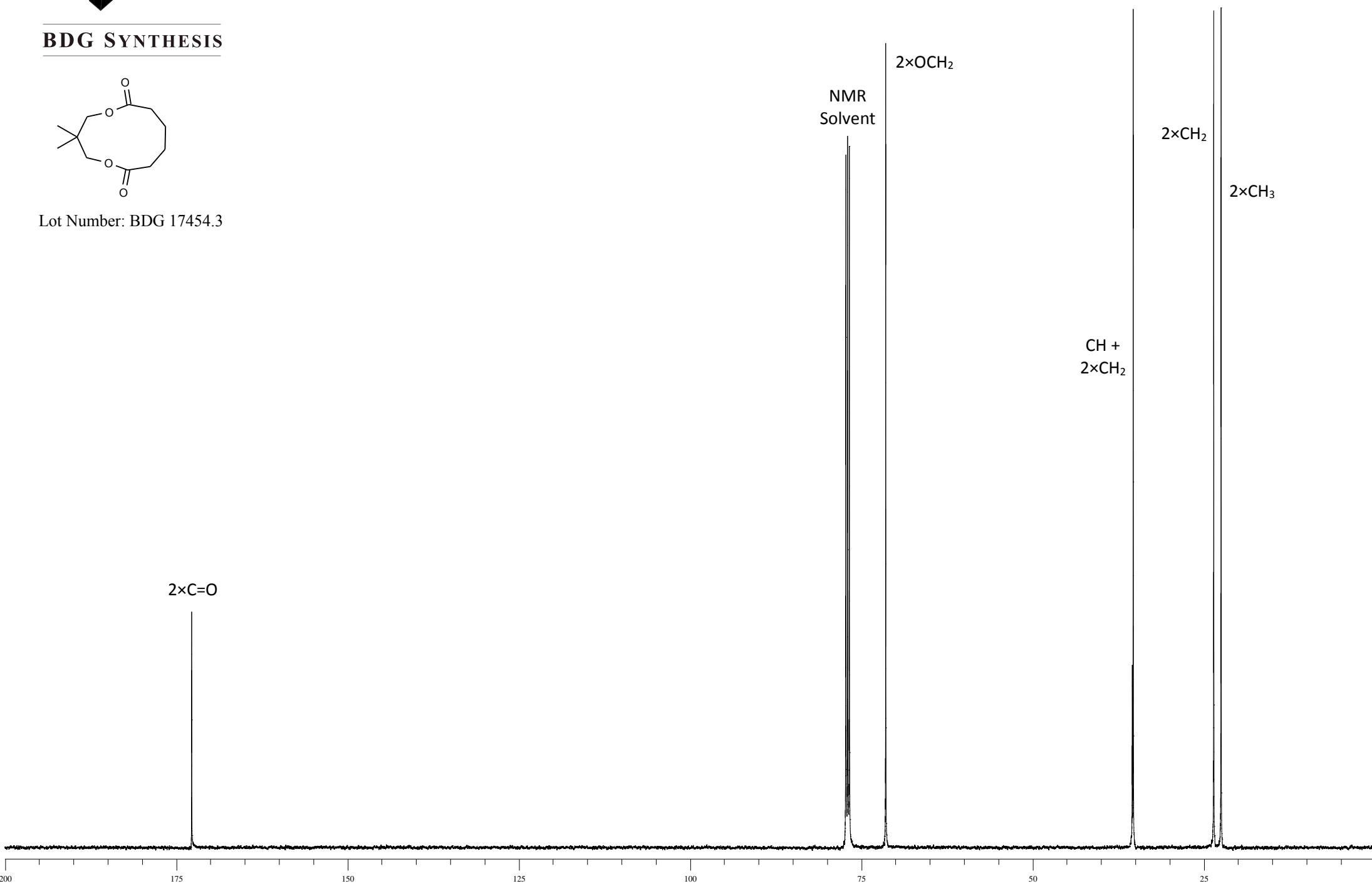


Carbon-13 NMR Spectrum of 3,3-Dimethyl-1,5-dioxacycloundecane-6,11-dione in CDCl<sub>3</sub>

**BDG SYNTHESIS**



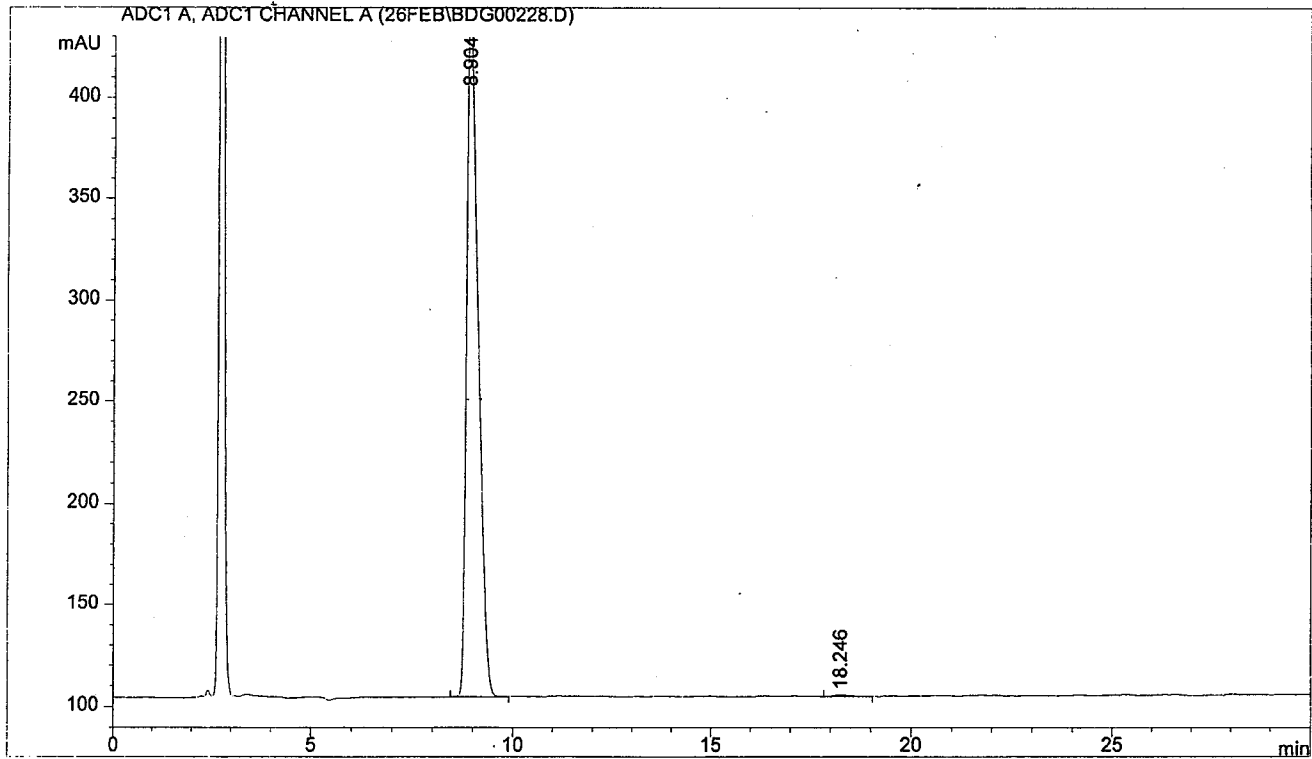
Lot Number: BDG 17454.3



```

=====
Acq. Operator   : Bruce Hamilton           Seq. Line :    1
Acq. Instrument : Instrument 1             Location  : Vial 1
Injection Date  : 2/26/2019 3:42:53 PM    Inj       :    1
                                           Inj Volume: 40 µl
Method         : C:\CHEM32\1\METHODS\2018\LC20068B.M
Last changed   : 2/26/2019 2:25:45 PM by Bruce Hamilton
Method Info    : BDG - Analysis of 3,3-Dimethyl-1,5-dioxacycloundecane-6,11-dione
                Column : Phenomenex Luna C18(2) 5 µm 250 x 4.6 mm : Guard C18RP 4 x 3 mm
                Mobile Phase : 40:60:0.05 Water : Methanol : Trifluoroacetic Acid
                Sample Solvent : 30:70 Water : Methanol
                Flow : 1 ml/min., Column Temperature : 30 C, Injection : 40 µl,
                Detection : Refractive Index
=====

```



=====  
Area Percent Report  
=====

```

Sorted By      : Signal
Multiplier    : 1.0000
Dilution      : 1.0000

```

Signal 1: ADC1 A, ADC1 CHANNEL A

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area %  |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1      | 8.904         | BV   | 0.2932      | 6766.66846   | 344.41644    | 99.6171 |
| 2      | 18.246        | PB   | 0.3935      | 26.01005     | 8.30277e-1   | 0.3829  |

Totals :                                   6792.67850   345.24672

Results obtained with enhanced integrator!

=====  
\*\*\* End of Report \*\*\*