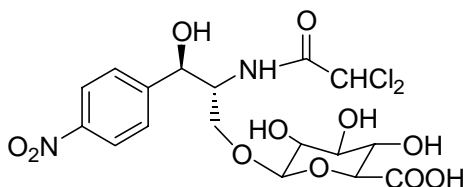




REFERENCE MATERIAL ANALYSIS REPORT

Compound Name: **Chloramphenicol glucuronide**
Collection No.: D714
Chemical Formula: $C_{17}H_{20}N_2O_{11}Cl_2$
CAS Number: 39751-33-2
Structure:

Description: Tan powder
Batch No: 01-AV-12
Molecular Weight: 499.3
Batch production completed: June 2002



Chloramphenicol glucuronide content of ampouled material = $65.7 \pm 5.0\%$ (mass fraction)

Organic purity of chloramphenicol glucuronide in ampouled D714 is 92.3%

Note: Moisture, solvent and acetate salt associated with the material comprise approximately 30% by mass of D714. Each ampoule contains the equivalent of $657 \pm 50 \mu\text{g}$ of chloramphenicol glucuronide.

QNMR: Instrument: Bruker DMX-500
Field strength: 500 MHz Solvent: D_2O
Internal standard: Dimethyl sulfone
Purity estimate: 69% (mass fraction %, mean of seven samples, $s = 2.7\%$, June 2002)

HPLC: Column: Alltima C18, 5 μm (4 mm \times 150 mm)
Mobile Phase: NH_4OAc/NH_3 (5mM, pH 8.0) / methanol (85:15)
Flow Rate: 0.8 ml/min
Detector: Photo diode array, monitoring at 276 nm
Retention time: 7.2 min
Relative peak area response of main component:
Initial analysis: 97.0%, $s = 0.15$ (7 sub samples in duplicate, June 2002)
Current re-analysis: 92.3%, $s = 0.06$ (1 sub sample in duplicate, February 2006)

ESI-MS: Instrument: Finnigan TSQ-700
Operation: Positive ion mode, direct infusion in aqueous MeOH.
Scan: Scan range m/z 50-600, spray voltage: 2.5 kV.
Major ions: 521 (100, $[MNa]^+$), 499 ($[MH]^+$) a.m.u.
Operation: Negative ion mode, direct infusion in aqueous MeOH.
Scan: Scan range m/z 50-600, spray voltage: 2.5 kV.
Major ion: 497 (100, $[M-H]^-$) a.m.u.
Ions reported are for the major ($^{35}Cl_2$) species

IR: Instrument: Biorad FT-IR FTS 3000 MX
Range: 4000-400 cm^{-1} , KBr.
Peaks: 3300 (broad), 1729, 1678, 1515, 1350, 1245, 1074, 1054, 814 cm^{-1}

1H NMR: Instrument: Bruker DMX-500
Field strength: 500 MHz Solvent: D_2O
Key spectral data: δ 4.41 (1H, d); 6.18 (1H, s); 7.59 (2H, d); 8.18 (2H, d) ppm

Expiration of certification: The property values are valid till 21st February 2007, i.e. one year from the date of re-certification, provided the material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body.

The long-term stability of the compound in solution has not been examined.

WARNING: **The stability of chloramphenicol glucuronide is reduced when it is taken into solution. Reference solutions containing this compound should be prepared immediately prior to use. If this is not feasible then solutions containing D714 should be stored out of direct light at 4 °C and monitored for evidence of decomposition.**

Recommended storage: At or below 4 °C in a closed container in a dry, dark area.

Intended Use: For *in vitro* laboratory analysis only.

WARNING: **Treat as hazardous substance.**
Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Authorised by:



Dr Laurie Besley,
General Manager,
Chemical & Biological Metrology, NMI
Dated: 17 November, 2006.

Report ID: D714.2006.02 Ampoules

Characterisation data and certified property values specified in this report supercede that in all reports issued prior to 16th June 2006.



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