

Certificate of analysis

 Batch: 114176680
 Article: 85744 Glucocamelinin (supplied as K salt)

Test	Unit	Limit	Testresult
Solubility, SOP 105001:			Conform
Methanol		soluble	Conform
Water		soluble	Conform
Identification (HPLC-HR/MS), SOP 204125		Conform	Conform
Identification (UV spectrum from HPLC-DAD analysis) according to specification, SOP 204311		Conform	Conform
Identification (IR-spectroscopy, Ph.Eur. 10.3, 2.2.24)/USP 43 NF 37 <197>), SOP 206000		Conform	Conform
Identification (1H-NMR-spectroscopy), (outsourced), SOP 206010		Conform	Conform
Identification (13C-NMR-spectroscopy), (outsourced), SOP 206020		Conform	Conform
Water content, (micro determination, coulometric titration), Ph.Eur. 10.0., 2.5.32, SOP 304291:			
Mean value	%		3.5
Peakpurity, (HPLC), SOP 401367		Conform	Conform
Glucosinolates (HPLC), multi-method, SOP 441823:			
Glucocamelinin	%	> = 90.00	97.44
Glucoarabin			

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Test	Unit	Limit	Testresult
Residual solvents, (headspace-GC), SOP 805765:			
Residual solvents	%		0.45
Inorganic impurities, (ICP-MS), for reference substances, SOP 811701:			
Sodium	%	< 0.1	
Potassium	%	7.3	
Magnesium	%	< 0.1	
Calcium	%	< 0.1	
Aluminium	%	< 0.1	
Phosphorus	%	< 0.1	
Sulfur	%	18.5	
Content* (regarding the counterion as an inorganic impurity. For detailed information please see the attached data sheet!), SOP 890002	%		86

Assessment:

The above mentioned reference substance meets the specification.

*The absolute content is calculated considering the chromatographic purity, and if available, the content of water, residual solvents and inorganic impurities according to the following formula:
Content = (100% - water content (%) - residual solvents (%) - inorganic impurities (%)) x chromatographic purity (%) / 100.

The chromatographic purity is checked regularly: the last analysis has been performed in August 2020.

The reference substance cannot be documented with an expiry date. The pack is closed and is recommended to be stored as indicated. The unopened product is guaranteed to fulfill the specifications of this analytical report for a period of 60 months. Once opened we can no longer guarantee the stability of the material.

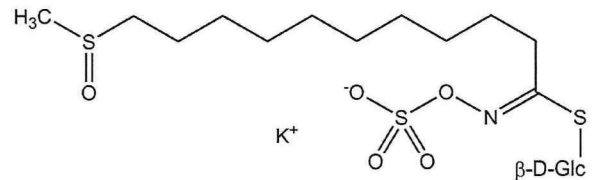
Vestenbergsreuth, 16.12.20


Dr. Michael Schwarz
Head of Reference Substances

Product Data Sheet

Glucocamelinin (supplied as K salt)

Product #: 85744



Physicochemical Data

CAS #: 67884-10-0
Molecular formula: C₁₈H₃₄NO₁₀S₃K
Molecular weight [g/mol]: 559.76
Synonyms: 10-Methylsulfinyldecylglucosinolate potassium salt

Substance class: Glucosinolates

Solubility: Soluble in methanol and water. Please note that this solubility information is based on in-house experience or taken from published data. It is not meant to guarantee solubility up to a specific concentration, nor does it guarantee stability of the reference substance in solution.

Additional Information

Please note: Please note that the given CAS # refers to the free acid as there is no CAS # for the potassium salt available. The counterion is determined quantitatively and treated as an impurity in the calculation of the absolute purity. The assigned absolute purity on the certificate of analysis therefore refers to the pure glucocamelinin.

Handling instructions: The substance is hygroscopic! The appearance of the substance in the vial may therefore differ from the information given on the certificate of analysis due to absorption of humidity. The exact weight is given on the label of the vial. In order to produce a stock solution of known concentration please transfer the contents of the vial quantitatively into a volumetric flask by repeated rinsing with excess solvent and then fill to the mark.

Source: botanical origin

Long-term storage conditions: 2-8 °C

Manufacturer: PhytoLab GmbH & Co.KG
Dutendorfer Straße 5-7
91487 Vestenbergsgreuth
Germany

Tel.: +49 9163 88-395
Fax: +49 9163 88-456
Mail: ref-substances@phytolab.de
Shop: <http://phyproof.phytolab.de>



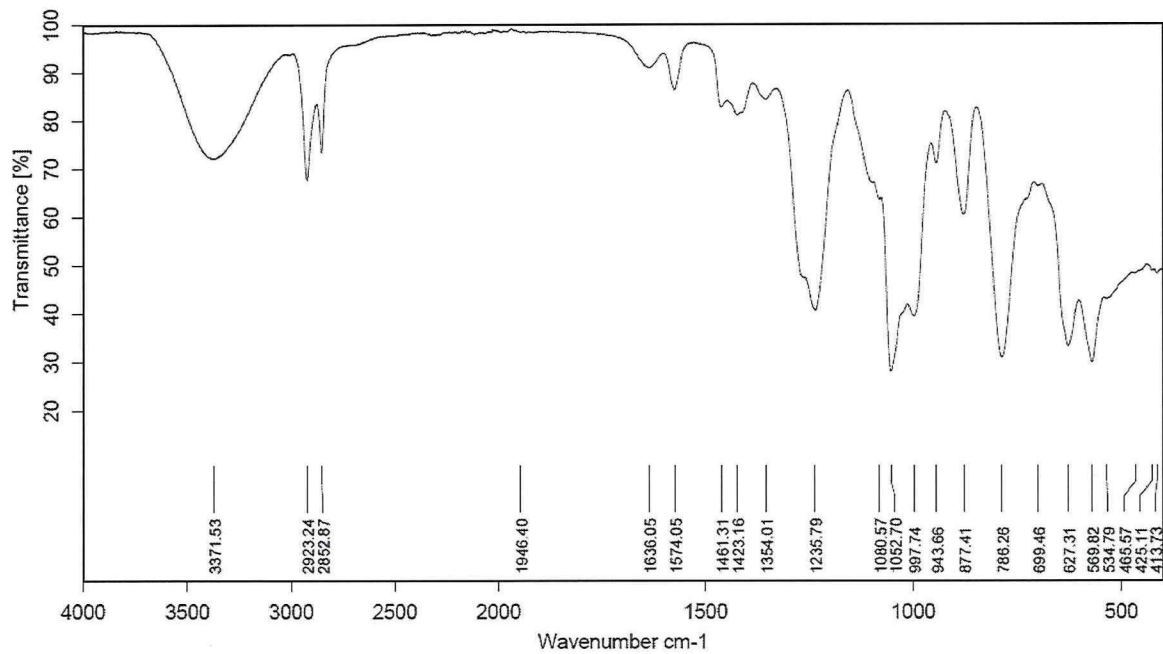
Supplements

Glucocamelinin (supplied as K salt)
Product # 85744

Batch # 16431

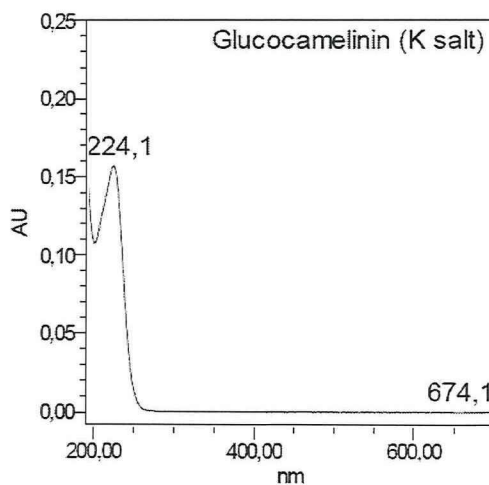
Identity tests:

IR spectrum



C:\Users\Public\Documents\Bruker\OPUS_ProtectedPool\MEAS\85744_Glucocamelinin_16431.0

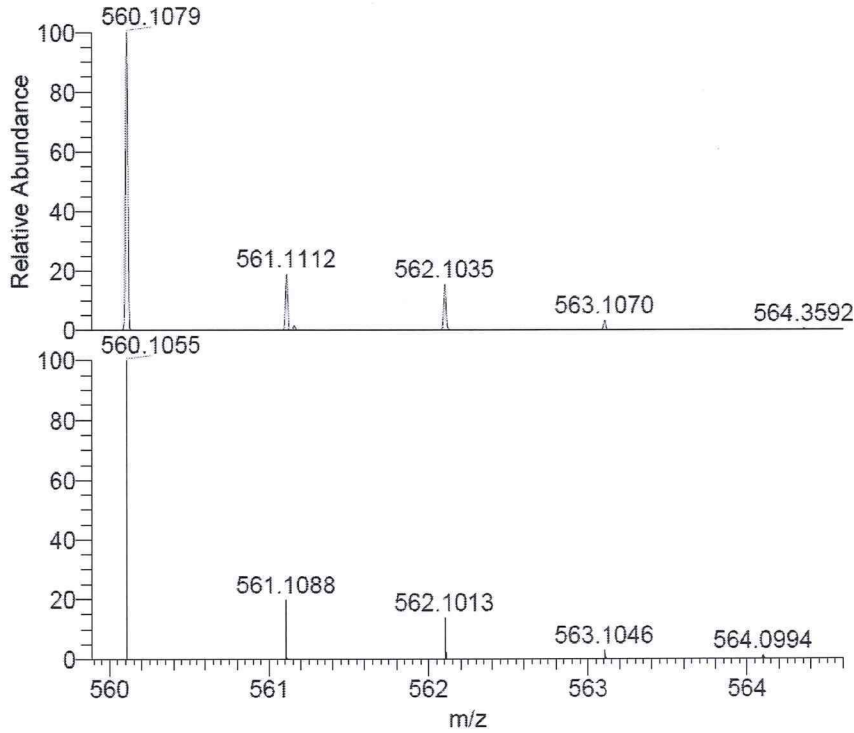
UV spectrum (derived from HPLC/PDA)





MS spectrum (ESI)

Detection: positive mode (compared with predicted spectrum)



NL:
2.03E5
200820_032#1114 RT: 12.30
AV: 1 SB: 167 7.72-8.47 ,
10.12-11.16 T: FTMS {1,1} +
p ESI Full ms
[100.00-1500.00]

NL:
6.37E5
C₁₈ H₃₅ NO₁₀ S₃ K:
C₁₈ H₃₅ N₁ O₁₀ S₃ K₁
pa Chrg 1

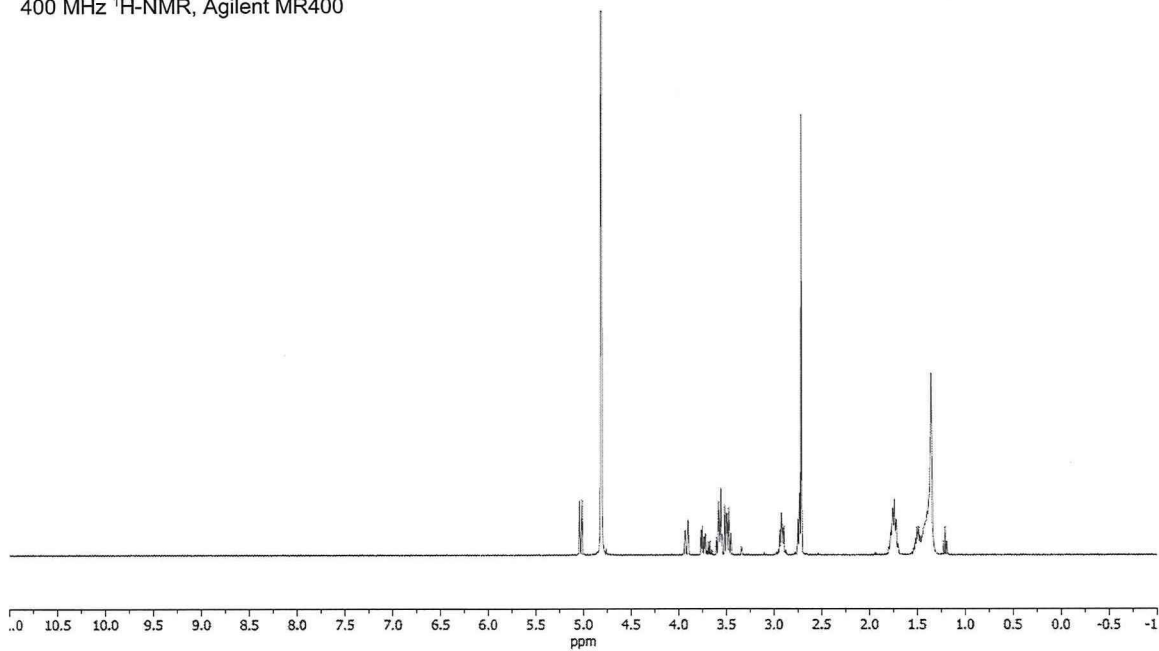
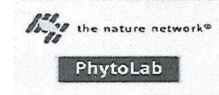


NMR spectra

¹H-NMR

PhytoLab GmbH & Co. KG
Glucocamelinin (K-Salz), Charge 16431
10.4 mg ad 0.7 ml D₂O + 3 Tr. CD₃OD

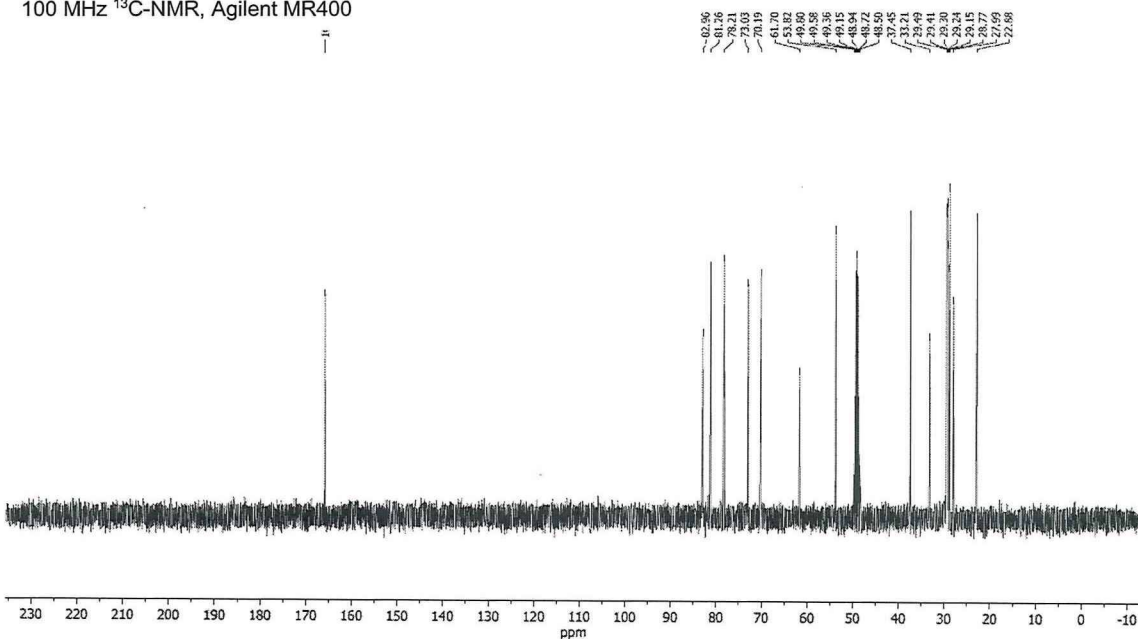
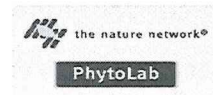
400 MHz ¹H-NMR, Agilent MR400



¹³C-NMR

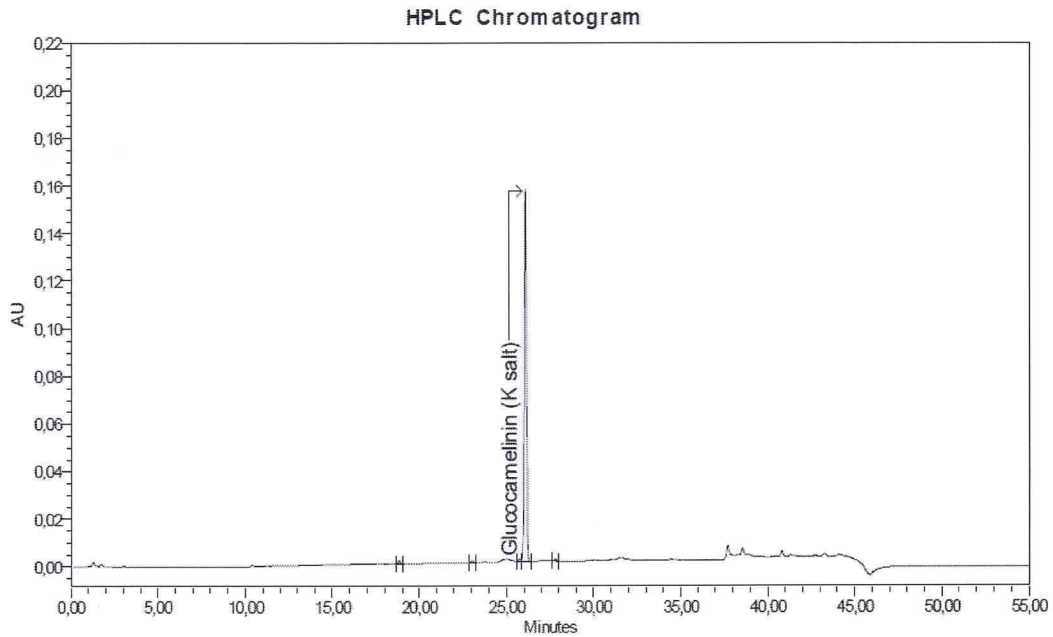
PhytoLab GmbH & Co. KG
Glucocamelinin (K-Salz), Charge 16431
10.4 mg ad 0.7 ml D₂O + 3 Tr. CD₃OD

100 MHz ¹³C-NMR, Agilent MR400





Chromatographic purity:



Peak Results

	Name	RT	Area	% Area
1		18,829	11252	0,83
2		23,035	6422	0,47
3		25,757	8863	0,66
4	Glucocamelinin (K salt)	26,072	1317528	97,43
5		27,843	8239	0,61

	Name	RT	Area	% Area
Sum				100,00

Analytical conditions

Column: Aqua C18 125A, 150 x 4.6 mm, 5 µm
 Mobile Phase: eluent A: 0.05M KH₂PO₄ pH 2.3
 eluent B: CH₃CN
 Mode: gradient

Time [min]	Eluent A [%]	Eluent B [%]
0	100	0
6	100	0
35	67	33
36	30	70
42	30	70
43	100	0
55	100	0

Flow: 1.1 ml/min
 Injection Volume: 20 µl
 Column Temperature: 25 °C
 Sample concentration: approx. 10.6 mg/100 ml
 Sample preparation: dissolved in H₂O
 Detection: UV, 225 nm
 Special note: -

Please note: Values on the certificate of analysis may vary as these are average values of at least six injections while above chromatogram and report is only one example. Non-integrated peaks originate from the blank injection.