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Reference Substances
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Certificate of analysis

Article:	84703 Usaramine N-oxide
Certificate # / Lot Number:	98867
Material batch:	20086
Sample-ID:	90887
End of analysis:	11/2023
Expiry date:	05/2027

Test	Unit	Specified value	Testresult
Appearance, SOP 100005		powder	conform
Color, SOP 100006		faint grey	conform
Identification (UV spectrum from HPLC-DAD analysis) according to specification, SOP 204311		conform	conform
Identification (1H-NMR-spectroscopy), (outsourced), SOP 206010		conform	conform
Identification (13C-NMR-spectroscopy), (outsourced), SOP 206020		conform	conform
Identification (HPLC-HR/MS), SOP 204125		conform	conform
Water content, (micro determination, coulometric titration), Ph.Eur. 10.0., 2.5.32, SOP 304291 Vers. 2018-01: Mean value	%		9.5
Pyrrolizidine alkaloid N-oxides (HPLC), multi-method, SOP 442141: Usaramine N-oxide	%	≥ 90.00	97.52
Peakpurity, (HPLC), SOP 401367		conform	conform

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Test	Unit	Specified value	Testresult
Inorganic impurities, (ICP-MS), for reference substances, SOP 811701: Calcium	%		<0.1
Potassium	%		<0.1
Magnesium	%		<0.1
Sulfur	%		<1.0
Sodium	%		<0.1
Phosphorus	%		<0.1
Aluminium	%		<0.1
Residual solvents, (headspace-GC), SOP 805765:	%		
Residual solvents (LOQ: 0.050)			0.254
Content, SOP 890000, calculated in (%): (100 - water - residual solvents - inorganic impurities) x chromatographic purity / 100	%		88

This PhytoLab phyproof© reference standard is by definition a primary reference standard and does not need to be qualified against any other reference standard. The identity of the reference standard has been substantiated by at least two independent analytical methods such as IR, NMR, UV or MS analysis. A mass balance approach, which takes chromatographic purity into account, as well as the contents of water, residual solvents, inorganic impurities, and the counter ion (if the reference standard is present as a salt) is applied in the calculation of the absolute purity as given in this COA (see description of SOP 8900XX).

The absolute purity value (and not just the chromatographic purity result obtained by means of HPLC or GC) must be used in all quantitative calculations as the chromatographic techniques do not yet account for water, residual solvents and inorganic impurities.

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Vestenbergsgreuth, 13/Nov/2023

Nicole Fuchs

QC Reference Substances

This is a computer print and valid without signature. A signed certificate of analysis can be taken on request.

PhytoLab
SAFEGUARDING BOTANICAL QUALITY.

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Further information:

Shelf life/stability: The stated expiry date applies when the reference substance is stored in the original unopened container within the specified temperature range. PhytoLab does not guarantee the stability of the reference substance once the vial has been opened.

Long-term storage and handling: The reference standard should be stored in the original unopened vial, protected against light and humidity in an airtight container, within the temperature range given on the label and accompanying data sheet. If stored below room temperature, the vial should be warmed up to room temperature in a desiccator before it is opened in order to avoid condensation of humidity. The user assumes responsibility for deciding how previously opened reference standard vials should be used and the user must ensure that the contents of opened vials are still suitable for their intended use.

Exact weight: the exact weight of each vial is given on the label of the inner vial to two decimal places. This information may be used to produce stock solutions of a known concentration without having to weigh in the reference substance again. If used for this purpose, the content of the vial must be quantitatively transferred to a volumetric flask and filled up to the required level. Please note that PhytoLab is unable to guarantee the stability of the reference standard in solution.

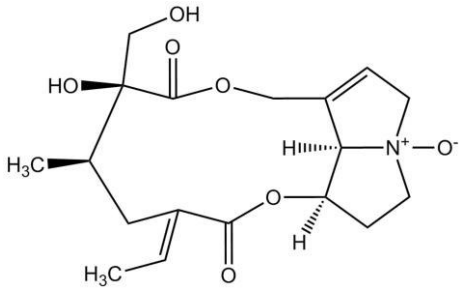
Intended use: this reference standard is solely intended for laboratory analytical purposes, research & development, and scientific teaching and training purposes. It may not be used for any other purpose and particularly not for use in, or the production of, food, animal feed, human or veterinary drugs, cosmetics, medicinal products or diagnostic agents, including in-vitro diagnostic agents. PhytoLab is unable to guarantee the suitability of this reference standard for any particular application other than its qualitative and quantitative use in chromatography and identification testing.

Further information about this reference standard can be found on the accompanying data sheet or in our webshop. Spectral and chromatographic data, and a description of the applied chromatographic method, are provided in the attachments to this COA. A detailed explanation of all data given on the COA can be found in the guide that is available from the download area in our webshop, where you can also download all of the safety data sheets.

Product Data Sheet

Usaramine N-oxide

Product #: 84703



Physicochemical Data

CAS #:	117020-54-9
Molecular formula:	C18H25NO7
Molecular weight [g/mol]:	367.40
Substance class:	Nitrogen-containing Compounds
Subgroup 1:	Alkaloids
Subgroup 2:	Pyrrolizidine alkaloids
Solubility:	soluble in chloroform, hot 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

Source:	botanical origin
Long-term storage conditions:	< -15 °C

Manufacturer:

Phytolab GmbH & Co.KG
Dutendorfer Straße 5-7
91487 Vestenbergsgreuth
Germany

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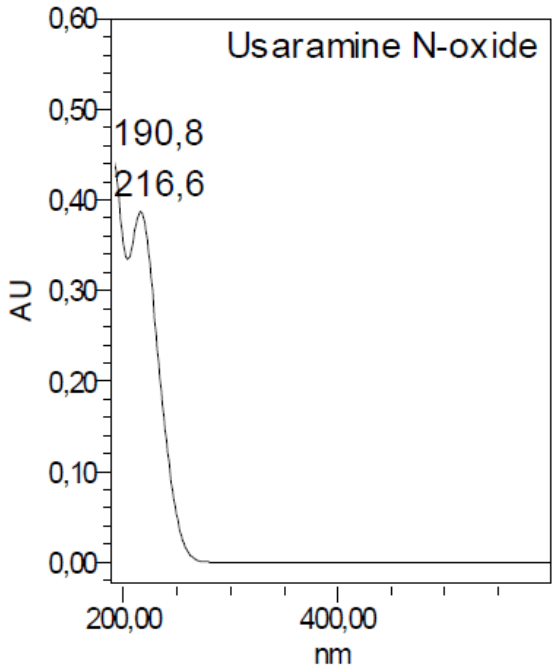
Mail: ref-substances@phytolab.de
Shop: https://phyproof.phytolab.com

Supplements

Usaramine N-oxide **Batch # 20086**
Product # 84703

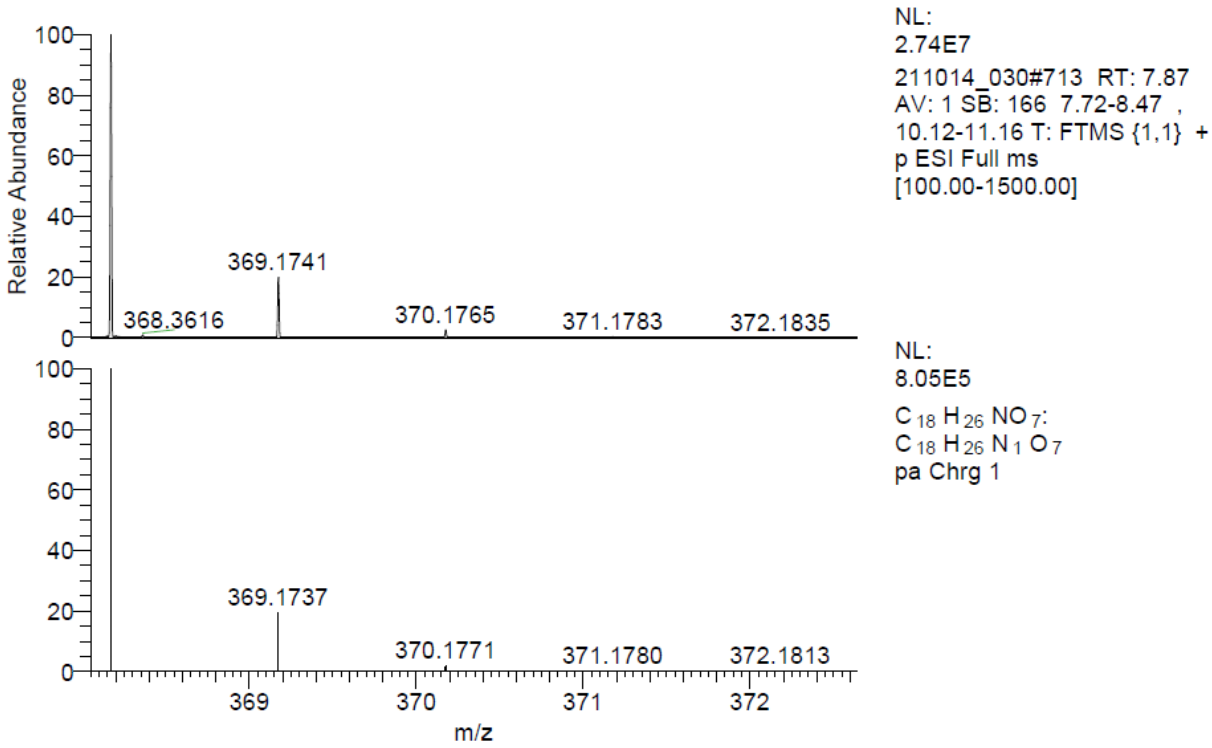
Identity tests:

UV spectrum (derived from HPLC/PDA)



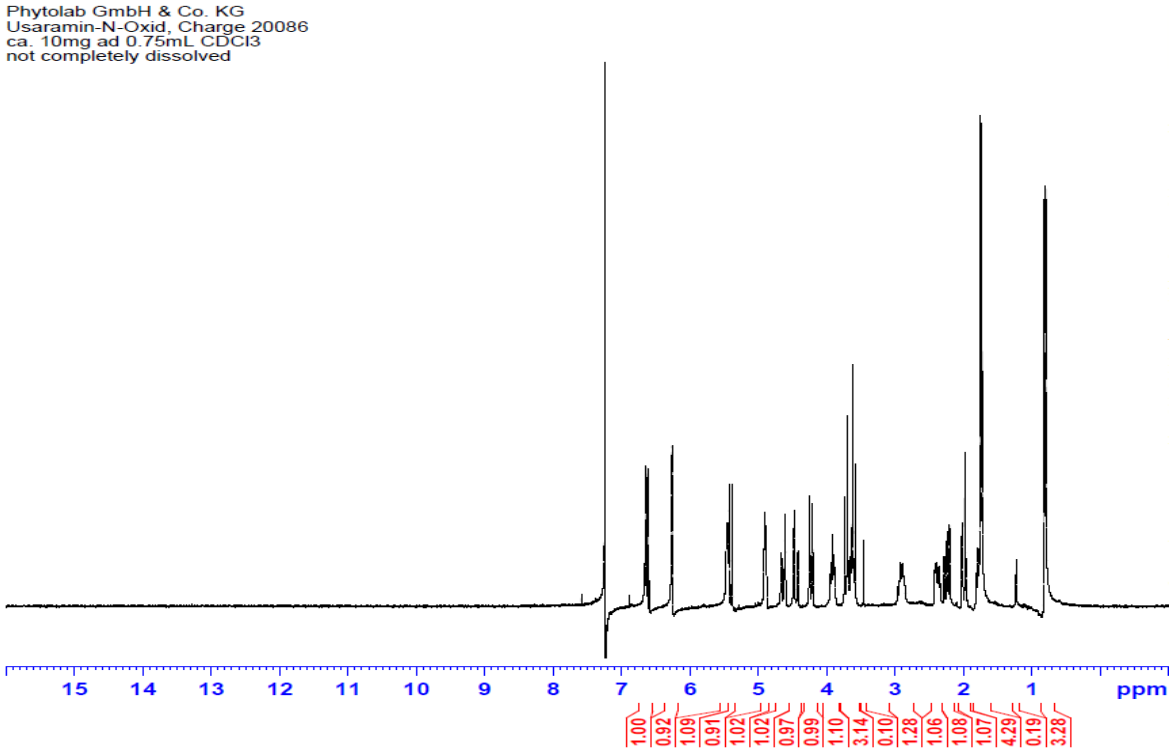
MS spectrum (ESI)

Detection: positive mode (compared with predicted spectrum)

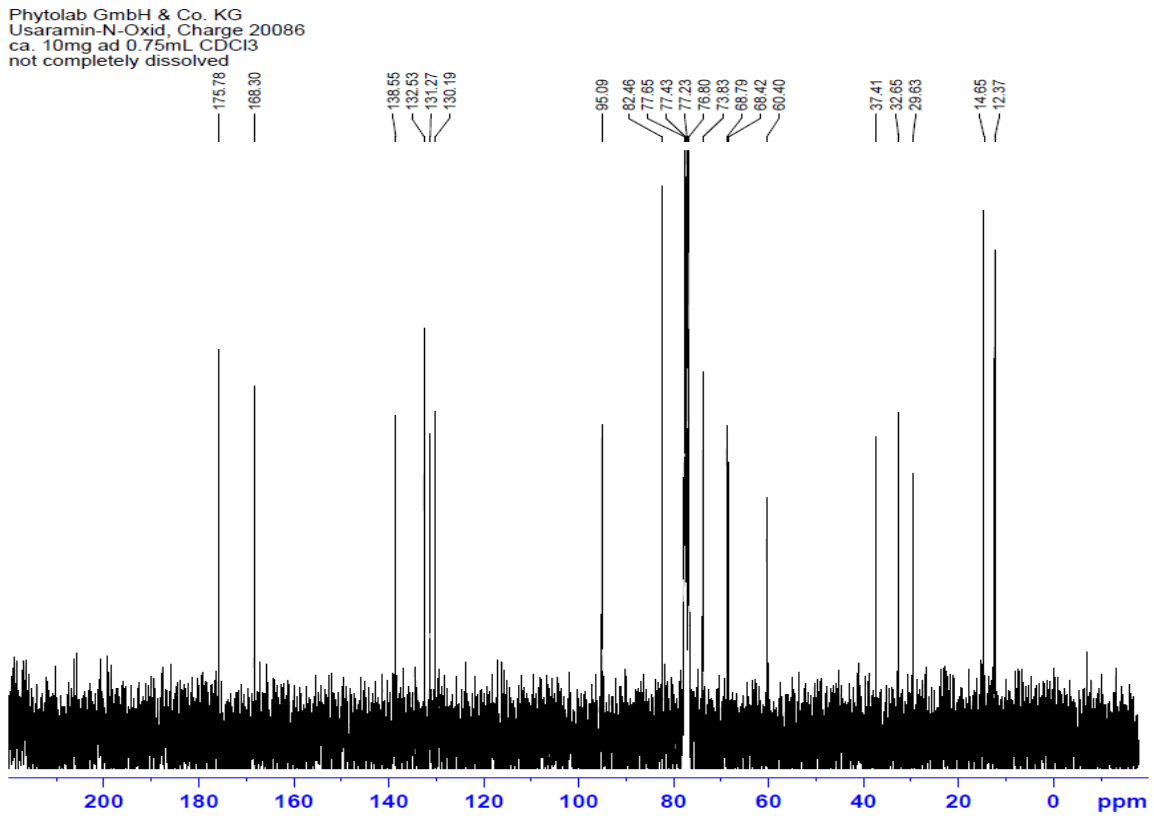


NMR spectra

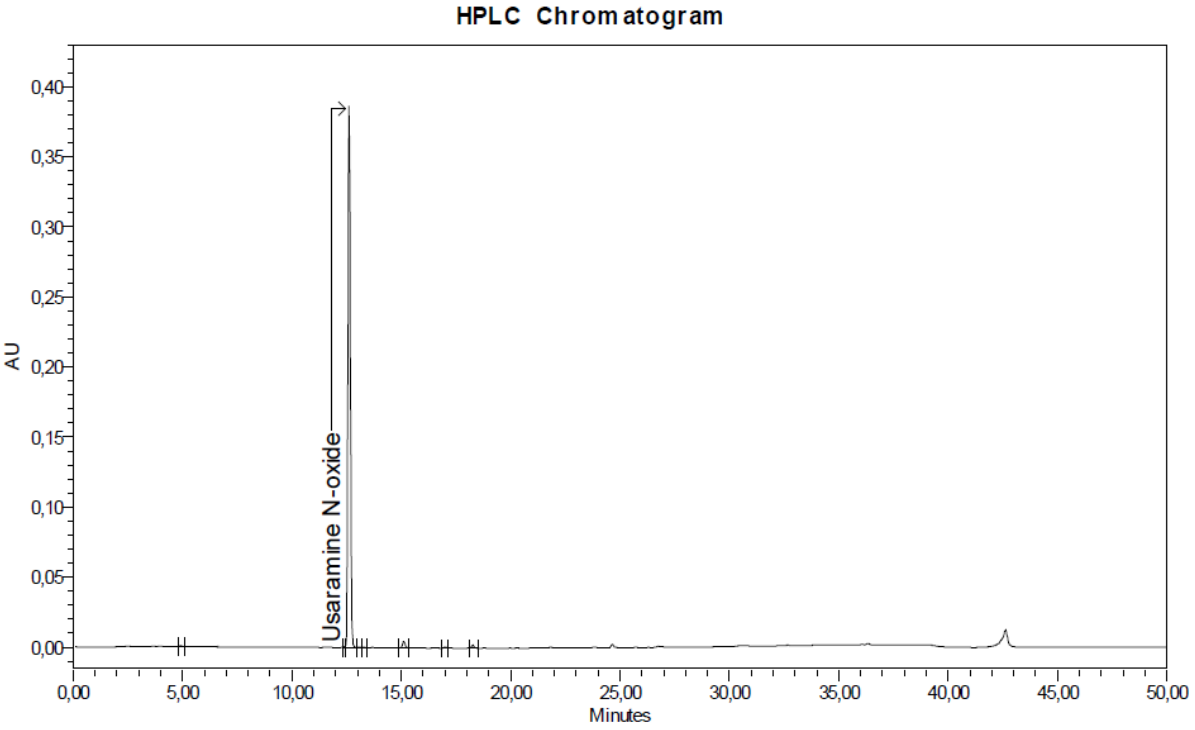
^{1}H-NMR



^{13}C-NMR



Chromatographic purity:



Peak Results

	Name	RT	Area	% Area
1		4,908	5981	0,19
2		12,433	3907	0,12
3	Usaramine N-oxide	12,613	3079796	97,32
4		13,088	5593	0,18
5		13,236	3666	0,12
6		15,114	40015	1,26
7		17,008	5696	0,18
8		18,269	19832	0,63
Sum				100,00

Analytical conditions

Column: Gemini C18 110A, 250 x 4.6 mm, 5 µm
Mobile Phase: eluent A: H_2O pH 2.0
eluent B: CH_3CN
Mode: gradient

Time [min]	Eluent A [%]	Eluent B [%]
0	98	2
10	80	20
30	20	80
35	20	80
37	98	2
50	98	2

Flow: 0.8 ml/min
Injection Volume: 20 µl
Column Temperature: 23 °C
Sample concentration: approx. 10.3 mg/100 ml
Sample preparation: dissolved in eluent A
Detection: UV, 215 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.