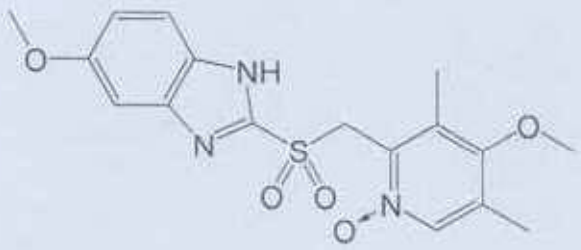
Setting standards
in analytical science

CERTIFICATE

Reference Substance

4-Methoxy-2-[[(5-methoxy-1H-benzimidazol-2-yl)sulphonyl]methyl]-3,5-dimethylpyridine 1-Oxide (Omeprazole Sulphone N-Oxide)



Molecular Formula:	$C_{17}H_{19}N_3O_5S$
Molecular Weight:	377.42
CAS Number:	[158812-85-2]

Catalogue Number:	MM0095.16
Lot Number:	15573
Long-term Storage:	2 to 8 °C, dark
Appearance:	white solid
Melting Point:	185 °C (dec.)
Assay 'as is':	99.2 %

Date of shipment: **2012-August-08**

This certificate is valid for two years from the date of shipment provided the substance is stored under the recommended conditions.

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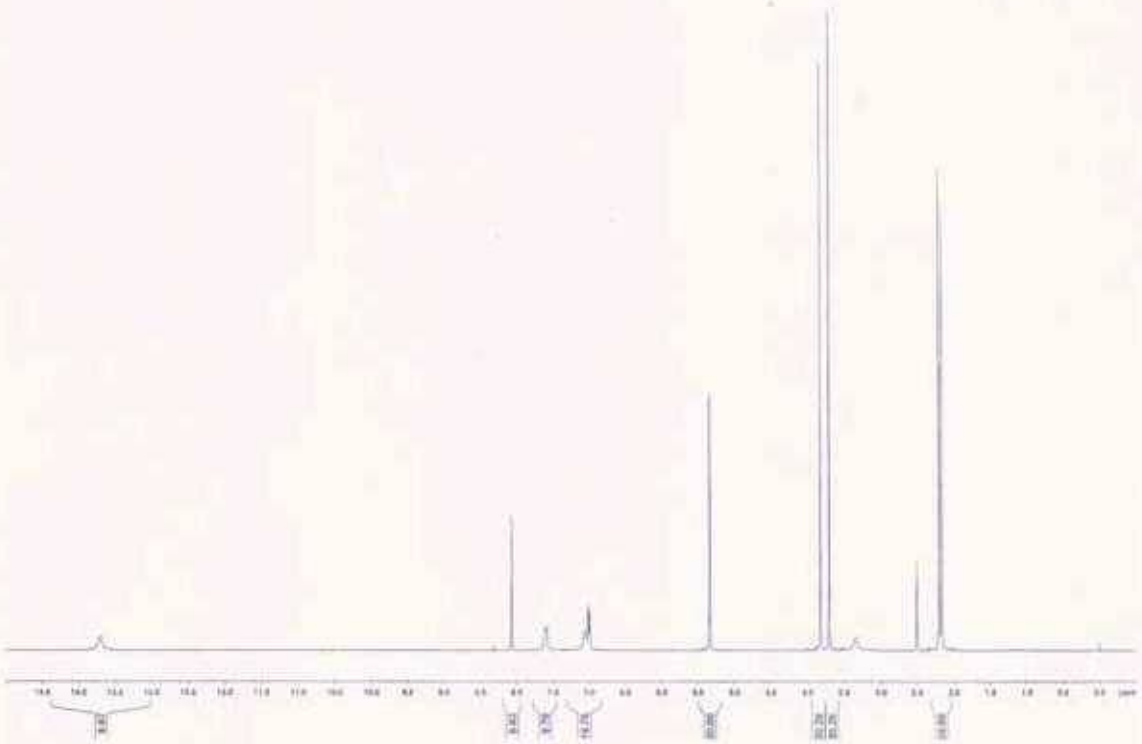
I. Identity

The identity of the reference substance was established by following analyses.

Ia. ^{1}H-NMR Spectrum

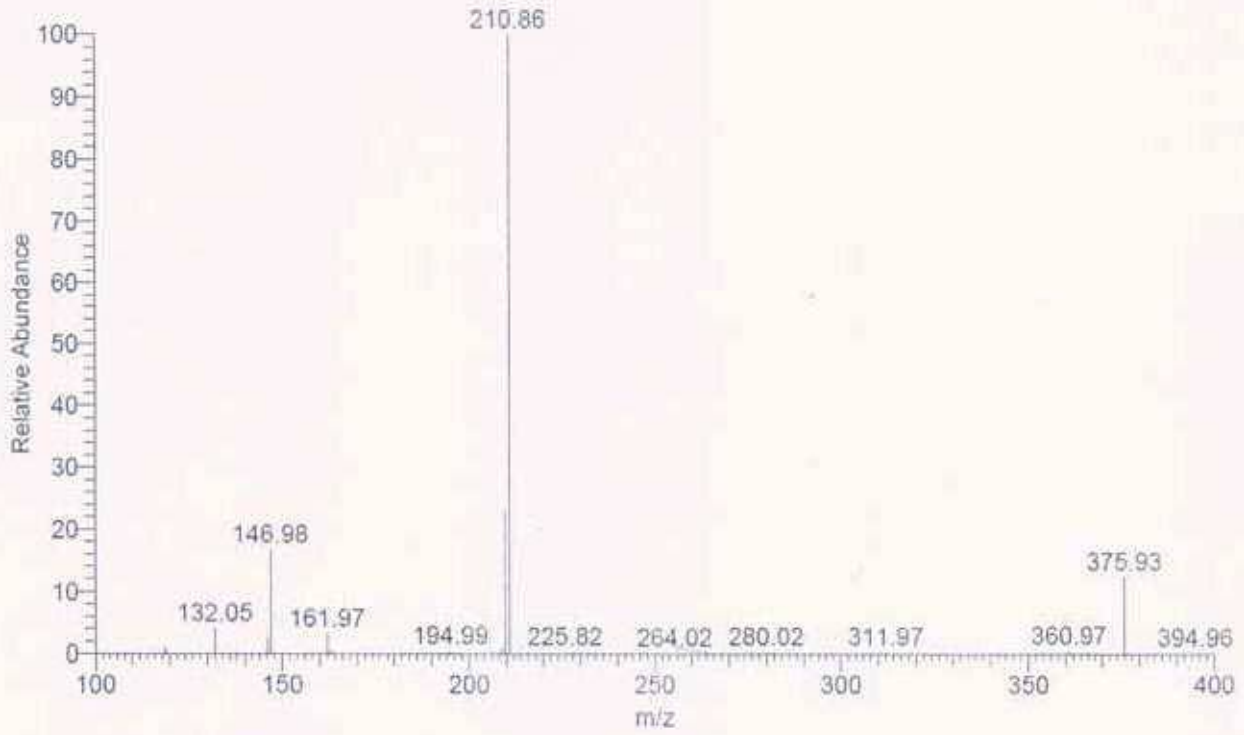
Conditions: 400 MHz, DMSO-d_6

The structure is confirmed with the signals of the spectrum and their interpretation.



Ib. Mass Spectrum

Method: 4.5 kV ESI; vaporization temperature: 200 °C, direct inlet



m/z	fragments
376	[M – H]
211	[$C_8H_7N_2O_3S$]
147	[$C_8H_7N_2O$]

The signals of the mass spectrum and their interpretation are consistent with the structural formula.

Ic. IR Spectrum

Method: Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy

Result of Peak Picking

No.	Position	Intensity
1	2925.48	64.8525
2	1624.73	82.3126
3	1458.89	76.1453
4	1400.07	77.3327
5	1326.79	61.3715
6	1204.33	70.5993
7	1132.01	53.7549
8	1028.04	70.1768
9	996.053	66.711
10	969.055	64.5746
11	828.277	60.3428
12	735.71	71.0276
13	682.677	74.931

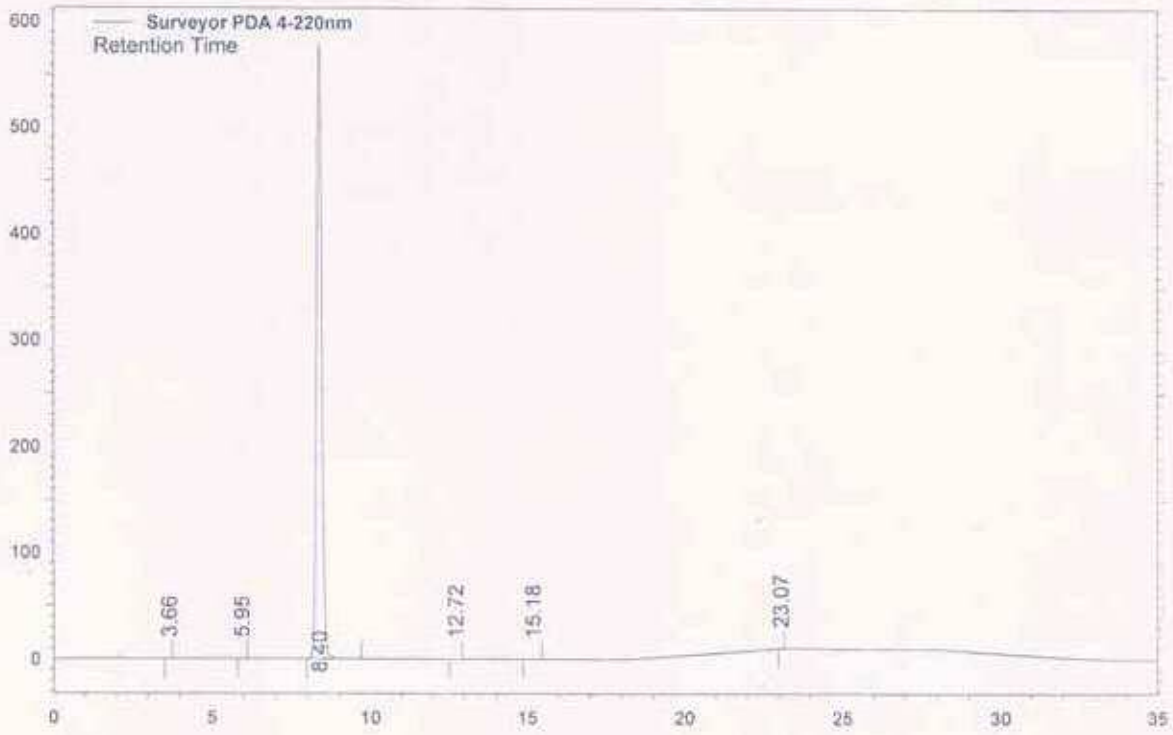
The signals of the IR spectrum and their interpretation are consistent with the structural formula.

II. Purity

The purity of the reference substance was analysed by high performance liquid chromatography (HPLC).

HPLC Conditions:

Column:	Conditions:	Detector:	Injector:
Hypersil Gold (C18)	1.0 ml/min, 40 °C	DAD	Auto
5 µm, 150 x 4.6 mm	0 – 15 min Water/Acetonitrile 80/20 15 – 20 min Water/Acetonitrile to 60/40 20 – 25 min Water/Acetonitrile 60/40 25 – 30 min Water/Acetonitrile to 80/20 30 – 35 min Water/Acetonitrile 80/20 (v/v); 0.1 % H_3PO_4	220 nm	5 µl; 0.0654 mg/ml in Water/Acetonitrile 50/50 (v/v)



Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	3.66	1422	0.02
2	5.95	2481	0.04
3	8.40	6745401	99.85
4	12.72	1480	0.02
5	15.18	3383	0.05
6	23.07	1693	0.03
Totals		6755860	100.00

For the calculation the system peaks were ignored. The content of the analyte was determined as ratio of the peak area of the analyte and the cumulative areas of the purities, added up to 100 %.

Results:

Average	99.85 %
Number of results	n=3
Standard deviation	0.01 %

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III. Water Content

Method: Karl Fischer titration

Results:

Average	0.20 %
Number of results	n=3
Standard deviation	0.01 %

IV. Residual Solvents

Method: ^{1}H-NMR

Result: 0.46 % Chloroform
0.04 % *n*-Hexane

V. Final Result

Total impurities (HPLC)	0.15 %
Water content	0.20 %
Residual solvents	0.50 %
Assay (100 % method)[1]	99.15 %

The assay is assessed to be 99.2 % 'as is'

The assay 'as is' is equivalent to the assay based on the not anhydrous and not dried substance respectively.

Release Date: 2012-05-24

LGC GmbH

Dr. Sabine Schroder
Product Release

[1] The calculation of the 100 % method follows the formula:

$$\text{Assay (\%)} = (100\ \% - \text{KF} - \text{RES}) \cdot \frac{\text{Purity HPLC (\%)}}{100\ \%}$$

Water (KF) and Residual solvents (RES) are considered as absolute contributions, HPLC purity is considered as relative contribution.