

STUDY REPORT ASL88432

- C₂₁H₃₉Br -

- Content -

Sponsor: ASAS Analytische Standards
Andre Stratmann Service Labor GmbH
Gottfried-Hagen-Str. 60-62
D-51105 Köln
Germany

Monitor: Mr. André Stratmann

Test Facility: Spectral Service AG
Emil-Hoffmann-Straße 33
D-50996 Köln
Germany

Date: 20 May 2020

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1 OBJECTIVES

The purpose of analysis is to determine the content of test item using a NMR spectroscopic method.

2 TEST ITEM

Tab. 1 Test item data

Sample name	Lot no.	Spectral Service code	Arrival
C ₂₁ H ₃₉ Br	#202005 16	ASL88432-1	18.05.2020

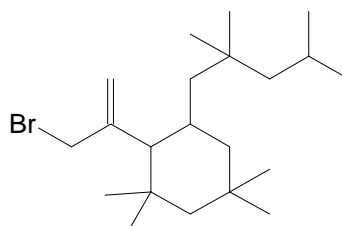


Fig. 1 Chemical structure of the test item

3 MATERIALS

3.1 Reference and Calibration Items

Tab. 2 Chemicals actually used as reference and calibration items

No	Substance name	Distributor	Order no.
85	Dimethyl sulfone (DMS)	Sigma-Aldrich, Steinheim (D)	41867

3.2 Chemicals and Materials

Tab. 3 Chemicals and materials used in the study

Substance name	Distributor	Order no.
Chloroform-d ₁ (CDCl ₃), Degree of deuteration 99.8% with 0.03% v/v TMS	Euriso-top, Saarbrücken (D)	D307K

3.3 Instruments

NMR spectrometer Avance III HD 500 (Bruker, Karlsruhe, D), magnetic flux density 11.7 Tesla
 BBFO^{PLUS} SmartProbe (Z119470 0070); automated sample changer Bruker B-ACS 120
 Computer Intel Xeon Dual Core 2.5 GHz under MS Windows 7, Bruker TopSpin 3.5
 Standard operation procedure SAA-GMR045-04

Micro balance Mettler-Toledo XPE 26 (Greifensee, CH)
 Balance printer Mettler Toledo LC-P45 (Greifensee, CH)
 Standard operation procedure SAA-GMR047-03

4 METHODS

4.1 NMR Spectroscopy

For quantification according to standard operation procedure SAA-MET001-04 appropriate amounts of test item and of internal standard have been exactly weighed (s. Table "Calculation of content", Chapter 5.1 - NMR Spectroscopy), dissolved in 0.7 ml CDCl₃ and measured. Integrated signals of test item and internal standard have been used for calculation.

The actually used NMR parameters appear on the spectrum plots.

The ratio of integrals per atom corresponds to the molar ratio of the compared substances. For calculation software Microsoft Excel was used.

Calculation:

$$\text{Equation 1} \quad \text{MOL}_{\text{IS}} [\text{mMol}] = \frac{\text{IW}_{\text{IS}} [\text{mg}] * \text{C}_{\text{IS}} [\%]}{\text{MW}_{\text{IS}} [\text{g/mol}] * 100}$$

$$\text{Equation 2} \quad \text{MOL}_{\text{TI}} [\text{mMol}] = \frac{\text{I}_{\text{TI}} * \text{NA}_{\text{IS}} * \text{MOL}_{\text{IS}} [\text{mMol}]}{\text{I}_{\text{IS}} * \text{NA}_{\text{TI}}}$$

$$\text{Equation 3} \quad \text{C}_{\text{TI}} [\% \text{-by weight}] = \frac{\text{MW}_{\text{TI}} [\text{g/mol}] * \text{MOL}_{\text{TI}} [\text{mMol}] * 100}{\text{IW}_{\text{TI}} [\text{mg}]}$$

Tab. 4 Declaration of variables

	test item (TI)	internal standard (IS)
molecular weight [g/mol]	MW _{TI}	MW _{IS}
initial weight [mg]	IW _{TI}	IW _{IS}
content [%-by weight]	C _{TI}	C _{IS}
Mol [mMol]	MOL _{TI}	MOL _{IS}
integral	I _{TI}	I _{IS}
number of atoms *)	NA _{TI}	NA _{IS}

*) atom refers to NMR active nucleus measured (e.g. ¹H, ¹³C, ¹⁹F, ³¹P)

5 RESULTS AND DISCUSSION

5.1 NMR Spectroscopy

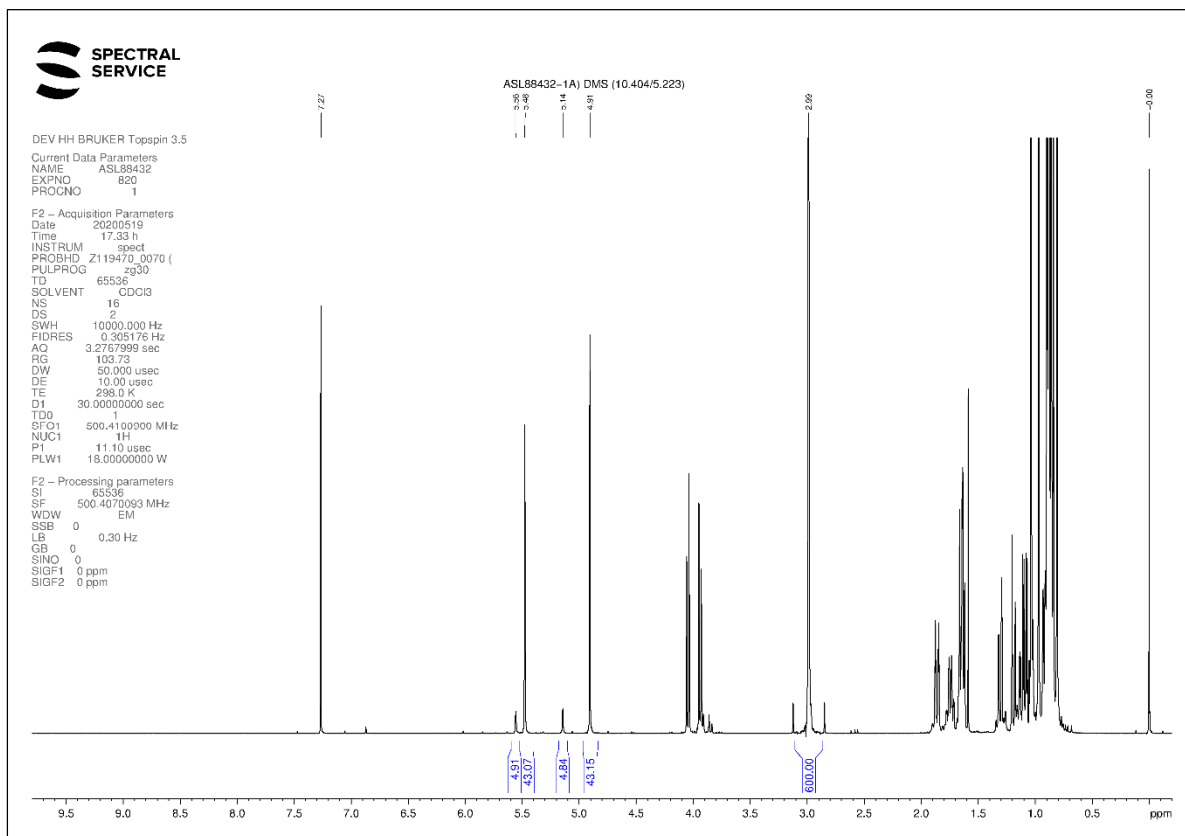
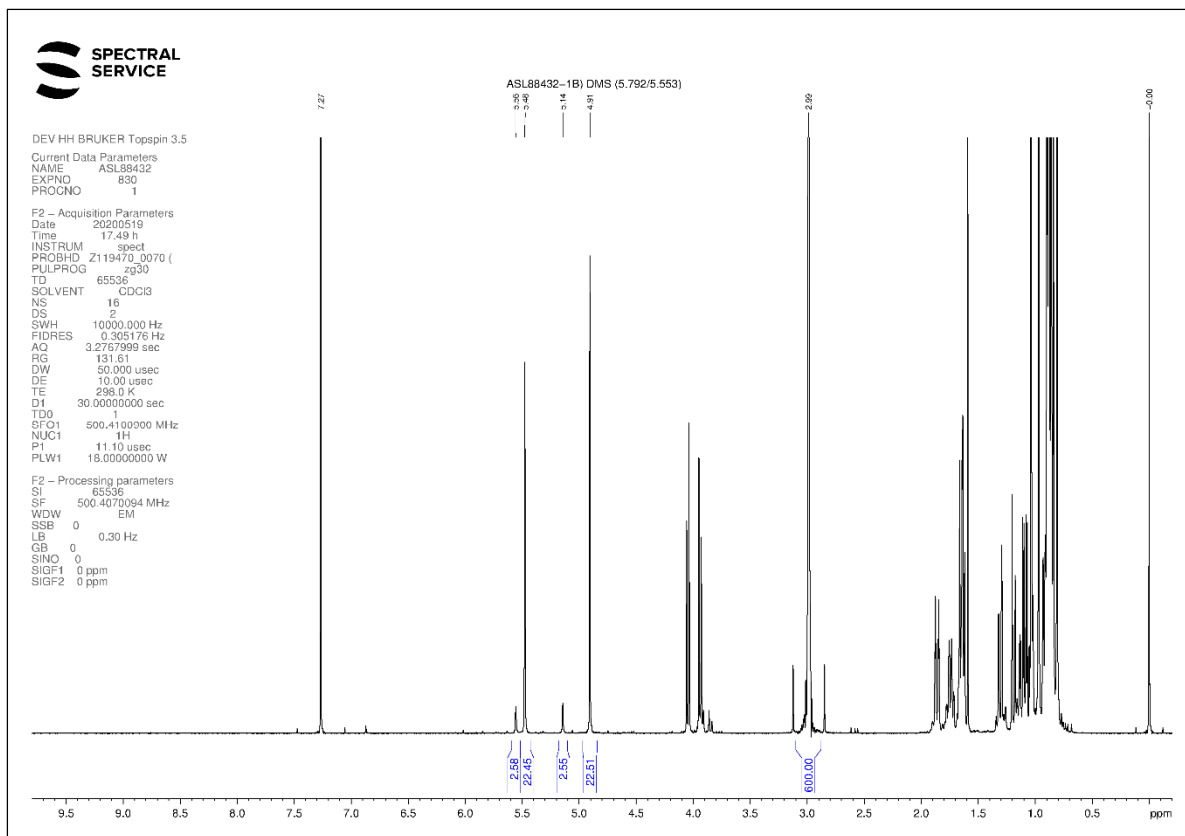
¹H-NMR signals not caused by the test item:

0.0 ppm (singlet of TMS); 7.27 ppm (singlet of CHCl₃ in CDCl₃).

The test item appears as diastereomers. For quantification, the integrals of corresponding signals at 5.14 ppm resp. 5.56 ppm were added to integrals of methylene group main signals at 4.91 ppm resp. 5.48 ppm.

Tab. 5 Calculation of content

Excel-Version 16.0				Version 04.01		valid from: 15/09/2018		
Test item	Integral	Initial weight [mg]	Integral	Initial weight [mg]	mMol	mMol	Content [mg]	Content [%]
	TI	TI	IS	IS	IS	TI	TI	TI
A	95.97	10.404	600.00	5.223	0.0554	0.0266	9.8390	94.6
B	50.09	5.792	600.00	5.553	0.0589	0.0147	5.4598	94.3
Int. Standard	Molecular weight TI [g/Mol]		370.20				Average	94.4
DMS	Molecular weight IS [g/Mol]		94.13				Std.-dev.	0.2
Rounding	Number of atoms TI		2				%RSD	0.2
2	Number of atoms IS		6				Balance	XPE26
en	Content [%] IS		99.82				Mettler-Toledo XPE26/M	
Comment: Initial weight is higher than required MinWeigh of 2 mg.								
Annotation: The integral value may be equivalent to a single value, a sum or a subtraction								

Fig. 2 ¹H-NMR spectrum of test item ASL88432 + DMS (A)Fig. 3 ¹H-NMR spectrum of test item ASL88432 + DMS (B)

6 SUMMARY

The average content of the test item was determined to 94.4 weight-% by ¹H-NMR spectroscopy.

The result refers exclusively to test item analysed by Spectral Service AG. Because no content specification was given to the Spectral Service AG for this analysis order, the assessment of the plausibility of this result is the responsibility of the sponsor.

7 PERSONNEL

Study director:	Helmut Herling,	Chemical engineer
Co-worker:	Christina Kohlhaas,	Chemical technical assistant
Quality assurance:	Karen Gedig,	Food chemist

All are staff members of the test facility.

8 CONFIRMATION OF THE STUDY REPORT

<p>Date: 20 May 2020</p> <p>Study director:</p> <p></p> <p>_____ Helmut Herling</p>	<p>Company stamp:</p> <p>Spectral Service AG Emil-Hoffmann-Str. 33 · D-50996 Köln ☎ 0 22 36 / 9 69 47-0 · Fax 0 22 36 / 9 69 47-11 www.spectralservice.de</p>
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