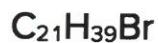


## STUDY REPORT ASL84242



### - Identity -

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

Monitor: Mr. André Stratmann

Quality standard: non GxP

Order no.: ---

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

Date: 28 October 2019

**TABLE OF CONTENTS**

1	OBJECTIVES .....	3
2	TEST ITEM.....	3
3	MATERIALS.....	3
3.1	Reference and Calibration Items (not applicable).....	3
3.2	Chemicals and Materials .....	3
3.3	Instruments.....	3
4	METHODS.....	3
4.1	NMR Spectroscopy .....	3
5	RESULTS AND DISCUSSION.....	4
5.1	NMR Spectroscopy .....	4
6	SUMMARY .....	9
7	PERSONNEL.....	9
8	CONFIRMATION OF THE STUDY REPORT .....	9

**FIGURES**

Fig. 1	Proposed chemical structure of the test item .....	3
Fig. 2	Signal assignment [ppm] $^1\text{H}$ -NMR spectrum (Fig. 4, page 5).....	4
Fig. 3	Signal assignment [ppm] $^{13}\text{C}$ -NMR spectrum (Fig. 5, page 6) .....	4
Fig. 4	$^1\text{H}$ -NMR spectrum of test item.....	5
Fig. 5	$^{13}\text{C}$ -NMR spectrum of test item .....	6
Fig. 6	2D-HSQC-Dept NMR spectrum of test item .....	7
Fig. 7	2D-HSQC Dept NMR spectrum (detail) of test item .....	7
Fig. 8	2D-HMBC NMR spectrum of test item .....	8
Fig. 9	2D-HMBC NMR spectrum (detail) of test item .....	8

**TABLES**

Tab. 1	Test item data .....	3
Tab. 2	Chemicals and materials used in the study .....	3

## 1 OBJECTIVES

The purpose of analysis is to determine identity of test item using NMR spectroscopic methods.

## 2 TEST ITEM

Tab. 1 Test item data

Sample name	Lot no.	Spectral Service code	Arrival
C <sub>21</sub> H <sub>39</sub> Br	#20191019	ASL84242-1	24.10.2019

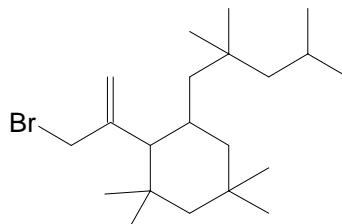


Fig. 1 Proposed chemical structure of the test item

## 3 MATERIALS

### 3.1 Reference and Calibration Items (not applicable)

### 3.2 Chemicals and Materials

Tab. 2 Chemicals and materials used in the study

Substance name	Distributor	Order no.
Chloroform-d <sub>1</sub> (CDCl <sub>3</sub> ), 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  
BBO Prodigy cryo probe (Z125869 0006); automated sample changer Bruker SampleXpress 60  
Computer Intel Xeon E5 8-Core 3.7 GHz under MS Windows 7, Bruker TopSpin 3.5  
Standard operation procedure SAA-GMR045-04

## 4 METHODS

### 4.1 NMR Spectroscopy

<sup>1</sup>H-NMR-, <sup>13</sup>C-NMR- and 2D-NMR (HSQC, HMBC) spectra were recorded to characterise the test item. Approx. 5 mg of the test item have been dissolved in 0.6 ml CDCl<sub>3</sub>. The actually used NMR parameters appear on the spectrum plot.

## 5 RESULTS AND DISCUSSION

### 5.1 NMR Spectroscopy

Number, multiplicity and chemical shift of the  $^1\text{H}$ -NMR and  $^{13}\text{C}$ -NMR signals as well as 2D-NMR cross peaks (Fig. 6ff) are in accordance with the proposed structure of the test item.

$^1\text{H}$ -NMR signals not caused by the test item:

0.0 ppm (singlet of TMS); 1.54 ppm (signal of water); 7.26 ppm (singlet of  $\text{CHCl}_3$  in  $\text{CDCl}_3$ ).

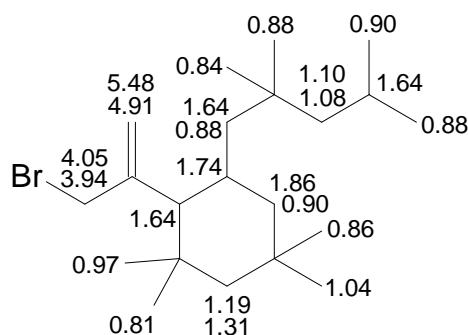


Fig. 2 Signal assignment [ppm]  $^1\text{H}$ -NMR spectrum (Fig. 4, page 5)

$^{13}\text{C}$ -NMR signals not caused by the test item:

0.0 ppm (singlet of TMS); 77.0 ppm (triplet of solvent  $\text{CDCl}_3$ ).

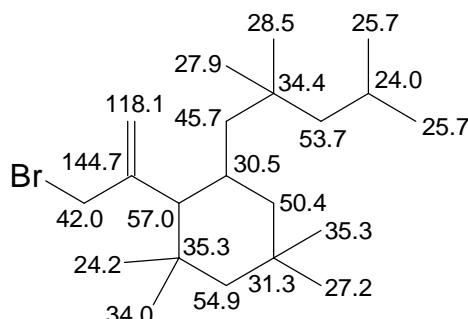
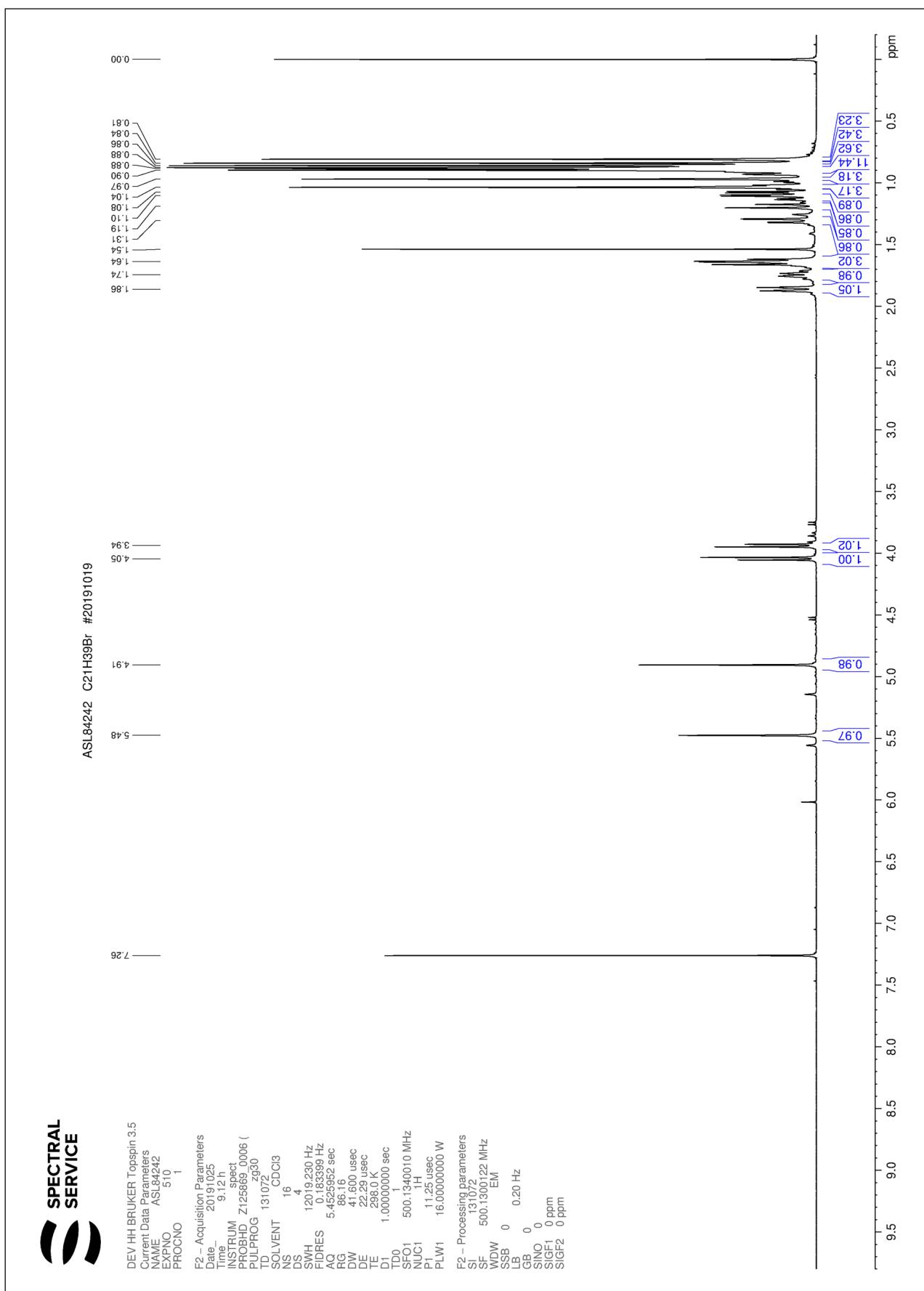


Fig. 3 Signal assignment [ppm]  $^{13}\text{C}$ -NMR spectrum (Fig. 5, page 6)

Fig. 4 <sup>1</sup>H-NMR spectrum of test item

**SPECTRAL  
SERVICE**

ASL84242 C21H39Br #20191019

DEV HH BRUKER Topspin 3.5  
Current Data Parameters  
NAME ASL84242  
EXPNO 511  
PROCNO 1

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PLW13 0.1561500 W  
wait64

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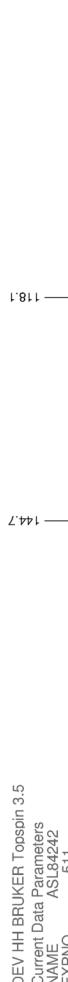


Fig. 5 13C-NMR spectrum of test item

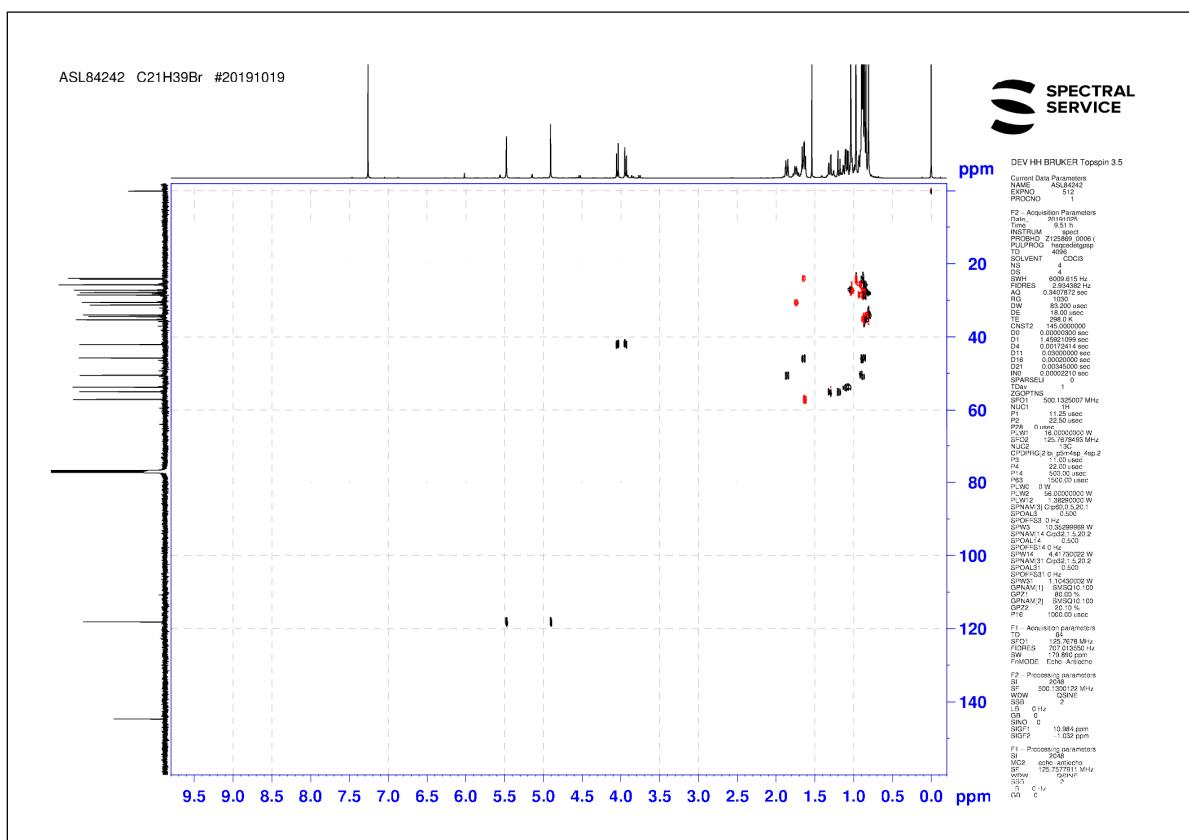


Fig. 6 2D-HSQC-Dept NMR spectrum of test item

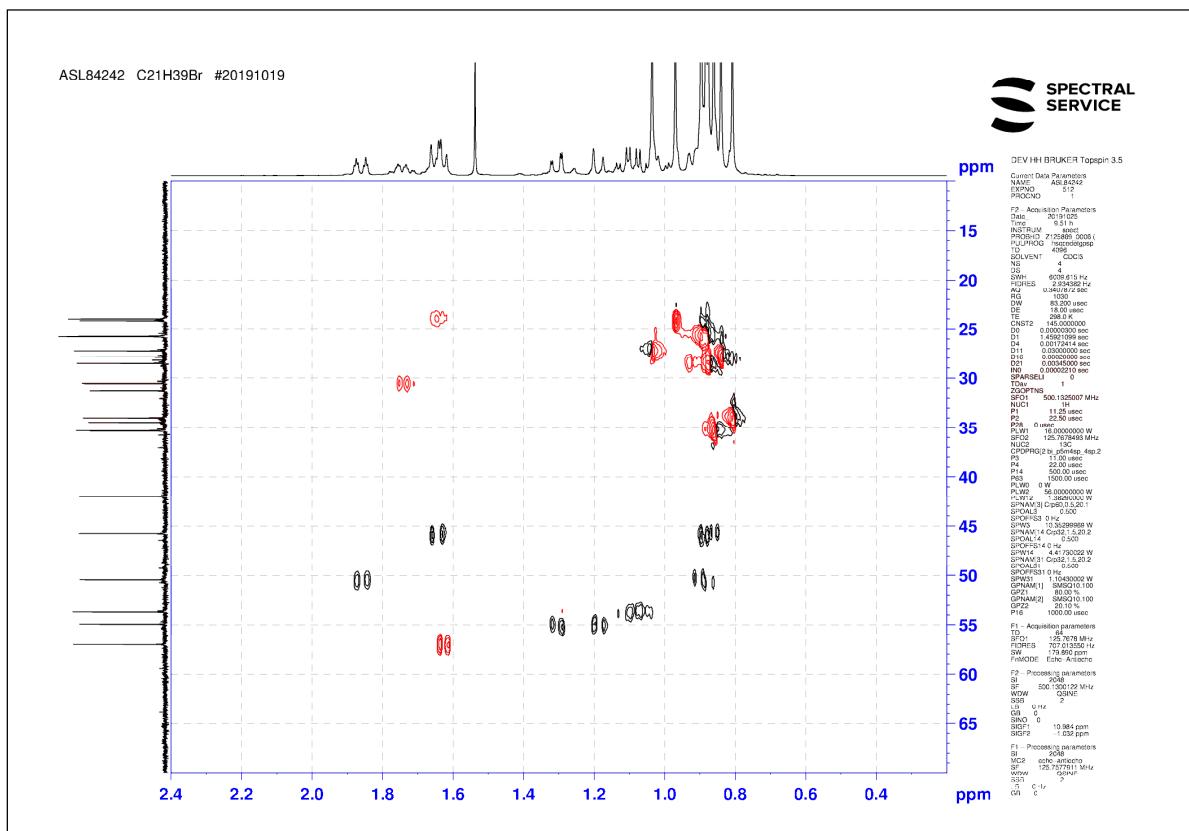


Fig. 7 2D-HSQC Dept NMR spectrum (detail) of test item

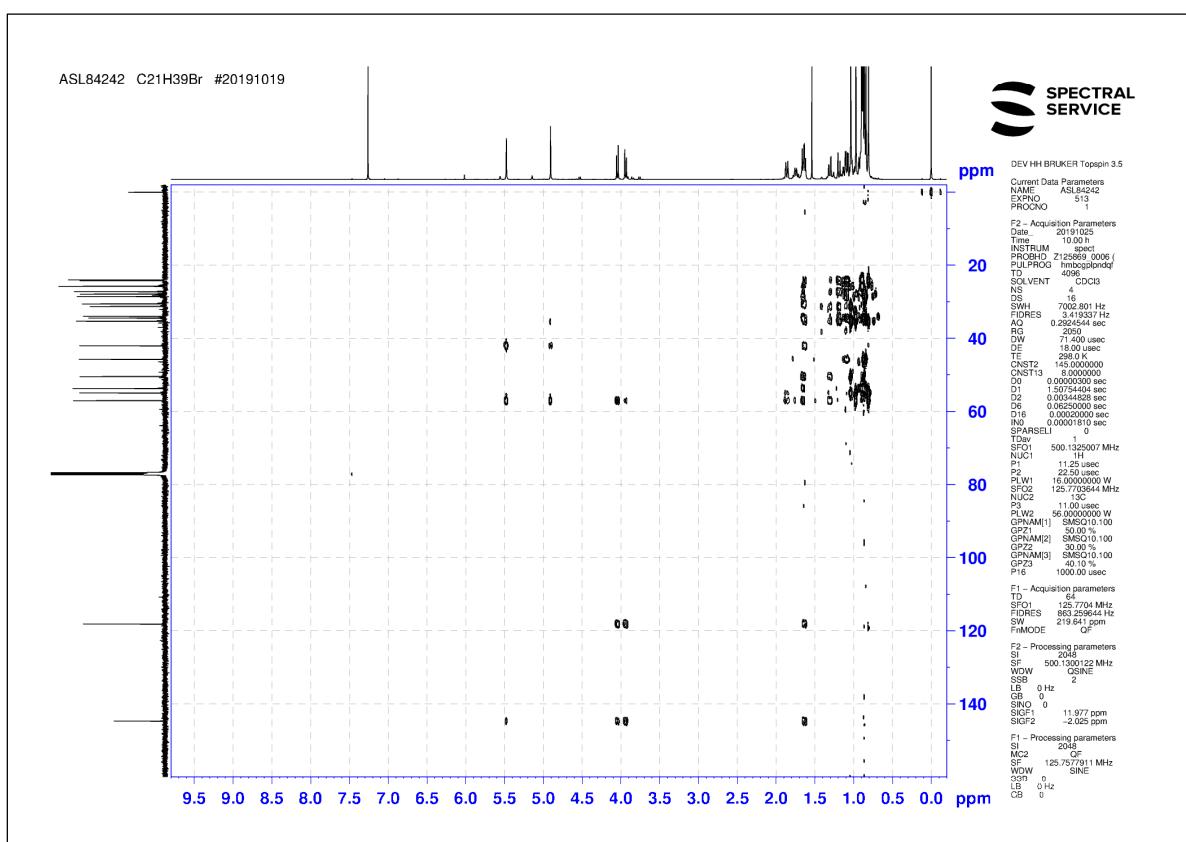


Fig. 8 2D-HMBC NMR spectrum of test item

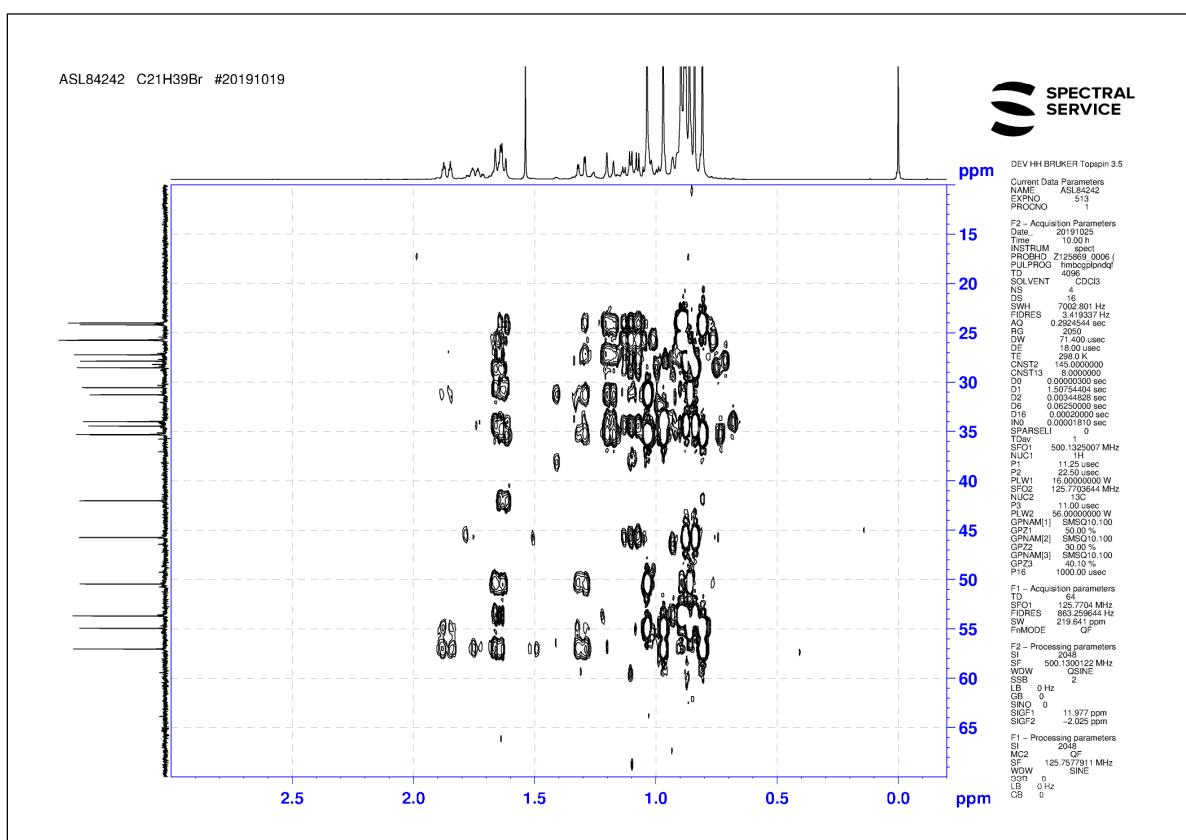


Fig. 9 2D-HMBC NMR spectrum (detail) of test item

**6 SUMMARY**

The proposed structure of the test item was confirmed by one- and two-dimensional NMR spectroscopy.

The result refers exclusively to test item analysed by Spectral Service AG and corresponds to the data given by the sponsor.

**7 PERSONNEL**

Study director:	Helmut Herling,	Chemical engineer
Co-worker:	Christina Kohlhaas,	Chemical technical assistant
Quality assurance:	Olga Kniffert,	Biological technical assistant

All are staff members of the test facility.

**8 CONFIRMATION OF THE STUDY REPORT**

Date: 28 October 2019  Study director:   _____ Helmut Herling	Company stamp:  <b>Spectral Service AG</b> Emil-Hoffmann-Str. 33 · D-50996 Köln ☎ 0 22 36 / 9 69 47-0 · Fax 0 22 36 / 9 69 47-11 <a href="http://www.spectralservice.de">www.spectralservice.de</a>
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