Supplementary Material

(*R*)-*N*-benzyl-*N*-(1-phenylethyl)cyclohexanamine

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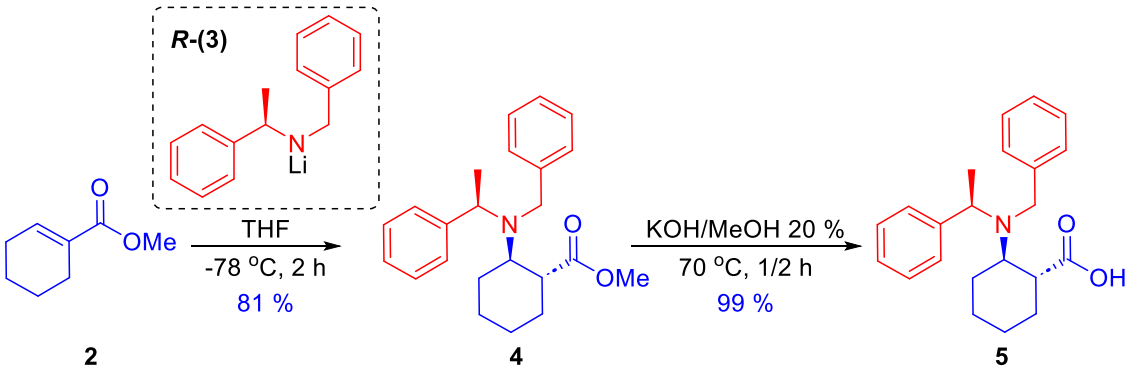
Contents

1. Synthetic procedure for the preparation of (1*R*,2*R*)-2-(benzyl((*R*)-1-phenylethyl)amino)cyclohexane-1-carboxylic acid (**5**).

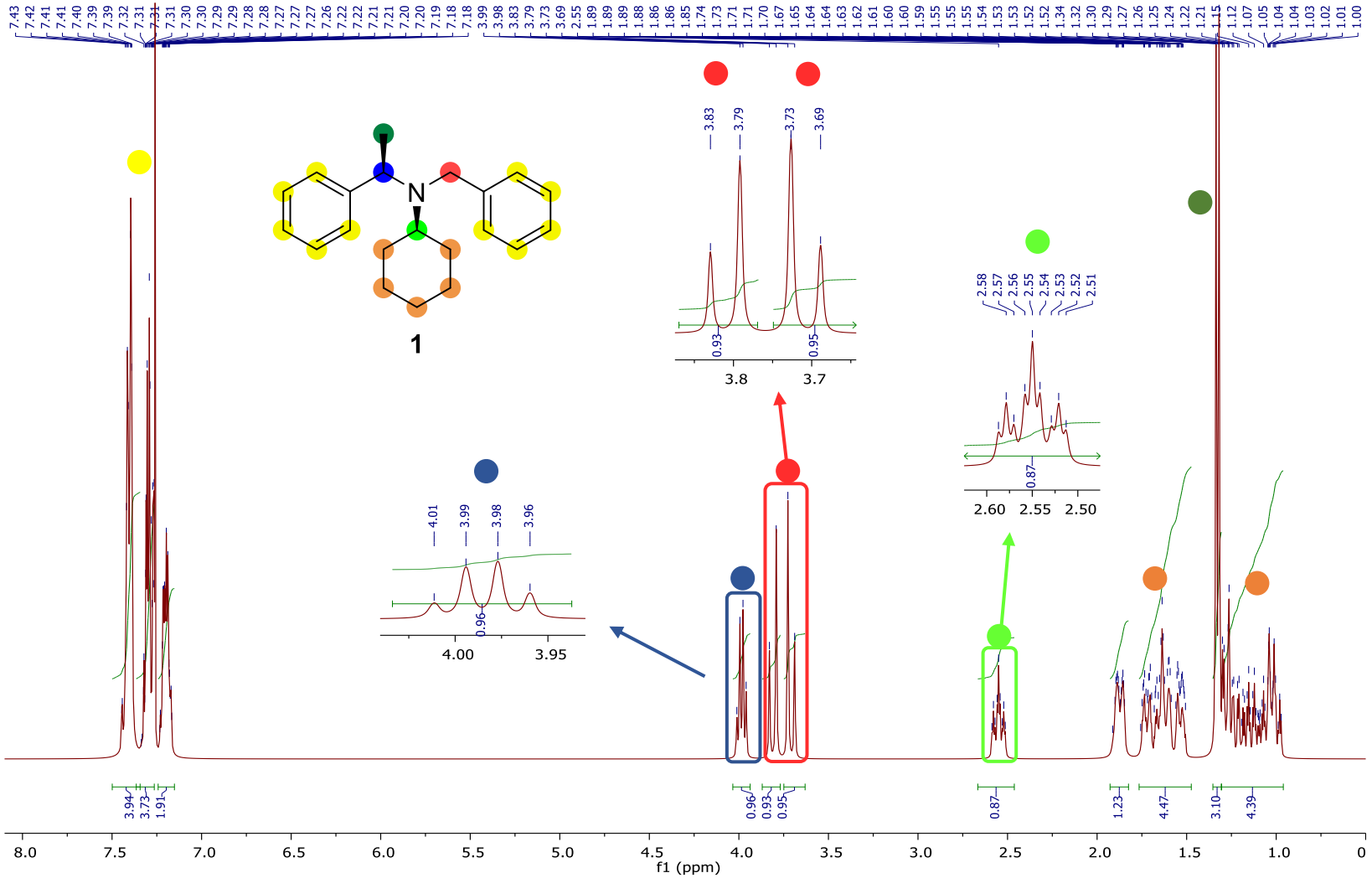
• *Preparation of (1R,2R)-2-(benzyl((R)-1-phenylethyl)amino)cyclohexane-1-carboxylic acid (**5**):* to a mixture of 0.60 g of methyl cyclohex-1-ene-1-carboxylate (**2**) (4.29 mmol) and 10.00 mL of THF was added via cannula the lithium amide ***R*-(3)** (2.00 g, 9.86 mmol), leaving in stirring for 2 hours at -78 ºC. Then, the reaction stopped with NH_4Cl and was left until it reached r.t. The organic phase was washed with citric acid, water and a saturated solution of NaCl. It crude was dried on anhydrous Na_2SO_4, filtered and evaporated the solvent. The reaction crude was chromatographed on silica gel column and increasing eluent (Hex/ AcOEt 99/1 to 95/5) to obtain the Michael adduct methyl (1*R*,2*R*)-2-(benzyl((*R*)-1-phenylethyl)amino)cyclohexane-1-carboxylate (**4**) with a 81 % yield. Its physical and spectroscopic properties agreed those reported in the literature [1]. To 0.20 g of Michael adduct was added 20.00 mL of KOH/ MeOH (20 % V/V) and the mixture was kept in stirring at 70 ºC and reflux for 24h. The reaction mixture was brought to pH 6 and extracted with AcOEt, washed with water and saturated NaCl. It was finally dried on anhydrous Na_2SO_4, filtered and evaporated the solvent. The crude acid was obtained as a beige solid that is sufficiently pure to be used without further purification.



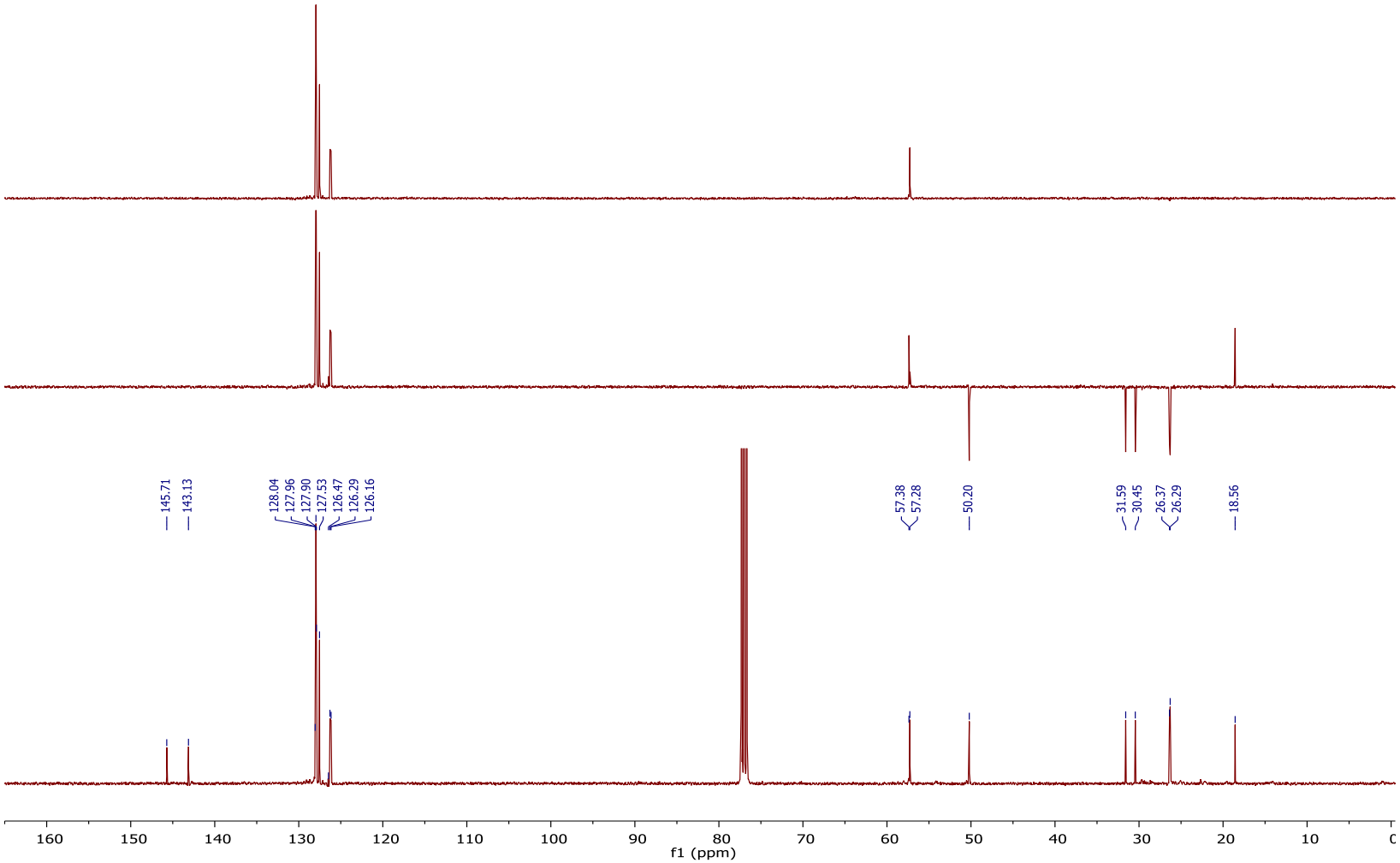
Reference

[1] Manchado, A.; García, M.; Salgado, M. M.; Díez, D.; Garrido, N. M. A Novel Barton Decarboxylation Produces a 1,4-Phenyl Radical Rearrangement Domino Reaction. *Tetrahedron* **2018**, *74* (38), 5240–5247. https://doi.org/10.1016/j.tet.2018.05.043.

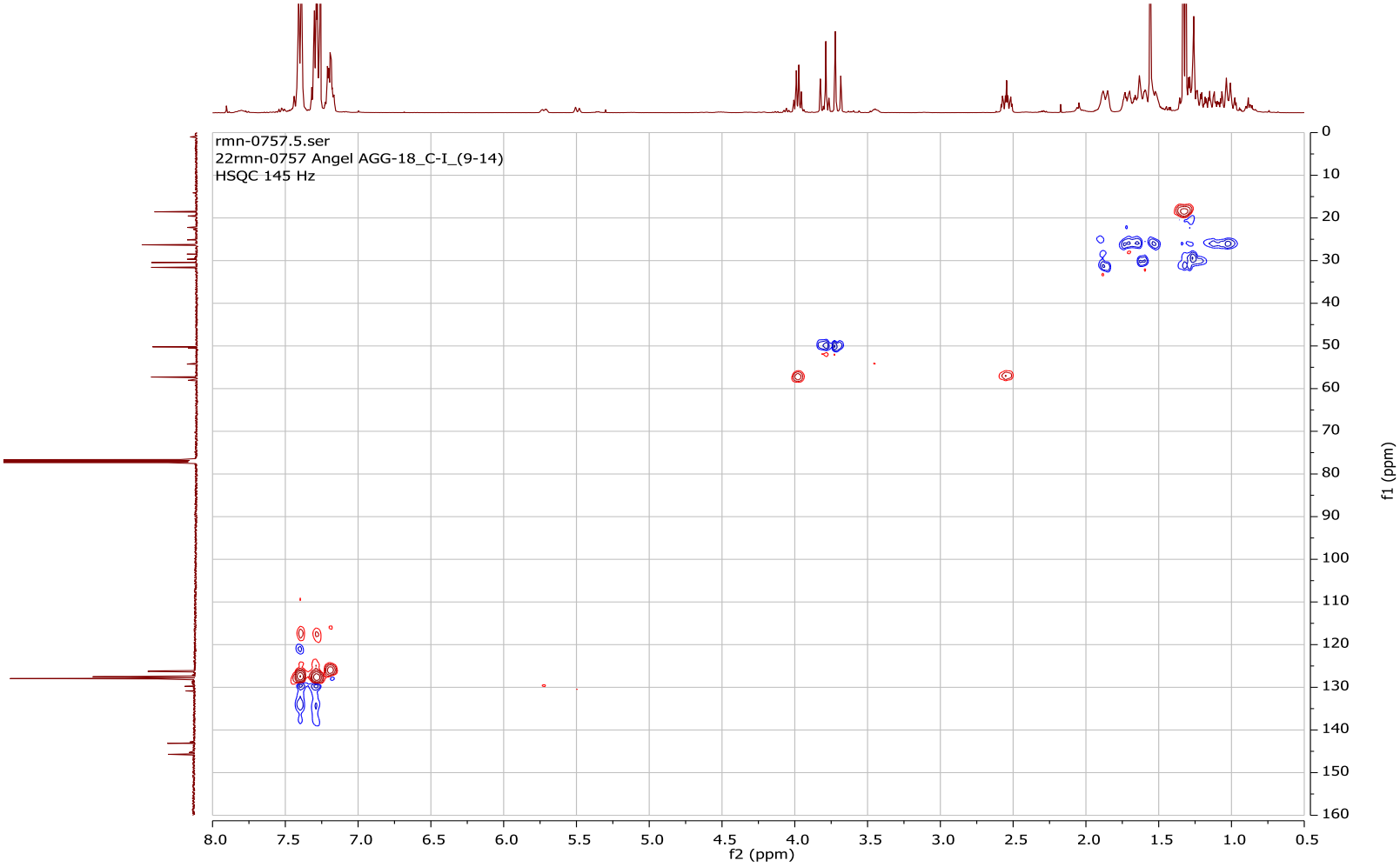
2. ^{1}H NMR of (*R*)-*N*-benzyl-*N*-(1-phenylethyl)cyclohexanamine (**1**).

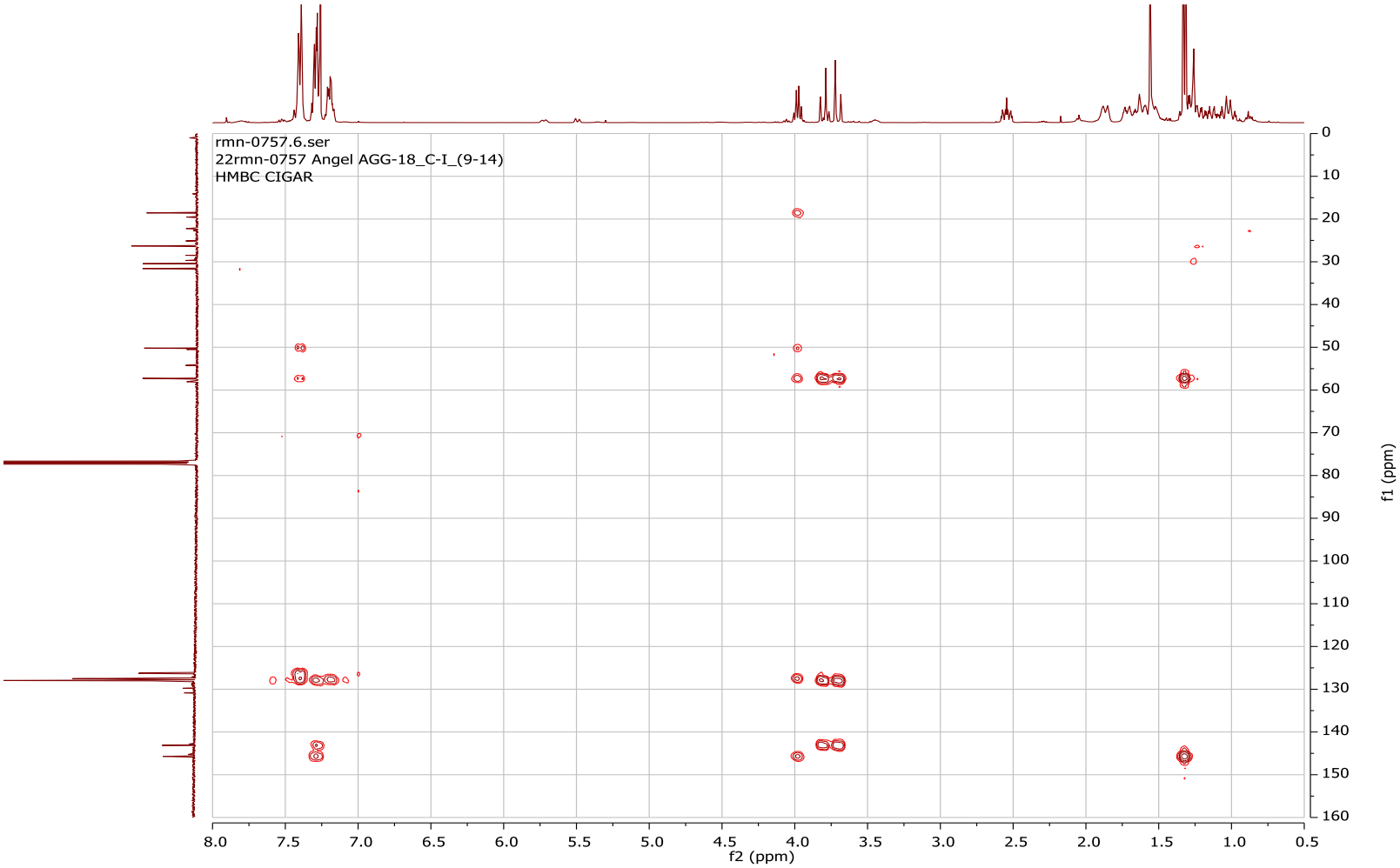


3. ^{13}C NMR of (*R*)-*N*-benzyl-*N*-(1-phenylethyl)cyclohexanamine (**1**).



4. Bidimensional NMR spectra (HSQC, HMBC, and COSY) of (*R*)-*N*-benzyl-*N*-(1-phenylethyl)cyclohexanamine (**1**).

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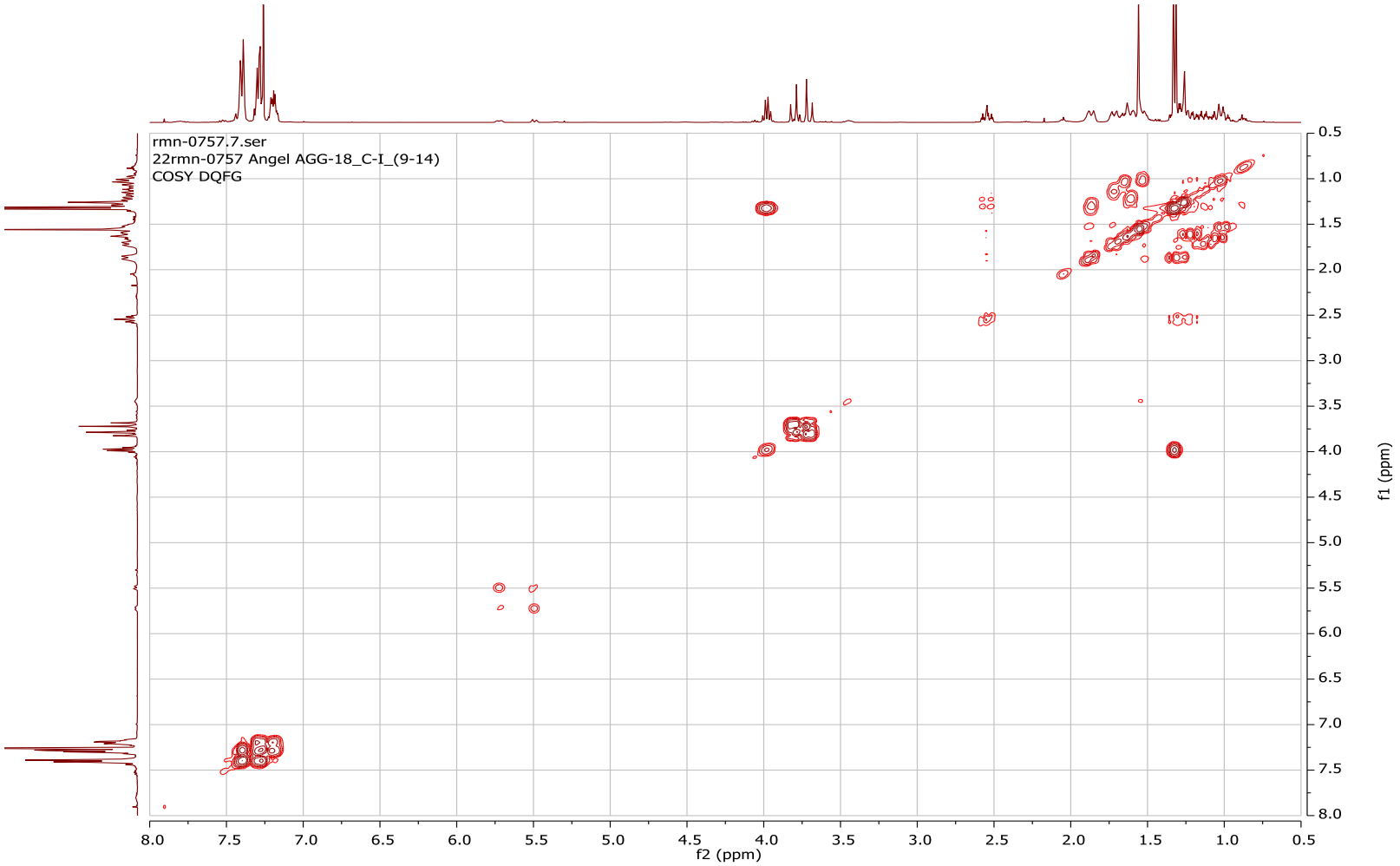
22rmn-0757 Angel AGG-18_C-I_(9-14)
HMBC CIGAR
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f2 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
f1 (ppm)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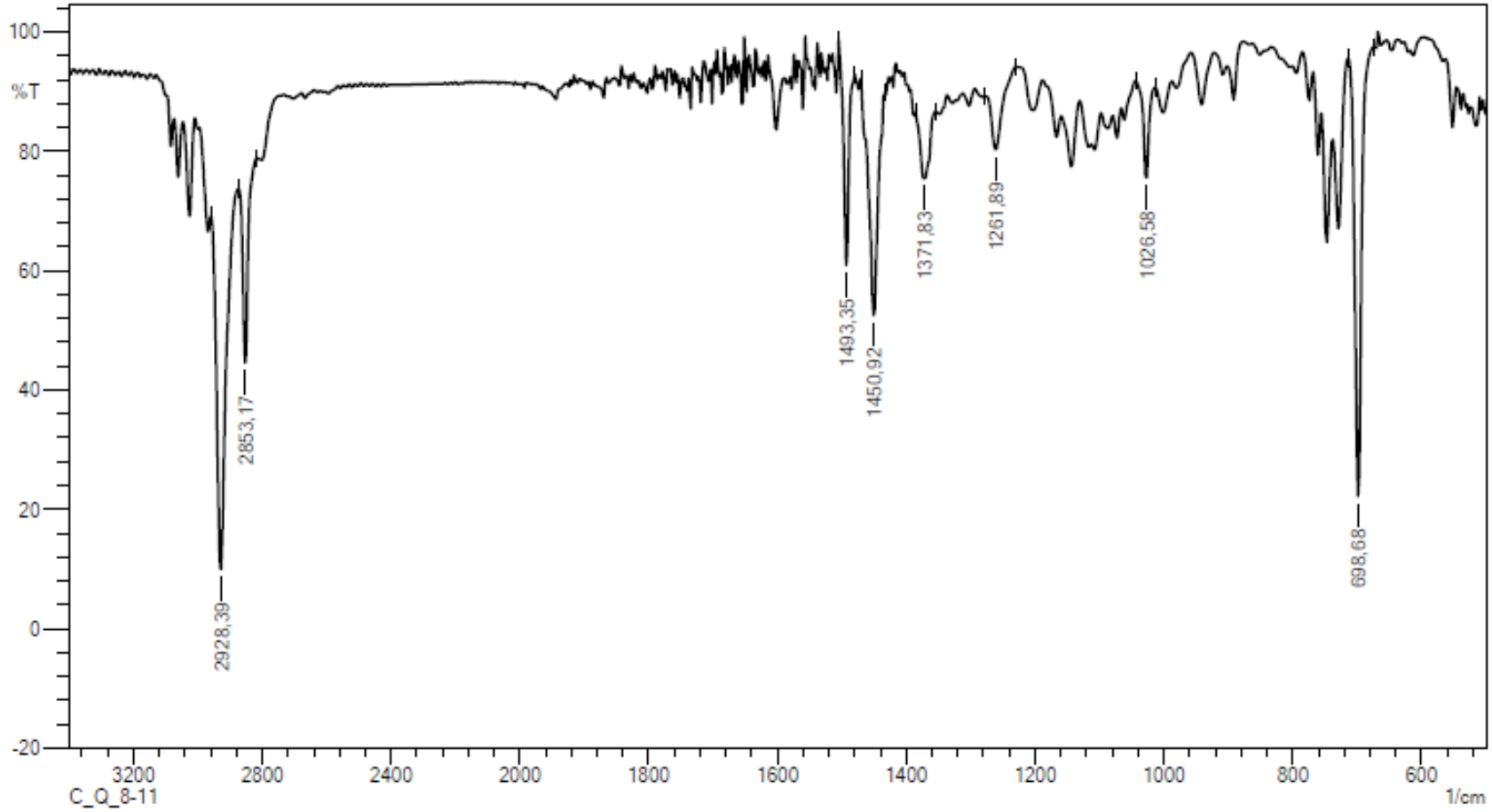
22rmn-0757 Angel AGG-18_C-I_(9-14)
COSY DQFG
f2 (ppm)
f1 (ppm)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

5. Infrared spectrum for (*R*)-*N*-benzyl-*N*-(1-phenylethyl)cyclohexanamine (**1**).



6. Mass spectrum of (*R*)-*N*-benzyl-*N*-(1-phenylethyl)cyclohexanamine (**1**).

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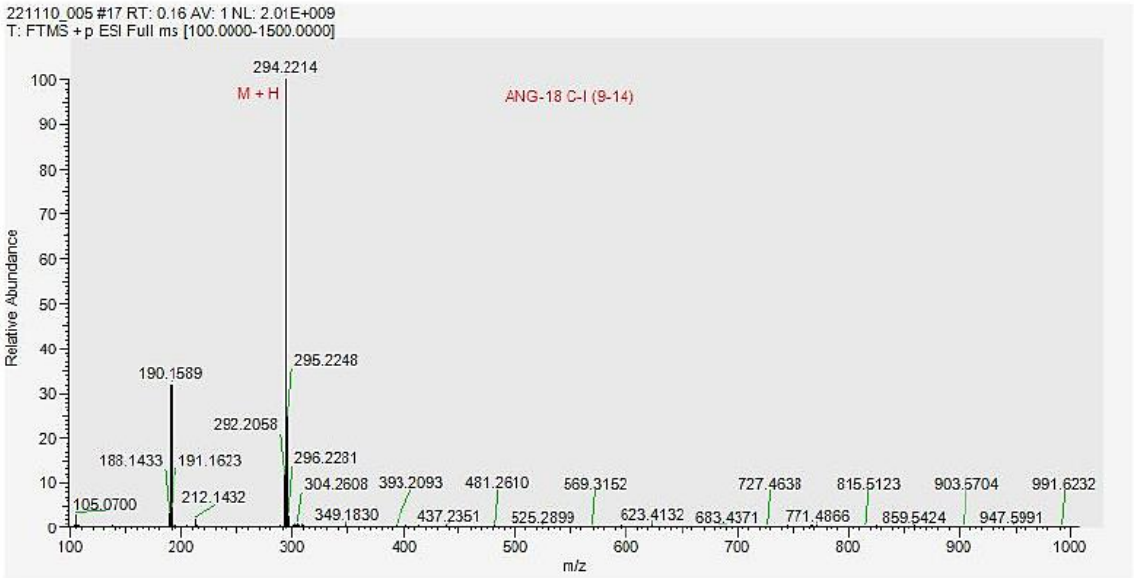
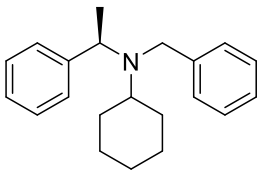


Display Formula	S Fit	RDB	Delta [ppm]	Theo. mass	Combined Score	# Matched Iso.
$C_{21}H_{28}N$	78,88	8,5	-0,75	294,22163	98,89	4
$C_{13}H_{32}O_2N_3{}^{32}S$	10,08	-0,5	1,46	294,22097	75,91	3
$C_{16}H_{33}N^{23}Na^{32}S$	6,989	0,5	-4,03	294,22259	75,75	3



Chemical Formula: $C_{21}H_{27}N$
Molecular Weight: 293,45
Elemental Analysis: C, 85.95; H, 9.27; N, 4.77

The spectrum shows one main peak at m/z = 294.2214 corresponding to the $[M+H]^+$ ion of the presented compound. The isotopic mass and the measured mass of this peak confirm the proposed gross formula $C_{21}H_{27}N$.