Supporting information

Self-organization ability of chiral N^{α}-substituted, N^{β}-Boc protected α-hydrazinoacetamides in the crystal and solution states.

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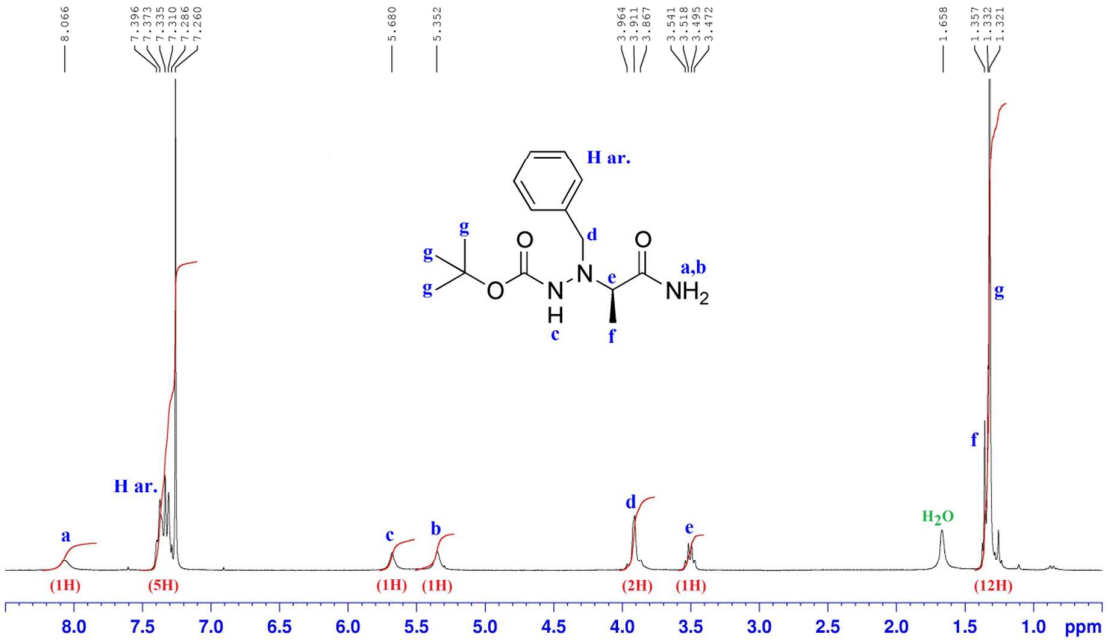
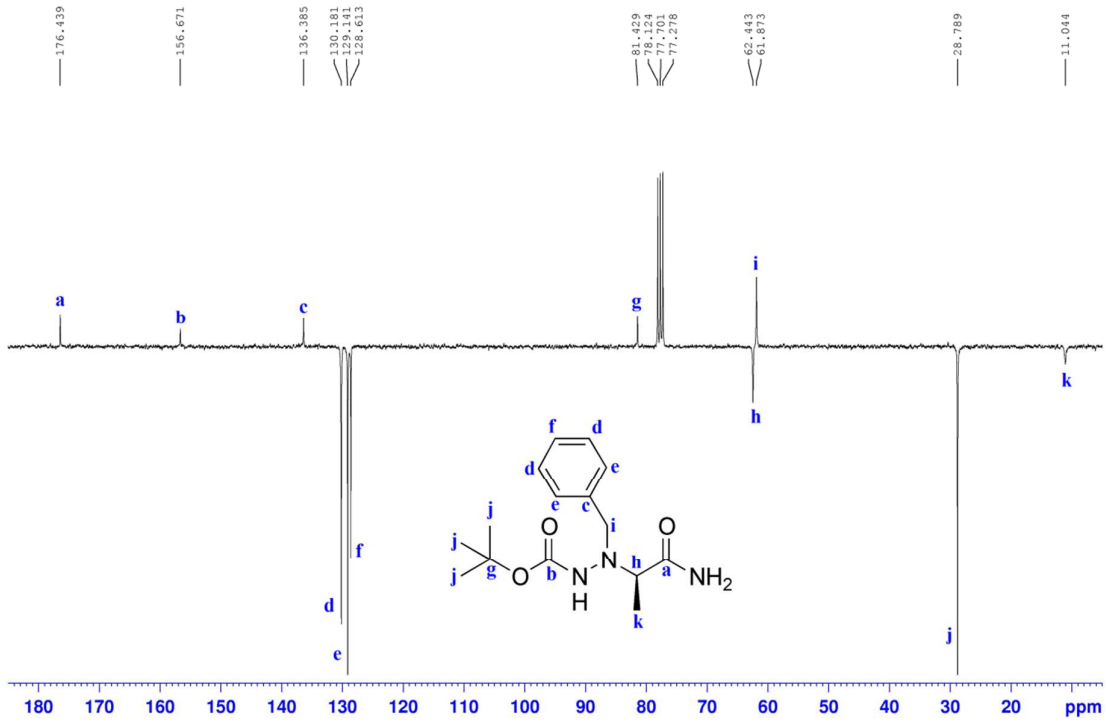
^{1}H NMR (300 MHz) spectrum of 1 ($CDCl_3$/10 mM, 298K)

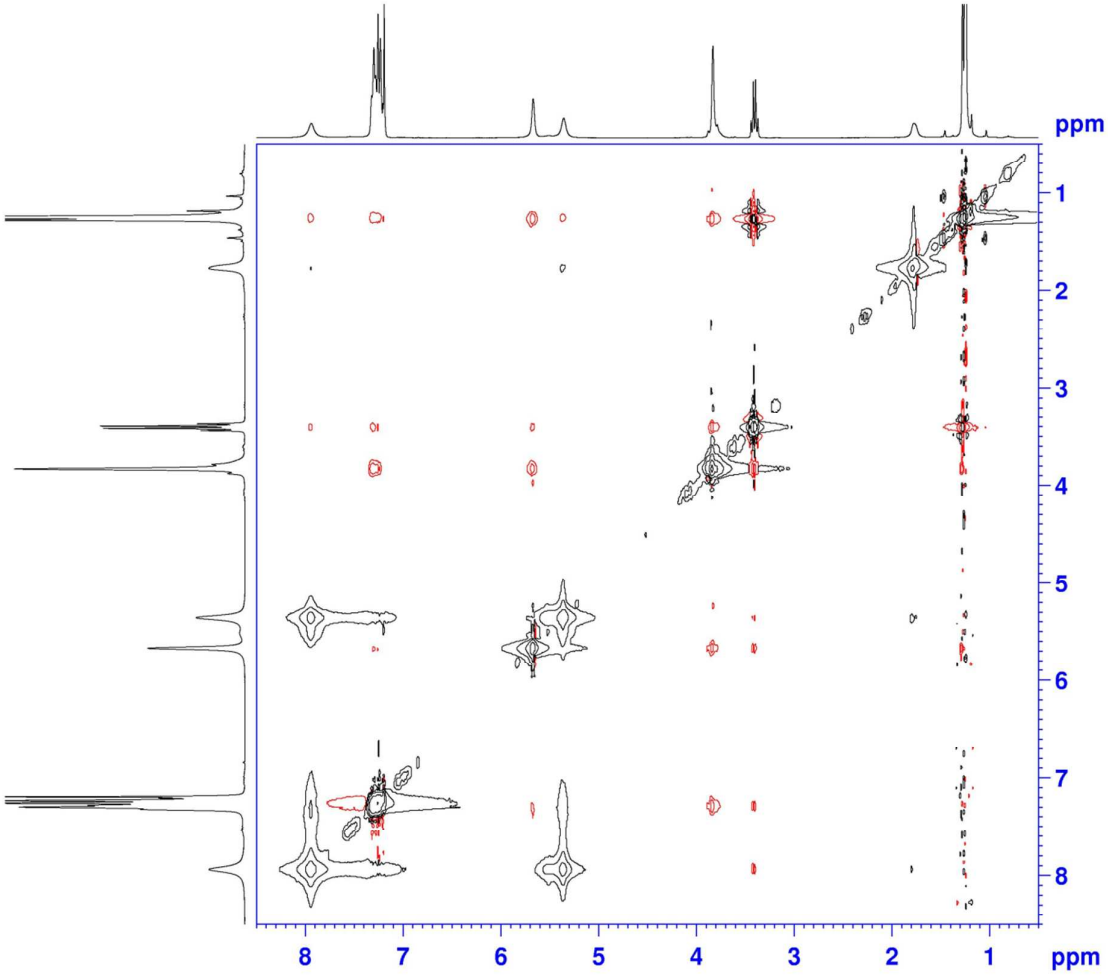


***J* mod^{13}C NMR (75 MHz) spectrum of 1 ($CDCl_3$, 298K)(C and CH_2 up; CH_3 and CH down)**

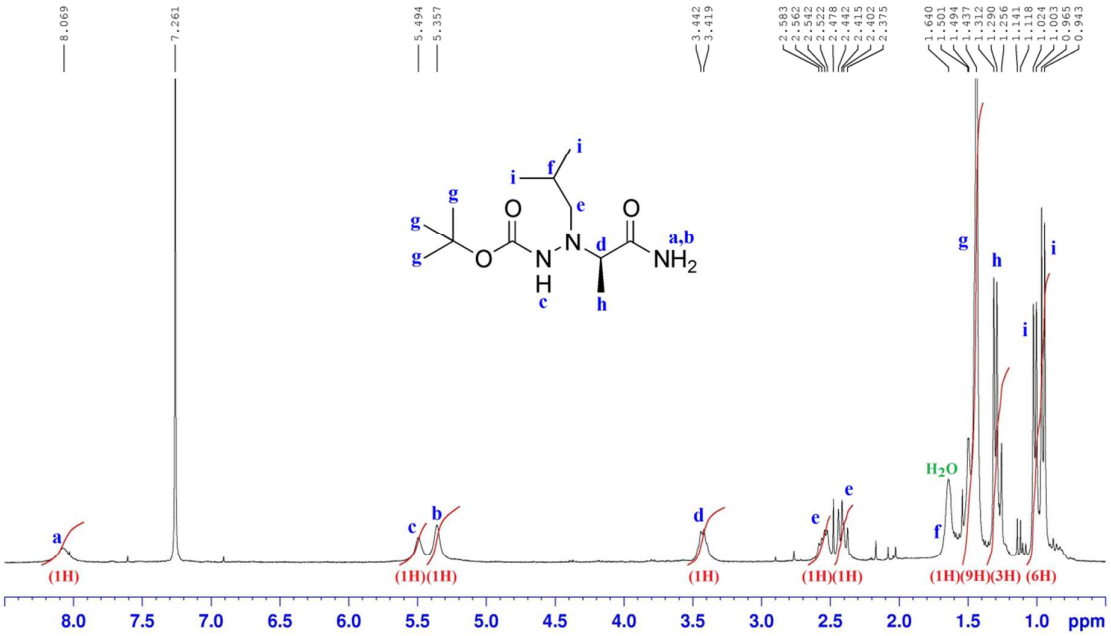


(**d** and **e** are interchangeable carbon chemical shift assignments)

2D NOESYNMR(300 MHz) spectrum of 1 ($CDCl_3$/10 mM, 298K)



^{1}H NMR (300 MHz) spectrum of 2 ($CDCl_3$/10 mM, 298K)



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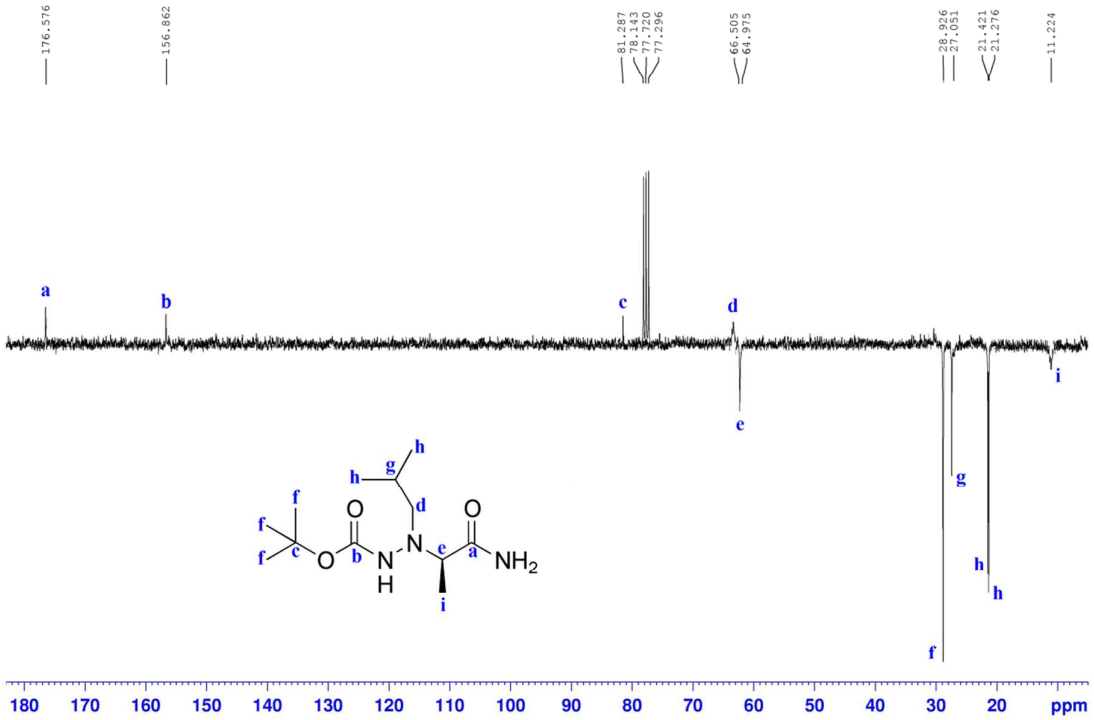
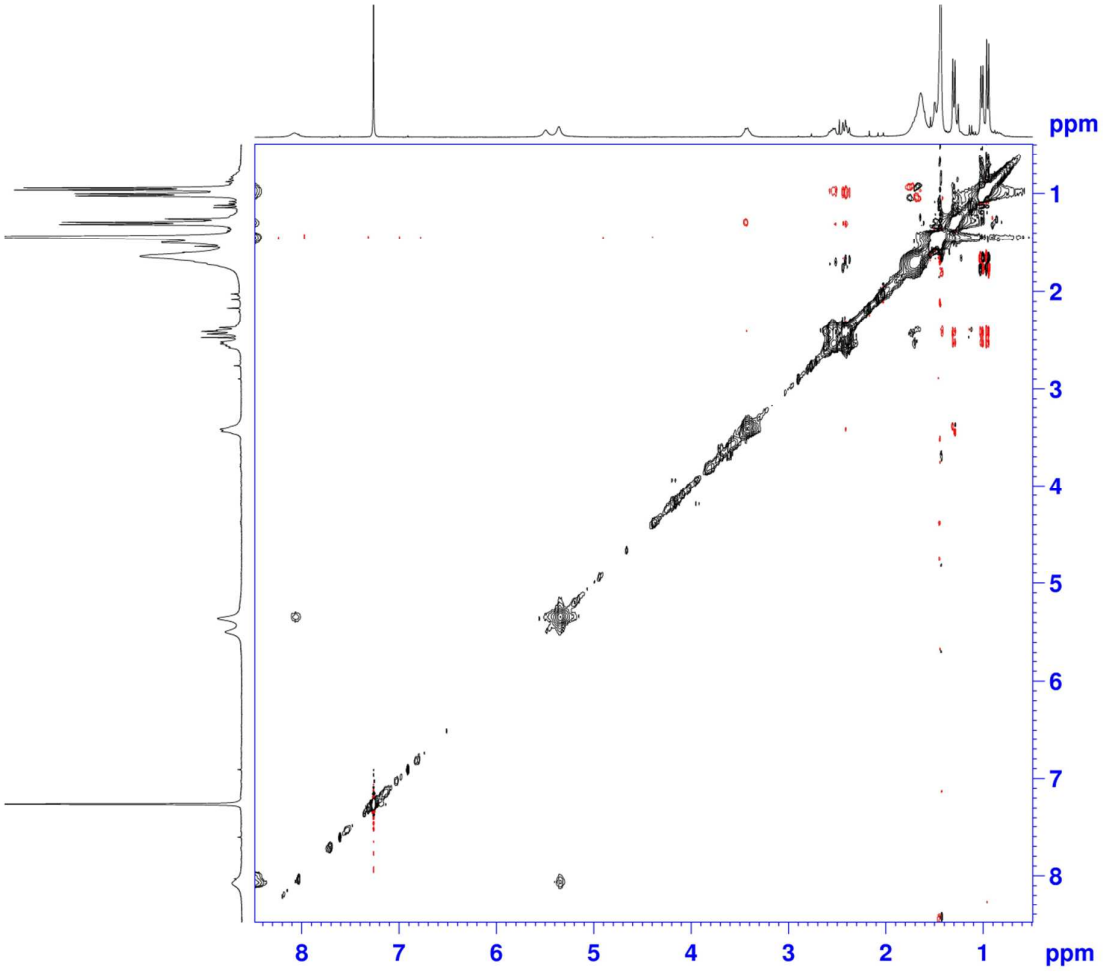
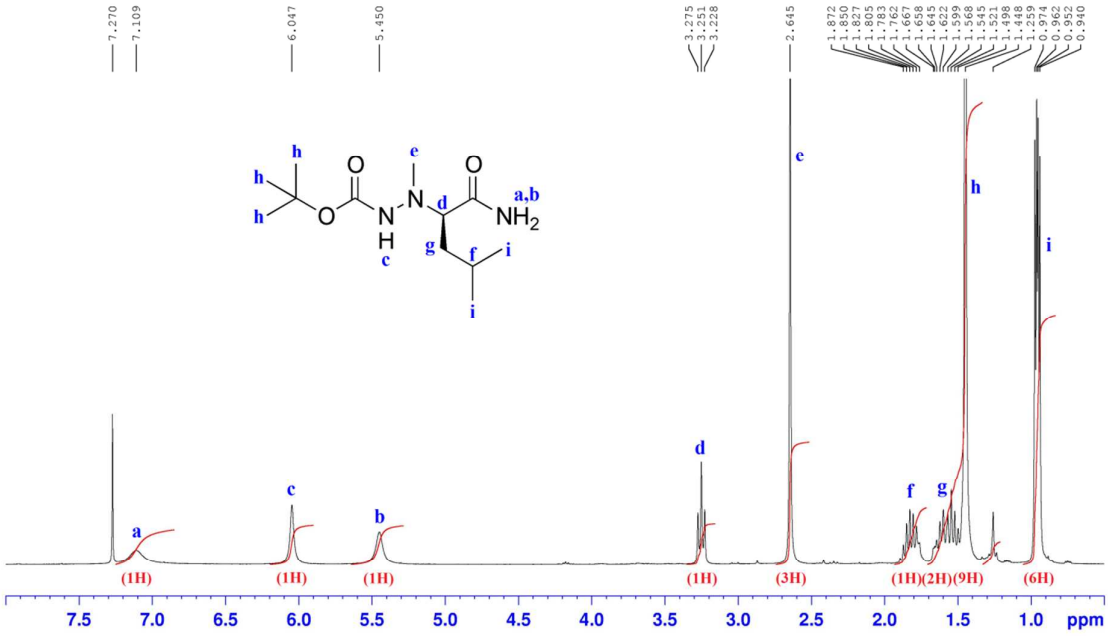


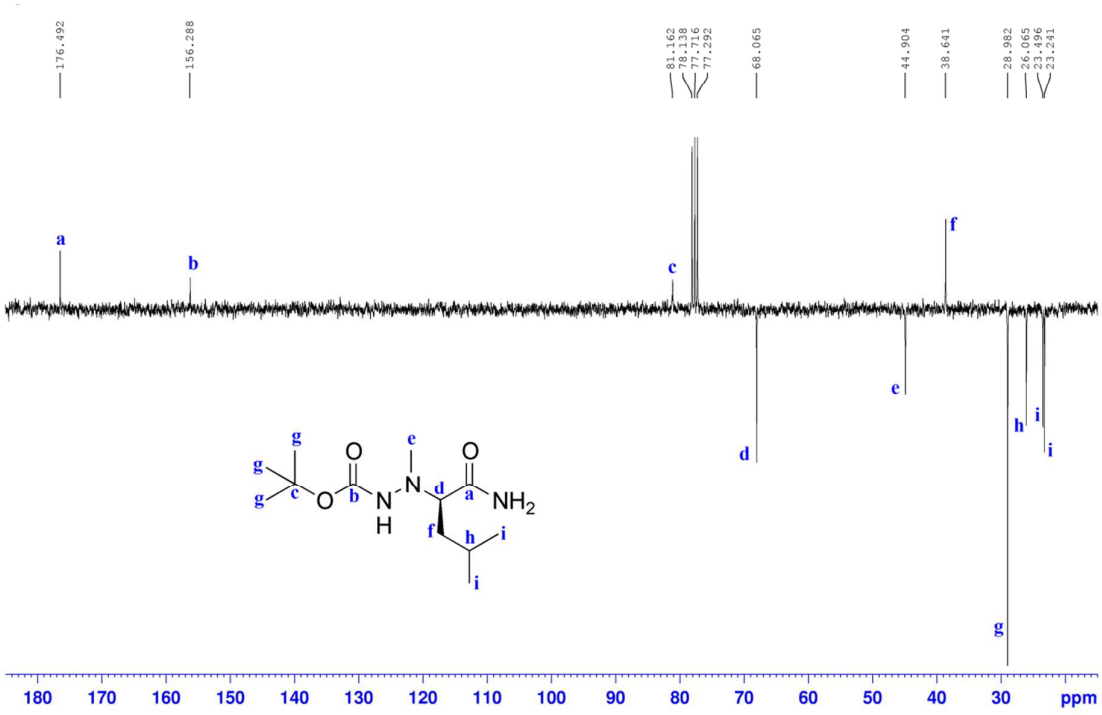
2D NOESYNMR(300 MHz) spectrum of 2 ($CDCl_3$/10 mM, 298K)



^{1}H NMR (300 MHz) spectrum of 3 ($CDCl_3$/10 mM, 298K)



J mod^{13}C NMR (75 MHz) spectrum of 3 ($CDCl_3$, 298K)(C and CH_2 up; CH_3 and CH down)



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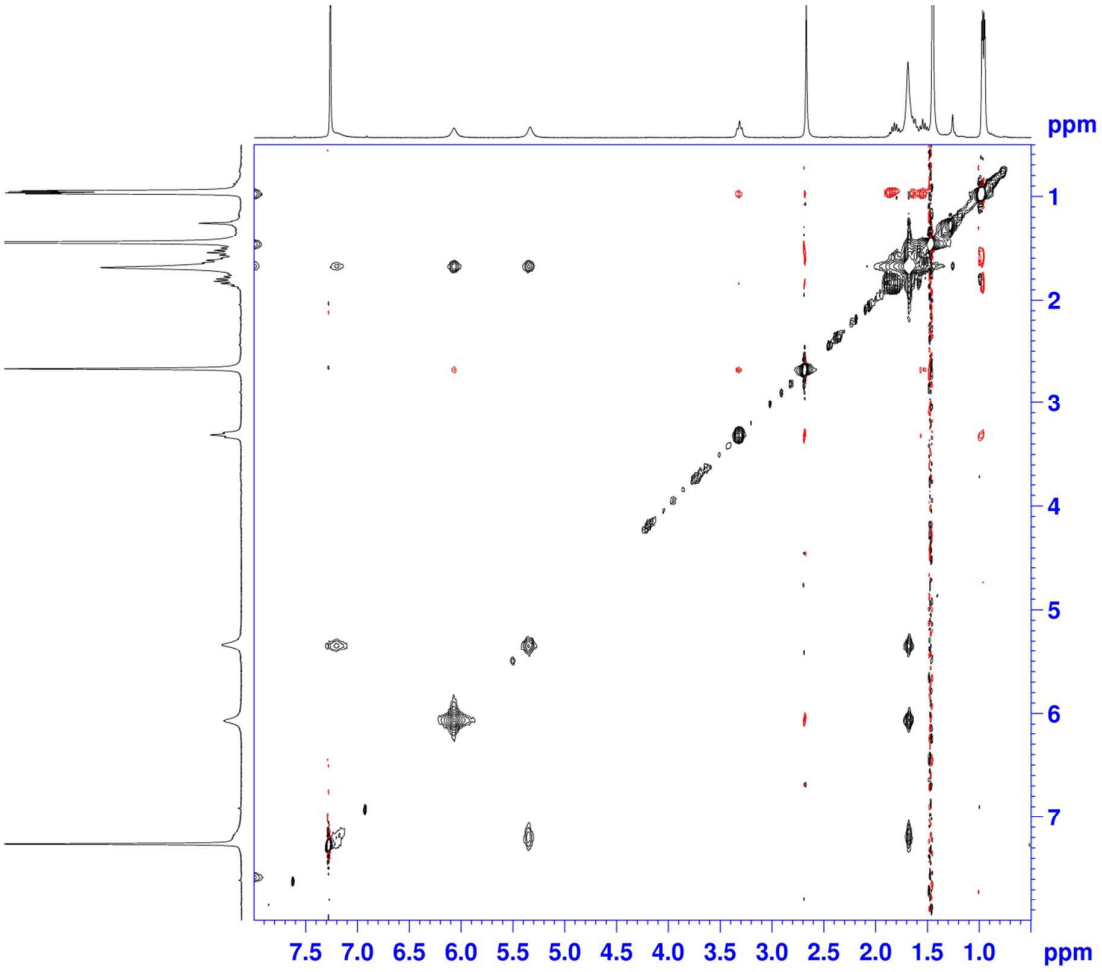


General information for X-ray diffraction data

- ✓ X-ray data from single crystals of compounds **1** and **3** were collected at 110K with a Rigaku Oxford Diffraction SuperNovadiffractometer equipped with a copper microfocus source (λ = 1.54184 Å) and an Atlas CCD detector. Diffraction data were processed using CrysAlis PRO (Rigaku Oxford Diffraction, 2014).

- ✓ X-ray data from a single crystal of compound **2** were collected at 100 K with a D8 Venture Bruker-AXS diffractometer equipped with a molybdenum microfocus source (λ = 0.71073 Å) with a PHOTON100 detector.

- ✓ Diffraction data were processed with the Bruker"SAINT"software package.

- ✓ The three structures were solved by direct methods with SIR2014 (M. C. Burla, R. Caliandro, B. Carrozzini, G. L. Cascarano, C. Cuocci, C. Giacovazzo, M. Mallamo, A. Mazzone, G. Polidori*J. Appl. Cryst.* **2015**, *48*, 306-309).

- ✓ The structures models refinements were conducted using a spherical atom model with ShelXle (C. B. Hübschle, G. M. Sheldrick, B. Dittrich *J. Appl. Cryst.* **2011**, *44*, 1281-1284).

- ✓ All X-ray diffraction data for each compound (**1-3**) are deposited in CCDC database under accession numbers: 1484989, 1484990 and 1484991 respectively.

Crystallographic parameters of 1-3

Compound **1-3** were recrystallized from a mixture petroleum ether/dichloromethane, by slow evaporation.

- ✓ **Crystal structure determination of compound 1:**

$C_{15}H_{23}N_3O_3$, M = 293.36, orthorhombic, a = 5.14336(16), b= 16.7956(5), c = 18.7002(6) Å, U = 1615.43(9) Å^3, T = 110 K, space group $P2_12_12_1$ (no.19), Z = 4, 13322 reflections measured, 3385 unique (Rint = 0.0473), which were used in all calculations. The final w$R(F^2)$ was 0.0905 (all data)

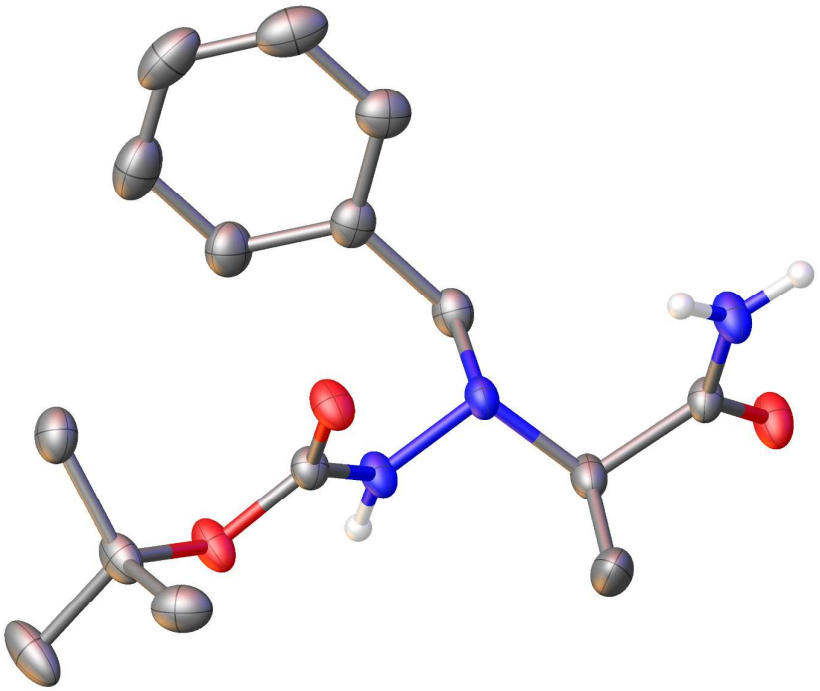
- ✓ **Crystal structure determination of compound 2:**

$C_{24}H_{50}N_6O_6$, M = 518.70, monoclinic, a = 10.5287(4), b= 9.9246(4), c = 14.8794(6) Å^3 = 104.141(2) °, U = 1507.68(10) Å^3, T = 100 K, space group $P2_1$ (no.4), Z = 2, 25270 reflections measured, 5125 unique (Rint = 0.0450), which were used in all calculations. The final w$R(F^2)$ was 0.0869 (all data).

- ✓ **Crystal structure determination of compound 3:**

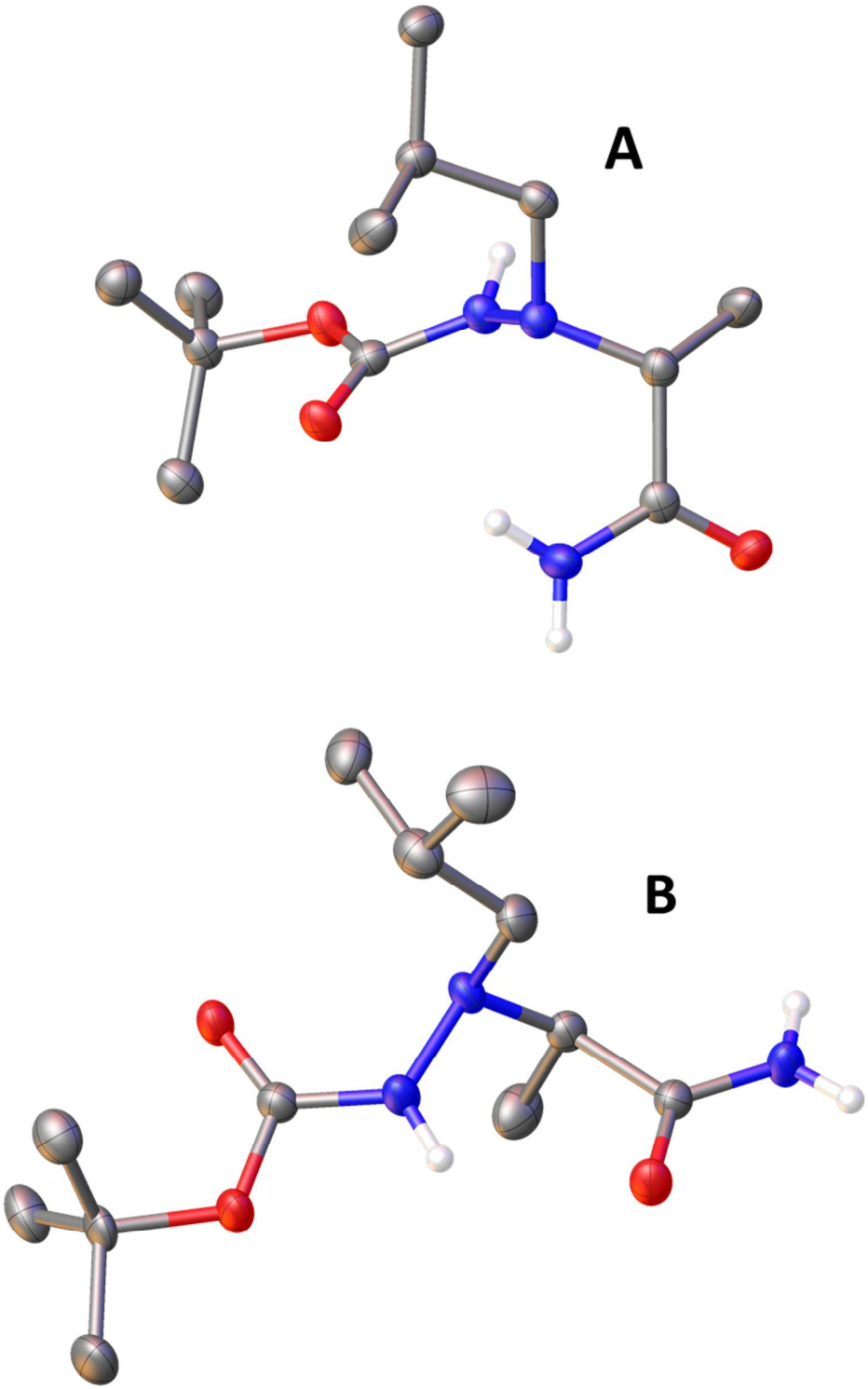
$C_{12}H_{25}N_3O_3$, M = 259.35, orthorhombic, a = 9.787(5), b= 12.007(5), c = 27.226(5) Å, U = 3199(2) Å^3, T = 100 K, space group $P2_12_12_1$ (no.19), Z = 8, 33994 reflections measured, 6686 unique (Rint = 0.0232), which were used in all calculations. The final w$R(F^2)$ was 0.0986 (all data).

ORTEP view of the unique molecule in the asymmetric unit of 1



Displacement spheres are drawn at the 50% probability level
Hydrogen atoms except NHs are omitted for clarity

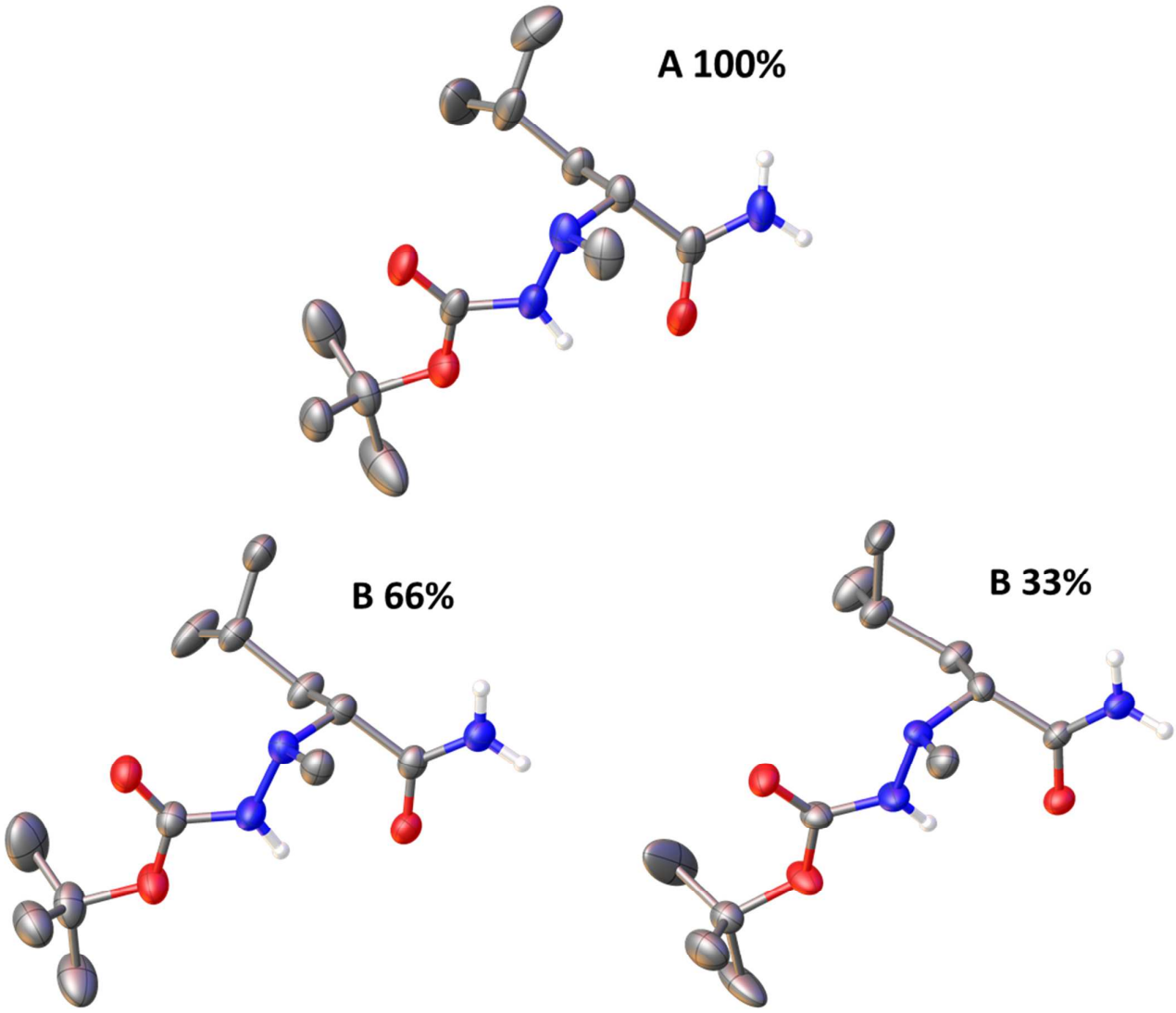
ORTEP view of thetwomolecules(A and B) in the asymmetric unitof 2



A (hydrazinoturn conformation) and B (C_6 conformation)
Displacement spheres are drawn at the 50% probability level.
Hydrogen atoms except NHs are omitted for clarity

ORTEP view of the twomolecules(A and B) in the asymmetric unit of 3

✓ Molecule A is ordered (100% occupancy)
✓ Molecule B is slightly disordered (occupancy ratios of 0.66:0.33)



A and B (C_6 conformations)
Displacement spheres are drawn at the 50% probability level.
Hydrogen atoms except NHs are omitted for clarity