Electronic Supplementary Material for CrystEngComm

Supporting information

Solvent-induced dynamic single-crystal-to-single-crystal transformation of a synthetic peptide-based cyclic compound

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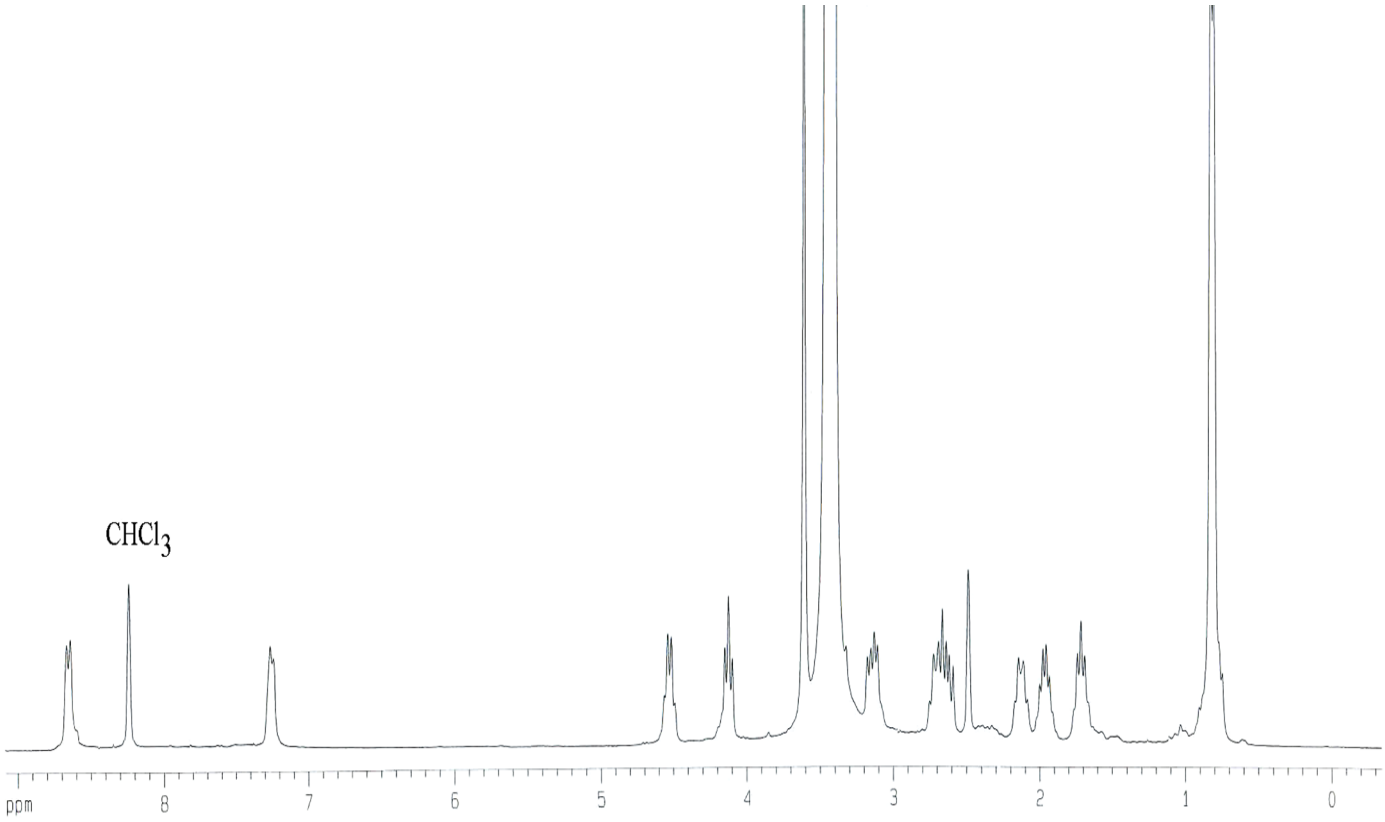


Figure S1: ^{1}H NMR (300 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide with co-crystallized $CHCl_3$ molecules (**1.CHCl$_3$**).

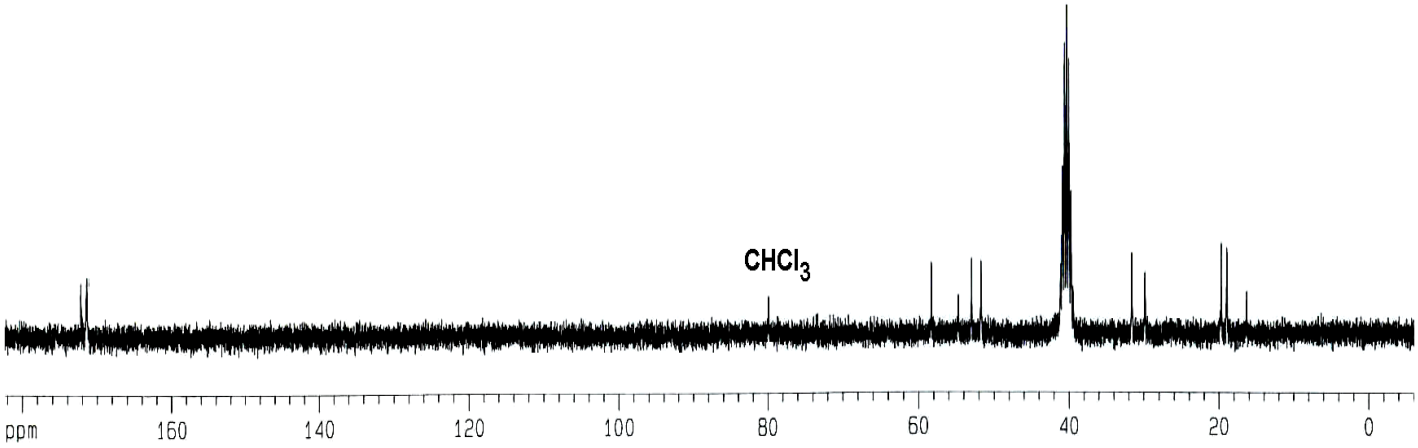


Figure S2: ^{13}C NMR (75 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide with co-crystallized $CHCl_3$ molecules (**1.CHCl$_3$**).

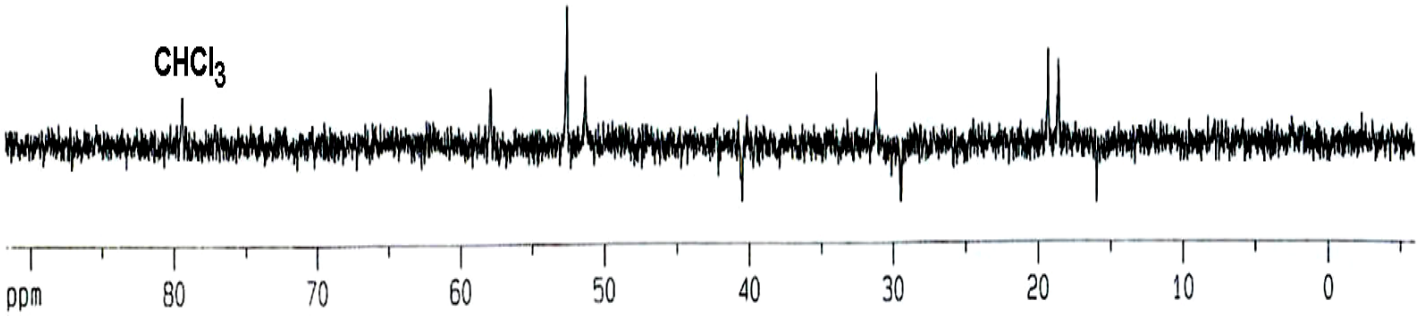


Figure S3: DEPT 135 (75 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide with co-crystallized $CHCl_3$ molecules (**1.CHCl$_3$**).

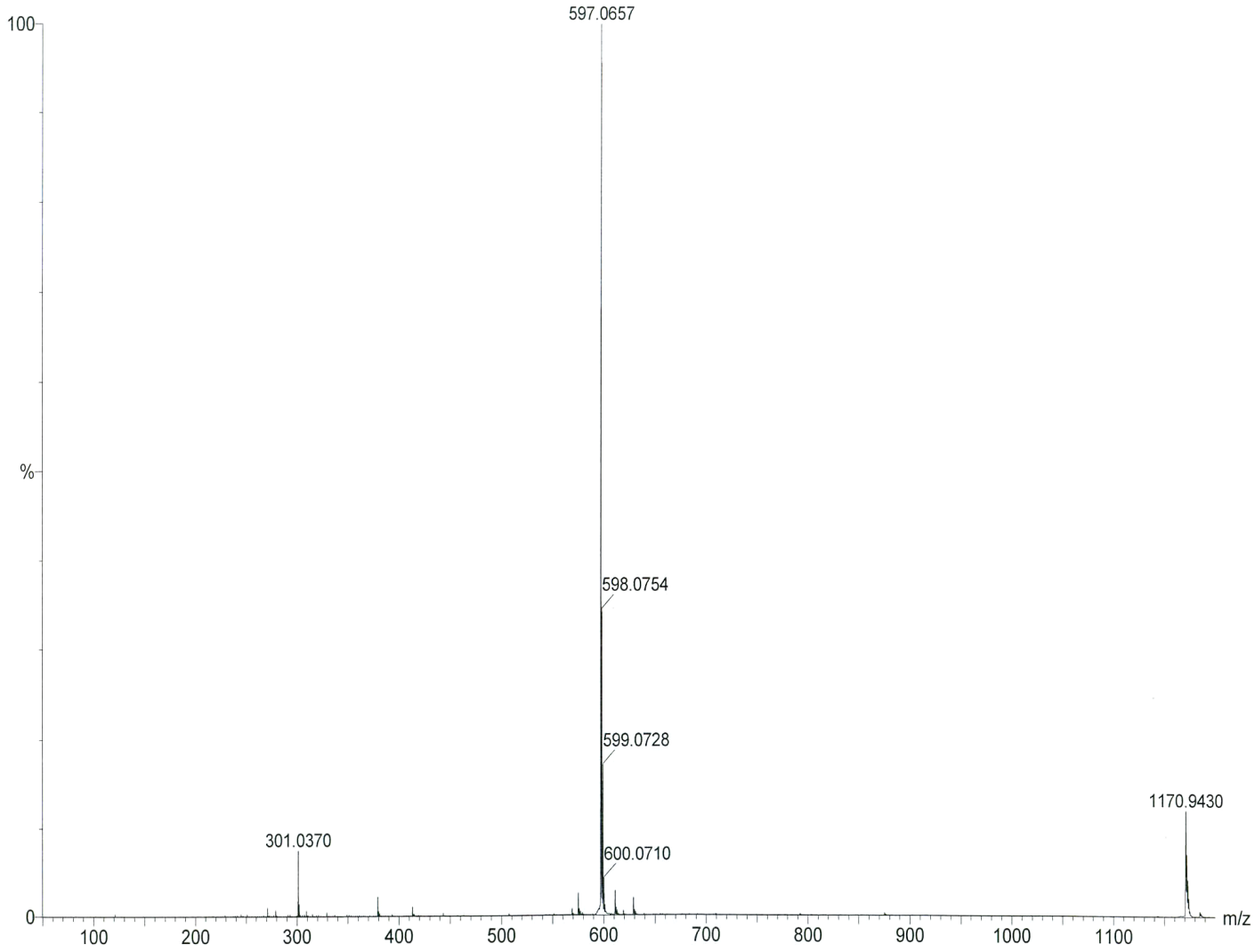


Figure S4: HRMS (ESI) data of the cyclic peptide with co-crystallized $CHCl_3$ molecules (**1.$CHCl_3$**).

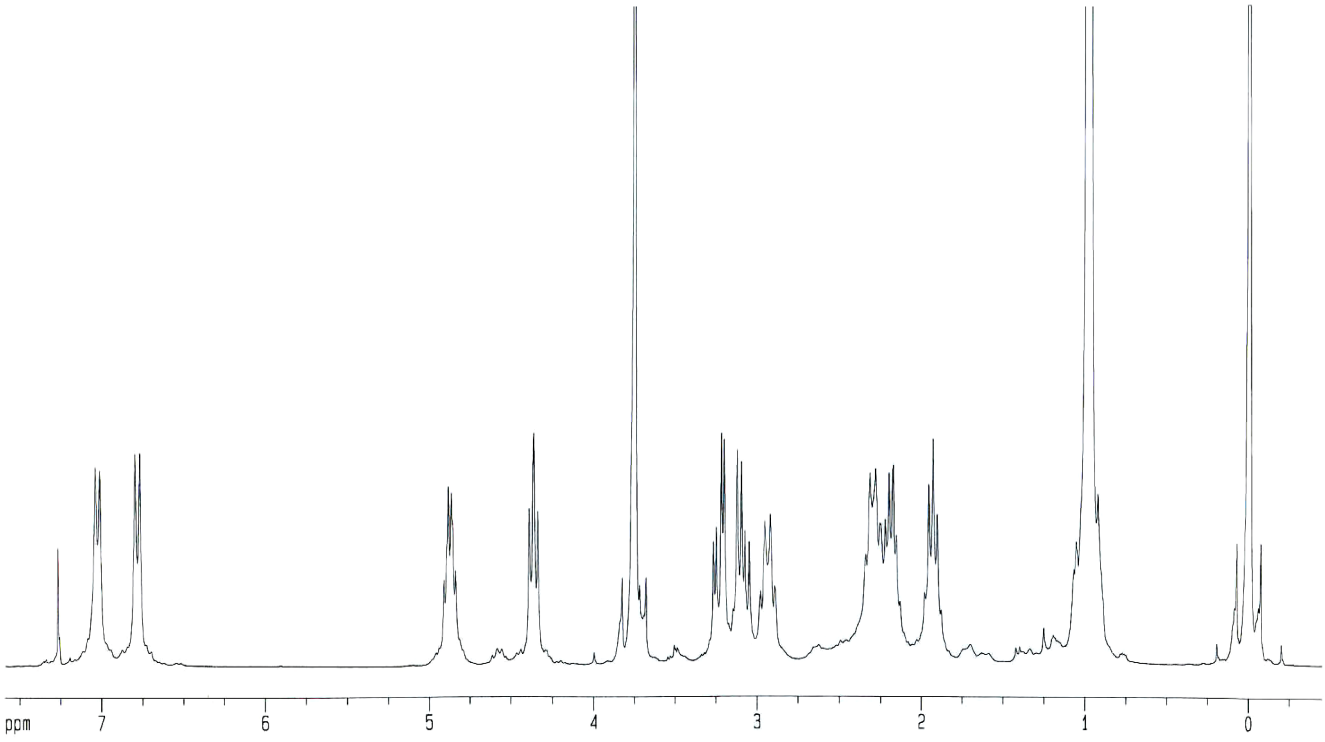


Figure S5: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C, TMS) spectrum of the cyclic peptide **1**.

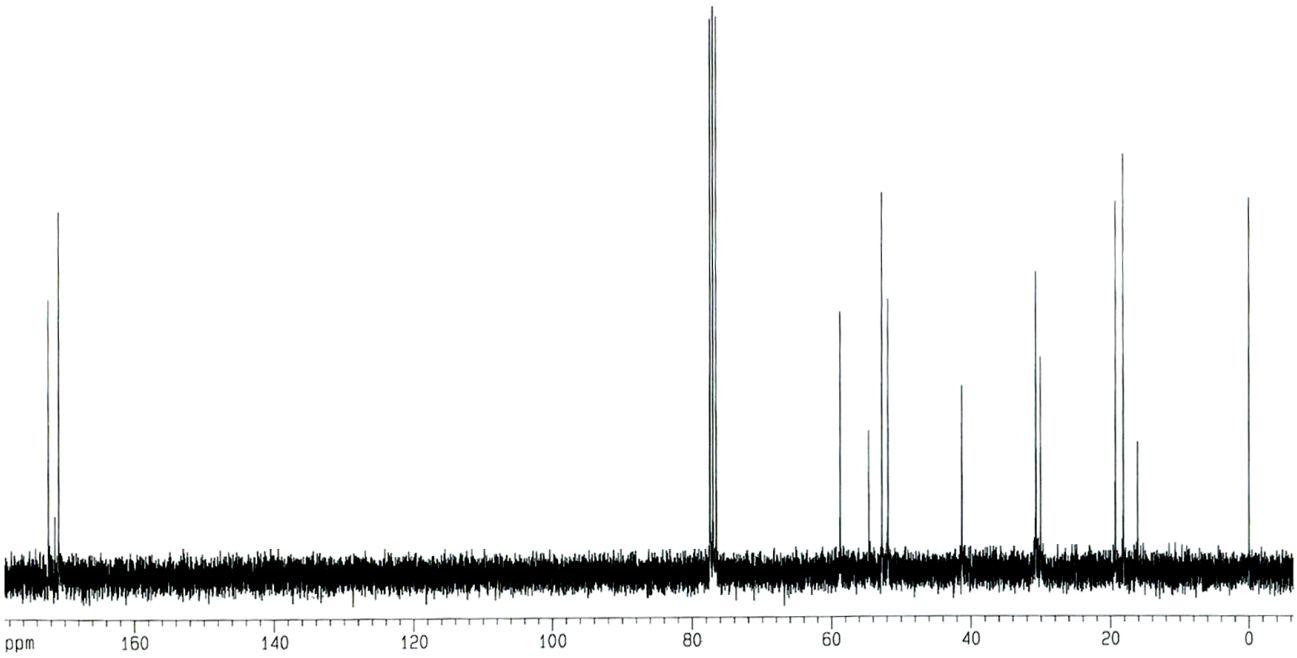


Figure S6: ^{13}C NMR (75 MHz, $CDCl_3$, 25 °C, TMS) spectrum of the cyclic peptide **1**.

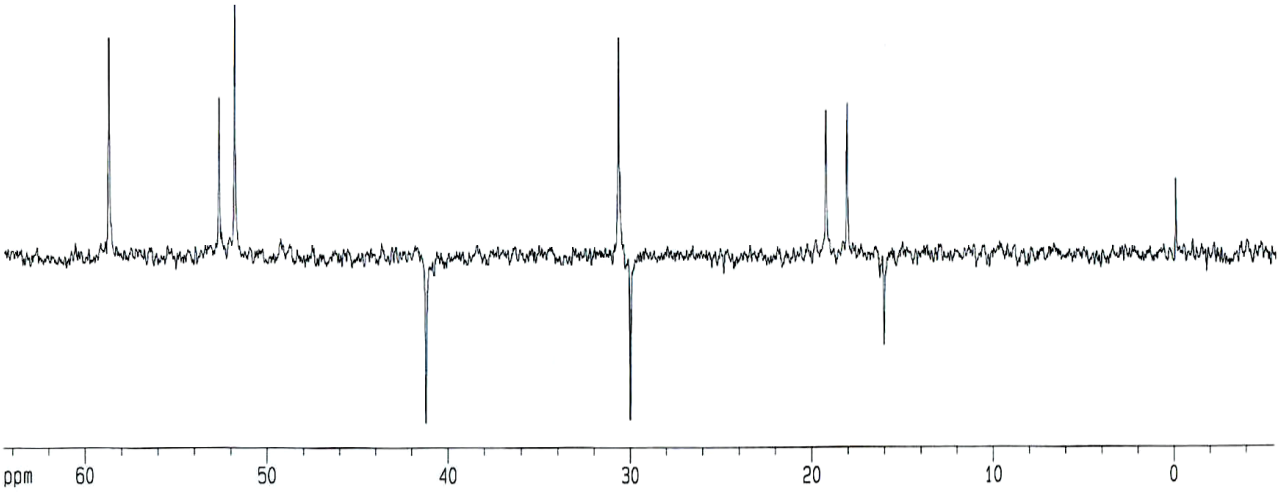


Figure S7: DEPT 135 (75 MHz, $CDCl_3$, 25 °C, TMS) spectrum of the cyclic peptide **1**.

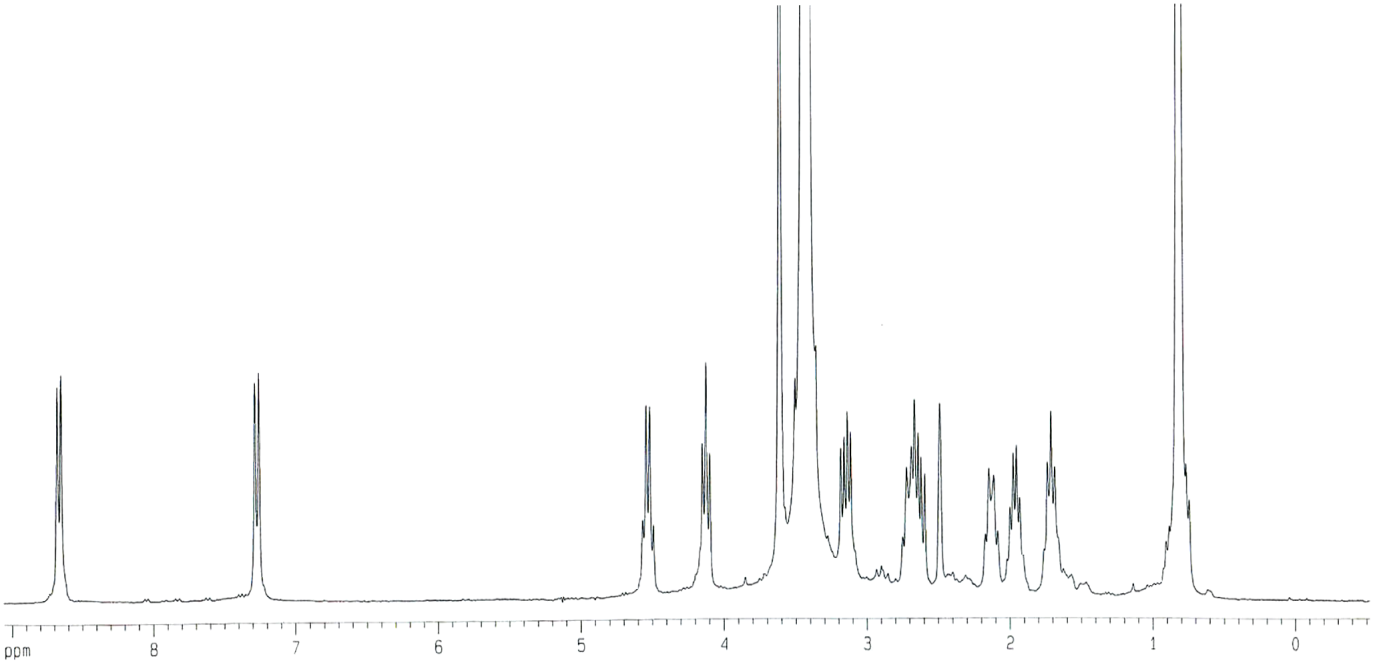


Figure S8: ^{1}H NMR (300 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide **1**.

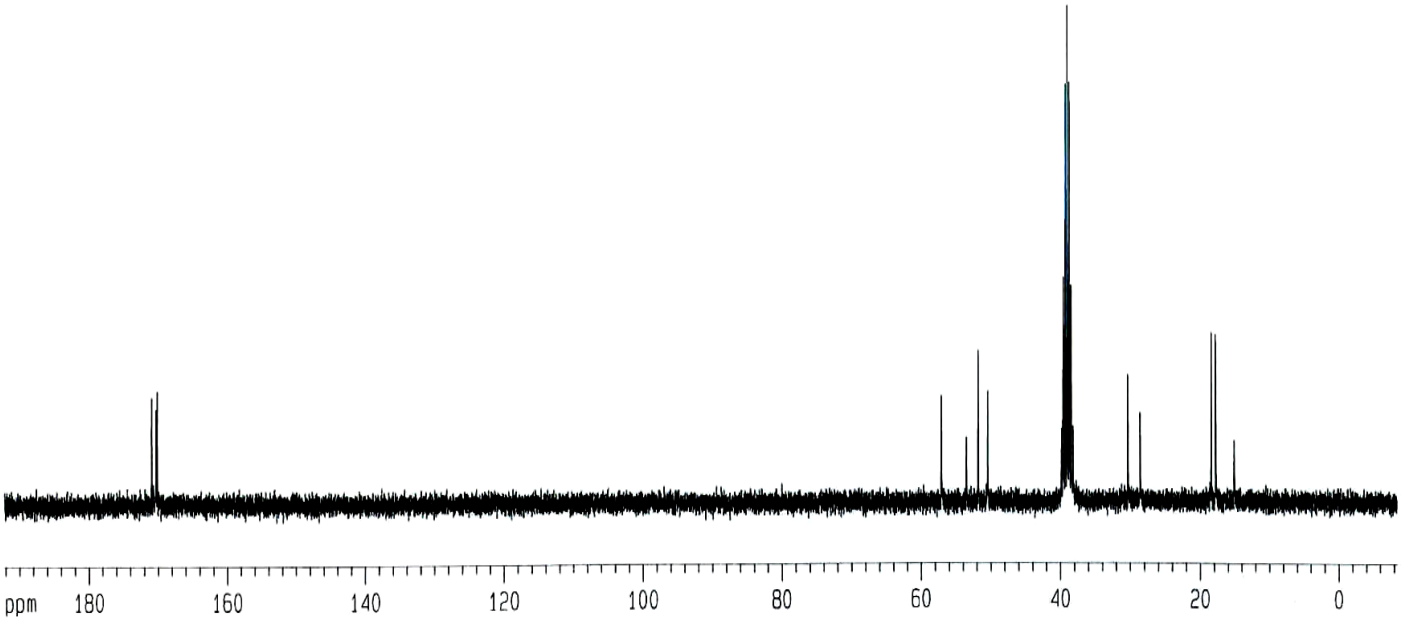


Figure S9: ^{13}C NMR (75 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide **1**.

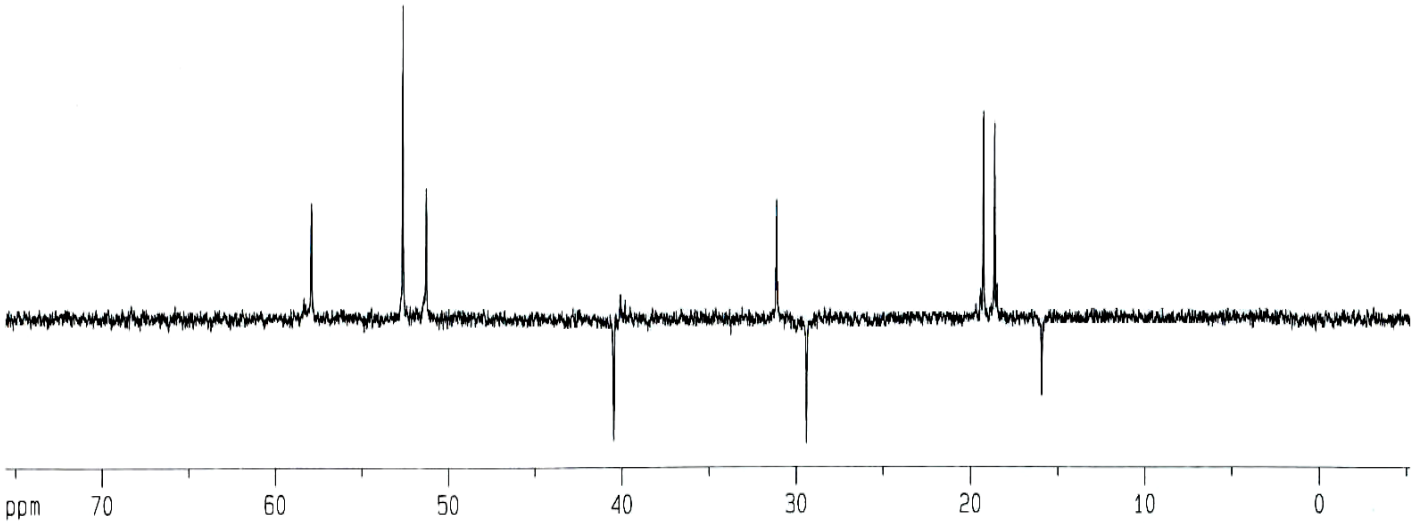


Figure S10: DEPT 135 (75 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide **1**.

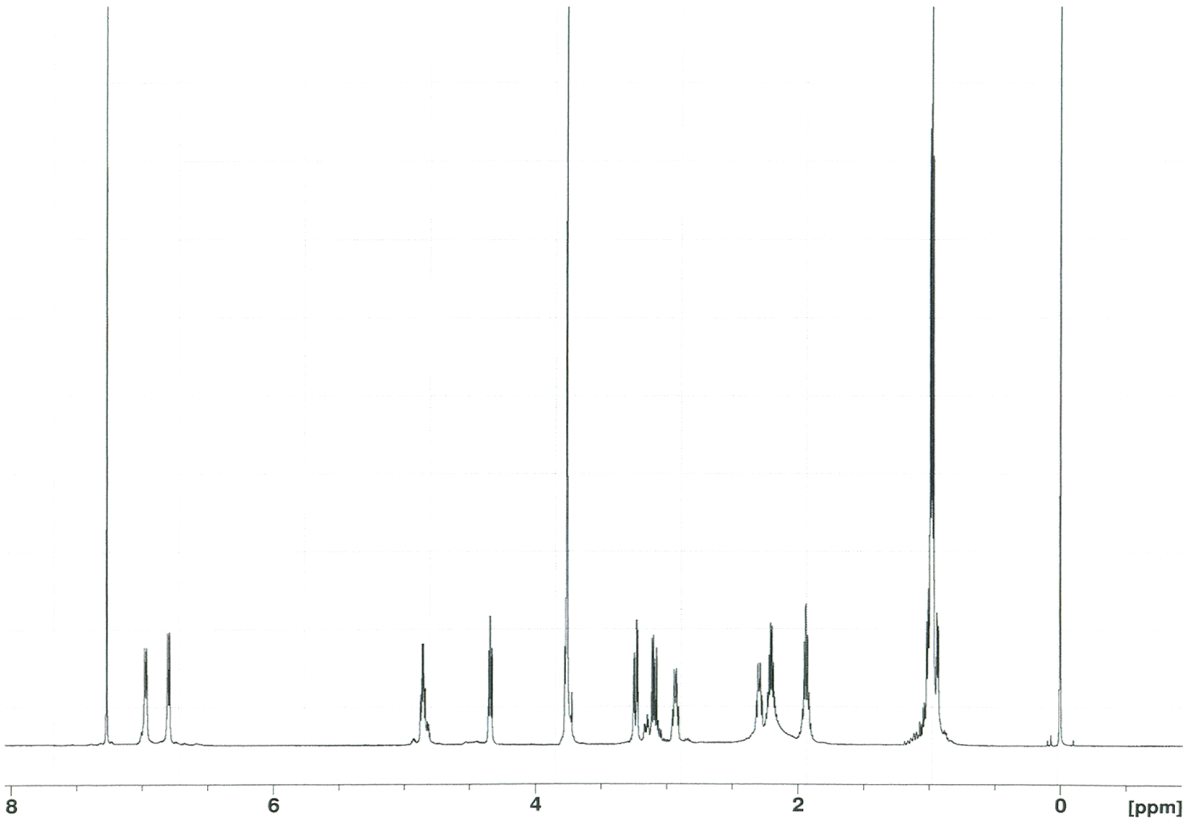


Figure S11: ^{1}H NMR (600 MHz, $CDCl_3$, 25 °C, TMS) spectrum of the cyclic peptide **1**.

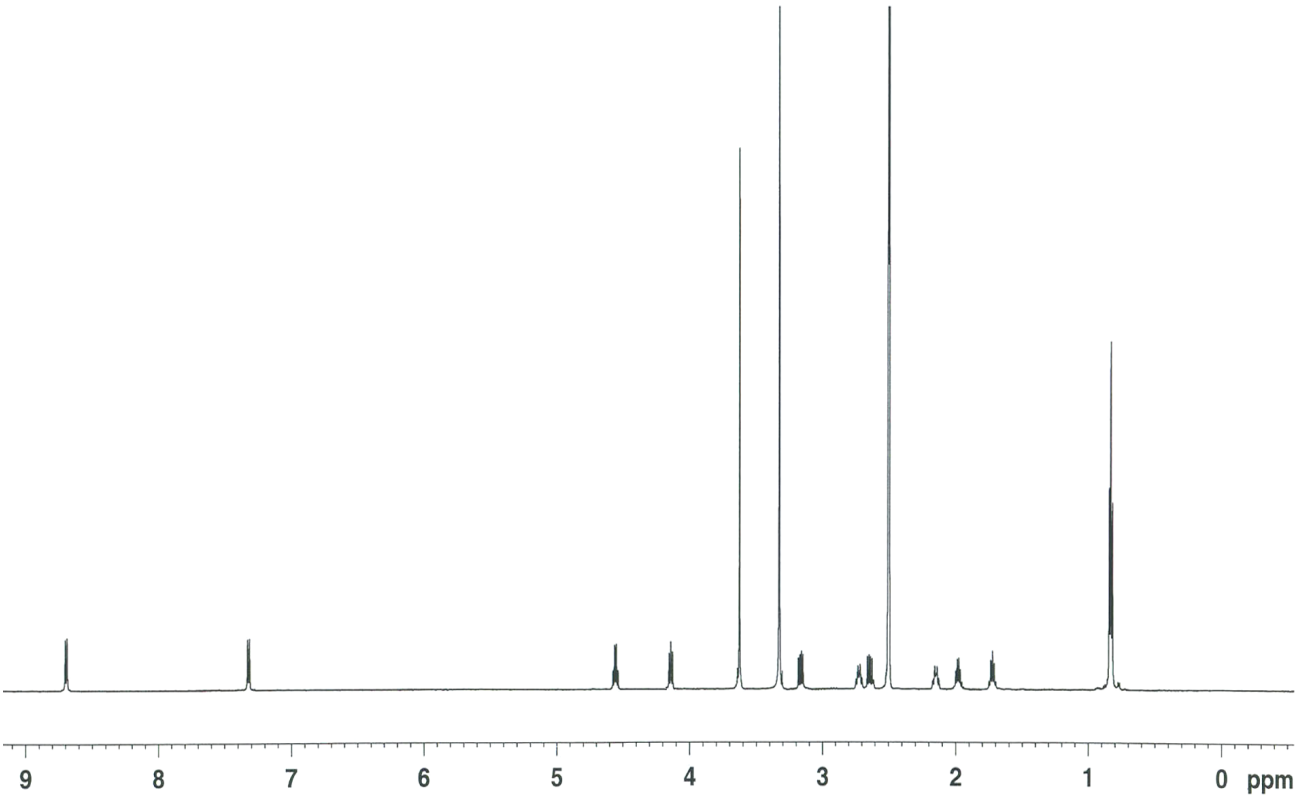


Figure S12: ^{1}H NMR (600 MHz, DMSO-d_6, 25 °C) spectrum of the cyclic peptide **1**.

Cyclic peptide **1.CH_3CN:**

^{1}H NMR (300 MHz, $CDCl_3$, 25 °C, TMS): δ = 6.69 (d, J = 8.4 Hz, 2H; L-Cys NH), 6.64 (d, J = 8.4 Hz, 2H; L-Val NH), 4.78–4.85 (m, 2H; L-Cys C^{α} H), 4.23–4.28 (m, 2H; L-Val C^{α} H), 3.74 (s, 6H; $-OCH_3$), 3.20–3.26 (m, 2H; L-Cys diastereotopic C^{β} H), 3.07–3.14 (m, 2H; L-Cys diastereotopic C^{β} H), 2.87–2.96 (m, 2H; α-CH_2 of 1,1-cyclobutane dicarboxamide), 2.17–2.30 (m, 2H; α-CH_2 of 1,1-cyclobutane dicarboxamide and 2H; L-Val C^{β} H), 2.15 (s, 3H; CH_3CN), 1.91–1.99 (m, 2H; β-CH_2 of 1,1-cyclobutane dicarboxamide), 0.93–0.97 (m, 12H; L-Val C^{γ} Hs).

Anal. calcd for $C_{26}H_{41}N_5O_8S_2$ (615): C 50.71, H 6.71, N 11.37, S 10.42 found: C 50.64, H 6.80, N 11.41, S 10.39.

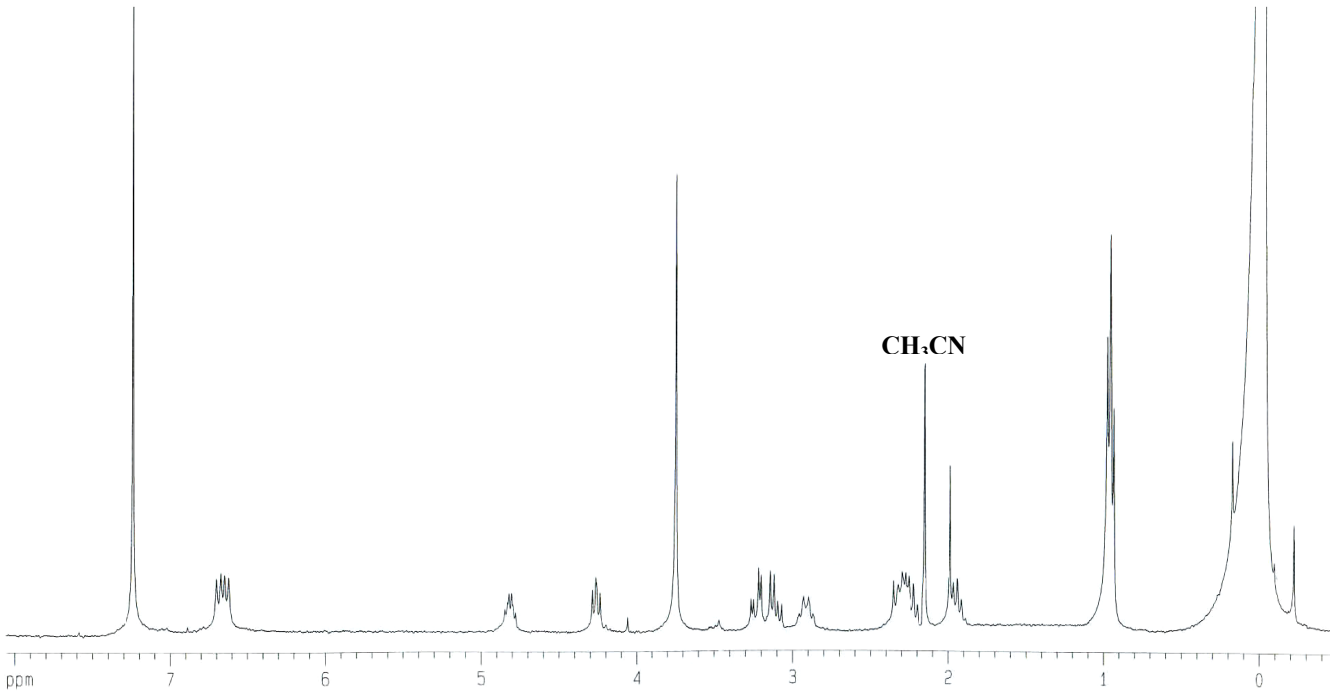
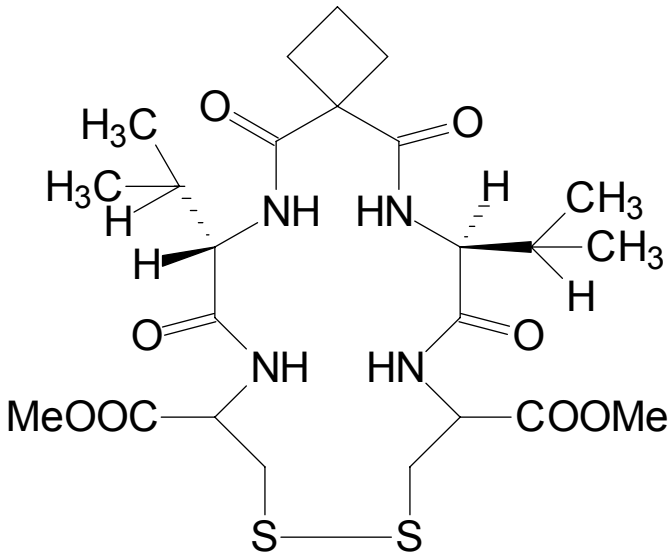
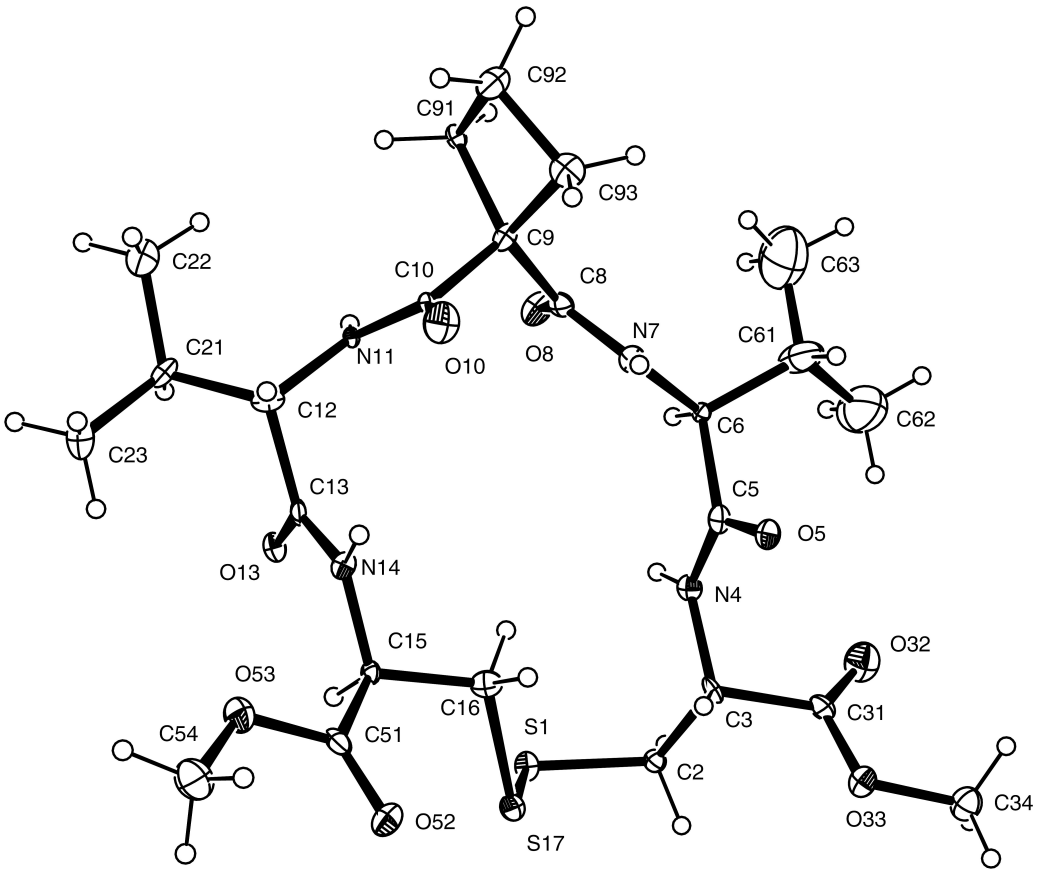


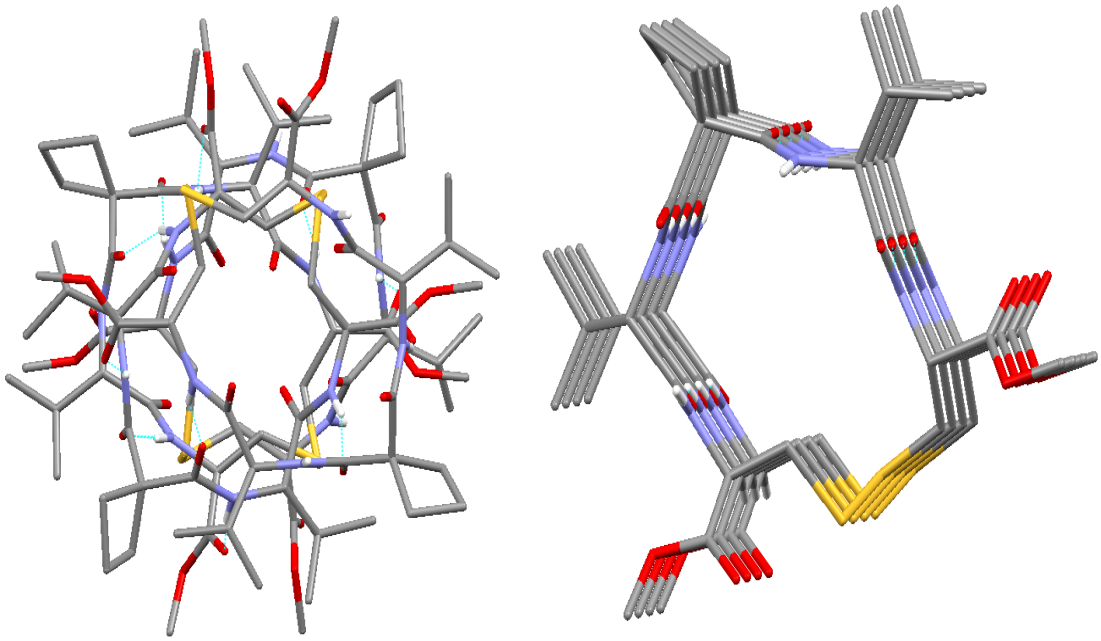
Figure S13: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C, TMS) spectrum of the cyclic peptide 1 with co-crystallized acetonitrile molecule (**1.CH_3CN)** (1:1).



ESI Fig. S1 Schematic representation of cyclic peptide **1**.

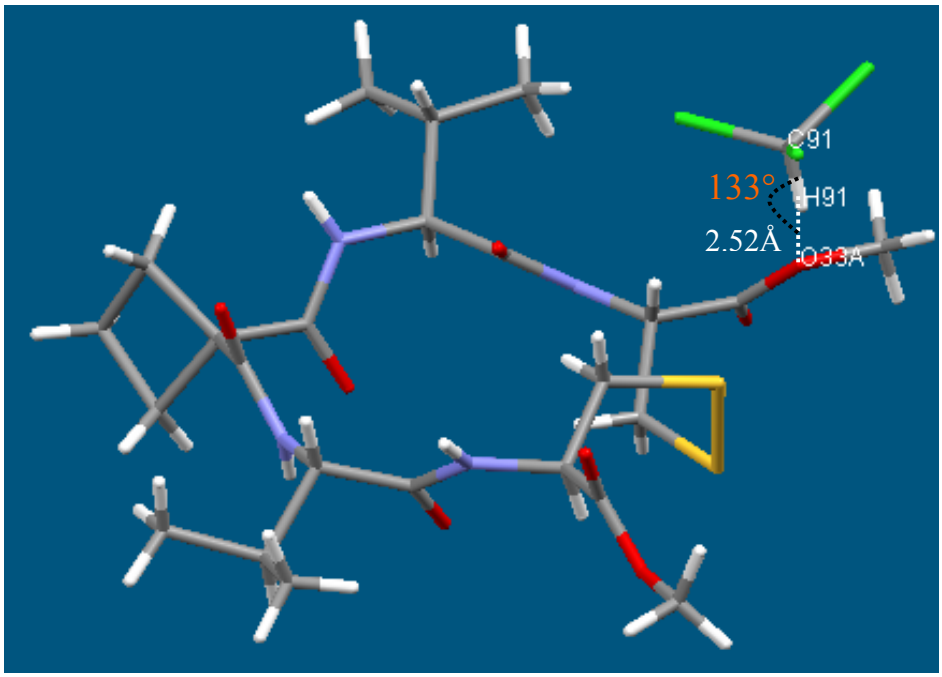


ESI Fig. S2 ORTEP diagram with atomic numbering scheme of the cyclic peptide **1**. Ellipsoids are at 30 % probability.

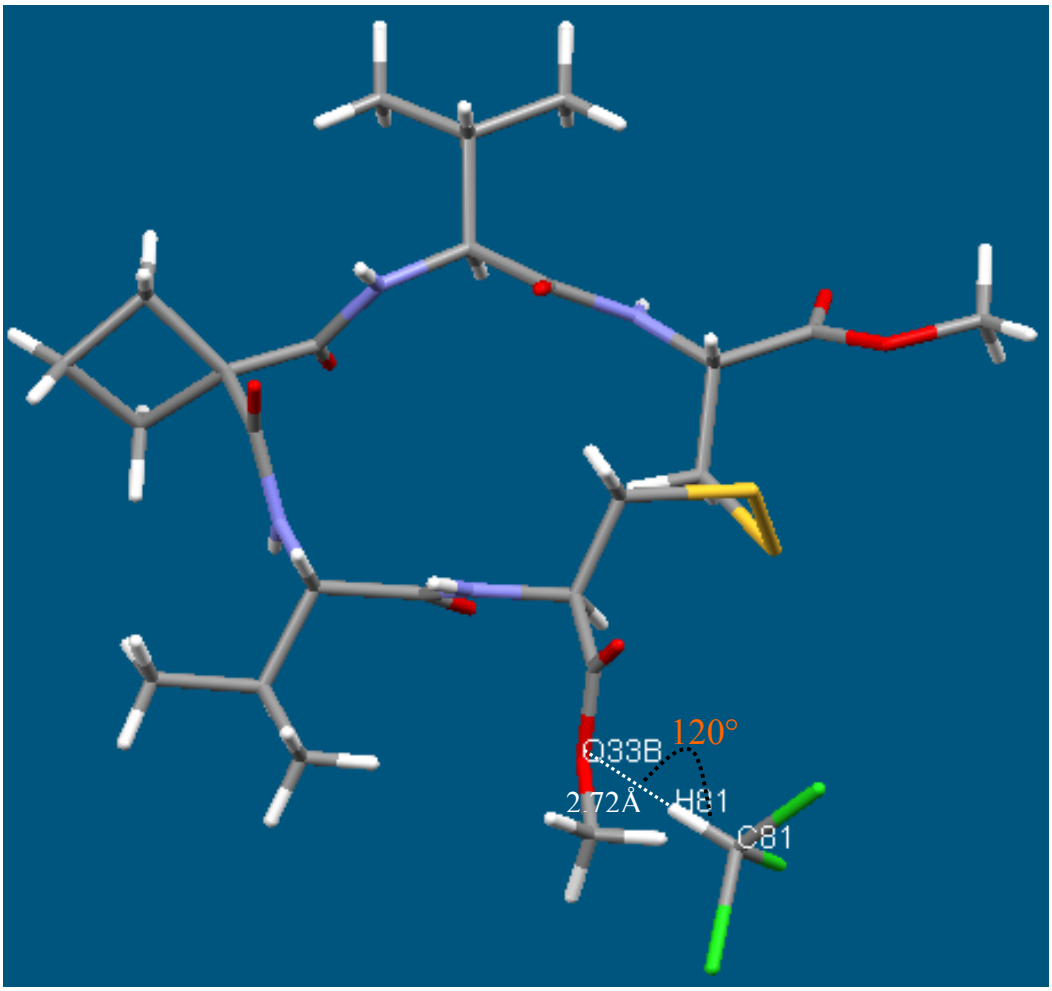


(a) (b)

ESI Fig. S3 Top view of the one-dimensional array formed by the (a) cyclic peptide **1.CHCl$_3$** and the (b) cyclic peptide **1** involving intermolecular hydrogen bonding. In case of **1.CHCl$_3$** the cyclic peptide rings are mutually staggered with each other while in the cyclic peptide **1**, they are eclipsed. Hydrogen atoms, which are not involved in hydrogen bonding, are omitted for clarity.



ESI Fig. S4 The closest contact of one C–H group of $CHCl_3$ molecule to the cyclic peptide **1.$CHCl_3$** is to O33A with a H91····O33A distance of 2.52 Å and C91–H91···O33A angle of 133°.



ESI Fig. S5 The closest contact of one C–H group of $CHCl_3$ molecule to the cyclic peptide **1.$CHCl_3$** is to O33B with a H81····O33B distance of 2.72 Å and C81–H81···O33B angle of 120°.