

Self-assembled hydrophobic Ala-Aib peptide encapsulating Curcumin: a convenient system for water insoluble drugs.

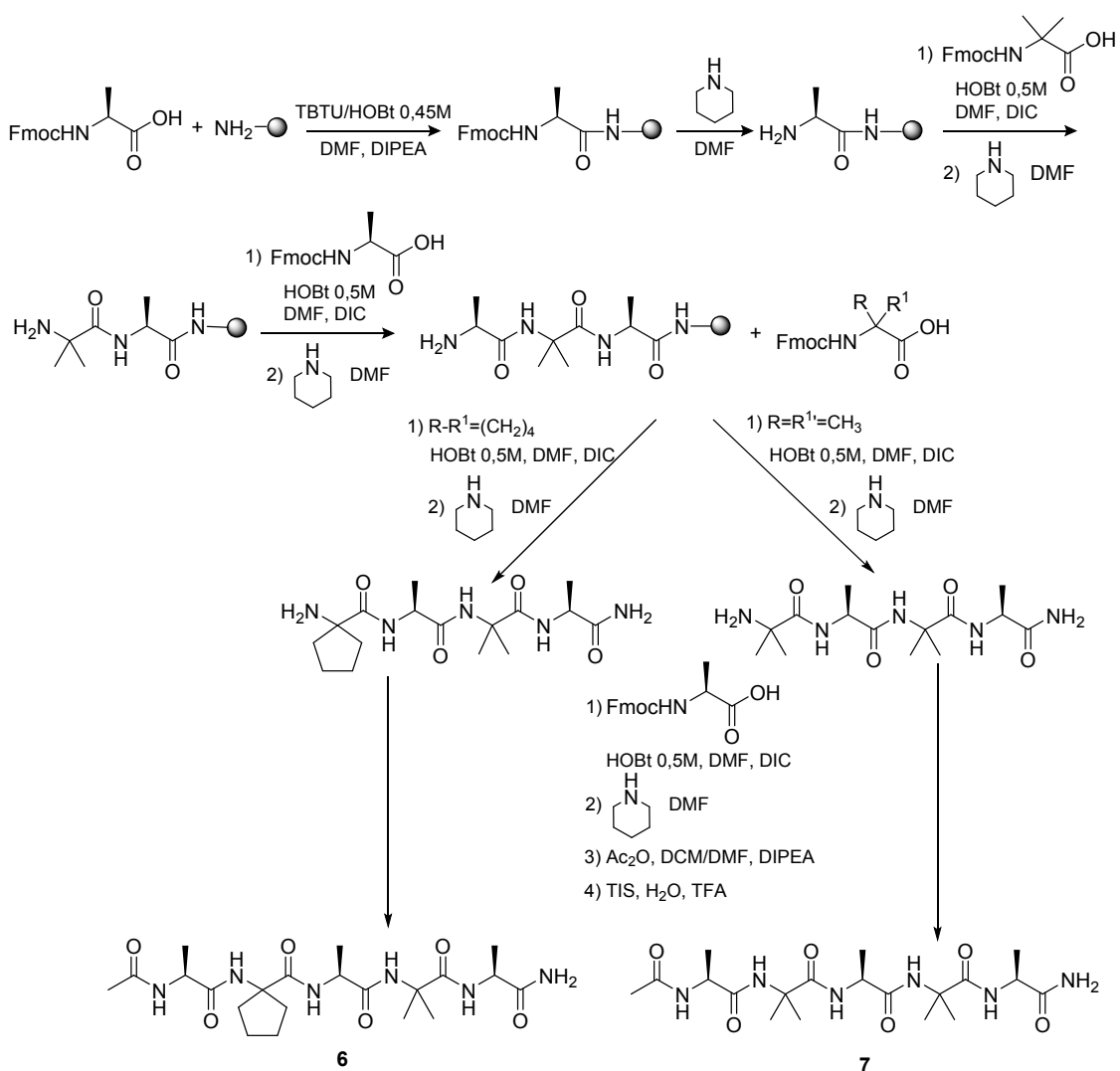
Silvia Locarno^{a}, Simona Argentiere^b, Alessandro Ruffoni^c, Daniela Maggioni^d, Raffaella Soave^e, Raffaella Bucci^a, Emanuela Erba^a, Cristina Lenardi^b, Maria Luisa Gelmi^a, Francesca Clerici^{a*}*

^a Department of Pharmaceutical Sciences, General and Organic Chemistry Section "A. Marchesini", University of Milan, Via Venezian 21, 20133 Milano Italy; ^b CIMAINA, Interdisciplinary Center for Nanostructured materials and Interfaces, Department of Physics, Via Celoria 16, 20133 Milano Italy, ^c School of Chemistry, University of Manchester, Manchester, M13 9PL, UK; ^d Department of Chemistry, Università degli Studi di Milano, Via Golgi 19, 20133 Milano Italy; ^e Institute of Chemical Sciences and Technologies "Giulio Natta", Italian National Research Council, CNR-SCITEC, Via Golgi 19, 20133 Milano.

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General Synthetic Procedures: Solid Phase Peptide Synthesis



Scheme S11. Synthetic scheme of compounds **6** and **7**.

Resin swelling: Fmoc-Rink Amide MBHA resin (100-200 mesh, 0.15 mmol) is swelled in a mixture of DCM (6.5 ml) and NMP (1 ml) for 30 minutes before use.

Fmoc-Deprotection: Fmoc-protected peptidyl resin is treated with 20% (v/v) piperidine in DMF for 5 minutes. Then, the resin is drained and the treatment is repeated for 15 minutes. The resin is finally washed six times with DMF.

Coupling Reaction: Method A: the Fmoc-protected amino acid (3 eq) is dissolved in 0.45M solution of HOBt (5 eq), HBTU (5 eq) and DIPEA (8 eq) in DMF. The peptidyl resin is treated with the mixture for 1h, drained and washed six times with DMF. This method applies for the first amino acid (Ala) and the second one (Aib).

Method B: the Fmoc-protected amino acid (3 eq) is dissolved in 0.5M solution of HOBt (5 eq) and DIC (5 eq) in DMF and stirred for 20 minutes. The peptidyl resin is treated with the mixture for 1 hour, drained and washed six times with DMF and six times with DCM. This method applies for the third amino acid (Ala), the fourth (Ac5C, or Aib) and the fifth (Ala).

Acetylation: peptidyl resin is treated with a mixture of DCM (1 ml), DMF (1 ml), acetic anhydride (0.1 ml) and DIPEA (0.2 ml) for 30 minutes. Then, the resin is drained and the treatment is repeated for another 30 minutes. The resin is finally washed six times with DMF.

Cleavage: Peptidyl resin is washed six times with DCM and dried with ether before starting the cleavage. Then, the resin is treated with a mixture (2 ml) of TFA/TIS/water (92:4.5:3.5) for 2 hours under magnetic stirring. Subsequently, the resin is drained and washed with methanol. The filtrate is concentrated in vacuum and extruded in 40 ml of cold ether. A white fluffy precipitated is formed and collected by centrifugation (6000 rpm, 5 min). The supernatant is decanted, the residue is resuspended in 20 ml of ether and centrifuged again. This is repeated twice. The residue is finally purified by preparative HPLC according to the following method and freeze-dried.

Time (min)	% A	% B	Flow (ml/min)	Gradient
0	100	0	20	linear
1	100	0	20	linear
20	50	50	20	linear
28	0	100	20	linear
29	0	100	20	linear
33	100	0	20	linear
34	100	0	0	linear

- phase A: H₂O/CH₃CN/TFA (95:5:0.1)
- phase B: CH₃CN/H₂O/TFA (95:5:0.1)

Peptide purity was measured by reverse phase HPLC according to the method described below:

Time (min)	% A	% B	Flow (ml/min)
0	100	0	0
1	100	0	1
18	50	50	1
22	0	100	1
23	0	100	1
25	100	0	1
31	100	0	0

- phase A: H₂O/CH₃CN/TFA 97:3:0.1
- phase B: H₂O/CH₃CN/TFA 30:70:0.1

Peptide 6: White solid, HPLC 97%, R_f: 17.78'. mp: 129.8°C. [α]_D: -50 (H₂O). IR (KBr) 3430(NH), 1658, 1534 (CO) cm⁻¹. ¹H NMR (D₂O+H₂O, 500 MHz) δ 1.30 (6H, d, J=7.4 Hz, CH₃ Ala₁,Ala₃), 1.35 (3H, d, J=7.4 Hz, CH₃ Ala₅), 1.41 (6H, s, CH₃ Aib), 1.67-1.71 (4H, m, CH₂), 1.82-1.86 (2H, m, CH₂), 1.97 (3H, s, CH₃), 2.04-2.10 (2H, m, CH₂), 4.10 (1H, m, CH_{Ala3}), 4.16 (1H, m, CH_{Ala1}), 4.20 (1H, m, CH_{Ala5}), 6.99 (1H, s, CONH), 7.33 (1H, s, CONH), 7.73 (1H, d, J=6.5 Hz, NH_{Ala5}), 7.77 (1H, d, J=5.6 Hz, NH_{Ala3}), 7.90 (1H, s, NH_{Aib}), 8.18 (1H, d, J=5.1 Hz, NH_{Ala1}), 8.43 (1H, s, NH_{Ac5C}). ¹³C NMR (D₂O, 500 MHz) δ 15.78 (CH₃ Ala₃), 15.95 (CH₃ Ala₁), 16.39 (CH₃ Ala₅), 21.53 (CH₃), 23.86, 23.93 (CH₂), 24.30 (CH₃ Aib), 36.03, 36.64 (CH₂), 49.88 (CH_{Ala5}), 50.23 (CH_{Ala1}), 50.78 (CH_{Ala3}), 56.62 (C_{Aib}), 66.66 (C_{Ac5C}), 174.22 (CO_{Me}), 175.10 (CO_{Ala3}), 175.61 (CO_{Ala1}), 176.72 (CO_{Ac5C}), 177.02 (CO_{Aib}), 178.17 (CO_{Ala5}). ESI-MS (m/z): 491.4 [M+Na]⁺

Peptide 7: White solid, HPLC 95%, R_f: 14.12'. mp: 73.2°C. [α]_D: +7.6 (CH₃OH). IR (KBr) 3429, 3276 (NH), 1683, 1658, 1543 (CO) cm⁻¹. ¹H NMR (D₂O+H₂O, 500 MHz) δ 1.30 (6H, d, J=7.4 Hz, CH₃ Ala₁,Ala₃), 1.33 (3H, d, J=7.5 Hz, CH₃ Ala₅), 1.37 (6H, d, J=5.6 Hz, CH₃ Aib₂), 1.40 (6H, s, CH₃ Aib₄), 1.95 (3H, s, CH₃), 4.08 (1H, m, CH_{Ala3}), 4.13 (1H, m, CH_{Ala1}), 4.17 (1H, m, CH_{Ala5}), 6.98 (1H, s, CONH), 7.33 (1H, s, CONH), 7.73 (1H, d, J=6.6 Hz, NH_{Ala5}), 7.75 (1H, d, J=5.7 Hz, NH_{Ala3}), 7.90 (1H, s, NH_{Aib4}), 8.18 (1H, d, J=5.4 Hz, NH_{Ala1}), 8.35 (1H, s, NH_{Aib2}). ¹³C NMR (D₂O, 500 MHz) δ 16.17 (CH₃ Ala₃), 16.51 (CH₃ Ala₁), 16.78 (CH₃ Ala₅), 21.96 (CH₃), 24.24, 24.35 (CH₃ Aib₄), 24.53, 24.64 (CH₃ Aib₂), 50.24 (CH_{Ala5}), 50.57 (CH_{Ala1}), 51.05 (CH_{Ala3}), 56.81 (C_{Aib4}), 57.01 (C_{Aib2}), 174.56 (CO_{Me}), 175.37 (CO_{Ala3}), 175.45 (CO_{Ala1}), 177.41 (CO_{Aib2}), 177.67 (CO_{Aib4}), 178.55 (CO_{Ala5}). ESI-MS (m/z): 466.4 [M+Na]⁺

NOESY experiments in CD_3OH and in D_2O/H_2O for peptide **6**

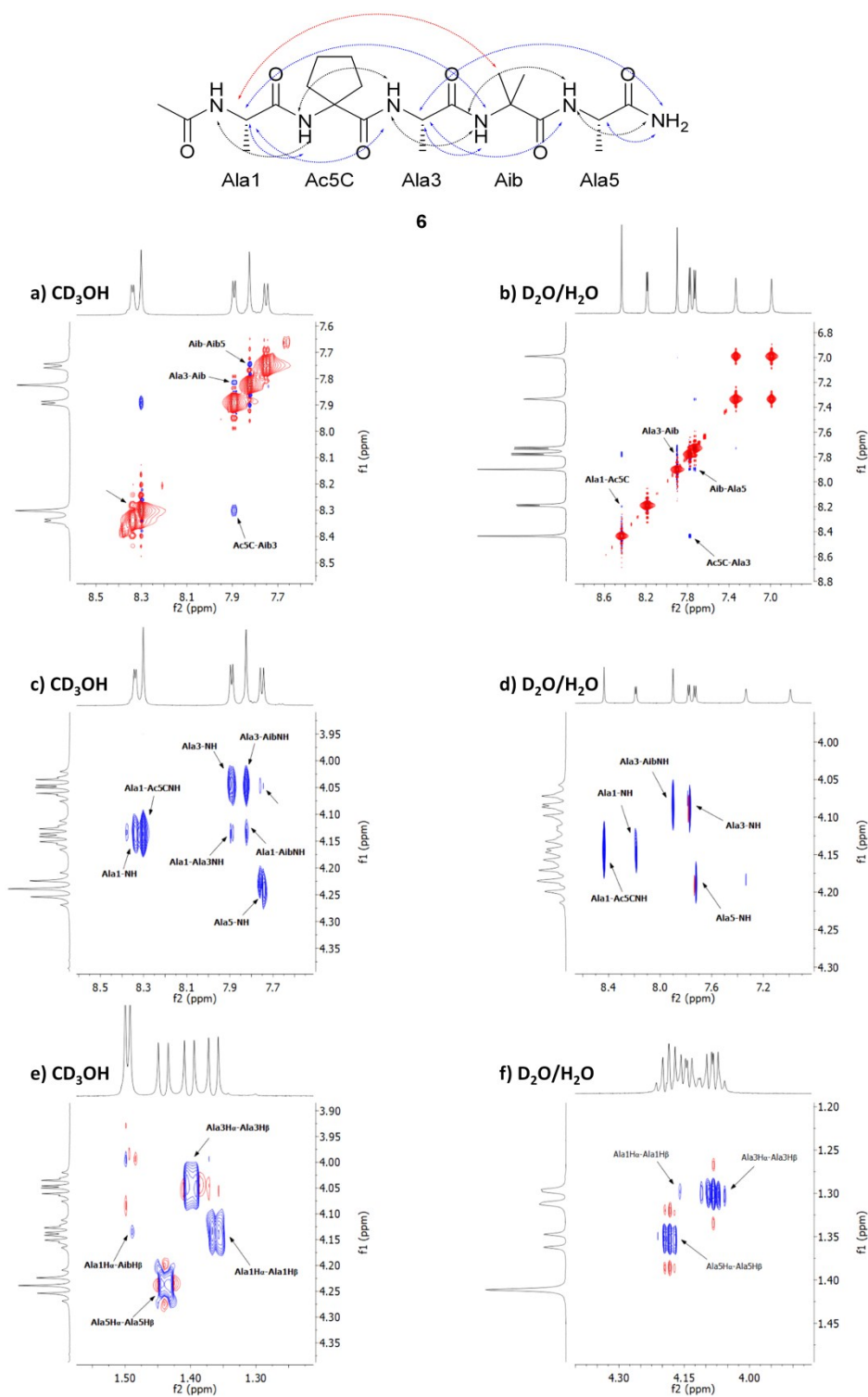


Figure S11. NOESY experiments analysis for **6** of: NH_i, NH_{i+1} NOE cross peaks region in a) CD_3OH and in b) D_2O/H_2O ; $C^\alpha H_i-NH$ region in c) CD_3OH and in d) D_2O/H_2O ; $C^\alpha H-C^\beta$ region in e) CD_3OH and in f) D_2O/H_2O .

Temperature dependence of amide chemical shift ($\Delta\delta/\Delta T$)

Confirmation of the helical conformation of peptides **6** and **7** was given estimating the temperature dependence of amide chemical shift ($\Delta\delta/\Delta T$, 273–300 K). In CD_3OH , $\Delta\delta/\Delta T$ NH values of Ala3, Aib4, Ala5 are around 4.0 ppb/K (Fig. SI2a,b). The lowest value of $\Delta\delta/\Delta T$ is attributed to one NH of CONH_2 , this means that the most stable part is amide at C-terminus. On the other hand, the other NHs (Ala1, Aib2 or Ac5c at N-terminus and the other NH proton of the primary amide) are not involved in any intramolecular H-bond ($\Delta\delta/\Delta T$ 6–8 ppb/K).

A different behaviour was found in $\text{D}_2\text{O}/\text{H}_2\text{O}$ (Fig. SI2c,d) where higher $\Delta\delta/\Delta T$ values were detected for all NHs. Moreover it is interesting to notice that in water the differences between the two primary amide protons $\Delta\delta/\Delta T$ values are smaller than in methanol. Especially one of the two primary amides of peptides **6** and **7** with a values of 4 ppb/K seems to be involved in a H-bond but it's not possible to determinate the inter- or intra-molecular nature of that bond.

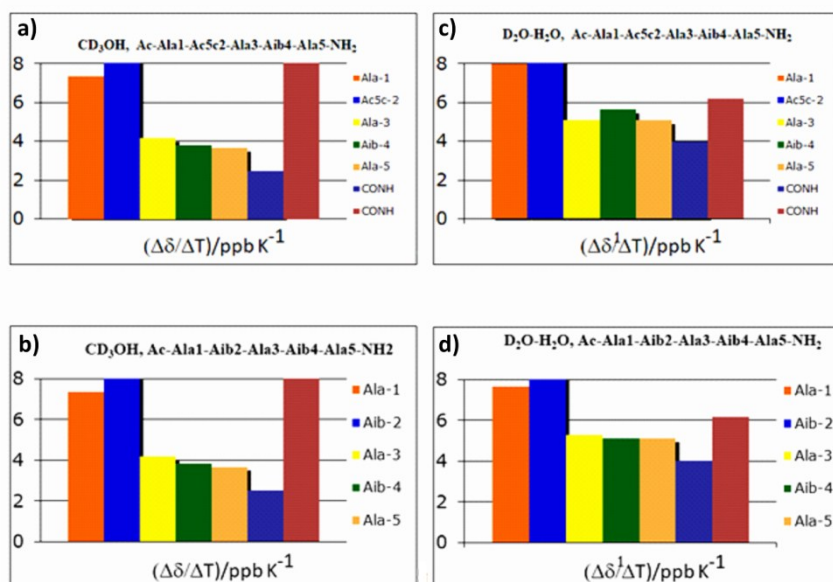


Figure SI2. Temperature dependence of amide chemical shift ($\Delta\delta/\Delta T$ 273–300 K) of peptides **6** and **7** in a,b) CD_3OH and c,d) $\text{D}_2\text{O}/\text{H}_2\text{O}$.

^{13}C Magnetic Nonequivalence

The evaluation of the ^{13}C -Magnetic Nonequivalence (MNE) of the signals related to the diastereotopic methyl groups of Aib4 was performed by HSQC experiments in CD_3CN , CD_3OD and D_2O as shown in table SI1. ¹

Solvent	Peptide 6		Peptide 7	
	Aib4 δ	MNE δ	Aib4 δ	MNE δ
CD_3CN	26.384-22.894	3.49	26.79-23.31	3.48

CD₃OD	25.237-23.112	1.125	25.65-23.50	2.15
D₂O	24.300-23.943	0.36	24.65-24.24	0.41

Table S11. Chemical shift in ppm of Aib-4 in peptides **6** and **7** and MNE.

Circular dichroism

Circular dichroism (CD) spectra were acquired in 2,2,2-trifluoroethanol (TFE), MeOH and water at 25°C with a peptides concentration of 0.1 mM (Fig. S13).

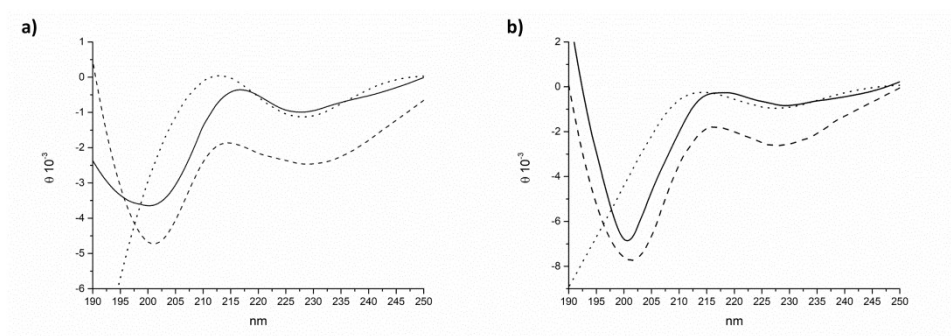


Figure S13. CD spectra (0.1 mM) of peptides a) **6** and b) **7** in MeOH (straight), TFE (dash), and water (dot).

IR spectroscopy

peptide	α -helix IR-band ($1656 \pm 2 \text{ cm}^{-1}$)	Random coil IR-band ($1648 \pm 2 \text{ cm}^{-1}$)
6	1657	1648
7	1654	1648

Table S12. IR signals after deconvolution of N-H stretching vibration region of the amides. ²

DLS spectra of peptide 7 in water and PBS

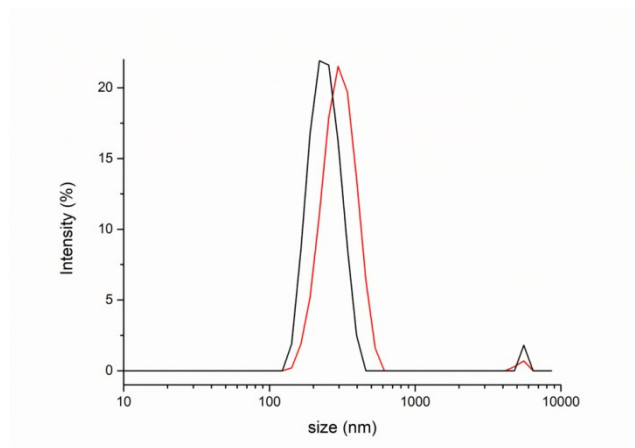


Figure S14. Size distribution by intensity of peptide **7** in water (black) and in PBS with 0.34% v/v of DMSO (red). Mean results are given for three different measurements.

Peptide aggregates stability in the two approaches

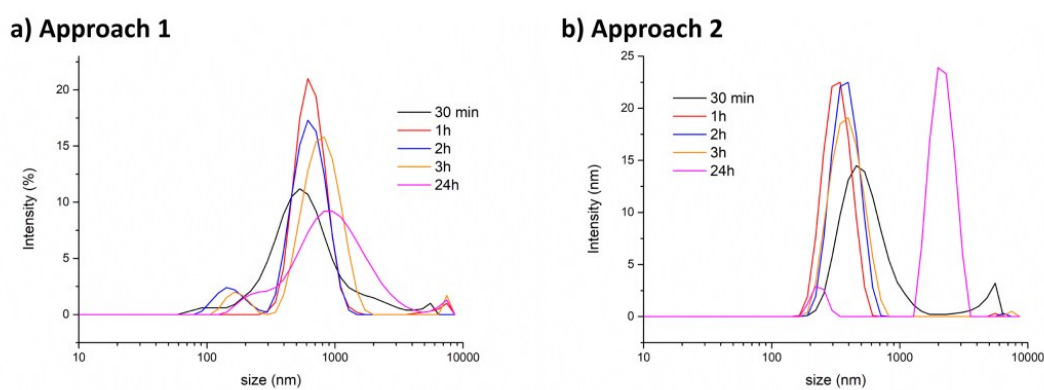


Figure S15. Stability test of peptide aggregates prepared according a) approach 1 and b) approach 2.

Dependence of absorption and emission of curcumin from concentration and pH

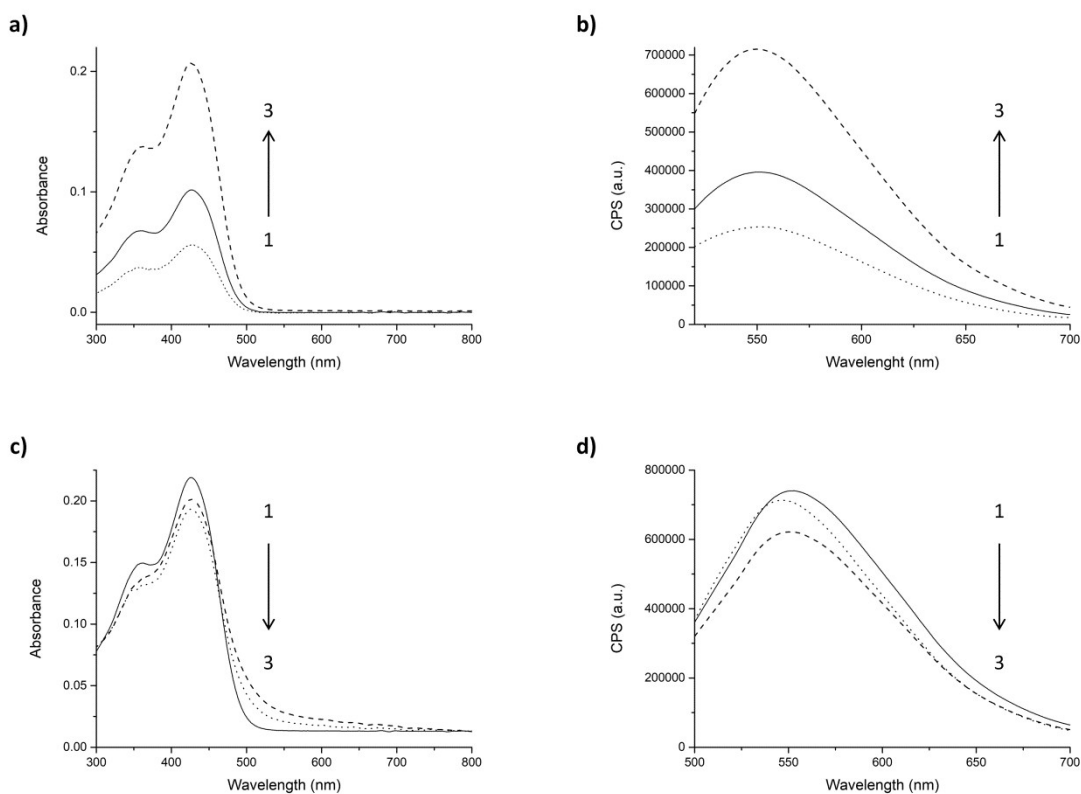


Figure S16. a) Absorption and b) fluorescence spectra at different concentrations of curcumin: (1) 2.4 μM (dot), (2) 4.8 μM (straight), (3) 9.6 μM (dash) in water with 0.34% v/v of DMSO. c) Absorption and d) fluorescence spectra of curcumin at decreasing pH in water with 0.34% v/v of DMSO.

Optical properties of curcumin after addition of TW20

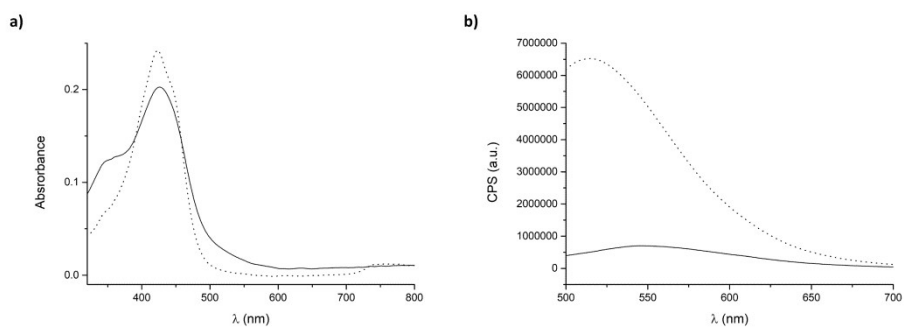


Figure S17. a) Absorption and b) fluorescence spectra of curcumin (9.6 μM) in PBS (straight) and in presence of 0.005% of TW20 (dot).

Calibration curve of curcumin in CHCl_3

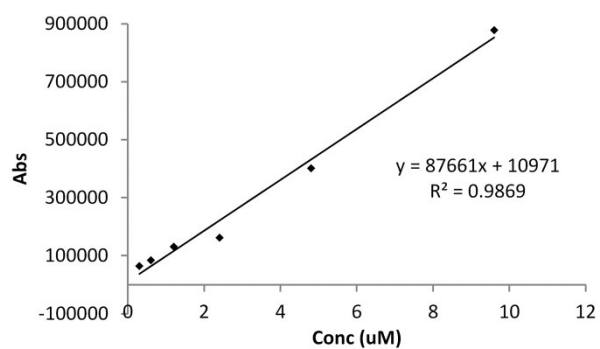
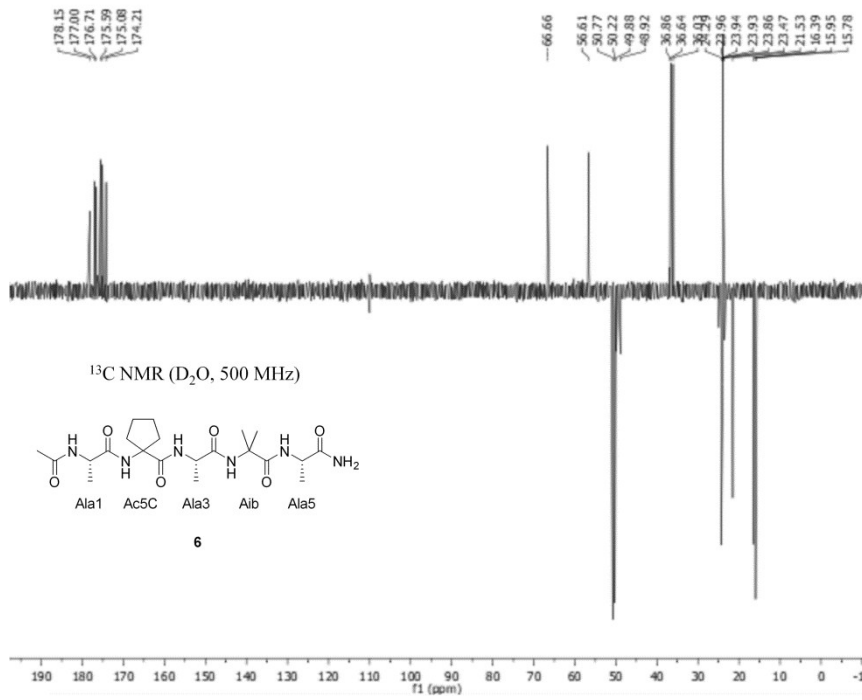
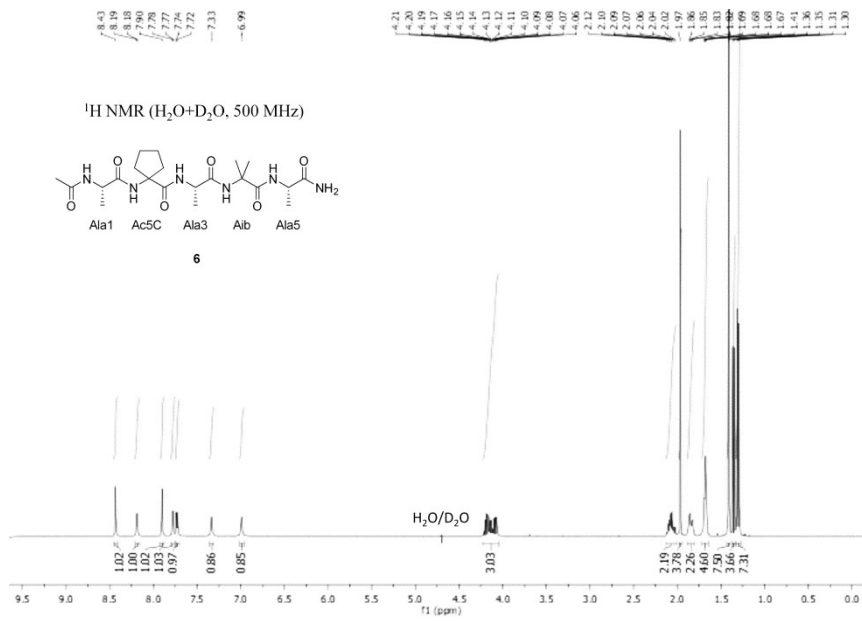
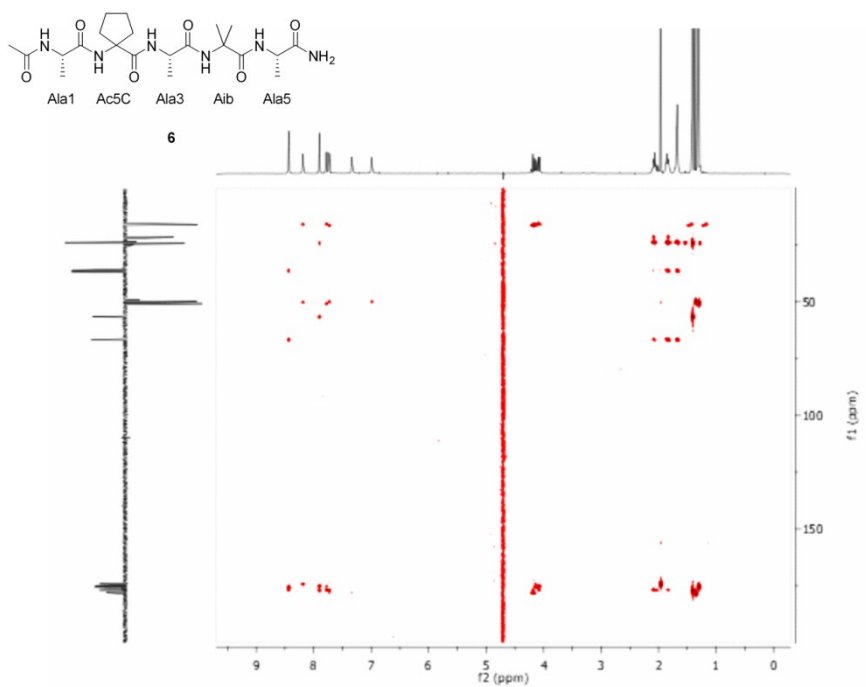


Figure S18. Calibration curve of curcumin in CHCl_3 .

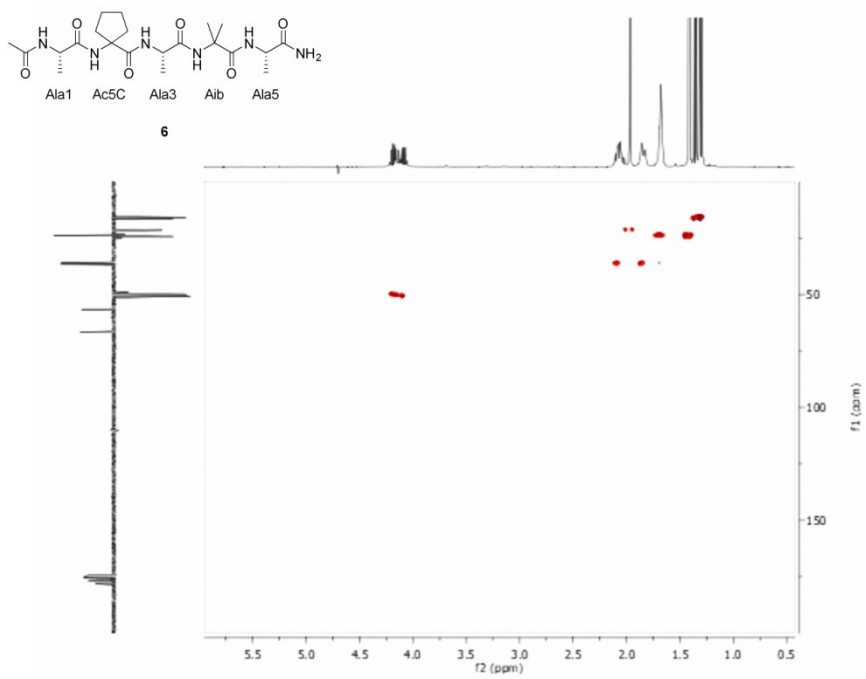
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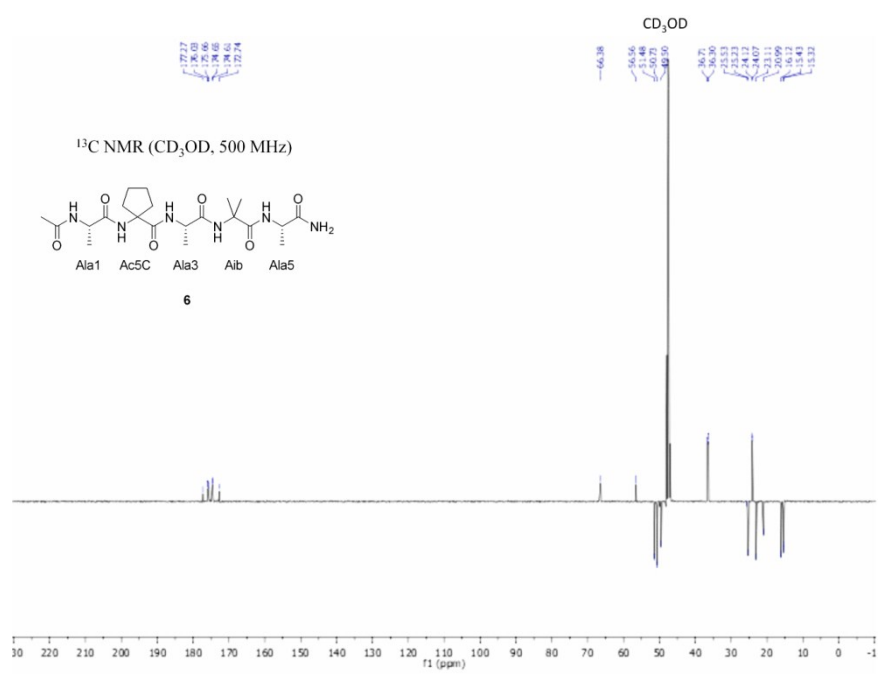
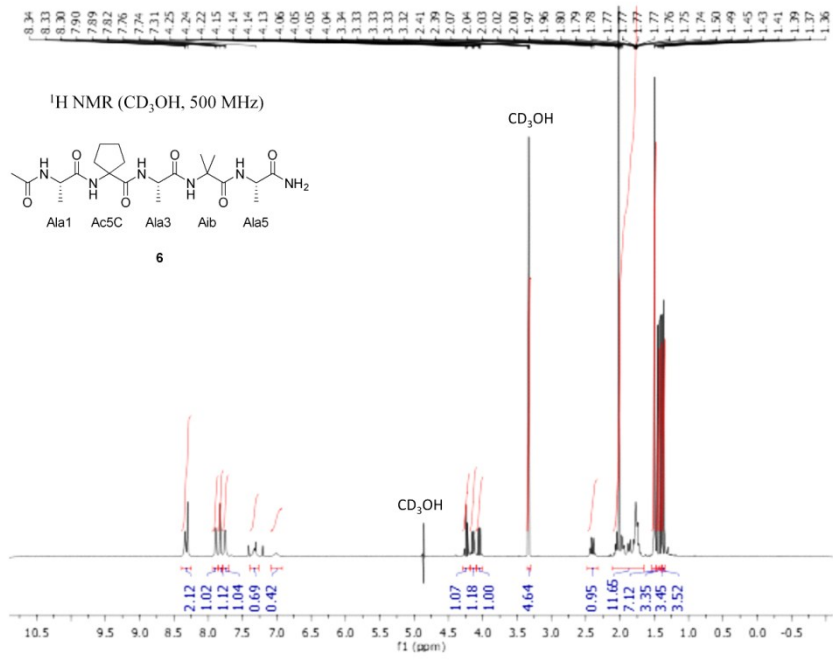


HMBC (H₂O+D₂O, 500 MHz)

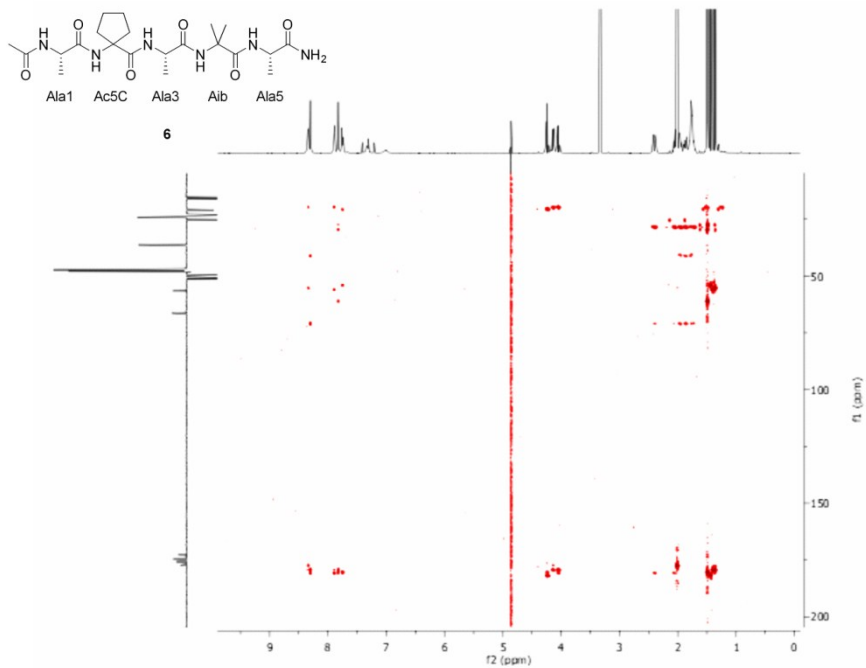


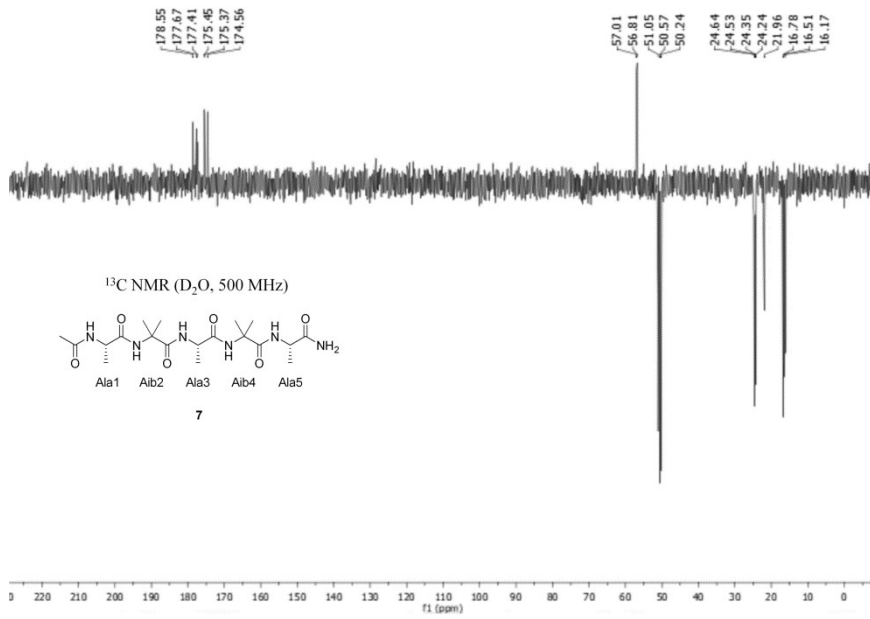
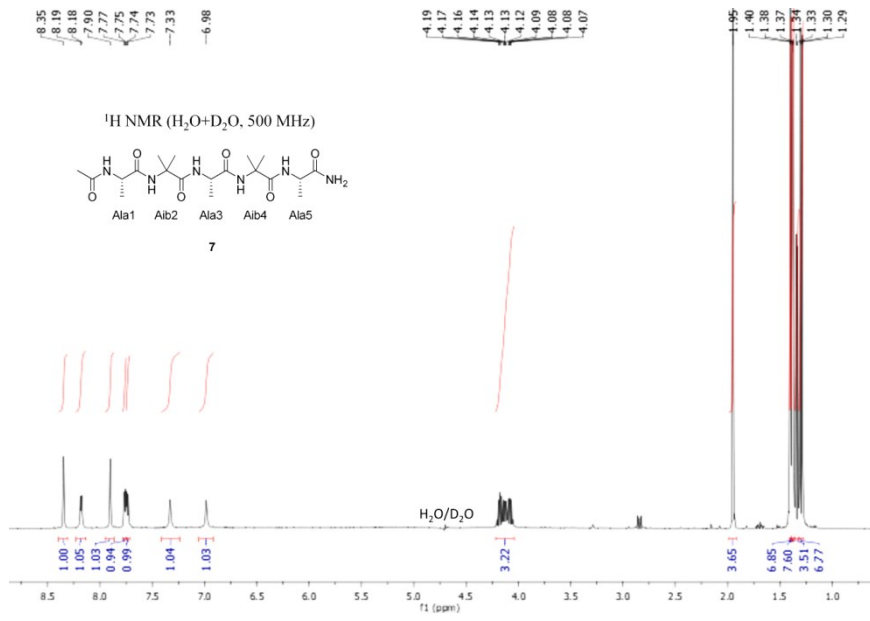
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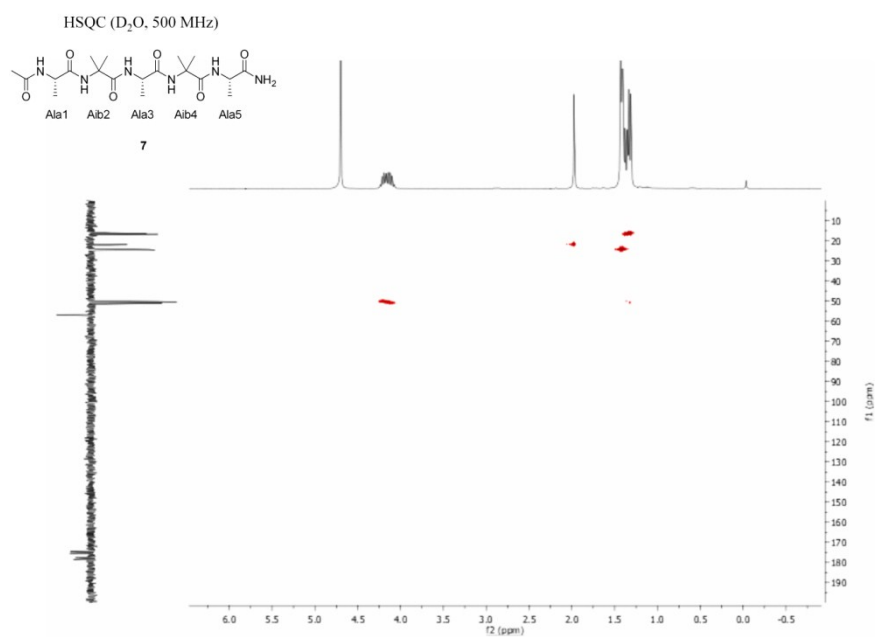
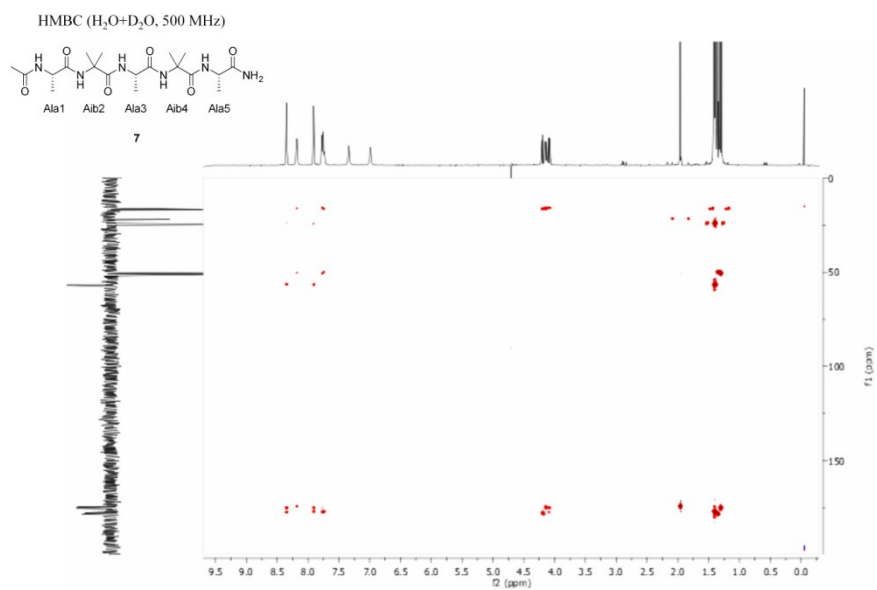


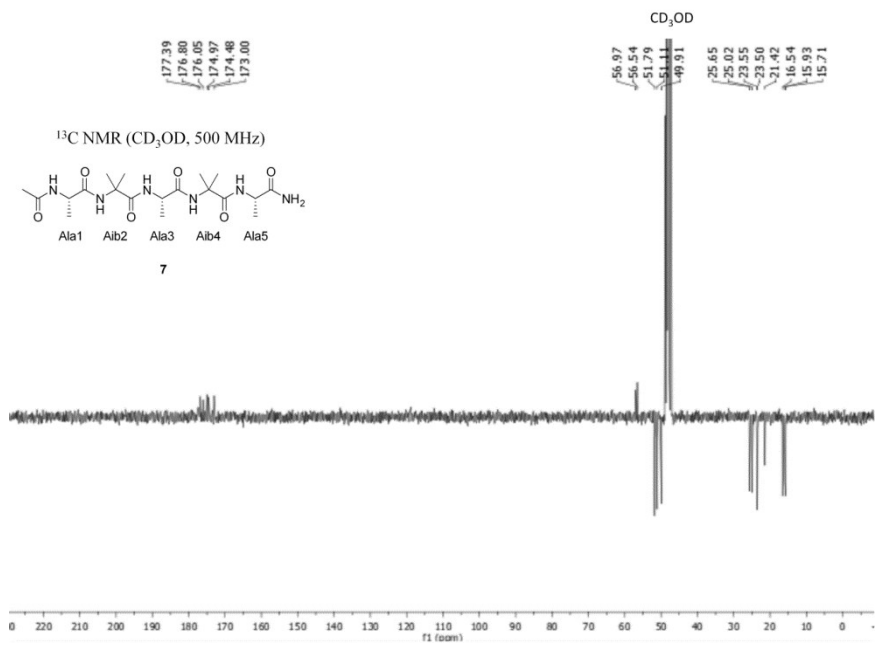
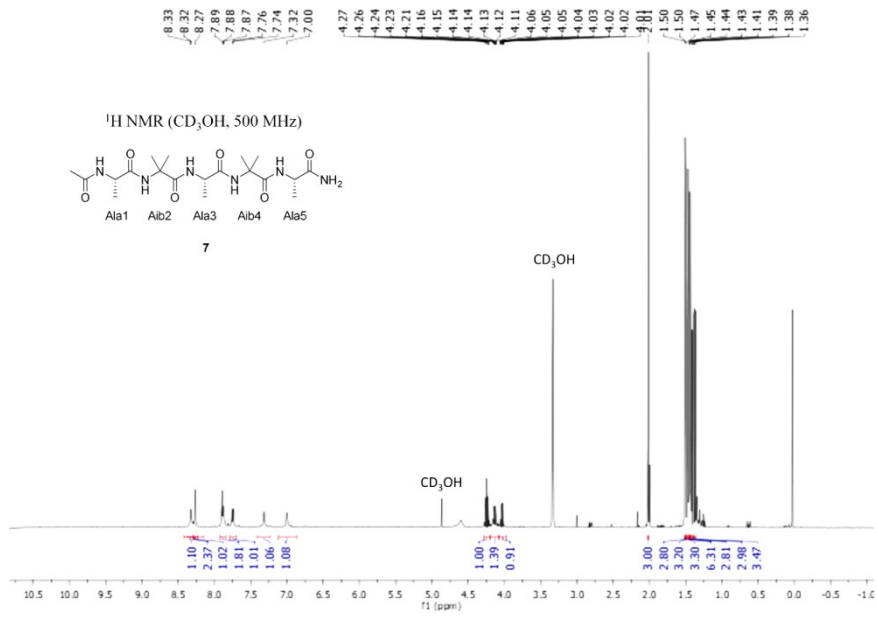


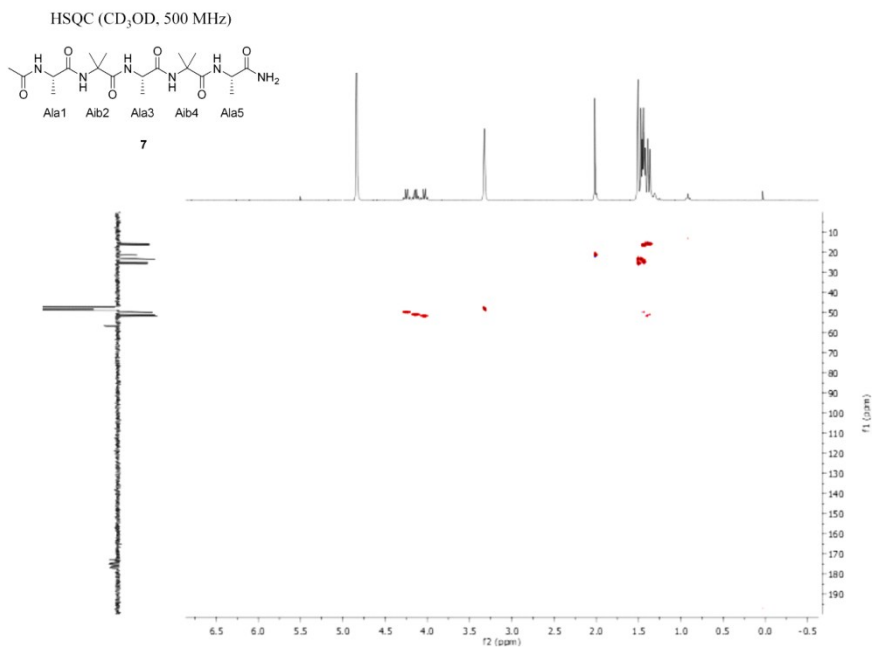
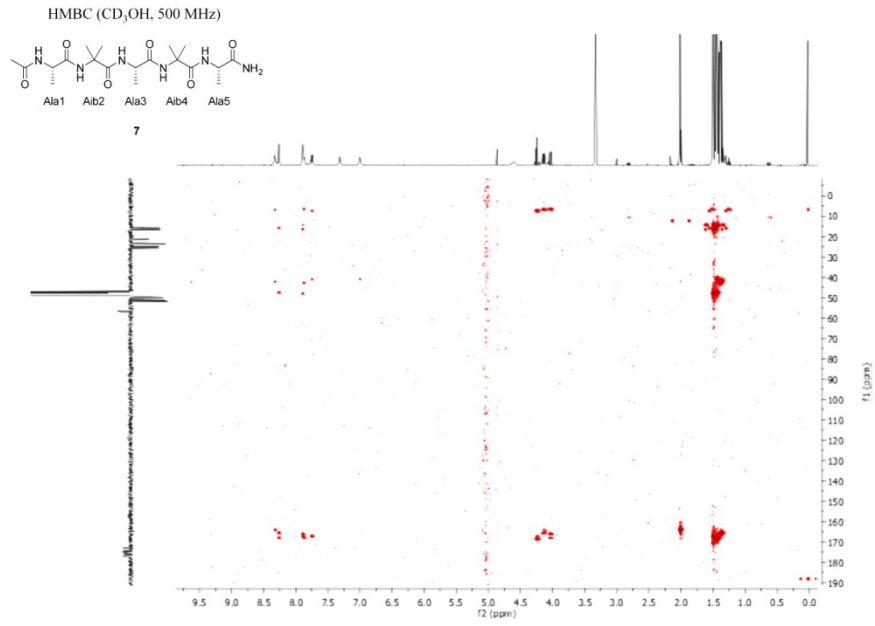
HMBC (CD₃OH, 500 MHz)











Abbreviations

aa, amino acid; Ac5C, aminocyclopentanecarboxylic acid; Aib, α -aminoisobutyric acid; Ala, Alanine; CD, circular dichroism; DCM, dichloromethane; DIC, N,N'-diisopropylcarbodiimide; DIPEA, N,N-Diisopropylethylamine; DLS, Dynamic Light Scattering; DMF, N,N-dimethylformamide; DMSO, dimethyl sulfoxide; Fmoc, fluorenylmethyloxycarbonyl; HBTU, hexafluorophosphate benzotriazole tetramethyl uranium; HOBt, 1-hydroxybenzotriazole; HPLC, high-performance liquid chromatography; IR, infrared spectroscopy; MBHA, 4-methylbenzhydrylamine; MNE, magnetic non-equivalence; NMP, N-methyl-2-pyrrolidone; NMR, nuclear magnetic resonance; NOESY, nuclear overhauser effect spectroscopy; PBS, phosphate buffer saline; TFA, trifluoroacetic acid; TFE, trifluoroethanol; TIS, triisopropylsilane; TW20, Tween 20.

References

- (1) Longo, E.; Moretto, A.; Formaggio, F.; Toniolo, C. *Chirality* **2011**, *23*, 756-760.
- (2) Kong, J.; Yu, S. *Acta Biochimica et Biophysica Sinica* **2007**, *39*, 549-559.

CIF file with X-ray data for 7

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#----- SUBMISSION DETAILS -----#

Name and address of author for correspondence

_publ_contact_author_name 'Raffaella Soave'
_publ_contact_author_address ;Istituto di Scienze e Tecnologie Chimiche "Giulio Natta"
CNR Via C. Golgi, 19 I-20133 Milano Italy;
_publ_contact_author_email 'raffaella.soave@istm.cnr.it'
_publ_contact_author_fax '+39(2)503142300'
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_publ_author_name
_publ_author_address
'Locarno, Silvia'

;

Department of Pharmaceutical Sciences, General and Organic
Chemistry Section A. Marchesini
Universit`a degli Studi di Milano
Via Venezian 21
I-20133 Milano
Italy

;

'Argentiere, Simona'

;

CIMAINA, Interdisciplinary Center for Nanostructured materials and Interfaces
Department of Physics
Via Celoria 16
20133 Milano
Italy

;

'Ruffoni, Alessandro'

;

School of Chemistry
University of Manchester
Manchester, M13 9PL, UK

;

'Maggioni, Daniela'

;

Department of Chemistry
Universit`a degli Studi di Milano
Via Golgi 19
I-20133 Milano
Italy

;

'Soave, Raffaella'

;

Istituto di Scienze e Tecnologie Chimiche "Giulio Natta"
Consiglio Nazionale delle Ricerche
Via C. Golgi, 19
I-20133 Milano
Italy

;

'Bucci, Raffaella'

;

Department of Pharmaceutical Sciences, General and Organic
Chemistry Section A. Marchesini
Universit`a degli Studi di Milano
Via Venezian 21
I-20133 Milano
Italy

;

'Erba, Emanuela'

;

Department of Pharmaceutical Sciences, General and Organic
Chemistry Section A. Marchesini
Universit`a degli Studi di Milano
Via Venezian 21
I-20133 Milano
Italy

;

'Lenardi, Cristina'

;

CIMAINA, Interdisciplinary Center for Nanostructured materials and Interfaces
Department of Physics
Via Celoria 16
20133 Milano
Italy

;
'Gelmi, Maria Luisa'
;
Department of Pharmaceutical Sciences, General and Organic
Chemistry Section A. Marchesini
Universit`a degli Studi di Milano
Via Venezian 21
I-20133 Milano
Italy

;
'Clerici, Francesca'
;
Department of Pharmaceutical Sciences, General and Organic
Chemistry Section A. Marchesini
Universit`a degli Studi di Milano
Via Venezian 21
I-20133 Milano
Italy

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_diffrn_radiation_monochromator graphite
_diffrn_measurement_device_type 'Bruker AXS APEX CCD area-detector'
_diffrn_measurement_method '\w-scan'
_diffrn_detector_area_resol_mean ?
_diffrn_reflns_number 22941
_diffrn_reflns_av_unetl/netl 0.0476
_diffrn_reflns_av_R_equivalents 0.0336
_diffrn_reflns_limit_h_min -14
_diffrn_reflns_limit_h_max 14
_diffrn_reflns_limit_k_min -16
_diffrn_reflns_limit_k_max 16
_diffrn_reflns_limit_l_min -16
_diffrn_reflns_limit_l_max 16
_diffrn_reflns_theta_min 1.844
_diffrn_reflns_theta_max 29.997
_diffrn_reflns_theta_full 25.242
_diffrn_measured_fraction_theta_max 1.000
_diffrn_measured_fraction_theta_full 1.000
_diffrn_reflns_Laue_measured_fraction_max 1.000
_diffrn_reflns_Laue_measured_fraction_full 1.000
_diffrn_reflns_point_group_measured_fraction_max 0.999
_diffrn_reflns_point_group_measured_fraction_full 1.000
_reflns_number_total 7475
_reflns_number_gt 4289
_reflns_threshold_expression 'I > 2\sigma(I)'
_reflns_Friedel_coverage 0.910
_reflns_Friedel_fraction_max 0.999
_reflns_Friedel_fraction_full 1.000

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_reflns_special_details
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Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

_reflns_Friedel_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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_computing_cell_refinement 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'
_computing_data_reduction 'SAINT+ (Bruker AXS, Madison (USA) 1994-1996)'
_computing_structure_solution 'SHELXS97 (Sheldrick, 1997)'
_computing_structure_refinement 'SHELXL-2014/4 (Sheldrick, 2014)'
_computing_molecular_graphics 'Diamond v3.2i (Brandenburg, 2012)'
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_refine_ls_structure_factor_coef Fsqd
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_refine_ls_weighting_details
'w=1/[\s^2^(Fo^2^)+(0.0622P)^2^] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary ?
_atom_sites_solution_secondary ?
_atom_sites_solution_hydrogens mixed
_refine_ls_hydrogen_treatment mixed
_refine_ls_extinction_method SHELXL
_refine_ls_extinction_coef 0.009(2)
_refine_ls_extinction_expression
'Fc^*=kFc[1+0.001xFc^2^/|s^3^/sin(2\q)]^-1/4^'
_refine_ls_abs_structure_details
;
Flack x determined using 1616 quotients [(I+)-(I-)]/[(I+)+(I-)]
(Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
;
_refine_ls_abs_structure_Flack -0.1(4)
_chemical_absolute_configuration syn
_refine_ls_number_reflns 7475
_refine_ls_number_parameters 319
_refine_ls_number_restraints 13
_refine_ls_R_factor_all 0.0931
_refine_ls_R_factor_gt 0.0417
_refine_ls_wR_factor_ref 0.1200
_refine_ls_wR_factor_gt 0.0976
_refine_ls_goodness_of_fit_ref 0.962
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_refine_ls_shift/su_max 0.027
_refine_ls_shift/su_mean 0.001

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O1 O 0.7612(2) 0.08422(18) 0.73189(17) 0.0524(5) Uani 1 1 d . . . . .
O2 O 0.9834(2) 0.2964(2) 0.95416(16) 0.0542(5) Uani 1 1 d . . . . .
O3 O 0.7128(2) 0.1055(2) 1.20373(17) 0.0590(6) Uani 1 1 d . . . . .
O4 O 1.0004(2) 0.2252(2) 1.45402(16) 0.0577(6) Uani 1 1 d . . . . .
O5 O 0.6807(2) 0.3791(2) 1.02669(18) 0.0592(6) Uani 1 1 d . . . . .
O6 O 0.5597(3) 0.2754(2) 0.5192(2) 0.0753(7) Uani 1 1 d . . . . .
N2 N 0.8947(2) 0.1888(2) 0.65262(18) 0.0409(5) Uani 1 1 d . . . . .
H1 H 0.9022 0.2134 0.5869 0.049 Uiso 1 1 calc R U . . .
N3 N 0.8044(2) 0.3474(2) 0.78350(18) 0.0420(5) Uani 1 1 d . . . . .
H2 H 0.7691 0.3450 0.7057 0.050 Uiso 1 1 calc R U . . .
N5 N 0.8777(3) 0.1732(2) 1.13675(18) 0.0452(6) Uani 1 1 d . . . . .
H3 H 0.8980 0.1952 1.0759 0.054 Uiso 1 1 calc R U . . .
N1 N 0.6638(2) 0.1589(2) 0.42462(19) 0.0451(5) Uani 1 1 d . . . . .
H4 H 0.6927 0.1514 0.3655 0.054 Uiso 1 1 calc R U . . .
N6 N 0.8794(3) 0.3464(2) 1.3034(2) 0.0532(6) Uani 1 1 d . . . . .
H5A H 0.8630 0.3944 1.3524 0.064 Uiso 1 1 calc R U . . .
H5B H 0.8478 0.3611 1.2271 0.064 Uiso 1 1 calc R U . . .
N4 N 0.6779(2) 0.2192(2) 0.91331(19) 0.0433(5) Uani 1 1 d . . . . .
H6 H 0.6912 0.1930 0.8505 0.052 Uiso 1 1 calc R U . . .
C1 C 0.7870(3) 0.1167(2) 0.6435(2) 0.0401(6) Uani 1 1 d . . . . .
C2 C 0.9281(3) 0.2925(2) 0.8442(2) 0.0416(6) Uani 1 1 d . . . . .
C3 C 0.7448(3) 0.1398(3) 1.1192(2) 0.0449(6) Uani 1 1 d . . . . .
C4 C 0.9527(3) 0.2505(2) 1.3452(2) 0.0434(6) Uani 1 1 d . . . . .
C5 C 0.6947(3) 0.3343(3) 0.9377(2) 0.0438(6) Uani 1 1 d . . . . .
C6 C 0.5977(4) 0.4651(3) 0.7591(3) 0.0633(9) Uani 1 1 d . . . . .
H6A H 0.6228 0.5213 0.7103 0.095 Uiso 1 1 calc R U . . .
H6B H 0.5466 0.5036 0.8022 0.095 Uiso 1 1 calc R U . . .
H6C H 0.5392 0.4053 0.7080 0.095 Uiso 1 1 calc R U . . .
C7 C 0.6379(3) 0.1364(3) 0.9895(2) 0.0487(7) Uani 1 1 d . . . . .
C8 C 0.7003(3) 0.0698(2) 0.5180(2) 0.0431(6) Uani 1 1 d . . . . .
H8 H 0.6116 0.0387 0.5208 0.052 Uiso 1 1 calc R U . . .
C9 C 0.9872(3) 0.1726(3) 1.2564(2) 0.0492(7) Uani 1 1 d . . . . .
H9 H 0.9979 0.0921 1.2873 0.059 Uiso 1 1 calc R U . . .
C10 C 1.0005(3) 0.2284(3) 0.7683(2) 0.0423(6) Uani 1 1 d . . . . .
C11 C 0.7302(3) 0.4104(2) 0.8481(2) 0.0441(6) Uani 1 1 d . . . . .
H11 H 0.7921 0.4739 0.8936 0.053 Uiso 1 1 calc R U . . .

```

C12 C 0.6458(5) 0.0116(3) 0.9429(3) 0.0733(11) Uani 1 1 d
 H12A H 0.5839 0.0059 0.8595 0.110 Uiso 1 1 calc R U . . .
 H12B H 0.6170 -0.0439 0.9900 0.110 Uiso 1 1 calc R U . . .
 H12C H 0.7413 -0.0048 0.9502 0.110 Uiso 1 1 calc R U . . .
 C13 C 0.4890(4) 0.1637(4) 0.9848(3) 0.0781(11) Uani 1 1 d
 H13A H 0.4885 0.2392 1.0202 0.117 Uiso 1 1 calc R U . . .
 H13B H 0.4605 0.1051 1.0291 0.117 Uiso 1 1 calc R U . . .
 H13C H 0.4243 0.1640 0.9020 0.117 Uiso 1 1 calc R U . . .
 C14 C 1.0957(3) 0.3174(3) 0.7405(3) 0.0587(8) Uani 1 1 d
 H14A H 1.1492 0.2792 0.6993 0.088 Uiso 1 1 calc R U . . .
 H14B H 1.1594 0.3510 0.8146 0.088 Uiso 1 1 calc R U . . .
 H14C H 1.0386 0.3782 0.6898 0.088 Uiso 1 1 calc R U . . .
 C15 C 0.7764(4) -0.0300(3) 0.4850(3) 0.0577(8) Uani 1 1 d
 H15A H 0.7206 -0.0577 0.4053 0.087 Uiso 1 1 calc R U . . .
 H15B H 0.7904 -0.0926 0.5423 0.087 Uiso 1 1 calc R U . . .
 H15C H 0.8666 -0.0033 0.4864 0.087 Uiso 1 1 calc R U . . .
 C16 C 0.5349(4) 0.3287(3) 0.3180(3) 0.0723(10) Uani 1 1 d
 H16A H 0.4380 0.3100 0.2707 0.108 Uiso 1 1 calc R U . . .
 H16B H 0.5921 0.3155 0.2705 0.108 Uiso 1 1 calc R U . . .
 H16C H 0.5417 0.4095 0.3420 0.108 Uiso 1 1 calc R U . . .
 C17 C 0.5860(3) 0.2528(3) 0.4280(3) 0.0534(7) Uani 1 1 d
 C18 C 1.0872(3) 0.1259(3) 0.8400(3) 0.0601(8) Uani 1 1 d
 H18A H 1.0258 0.0718 0.8587 0.090 Uiso 1 1 calc R U . . .
 H18B H 1.1575 0.1543 0.9138 0.090 Uiso 1 1 calc R U . . .
 H18C H 1.1330 0.0870 0.7928 0.090 Uiso 1 1 calc R U . . .
 C19 C 1.1276(3) 0.2106(4) 1.2495(3) 0.0737(10) Uani 1 1 d
 H19A H 1.1518 0.1590 1.1964 0.111 Uiso 1 1 calc R U . . .
 H19B H 1.2004 0.2076 1.3289 0.111 Uiso 1 1 calc R U . . .
 H19C H 1.1192 0.2892 1.2190 0.111 Uiso 1 1 calc R U . . .
 OW1 O 1.1732(3) 0.0241(3) 1.5426(3) 0.1088(11) Uani 1 1 d D
 HW1 H 1.258(3) 0.068(5) 1.582(5) 0.163 Uiso 1 1 d D U . . .
 HW2 H 1.106(4) 0.077(4) 1.496(5) 0.163 Uiso 1 1 d D U . . .
 OW2 O 0.4230(4) 0.1502(5) 0.6390(3) 0.1388(16) Uani 1 1 d D
 HW3 H 0.433(8) 0.197(6) 0.579(4) 0.208 Uiso 1 1 d D U . . .
 HW4 H 0.469(7) 0.183(6) 0.716(3) 0.208 Uiso 1 1 d D U . . .

loop_

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 _atom_site_aniso_U_33
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 _atom_site_aniso_U_13
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O1 0.0691(13) 0.0542(13) 0.0394(10) 0.0012(9) 0.0265(9) -0.0092(10)
 O2 0.0513(11) 0.0750(14) 0.0319(10) -0.0035(9) 0.0103(8) -0.0011(10)

O3 0.0817(15) 0.0637(14) 0.0375(10) -0.0014(9) 0.0290(10) -0.0169(11)
O4 0.0668(13) 0.0719(14) 0.0334(10) 0.0028(10) 0.0173(9) -0.0051(12)
O5 0.0783(15) 0.0598(13) 0.0495(11) -0.0059(10) 0.0353(11) 0.0093(11)
O6 0.0969(18) 0.0729(16) 0.0681(15) 0.0054(12) 0.0445(14) 0.0271(14)
N2 0.0467(12) 0.0461(13) 0.0307(10) 0.0009(9) 0.0152(9) -0.0024(10)
N3 0.0498(13) 0.0459(13) 0.0309(10) 0.0016(10) 0.0158(10) 0.0028(11)
N5 0.0564(14) 0.0481(14) 0.0302(10) -0.0019(10) 0.0148(10) -0.0022(11)
N1 0.0487(13) 0.0516(14) 0.0335(11) 0.0004(10) 0.0136(10) 0.0036(11)
N6 0.0727(17) 0.0465(14) 0.0408(12) -0.0040(11) 0.0213(12) 0.0042(13)
N4 0.0541(14) 0.0456(13) 0.0329(11) -0.0042(10) 0.0191(10) -0.0078(11)
C1 0.0472(15) 0.0392(14) 0.0351(12) 0.0013(11) 0.0164(11) 0.0012(12)
C2 0.0461(15) 0.0434(15) 0.0352(13) 0.0005(11) 0.0149(12) -0.0029(12)
C3 0.0617(17) 0.0411(15) 0.0349(13) -0.0043(11) 0.0216(12) -0.0062(13)
C4 0.0498(16) 0.0461(16) 0.0342(13) 0.0003(12) 0.0153(12) -0.0084(13)
C5 0.0466(15) 0.0457(16) 0.0394(14) 0.0008(12) 0.0162(12) 0.0045(13)
C6 0.072(2) 0.063(2) 0.0540(18) 0.0080(16) 0.0233(16) 0.0256(18)
C7 0.0604(17) 0.0531(17) 0.0347(13) -0.0009(12) 0.0200(12) -0.0176(15)
C8 0.0438(15) 0.0437(16) 0.0400(14) -0.0028(12) 0.0135(12) -0.0027(12)
C9 0.0603(18) 0.0449(16) 0.0356(13) 0.0001(12) 0.0098(12) 0.0083(14)
C10 0.0426(14) 0.0494(16) 0.0335(13) -0.0005(12) 0.0124(11) -0.0018(12)
C11 0.0549(17) 0.0392(15) 0.0394(14) -0.0011(12) 0.0190(13) 0.0026(13)
C12 0.119(3) 0.055(2) 0.0442(17) -0.0099(15) 0.0282(19) -0.032(2)
C13 0.059(2) 0.115(3) 0.063(2) 0.013(2) 0.0249(16) -0.017(2)
C14 0.0536(17) 0.070(2) 0.0565(18) -0.0113(16) 0.0252(15) -0.0174(16)
C15 0.0621(19) 0.0533(18) 0.0522(17) -0.0097(14) 0.0144(15) 0.0089(15)
C16 0.078(2) 0.068(2) 0.066(2) 0.0222(19) 0.0220(19) 0.0155(19)
C17 0.0572(18) 0.0543(19) 0.0471(17) 0.0030(14) 0.0175(14) 0.0052(15)
C18 0.0579(18) 0.064(2) 0.0502(16) 0.0038(15) 0.0108(14) 0.0132(16)
C19 0.0526(19) 0.111(3) 0.0540(19) -0.016(2) 0.0160(15) 0.011(2)
OW1 0.087(2) 0.113(3) 0.117(3) 0.060(2) 0.0260(19) 0.0004(18)
OW2 0.128(3) 0.173(4) 0.110(3) 0.012(3) 0.037(2) -0.059(3)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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O2 C2 1.219(3) . ?
O3 C3 1.229(3) . ?
O4 C4 1.237(3) . ?
O5 C5 1.230(3) . ?
O6 C17 1.236(4) . ?
N2 C1 1.341(3) . ?
N2 C10 1.469(3) . ?
N2 H1 0.8600 . ?
N3 C2 1.350(4) . ?
N3 C11 1.450(3) . ?
N3 H2 0.8600 . ?
N5 C3 1.342(4) . ?
N5 C9 1.447(3) . ?
N5 H3 0.8600 . ?
N1 C17 1.340(4) . ?
N1 C8 1.450(4) . ?
N1 H4 0.8600 . ?
N6 C4 1.313(4) . ?
N6 H5A 0.8600 . ?
N6 H5B 0.8600 . ?
N4 C5 1.342(4) . ?
N4 C7 1.467(4) . ?
N4 H6 0.8600 . ?
C1 C8 1.522(4) . ?
C2 C10 1.542(4) . ?
C3 C7 1.523(4) . ?
C4 C9 1.516(4) . ?
C5 C11 1.518(4) . ?
C6 C11 1.506(4) . ?
C6 H6A 0.9600 . ?
C6 H6B 0.9600 . ?
C6 H6C 0.9600 . ?
C7 C13 1.524(5) . ?
C7 C12 1.541(5) . ?
C8 C15 1.507(4) . ?
C8 H8 0.9800 . ?
C9 C19 1.521(5) . ?
C9 H9 0.9800 . ?
C10 C18 1.522(4) . ?
C10 C14 1.521(4) . ?
C11 H11 0.9800 . ?
C12 H12A 0.9600 . ?
C12 H12B 0.9600 . ?

C12 H12C 0.9600 . ?
C13 H13A 0.9600 . ?
C13 H13B 0.9600 . ?
C13 H13C 0.9600 . ?
C14 H14A 0.9600 . ?
C14 H14B 0.9600 . ?
C14 H14C 0.9600 . ?
C15 H15A 0.9600 . ?
C15 H15B 0.9600 . ?
C15 H15C 0.9600 . ?
C16 C17 1.493(5) . ?
C16 H16A 0.9600 . ?
C16 H16B 0.9600 . ?
C16 H16C 0.9600 . ?
C18 H18A 0.9600 . ?
C18 H18B 0.9600 . ?
C18 H18C 0.9600 . ?
C19 H19A 0.9600 . ?
C19 H19B 0.9600 . ?
C19 H19C 0.9600 . ?
OW1 HW1 0.955(19) . ?
OW1 HW2 0.924(19) . ?
OW2 HW3 0.931(19) . ?
OW2 HW4 0.936(19) . ?

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C1 N2 H1 118.1 . . ?
C10 N2 H1 118.1 . . ?
C2 N3 C11 120.7(2) . . ?
C2 N3 H2 119.6 . . ?
C11 N3 H2 119.6 . . ?
C3 N5 C9 121.0(2) . . ?
C3 N5 H3 119.5 . . ?
C9 N5 H3 119.5 . . ?
C17 N1 C8 121.6(2) . . ?
C17 N1 H4 119.2 . . ?
C8 N1 H4 119.2 . . ?
C4 N6 H5A 120.0 . . ?

C4 N6 H5B 120.0 . . ?
H5A N6 H5B 120.0 . . ?
C5 N4 C7 122.6(2) . . ?
C5 N4 H6 118.7 . . ?
C7 N4 H6 118.7 . . ?
O1 C1 N2 122.7(2) . . ?
O1 C1 C8 120.1(2) . . ?
N22 C1 C8 117.1(2) . . ?
O2 C2 N3 121.6(3) . . ?
O2 C2 C10 121.0(2) . . ?
N3 C2 C10 117.3(2) . . ?
O3 C3 N5 121.2(3) . . ?
O3 C3 C7 121.0(2) . . ?
N5 C3 C7 117.6(2) . . ?
O4 C4 N6 122.5(3) . . ?
O4 C4 C9 119.3(3) . . ?
N6 C4 C9 118.1(2) . . ?
O5 C5 N4 123.2(3) . . ?
O5 C5 C11 119.9(3) . . ?
N4 C5 C11 116.9(2) . . ?
C11 C6 H6A 109.5 . . ?
C11 C6 H6B 109.5 . . ?
H6A C6 H6B 109.5 . . ?
C11 C6 H6C 109.5 . . ?
H6A C6 H6C 109.5 . . ?
H6B C6 H6C 109.5 . . ?
N4 C7 C13 110.1(3) . . ?
N4 C7 C3 110.6(2) . . ?
C13 C7 C3 111.0(3) . . ?
N4 C7 C12 108.1(2) . . ?
C13 C7 C12 111.0(3) . . ?
C3 C7 C12 106.0(3) . . ?
N1 C8 C15 110.0(2) . . ?
N1 C8 C1 113.3(2) . . ?
C15 C8 C1 110.8(2) . . ?
N1 C8 H8 107.5 . . ?
C15 C8 H8 107.5 . . ?
C1 C8 H8 107.5 . . ?
N5 C9 C4 113.0(2) . . ?
N5 C9 C19 109.9(2) . . ?
C4 C9 C19 109.4(3) . . ?
N5 C9 H9 108.1 . . ?
C4 C9 H9 108.1 . . ?
C19 C9 H9 108.1 . . ?
N2 C10 C18 111.1(2) . . ?
N2 C10 C14 107.8(2) . . ?

C18 C10 C14 110.6(3) . . ?
N2 C10 C2 110.6(2) . . ?
C18 C10 C2 109.8(2) . . ?
C14 C10 C2 106.8(2) . . ?
N3 C11 C6 109.7(2) . . ?
N3 C11 C5 113.1(2) . . ?
C6 C11 C5 110.3(2) . . ?
N3 C11 H11 107.8 . . ?
C6 C11 H11 107.8 . . ?
C5 C11 H11 107.8 . . ?
C7 C12 H12A 109.5 . . ?
C7 C12 H12B 109.5 . . ?
H12A C12 H12B 109.5 . . ?
C7 C12 H12C 109.5 . . ?
H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
C7 C13 H13A 109.5 . . ?
C7 C13 H13B 109.5 . . ?
H13A C13 H13B 109.5 . . ?
C7 C13 H13C 109.5 . . ?
H13A C13 H13C 109.5 . . ?
H13B C13 H13C 109.5 . . ?
C10 C14 H14A 109.5 . . ?
C10 C14 H14B 109.5 . . ?
H14A C14 H14B 109.5 . . ?
C10 C14 H14C 109.5 . . ?
H14A C14 H14C 109.5 . . ?
H14B C14 H14C 109.5 . . ?
C8 C15 H15A 109.5 . . ?
C8 C15 H15B 109.5 . . ?
H15A C15 H15B 109.5 . . ?
C8 C15 H15C 109.5 . . ?
H15A C15 H15C 109.5 . . ?
H15B C15 H15C 109.5 . . ?
C17 C16 H16A 109.5 . . ?
C17 C16 H16B 109.5 . . ?
H16A C16 H16B 109.5 . . ?
C17 C16 H16C 109.5 . . ?
H16A C16 H16C 109.5 . . ?
H16B C16 H16C 109.5 . . ?
O6 C17 N1 120.9(3) . . ?
O6 C17 C16 122.0(3) . . ?
N1 C17 C16 117.1(3) . . ?
C10 C18 H18A 109.5 . . ?
C10 C18 H18B 109.5 . . ?
H18A C18 H18B 109.5 . . ?

C10 C18 H18C 109.5 . . ?
H18A C18 H18C 109.5 . . ?
H18B C18 H18C 109.5 . . ?
C9 C19 H19A 109.5 . . ?
C9 C19 H19B 109.5 . . ?
H19A C19 H19B 109.5 . . ?
C9 C19 H19C 109.5 . . ?
H19A C19 H19C 109.5 . . ?
H19B C19 H19C 109.5 . . ?
HW1 OW1 HW2 107(3) . . ?
HW3 OW2 HW4 112(5) . . ?

loop_

_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
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_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
C10 N2 C1 O1 5.7(4) ?
C10 N2 C1 C8 -171.0(2) ?
C11 N3 C2 O2 -2.0(4) ?
C11 N3 C2 C10 -179.5(2) ?
C9 N5 C3 O3 -1.6(4) ?
C9 N5 C3 C7 -176.6(3) ?
C7 N4 C5 O5 1.5(4) ?
C7 N4 C5 C11 -177.5(2) ?
C5 N4 C7 C13 65.2(4) ?
C5 N4 C7 C3 -57.9(4) ?
C5 N4 C7 C12 -173.5(3) ?
O3 C3 C7 N4 158.5(3) ?
N5 C3 C7 N4 -26.4(4) ?
O3 C3 C7 C13 36.0(4) ?
N5 C3 C7 C13 -148.9(3) ?
O3 C3 C7 C12 -84.6(3) ?
N5 C3 C7 C12 90.5(3) ?
C17 N1 C8 C15 174.9(3) ?
C17 N1 C8 C1 -60.6(3) ?
O1 C1 C8 N1 138.2(3) ?
N2 C1 C8 N1 -45.0(3) ?
O1 C1 C8 C15 -97.7(3) ?
N2 C1 C8 C15 79.1(3) ?

C3 N5 C9 C4 -59.2(3) ?
 C3 N5 C9 C19 178.3(3) ?
 O4 C4 C9 N5 150.8(3) ?
 N6 C4 C9 N5 -33.1(4) ?
 O4 C4 C9 C19 -86.3(3) ?
 N6 C4 C9 C19 89.8(3) ?
 C1 N2 C10 C18 63.8(3) ?
 C1 N2 C10 C14 -174.8(3) ?
 C1 N2 C10 C2 -58.4(3) ?
 O2 C2 C10 N2 154.2(3) ?
 N3 C2 C10 N2 -28.2(3) ?
 O2 C2 C10 C18 31.3(4) ?
 N3 C2 C10 C18 -151.2(3) ?
 O2 C2 C10 C14 -88.8(3) ?
 N3 C2 C10 C14 88.8(3) ?
 C2 N3 C11 C6 178.8(3) ?
 C2 N3 C11 C5 -57.5(3) ?
 O5 C5 C11 N3 153.6(3) ?
 N4 C5 C11 N3 -27.4(3) ?
 O5 C5 C11 C6 -83.0(3) ?
 N4 C5 C11 C6 96.0(3) ?
 C8 N1 C17 O6 10.4(4) ?
 C8 N1 C17 C16 -170.3(3) ?

loop_

_geom_hbond_atom_site_label_D
 _geom_hbond_atom_site_label_H
 _geom_hbond_atom_site_label_A
 _geom_hbond_distance_DH
 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
 N2 H1 O4 0.86 2.16 2.962(3) 154.8 1_554
 N3 H2 O6 0.86 2.57 3.309(4) 145.2 .
 N5 H3 O2 0.86 2.26 3.093(3) 163.0 .
 N1 H4 O3 0.86 2.08 2.912(3) 164.3 1_554
 N6 H5A OW1 0.86 2.05 2.911(4) 173.5 2_758
 N6 H5B O5 0.86 2.37 3.182(3) 158.0 .
 N4 H6 O1 0.86 2.19 3.015(3) 161.9 .
 C13 H13A O5 0.96 2.50 3.060(5) 117.1 .
 C18 H18A O1 0.96 2.55 3.114(4) 117.4 .
 OW1 HW1 OW2 0.955(19) 1.82(2) 2.770(5) 173(6) 1_656
 OW1 HW2 O4 0.924(19) 1.97(3) 2.844(4) 158(5) .
 OW2 HW3 O6 0.931(19) 1.90(4) 2.731(5) 147(6) .
 OW2 HW4 N4 0.936(19) 2.55(4) 3.423(4) 155(6) .

_refine_diff_density_max 0.163
_refine_diff_density_min -0.214
_refine_diff_density_rms 0.035

_shelxl_version_number 2014/6

_shelx_res_file

;

TITL compound 7

CELL 0.71073 10.1561 11.4127 11.8880 90.000 111.692 90.000

ZERR 2.00 0.0020 0.0023 0.0024 0.000 0.030 0.000

LATT -1

SYMM -X, 0.5+Y, -Z

SFAC C H N O

UNIT 38 76 12 16

OMIT -3. 60.

ACTA

L.S. 8

BOND\$H

MERG 2

LIST 4

EQIV \$1 x, y, z-1

HTAB N22 O4_\$1

HTAB N3 O6

HTAB N5 O2

HTAB N1 O3_\$1

EQIV \$2 -x+2, y+1/2, -z+3

HTAB N6 OW1_\$2

HTAB N6 O5

HTAB N4 O1

HTAB C13 O5

HTAB C18 O1

EQIV \$3 x+1, y, z+1

HTAB OW1 OW2_\$3

HTAB OW1 O4

HTAB OW2 O6

HTAB OW2 N6

CONF

FMAP 2

PLAN 20

WGHT 0.062200

EXTI 0.009052

FVAR 0.28393

O1 4 0.761167 0.084216 0.731895 11.00000 0.06906 0.05425 =
0.03936 0.00125 0.02647 -0.00924

O2 4 0.983409 0.296421 0.954160 11.00000 0.05133 0.07496 =
0.03189 -0.00351 0.01028 -0.00110
O3 4 0.712764 0.105452 1.203733 11.00000 0.08169 0.06374 =
0.03752 -0.00143 0.02896 -0.01687
O4 4 1.000413 0.225191 1.454017 11.00000 0.06677 0.07193 =
0.03343 0.00276 0.01732 -0.00514
O5 4 0.680664 0.379128 1.026688 11.00000 0.07834 0.05983 =
0.04946 -0.00591 0.03529 0.00931
O6 4 0.559730 0.275405 0.519250 11.00000 0.09687 0.07289 =
0.06809 0.00540 0.04454 0.02706
N22 3 0.894671 0.188839 0.652620 11.00000 0.04668 0.04613 =
0.03068 0.00089 0.01521 -0.00237
AFIX 43
H1 2 0.902212 0.213409 0.586876 11.00000 -1.20000
AFIX 0
N3 3 0.804380 0.347422 0.783502 11.00000 0.04985 0.04593 =
0.03089 0.00156 0.01576 0.00278
AFIX 43
H2 2 0.769150 0.345016 0.705700 11.00000 -1.20000
AFIX 0
N5 3 0.877682 0.173156 1.136747 11.00000 0.05639 0.04807 =
0.03025 -0.00193 0.01485 -0.00218
AFIX 43
H3 2 0.897997 0.195198 1.075890 11.00000 -1.20000
AFIX 0
N1 3 0.663765 0.158913 0.424623 11.00000 0.04874 0.05165 =
0.03347 0.00041 0.01359 0.00357
AFIX 43
H4 2 0.692693 0.151378 0.365540 11.00000 -1.20000
AFIX 0
N6 3 0.879413 0.346375 1.303417 11.00000 0.07267 0.04646 =
0.04081 -0.00401 0.02126 0.00416
AFIX 93
H5A 2 0.863039 0.394436 1.352427 11.00000 -1.20000
H5B 2 0.847824 0.361118 1.227078 11.00000 -1.20000
AFIX 0
N4 3 0.677918 0.219248 0.913311 11.00000 0.05410 0.04560 =
0.03288 -0.00421 0.01906 -0.00784
AFIX 43
H6 2 0.691172 0.193020 0.850499 11.00000 -1.20000
AFIX 0
C1 1 0.787033 0.116675 0.643495 11.00000 0.04718 0.03919 =
0.03509 0.00133 0.01640 0.00121
C2 1 0.928143 0.292467 0.844150 11.00000 0.04611 0.04339 =
0.03515 0.00051 0.01487 -0.00293
C3 1 0.744788 0.139805 1.119229 11.00000 0.06173 0.04114 =

0.03490 -0.00430 0.02161 -0.00621
 C4 1 0.952662 0.250459 1.345154 11.00000 0.04978 0.04613 =
 0.03423 0.00026 0.01535 -0.00844
 C5 1 0.694683 0.334295 0.937712 11.00000 0.04659 0.04568 =
 0.03937 0.00083 0.01619 0.00449
 C6 1 0.597680 0.465125 0.759090 11.00000 0.07249 0.06344 =
 0.05401 0.00801 0.02330 0.02557
 AFIX 137
 H6A 2 0.622771 0.521312 0.710313 11.00000 -1.50000
 H6B 2 0.546613 0.503625 0.802224 11.00000 -1.50000
 H6C 2 0.539173 0.405290 0.707990 11.00000 -1.50000
 AFIX 0
 C7 1 0.637879 0.136441 0.989535 11.00000 0.06042 0.05312 =
 0.03469 -0.00092 0.02004 -0.01760
 C8 1 0.700290 0.069759 0.518043 11.00000 0.04380 0.04374 =
 0.03997 -0.00283 0.01352 -0.00272
 AFIX 13
 H8 2 0.611565 0.038679 0.520765 11.00000 -1.20000
 AFIX 0
 C9 1 0.987223 0.172576 1.256450 11.00000 0.06032 0.04488 =
 0.03561 0.00009 0.00978 0.00831
 AFIX 13
 H9 2 0.997919 0.092105 1.287313 11.00000 -1.20000
 AFIX 0
 C10 1 1.000490 0.228409 0.768324 11.00000 0.04263 0.04936 =
 0.03346 -0.00053 0.01237 -0.00182
 C11 1 0.730240 0.410441 0.848121 11.00000 0.05487 0.03923 =
 0.03943 -0.00112 0.01898 0.00265
 AFIX 13
 H11 2 0.792131 0.473874 0.893616 11.00000 -1.20000
 AFIX 0
 C12 1 0.645759 0.011648 0.942920 11.00000 0.11854 0.05543 =
 0.04421 -0.00988 0.02819 -0.03227
 AFIX 137
 H12A 2 0.583938 0.005919 0.859504 11.00000 -1.50000
 H12B 2 0.616973 -0.043910 0.989992 11.00000 -1.50000
 H12C 2 0.741277 -0.004808 0.950225 11.00000 -1.50000
 AFIX 0
 C13 1 0.489045 0.163704 0.984752 11.00000 0.05898 0.11476 =
 0.06269 0.01292 0.02490 -0.01730
 AFIX 137
 H13A 2 0.488508 0.239213 1.020167 11.00000 -1.50000
 H13B 2 0.460546 0.105074 1.029056 11.00000 -1.50000
 H13C 2 0.424304 0.164004 0.902020 11.00000 -1.50000
 AFIX 0
 C14 1 1.095712 0.317412 0.740455 11.00000 0.05364 0.07016 =

0.05651 -0.01126 0.02517 -0.01736
 AFIX 137
 H14A 2 1.149235 0.279230 0.699283 11.00000 -1.50000
 H14B 2 1.159416 0.350996 0.814648 11.00000 -1.50000
 H14C 2 1.038584 0.378183 0.689843 11.00000 -1.50000
 AFIX 0
 C15 1 0.776366 -0.030014 0.485010 11.00000 0.06210 0.05326 =
 0.05217 -0.00967 0.01444 0.00893
 AFIX 137
 H15A 2 0.720552 -0.057727 0.405335 11.00000 -1.50000
 H15B 2 0.790420 -0.092574 0.542264 11.00000 -1.50000
 H15C 2 0.866576 -0.003284 0.486393 11.00000 -1.50000
 AFIX 0
 C16 1 0.534877 0.328730 0.317990 11.00000 0.07849 0.06838 =
 0.06598 0.02223 0.02202 0.01550
 AFIX 137
 H16A 2 0.437954 0.310011 0.270694 11.00000 -1.50000
 H16B 2 0.592119 0.315483 0.270532 11.00000 -1.50000
 H16C 2 0.541676 0.409510 0.342000 11.00000 -1.50000
 AFIX 0
 C17 1 0.586030 0.252793 0.428043 11.00000 0.05715 0.05426 =
 0.04712 0.00302 0.01747 0.00525
 C18 1 1.087189 0.125923 0.840044 11.00000 0.05791 0.06423 =
 0.05019 0.00379 0.01079 0.01316
 AFIX 137
 H18A 2 1.025804 0.071797 0.858695 11.00000 -1.50000
 H18B 2 1.157487 0.154334 0.913848 11.00000 -1.50000
 H18C 2 1.132975 0.086980 0.792838 11.00000 -1.50000
 AFIX 0
 C19 1 1.127616 0.210602 1.249542 11.00000 0.05260 0.11143 =
 0.05401 -0.01578 0.01600 0.01080
 AFIX 137
 H19A 2 1.151803 0.158966 1.196392 11.00000 -1.50000
 H19B 2 1.200386 0.207600 1.328876 11.00000 -1.50000
 H19C 2 1.119246 0.289235 1.218999 11.00000 -1.50000
 AFIX 0
 SADI OW1 HW1 OW1 HW2 OW2 HW3 OW2 HW4
 DFIX 0.900 0.02 OW1 HW1 OW1 HW2 OW2 HW3 OW2 HW4
 DANG 1.550 0.02 HW1 HW2
 DANG 1.350 0.04 HW3 HW4
 OW1 4 1.173199 0.024085 1.542626 11.00000 0.08656 0.11270 =
 0.11709 0.06021 0.02599 0.00044
 HW1 2 1.257711 0.068155 1.582083 11.00000 -1.50000
 HW2 2 1.105709 0.076660 1.496218 11.00000 -1.50000
 OW2 4 0.422983 0.150213 0.639015 11.00000 0.12767 0.17282 =
 0.10972 0.01236 0.03675 -0.05946

HW3 2 0.433137 0.197434 0.578893 11.00000 -1.50000
HW4 2 0.468862 0.182505 0.716014 11.00000 -1.50000
HKLF 4

REM sad1 in P2(1)

REM R1 = 0.0417 for 4289 Fo > 4sig(Fo) and 0.0931 for all 7475 data

REM 319 parameters refined using 13 restraints

END

WGHT 0.0636 0.0000

REM Highest difference peak 0.163, deepest hole -0.214, 1-sigma level 0.035

Q1 1 0.4755 0.2165 0.4789 11.00000 0.05 0.16
Q2 1 0.4306 0.3828 0.3259 11.00000 0.05 0.15
Q3 1 0.7776 0.0993 0.3224 11.00000 0.05 0.15
Q4 1 0.6847 0.2402 1.1919 11.00000 0.05 0.14
Q5 1 0.8626 0.1194 0.8227 11.00000 0.05 0.13
Q6 1 0.8508 0.4560 0.9363 11.00000 0.05 0.13
Q7 1 0.7415 0.2258 0.7395 11.00000 0.05 0.13
Q8 1 0.7649 0.2164 0.4485 11.00000 0.05 0.13
Q9 1 0.8676 0.1770 0.7837 11.00000 0.05 0.13
Q10 1 0.7903 0.2795 0.5336 11.00000 0.05 0.12
Q11 1 0.7701 0.0964 1.2314 11.00000 0.05 0.12
Q12 1 0.8259 0.1931 0.7207 11.00000 0.05 0.12
Q13 1 0.4985 0.2143 0.5639 11.00000 0.05 0.12
Q14 1 0.7925 0.2624 1.3144 11.00000 0.05 0.12
Q15 1 0.8419 0.1397 1.2954 11.00000 0.05 0.12
Q16 1 0.6758 -0.0571 1.0188 11.00000 0.05 0.12
Q17 1 0.7084 0.3139 0.6934 11.00000 0.05 0.12
Q18 1 0.6399 0.4124 0.3323 11.00000 0.05 0.12
Q19 1 0.3600 0.0331 0.7804 11.00000 0.05 0.11
Q20 1 0.5976 0.0637 0.6978 11.00000 0.05 0.11

;

_shelx_res_checksum 72766

_shelx_hkl_file

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1 0 0 76.03 1.43
1 0 0 76.15 1.53
-1 0 0 77.00 1.52
-1 0 0 73.53 1.66
-2 0 0 0.43 0.13
2 0 0 0.42 0.13
2 0 0 0.49 0.14
-2 0 0 0.55 0.11

2	0	0	0.44	0.07
-3	0	0	57.73	1.44
3	0	0	57.54	1.31
3	0	0	57.88	1.14
3	0	0	57.00	1.22
3	0	0	56.31	1.43
-3	0	0	59.79	1.28
-4	0	0	5.88	0.32
4	0	0	6.38	0.37
4	0	0	6.31	0.42
4	0	0	6.42	0.24
-4	0	0	6.63	0.42
4	0	0	6.24	0.31
5	0	0	3.66	0.34
5	0	0	3.52	0.37
5	0	0	4.02	0.22
-5	0	0	3.72	0.30
-5	0	0	3.55	0.29
-5	0	0	3.64	0.38
5	0	0	3.77	0.30
-6	0	0	2.59	0.28
-6	0	0	1.81	0.29
6	0	0	1.55	0.35
6	0	0	2.13	0.35
6	0	0	2.37	0.21
-6	0	0	2.27	0.39
-7	0	0	9.09	0.71
7	0	0	10.56	0.71
-7	0	0	8.75	0.56
7	0	0	9.83	0.38
-7	0	0	9.56	0.48
7	0	0	10.37	0.64
-8	0	0	0.67	0.29
-8	0	0	0.84	0.31
8	0	0	0.36	0.38
9	0	0	11.90	0.70
-9	0	0	11.53	0.65
-9	0	0	11.78	0.60
10	0	0	5.89	0.61
-10	0	0	5.28	0.55
-11	0	0	-0.65	0.32
11	0	0	-0.08	0.40
-12	0	0	0.15	0.36
12	0	0	0.29	0.47
13	0	0	0.71	0.53
-13	0	0	0.03	0.43

0 -1 0 -0.94 0.05
0 -1 0 -0.95 0.05
0 -1 0 -0.75 0.05
0 -1 0 -0.85 0.06
0 1 0 -0.83 0.05
1 -1 0 986.04 19.27
-1 -1 0 953.93 20.64
-1 -1 0 975.99 19.31
1 -1 0 961.81 19.43
1 -1 0 950.10 20.72
-1 -1 0 944.36 18.96
-1 1 0 960.21 17.89
-1 1 0 964.40 19.06
-1 1 0 947.84 20.68
1 1 0 982.31 19.41
1 1 0 953.27 20.66
1 1 0 984.94 18.04
2 -1 0 88.20 2.02
-2 -1 0 88.15 2.01
-2 -1 0 87.73 1.82
2 -1 0 90.11 1.83
2 -1 0 86.09 1.87
2 1 0 88.85 2.01
2 1 0 90.38 1.87
-2 1 0 91.22 1.84
-2 1 0 88.21 1.67
3 -1 0 103.16 2.15
3 -1 0 103.97 2.41
-3 -1 0 105.09 2.16
-3 -1 0 104.05 2.40
3 -1 0 100.30 2.22
-3 1 0 104.02 1.97
3 1 0 105.22 2.05
3 1 0 102.08 2.38
-3 1 0 104.81 2.41
-3 1 0 100.89 2.18
3 1 0 105.38 2.21
4 -1 0 116.71 2.23
4 -1 0 113.70 2.44
-4 -1 0 117.10 2.46
4 -1 0 114.91 2.74
-4 -1 0 117.64 2.73
4 -1 0 113.56 2.52
-4 1 0 119.50 2.49
4 1 0 115.42 2.27
4 1 0 120.33 2.73

4	1	0	116.53	2.52
-4	1	0	114.16	2.74
5	-1	0	7.85	0.43
5	-1	0	7.59	0.27
-5	-1	0	8.12	0.41
-5	-1	0	7.90	0.52
5	-1	0	7.81	0.53
5	-1	0	8.05	0.36
-5	1	0	7.78	0.52
5	1	0	8.30	0.51
5	1	0	7.69	0.30
-5	1	0	7.08	0.39
5	1	0	8.19	0.45
-6	-1	0	2.14	0.36
6	-1	0	2.66	0.30
-6	-1	0	2.59	0.30
6	-1	0	2.67	0.20
6	-1	0	1.87	0.34
6	-1	0	3.09	0.42
6	1	0	1.96	0.35
-6	1	0	2.53	0.39
-6	1	0	2.31	0.30
-6	1	0	2.83	0.28
6	1	0	2.43	0.34
6	1	0	2.45	0.22
-7	-1	0	11.25	0.52
7	-1	0	10.88	0.55
7	-1	0	10.94	0.65
-7	-1	0	11.80	0.59
7	-1	0	11.30	0.39
-7	-1	0	12.62	0.78
-7	1	0	10.23	0.49
7	1	0	12.64	0.76
-7	1	0	10.21	0.55
7	1	0	12.20	0.45
7	1	0	11.29	0.65
-7	1	0	11.41	0.78
8	-1	0	3.06	0.49
-8	-1	0	2.29	0.37
-8	-1	0	3.22	0.38
8	1	0	3.12	0.29
-8	1	0	3.11	0.34
-8	1	0	2.44	0.37
8	1	0	2.86	0.43
-9	-1	0	0.87	0.31
9	-1	0	0.81	0.40

-9 -1 0 0.63 0.31
9 1 0 0.59 0.34
-9 1 0 0.89 0.31
-9 1 0 1.27 0.38
-10 -1 0 2.51 0.41
10 -1 0 2.89 0.49
10 1 0 2.38 0.49
-10 1 0 3.68 0.49
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6	1	-1	12.23	0.57
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3	2	14	-0.77	0.92
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6	5	-15	-0.12	0.45
5	-5	-15	0.42	0.56
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5	5	-15	0.00	0.43
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3	-5	-15	1.38	0.57
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3	5	-15	1.15	0.44
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2	-5	-15	0.92	0.51
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1	-5	-15	0.67	0.46
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1	5	-15	0.78	0.46
0	-5	-15	0.00	0.44
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8	-6	-15	-0.60	0.55
8	6	-15	0.53	0.53
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7	-6	-15	0.10	0.60
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1	6	-15	-0.50	0.41
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7	7	-15	-0.35	0.41
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4	-7	-15	1.27	0.59
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3	-7	-15	0.58	0.93
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1	7	-15	-0.21	0.47
7	8	-15	-0.06	0.50
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7	0	-16	-0.32	0.52
-7	0	16	0.09	0.47
6	0	-16	0.92	0.58
-6	0	16	0.04	0.46
5	0	-16	-0.22	0.50

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5	2	-16	0.36	0.45
4	-2	-16	0.90	0.54
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3	-2	-16	0.57	0.53
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1	2	-16	0.69	0.42
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8	-4	-16	0.00	0.60
8	4	-16	-0.21	0.40
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4	0	-17	-0.07	0.52
3	0	-17	0.04	0.52
8	-1	-17	0.16	0.61
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7	1	-17	-0.34	0.46
6	-1	-17	0.23	0.61
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5	-1	-17	-0.21	0.55
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4	-1	-17	0.94	0.58
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