

Supplementary Information

Photoinduced Morphological Transformation in the Azobenzene-Cored Hybrid Peptides

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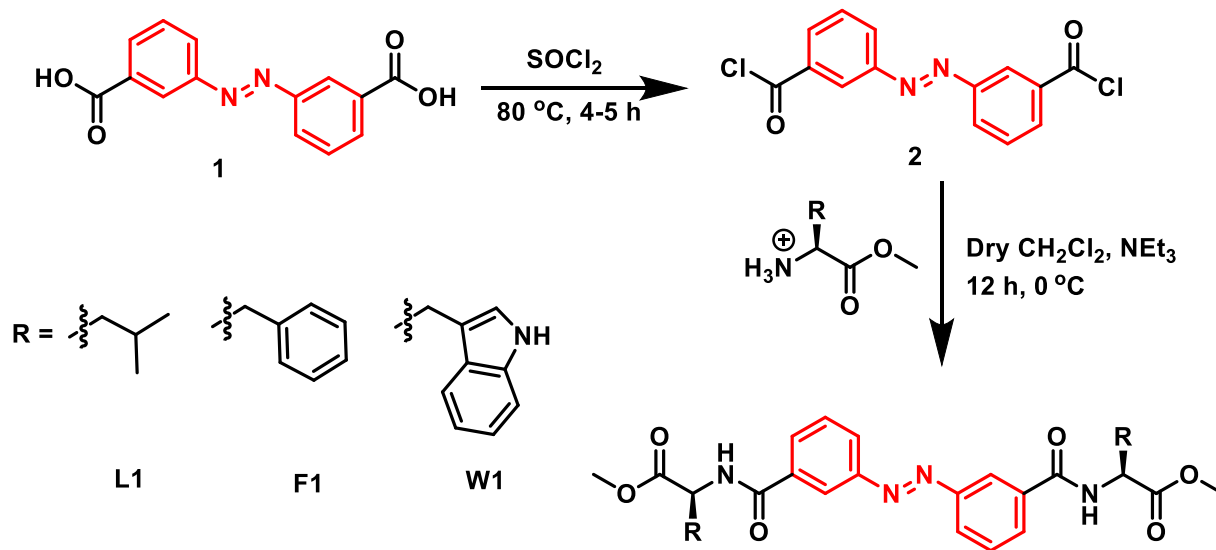
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General Experimental Section:

Reagents were purchased from Sigma-Aldrich (USA), and Alfa Aesar (India). All amino acids used were of L-configuration. Silica gel thin-layer chromatography (TLC) was used to monitor all reactions. All synthesized compounds were purified by silica gel (100–200 mesh, SRL, India) column chromatography. Standard methods were utilised to dry the solvents. ^1H and ^{13}C NMR spectra were obtained on a Bruker-DPX-500MHz and Zeol 400MHz spectrometer and the chemical shifts were reported relative to tetramethylsilane (TMS) as the internal standard. Coupling constants are recorded in Hz, and the ^1H NMR data are reported as s (singlet), d (doublet), br (broad), t (triplet) and m (multiplet), dd (doublet of doublets), ddd (doublet of doublet of doublets), br td (broad triplet of doublets). High resolution mass spectra (HRMS) were recorded in a Bruker MicrO-TOF-QII model using the ESI technique. Melting points were recorded by a Fisher scientific melting point apparatus. X-ray diffraction analysis was carried out on a Bruker AXS SMART APEX diffractometer with a CCD area detector (Mo $K\alpha = 0.71073\text{\AA}$, monochromator: S3 graphite). Structure solution, refinement, and data output were carried out with the ShelXTL program. Photoisomerization of *trans-to-trans* isomer was performed by using a high-intensity mercury lamp (OmniCure S2000 UV curing system, 10 mW/cm^2), with 365 nm and 400-500 nm filters (Lumen Dynamics). Specific rotation was measured on a Rudolph Research Analytical Polarimeter (Model: Autopol V) using sodium D-line (589 nm).

Synthetic Scheme:



Scheme S1: Synthetic scheme of L1, F1 and W1.

Preparation of Compounds:

Synthesis of 2

To azobenzene-3,3'-dicarboxylic acid **1** (1.50 g, 5.55 mmol) was added thionyl chloride (SOCl_2) (4.03 mL, 55.50 mmol), and the reaction mixture was heated at $80\text{ }^\circ\text{C}$ in an oil bath for 4 h. The resultant mixture was evaporated under high vacuum to obtain **2** as a yellow powder.

Yield: quantitative.

Synthesis of L1

To an ice-cooled solution of leucine methyl ester hydrochloride (0.500 g, 2.76 mmol) in 20 mL of dry dichloromethane (DCM), triethylamine (0.77 mL, 5.52 mmol) was added and stirred for about 10 min. To the reaction mixture, a solution of **2** (0.424 g, 1.38 mmol) in 30 mL of dry DCM was added dropwise over a period of 30 minutes. The resultant mixture was stirred for 12 h at room

temperature. After completion of reaction (as monitored by TLC), the reaction mixture was washed with 0.2 N H₂SO₄, saturated aq. NaHCO₃ solution and water. The organic layer was collected and dried over anhydrous Na₂SO₄ and evaporated under vacuum to obtain the crude product, which was purified by using silica gel column chromatography (Ethyl acetate/Hexane in 3:7) to obtain 0.650 g of **L1** as a yellow powder.

Yield: 90 %

MP: 180–182 °C; [α]_D²¹ = -30° (c 0.05 g/mL, DMSO)

¹H NMR (400 MHz, DMSO-*d*₆) δ 0.90 (d, *J* = 6.5 Hz, 6H), 0.94 (d, *J* = 6.5 Hz, 6H), 1.60 (br m, 2H), 1.72 (br m, 2H), 1.82 (br m, 2H), 3.66 (s, 6H), 4.55 (m, 2H), 7.74 (t, *J* = 8 Hz, 2H), 8.10 (m, 4H), 8.45 (t, *J* = 2 Hz, 2H), 9.00 (d, *J* = 8 Hz, 2H); ¹³C NMR (100 MHz, DMSO- *d*₆) δ 21.7, 23.4, 25.0, 51.6, 52.5, 53.5, 122.3, 125.8, 130.3, 131.2, 135.6, 152.3, 166.4, 173.5; IR (KBr): 3325, 3060, 2957, 1749, 1641, 1548, 1345, 1272, 1213, 1156 cm⁻¹; HRMS calcd. for C₂₈H₃₆N₄NaO₆ m/z 547.2533, found m/z 547.2536.

Synthesis of F1

To an ice-cooled solution of phenylalanine methyl ester hydrochloride (0.500 g, 2.32 mmol) in 20 mL of dry DCM, triethylamine (0.65 mL, 4.6 mmol) was added and stirred for about 10 min. To the reaction mixture, a solution of **2** (0.355 g, 1.16 mmol) in 30 mL of dry DCM was added dropwise over a period of 30 minutes. The resultant mixture was stirred for 12 h at room temperature. After the completion of reaction (as monitored by TLC), the reaction mixture was washed with 0.2 N H₂SO₄, saturated aq. NaHCO₃ solution and water. The organic layer was collected and dried over anhydrous Na₂SO₄ and evaporated under vacuum to obtain the crude

product, which was purified by silica gel column chromatography (Ethyl acetate/Hexane in 3.5:6.5) to obtain 0.550 g of **F1** as a yellow powder.

Yield: 89.3 %

MP: 185–187 °C; $[\alpha]_D^{21} = -46^\circ$ (c 0.05 g/mL, DMSO)

^1H NMR (400 MHz, DMSO- d_6) δ 3.16 (m, 4H), 3.65 (s, 6H), 4.71 (m, 2H), 7.19 (tt, $J = 6.9$ Hz, $J = 1.8$ Hz, 2H), 7.30 (m, 8H), 7.72 (t, $J = 8$ Hz, 2H), 8.00 (dt, $J = 8$ Hz, $J = 1$ Hz, 2H), 8.08 (br d, $J = 8$ Hz, 2H), 8.35 (t, $J = 1$ Hz, 2H), 9.13 (d, $J = 8$ Hz, 2H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 36.7, 52.6, 55.0, 122.0, 126.1, 127.1, 128.8, 129.6, 130.3, 131.0, 135.5, 138.2, 152.2, 166.2, 172.6; IR (KBr): 3282, 3029, 2923, 2852, 1740, 1642, 1534, 1441, 1280, 1215, 1174 cm^{-1} ; HRMS calcd. for $\text{C}_{34}\text{H}_{32}\text{N}_4\text{NaO}_6$ m/z 615.2220, found m/z 615.2215.

Synthesis of **W1**

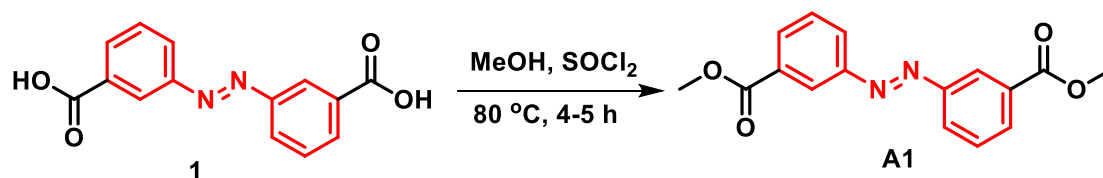
To an ice-cooled solution of tryptophan methyl ester hydrochloride (0.500 g, 1.96 mmol) in 20 mL of dry DCM, triethylamine (0.55 mL, 3.97 mmol) was added and stirred for about 10 min. Then the solution of **2** (0.301 g, 0.98 mmol) in 30 mL of dry DCM was gently added to it dropwise over a period of 30 minutes. The resultant mixture was stirred for 12 h at room temperature. After completion of reaction, the reaction mixture was washed with 0.2 N H_2SO_4 , saturated aq. NaHCO_3 solution and water. The organic layer was collected and dried over anhydrous Na_2SO_4 and evaporated under vacuum to obtain the crude product, which was purified by silica gel column chromatography (Ethyl acetate/Hexane in 5:5) to obtain 0.500 g of **W1** as a yellow powder.

Yield: 76 %

MP: 196–198 °C; $[\alpha]_D^{21} = -106^\circ$ (c 0.05 g/mL, DMSO)

^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 3.29 (m, 4H), 3.68 (s, 6H), 4.73 (br s, $J = 8$ Hz, 2H), 6.99 (t, $J = 7.5$ Hz, 2H), 7.06 (t, $J = 7.5$ Hz, 2H), 7.22 (s, 2H), 7.33 (d, $J = 7.5$ Hz, 2H), 7.58 (d, $J = 7.5$ Hz, 2H), 7.72 (t, $J = 7.5$ Hz, 2H), 8.03 (d, $J = 7.5$ Hz, 2H), 8.09 (d, $J = 7.5$ Hz, 2H), 8.38 (s, 2H), 9.12 (d, $J = 7.5$ Hz, 2H), 10.85 (s, 2H); ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$) δ 27.1, 52.5, 54.5, 110.4, 111.9, 118.5, 118.9, 121.5, 122.0, 124.1, 126.0, 127.5, 130.2, 131.0, 135.5, 136.6, 152.2, 166.1, 172.9; IR (KBr): 3405, 3298, 3056, 2924, 2853, 1746, 1642, 1528, 1457, 1437, 1353, 1276, 1212, 1175 cm^{-1} ; HRMS calcd. for $\text{C}_{38}\text{H}_{34}\text{N}_6\text{NaO}_6$ m/z 693.2438, found m/z 693.2435.

Synthesis of A1



Scheme S2: Synthetic scheme of A1.

To an ice-cooled solution of azobenzene-3,3'-dicarboxylic acid **1** (0.500 g, 1.85 mmol) in 10 mL of methanol, SOCl_2 (1.34 mL, 18.50 mmol) was added dropwise over a span of 20–30 min. The reaction mixture was heated at 80 °C in an oil bath for 4 h. After that, methanol was evaporated to get the product.

Yield: quantitative

MP: 157-159 °C

^1H NMR (400 MHz, CDCl_3) δ 3.98 (s, 6H), 7.62 (br td, $J = 8$ Hz, $J = 0.4$ Hz, 2H), 8.13 (ddd, $J = 8$ Hz, $J = 2$ Hz, $J = 2$ Hz, $J = 1.2$ Hz, 2H), 8.18 (ddd, $J = 8$ Hz, $J = 2$ Hz, $J = 1.2$ Hz, 2H), 8.60 (br td, $J = 2$ Hz, $J = 0.4$ Hz, 2H); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 52.48, 124.24, 127.19, 129.36, 131.47, 132.17, 152.46, 166.55; IR (KBr): 3427, 3020, 2962, 2925, 1729, 1435, 1298, 1270, 1207, 1152, 1098, 1072 cm^{-1} ; HRMS calcd. for $\text{C}_{16}\text{H}_{15}\text{N}_2\text{O}_4$ m/z 299.1032, found m/z 299.1032.

Methods: -

1. Scanning Electron Microscopy (SEM):

The sample solution (1 mg/mL) prepared in 1:1 MeOH/CHCl₃ mixture, was drop-casted onto the glass slide attached to the stub by carbon tape. The solution was allowed to dry in the air and coated with 10 nm of gold. All the samples were analyzed using ZEISS EVO 50 microscope. For the imaging of *cis* isomer, the same sample solution was first irradiated with 365 nm light and then drop-casted onto the glass slide attached to the stub by carbon tape.

2. High Resolution-Transmission Electron Microscopy (HR-TEM):

Samples for HR-TEM were prepared by dissolving the 1 mg of compound in 1:1 MeOH/CHCl₃ mixture. A 5 μL aliquot of the sample solution was placed on a 200-mesh copper grid, and samples were viewed using a TECHNAI G2 (20S-TWIN) electron microscope. For the imaging of *cis* isomer, the same sample solution was first irradiated with a 365 nm and then placed on a 200-mesh copper grid.

3. Atomic Force Microscopy (AFM):

Solutions of the compounds were prepared by dissolving 1 mg of each compound in 1 mL of a 1:1 MeOH/CHCl₃ mixture. Approximately 10 μL of the solution was drop-casted onto a glass slide and allowed to dry at room temperature. The samples were then examined using a BRUKER Dimension Icon XR Atomic Force Microscope in a tapping mode. The AFM images acquired at room temperature were further processed by using ImageJ software.

4. UV–vis photoisomerization study:

All the solutions were made at a concentration of 25 μM in DMSO. Agilent Technologies Cary 60 UV-vis spectrophotometer with a 1 cm quartz cell length was used to record the UV-vis spectra for each irradiation duration. The percentage conversion of the *trans*-to-*cis* isomer was determined by monitoring the change in absorbance at ~ 325 nm, using the following equation ^[1]:

$$\%cis = \frac{A_{trans} - A_{cis}}{A_{trans}} \times 100 \dots\dots\dots (\text{Eq. S1})$$

Where, % *cis* = Fraction of *cis*-isomer upon UV-light (365 nm) irradiation.

A_{trans} = Absorbance maximum at ~ 325 nm before irradiation. A_{cis} = Absorbance maximum at ~ 325 nm after the photostationary state (PSS) is obtained upon UV-irradiation.

5. ¹H NMR photoisomerization study:

A 1×10^{-3} M solution of the pseudopeptides **L1** and **F1** was prepared in CDCl_3 , while **W1** was prepared in $\text{DMSO-}d_6$. The solutions were irradiated with 365 nm light, and the resultant changes in the ¹H NMR spectra were monitored. To revert the systems to the *trans* form, the same solutions were subsequently irradiated with visible light (400–500 nm). For each pseudopeptide, the *trans* and *cis* isomer ratios were determined by integrating the α -proton signals at 4.92 and 4.76 ppm for **L1**, 5.14 and 5.00 ppm for **F1**, and 4.74 and 4.63 ppm for **W1**, respectively.

6. Confocal Microscopic Studies:

Solutions of compounds were prepared by dissolving them in 1:1 MeOH/ CHCl_3 mixture. About 10 μL of the solution was drop-casted onto a rectangular glass coverslip and imaged by FV3000, Olympus Confocal Microscope. While imaging, all the volatile solvents were evaporated and a thin layer of residue was formed on the coverslip. To study the effects of UV-light irradiation, some of the sample solution was exposed to 365 nm light for 30 minutes, drop-casted onto the coverslip

and imaged by confocal microscope. We used a 405 nm laser for the excitation and took time lapse images of different time intervals with the help of 100× objective lens. The images were captured at room temperature and were processed using ImageJ software. The average number density was calculated using the thresholding and particle analysis functions in ImageJ software.

Additionally, for the confocal microscopic analysis of the *cis* form of **W1**, two 1 mg/mL solutions of **W1** were prepared in a 1:1 MeOH/CHCl₃ solvent mixture. To one of these solutions, 0.04 equiv. of rhodamine B dye were added. The mixture was vortexed for 30 seconds to ensure uniform mixing. The resulting solutions were then irradiated with 365 nm light to obtain the *cis* form. Subsequently, approximately 20 μL of the irradiated samples were drop-casted onto 20 × 60 mm glass slides and dried. The prepared sample was continuously irradiated with visible light for 1 minute, during which confocal images were captured using an Olympus FV1200 microscope equipped with a UPlanSApo 100× objective lens. Excitation laser of 488 nm wavelength was used. Furthermore, a time-lapse video of the complete 1-minute irradiation process was recorded to capture and analyse the real-time morphological transformations.

7. Dynamic Light Scattering:

Solutions of the compounds were prepared by dissolving 1 mg of each compound in 1 mL of a 1:1 MeOH/CHCl₃ mixture. Dynamic light scattering (DLS) studies were performed using a Malvern Zetasizer ZS 90 unit at 25 °C, which was fitted with a 633 nm “red” laser. Quartz cuvettes were used for the measurements, with the path length of 1 cm. The data were analyzed using OriginPro 2020b.

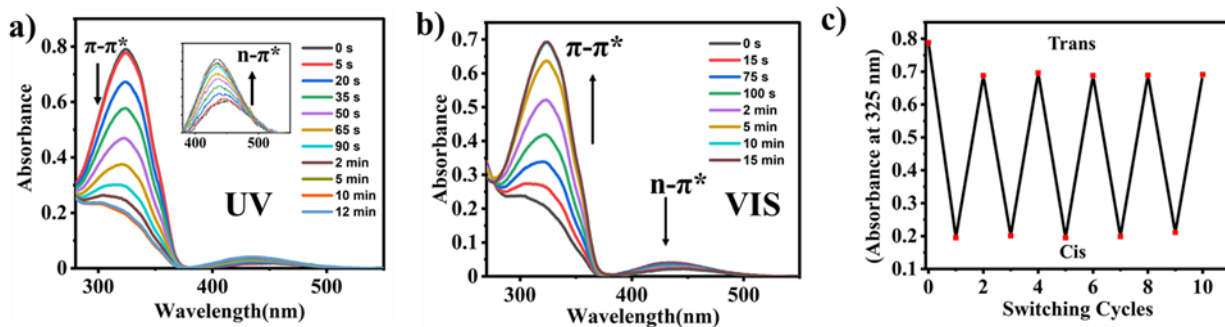


Figure S1: UV-vis absorption spectra of **L1** upon a) UV irradiation at 365 nm, inset is zoomed view of absorbance corresponding to $n-\pi^*$ in the region of 400-500 nm, b) Visible light irradiation at 400-500 nm, c) Photoswitchability of **L1** with alternating UV and vis irradiation.

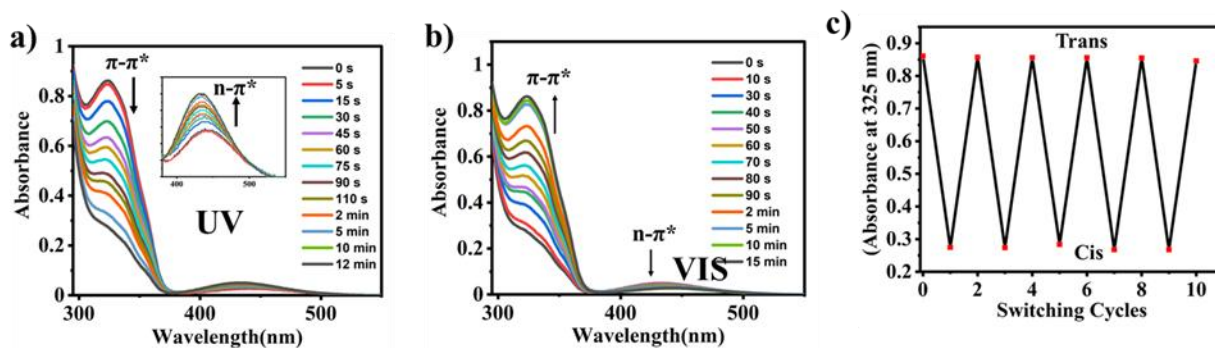


Figure S2: UV-vis absorption spectra of **W1** upon a) UV irradiation at 365 nm, inset is zoomed view of absorbance corresponding to $n-\pi^*$ in the region of 400-500 nm, b) Visible light irradiation at 400-500 nm, c) Photoswitchability of **W1** with alternating UV and vis irradiation.

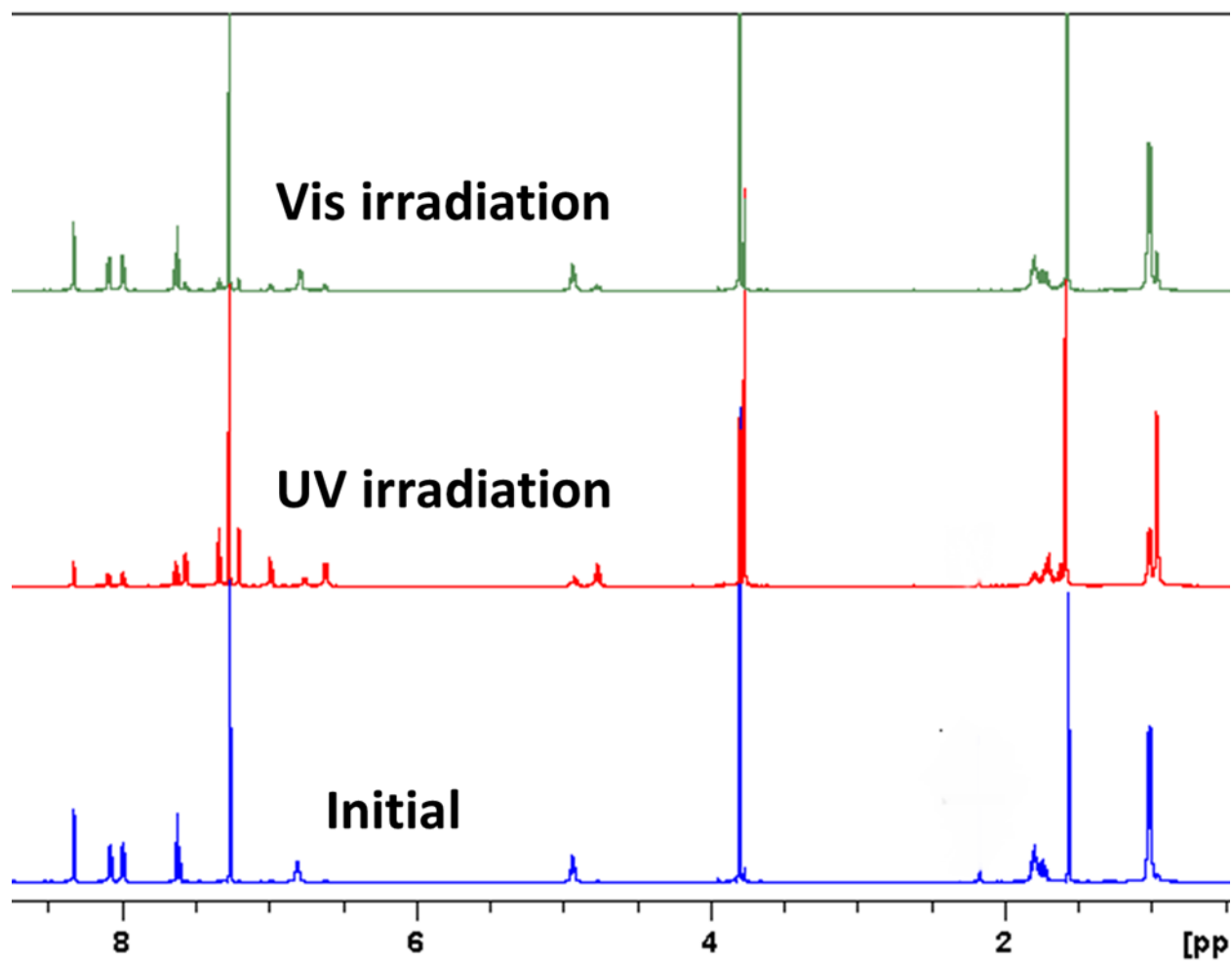


Figure S3: ¹H NMR (400 MHz, CDCl₃) spectra of L1 showing the photoisomerization upon UV (365 nm) and vis light (400-500 nm) irradiation.

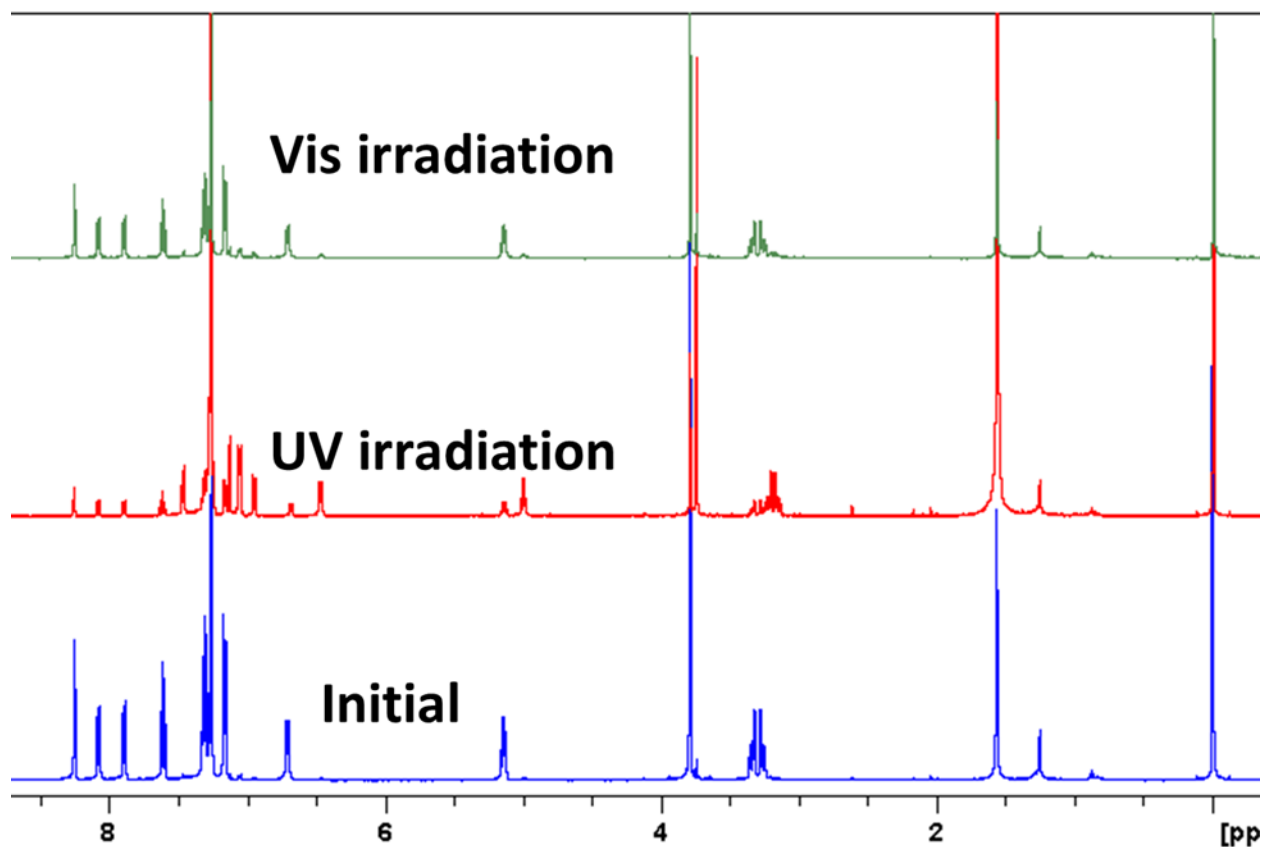


Figure S4: ¹H NMR (400 MHz, CDCl₃) spectra of F1 showing the photoisomerization upon UV (365 nm) and vis light irradiation (400-500 nm).

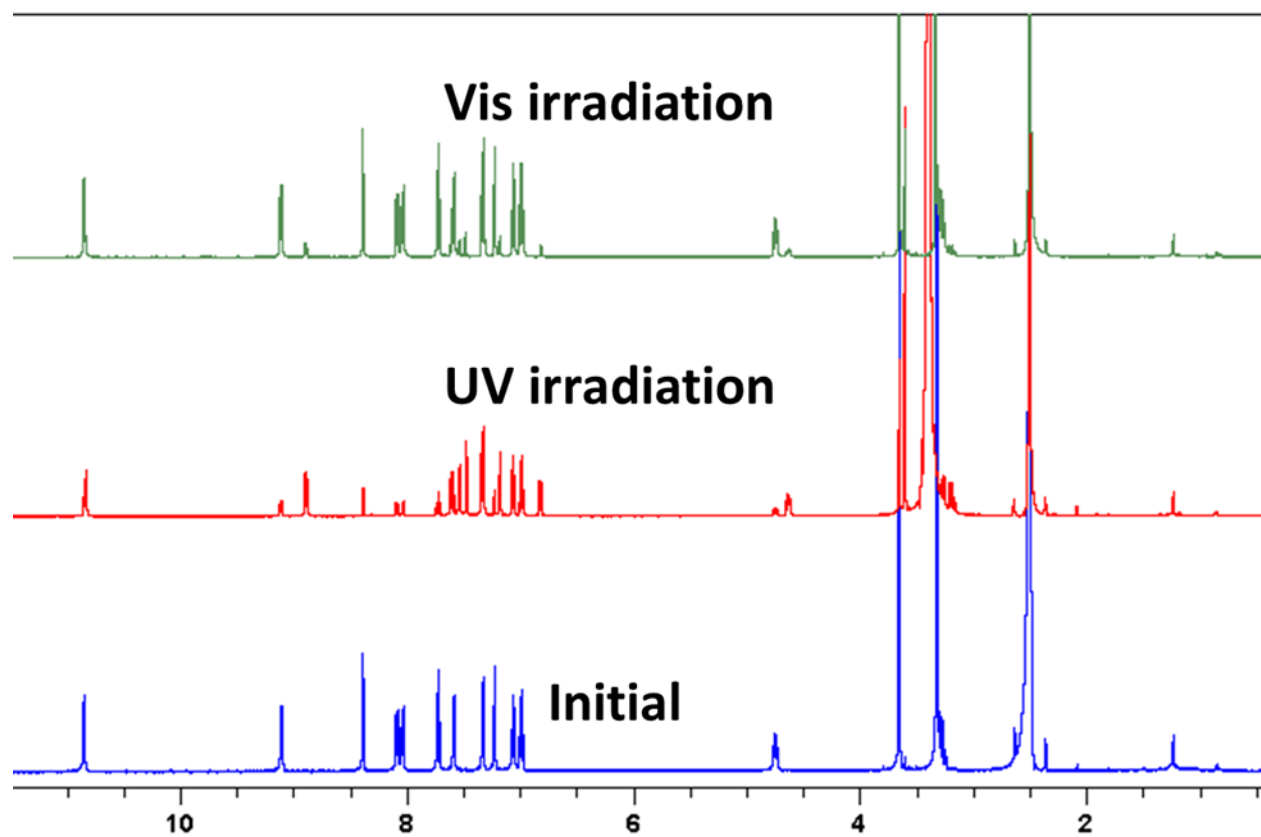


Figure S5: ^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectra of **W1** showing the photoisomerization upon UV (365 nm) and vis light irradiation (400-500 nm).

Table S1: Percentage conversions of *trans* to *cis* conformer for **L1**, **F1**, and **W1** as calculated using ^1H NMR spectra after irradiation with 365 nm light.

Compound	% <i>Trans</i>	% <i>Cis</i>
L1	26	74
F1	27	73
W1	27	73

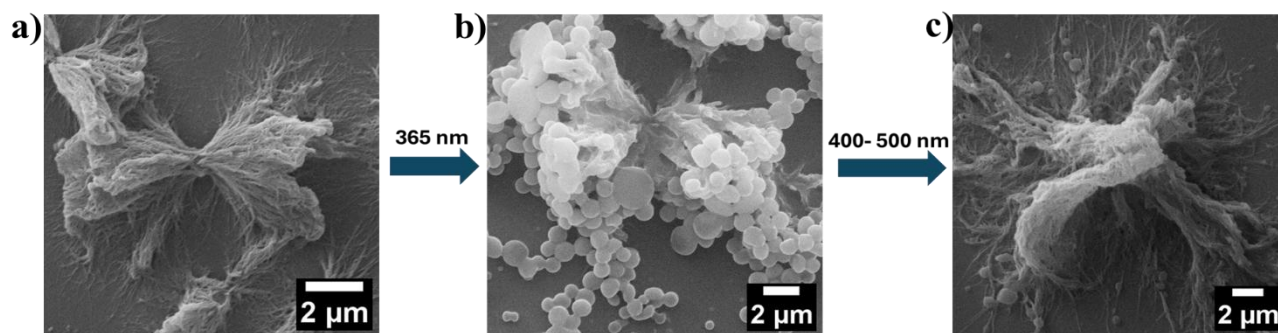


Figure S6: SEM images showing the reversible morphological transformation of **F1** (1 mg/mL in 1:1 MeOH/ CHCl_3). (a) Initial morphology before irradiation, (b) after UV irradiation at 365 nm, and (c) upon subsequent visible light irradiation (400–500 nm).

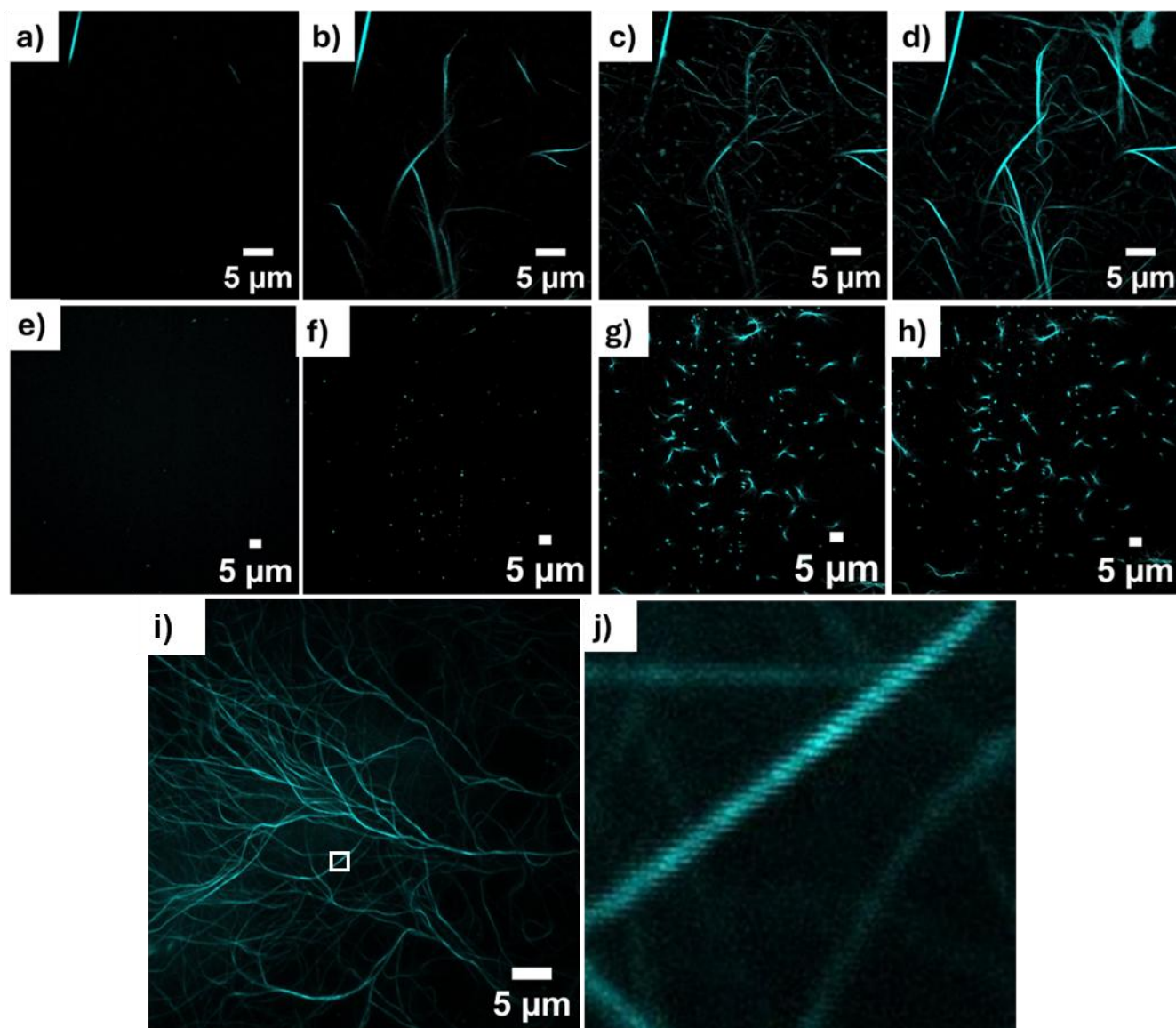


Figure S7: Confocal images illustrating the light-responsive morphological transformation of F1 assembly. (a–d) Images captured before light irradiation reveal the formation of well-defined fibrous networks. (e–h) post-irradiation images showing a transition from purely fibrous structures to a coexistence of fibrous and vesicular morphologies, at different time interval suggesting light-induced structural reorganization, (i) A magnified view of the fibrous assembly highlighting, the fibers with noticeable helical twists, (j) A high-resolution confocal image of a single fiber strand.

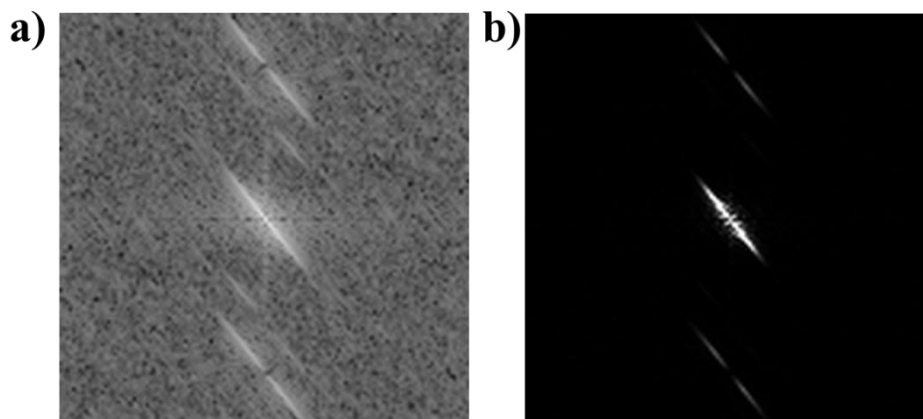


Figure S8: (a) FFT spectrum of the image shown in Fig. S7j, and (b) the corresponding power spectrum derived from the FFT analysis.

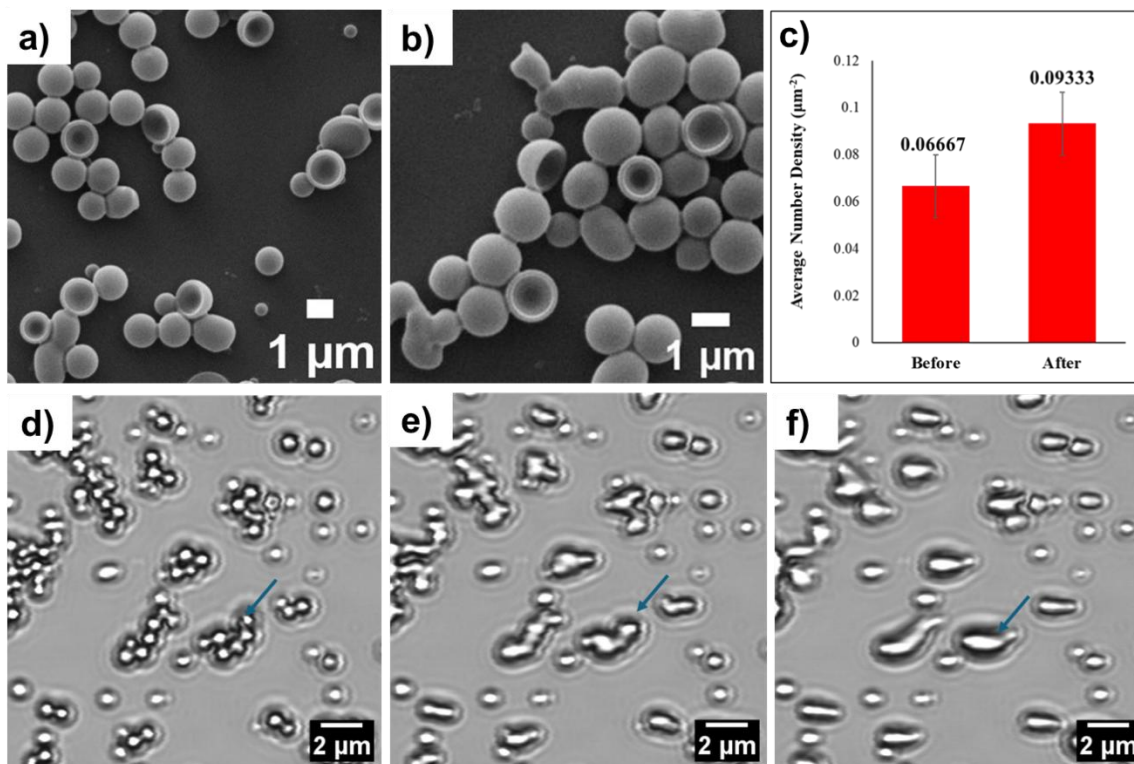


Figure S9: (a, b) SEM images of W1 recorded at a concentration of 0.8 mg/mL in a 1:1 MeOH/CHCl₃ solvent system. (c) Bar graph showing the average number density of vesicles (μm⁻²) before and after UV irradiation (d-f) Confocal bright field images of *cis*-W1 (1 mg/mL in 1:1 MeOH/CHCl₃ mixture) at different time intervals showing the fusion of vesicles upon excitation at ~465 nm light.

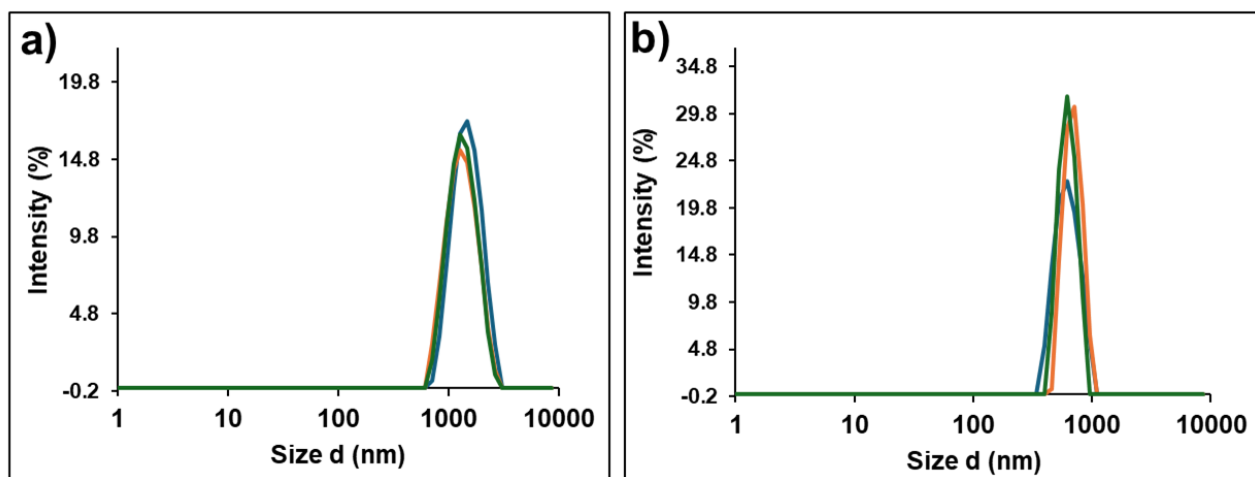


Figure S10: DLS measurement of **W1** (a) before irradiation, (b) after irradiation with 365 nm light.

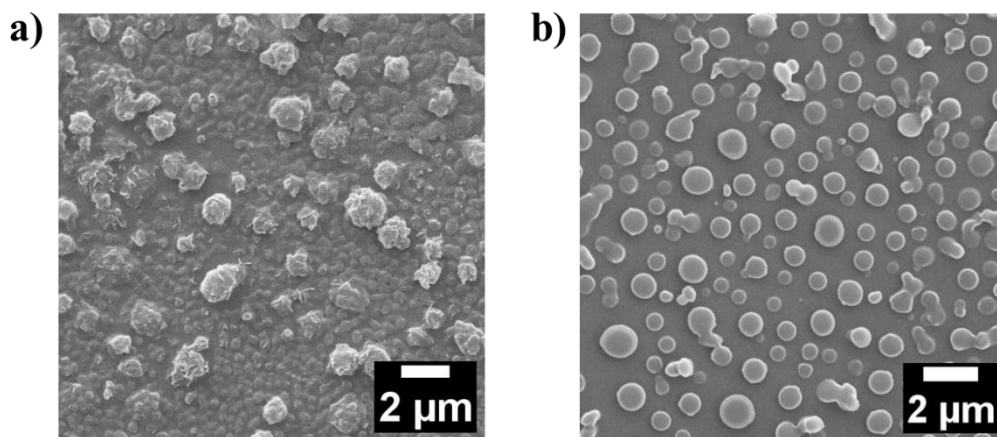


Figure S11: SEM microscopy showing the light induced morphological transformation in **L1** (1:1 MeOH/CHCl₃ at 1 mg/mL) a) Before UV-irradiation b) after irradiation with 365 nm light.

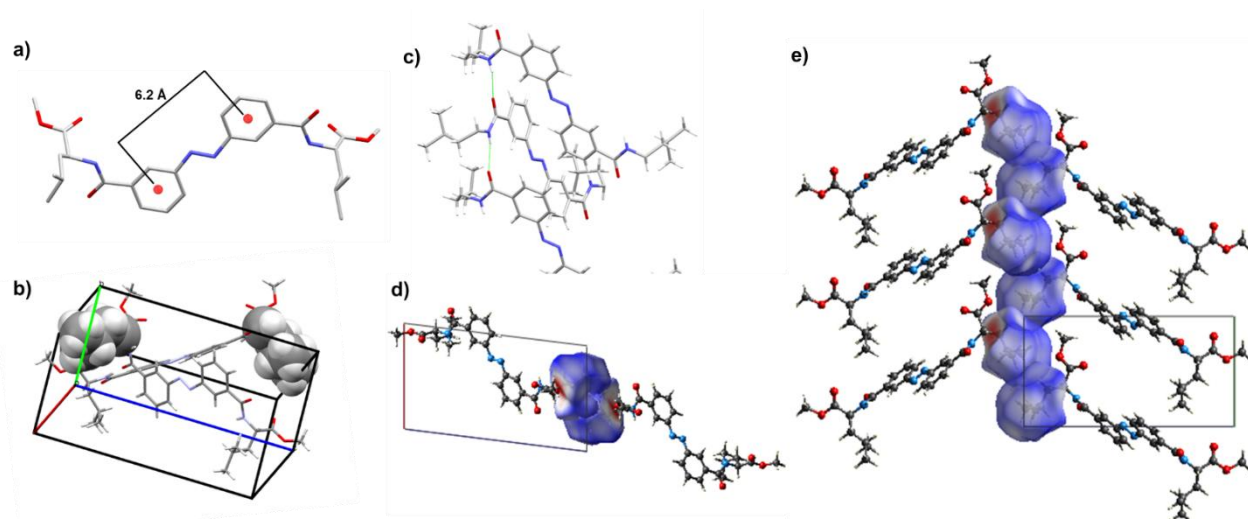


Figure S12: (a) Crystal structure of **L1** exhibiting a linear conformation, with two benzene rings 6.2 Å apart (b) unit cell of **L1** containing two molecules in the *trans* configuration, with the leucine side chain involved in the zipper formation are shown in the space-fill model (c) intermolecular hydrogen bonding between the amide carbonyl group of one molecule and the amide N–H of the other, (d) van der Waals radii overlap diagram, where the lighter regions indicate electron density overlap between the leucine side chains, (e) van der Waals radii overlap diagram of **L1** viewed along the b-c plane, showing a leucine–leucine zipper-like packing arrangement. Semi-transparent van der Waals surfaces are drawn around the molecular framework to indicate regions, where neighbouring leucine residues approach distances close to, or shorter than, the sum of their van der Waals radii. The lighter regions indicate electron density overlap between the leucine side chains, highlighting close intermolecular contacts that contribute to the stabilization of the zipper-like stacking motif. The diagram was generated using Crystal Explorer 17.5 with standard van der Waals radii to visualize non-bonded intermolecular interactions.

Table S2: Crystal data and structure refinement for **L1** (CCDC 2528782).

Identification code	DALO
Empirical formula	C ₂₈ H ₃₆ N ₄ O ₆
Formula weight	524.621
Temperature/K	299.00
Crystal system	monoclinic
Space group	P2 ₁
a (Å)	9.9662(8)
b (Å)	8.6628(7)
c (Å)	17.4082(14)
α (°)	90
β (°)	97.003(2)
γ (°)	90
Volume (Å ³)	1491.7(2)
Z	2
ρ _{calc} (g/cm ³)	1.168
μ (mm ⁻¹)	0.083
F (000)	560.3
Crystal (size/mm ³)	0.22 × 0.08 × 0.11
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection (°)	4.12 to 56.48
Index ranges	-13 ≤ h ≤ 13, -10 ≤ k ≤ 11, -23 ≤ l ≤ 23
Reflections collected	16037
Independent reflections	6910 [R _{int} = 0.0425, R _{sigma} = 0.0725]
Data/restraints/parameters	6910/1/350
Goodness-of-fit on F ²	1.055
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0666, wR ₂ = 0.1491
Final R indexes [all data]	R ₁ = 0.1396, wR ₂ = 0.1879
Largest diff. peak/hole (e Å ⁻³)	0.31/-0.25

Flack parameter

0.3(7)

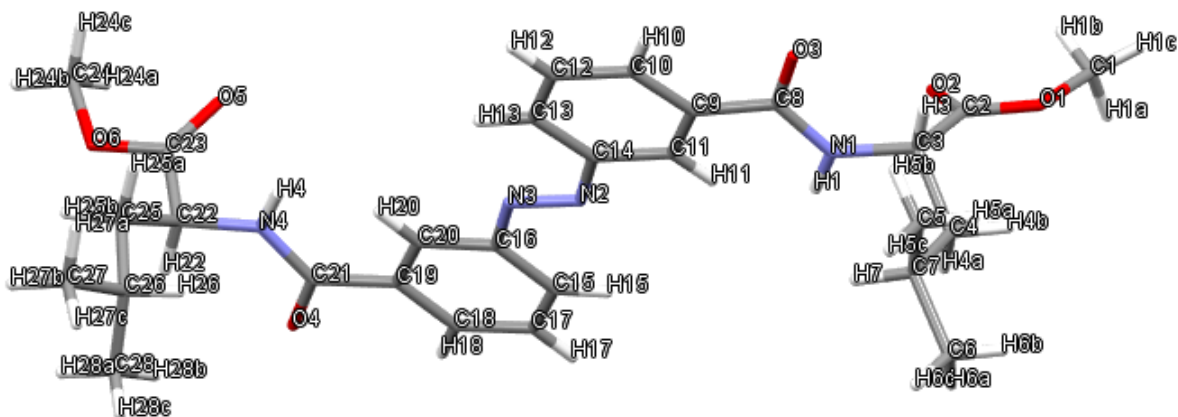


Table S3: Details related to intermolecular H-bonding interactions and CH- π interaction present in **L1**.

Donor (D)	H Label (H)	Acceptor (A)	DH...A (Å)	D...A (Å)	< D...H...A Angle (°)
N4	H4	O3	2.073	2.903	161.91
N1	H1	O4	2.036	2.856	159.16
C15	H15	C15C16C17C18C19C20	3.263	4.041	142.57

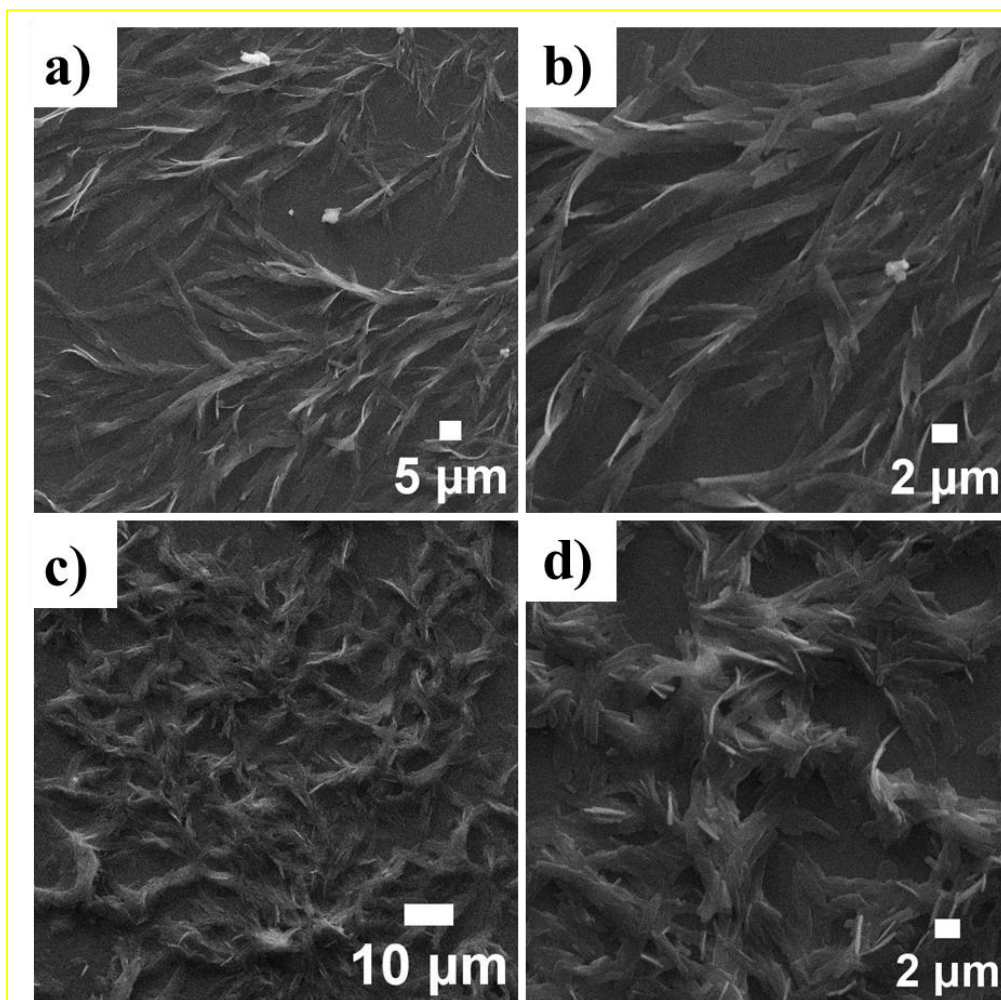
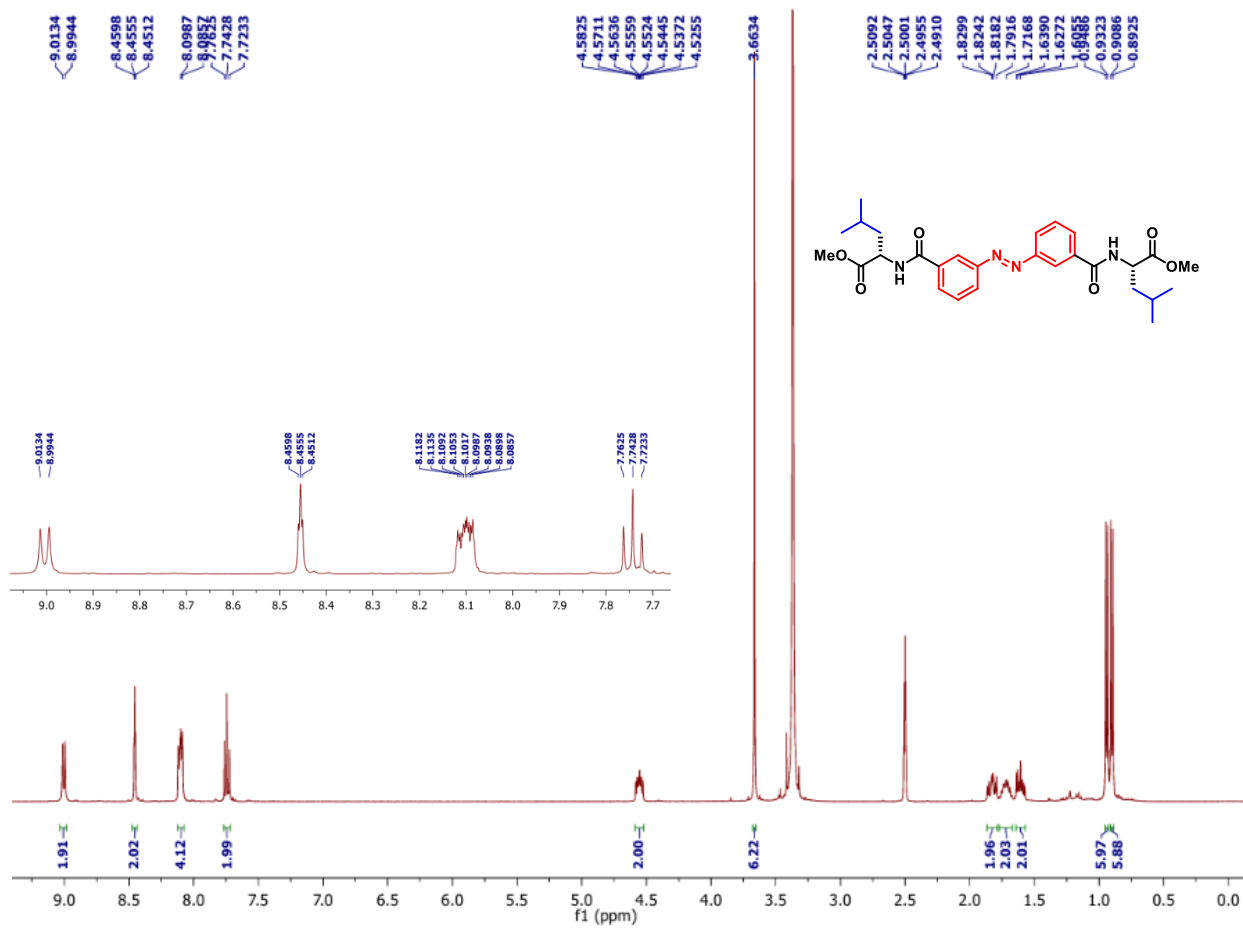
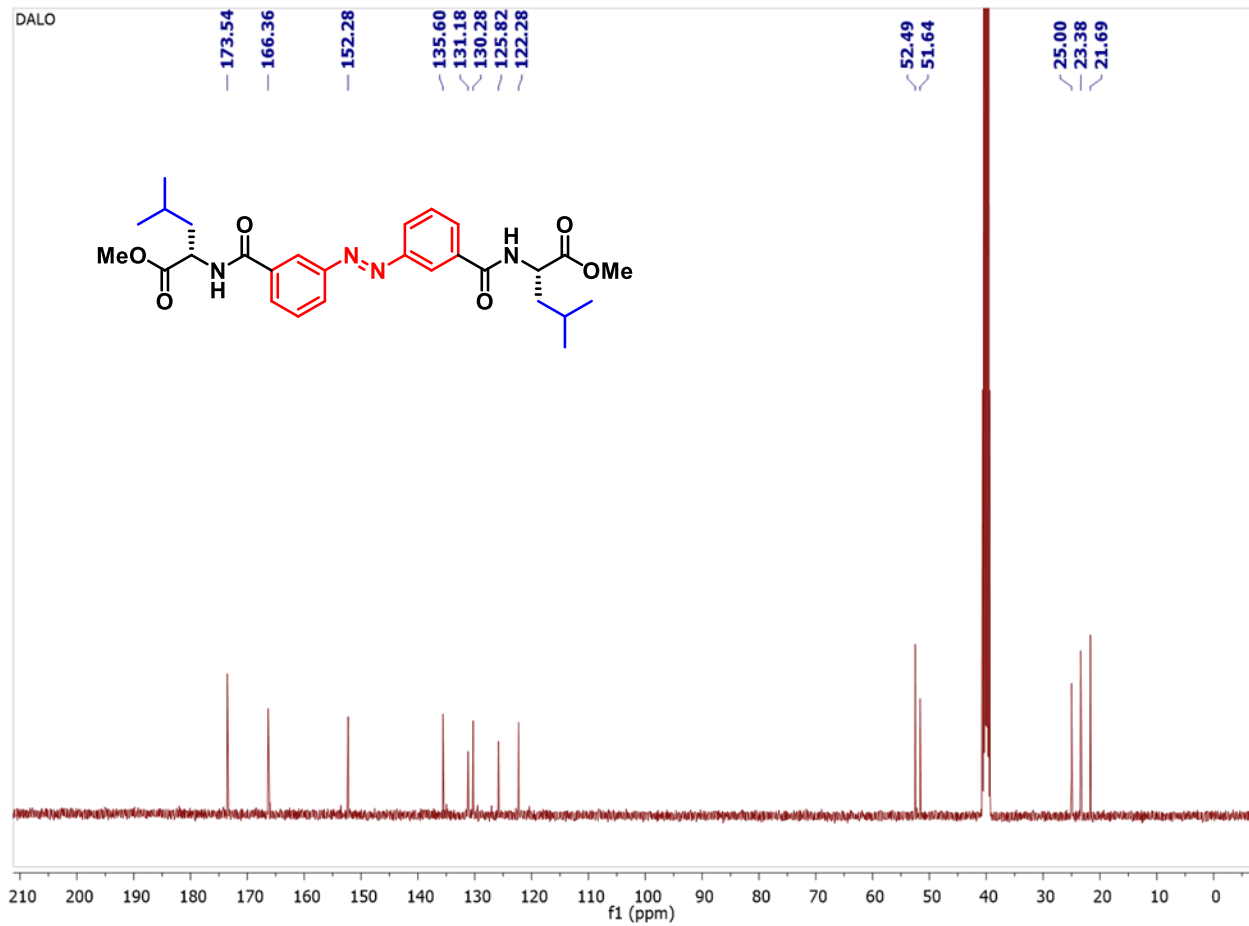


Figure S13: SEM images of A1 (1 mg/mL in 1:1 MeOH/CHCl₃) (a, b) before irradiation and (c, d) after irradiation with 365 nm light.



¹H NMR (400 MHz, DMSO-*d*₆) of L1



^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) of L1

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions

343 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-28 H: 0-42 N: 0-4 O: 0-8 Na: 0-1 S: 0-2

XEVO-G2XSQTOF#TFC2176

Capillary V 3, Cone V 40, Desolvation Gas 800

POSITIVE ION MODE

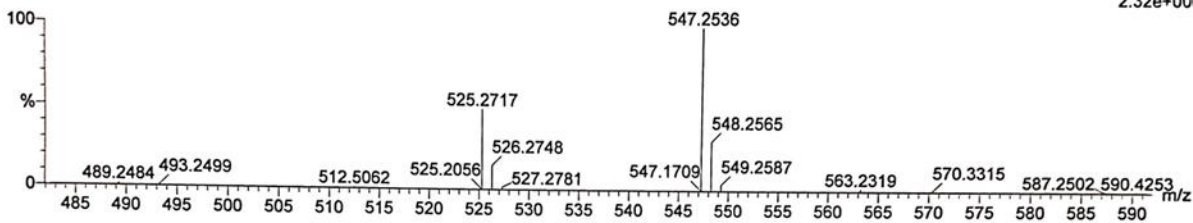
ESI

25-Jun-2021

VH 2

25062025_2 13 (0.276)

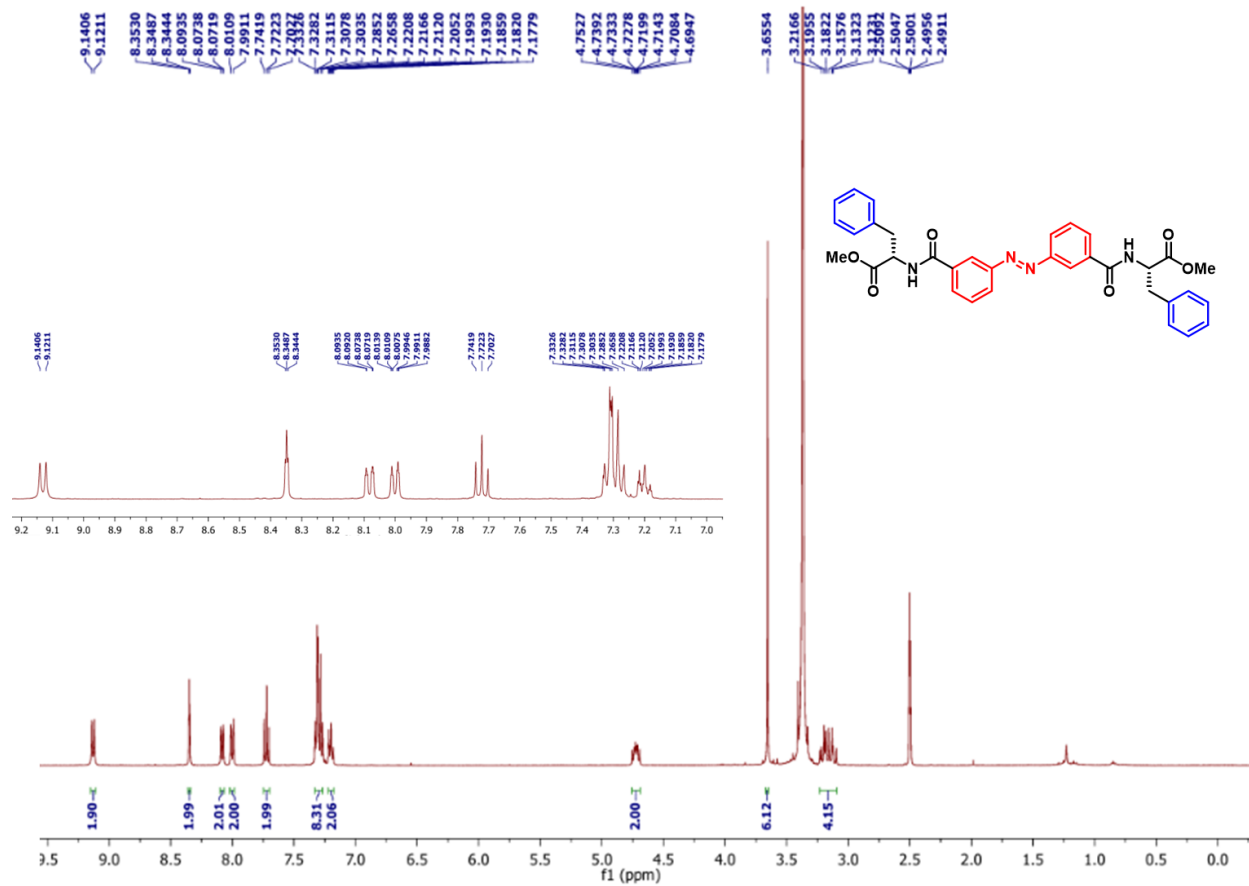
1: TOF MS ES+
2.32e+006



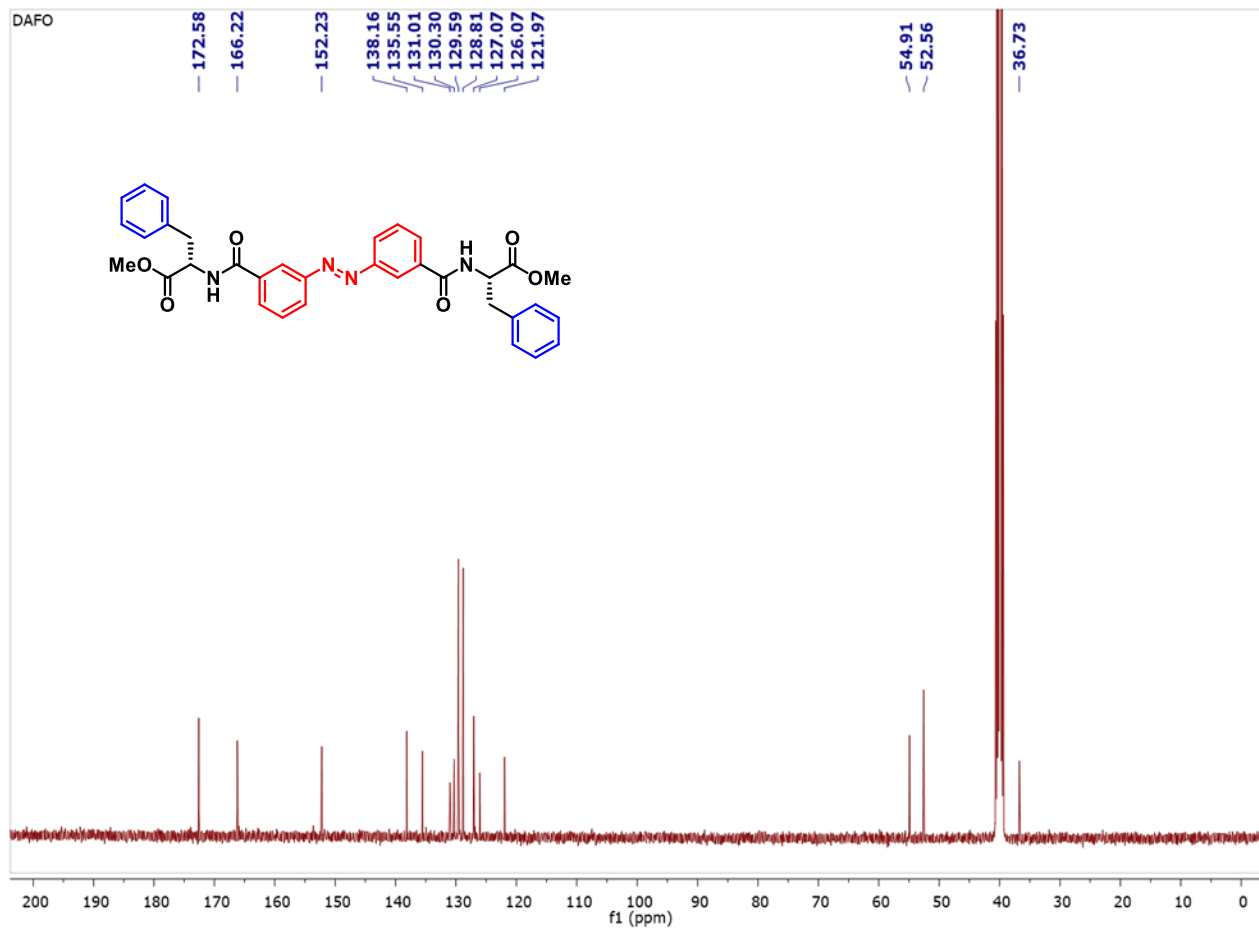
Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
547.2536	547.2533	0.3	0.5	12.5	400.1	n/a	n/a	C28 H36 N4 O6 Na

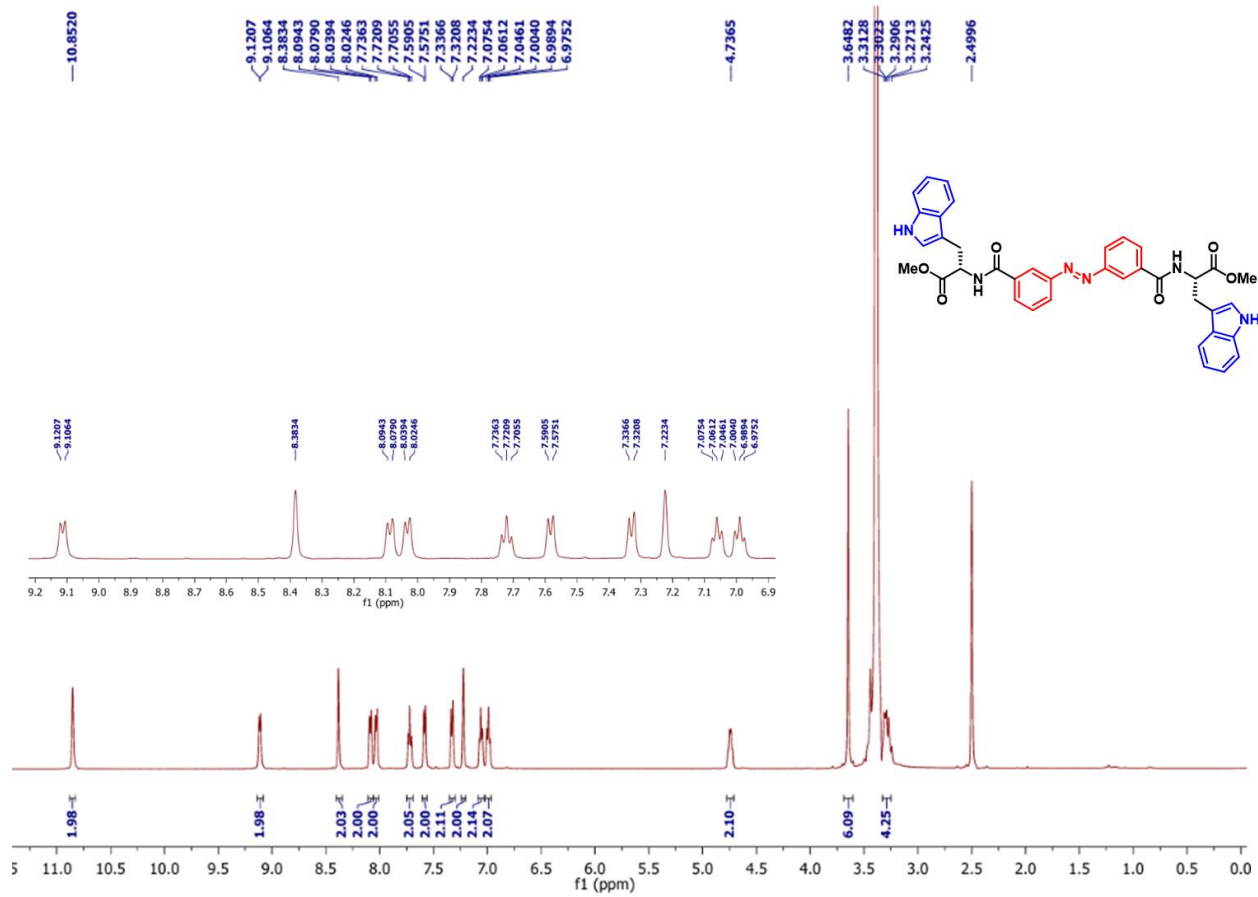
HRMS of L1



¹H NMR (400 MHz, DMSO-*d*₆) of F1

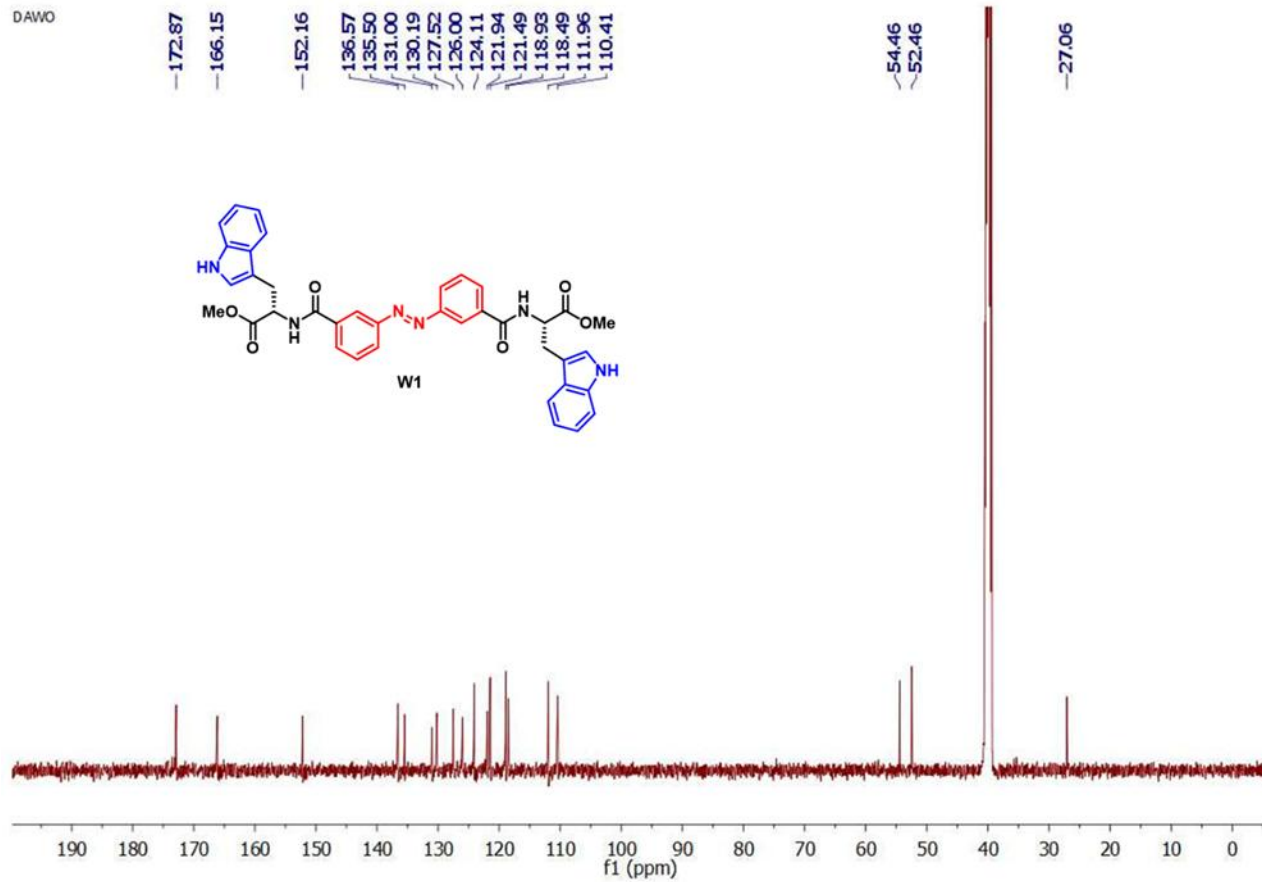


^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) of F1



¹H NMR (500 MHz, DMSO-d₆) of W1

DAWO



^{13}C NMR (125 MHz, $\text{DMSO-}d_6$) of **W1**

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

106 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-38 H: 0-42 N: 0-6 O: 0-8 Na: 0-1

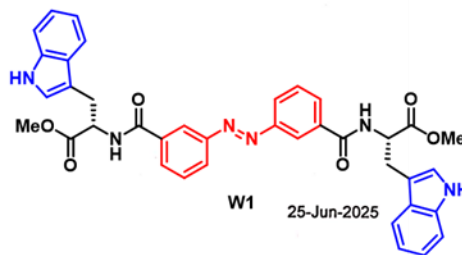
XEVO-G2XSQTOF#TFC2176

Capillary V 3, Cone V 40, Desolvation Gas 800
ESI

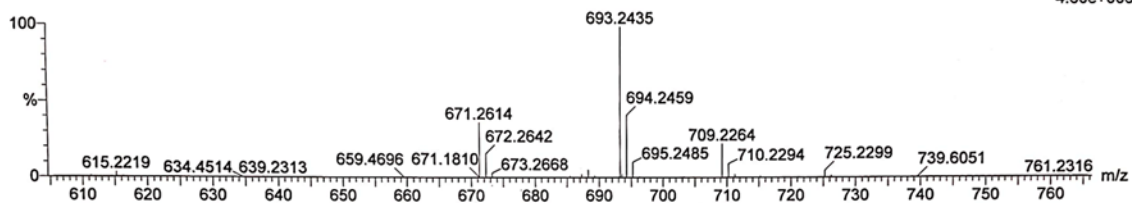
POSITIVE ION MODE

VH 4

25062025_4 47 (0.930)



1: TOF MS ES+
4.60e+006

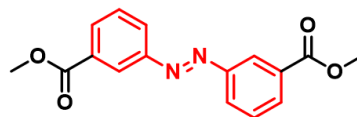
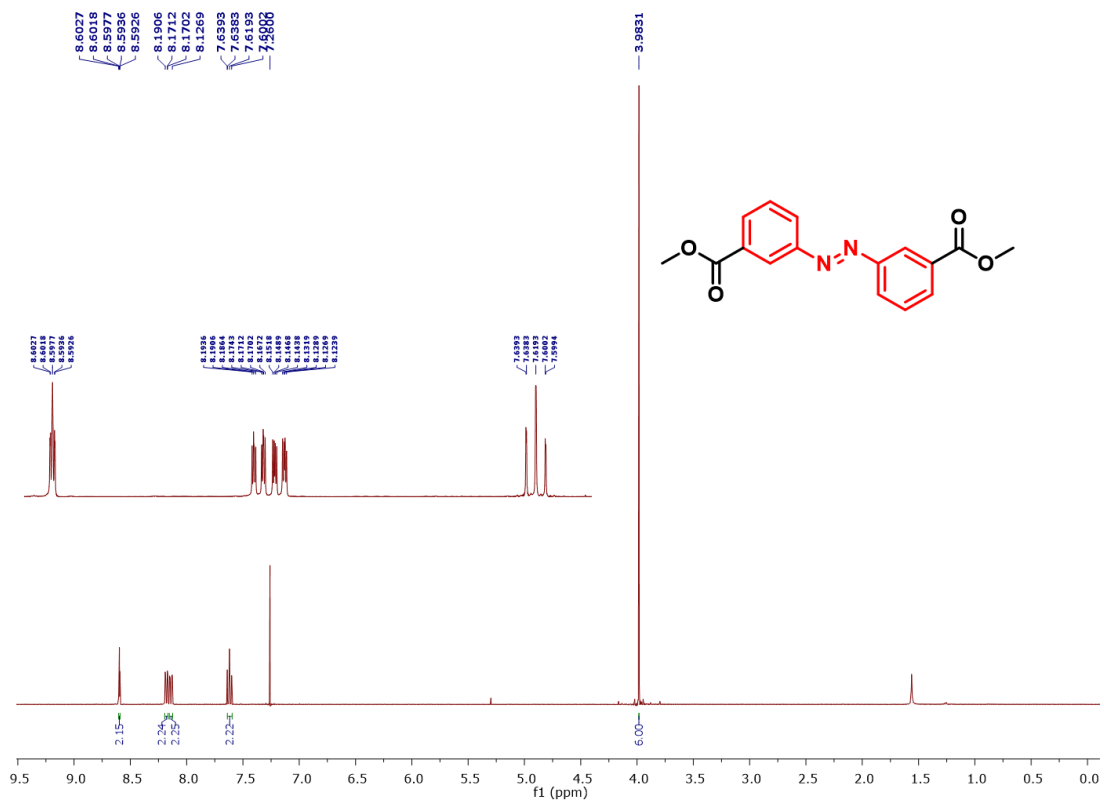


Minimum:

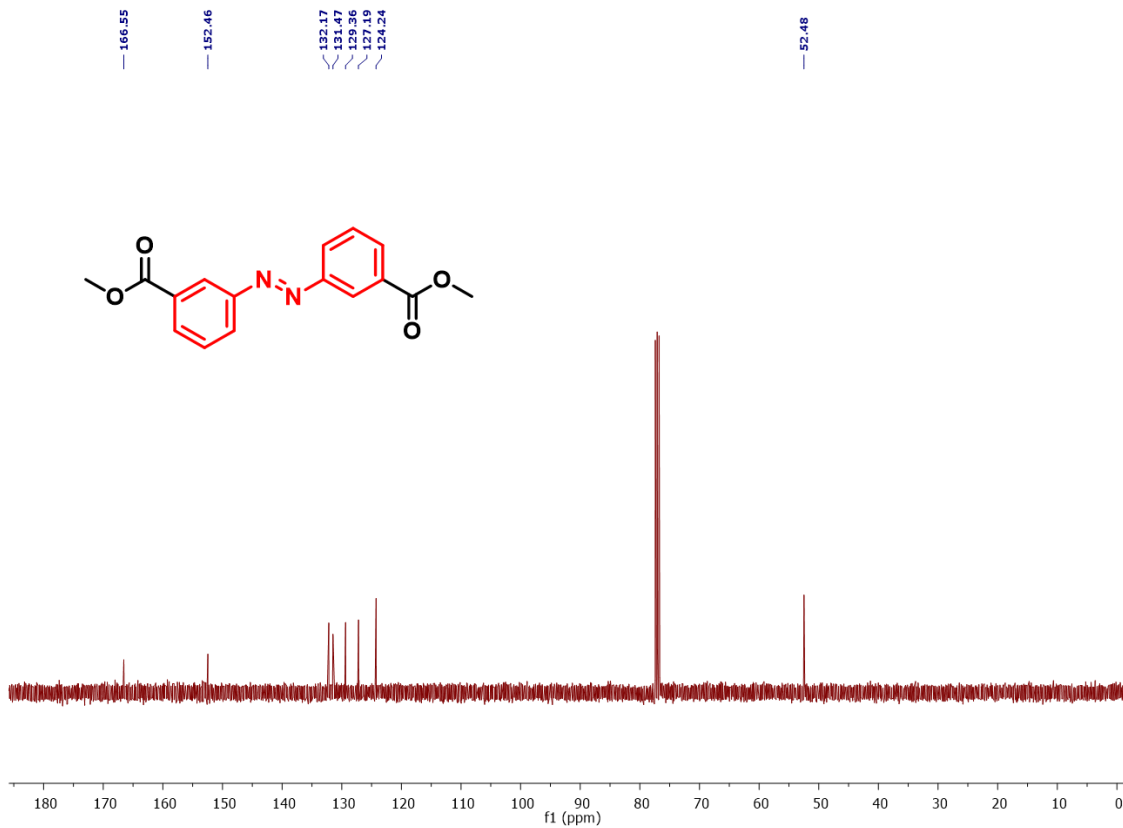
Maximum: 5.0 5.0 -1.5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
693.2435	693.2438	-0.3	-0.4	24.5	348.0	n/a	n/a	C38 H34 N6 O6 Na

HRMS of W1



¹H NMR (400 MHz, CDCl₃) of A1



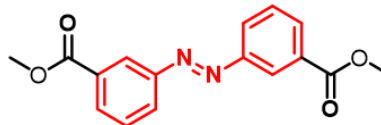
¹³C NMR (100 MHz, CDCl₃) of A1

Single Mass Analysis

Tolerance = 8.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions

285 formula(e) evaluated with 4 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-30 H: 0-25 N: 0-6 O: 0-8 Na: 0-1

XEVO -G2XSQTOF#TFC2176

Capillary V 3, Cone V 40, Desolvation Gas 800
ESI

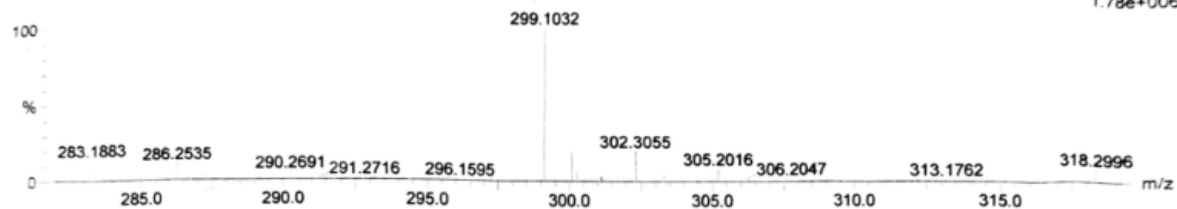
04-May-2026

POSITIVE ION MODE

JV DAAOME

04052026_14 40 (0.792)

1: TOF MS ES+
1.78e+006



Minimum: -1.5
Maximum: 5.0 8.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
299.1032	299.1032	0.0	0.0	10.5	932.2	0.003	99.66	C16 H15 N2 O4
	299.1021	1.1	3.7	12.5	938.5	6.308	0.18	C15 H12 N6 Na
	299.1045	-1.3	-4.3	15.5	938.9	6.659	0.13	C17 H11 N6
	299.1048	-1.6	-5.3	11.5	940.5	8.298	0.02	C19 H16 O2 Na

HRMS of A1

References:

[1] G. Das, T. Prakasam, M. A. Addicoat, S. K. Sharma, F. Ravaux, R. Mathew, M. Baias, R. Jagannathan, M. A. Olson, A. Trabolsi, “Azobenzene-Equipped Covalent Organic Framework: Light-Operated Reservoir” *J. Am. Chem. Soc.* **2019**, *141*, 19078–19087.