

Electronic Supporting Information

A Molecular Chalice With Hydrophobic Walls And Hydrophilic Rim: Self-Assembly And Complexation Properties

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General Remarks.

All chemicals were purchased from Aldrich Chemicals in their highest purity and used without further purification. D₂O (99.95% isotopic purity), DMSO-d⁶ or CDCl₃ were also purchased from Aldrich Chemicals. All solvents and liquid reagents were degassed by bubbling nitrogen for 15 min before each use or by two freeze-pump-thaw cycles before use. The GC analyses were performed on a TRACE GC Ultra (Thermo Scientific) coupled with a Windows® XP workstation.

NMR Measurements.

NMR experiments were recorded on Advance 400 Bruker, at 400.13 and 75.49 MHz, respectively, in the indicated solvents. All NMR experiments (1D experiments, Job's plot) were obtained by the use of the sequence commercially available on Bruker spectrometer. Chemical shifts are given in ppm (δ) and measured relative to residual solvent.

ESI/HRMS

Mass spectral data were obtained by the Université de Montréal Mass Spectrometry Facility and were recorded on a Mass spectrometer TSQ Quantum Ultra (Thermo Scientific) with accurate mass options instrument.

Compounds 1 to were were prepared following the synthetic procedure previously described: R. P. Sijbesma, R. J. M. Nolte *Recl. Trav. Chim. Pays Bas* **1993**, *112*, 643.

Diphenylglycoluril (1). To a solution of urea (6g, 100 mmol) and benzil (10.5g, 50 mmol) in toluene (200 mL) was added dropwise trifluoroacetic acid (10 mL). The reaction mixture was heated at reflux overnight and cooled to room temperature. 40 mL of ethanol was added to the mixture. After being stirred for 15 minutes, the mixture was filtered, and the substrate was washed with 40 mL of ethanol. The filtrate was solubilised in 100 mL of DMSO and poured in 500 mL of ethanol. The mixture was stirred for 15 minutes and filtered. The substrate was washed with 40 mL of ethanol to give white crystals (7.132g, 48%): ¹H NMR (400 MHz, DMSO-d⁶) δ 7.74 (s, 3 H, ArH), 7.04 (s, 7 H, ArH); ¹³C NMR (100 MHz, DMSO-d⁶) δ 160.25 (C=O), 137.83 (Ar), 127.29 (Ar), 126.87 (Ar), 126.56 (Ar), 81.30 (C-Ar).

1,3,4,6-Bis(2-oxapropylene)tetrahydro-3a,6a-diphenylimidazo[4,5-d]-imidazole-2,5(1H,3H)-dione (2). To a solution of diphenylglycoluril (1) (4g, 13.6 mmol) and

paraformaldehyde (2.04g, 68 mmol) in 20 mL of DMSO was added a solution of NaOH 1M until pH reaches 9, then the mixture was stirred at room temperature overnight. Concentrated HCl was added dropwise until the pH reaches 1 then the solution was stirred at 100°C for 2 hours. After cooling to room temperature the mixture was filtered. The solid was washed with water and dried in a vacuum desiccator to give white crystals (3.3393g, 65%). ¹H NMR (400 MHz, DMSO-d⁶) δ 7.17 (s, 10 H, ArH), 5.46 (d, 4 H, *J*_{gem} = 11.4 Hz, NCHHO), 4.56 (d, 4 H, *J*_{gem} = 11.4 Hz, NCHHO); ¹³C NMR (100 MHz, DMSO-d₆) δ 159.09 (C=O), 133.68 (Ar), 130.15 (Ar), 129.49 (Ar), 128.89 (Ar), 80.08 (C-Ph), 72.71 (NCH₂O).

1,3,4,6-tetrakis(acetyloxy)methyltetrahydro-3a,6a-diphenyl-imidazo[4,5-d]imidazole-2,5(1H,3H)-dione (3). A solution of (2) (3g, 7.94 mmol) and paratoluene sulfonic acid (0.3g, 1.75 mmol) in acetic anhydride (13 mL) was stirred at 110°C for 3 hours. After cooling down to room temperature, 13 mL of diethyl ether were added to the solution then the mixture was stirred at room temperature for 10 minutes. The mixture was filtered and the substrate was washed with diethyl ether to give white crystals (3.2501g, 70%). ¹H NMR (400 MHz, CDCl₃) δ 7.16 (t, 2 H, *J* = 7.2 Hz, ArH), 7.06 (t, 4 H, *J* = 7.2 Hz, ArH), 6.82 (d, 4 H, *J* = 7.2 Hz, ArH), 5.70 (d, 4 H, *J*_{gem} = 11.5 Hz, NCHHOAc), 5.26 (d, 4 H, *J*_{gem} = 11.5 Hz, NCHHOAc), 2.02 (s, 12 H, COCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 169.78 (CH₃C=OO), 156.40 (NC=ON), 130.86 (Ar), 129.76 (Ar), 128.35 (Ar), 128.06 (Ar), 87.01 (C-Ph), 66.81 (NCH₂O), 20.65 (CH₃).

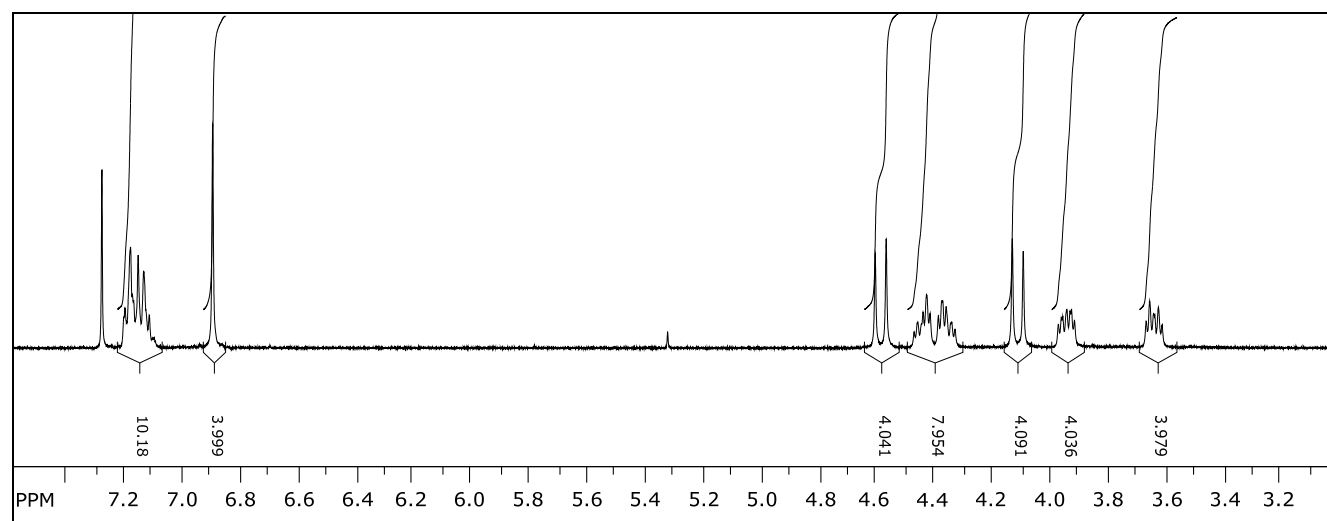
1,3,4,6-tetrakis(chloromethyl)tetrahydro-3a,6a-diphenyl-imidazo[4,5-d]imidazole-2,5(1H,3H)-dione (4). A solution of (3) (1g, 1.72 mmol) and thionyl chloride (1.26 mL, 17.34 mmol) in 1.5 mL of dry dichloromethane was stirred at room temperature overnight, then filtered. The substrate was washed with diethyl ether to give white crystals (724mg, 86%). ¹H NMR (400 MHz, CDCl₃) δ 7.25 (t, 2 H, *J* = 7.5 Hz, ArH), 7.15 (t, 4 H, *J* = 7.5 Hz, ArH), 6.91 (d, 4 H, *J* = 7.5 Hz, ArH), 5.36 (d, 4 H, *J*_{gem} = 11.53 Hz, NCHHCl), 5.27 (d, 4 H, *J*_{gem} = 11.3 Hz, NCHHCl); ¹³C NMR (100 MHz, CDCl₃) δ 154.11(C=O), 130.47 (Ar), 128.99 (Ar), 128.79 (Ar), 128.34 (Ar), 87.26 (C-Ph), 52.60 (NCH₂Cl).

Molecular chalice (5). To a solution of (4) (500mg, 1.02 mmol) and DB18C6 (553.6mg, 1.54 mmol) in 65 mL of anhydrous 1,2-dichloroethane under nitrogen atmosphere was added tin (IV) chloride (0.84 mL, 7.17 mmol). The solution was heated at reflux for 24h then cooled to room temperature. 10 mL of HCl 6N were added and the mixture was heated at reflux for 20

minutes. After cooling to room temperature, 20 mL of dichloromethane and 20 mL of water were added. Organic phase was separated and washed two times with 20 mL of HCl 1N, dried over magnesium sulphate and evaporated. The crude product was purified by chromatography on a silica gel column with an eluent CH₂Cl₂/MeOH 9:1. The yellow oil obtained was dissolved in dichloromethane. White crystals were obtained by diffusion of ether in the dichloromethane solution during 2 days (138.1 mg, 19%) : ¹H NMR (400 MHz, CDCl₃) δ 7.19-7.06 (m, 10 H, ArH), 6.87 (s, 4 H, Ar_{DB18C6}H), 4.54 (d, 4 H, *J*_{gem} = 15.2 Hz, NCHHAr_{DB18C6}), 4.45-4.36 (m, 4 H, CH₂), 4.36-4.28 (m, 4 H, CH₂), 4.07 (d, 4 H, *J*_{gem} = 15.2 Hz, NCHHAr_{DB18C6}), 3.95-3.86 (m, 4 H, CH₂), 3.64-3.55 (m, 4 H, CH₂); ¹³C NMR (100 MHz, CDCl₃) δ 157.54 (C=O), 145.67 (Ar_{DB18C6}O), 134.79 (Ar_{DB18C6}CH₂), 129.13 (Ph), 128.48 (Ph), 128.11 (Ph), 127.83 (Ph), 117.29 (Ar_{DB18C6}H), 85.27 (C-Ph), 68.02(OCH₂CH₂O), 67.51 (OCH₂CH₂O), 43.96 (NCH₂Ar_{DB18C6}). HR-MS: calcd for [M+H]⁺ C₄₀H₃₉N₄O₈ 703.27624, found 703.27689.

Dibenzo24-crown-8 crown (DB24C8) ether has been tried in the same quadruple Friedel-Crafts reaction. In this case only the mono and double-attached DB24C8 were obtained.

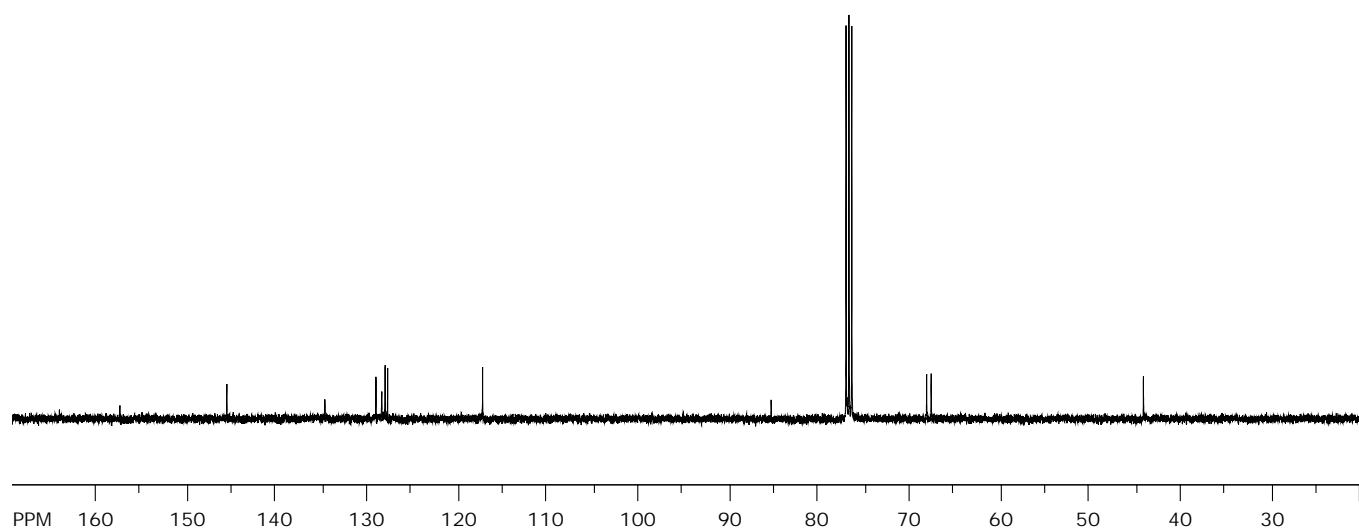
¹H NMR (400 MHz) of **5** in CDCl₃ at 298K.



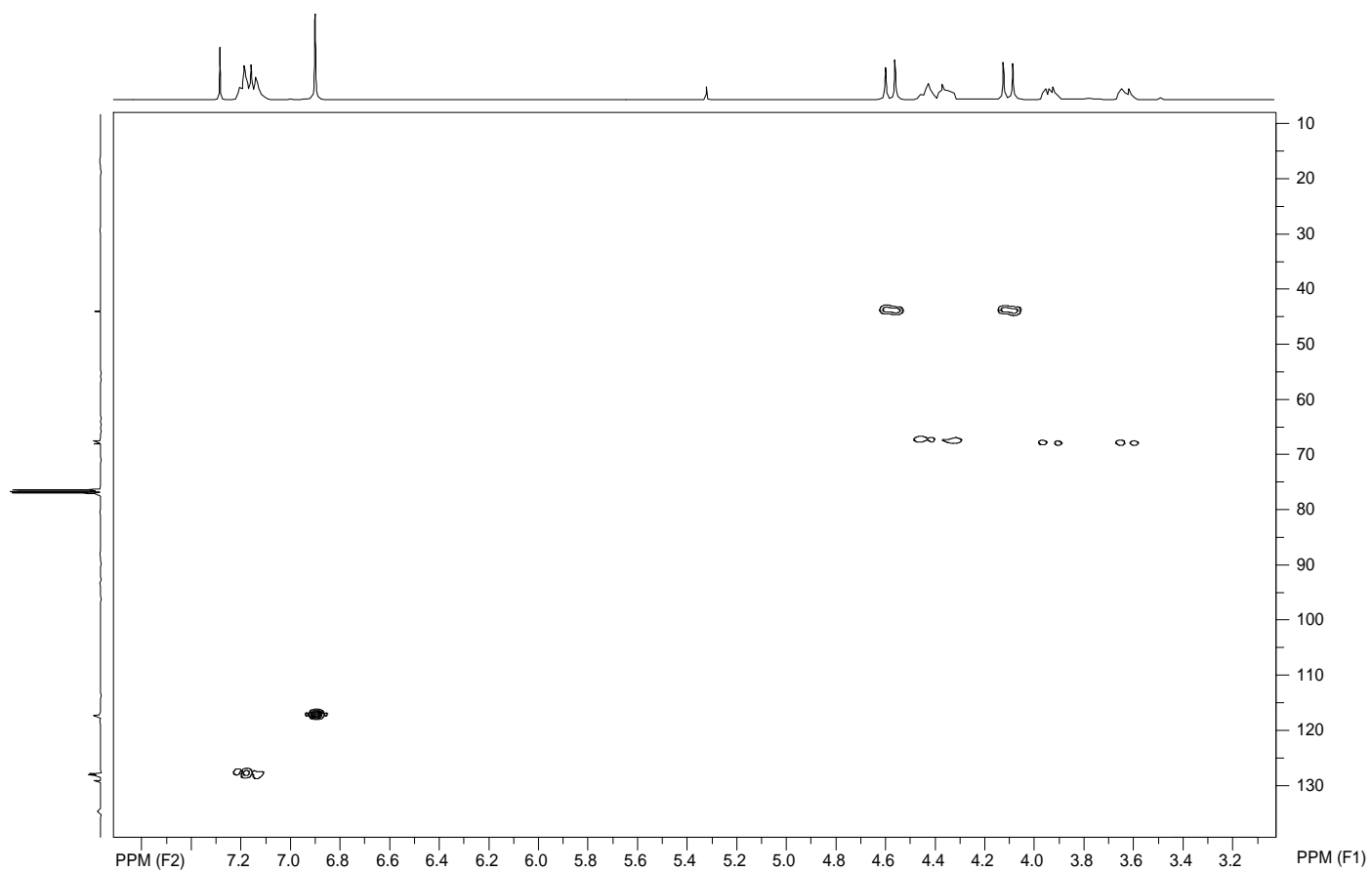
For the continuous variation method, solutions were prepared, where the sum of **5** and metal salt concentrations was kept constant (5 mM), but the concentrations of metal salt and **5** were systematically varied.⁹ The rate of complexation was fast on the NMR time scale, as only the induced chemical shift was observed in the ¹H NMR spectrum. The ¹H NMR spectrum of **5** shows four multiplets at 4.45-4.36 ppm, 4.36-4.28 ppm, 3.95-3.86 ppm and 3.64-3.55 ppm corresponding to the crown ether moiety. On the ¹³C NMR spectrum, carbons of the crown ether part of the molecule can be observed at 68.02 ppm for C₁ and 67.51 ppm for C₂. A 2D HMQC experiment allowed us to assign the two multiplets at 3.95-3.86 ppm and 3.64-3.55

ppm for the protons H_1 , while the two multiplets at 4.45-4.36 ppm and 4.36-4.28 ppm for protons H_2 .

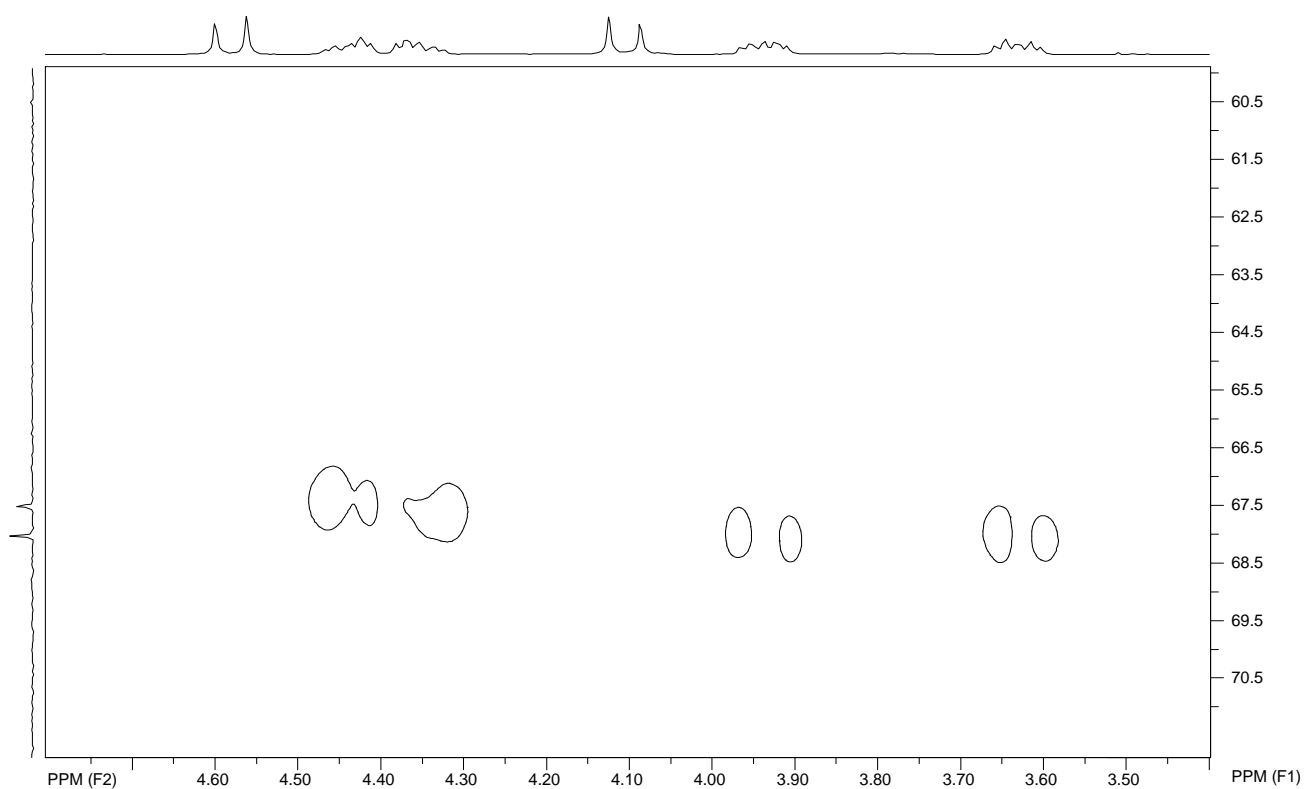
^{13}C NMR (100 MHz) of 5 in CDCl_3 at 298K.



HMQC of 5 in CDCl_3 at 298K



HMQC of 5 in CDCl₃ at 298K (close up view of crown ether moiety signals).



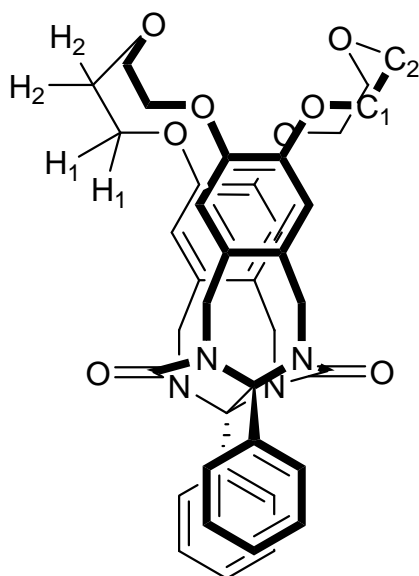


Figure S1. Continuous variation plot (Job's plot) derived from the ¹H NMR data for **5** and NaPF₆.

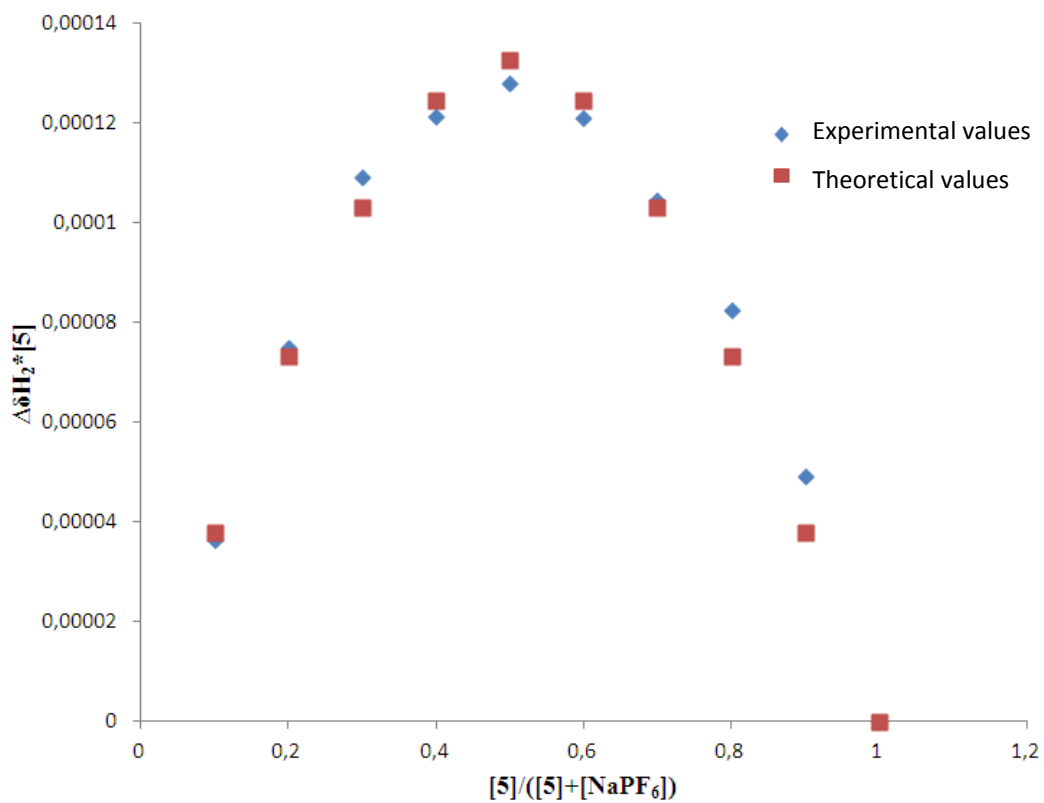
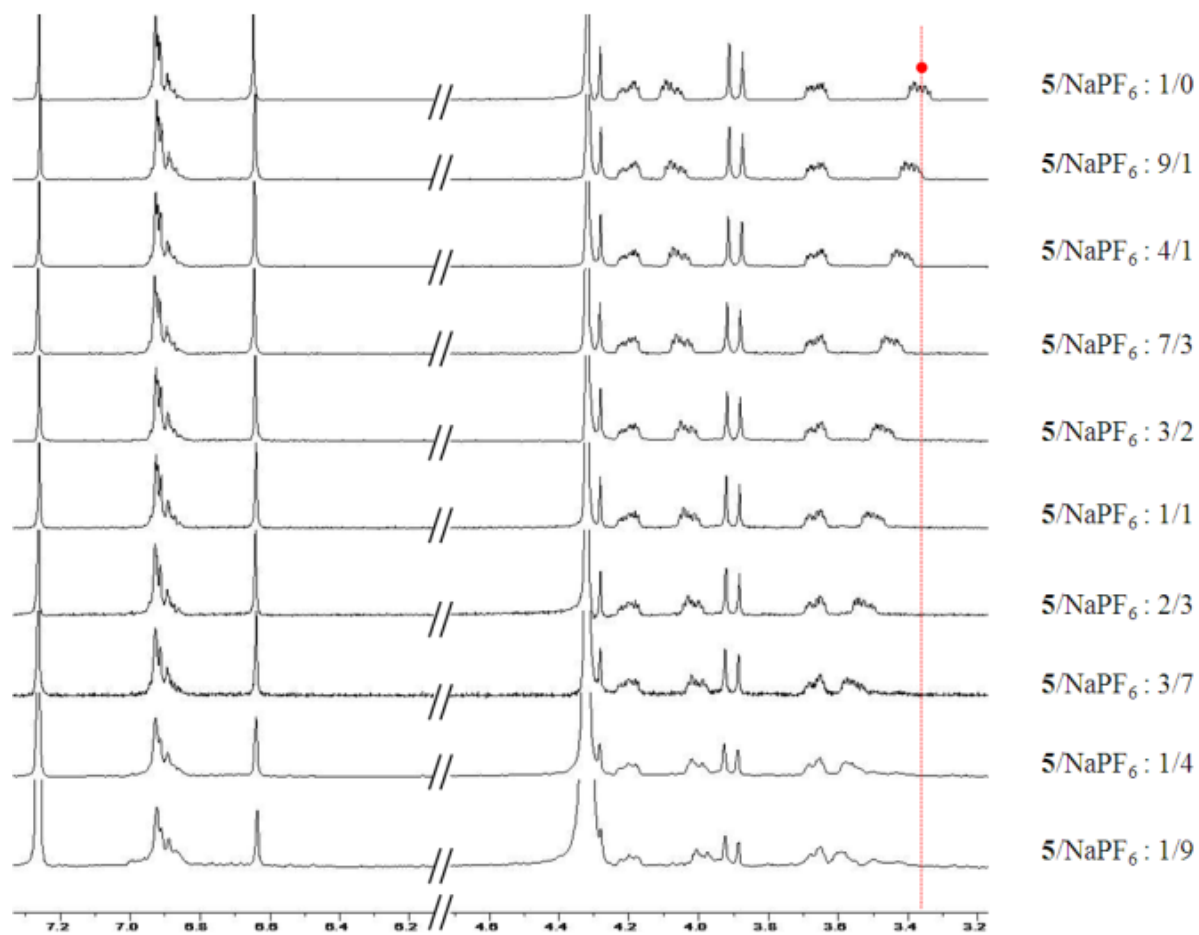
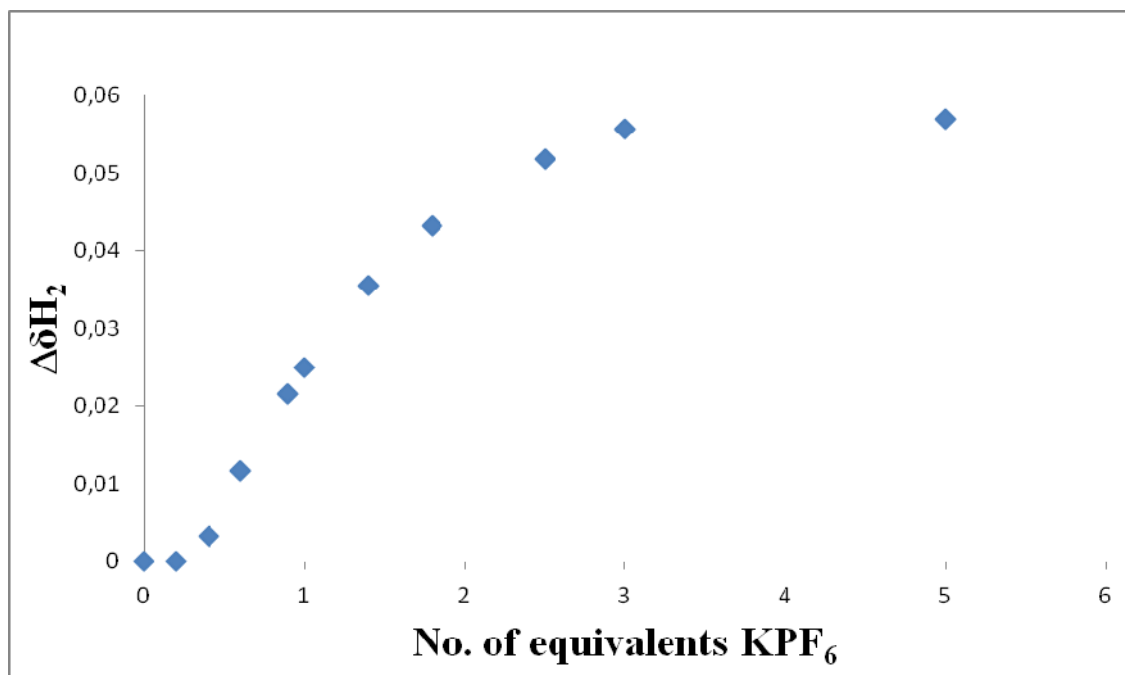
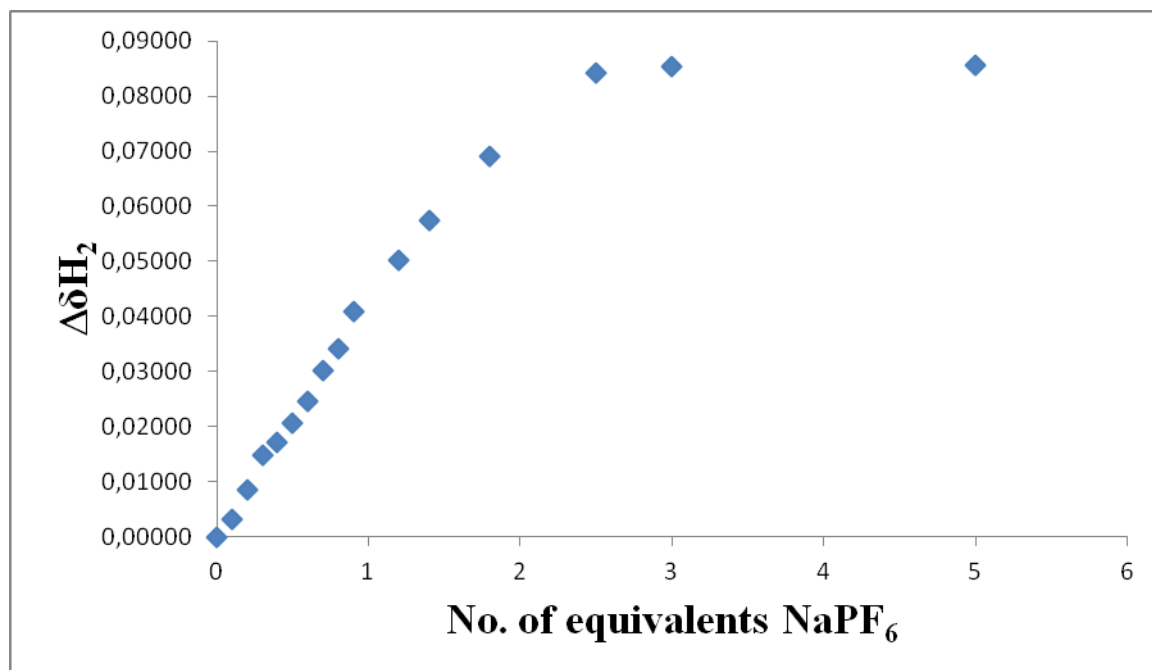


Figure S2: A. 1H NMR (400 MHz) titration a 5 mM solution of 5 in MeOD:CDCl₃ 3:5 by a 5 mM solution of NaPF₆ in MeOD:CDCl₃ 3:5.

A.

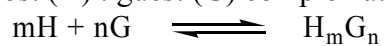


B. Binding isotherms for the 5 with NaPF₆ and KPF₆. Non-linear least-squares analysis for K_a determination (K. Connors, Binding Constants. The Measurement of Molecular Complex Stability; Ed. Wiley: New York, 1987.)



Calculation of the association constants from Job's plot data:

The general equation for the host (H) : guest (G) complex at the equilibrium is:



The association constant of this equilibrium is defined by equation 1:

$$K_a = \frac{[H_m G_n]}{[H]^m [G]^n}$$

During the continuous variation experiment, the total concentration C_t of G and H is constant and defined by the equation 2:

$$C_t = [H]_0 + [G]_0$$

where $[H]_0$ is the initial concentration of the host and $[G]_0$ is the initial concentration of the guest. During the experiment, initial concentrations of H and G are varied, and their ratio r is defined by the equation 3:

$$r = \frac{[H]_0}{[H]_0 + [G]_0}$$

The ratio x of the H:G complex can be defined by the equation 4:

$$x = \frac{m[H_m G_n]}{[H]_0}$$

The balance of the reaction can be written:

| | H | G | $H_m G_n$ |
|---------------|----------------------------|----------------------------|-------------|
| Initial state | $[H]_0$ | $[G]_0$ | |
| Equilibrium | $[H] = [H]_0 - m[H_m G_n]$ | $[G] = [G]_0 - n[H_m G_n]$ | $[H_m G_n]$ |

The association constant K_a can be written as equation 5:

$$K_a = \frac{[H_m G_n]}{([H]_0 - m[H_m G_n])^m ([G]_0 - n[H_m G_n])^n}$$

In the case of a 1:1 complex equation 5 becomes:

$$K_a = \frac{[HG]}{([H]_0 - [HG])([G]_0 - [HG])}$$

The relation between $[HG]$ and K_a can be written as the quadratic equation 6:

$$[HG] = K_a([H]_0 - [HG])([G]_0 - [HG])$$

which solution is:

$$[HG] = \frac{C_t + \frac{1}{K_a} - \sqrt{(C_t + \frac{1}{K_a})^2 - 4rC_t^2(1-r)}}{2}$$

Performing the continuous variation experiment by NMR, $\delta_{H,free}$ and $\delta_{H,comp}$ are defined as the chemical shifts of the H protons in its free form and in its complexed form.

$\delta_{H,free}$ was obtained by NMR at a concentration of 2.5 mM in a $CDCl_3/MeOD$ 5/3 mixture. $\delta_{H,comp}$ was obtained by NMR of the host at a concentration of 2.5 mM with 3 equivalents of $NaPF_6$ in a $CDCl_3/MeOD$ 5/3 solvent (which represents the solubility limits of $NaPF_6$).

In the case of a fast exchange, the observed chemical shift δ_{obs} is defined by the equation 7:

$$\delta_{obs} = x\delta_{H,comp} + (1-x)\delta_{H,free}$$

From 4 in equation 7:

$$\delta_{obs} = \frac{m[H_mG_n]\delta_{H,comp} + ([H]_0 - m[H_mG_n])\delta_{H,free}}{[H]_0}$$

This equation can be rearranged as to give equation 8:

$$[H]_0(\delta_{obs} - \delta_{H,free}) = m[H_mG_n](\delta_{H,comp} - \delta_{H,free})$$

In the case of a 1:1 complex, equation 8 becomes:

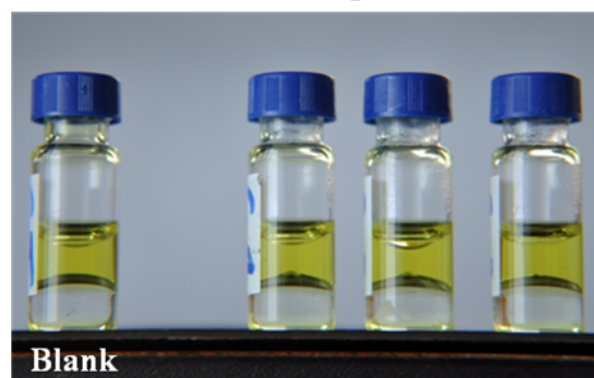
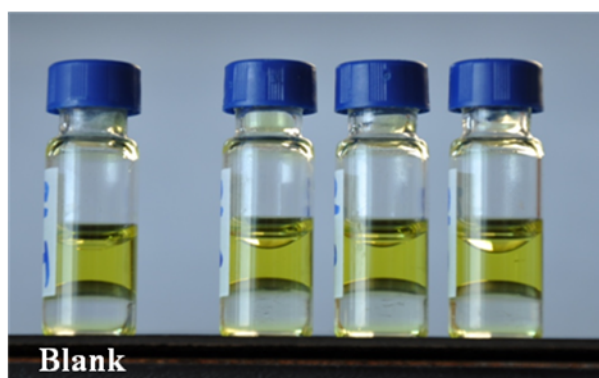
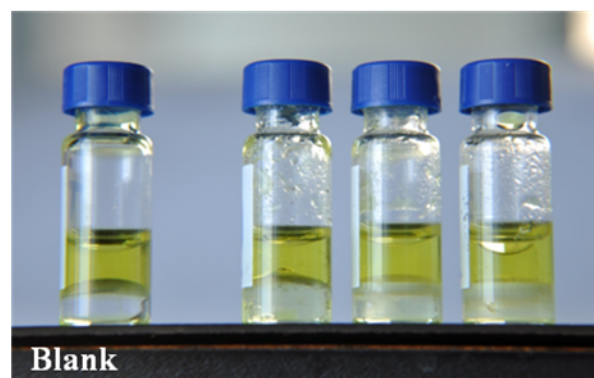
$$[H]_0(\delta_{obs} - \delta_{H,free}) = [HG](\delta_{H,comp} - \delta_{H,free})$$

By combining equation 8 and the solution of the quadratic equation, equation 9 can be written with a single variable, r:

$$[H]_0(\delta_{obs} - \delta_{H,free}) = \frac{(\delta_{H,comp} - \delta_{H,free})}{2} \left(C_t + \frac{1}{K_a} - \sqrt{\left(C_t + \frac{1}{K_a} \right)^2 - 4rC_t^2(1-r)} \right)$$

Non-linear regression was used for equation 9 for protons H₁ and H₂ of chalice 5 in order to estimate the association constant K_a. The association constant for Na⁺ was calculated using this method in order to validate the values obtained from the titration curves, K_a = 1340 M⁻¹.

Figure S3: Metal picrate extraction by 5 : 5 mM solutions of metal picrate in water were extracted by 5 mM solutions of 5 in chloroform.



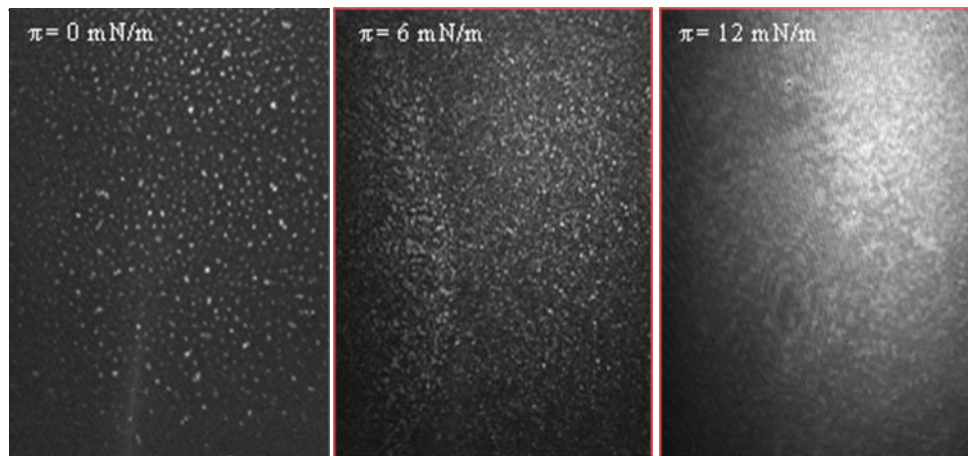
Surface pressure and area measurement: Solutions of **5** were spread on the water surface (area 80 cm²) of a KSV L-B trough (KSV Instruments, Finland) thermostated at room temperature (20±0.5°C). Following by solvent evaporation (10 min), the molecules were symmetrically compressed at a rate of 5 Å²/molecule/min. The surface pressure was measured using a Wilhelmy balance and a platinum plate (precision of 0.1 mN/m). The distilled water was purified with a Milli-Q Gradient System (Millipore, Bedford, MA) to produce deionized water with a resistivity of 18.2 MV cm. For complexation data, solutions of **5** were spread on a 10 mM NaCl or KCl surface. Same procedure as described before was followed.

Langmuir isotherms and BAM imaging: Brewster angle microscopy (BAM) measurements at the A/W interface were carried out with a commercial I-Elli2000 imaging ellipsometer (Nanofilm Technologie GmbH, Göttingen, Germany) equipped with a 50mW Nd:YAG laser ($\lambda = 532$ nm) and a 702 BAM film balance (area of 700 cm²) from Nima Technology Ltd. (Coventry, England). Both the imaging ellipsometer and film balance were mounted on an AVI-150M vibration isolation system (Herzan Industries, Laguna Niguel, CA). Monolayers were symmetrically compressed at 20 cm² min⁻¹. Imaging was performed with a 10x objective (corresponding to lateral resolution of approximately 2 μ m, incident angle of 53.15° (Brewster angle of water) and a laser output of 50%. The polarizer, analyzer and compensator angles were all set to 0°.

Monolayer deposition: Solutions of **5** were spread on the water surface (area 287 cm²) of a KSV 2000 mini-alternate multilayer trough (KSV Instruments Ltd., Helsinki, Finland). The subphase temperature was maintained at 20°C using a Julabo model F12-MC circulation bath. Following solvent evaporation (30 min), the molecules were symmetrically compressed at a rate of 1 Å² molecule⁻¹min⁻¹ to the desired surface pressure. The surface pressure was measured using a platinum Wilhelmy plate sensing device (precision of 0.1 mN/m). High purity water (18.2M Ω cm), obtained by further purification of distilled water with a Milli-Q Gradient system (Millipore, Bedford, MA), was used as the subphase of the Langmuir-Blodgett trough. The deposition method was the Langmuir-Blodgett technique (vertical deposition). The monolayer was compressed to a target surface pressure and held at that pressure for 20 min before vertical deposition onto mica (AFM) or silicon (spectroscopic ellipsometry).

Spectroscopic Ellipsometry data: Film thickness of 12.3 Å was obtained for a monolayer of **5** deposited on silicon substrates at $\pi = 5 \text{ mN}\cdot\text{m}^{-1}$. All measurements were performed in air at an incident angle of 75° and a wavelength range of 370–1000 nm on a multiwavelength ellipsometer equipped with a QTH lamp and rotating compensator (Model M-2000V, J.A. Woollam Co, Inc., Lincoln, NE). Six different macroscopic areas were analyzed on each sample. The average film thickness was calculated from plots of Ψ ($\pm 0.065^\circ$) and Δ ($\pm 1.51^\circ$) versus wavelength and a three-layer Si/SiO_x/cage model using the Levenberg-Marquardt non-linear optimization algorithm of the vendor's WVASE32® software. Selected parameters were optimized to best fit the experimental data and the calculation terminated when the mean standard error (MSE) was between 2.148 and 2.268 (for comparison, a perfect fit is achieved when MSE= 1.0). The thickness (d) of the native oxide (SiO_x) layer on the bare silicon substrate was first independently determined using the complex refractive index values ($\hat{N} = n - ik$) as a function of wavelength found in the materials data files provided by the vendor. The following Si/SiO_x optical parameters were obtained at 532.3 nm: layer 1 (Si): $d = 1 \text{ mm}$, $n = 4.142$, $k = 0.028$ and layer 2 (SiO_x): $d = 1.8 \text{ nm}$, $n = 1.466$, $k = 0$. The Si/SiO_x parameters were held fix in subsequent calculations of the cage monolayer thicknesses. Each cage monolayer (layer 3) was modeled as a Cauchy layer. The Cauchy dispersion equation describes the film refractive index as a function of wavelength (λ) in the transparent spectral range: $n(\lambda/\mu\text{m}) = A + B/\lambda^2 + C/\lambda^4$, where A , B , and C are the Cauchy parameters. The cage monolayer data was fit using values for the Cauchy parameters of $A = 1.44$, $B = 0.0045$, and $C = 0$ and, at 532.3 nm, $n = 1.456$ and $k = 0$.

Figure S4. Brewster Angle Microscopy at 0 mN/m, 6,19 mN/m and 12.12 mN/m (538 μm \times 430 μm images)



AFM imaging: The solid-supported monolayer of **5** was imaged under ambient conditions using a Dimension 5000 scanning probe microscope and Nanoscope V controller (Digital Instruments, Santa Barbara, CA). Height and phase contrast images were simultaneously acquired in intermittent-contact (“tapping”) mode with silicon probes (Nanoworld type Arrow-NCR) of nominal spring constant of 20-100 Nm^{-1} , resonant frequency of 200-300 kHz and tip radius < 10 nm. Imaging was performed at drive amplitudes of ~ 50 – 70 mV and medium oscillation damping ($17 \pm 3\%$, where the percent oscillation damping = (free air amplitude–imaging set point amplitude)/free air amplitude $\times 100$). All images were captured at a scan rate of 1.0 Hz and resolution of 512×512 pixels. For each cage composition and surface pressure, images were obtained from 2 independently prepared samples, and several macroscopically separated areas were imaged on each sample.

CRYSTAL AND MOLECULAR STRUCTURE OF 5
C₄₀H₃₈N₄O₈ COMPOUND

| | |
|-----------------------------------|---|
| Empirical formula | C ₄₀ H ₃₈ N ₄ O ₈ |
| Formula weight | 702.74 |
| Temperature | 150K |
| Wavelength | 1.54178 Å |
| Crystal system | Triclinic |
| Space group | P-1 |
| Unit cell dimensions | a = 8.4941(4) Å α = 103.151(2)° b = 14.0115(6) Å β = 92.589(2)° c = 14.3666(6) Å γ = 99.426(2)° |
| Volume | 1636.46(12) Å ³ |
| Z | 2 |
| Density (calculated) | 1.426 g/cm ³ |
| Absorption coefficient | 0.824 mm ⁻¹ |
| F(000) | 740 |
| Crystal size | 0.12 x 0.03 x 0.02 mm |
| Theta range for data collection | 3.17 to 67.80° |
| Index ranges | -9 ≤ h ≤ 8, -16 ≤ k ≤ 16, -17 ≤ l ≤ 17 |
| Reflections collected | 26274 |
| Independent reflections | 5140 [R _{int} = 0.054] |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.9837 and 0.8532 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 5140 / 0 / 469 |
| Goodness-of-fit on F ² | 1.047 |
| Final R indices [I > 2σ(I)] | R ₁ = 0.0380, wR ₂ = 0.0978 |
| R indices (all data) | R ₁ = 0.0518, wR ₂ = 0.1027 |
| Largest diff. peak and hole | 0.294 and -0.215 e/Å ³ |

Table S1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C40 H38 N4 O8.

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

| | x | y | z | U_{eq} |
|-------|----------|----------|---------|----------|
| O(1) | 5474(2) | 6164(1) | 3516(1) | 54(1) |
| O(2) | 2893(2) | 6499(1) | 2477(1) | 49(1) |
| O(3) | 2862(2) | 6694(1) | 499(1) | 54(1) |
| O(4) | 5709(2) | 6870(1) | -728(1) | 48(1) |
| O(5) | 7863(2) | 6228(1) | 205(1) | 49(1) |
| O(6) | 7297(2) | 5327(1) | 1934(1) | 67(1) |
| O(7) | 9884(2) | 8961(1) | 3427(1) | 48(1) |
| O(8) | 5005(2) | 10025(1) | 1380(1) | 46(1) |
| N(1) | 8459(2) | 10243(1) | 3878(1) | 35(1) |
| N(2) | 6219(2) | 10508(1) | 2917(1) | 34(1) |
| N(3) | 7678(2) | 10752(1) | 1716(1) | 34(1) |
| N(4) | 9720(2) | 10127(1) | 2546(1) | 35(1) |
| C(1) | 5329(3) | 7129(1) | 3536(1) | 42(1) |
| C(2) | 4047(3) | 7281(1) | 2988(1) | 42(1) |
| C(3) | 3424(3) | 5828(1) | 1685(1) | 49(1) |
| C(4) | 4101(3) | 6326(1) | 937(1) | 48(1) |
| C(5) | 3405(3) | 7482(1) | 60(1) | 50(1) |
| C(6) | 4186(3) | 7162(2) | -857(1) | 52(1) |
| C(7) | 6915(2) | 7585(1) | -191(1) | 41(1) |
| C(8) | 8084(2) | 7238(1) | 305(1) | 40(1) |
| C(9) | 8923(3) | 5842(1) | 771(1) | 49(1) |
| C(10) | 8647(3) | 5997(2) | 1815(1) | 59(1) |
| C(11) | 7287(3) | 5105(2) | 2857(1) | 61(1) |
| C(12) | 7058(3) | 5954(2) | 3652(2) | 56(1) |
| C(13) | 7542(2) | 9775(1) | 4552(1) | 39(1) |
| C(14) | 6248(2) | 8912(1) | 4045(1) | 38(1) |
| C(15) | 6404(3) | 7952(1) | 4073(1) | 41(1) |
| C(16) | 3866(2) | 8244(1) | 2980(1) | 42(1) |
| C(17) | 4965(2) | 9066(1) | 3488(1) | 38(1) |
| C(18) | 4860(2) | 10107(1) | 3386(1) | 38(1) |
| C(19) | 10544(2) | 9673(1) | 1734(1) | 38(1) |
| C(20) | 9361(2) | 8944(1) | 991(1) | 37(1) |
| C(21) | 9309(2) | 7922(1) | 871(1) | 38(1) |
| C(22) | 6999(2) | 8596(1) | -97(1) | 40(1) |
| C(23) | 8193(2) | 9284(1) | 511(1) | 37(1) |
| C(24) | 8088(2) | 10383(1) | 726(1) | 39(1) |
| C(25) | 7794(2) | 10923(1) | 3410(1) | 33(1) |
| C(26) | 6173(2) | 10375(1) | 1945(1) | 35(1) |
| C(27) | 8872(2) | 10949(1) | 2528(1) | 33(1) |
| C(28) | 9390(2) | 9687(1) | 3287(1) | 36(1) |
| C(29) | 7847(3) | 13796(2) | 5286(2) | 62(1) |
| C(30) | 8751(3) | 13155(2) | 5548(1) | 58(1) |
| C(31) | 8737(3) | 12226(1) | 4957(1) | 47(1) |
| C(32) | 7800(2) | 11930(1) | 4094(1) | 36(1) |
| C(33) | 6872(3) | 12571(1) | 3846(1) | 48(1) |
| C(34) | 6903(3) | 13502(1) | 4443(2) | 57(1) |
| C(35) | 12243(3) | 13674(2) | 2817(2) | 72(1) |
| C(36) | 12559(3) | 12970(2) | 3293(2) | 73(1) |
| C(37) | 11484(3) | 12083(2) | 3182(2) | 57(1) |

| | | | | |
|--------|-----------|-----------|----------|--------|
| C (38) | 10066 (2) | 11909 (1) | 2611 (1) | 38 (1) |
| C (39) | 9752 (3) | 12635 (1) | 2157 (1) | 53 (1) |
| C (40) | 10839 (4) | 13504 (2) | 2250 (2) | 70 (1) |

Table S2. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C40 H38 N4 O8.

| | x | y | z | U_{eq} |
|--------|-------|-------|-------|----------|
| H(39A) | 2507 | 5298 | 1382 | 59 |
| H(39B) | 4250 | 5506 | 1935 | 59 |
| H(38A) | 4982 | 6882 | 1235 | 58 |
| H(38B) | 4538 | 5843 | 446 | 58 |
| H(40A) | 4183 | 7995 | 520 | 60 |
| H(40B) | 2483 | 7791 | -82 | 60 |
| H(35A) | 3451 | 6596 | -1288 | 62 |
| H(35B) | 4331 | 7719 | -1181 | 62 |
| H(36A) | 10037 | 6157 | 719 | 59 |
| H(36B) | 8825 | 5118 | 493 | 59 |
| H(48A) | 9599 | 5894 | 2177 | 71 |
| H(48B) | 8483 | 6689 | 2070 | 71 |
| H(51A) | 8314 | 4900 | 3004 | 73 |
| H(51B) | 6418 | 4533 | 2833 | 73 |
| H(42A) | 7199 | 5780 | 4277 | 68 |
| H(42B) | 7866 | 6550 | 3653 | 68 |
| H(12A) | 7043 | 10281 | 4977 | 47 |
| H(12B) | 8284 | 9534 | 4959 | 47 |
| H(20) | 7261 | 7850 | 4466 | 49 |
| H(22) | 2970 | 8345 | 2619 | 50 |
| H(21A) | 3852 | 10089 | 3004 | 46 |
| H(21B) | 4841 | 10548 | 4029 | 46 |
| H(31A) | 11355 | 9324 | 1960 | 46 |
| H(31B) | 11105 | 10197 | 1446 | 46 |
| H(33) | 10130 | 7693 | 1184 | 46 |
| H(15) | 6231 | 8825 | -452 | 48 |
| H(19A) | 9128 | 10770 | 633 | 46 |
| H(19B) | 7264 | 10490 | 269 | 46 |
| H(50) | 7880 | 14440 | 5691 | 74 |
| H(41) | 9393 | 13354 | 6142 | 69 |
| H(45) | 9371 | 11791 | 5143 | 56 |
| H(28) | 6207 | 12370 | 3261 | 58 |
| H(43) | 6263 | 13938 | 4263 | 68 |
| H(47) | 12991 | 14273 | 2882 | 86 |
| H(52) | 13520 | 13091 | 3699 | 87 |
| H(44) | 11725 | 11594 | 3501 | 68 |
| H(46) | 8771 | 12534 | 1774 | 64 |
| H(49) | 10613 | 13988 | 1919 | 84 |

Table S3. Anisotropic parameters ($\text{\AA}^2 \times 10^3$) for C40 H38 N4 O8.

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

| | U11 | U22 | U33 | U23 | U13 | U12 |
|-------|-------|-------|-------|-------|-------|--------|
| O(1) | 57(1) | 37(1) | 66(1) | 13(1) | 4(1) | 2(1) |
| O(2) | 44(1) | 45(1) | 49(1) | 2(1) | 8(1) | -6(1) |
| O(3) | 44(1) | 58(1) | 62(1) | 23(1) | 2(1) | 0(1) |
| O(4) | 46(1) | 46(1) | 42(1) | -1(1) | 0(1) | 2(1) |
| O(5) | 54(1) | 36(1) | 51(1) | 5(1) | 0(1) | 5(1) |
| O(6) | 75(1) | 60(1) | 58(1) | 11(1) | 12(1) | -6(1) |
| O(7) | 57(1) | 45(1) | 48(1) | 18(1) | 3(1) | 18(1) |
| O(8) | 40(1) | 59(1) | 37(1) | 9(1) | -4(1) | 3(1) |
| N(1) | 40(1) | 35(1) | 29(1) | 10(1) | 3(1) | 5(1) |
| N(2) | 32(1) | 40(1) | 30(1) | 9(1) | 3(1) | 1(1) |
| N(3) | 36(1) | 38(1) | 27(1) | 9(1) | 3(1) | 4(1) |
| N(4) | 37(1) | 36(1) | 32(1) | 9(1) | 5(1) | 7(1) |
| C(1) | 49(2) | 35(1) | 41(1) | 12(1) | 11(1) | 2(1) |
| C(2) | 42(1) | 40(1) | 40(1) | 7(1) | 9(1) | -4(1) |
| C(3) | 55(2) | 41(1) | 46(1) | 7(1) | 2(1) | -1(1) |
| C(4) | 47(2) | 51(1) | 46(1) | 11(1) | 4(1) | 8(1) |
| C(5) | 48(2) | 46(1) | 55(1) | 15(1) | 3(1) | 6(1) |
| C(6) | 53(2) | 52(1) | 45(1) | 9(1) | -8(1) | -1(1) |
| C(7) | 45(1) | 42(1) | 30(1) | 3(1) | 6(1) | 1(1) |
| C(8) | 46(1) | 37(1) | 36(1) | 5(1) | 10(1) | 5(1) |
| C(9) | 49(2) | 39(1) | 58(1) | 7(1) | 9(1) | 9(1) |
| C(10) | 67(2) | 48(1) | 58(1) | 12(1) | 5(1) | 1(1) |
| C(11) | 77(2) | 49(1) | 60(1) | 16(1) | 15(1) | 11(1) |
| C(12) | 67(2) | 48(1) | 56(1) | 13(1) | 0(1) | 13(1) |
| C(13) | 49(1) | 40(1) | 28(1) | 11(1) | 5(1) | 2(1) |
| C(14) | 44(1) | 38(1) | 29(1) | 10(1) | 8(1) | 2(1) |
| C(15) | 47(1) | 42(1) | 34(1) | 12(1) | 4(1) | 3(1) |
| C(16) | 36(1) | 46(1) | 42(1) | 12(1) | 6(1) | 3(1) |
| C(17) | 39(1) | 40(1) | 34(1) | 10(1) | 11(1) | 3(1) |
| C(18) | 33(1) | 44(1) | 38(1) | 11(1) | 9(1) | 5(1) |
| C(19) | 34(1) | 41(1) | 40(1) | 8(1) | 8(1) | 7(1) |
| C(20) | 38(1) | 41(1) | 30(1) | 8(1) | 12(1) | 4(1) |
| C(21) | 35(1) | 43(1) | 38(1) | 8(1) | 9(1) | 11(1) |
| C(22) | 45(1) | 47(1) | 29(1) | 9(1) | 6(1) | 7(1) |
| C(23) | 42(1) | 40(1) | 27(1) | 9(1) | 9(1) | 4(1) |
| C(24) | 48(1) | 41(1) | 27(1) | 11(1) | 7(1) | 5(1) |
| C(25) | 35(1) | 34(1) | 29(1) | 7(1) | 3(1) | 2(1) |
| C(26) | 39(1) | 34(1) | 30(1) | 8(1) | 2(1) | 4(1) |
| C(27) | 32(1) | 35(1) | 30(1) | 9(1) | 3(1) | 4(1) |
| C(28) | 38(1) | 36(1) | 32(1) | 6(1) | -3(1) | 2(1) |
| C(29) | 74(2) | 42(1) | 59(1) | -7(1) | 16(1) | 3(1) |
| C(30) | 54(2) | 62(1) | 44(1) | -8(1) | 0(1) | 2(1) |
| C(31) | 49(2) | 49(1) | 38(1) | 3(1) | 1(1) | 6(1) |
| C(32) | 41(1) | 35(1) | 31(1) | 7(1) | 8(1) | 2(1) |
| C(33) | 60(2) | 42(1) | 42(1) | 10(1) | 4(1) | 8(1) |
| C(34) | 70(2) | 41(1) | 61(1) | 10(1) | 11(1) | 13(1) |
| C(35) | 65(2) | 46(1) | 94(2) | 8(1) | 26(2) | -13(1) |
| C(36) | 44(2) | 65(2) | 95(2) | 3(1) | -1(1) | -10(1) |
| C(37) | 48(2) | 50(1) | 67(1) | 12(1) | -2(1) | -1(1) |

| | | | | | | |
|-------|-------|-------|-------|-------|-------|-------|
| C(38) | 37(1) | 37(1) | 37(1) | 7(1) | 8(1) | 2(1) |
| C(39) | 58(2) | 46(1) | 57(1) | 20(1) | 6(1) | 1(1) |
| C(40) | 84(2) | 45(1) | 80(2) | 25(1) | 18(2) | -6(1) |

Table S4. Bond lengths [Å] and angles [°] for C40 H38 N4 O8

| | |
|-------------|----------|
| O(1)-C(1) | 1.372(2) |
| O(1)-C(12) | 1.437(3) |
| O(2)-C(2) | 1.385(2) |
| O(2)-C(3) | 1.442(2) |
| O(3)-C(5) | 1.415(2) |
| O(3)-C(4) | 1.427(3) |
| O(4)-C(7) | 1.375(2) |
| O(4)-C(6) | 1.436(3) |
| O(5)-C(8) | 1.370(2) |
| O(5)-C(9) | 1.435(3) |
| O(6)-C(10) | 1.402(3) |
| O(6)-C(11) | 1.430(2) |
| O(7)-C(28) | 1.214(2) |
| O(8)-C(26) | 1.212(2) |
| N(1)-C(28) | 1.383(2) |
| N(1)-C(25) | 1.454(2) |
| N(1)-C(13) | 1.471(2) |
| N(2)-C(26) | 1.364(2) |
| N(2)-C(25) | 1.450(2) |
| N(2)-C(18) | 1.461(2) |
| N(3)-C(26) | 1.385(2) |
| N(3)-C(27) | 1.456(2) |
| N(3)-C(24) | 1.476(2) |
| N(4)-C(28) | 1.366(2) |
| N(4)-C(27) | 1.459(2) |
| N(4)-C(19) | 1.462(2) |
| C(1)-C(2) | 1.387(3) |
| C(1)-C(15) | 1.395(2) |
| C(2)-C(16) | 1.385(3) |
| C(3)-C(4) | 1.495(2) |
| C(5)-C(6) | 1.512(3) |
| C(7)-C(22) | 1.381(2) |
| C(7)-C(8) | 1.407(3) |
| C(8)-C(21) | 1.380(3) |
| C(9)-C(10) | 1.500(3) |
| C(11)-C(12) | 1.495(3) |
| C(13)-C(14) | 1.514(2) |
| C(14)-C(15) | 1.383(2) |
| C(14)-C(17) | 1.397(3) |
| C(16)-C(17) | 1.391(2) |
| C(17)-C(18) | 1.515(2) |
| C(19)-C(20) | 1.504(2) |
| C(20)-C(23) | 1.389(3) |
| C(20)-C(21) | 1.396(2) |
| C(22)-C(23) | 1.392(2) |
| C(23)-C(24) | 1.518(2) |
| C(25)-C(32) | 1.524(2) |
| C(25)-C(27) | 1.600(2) |
| C(27)-C(38) | 1.523(2) |
| C(29)-C(34) | 1.363(3) |
| C(29)-C(30) | 1.375(3) |
| C(30)-C(31) | 1.381(3) |
| C(31)-C(32) | 1.382(3) |
| C(32)-C(33) | 1.381(3) |
| C(33)-C(34) | 1.385(3) |
| C(35)-C(40) | 1.368(4) |
| C(35)-C(36) | 1.374(4) |
| C(36)-C(37) | 1.389(3) |

| | |
|-----------------------|-------------|
| C(37) - C(38) | 1.381 (3) |
| C(38) - C(39) | 1.381 (3) |
| C(39) - C(40) | 1.378 (3) |
| | |
| C(1) - O(1) - C(12) | 117.86 (15) |
| C(2) - O(2) - C(3) | 116.09 (15) |
| C(5) - O(3) - C(4) | 114.84 (16) |
| C(7) - O(4) - C(6) | 116.81 (15) |
| C(8) - O(5) - C(9) | 118.19 (14) |
| C(10) - O(6) - C(11) | 114.57 (17) |
| C(28) - N(1) - C(25) | 111.79 (12) |
| C(28) - N(1) - C(13) | 118.20 (14) |
| C(25) - N(1) - C(13) | 120.48 (15) |
| C(26) - N(2) - C(25) | 114.05 (14) |
| C(26) - N(2) - C(18) | 121.85 (14) |
| C(25) - N(2) - C(18) | 123.30 (13) |
| C(26) - N(3) - C(27) | 111.57 (12) |
| C(26) - N(3) - C(24) | 117.28 (13) |
| C(27) - N(3) - C(24) | 120.94 (15) |
| C(28) - N(4) - C(27) | 113.80 (14) |
| C(28) - N(4) - C(19) | 122.12 (15) |
| C(27) - N(4) - C(19) | 122.66 (13) |
| O(1) - C(1) - C(2) | 117.65 (16) |
| O(1) - C(1) - C(15) | 123.19 (19) |
| C(2) - C(1) - C(15) | 119.16 (17) |
| C(16) - C(2) - O(2) | 118.43 (19) |
| C(16) - C(2) - C(1) | 119.49 (16) |
| O(2) - C(2) - C(1) | 122.02 (17) |
| O(2) - C(3) - C(4) | 113.64 (15) |
| O(3) - C(4) - C(3) | 109.01 (17) |
| O(3) - C(5) - C(6) | 113.95 (16) |
| O(4) - C(6) - C(5) | 114.64 (16) |
| O(4) - C(7) - C(22) | 123.92 (19) |
| O(4) - C(7) - C(8) | 116.16 (16) |
| C(22) - C(7) - C(8) | 119.89 (16) |
| O(5) - C(8) - C(21) | 125.23 (18) |
| O(5) - C(8) - C(7) | 115.86 (16) |
| C(21) - C(8) - C(7) | 118.86 (16) |
| O(5) - C(9) - C(10) | 115.61 (18) |
| O(6) - C(10) - C(9) | 109.96 (16) |
| O(6) - C(11) - C(12) | 114.04 (17) |
| O(1) - C(12) - C(11) | 109.15 (18) |
| N(1) - C(13) - C(14) | 112.44 (12) |
| C(15) - C(14) - C(17) | 119.49 (16) |
| C(15) - C(14) - C(13) | 119.51 (18) |
| C(17) - C(14) - C(13) | 120.81 (16) |
| C(14) - C(15) - C(1) | 121.38 (19) |
| C(2) - C(16) - C(17) | 121.6 (2) |
| C(16) - C(17) - C(14) | 118.82 (17) |
| C(16) - C(17) - C(18) | 121.14 (18) |
| C(14) - C(17) - C(18) | 119.88 (15) |
| N(2) - C(18) - C(17) | 111.10 (15) |
| N(4) - C(19) - C(20) | 110.17 (15) |
| C(23) - C(20) - C(21) | 119.77 (16) |
| C(23) - C(20) - C(19) | 119.72 (15) |
| C(21) - C(20) - C(19) | 120.03 (17) |
| C(8) - C(21) - C(20) | 121.14 (19) |
| C(7) - C(22) - C(23) | 120.99 (19) |
| C(20) - C(23) - C(22) | 119.23 (16) |
| C(20) - C(23) - C(24) | 120.96 (15) |
| C(22) - C(23) - C(24) | 119.43 (18) |

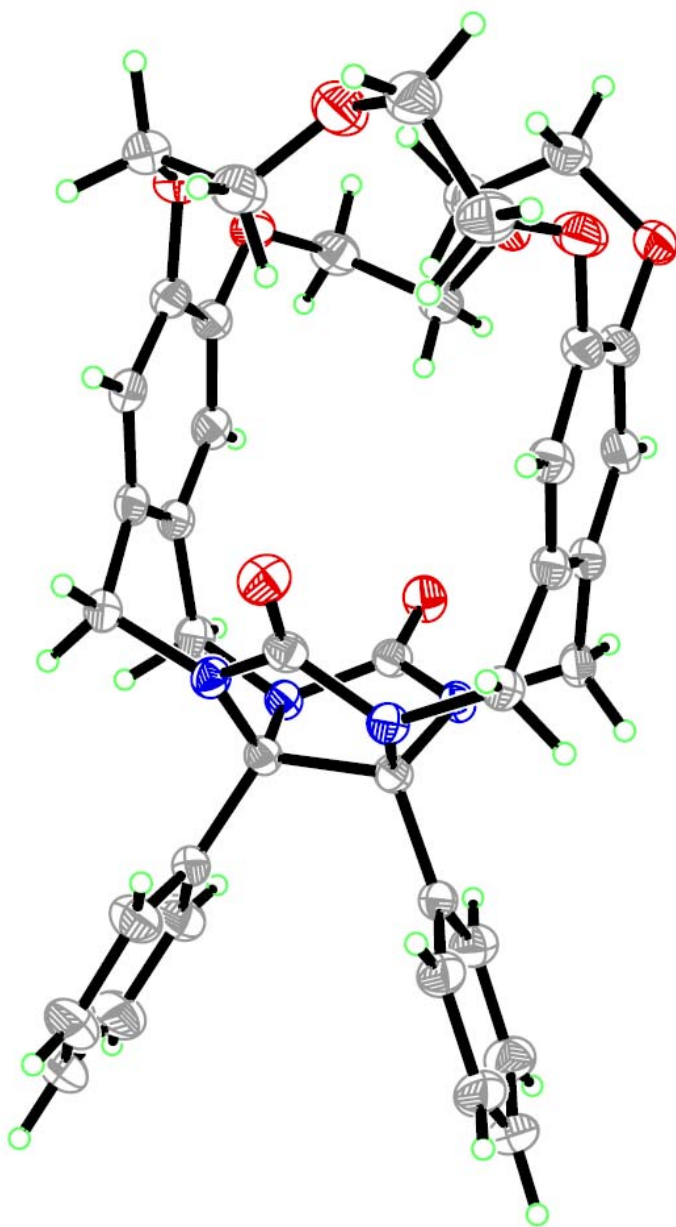
| | |
|-------------------|------------|
| N(3)-C(24)-C(23) | 111.98(12) |
| N(2)-C(25)-N(1) | 113.45(12) |
| N(2)-C(25)-C(32) | 111.13(15) |
| N(1)-C(25)-C(32) | 112.22(13) |
| N(2)-C(25)-C(27) | 101.49(12) |
| N(1)-C(25)-C(27) | 102.58(14) |
| C(32)-C(25)-C(27) | 115.34(12) |
| O(8)-C(26)-N(2) | 126.18(16) |
| O(8)-C(26)-N(3) | 125.68(14) |
| N(2)-C(26)-N(3) | 108.08(15) |
| N(3)-C(27)-N(4) | 114.04(13) |
| N(3)-C(27)-C(38) | 112.43(13) |
| N(4)-C(27)-C(38) | 110.08(15) |
| N(3)-C(27)-C(25) | 102.59(14) |
| N(4)-C(27)-C(25) | 101.40(12) |
| C(38)-C(27)-C(25) | 115.76(13) |
| O(7)-C(28)-N(4) | 126.06(17) |
| O(7)-C(28)-N(1) | 125.99(15) |
| N(4)-C(28)-N(1) | 107.88(15) |
| C(34)-C(29)-C(30) | 119.54(19) |
| C(29)-C(30)-C(31) | 120.7(2) |
| C(30)-C(31)-C(32) | 120.0(2) |
| C(33)-C(32)-C(31) | 118.88(16) |
| C(33)-C(32)-C(25) | 119.67(15) |
| C(31)-C(32)-C(25) | 121.45(18) |
| C(32)-C(33)-C(34) | 120.56(19) |
| C(29)-C(34)-C(33) | 120.3(2) |
| C(40)-C(35)-C(36) | 119.4(2) |
| C(35)-C(36)-C(37) | 120.4(2) |
| C(38)-C(37)-C(36) | 120.4(2) |
| C(39)-C(38)-C(37) | 118.34(17) |
| C(39)-C(38)-C(27) | 121.96(18) |
| C(37)-C(38)-C(27) | 119.66(16) |
| C(40)-C(39)-C(38) | 121.1(2) |
| C(35)-C(40)-C(39) | 120.3(2) |

Table S5. Torsion angles [°] for C40 H38 N4 O8.

| | |
|-------------------------|--------------|
| C(12)-O(1)-C(1)-C(2) | 149.98 (17) |
| C(12)-O(1)-C(1)-C(15) | -29.7 (2) |
| C(3)-O(2)-C(2)-C(16) | 115.92 (19) |
| C(3)-O(2)-C(2)-C(1) | -66.9 (2) |
| O(1)-C(1)-C(2)-C(16) | -179.27 (15) |
| C(15)-C(1)-C(2)-C(16) | 0.4 (3) |
| O(1)-C(1)-C(2)-O(2) | 3.6 (2) |
| C(15)-C(1)-C(2)-O(2) | -176.75 (15) |
| C(2)-O(2)-C(3)-C(4) | -59.4 (2) |
| C(5)-O(3)-C(4)-C(3) | 157.20 (15) |
| O(2)-C(3)-C(4)-O(3) | -64.9 (2) |
| C(4)-O(3)-C(5)-C(6) | 71.9 (2) |
| C(7)-O(4)-C(6)-C(5) | -61.9 (2) |
| O(3)-C(5)-C(6)-O(4) | -69.5 (2) |
| C(6)-O(4)-C(7)-C(22) | -26.7 (2) |
| C(6)-O(4)-C(7)-C(8) | 151.28 (15) |
| C(9)-O(5)-C(8)-C(21) | 3.9 (2) |
| C(9)-O(5)-C(8)-C(7) | -173.68 (15) |
| O(4)-C(7)-C(8)-O(5) | -1.0 (2) |
| C(22)-C(7)-C(8)-O(5) | 177.03 (15) |
| O(4)-C(7)-C(8)-C(21) | -178.77 (15) |
| C(22)-C(7)-C(8)-C(21) | -0.7 (2) |
| C(8)-O(5)-C(9)-C(10) | 74.2 (2) |
| C(11)-O(6)-C(10)-C(9) | 155.03 (19) |
| O(5)-C(9)-C(10)-O(6) | 75.9 (2) |
| C(10)-O(6)-C(11)-C(12) | 69.7 (3) |
| C(1)-O(1)-C(12)-C(11) | -128.53 (16) |
| O(6)-C(11)-C(12)-O(1) | 66.6 (2) |
| C(28)-N(1)-C(13)-C(14) | -65.5 (2) |
| C(25)-N(1)-C(13)-C(14) | 78.00 (19) |
| N(1)-C(13)-C(14)-C(15) | 110.59 (18) |
| N(1)-C(13)-C(14)-C(17) | -64.3 (2) |
| C(17)-C(14)-C(15)-C(1) | 1.7 (3) |
| C(13)-C(14)-C(15)-C(1) | -173.30 (15) |
| O(1)-C(1)-C(15)-C(14) | 177.50 (16) |
| C(2)-C(1)-C(15)-C(14) | -2.2 (3) |
| O(2)-C(2)-C(16)-C(17) | 179.09 (15) |
| C(1)-C(2)-C(16)-C(17) | 1.8 (3) |
| C(2)-C(16)-C(17)-C(14) | -2.3 (3) |
| C(2)-C(16)-C(17)-C(18) | 173.06 (15) |
| C(15)-C(14)-C(17)-C(16) | 0.5 (2) |
| C(13)-C(14)-C(17)-C(16) | 175.45 (15) |
| C(15)-C(14)-C(17)-C(18) | -174.88 (15) |
| C(13)-C(14)-C(17)-C(18) | 0.0 (2) |
| C(26)-N(2)-C(18)-C(17) | 90.0 (2) |
| C(25)-N(2)-C(18)-C(17) | -79.08 (19) |
| C(16)-C(17)-C(18)-N(2) | -112.68 (18) |
| C(14)-C(17)-C(18)-N(2) | 62.64 (19) |
| C(28)-N(4)-C(19)-C(20) | 85.2 (2) |
| C(27)-N(4)-C(19)-C(20) | -80.34 (18) |
| N(4)-C(19)-C(20)-C(23) | 64.88 (19) |
| N(4)-C(19)-C(20)-C(21) | -107.16 (18) |
| O(5)-C(8)-C(21)-C(20) | -174.54 (15) |
| C(7)-C(8)-C(21)-C(20) | 3.0 (2) |
| C(23)-C(20)-C(21)-C(8) | -2.1 (2) |
| C(19)-C(20)-C(21)-C(8) | 169.90 (16) |

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|-------------------------------|--------------|
| O(4) - C(7) - C(22) - C(23) | 175.50 (16) |
| C(8) - C(7) - C(22) - C(23) | -2.4 (2) |
| C(21) - C(20) - C(23) - C(22) | -1.0 (2) |
| C(19) - C(20) - C(23) - C(22) | -173.04 (15) |
| C(21) - C(20) - C(23) - C(24) | 171.87 (14) |
| C(19) - C(20) - C(23) - C(24) | -0.2 (2) |
| C(7) - C(22) - C(23) - C(20) | 3.2 (2) |
| C(7) - C(22) - C(23) - C(24) | -169.71 (15) |
| C(26) - N(3) - C(24) - C(23) | -66.1 (2) |
| C(27) - N(3) - C(24) - C(23) | 76.21 (19) |
| C(20) - C(23) - C(24) - N(3) | -64.8 (2) |
| C(22) - C(23) - C(24) - N(3) | 108.00 (18) |
| C(26) - N(2) - C(25) - N(1) | -114.47 (15) |
| C(18) - N(2) - C(25) - N(1) | 55.4 (2) |
| C(26) - N(2) - C(25) - C(32) | 117.98 (15) |
| C(18) - N(2) - C(25) - C(32) | -72.15 (18) |
| C(26) - N(2) - C(25) - C(27) | -5.17 (17) |
| C(18) - N(2) - C(25) - C(27) | 164.71 (15) |
| C(28) - N(1) - C(25) - N(2) | 92.25 (17) |
| C(13) - N(1) - C(25) - N(2) | -53.42 (18) |
| C(28) - N(1) - C(25) - C(32) | -140.76 (15) |
| C(13) - N(1) - C(25) - C(32) | 73.57 (18) |
| C(28) - N(1) - C(25) - C(27) | -16.36 (17) |
| C(13) - N(1) - C(25) - C(27) | -162.04 (12) |
| C(25) - N(2) - C(26) - O(8) | 178.60 (17) |
| C(18) - N(2) - C(26) - O(8) | 8.6 (3) |
| C(25) - N(2) - C(26) - N(3) | -4.12 (19) |
| C(18) - N(2) - C(26) - N(3) | -174.16 (15) |
| C(27) - N(3) - C(26) - O(8) | -169.78 (17) |
| C(24) - N(3) - C(26) - O(8) | -24.1 (3) |
| C(27) - N(3) - C(26) - N(2) | 12.92 (19) |
| C(24) - N(3) - C(26) - N(2) | 158.59 (14) |
| C(26) - N(3) - C(27) - N(4) | 93.37 (17) |
| C(24) - N(3) - C(27) - N(4) | -50.88 (18) |
| C(26) - N(3) - C(27) - C(38) | -140.42 (15) |
| C(24) - N(3) - C(27) - C(38) | 75.34 (18) |
| C(26) - N(3) - C(27) - C(25) | -15.39 (17) |
| C(24) - N(3) - C(27) - C(25) | -159.63 (13) |
| C(28) - N(4) - C(27) - N(3) | -112.64 (15) |
| C(19) - N(4) - C(27) - N(3) | 54.0 (2) |
| C(28) - N(4) - C(27) - C(38) | 119.92 (15) |
| C(19) - N(4) - C(27) - C(38) | -73.41 (18) |
| C(28) - N(4) - C(27) - C(25) | -3.15 (17) |
| C(19) - N(4) - C(27) - C(25) | 163.51 (14) |
| N(2) - C(25) - C(27) - N(3) | 11.78 (15) |
| N(1) - C(25) - C(27) - N(3) | 129.26 (13) |
| C(32) - C(25) - C(27) - N(3) | -108.44 (16) |
| N(2) - C(25) - C(27) - N(4) | -106.31 (14) |
| N(1) - C(25) - C(27) - N(4) | 11.16 (15) |
| C(32) - C(25) - C(27) - N(4) | 133.47 (15) |
| N(2) - C(25) - C(27) - C(38) | 134.59 (15) |
| N(1) - C(25) - C(27) - C(38) | -107.93 (16) |
| C(32) - C(25) - C(27) - C(38) | 14.4 (2) |
| C(27) - N(4) - C(28) - O(7) | 176.13 (17) |
| C(19) - N(4) - C(28) - O(7) | 9.4 (3) |
| C(27) - N(4) - C(28) - N(1) | -6.80 (19) |
| C(19) - N(4) - C(28) - N(1) | -173.55 (15) |
| C(25) - N(1) - C(28) - O(7) | -167.78 (17) |
| C(13) - N(1) - C(28) - O(7) | -21.2 (3) |
| C(25) - N(1) - C(28) - N(4) | 15.14 (19) |
| C(13) - N(1) - C(28) - N(4) | 161.68 (14) |

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|-------------------------|-------------|
| C(34)-C(29)-C(30)-C(31) | 1.3(3) |
| C(29)-C(30)-C(31)-C(32) | -0.4(3) |
| C(30)-C(31)-C(32)-C(33) | -0.9(3) |
| C(30)-C(31)-C(32)-C(25) | 178.06(17) |
| N(2)-C(25)-C(32)-C(33) | -35.8(2) |
| N(1)-C(25)-C(32)-C(33) | -164.07(16) |
| C(27)-C(25)-C(32)-C(33) | 78.9(2) |
| N(2)-C(25)-C(32)-C(31) | 145.19(16) |
| N(1)-C(25)-C(32)-C(31) | 17.0(2) |
| C(27)-C(25)-C(32)-C(31) | -100.02(19) |
| C(31)-C(32)-C(33)-C(34) | 1.2(3) |
| C(25)-C(32)-C(33)-C(34) | -177.76(17) |
| C(30)-C(29)-C(34)-C(33) | -1.0(3) |
| C(32)-C(33)-C(34)-C(29) | -0.3(3) |
| C(40)-C(35)-C(36)-C(37) | 1.5(4) |
| C(35)-C(36)-C(37)-C(38) | -1.5(4) |
| C(36)-C(37)-C(38)-C(39) | 0.0(3) |
| C(36)-C(37)-C(38)-C(27) | -177.67(19) |
| N(3)-C(27)-C(38)-C(39) | 18.2(2) |
| N(4)-C(27)-C(38)-C(39) | 146.57(17) |
| C(25)-C(27)-C(38)-C(39) | -99.2(2) |
| N(3)-C(27)-C(38)-C(37) | -164.17(17) |
| N(4)-C(27)-C(38)-C(37) | -35.8(2) |
| C(25)-C(27)-C(38)-C(37) | 78.4(2) |
| C(37)-C(38)-C(39)-C(40) | 1.6(3) |
| C(27)-C(38)-C(39)-C(40) | 179.18(19) |
| C(36)-C(35)-C(40)-C(39) | 0.0(4) |
| C(38)-C(39)-C(40)-C(35) | -1.6(4) |



ORTEP view of the C₄₀ H₃₈ N₄ O₈ compound with the numbering scheme adopted. Ellipsoids drawn at 30% probability level. Hydrogen atoms are represented by sphere of arbitrary size.

REFERENCES

SAINT (2006) Release 7.34A; Integration Software for Single Crystal Data.
Bruker AXS Inc., Madison, WI 53719-1173.

Sheldrick, G.M. (1996). SADABS, Bruker Area Detector Absorption
Corrections.
Bruker AXS Inc., Madison, WI 53719-1173.

Sheldrick, G.M. (2008). *Acta Cryst.* A64, 112-122.

SHELXTL (2001) version 6.12; Bruker Analytical X-ray Systems Inc.,
Madison, WI 53719-1173.

APEX2 (2006) version 2.1-0; Bruker Molecular Analysis Research Tool.
Bruker AXS Inc., Madison, WI 53719-1173.

Spek, A.L. (2008). PLATON, A Multipurpose Crystallographic Tool,
Utrecht University, Utrecht, The Netherlands.

Maris, T. (2004). UDMX, University of Montréal, Montréal, QC, Canada.

XPREP (2005) Version 2005/2; X-ray data Preparation and Reciprocal space
Exploration Program. Bruker AXS Inc., Madison, WI 53719-1173.