

**Supporting Information of**

**Changing Volume of a Giant Macrocycle:  
The Swelling of the Macrocycle with Organic Solvents**

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1. Experimental procedures.

**General experiments.**

All melting points were measured on a Yanaco MP-S3 micro melting point apparatus.  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR spectra were determined with a JEOL JNM-AL 300 (300 MHz), JEOL JNM-EX400 (400 MHz), JEOL JNM-ECA500 (500 MHz), and JEOL JNM-ECA600 (600 MHz) spectrometers. In measuring  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra, residual solvent peaks were used as internal standards and chemical shifts ( $\delta$ ) are given relative to tetramethylsilane (TMS).  $\text{H}_3\text{PO}_4$  and  $\text{PPh}_3$  were used for the external standards for usual  $^{31}\text{P}$  NMR at room temperature and VT  $^{31}\text{P}$  NMR, each other. The  $^{31}\text{P}$  NMR spectrum in a non-deuterated solvent was performed with the support of the deuterated solvent enclosed in the capillary. Elemental analysis was performed at the Elemental Analytical Centre affiliated to Faculty of Science, Kyushu University. FAB mass spectra were recorded with a JEOL JMS-70 mass spectrometer with *m*-nitrobenzyl alcohol as a matrix. Analytical TLC was carried out on silica gel Merck 60 F<sub>254</sub>, aluminium oxide Merck 60 F<sub>254</sub>. Column chromatography was carried out on silica gel (KANTO 60N) and activated aluminum oxide (nacalai tesque 200mesh).

**Synthesis of the dialdehyde 3a.**

To a stirred mixture of 4-ethynylbenzaldehyde (0.13 g, 1.0 mmol) and *trans*-Pt(PEt<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (0.25 g, 0.50 mmol) in 100 mL of dry CH<sub>2</sub>Cl<sub>2</sub>, NEt<sub>3</sub> (0.13 g, 1.3 mmol) and CuI (9.5 mg, 0.050 mmol) were added, and the mixture was stirred for 14.5 h under an argon atmosphere. The solvent was removed under reduced pressure, and chromatography on alumina with CH<sub>2</sub>Cl<sub>2</sub> as an eluent gave the purified product as a yellow powder. Yield: 0.29 g

(83%); mp 164–166 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  (H) 1.14– 1.32 (18H, m,  $\text{CH}_3$ ), 2.07– 2.27 (12H, m,  $\text{CH}_2$ ), 7.37 (4H, d,  $J$ = 8.3 Hz, Ar– H), 7.73 (4H, d,  $J$ = 8.3 Hz, Ar– H), 9.93 (2H, s, CHO);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  (C) 8.31 ( $\text{CH}_3$ ), 16.4 (t,  $\text{PCH}_2$ ), 110.3– 110.4 (m,  $\text{PtC}\equiv\text{C}$ ), 116.0 (t,  $\text{PtC}\equiv\text{C}$ ), 129.6 ( $\text{Ph}$ ), 131.1– 131.3 (m,  $\text{Ph}$ ), 133.0 ( $\text{Ph}$ ), 135.3– 135.4 (m,  $\text{Ph}$ ), 191.5 (CHO);  $^{31}\text{P}$  { $^1\text{H}$ } NMR (202 MHz,  $\text{CDCl}_3$ ):  $\delta$  (P) 11.4 (s,  $^1J_{\text{PtP}}= 2346$  Hz); HRMS (FAB) m/z: Calcd. For  $\text{C}_{30}\text{H}_{41}\text{O}_2\text{P}_2{}^{195}\text{Pt}$ : 690.2230 [ $\text{M}^+$ ], found: 690.2227; Anal. Calcd. For  $\text{C}_{30}\text{H}_{40}\text{O}_2\text{P}_2\text{Pt}$ : C, 52.25; H, 5.85%; Found C, 52.33; H, 5.83%.

### Synthesis of the dialdehyde **3b** and **3c**.

To a solution of dialdehyde **3a** (0.41 g, 0.60 mmol) in  $\text{CH}_2\text{Cl}_2$  (100 ml), 2M triisopropylphosphine solution in toluene (1.5 ml) were added, and the mixture was stirred for about 3 h under an argon atmosphere. The solution has been further stirred for overnight under air atmosphere. The solvent was removed under reduced pressure, and chromatography on alumina with  $\text{CH}_2\text{Cl}_2$ : Hexane= 1:1 as an eluent gave the purified product **3b** and **3c** as a pale yellow powder.

**Dialdehyde 3b.** Yield: 0.11 g (24%); mp 171–173 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  (H) 1.18– 1.28 (9H, m,  $\text{PCH}_2\text{CH}_3$ ), 1.37– 1.46 (18H, m,  $\text{PCHCH}_3$ ), 2.10– 2.24 (6H, m,  $\text{PCH}_2\text{CH}_3$ ), 2.89– 3.02 (3H, m,  $\text{PCHCH}_3$ ), 7.32 (4H, d,  $J$ = 8.0 Hz, Ar– H), 7.72 (4H, d,  $J$ = 8.0 Hz, Ar– H), 9.92 (2H, s, CHO);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  (C) 8.362 ( $\text{PCH}_2\text{CH}_3$ ), 16.09– 16.54 (m,  $\text{PCH}_2\text{CH}_3$ ), 19.76 ( $\text{PCHCH}_3$ ), 24.36– 24.73 (m,  $\text{PCHCH}_3$ ), 111.1 ( $\text{PtC}\equiv\text{C}$ ), 117.6 (t,  $\text{PtC}\equiv\text{C}$ ), 129.7 ( $\text{Ph}$ ), 130.9 ( $\text{Ph}$ ), 132.8 ( $\text{Ph}$ ), 135.6 ( $\text{Ph}$ ), 191.5 (CHO);  $^{31}\text{P}$  { $^1\text{H}$ } NMR (202 MHz,  $\text{CDCl}_3$ ):  $\delta$  (P) 10.8 (d,  $^1J_{\text{PtP}}= 2344$  Hz,  $^2J_{\text{PP}}= 373$  Hz, PEt), 36.1 (d,  $^1J_{\text{PtP}}= 2406$  Hz,  $^2J_{\text{PP}}= 373$  Hz, P(*i*-Pr)); HRMS (FAB) m/z: Calcd. For  $\text{C}_{33}\text{H}_{46}\text{O}_2\text{P}_2{}^{195}\text{Pt}$ : 731.2621 [ $\text{M}^+$ ], found: 731.2624; Anal. Calcd. For  $\text{C}_{33}\text{H}_{46}\text{O}_2\text{P}_2\text{Pt}$ : C,

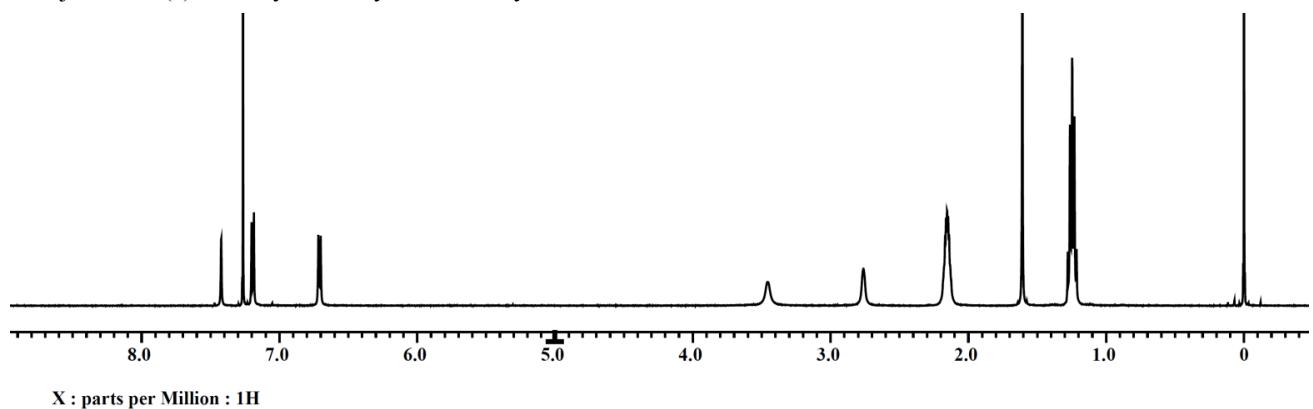
54.17; H, 6.34%; Found C, 54.34; H, 6.34%.

**Dialdehyde 3c.** Yield: 0.082 g (18%); mp >201 °C (decomp.);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  (H) 1.38–1.44 (36H, m,  $\text{CH}_3$ ), 2.90–3.10 (6H, m,  $\text{CH}$ ), 7.25 (4H, d,  $J$ = 8.0 Hz, Ar–H), 7.71 (4H, d,  $J$ = 8.0 Hz, Ar–H), 9.92 (2H, s,  $\text{CHO}$ );  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  (C) 20.41 ( $\text{CH}_3$ ), 24.93 (t, PCH), 112.4 (PtC≡C), 120.2 (t, PtC≡C), 130.4 (Ph), 131.3 (Ph), 133.3 (Ph), 136.6 (Ph), 192.2 (CHO);  $^{31}\text{P}$  { $^1\text{H}$ } NMR (202 MHz,  $\text{CDCl}_3$ ):  $\delta$  (P) 34.2 (s,  $^1J_{\text{PtP}}= 2401$  Hz); HRMS (FAB) m/z: Calcd. For  $\text{C}_{36}\text{H}_{52}\text{O}_2\text{P}_2{}^{195}\text{Pt}$ : 773.3090 [ $\text{M}^+$ ], found: 773.3124; Anal. Calcd. For  $\text{C}_{36}\text{H}_{52}\text{O}_2\text{P}_2\text{Pt}$ : C, 55.88; H, 6.77%; Found C, 55.80; H, 6.72%.

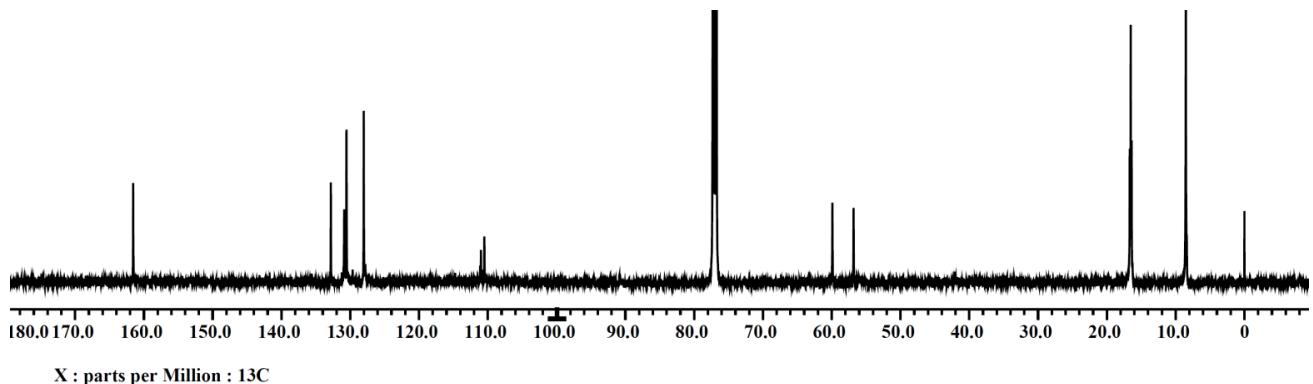
### General procedure for the synthesis of tetrahedral macrocycle 1.

A solution of tris(2-aminoethyl)amine **2** (0.10 mmol) in dry MeOH (100 mL) and a solution of dialdehyde **3** (0.15 mmol) in dry  $\text{CH}_2\text{Cl}_2$  (100 mL) were simultaneously added to dry MeOH (100 mL) at room temperature with stirring under an argon atmosphere. After the addition, the reaction mixture was stirred at room temperature for 2 days, and was concentrated under reduced pressure. The slightly yellow precipitate of the product was filtered off.

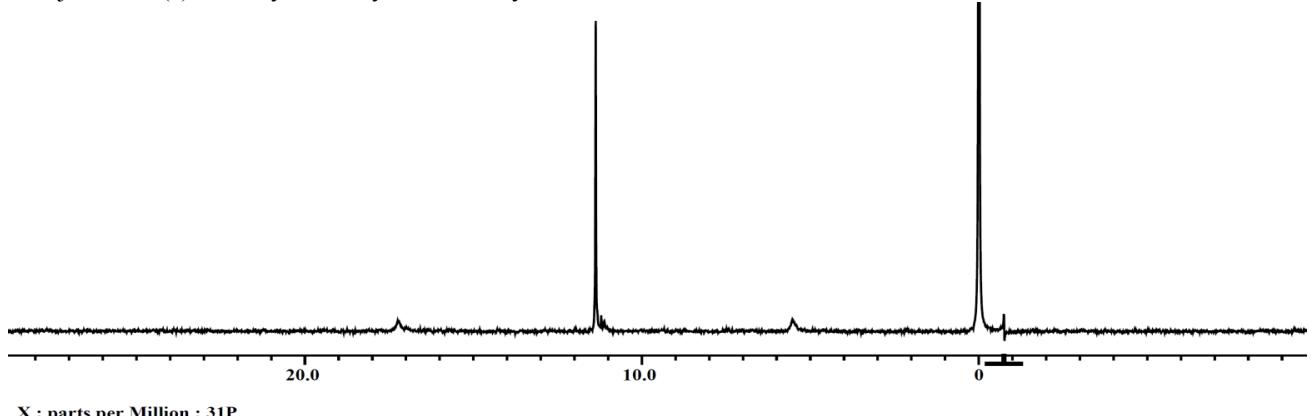
**Macrocycle 1a.** Yield: 0.069 g (64%); mp >115 °C (decomp.); LRMS (FAB) m/z: Calcd. For  $\text{C}_{204}\text{H}_{288}\text{N}_{16}\text{P}_{12}\text{Pt}_6$ : 4503.78 [ $\text{M}^+$ ], found: 4506.9; Anal. Calcd. For  $\text{C}_{204}\text{H}_{288}\text{N}_{16}\text{P}_{12}\text{Pt}_6$ : C, 54.37; H, 6.44; N, 4.97%; Found C, 54.18; H, 6.43; N, 5.00%.



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  (H) 1.15– 1.34 (108H, m,  $\text{CH}_3$ ), 2.06– 2.24 (72H, m,  $\text{PCH}_2$ ), 2.76 (24H, br,  $\text{CHNCH}_2\text{CH}_2$ ), 3.46 (24H, br,  $\text{CHNCH}_2\text{CH}_2$ ), 6.72 (24H, d,  $J= 8.0$  Hz, Ar– H), 7.19 (24H, d,  $J=8.0$  Hz, Ar– H), 7.43 (12H, s,  $\text{CH}=\text{N}$ ).

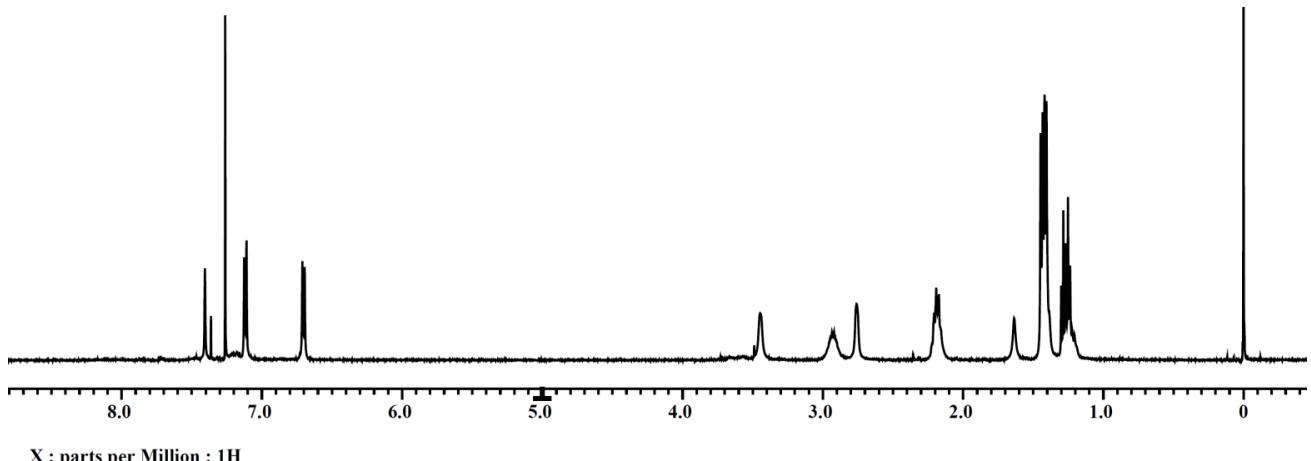


$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  (C) 8.518 ( $\text{CH}_3$ ), 16.54 (t,  $\text{PCH}_2$ ), 56.82 ( $\text{NCH}_2$ ), 59.90 ( $\text{NCH}_2$ ), 110.5 (PtC≡C), 111.0 (t, PtC≡C), 128.0 (Ph), 130.5 (Ph), 130.9 (Ph), 132.8 (Ph), 161.6 ( $\text{C}=\text{N}$ ).

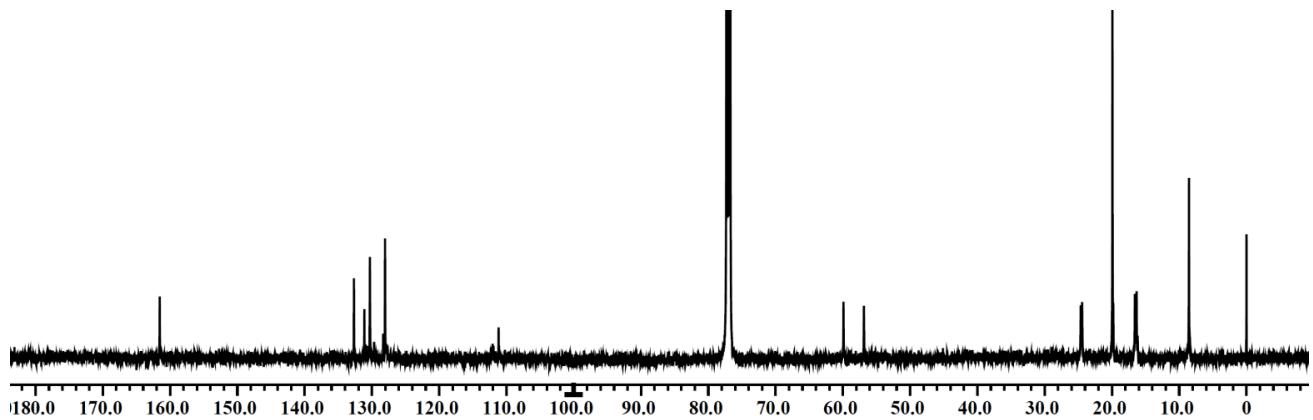


$^{31}\text{P}$  { $^1\text{H}$ } NMR (202 MHz,  $\text{CDCl}_3$ ):  $\delta$  (P) 11.5 (s,  $^1J_{\text{PtP}} = 2370$  Hz).

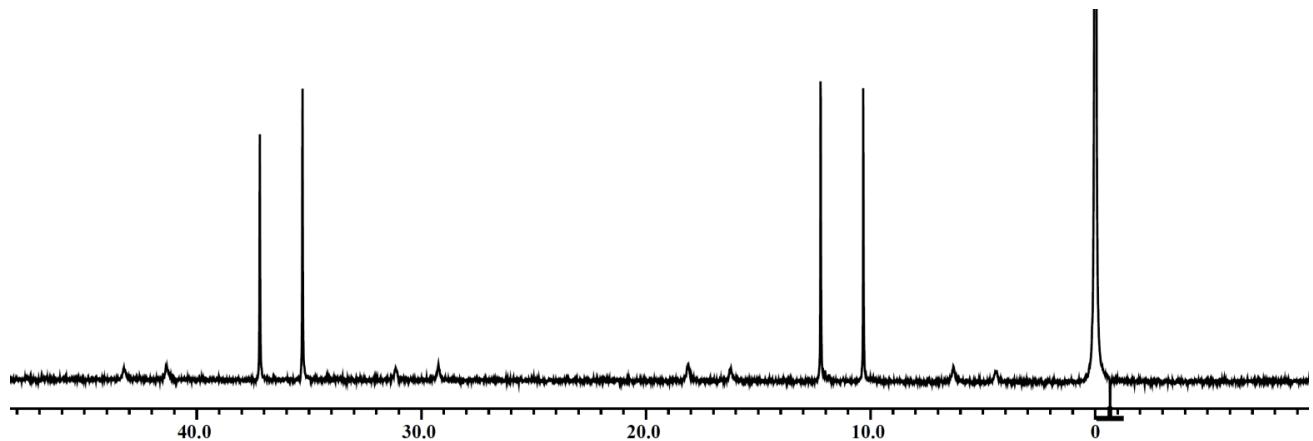
Macrocyclic **1b**. Yield: 48%; mp >80 °C (decomp.); LRMS (FAB) m/z: Calcd. For  $\text{C}_{222}\text{H}_{324}\text{N}_{16}\text{P}_{12}\text{Pt}_6$ : 4756.06 [ $\text{M}^+$ ], found: 4759.0; Anal. Calcd. For  $\text{C}_{222}\text{H}_{324}\text{N}_{16}\text{P}_{12}\text{Pt}_6$ : C, 56.03; H, 6.86; N, 4.71%; Found C, 55.73; H, 6.86; N, 4.71%.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (H) 1.21–1.32 (54H, m,  $\text{PCH}_2\text{CH}_3$ ), 1.38–1.47 (108H, m,  $\text{PCHCH}_3$ ), 2.13–2.24 (36H, m,  $\text{PCH}_2\text{CH}_3$ ), 2.76 (24H, br,  $\text{CHNCH}_2\text{CH}_2$ ), 2.87–2.98 (18H, m,  $\text{PCHCH}_3$ ), 3.45 (24H, br,  $\text{CHNCH}_2\text{CH}_2$ ), 6.69 (24H, d,  $J = 8.1$  Hz, Ar–H), 7.12 (24H, d,  $J = 7.8$  Hz, Ar–H), 7.40 (12H, s, CH=N).



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  (C) 8.546 ( $\text{PCH}_2\text{CH}_3$ ), 16.04– 16.81 (m,  $\text{PCH}_2\text{CH}_3$ ), 19.93 ( $\text{PCHCH}_3$ ), 24.29– 24.74 (m,  $\text{PCHCH}_3$ ), 56.87 ( $\text{NCH}_2$ ), 59.90 ( $\text{NCH}_2$ ), 111.2 ( $\text{PtC}\equiv\text{C}$ ), 112.1 (t,  $\text{PtC}\equiv\text{C}$ ), 128.1 (*Ph*), 130.3 (*Ph*), 131.1 (*Ph*), 132.7 (*Ph*), 161.5 ( $\text{C}=\text{N}$ ).



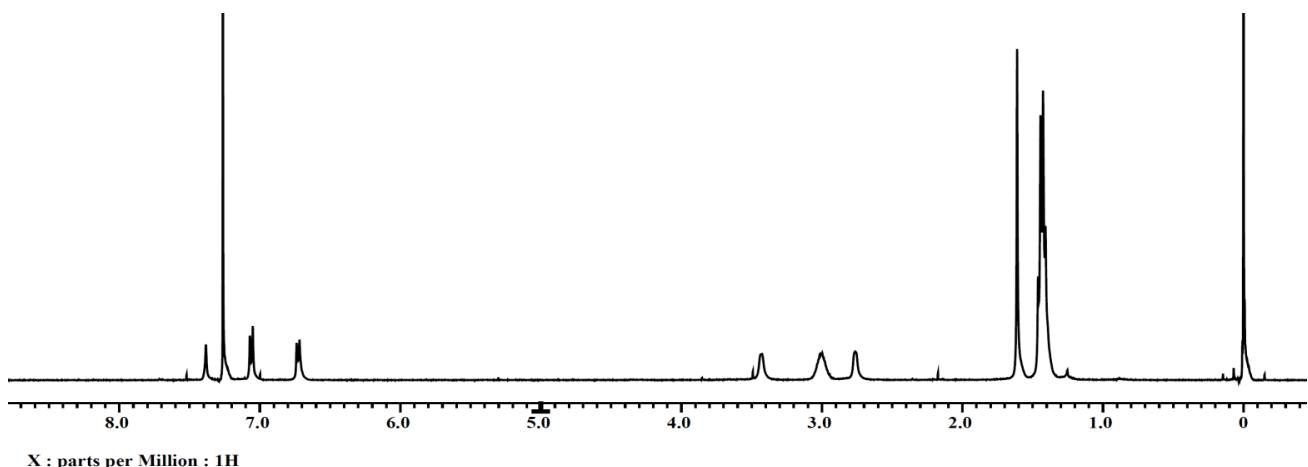
$^{31}\text{P}$  { $^1\text{H}$ } NMR (202 MHz,  $\text{CDCl}_3$ ):  $\delta$  (P) 11.3 (d,  $^1J_{\text{PtP}}=2382$  Hz,  $^2J_{\text{pp}}=384$  Hz, *PEt*), 36.2 (d,  $^1J_{\text{PtP}}=2445$  Hz,  $^2J_{\text{pp}}=384$  Hz, *P(i-Pr)*).

Macrocycle **1c**. Yield: 0.10 g (82%); mp >175 °C (decomp.); LRMS (ESI-MS) m/z: Calcd. For

$C_{240}H_{360}N_{16}P_{12}Pt_6$ : 5011.7 [M+H]<sup>+</sup>, found: 5016.8; Calcd.: 2506.8 [M+2H]<sup>2+</sup>, found: 2506.8; Calcd.: 1671.6

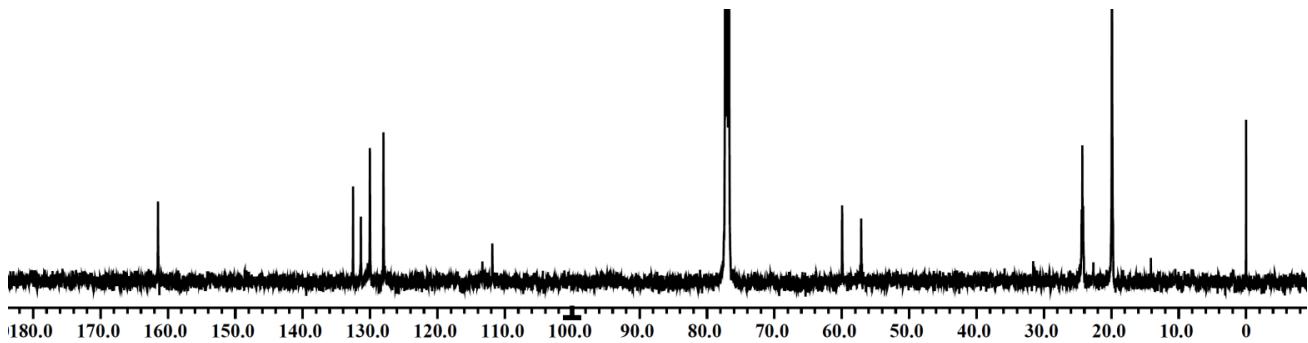
[M+3H]<sup>3+</sup>, found: 1671.5; Anal. Calcd. For  $C_{240}H_{360}N_{16}P_{12}Pt_6$ : C, 57.52; H, 7.24; N, 4.47%; Found C, 57.22; H,

7.24; N, 4.47%.



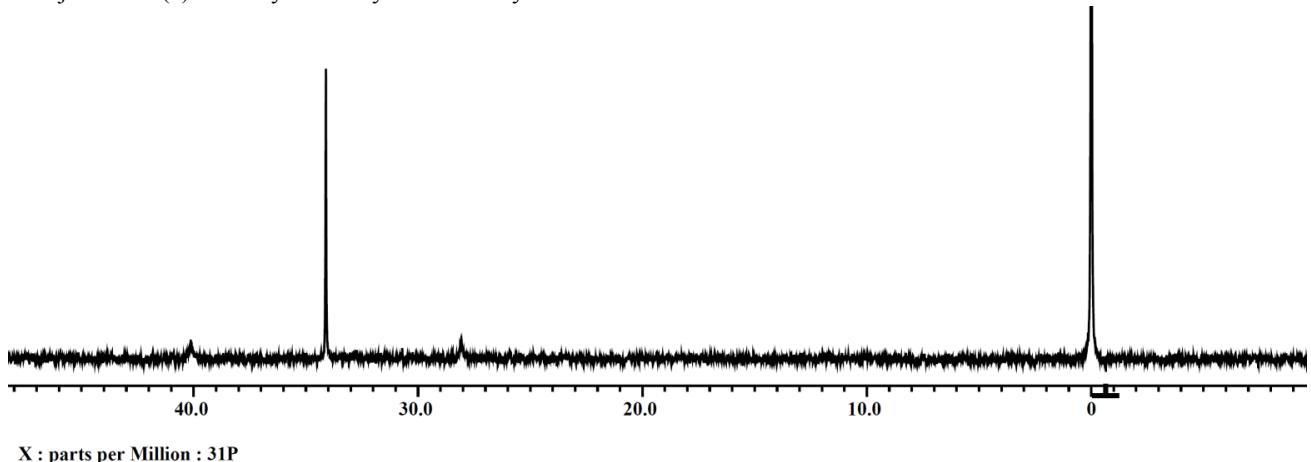
X : parts per Million : 1H

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ (H) 1.36– 1.50 (216H, m, CH<sub>3</sub>), 2.77 (24H, br, CHNCH<sub>2</sub>CH<sub>2</sub>), 2.95– 3.08 (36H, m, PCH), 3.44 (24H, br, CHNCH<sub>2</sub>CH<sub>2</sub>), 6.73 (24H, d, J= 8.3 Hz, Ar– H), 7.06 (24H, d, J=8.3 Hz, Ar– H), 7.39 (12H, s, CH=N).



X : parts per Million : 13C

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (C) 19.90 (CH<sub>3</sub>), 24.29 (t, PCH), 57.12 (NCH<sub>2</sub>), 59.95 (NCH<sub>2</sub>), 111.9 (PtC≡ C), 113.3 (PtC≡ C), 128.0 (Ph), 130.0 (Ph), 131.4 (Ph), 132.5 (Ph), 161.5 (C=N).



$^{31}\text{P} \{^1\text{H}\}$  NMR (202 MHz,  $\text{CDCl}_3$ ):  $\delta$  (P) 34.1 (s,  $^1J_{\text{PtP}} = 2432$  Hz).

#### PGSE-NMR measurements.

The PGSE NMR diffusion measurements were performed on a JEOL JNM-ECA600 (600 MHz) spectrometers equipped with a Z-axis field gradient system 30 A and TH5ATFG2 probe using bpp\_led\_dosy sequence. The shape of the gradient pulse was sine, and the gradient strength was calibrated against the value of the self-diffusion coefficient of HDO in  $\text{D}_2\text{O}$ . The values given below were used as the diffusion delay ( $\Delta$  [ms]) and gradient length ( $\delta$  [ms]),  $(\Delta, \delta) = (100, 2.1), (200, 1.5)$  and  $(300, 1.2)$  for dichloromethane- $d_2$  solution,  $(100, 2.0)$  and  $(200, 1.5)$  for benzene- $d_6$  solution,  $(100, 2.0), (200, 1.3)$  and  $(300, 1.12)$  for toluene- $d_8$  solution, and  $(100, 2.1), (200, 1.5)$  and  $(300, 1.2)$  for *p*-xylene- $d_{10}$  solution. The temperature was set to 298 K, and the samples were placed in a 3 mm tube (SHIGEMI corp.) for the fitting of the magnetization index. The  $D$  values were determined from the slope of the regression line  $\ln(I/I_0)$  versus  $g^2$ , according to the equation,  $\ln(I/I_0) = -(\gamma\delta)^2 g^2 (\Delta - \delta/3) (4/\pi^2) D$ , in which  $I/I_0$  = observed spin echo intensity/ intensity without gradients,  $g$  = gradient strength, and  $\gamma$  = gyromagnetic ratio, using the curve analysis tool of the software Delta vol. 4.3.6 (JEOL USA, Inc). The

determined value of  $D$  was used to calculate the hydrodynamic radius ( $R$ ) from the Stokes-Einstein equation,  $D = k_B T / 6\pi\eta R$ , in which the  $k_B$  is the Boltzmann constant,  $T$  is the absolute temperature, and  $\eta$  is the viscosity of the solvent. The internal standards, cyclohexane and trimethylsilane, were used to fit the viscosity of the deuterated solvents. By assuming that the hydrodynamic radius of the internal standards ( $R_{ref}$ ) were the same in various solvents, the viscosity of the solvent were fixed by using the measured diffusion coefficient ( $D_{ref}$ ) and the known value of the hydrodynamic radius ( $R_{ref}$ ) of the internal standard, as  $\eta = k_B T / 6\pi D_{ref} R_{ref}$ . Because of the low boiling point, the concentration of the trimethylsilane was not expected to be the same strict enough for internal standard. Thus cyclohexane was used as the internal standards to set the viscosity. The  $R_H$  values were calculated as  $R_H = D_{ref} R_{ref} / D$ .

### **DLS measurements.**

DLS measurements were conducted with an ALV DLS/ SLS-5000 light scattering system equipped with an ALV-5000 multiple  $\tau$  digital correlator for the collective diffusion coefficieant  $D$ . the measurements were taken in  $10^\circ$  angular increments from  $30^\circ$  to  $150^\circ$  for 30 s and 10 runs at each angle. The wave vector  $q$  is defined as  $q = (4n\pi/\lambda) \sin(\theta/2)$ , where  $n$  is the refractive index of the solution,  $\lambda$  is the wavelength of the incident beam ( $\lambda=635.8$  nm) operated by a 22 mW He-Ne laser, and the  $\theta$  is the scattering angle. The time-correlated signal decay was analyzed using CONTIN Laplace inversion routine. Fitting the relaxation rate versus  $q^2$  yielded the diffusion coefficients by  $\Gamma = D q^2$ . The relaxation rate  $\Gamma$  of each process was calculated as the inverse of its relaxation time. The hydrodynamic radius ( $R$ ) was calculated by as described above for PGSE-NMR. We used the

>99.8% pure solvent of dichloromethane after distillation and drying, >99.8% pure solvent of tetrahydrofuran after distillation and drying, >99.8% pure solvent of *p*-dioxane after drying, >99.8% pure solvent of benzene after distillation and drying, 99.7% pure solvent of toluene after drying, 98.0% pure solvent of *p*-xylene after distillation and drying, 99.8% pure solvent of ethylbenzene after drying, and 99% pure solvent of butylbenzene after distillation and drying. The known values of the viscosity of the pure solvent were used as the viscosity of the solvent.

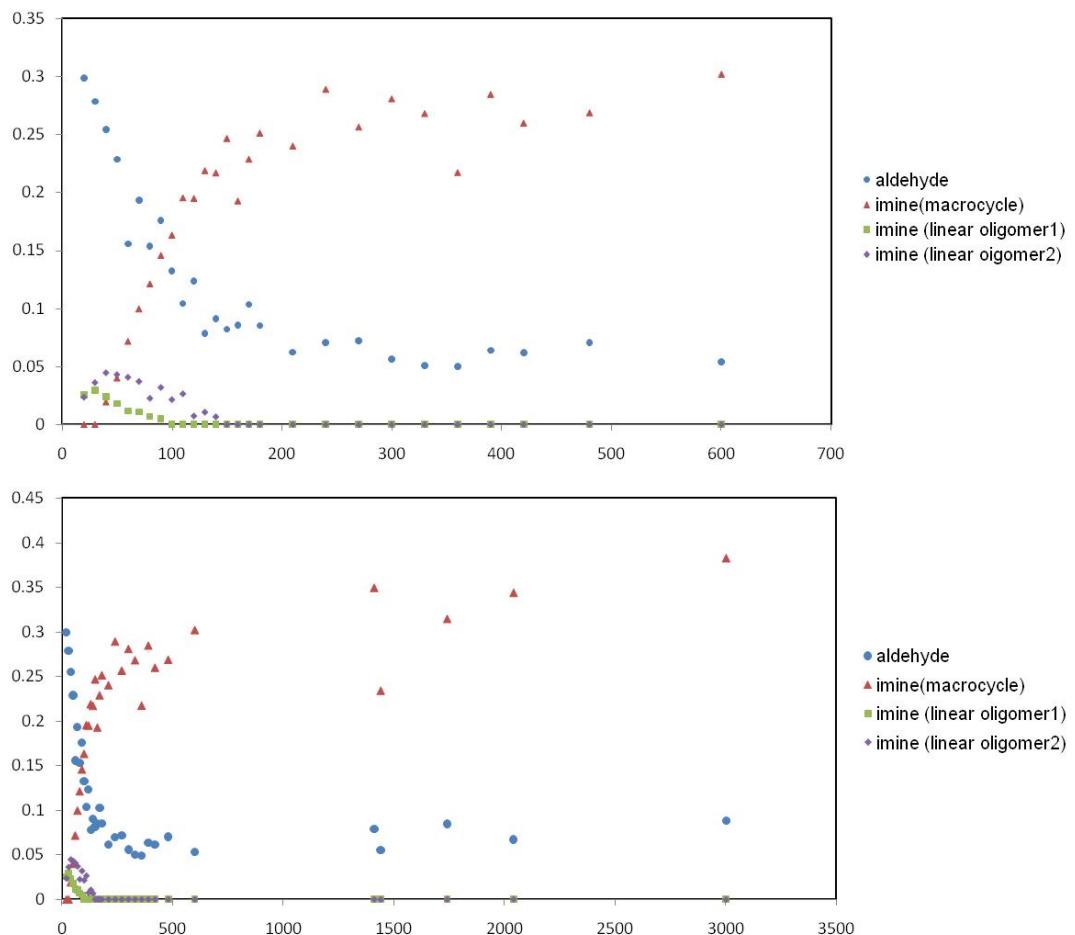
### Theoretical methods.

All calculations were performed using the Gaussian 03 suite of programs.<sup>1</sup> And the optimization of the structure of **1a** have been carried out at the HF level with the LANL2DZ basis set.<sup>2</sup>

<sup>1</sup> M. J. Frisch, G.W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, J. A. Montgomery, T. Vreven, K. N. Kudin, J. C. Burant, J.M. Millam, S. S. Iyengar, J. Tomasi, V. Barone, B. Mennucci, M. Cossi, G. Scalmani, N. Rega, G. A. Petersson, H. Nakatsuji, M. Hada, M. Ehara, K. Topyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Makajima, Y. Honda, O. Kitao, H. Nakai, M. Klene, X. Li, J. E. Know, H. P. Hratchian, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin,, R. Cammi, C. Pomelli, J. W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakrzewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Latham, C. Y. Peng, A. Nanayakkara, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C. Gonzalez, J. A. Pople, *Gaussian 03, Revision C. 02*, Gaussian, Inc.: Wallingford, CT, 2004.

<sup>2</sup> P. J. Hay, W. R. Wadt, *J. Chem. Phys.*, 1985, **82**, 270-283.

2.  $^1\text{H}$  NMR monitoring for the cyclization reaction.



**Fig. S1** Time-dependent intensity ratio of the reaction components on  $^1\text{H}$  NMR spectrum for the dehydrative condensation reaction between triamine **2** and dialdehyde **3a**. The reaction had been performed in a NMR tube, and the intensity has been estimated by calculating the relative intensity against internal standard, trimethylsilane. Because the higher concentration had been needed to measure the  $^1\text{H}$  NMR spectrum, the reaction attained less conversion (ca. 80%) than that of the case of high diluted cyclization reaction which has been routinely performed.

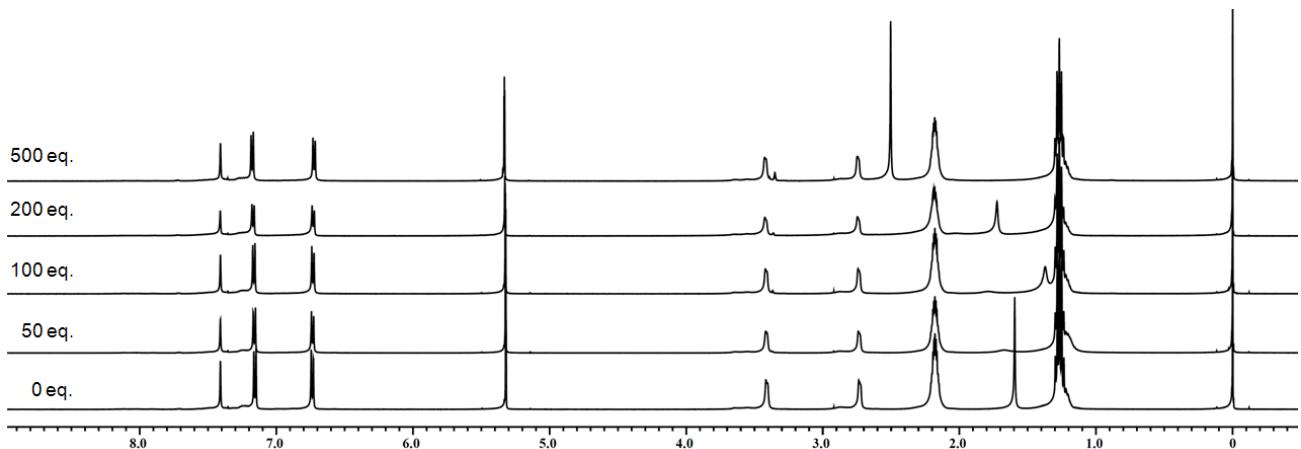
3. Crystal data.

**Table S1** Summary of crystallographic data and refinement details of **1a·MeOH**.

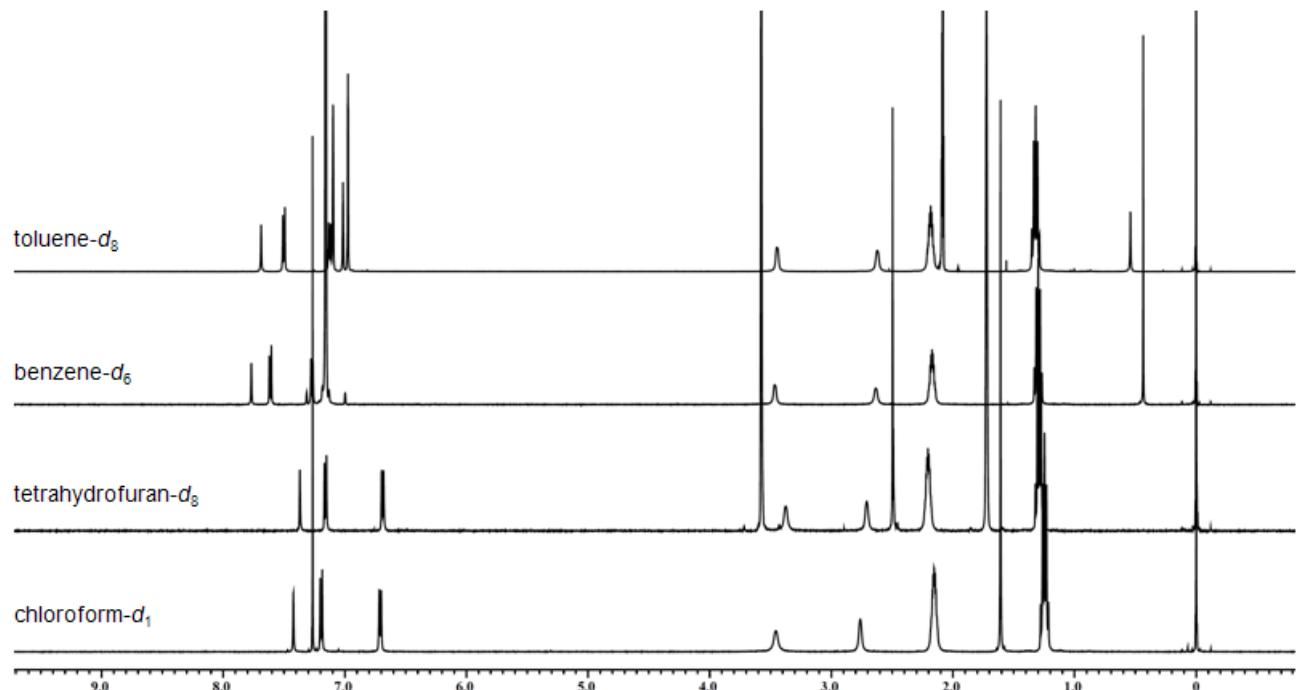
	<b>1a·MeOH</b>		<b>1a·MeOH</b>
Empirical formula	C <sub>205</sub> H <sub>292</sub> N <sub>16</sub> OP <sub>12</sub> Pt <sub>6</sub>	Z	2

M <sub>w</sub>	4538.89	D <sub>calc.</sub> [g/cm <sup>3</sup> ]	1.328
Crystal color and habit	colorless, block	F (000)	4580
Crystal size [mm]	0.15×0.12×0.07	μ [cm <sup>-1</sup> ]	38.02 (Mo-Kα)
Crystal system	triclinic	2θ <sub>max</sub>	54.96 ( I > 2.0σ)
Space group	P-1 (#2)	No. of reflections	115542
Temperature [K]	133	measured	51700
a [Å]	21.913(3)	R <sub>int</sub>	0.0540
b [Å]	23.156(3)	No. observations	51700
c [Å]	23.850(4)	No. of parameters	1435
α [°]	93.385(6)	Reflection/Parameter ratio	36.03
β [°]	108.751(6)	R	0.0888
γ [°]	95.706(5)	R <sub>w</sub>	0.2755
V = [Å <sup>3</sup> ]	11350(3)	GOF	1.031

4. NMR titration of **1a**.

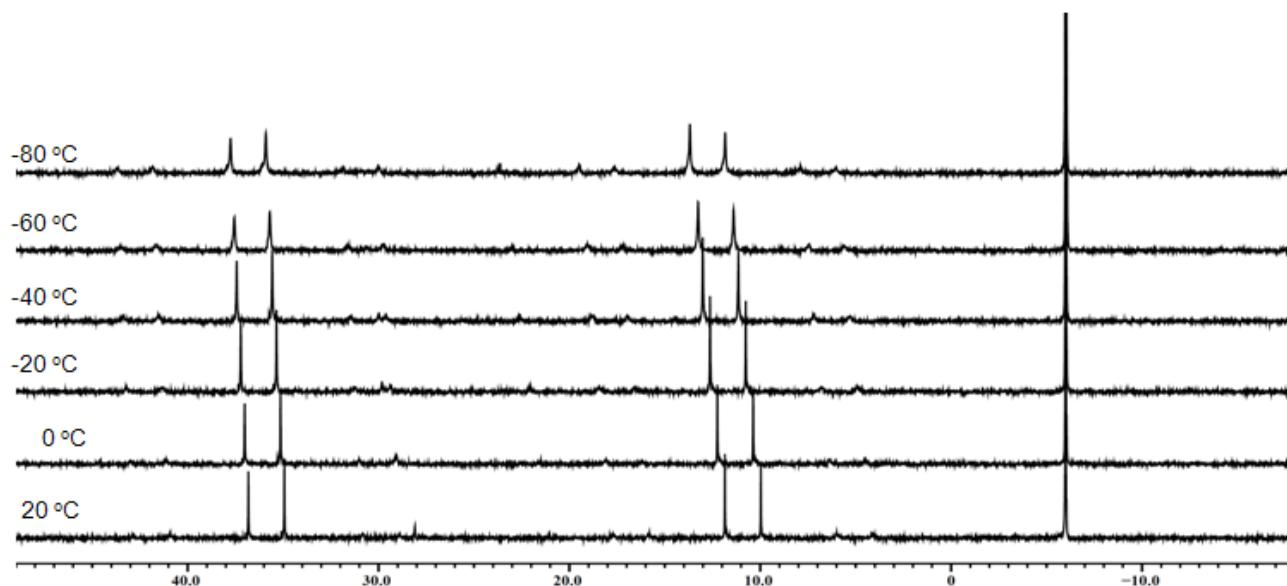


**Fig. S2** NMR titration study of **1a** in  $\text{CD}_2\text{Cl}_2$  (guest molecule: Me-d<sub>3</sub>OD, 500 MHz).



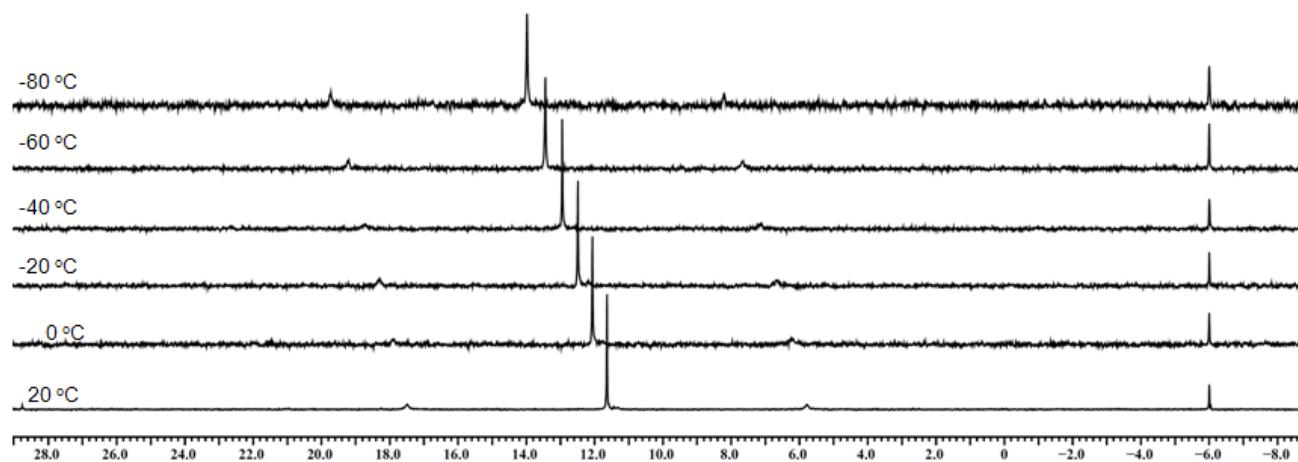
**Fig. S3** <sup>1</sup>H NMR spectra of **1a** in  $\text{CDCl}_3$ , THF-d<sub>8</sub>, benzene-d<sub>6</sub>, and toluene-d<sub>8</sub> (500 MHz).

5.  $^{31}\text{P}$  NMR spectrum of **1b**.

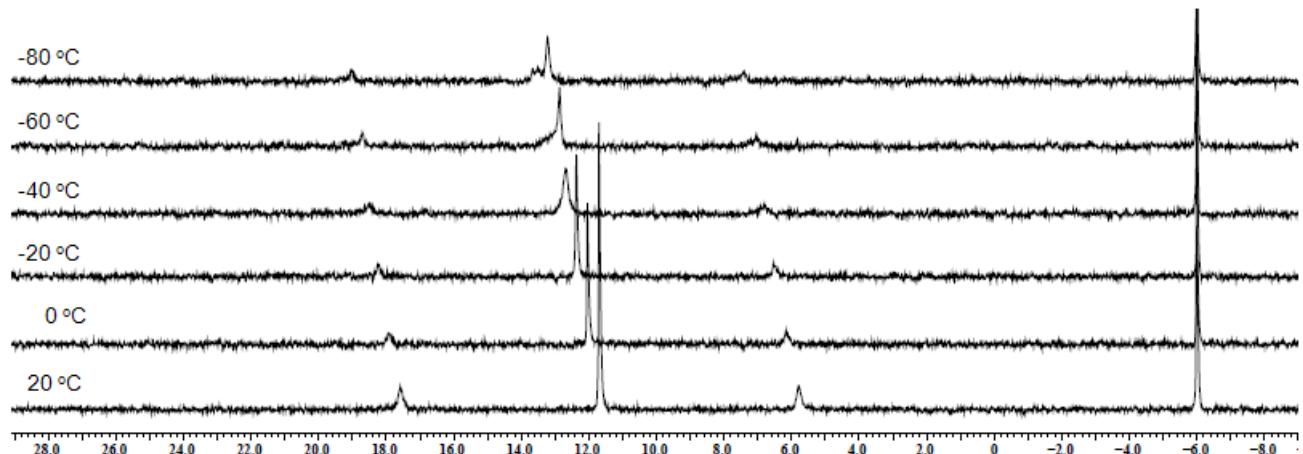


**Fig. S4** VT  $^{31}\text{P}$  NMR spectrum of **1b** in  $\text{CD}_2\text{Cl}_2$  (202 MHz).

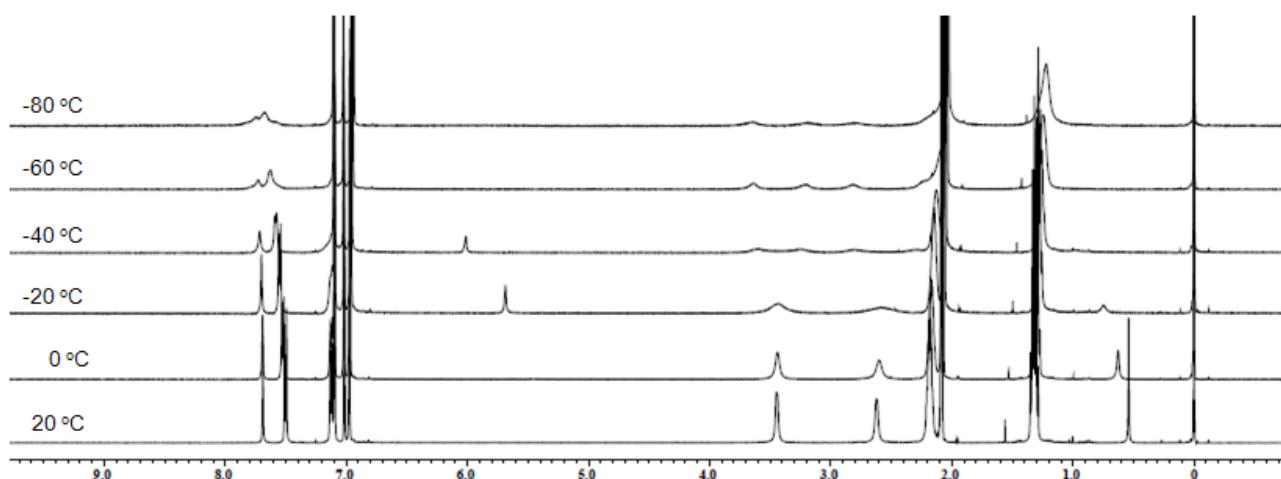
6. VT NMR spectra of **1a**.



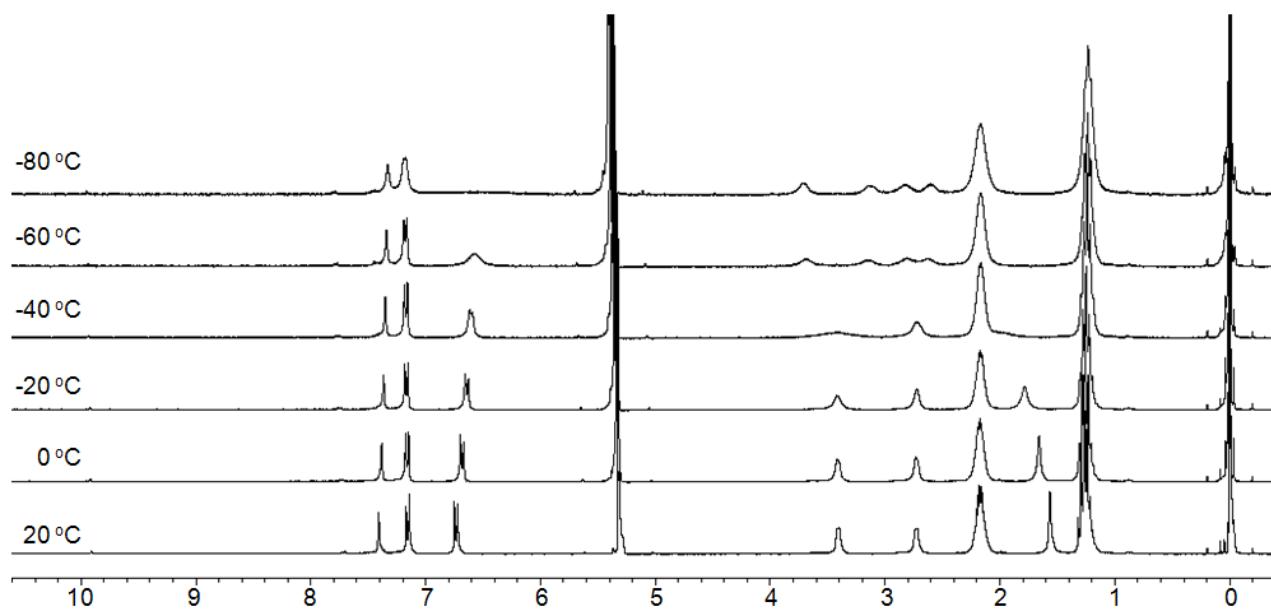
**Fig. S5** VT  $^{31}\text{P}$  NMR spectra of **1a** in  $\text{CD}_2\text{Cl}_2$  (202 MHz).



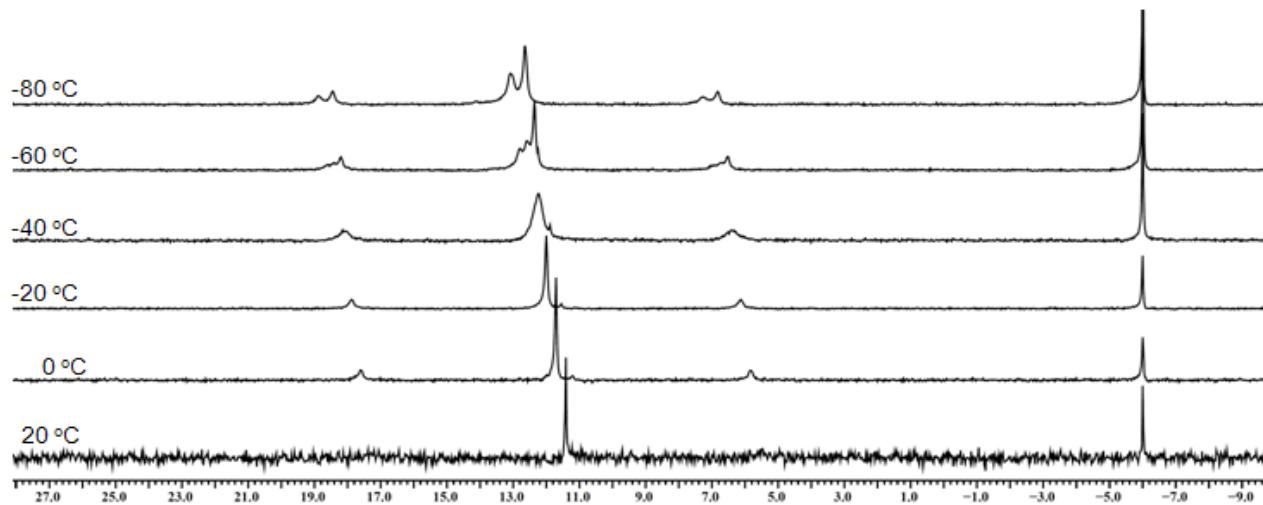
**Fig. S6** VT  $^{31}\text{P}$  NMR spectra of **1a** in toluene- $d_8$  (202 MHz).



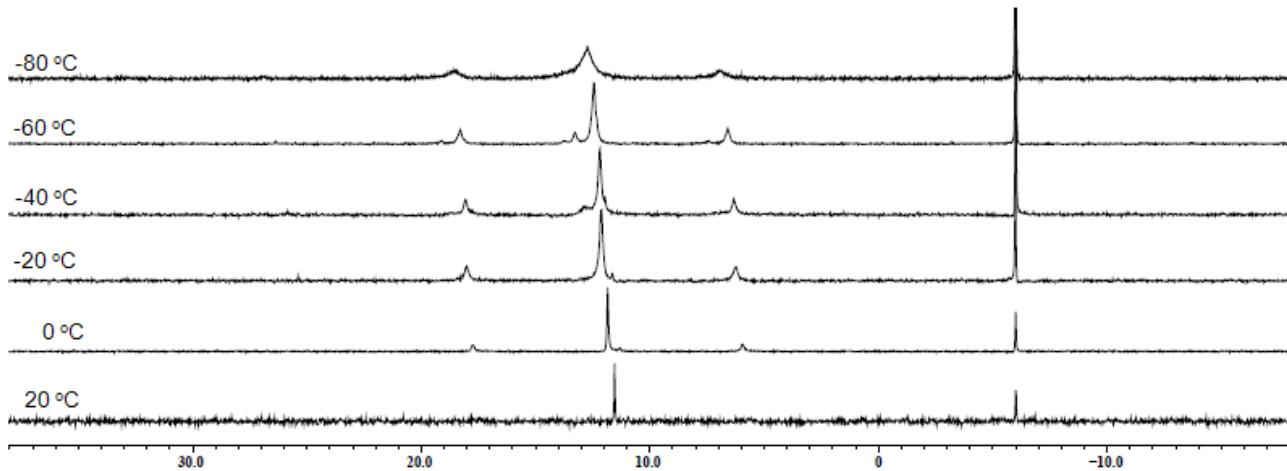
**Fig. S7** VT  $^1\text{H}$  NMR spectra of **1a** in toluene- $d_8$  (500 MHz).



**Fig. S8** VT  $^1\text{H}$  NMR spectra of **1a** in  $\text{CD}_2\text{Cl}_2$  (300 MHz).

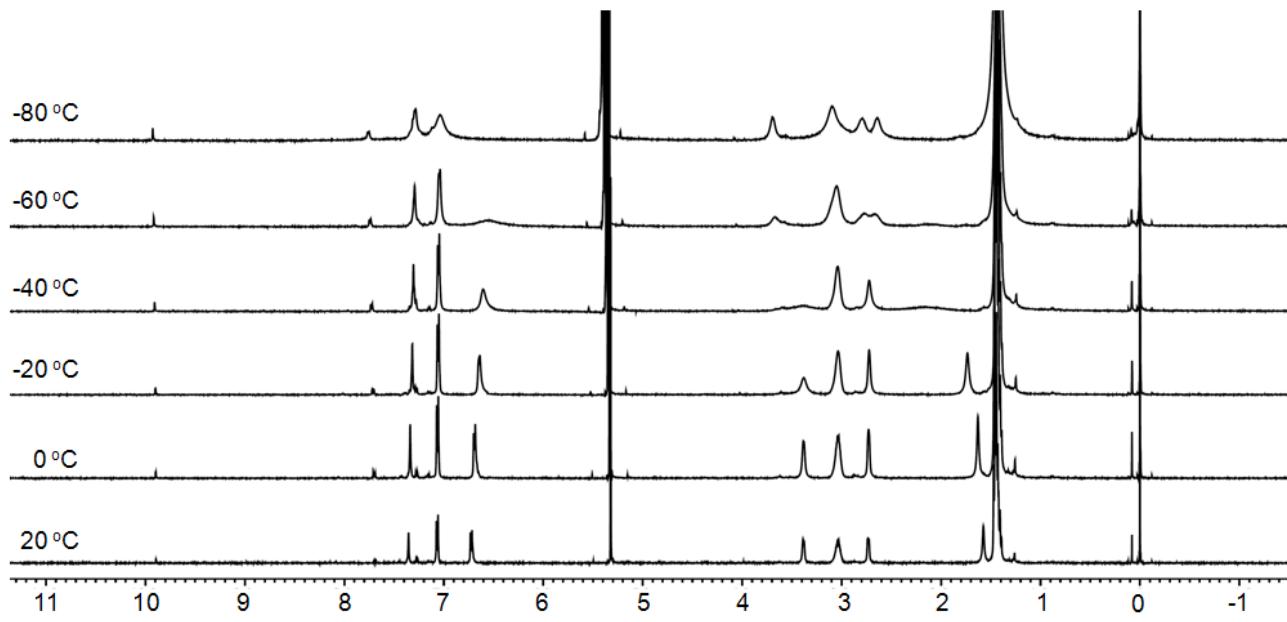


**Fig. S9** VT  $^{31}\text{P}$  NMR spectra of **1a** in ethylbenzene (202 MHz).



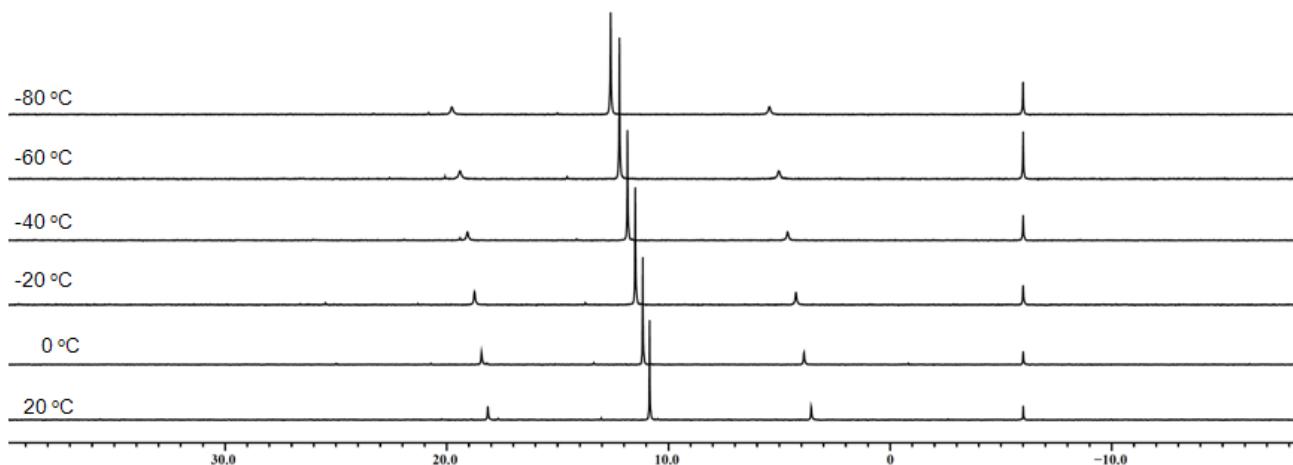
**Fig. S10** VT  $^{31}\text{P}$  NMR spectra of **1a** in butylbenzene (202 MHz)..

7. VT NMR spectra of **1c**.

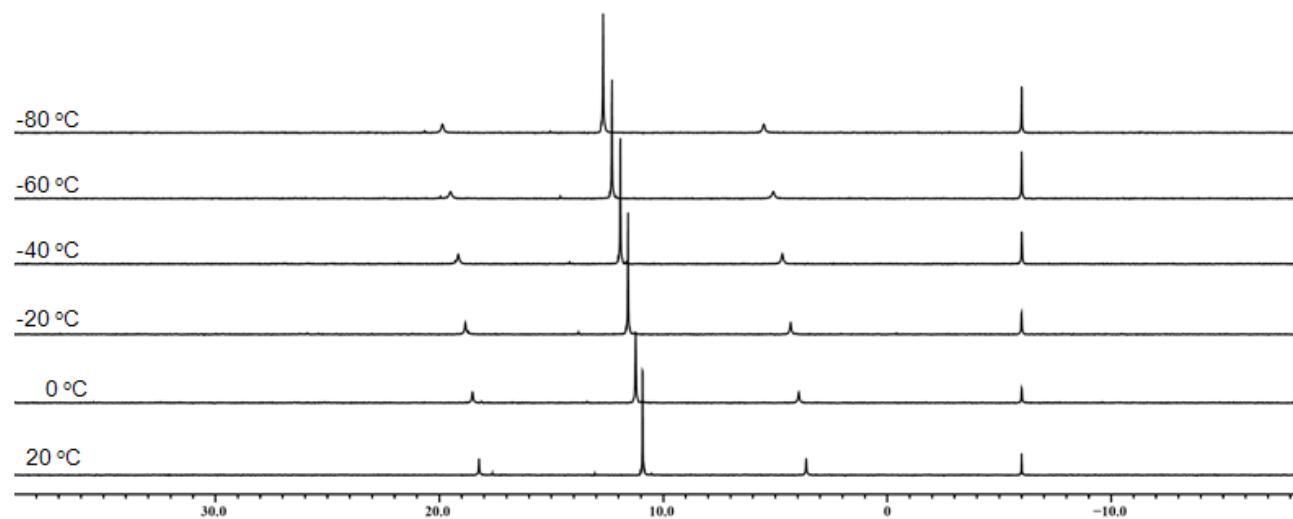


**Fig. S11** VT  $^1\text{H}$  NMR spectra of **1c** in  $\text{CD}_2\text{Cl}_2$  (500 MHz).

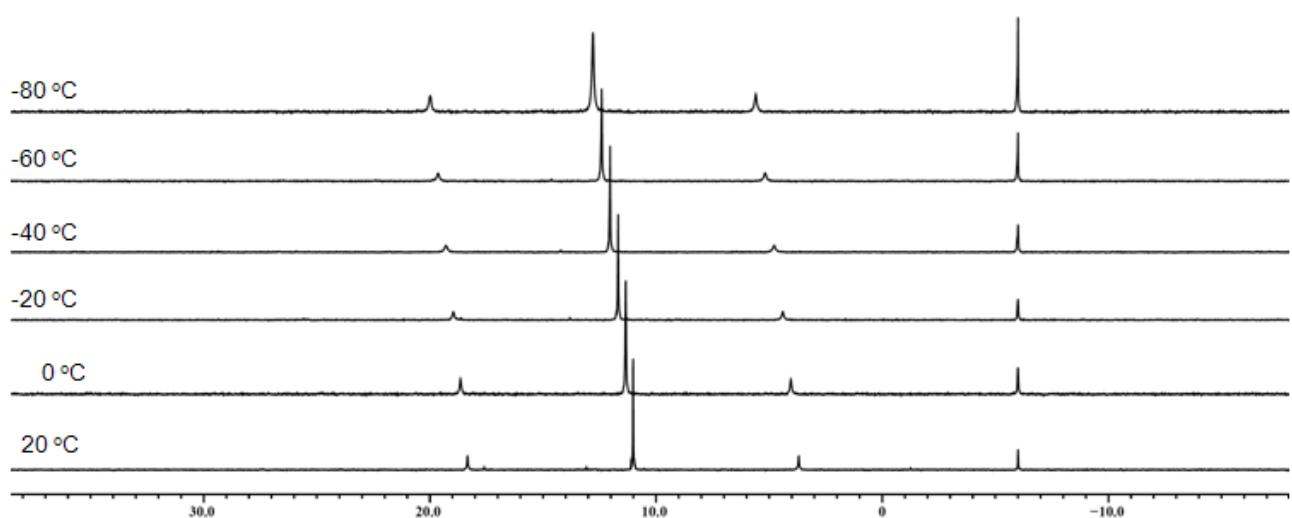
8. VT NMR spectra of **3a**.



**Fig. S12** VT  $^{31}\text{P}$  NMR spectra of **3a** in toluene- $d_8$  (162 MHz).



**Fig. S13** VT  $^{31}\text{P}$  NMR spectra of **3a** in ethylbenzene (162 MHz).



**Fig. S14** VT  $^{31}\text{P}$  NMR spectra of **3a** in butylbenzene (162 MHz).

9. Diffusion coefficients.

**Table S2** Measured  $D$  values of dichloromethane- $d_2$  solution of **1a** by PGSE-NMR measurements.

	$\mathcal{A}$ (ms)	$\delta$ (ms)		$D$ ( $\times 10^{-10}$ m/s)			averaged $D$	averaged
Entry			<b>1</b>	<i>c</i> -hexane	TMS	residual proton of $\text{CD}_2\text{Cl}_2$	( $\times 10^{-10}$ m/s)	$R_H$ (nm)
a1	100	2.1	4.11	25.9	24.5		33.0	
a2			4.07	25.6	24.5		34.0	
a3			4.11	25.6	24.3		32.5	
a4			3.98	26.0	23.8		33.0	
b1	200	1.5	3.98	25.1	23.1		31.1	
b2			3.90	24.8	23.0		31.9	4.00
b3			3.91	24.8	23.0		32.2	1.09
b4			3.87	24.8	23.2		31.5	
c1	300	1.2	4.00	25.5	24.6		33.7	
c2			4.08	25.2	25.0		33.4	
c3			3.97	25.0	24.9		33.3	
c4			4.03	24.7	25.0		32.3	

**Table S3** Measured  $D$  values of benzene- $d_6$  solution of **1a** by PGSE-NMR measurements.

	$\mathcal{A}$ (ms)	$\delta$ (ms)		$D$ ( $\times 10^{-10}$ m/s)			averaged $D$	averaged
Entry			<b>1</b>	<i>c</i> -hexane	TMS	residual proton of benzene- $d_6$	( $\times 10^{-10}$ m/s)	$R_H$ (nm)
a1	100	2.0	2.63	18.0	20.8	19.7		
a2			2.64	18.2	20.4	19.5		
a3			2.64	18.4	20.0	20.0		
b1	200	1.5	2.65	18.0	17.3	19.7		
b2			2.63	17.8	17.2	19.7	2.64	1.18
b3			2.64	17.9	16.9	19.6		
c1	200	1.5	2.65	18.0	16.7	19.7		
c2			2.64	18.1	17.4	19.6		
c3			2.65	17.8	16.8	19.5		

**Table S4** Measured  $D$  values of toluene- $d_8$  solution of **1a** by PGSE-NMR measurements.

	$\Delta$ (ms)	$\delta$ (ms)		$D$ ( $\times 10^{-10}$ m/s)			averaged $D$	averaged
Entry			<b>1</b>	<i>c</i> -hexane	TMS	residual proton of toluene- $d_8$	( $\times 10^{-10}$ m/s)	$R_H$ (nm)
a1	200	1.5	2.91	19.9	18.6	19.7		
a2			2.92	19.8	18.5	20.2		
a3			2.93	19.5	18.4	20.2		
a4			2.93	19.7	18.5	19.7		
b1	300	1.2	2.92	19.6	20.5	19.7		
b2			2.92	19.5	20.3	19.4		
b3			2.94	19.5	20.7	19.4	2.93	1.15
b4			2.91	19.7	20.4	19.2		
c1	400	1.0	2.96	19.3	18.7	18.5		
c2			2.93	19.0	18.7	19.5		
c3			2.96	19.7	19.3	19.3		
c4			2.89	19.1	19.4	18.0		

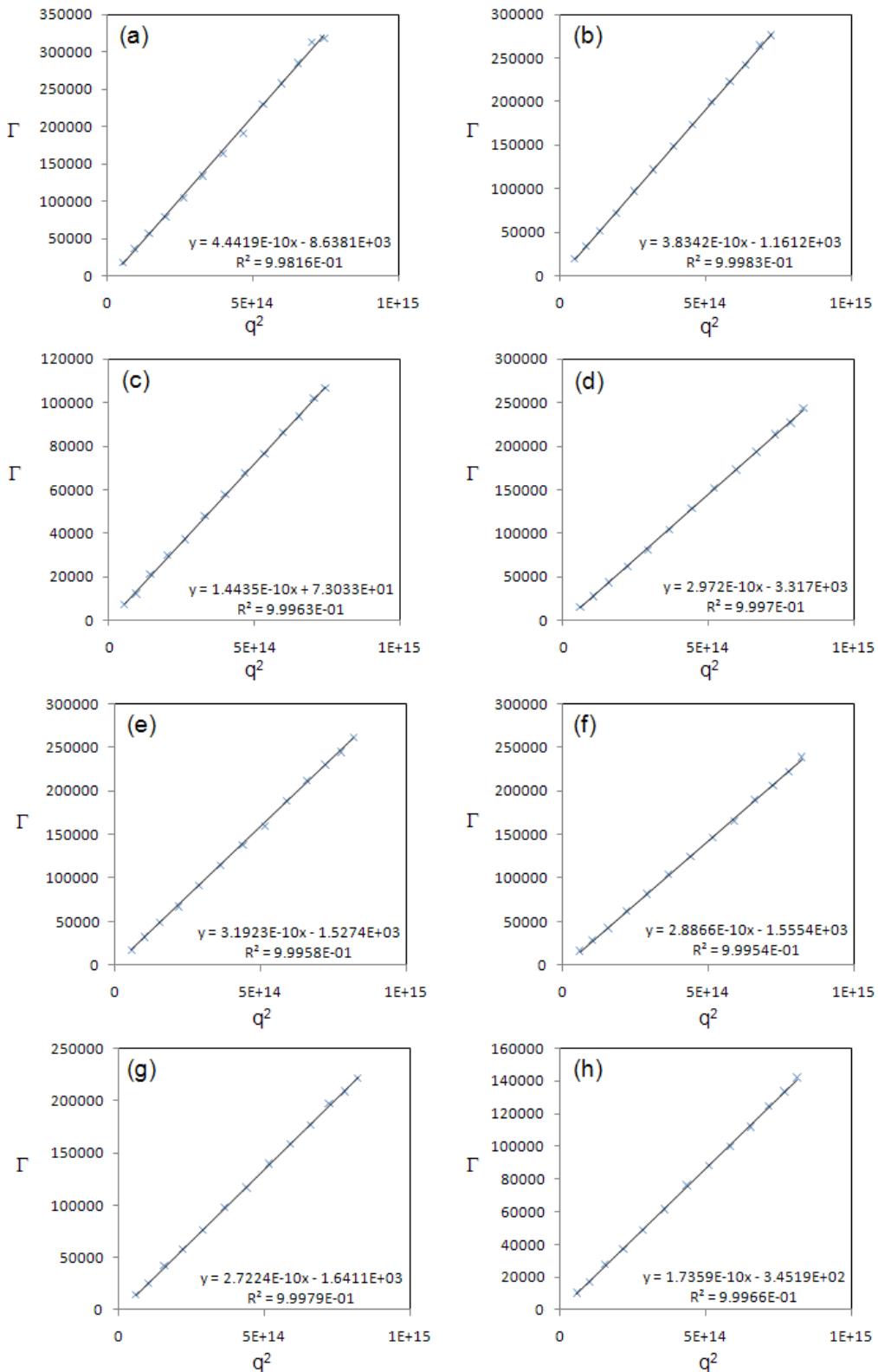
**Table S5** Measured  $D$  values of *p*-xylene- $d_{10}$  solution of **1a** by PGSE-NMR measurements.

	$\Delta$ (ms)	$\delta$ (ms)		$D$ ( $\times 10^{-10}$ m/s)			averaged $D$	averaged
Entry			<b>1</b>	<i>c</i> -hexane	TMS	residual proton of <i>p</i> -xylene- $d_{10}$	( $\times 10^{-10}$ m/s)	$R_H$ (nm)
a1	100	2.1	2.69	18.6	18.9	19.0		
a2			2.67	18.9	19.0	18.7		
a3			2.71	18.9	19.0	19.0		
a4			2.68	19.1	18.9	18.9		
b1	200	1.5	2.66	18.7	19.1	18.6		
b2			2.67	18.5	19.0	18.9	2.68	1.20
b3			2.70	18.5	19.5	18.6		
b4			2.69	18.6	19.8	18.9		
c1	300	1.2	2.64	18.8	18.9	18.4		
c2			2.71	18.8	18.8	18.5		
c3			2.70	18.7	19.0	18.3		
c4			2.68	18.6	18.7	18.5		

**Table S6** Measured  $D$  values of tetrahydrofuran- $d_8$  solution of **1a** by PGSE-NMR measurements.

Entry	$\Delta$ (ms)	$\delta$ (ms)	$D$ ( $\times 10^{-10}$ m/s)				averaged $D$ ( $\times 10^{-10}$ m/s)	$R_H$ (nm)
			<b>1</b>	<i>c</i> -hexane	TMS	residual proton of <i>p</i> -xylene- $d_{10}$		
a1	100	2.1	3.37	21.2	20.0		23.0	
a2			3.35	21.8	20.9		24.3	
a3			3.36	21.8	20.4		24.6	
a4			3.38	20.8	20.2		23.2	
b1	200	1.4	3.35	21.4	21.0		25.2	
b2			3.33	21.0	20.9		24.2	3.35
b3			3.30	22.0	21.8		25.0	1.10
b4			3.38	21.6	21.9		24.7	
c1	300	1.2	3.35	20.4	18.4		24.2	
c2			3.31	20.9	18.6		24.0	
c3			3.33	21.2	18.6		24.0	
c4			3.34	20.8	18.5		23.7	



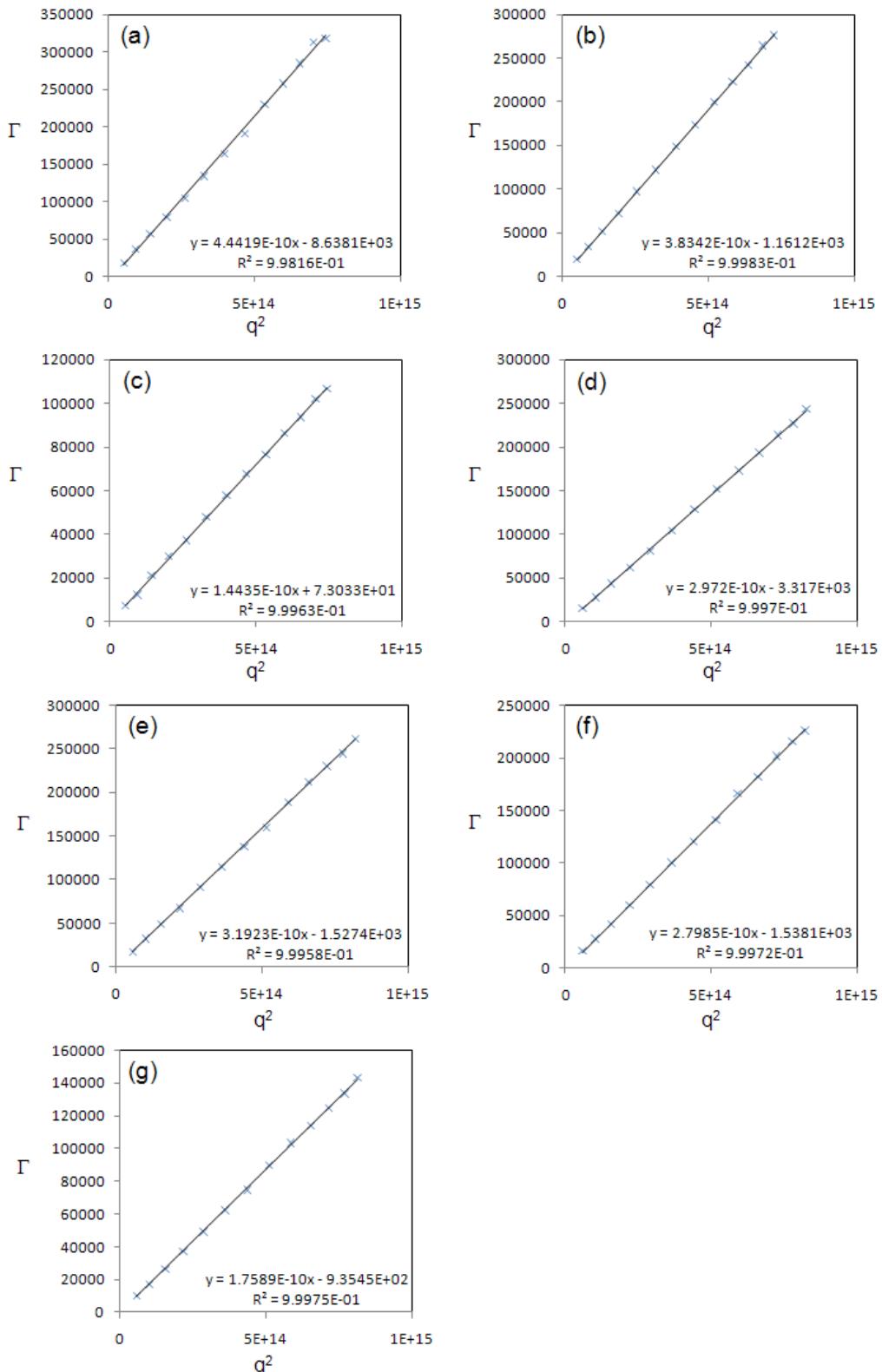


**Fig. S15** The results of DLS measurements ( $q^2$  vs.  $\Gamma$ ) for **1a**, dichloromethane (a), tetrahydrofuran (b),

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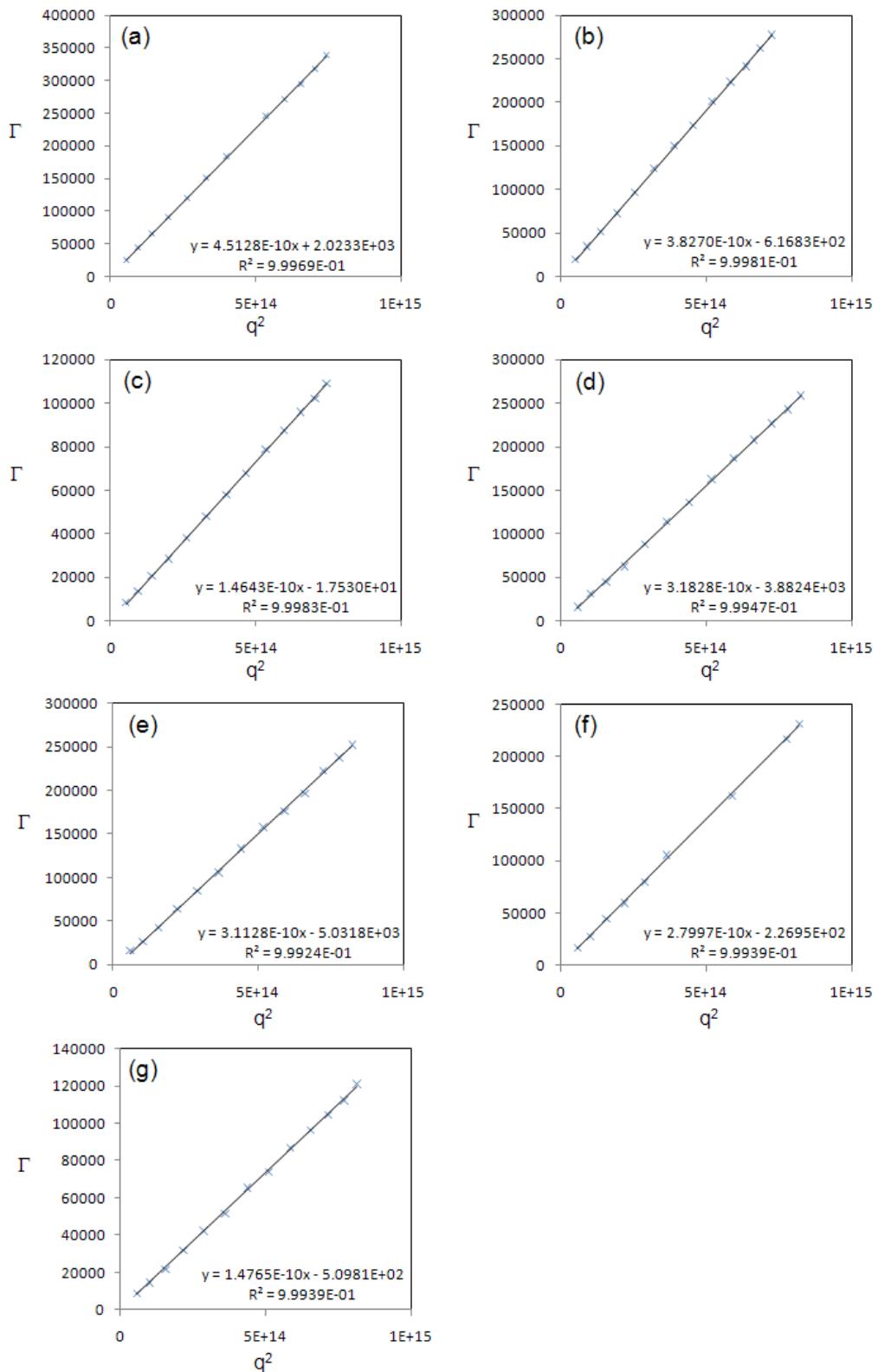
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*p*-dioxane (c), benzene (d), toluene (e), *p*-xylene (f), ethylbenzene (g), and butylbenzene (h).



**Fig. S16** The results of DLS measurements ( $q^2$  vs.  $\Gamma$ ) for **1c**, dichloromethane (a), tetrahydrofuran (b),

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*p*-dioxane (c), benzene (d), toluene (e), ethylbenzene (f), and butylbenzene (g).



**Fig. S17** The results of DLS measurements ( $q^2$  vs.  $\Gamma$ ) for **4**, dichloromethane (a), tetrahydrofuran (b),

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*p*-dioxane (c), benzene (d), toluene (e), *p*-xylene (f), and butylbenzene (g).

10. Theoretical methods.

**Table S7** Calculated coordinates of **1a**.

Center	Atomic Number	Coordinates (Angstroms)							
		X	Y	Z	34	6	-5.070131	4.100259	-5.494353
1	7	6.723542	11.890083	5.168931	35	6	-8.031617	4.067399	-7.835631
2	6	6.855401	12.800286	4.014573	36	7	-8.844306	4.840525	-8.427444
3	6	7.161804	12.081812	2.694840	37	6	-9.895327	4.320025	-9.298644
4	7	5.938784	11.574738	2.076417	38	6	-11.268775	4.835991	-8.860946
5	6	5.759717	10.333052	1.884995	39	7	-11.831941	4.084475	-7.723876
6	6	4.549455	9.795873	1.224351	40	6	5.850064	12.449036	6.219071
7	6	3.516109	10.638745	0.790879	41	6	4.354598	12.219227	5.972596
8	6	2.409316	10.120396	0.132768	42	7	3.978509	10.851701	6.316407
9	6	2.296504	8.739734	-0.118789	43	6	3.533431	10.038526	5.451249
10	6	3.322800	7.899563	0.334870	44	6	3.130763	8.657663	5.798188
11	6	4.434704	8.422581	0.992875	45	6	3.226606	8.180003	7.113023
12	6	1.170088	8.215235	-0.850509	46	6	2.841622	6.883888	7.423921
13	6	0.237565	7.790911	-1.513518	47	6	2.335800	6.019687	6.433015
14	78	-1.291939	7.067970	-2.666946	48	6	2.228572	6.508166	5.123689
15	15	-2.381106	6.305403	-0.639937	49	6	2.625611	7.807378	4.812028
16	6	-2.436246	7.611219	0.710740	50	6	1.937922	4.674973	6.764192
17	6	-1.423164	4.911841	0.177171	51	6	1.623264	3.532957	7.049592
18	6	-1.257322	3.669883	-0.714034	52	78	1.063189	1.614298	7.500947
19	6	-4.140761	5.676956	-0.821990	53	15	3.108479	1.424631	8.799500
20	15	-0.123870	7.826279	-4.666274	54	6	3.520073	2.909574	9.881226
21	6	1.037237	9.286431	-4.444091	55	6	2.333347	3.512897	10.651338
22	6	0.352412	10.571277	-3.949386	56	6	4.602665	1.336934	7.659440
23	6	-1.227451	8.314918	-6.110717	57	6	4.497572	0.260946	6.566468
24	6	-0.539936	8.460064	-7.478805	58	6	3.341171	-0.081928	9.897828
25	6	1.040248	6.496865	-5.306594	59	6	2.439027	-0.139980	11.140824
26	6	0.360495	5.161243	-5.648009	60	15	-0.912322	1.823954	6.105409
27	6	-2.813297	6.342875	-3.820876	61	6	-0.562785	1.242123	4.350266
28	6	-3.731598	5.934726	-4.509566	62	6	0.595061	1.972448	3.651200
29	6	-4.804440	5.469951	-5.351714	63	6	-1.598401	3.557697	5.897340
30	6	-5.615421	6.379353	-6.056304	64	6	-1.994479	4.223451	7.225739
31	6	-6.659439	5.934658	-6.856475	65	6	-2.367346	0.750212	6.631898
32	6	-6.922064	4.564295	-6.993041	66	6	-3.717125	1.045398	5.954499
33	6	-6.110166	3.655747	-6.308547	67	6	0.532061	-0.310282	7.936143

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69	6	-0.110880	-2.815795	8.554017	84	6	7.238181	7.097743	4.126038
70	6	-0.290534	-3.214967	9.892274	85	6	8.293417	7.031742	3.204637
71	6	-0.643231	-4.521274	10.207383	86	6	8.330825	6.032673	2.242112
72	6	-0.821279	-5.477178	9.197541	87	6	7.317359	5.057262	2.170731
73	6	-0.640450	-5.086750	7.868318	88	6	6.266226	5.126697	3.095536
74	6	-0.294146	-3.776521	7.549546	89	6	6.229750	6.133487	4.059037
75	6	-1.187906	-6.878564	9.495774	90	6	7.365966	4.018672	1.173081
76	7	-1.375586	-7.314020	10.672837	91	6	7.439733	3.160183	0.311097
77	6	-1.703730	-8.721127	10.907990	92	78	7.572343	1.800245	-1.212356
78	6	-3.021601	-8.846931	11.682710	93	15	5.474818	2.627988	-2.123046
79	7	-4.218074	-8.764665	10.821397	94	6	5.732558	4.078750	-3.293944
80	6	8.032779	11.462389	5.700639	95	6	6.200185	5.379214	-2.620376
81	6	8.008194	10.063142	6.325929	96	6	4.249365	3.205264	-0.819717
82	7	8.077461	9.041399	5.283189	97	6	2.912004	3.756768	-1.341613
83	6	7.181654	8.151743	5.162354	98	6	4.506367	1.423025	-3.188827
99	6	4.108077	0.126513	-2.465179	117	6	9.280829	-4.929280	-10.021026
100	15	9.678642	0.980513	-0.319488	118	6	10.062867	-6.209670	-9.709151
101	6	9.787543	1.059142	1.557038	119	7	9.349328	-7.132267	-8.807906
102	6	11.124842	0.626093	2.181336	120	6	8.368789	-7.972653	-9.518615
103	6	11.168503	1.912683	-0.986236	121	6	7.225360	-8.470754	-8.635039
104	6	11.218404	3.400126	-0.601989	122	7	6.181773	-7.452223	-8.492812
105	6	10.094595	-0.798628	-0.755950	123	6	5.579721	-7.285022	-7.387908
106	6	9.064649	-1.828620	-0.266952	124	6	4.409060	-6.394831	-7.230285
107	6	7.750071	0.528868	-2.803390	125	6	3.642597	-6.478609	-6.063781
108	6	7.847489	-0.177781	-3.791045	126	6	2.466414	-5.747127	-5.926467
109	6	7.930442	-1.050201	-4.934141	127	6	2.025672	-4.898055	-6.952834
110	6	9.174189	-1.389807	-5.498656	128	6	2.829351	-4.775856	-8.102706
111	6	9.255022	-2.243733	-6.589568	129	6	3.995799	-5.517638	-8.242263
112	6	8.094885	-2.788252	-7.158187	130	6	0.763628	-4.208840	-6.843749
113	6	6.856845	-2.449241	-6.606841	131	6	-0.341418	-3.699556	-6.756866
114	6	6.772687	-1.592095	-5.511886	132	78	-2.195200	-2.843735	-6.616149
115	6	8.157956	-3.698362	-8.321517	133	15	-1.229181	-0.805444	-7.506316
116	7	9.250854	-4.048467	-8.859115	134	6	-0.445640	-1.109744	-9.185897
135	6	-1.437061	-1.600885	-10.253036	141	6	-2.173108	-6.480423	-6.242248
136	6	0.200807	-0.148923	-6.483947	142	6	-2.701250	-7.803089	-5.660705
137	6	-0.197558	0.256805	-5.054656	143	6	-4.895147	-5.267309	-6.422088
138	6	-2.405370	0.639829	-7.748794	144	6	-5.022101	-5.344016	-7.951354
139	6	-1.801330	1.912387	-8.365295	145	6	-3.369409	-4.939172	-3.949157
140	15	-3.149209	-4.932808	-5.814666	146	6	-2.059077	-4.806701	-3.158767

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147	6	-4.046631	-1.990065	-6.476165	186	6	-6.132834	-3.253814	6.000291
148	6	-5.145604	-1.470085	-6.395125	187	6	-7.000885	-4.182746	6.603791
149	6	-6.432199	-0.822954	-6.341735	188	6	-6.636469	-4.850040	7.766359
150	6	-7.265848	-0.801248	-7.476409	189	6	-5.392131	-4.612009	8.366593
151	6	-8.504256	-0.176272	-7.442168	190	6	-4.519015	-3.705172	7.760590
152	6	-8.957554	0.448511	-6.271651	191	6	-4.880974	-3.037512	6.593400
153	6	-8.135380	0.429765	-5.141967	192	6	-4.991598	-5.274223	9.625947
154	6	-6.890426	-0.196859	-5.174087	193	7	-5.745743	-6.073346	10.259263
155	6	-10.279413	1.110900	-6.214187	194	6	-5.334606	-6.636377	11.543855
156	7	-11.105508	1.064522	-7.175292	195	6	-5.376372	-8.166452	11.510697
157	6	-12.406829	1.722944	-7.096206	196	6	-4.547531	-10.043841	10.164109
158	6	-12.504722	2.840055	-8.141433	197	6	-5.126161	-9.882027	8.753678
159	6	-12.676308	4.915798	-6.846908	198	7	-4.067539	-9.605727	7.787248
160	6	-11.871641	5.715304	-5.818297	199	6	-4.139431	-8.633819	6.975755
161	7	-11.496375	4.856069	-4.697799	200	6	-3.091887	-8.364173	5.964799
162	6	-10.285238	4.718841	-4.348891	201	6	-1.965690	-9.191557	5.845948
163	6	-9.869415	3.859911	-3.219359	202	6	-0.981043	-8.914923	4.909348
164	6	-10.803690	3.174968	-2.430495	203	6	-1.091524	-7.810203	4.041306
165	6	-10.386004	2.358000	-1.388152	204	6	-2.229882	-7.000413	4.150269
166	6	9.019716	2.205713	-1.089034	205	6	-3.213324	-7.273598	5.100434
167	6	-8.090355	2.902650	-1.874511	206	6	-0.057362	-7.539367	3.075364
168	6	-8.511198	3.716734	-2.923827	207	6	0.841441	-7.372887	2.269731
169	6	-8.582256	1.355191	-0.011245	208	78	2.345330	-7.126095	0.908112
170	6	-8.187734	0.629638	0.884751	209	15	1.983082	-4.722472	0.893434
171	78	-7.510464	-0.585155	2.387299	210	6	1.136291	-4.020452	2.415359
172	15	-6.061985	-1.652313	0.750550	211	6	1.963623	-4.154480	3.703950
173	6	-5.117094	-0.386146	-0.279769	212	6	0.876786	-4.121254	-0.502807
174	6	-4.016702	-0.891066	-1.228622	213	6	1.382678	-4.454456	-1.917144
175	6	-4.787485	-2.882070	1.358430	214	6	3.584562	-3.747209	0.744187
176	6	-3.705778	-2.253537	2.253297	215	6	3.449525	-2.214190	0.739350
177	15	-9.112201	0.382607	3.950547	216	15	2.841823	-9.499077	0.912553
178	6	-9.826768	2.062213	3.504853	217	6	2.557176	-10.326273	-0.745788
179	6	-8.780497	3.180004	3.370835	218	6	1.099107	-10.254217	-1.229018
180	6	-10.644893	-0.706120	4.149766	219	6	1.913380	-10.538864	2.174759
181	6	-10.854266	-1.735997	3.028169	220	6	2.170226	-12.056165	2.135759
182	6	-8.444265	0.617063	5.695191	221	6	4.660581	-9.830362	1.250489
183	6	-9.464364	1.035848	6.767442	222	6	5.158975	-9.258687	2.588136
184	6	-6.876894	-1.815679	3.890107	223	6	3.821051	-6.925842	-0.499435
185	6	-6.528973	-2.508334	4.832078	224	6	4.678687	-6.840555	-1.361419

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225	6	5.712758	-6.733506	-2.360686	264	1	-1.273154	8.757819	-8.219192
226	6	6.447187	-7.865703	-2.764754	265	1	-0.102368	7.528260	-7.813949
227	6	7.451048	-7.765461	-3.718108	266	1	0.238546	9.214053	-7.470153
228	6	7.759357	-6.528490	-4.301498	267	1	1.562477	6.888730	-6.170837
229	6	7.029887	-5.403540	-3.909743	268	1	1.777096	6.351486	-4.527236
230	6	6.020320	-5.502700	-2.954711	269	1	1.101742	4.450129	-5.996306
231	6	8.846606	-6.388603	-5.294906	270	1	-0.383647	5.274993	-6.426604
232	7	9.571009	-7.365039	-5.658950	271	1	-0.136283	4.743271	-4.783385
233	6	10.634148	-7.195017	-6.647338	272	1	-5.418712	7.428457	-5.964828
234	6	10.268493	-7.910992	-7.954391	273	1	-7.278051	6.631690	-7.383346
235	6	-3.415137	8.760961	0.446687	274	1	-6.291249	2.602371	-6.403228
236	6	-4.819986	5.197078	0.467957	275	1	-4.461940	3.391783	-4.969192
237	6	-7.217144	-2.586998	-0.409911	276	1	-8.106011	2.990128	-7.921798
238	6	-6.605610	-3.579166	-1.409850	277	1	-9.897517	3.233904	-9.348907
239	1	7.632525	13.543013	4.207765	278	1	-9.692663	4.702274	-10.293014
240	1	5.930776	13.340196	3.878892	279	1	-11.158233	5.875786	-8.590148
241	1	7.577233	12.809049	2.007981	280	1	-11.950126	4.800860	-9.712798
242	1	7.901266	11.300599	2.846787	281	1	6.032337	13.520387	6.327059
243	1	6.498034	9.598607	2.182145	282	1	6.089401	11.991640	7.167726
244	1	3.602755	11.690580	0.967988	283	1	3.808969	12.868178	6.648072
245	1	1.627466	10.773924	-0.197707	284	1	4.083293	12.488039	4.954559
246	1	3.246437	6.843654	0.172151	285	1	3.417432	10.317455	4.410829
247	1	5.213838	7.760081	1.317536	286	1	3.591408	8.839460	7.873139
248	1	-1.427429	7.999625	0.761247	287	1	2.919429	6.529214	8.432008
249	1	-2.635446	7.123241	1.664079	288	1	1.834794	5.872604	4.356278
250	1	-0.455638	5.321699	0.432130	289	1	2.543348	8.153875	3.799759
251	1	-1.922308	4.648897	1.101731	290	1	4.309306	2.611652	10.563050
252	1	-0.686302	2.911144	-0.191405	291	1	3.930728	3.657181	9.217179
253	1	-0.735870	3.914867	-1.629912	292	1	2.669796	4.365866	11.230363
254	1	-2.214396	3.237167	-0.981338	293	1	1.885735	2.805252	11.336350
255	1	-4.124260	4.898285	-1.570190	294	1	1.567948	3.849454	9.967347
256	1	-4.702641	6.486355	-1.269652	295	1	4.706281	2.314612	7.209275
257	1	1.790002	8.976608	-3.734237	296	1	5.479147	1.162427	8.273957
258	1	1.531437	9.463285	-5.392287	297	1	5.387753	0.276760	5.948228
259	1	1.088292	11.359095	-3.839034	298	1	3.640224	0.436934	5.930894
260	1	-0.116367	10.412553	-2.988494	299	1	4.401676	-0.733440	6.985673
261	1	-0.401436	10.924543	-4.643798	300	1	3.152763	-0.943930	9.275361
262	1	-2.021542	7.587052	-6.155807	301	1	4.384445	-0.104061	10.193742
263	1	-1.686778	9.253391	-5.828826	302	1	2.634774	-1.055781	11.686036

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303	1	1.394440	-0.129993	10.863633	322	1	-0.775621	-5.800892	7.080140
304	1	2.625604	0.686178	11.815339	323	1	-0.154942	-3.495453	6.525468
305	1	-0.341372	0.185057	4.424775	324	1	-1.283741	-7.524605	8.632371
306	1	-1.473384	1.342580	3.770310	325	1	-0.906578	-9.130743	11.517655
307	1	0.736057	1.567252	2.655076	326	1	-1.753440	-9.291578	9.984834
308	1	1.519531	1.857526	4.199616	327	1	-3.022311	-9.781822	12.244996
309	1	0.402869	3.033605	3.552232	328	1	-3.049698	-8.043983	12.404298
310	1	-0.832704	4.142451	5.411134	329	1	8.397566	12.183842	6.434251
311	1	-2.450940	3.504453	5.231568	330	1	8.753940	11.437034	4.897996
312	1	-2.369654	5.222898	7.039948	331	1	8.897278	9.952156	6.936555
313	1	-1.141497	4.303551	7.883836	332	1	7.139298	9.943171	6.967942
314	1	-2.772260	3.668330	7.738587	333	1	6.321090	8.117008	5.819428
315	1	-2.061384	-0.267333	6.435990	334	1	9.073075	7.762826	3.263215
316	1	-2.453376	0.836454	7.705354	335	1	9.138839	5.993784	1.539552
317	1	-4.465137	0.356504	6.326600	336	1	5.482815	4.396970	3.055299
318	1	-3.673394	0.920460	4.878480	337	1	5.416472	6.162106	4.757951
319	1	-4.067323	2.048509	6.162446	338	1	6.474489	3.749364	-4.009597
320	1	-0.150129	-2.495866	10.673799	339	1	4.809995	4.242820	-3.839187
321	1	-0.778452	-4.818104	11.227189					
340	1	6.346770	6.146031	-3.372558	360	1	12.089127	3.866182	-1.049697
341	1	7.134527	5.235636	-2.095107	361	1	11.283588	3.537303	0.470719
342	1	5.474748	5.751630	-1.908156	362	1	10.336869	3.922824	-0.948394
343	1	4.080457	2.359788	-0.165298	363	1	10.170614	-0.839277	-1.832992
344	1	4.764434	3.944786	-0.226638	364	1	11.074378	-1.021612	-0.349441
345	1	2.296417	4.070209	-0.507217	365	1	9.361090	-2.822483	-0.579650
346	1	2.350746	3.014453	-1.896224	366	1	8.088421	-1.624647	-0.684844
347	1	3.050394	4.620584	-1.980956	367	1	8.983442	-1.836204	0.814713
348	1	3.624985	1.937117	-3.552810	368	1	10.066703	-0.969954	-5.079558
349	1	5.128988	1.198328	-4.042358	369	1	10.204105	-2.497831	-7.014907
350	1	3.573475	-0.528444	-3.144256	370	1	5.956533	-2.857253	-7.025786
351	1	3.456891	0.322873	-1.620055	371	1	5.814906	-1.347107	-5.099135
352	1	4.981799	-0.400108	-2.108108	372	1	7.203536	-4.060934	-8.686111
353	1	9.544753	2.072416	1.836640	373	1	9.812426	-4.398514	-10.803390
354	1	8.981426	0.440560	1.929307	374	1	8.286324	-5.164426	-10.393767
355	1	11.066507	0.718241	3.259889	375	1	10.310979	-6.712079	-10.647292
356	1	11.947942	1.246826	1.846742	376	1	10.995283	-5.912345	-9.251937
357	1	11.367938	-0.405262	1.955949	377	1	8.870779	-8.826317	-9.982635
358	1	12.064793	1.402543	-0.652938	378	1	7.918254	-7.399566	-10.314651
359	1	11.122194	1.811243	-2.063003	379	1	6.760339	-9.313988	-9.137328

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380	1	7.594900	-8.821612	-7.675399	419	1	-6.271789	-0.202973	-4.299224
381	1	5.883100	-7.810944	-6.489013	420	1	-10.501600	1.641401	-5.296416
382	1	3.960930	-7.117766	-5.261733	421	1	-12.615755	2.113583	-6.103545
383	1	1.888960	-5.828170	-5.027053	422	1	-13.155190	0.975456	-7.331145
384	1	2.520966	-4.112055	-8.886841	423	1	-12.052082	2.474061	-9.051162
385	1	4.582560	-5.445475	-9.136046	424	1	-13.556039	3.031407	-8.363448
386	1	0.042085	-0.198469	-9.510314	425	1	-13.352282	4.278159	-6.297241
387	1	0.320662	-1.853862	-9.025024	426	1	-13.290448	5.596946	-7.438933
388	1	-0.917498	-1.769553	-11.188949	427	1	-11.010958	6.187586	-6.285415
389	1	-2.224008	-0.879288	-10.438918	428	1	-12.511448	6.495496	-5.422057
390	1	-1.898846	-2.532270	-9.952712	429	1	-9.482386	5.227193	-4.869277
391	1	0.930763	-0.945090	-6.452346	430	1	-11.845653	3.299410	-2.642150
392	1	0.644465	0.689001	-7.007552	431	1	-11.108214	1.829064	-0.799544
393	1	0.674342	0.604224	-4.512206	432	1	-7.045539	2.809335	-1.656676
394	1	-0.615612	-0.583881	-4.515777	433	1	-7.780780	4.237429	-3.511794
395	1	-0.928720	1.056924	-5.049026	434	1	-5.872469	0.158053	-0.826995
396	1	-3.219638	0.271476	-8.355193	435	1	-4.695117	0.313503	0.429521
397	1	-2.834525	0.855448	-6.780913	436	1	-3.572510	-0.045665	-1.742339
398	1	-2.570410	2.670575	-8.450667	437	1	-4.398137	-1.561479	-1.985427
399	1	-1.404723	1.736380	-9.358153	438	1	-3.224004	-1.402703	-0.697494
400	1	-1.007083	2.324365	-7.753707	439	1	-5.321827	-3.643411	1.909089
401	1	-2.143780	-6.529114	-7.322417	440	1	-4.330526	-3.358781	0.500657
402	1	-1.157103	-6.302192	-5.925994	441	1	-3.041099	-3.026878	2.620744
403	1	-2.062405	-8.617812	-5.981231	442	1	-4.150225	-1.762024	3.105439
404	1	-3.706406	-8.027615	-5.996795	443	1	-3.104664	-1.533698	1.709718
405	1	-2.703020	-7.798863	-4.577193	444	1	-10.356582	1.938201	2.571153
406	1	-5.239592	-6.187813	-5.966277	445	1	-10.555194	2.319638	4.264268
407	1	-5.506620	-4.461878	-6.043899	446	1	-9.265398	4.101631	3.071000
408	1	-6.054447	-5.526031	-8.225799	447	1	-8.043312	2.931458	2.621949
409	1	-4.424927	-6.147203	-8.368606	448	1	-8.270976	3.369278	4.309342
410	1	-4.711835	-4.414829	-8.409774	449	1	-10.564749	-1.212604	5.102128
411	1	-4.024353	-4.106307	-3.729949	450	1	-11.504318	-0.049449	4.214311
412	1	-3.894945	-5.843458	-3.666350	451	1	-11.688779	-2.382778	3.275498
413	1	-2.267710	-4.786927	-2.095077	452	1	-9.975783	-2.350981	2.896150
414	1	-1.536241	-3.896403	-3.421508	453	1	-11.069480	-1.253562	2.082887
415	1	-1.390644	-5.638269	-3.347014	454	1	-7.964033	-0.310789	5.963715
416	1	-6.931189	-1.280874	-8.374145	455	1	-7.660610	1.360340	5.623979
417	1	-9.134978	-0.166649	-8.307143	456	1	-8.958444	1.146036	7.719636
418	1	-8.460761	0.907896	-4.238113	457	1	-10.243671	0.296143	6.899751

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458	1	-9.935410	1.984285	6.538117	497	1	2.176881	-10.136472	3.142671
459	1	-7.960645	-4.359801	6.161149	498	1	0.863176	-10.327568	2.040269
460	1	-7.307621	-5.542703	8.231324	499	1	1.590473	-12.540071	2.913438
461	1	-3.561131	-3.510234	8.202363	500	1	3.211290	-12.302892	2.307984
462	1	-4.202101	-2.341340	6.144608	501	1	1.873294	-12.496536	1.191575
463	1	-4.005806	-5.015685	9.992509	502	1	4.827978	-10.899712	1.206788
464	1	-6.053389	-6.293211	12.279633	503	1	5.205820	-9.377477	0.435288
465	1	-4.350310	-6.291353	11.849488	504	1	6.211849	-9.482327	2.714601
466	1	-6.284055	-8.457032	11.002854	505	1	4.629910	-9.680032	3.435014
467	1	-5.445584	-8.543436	12.533067	506	1	5.038224	-8.183890	2.617159
468	1	-5.257168	-10.605386	10.773072	507	1	6.229315	-8.814664	-2.316344
469	1	-3.656306	-10.648159	10.081712	508	1	8.005787	-8.629455	-4.021789
470	1	-5.568323	-10.829952	8.466020	509	1	7.244158	-4.449132	-4.349715
471	1	-5.908882	-9.127546	8.744355	510	1	5.467290	-4.629596	-2.673629
472	1	-4.980577	-7.950949	6.976747	511	1	8.993785	-5.393043	-5.692916
473	1	-1.884079	-10.042784	6.489915	512	1	11.522733	-7.662311	-6.239745
474	1	-0.115101	-9.541778	4.842744	513	1	10.857117	-6.149407	-6.843871
475	1	-2.340027	-6.159303	3.495652	514	1	11.182877	-8.148015	-8.500472
476	1	-4.072994	-6.634277	5.166230	515	1	9.812063	-8.853074	-7.690673
477	1	0.901298	-2.980339	2.220353	516	1	-3.367581	9.481070	1.254517
478	1	0.204822	-4.556073	2.523576	517	1	-4.440785	8.421476	0.368241
479	1	1.401273	-3.770740	4.545716	518	1	-3.163798	9.278282	-0.471358
480	1	2.890423	-3.594994	3.649796	519	1	-5.828957	4.871142	0.248795
481	1	2.197823	-5.190565	3.905595	520	1	-4.886536	5.984720	1.208970
482	1	-0.089962	-4.580108	-0.336516	521	1	-4.299152	4.359756	0.918092
483	1	0.748665	-3.051156	-0.389998	522	1	-7.905174	-3.107680	0.240815
484	1	0.691619	-4.067529	-2.657226	523	1	-7.776293	-1.816968	-0.920791
485	1	1.475048	-5.521441	-2.062133	524	1	-7.399445	-4.054438	-1.974151
486	1	2.353637	-4.016354	-2.112380	525	1	-6.048215	-4.361801	-0.909545
487	1	4.211746	-4.069105	1.564393	526	1	-5.950645	-3.096297	-2.121429
488	1	4.068985	-4.090865	-0.156554					
489	1	4.428678	-1.763195	0.630280					
490	1	3.018827	-1.837562	1.659074					
491	1	2.840361	-1.863365	-0.085418					
492	1	3.205350	-9.820928	-1.447951					
493	1	2.887324	-11.355643	-0.678390					
494	1	1.003667	-10.742820	-2.191634					
495	1	0.778619	-9.226220	-1.338653					
496	1	0.419392	-10.743785	-0.541411					