Supporting Information for

Solution Superstructures: Truncated Cubeoctahedron Structures of Pyrogallol[4]arene Nanoassemblies

In the section, we report two sets of experiments:

a) SANS data analysis of holmium-seamed *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly in d6-DMSO
b) SANS data analysis of holmium-seamed *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly in d6-DMSO

For the SANS experiments, solid precipitates of PgC_1Ho or PgC_3Ho were dissolved in d6-DMSO at a mass fraction of 3%. Samples were held in cells of path length 2 mm and held at a constant temperature of 25 °C. SANS measurements were carried out at the NG7 SANS instrument at the NIST Center for Neutron Research using 6 Å neutrons and two different sample to detector distances to give an overall q-range of 0.01659 $Å^{-1}$ to 0.514$Å^{-1}$. Data was reduced and placed on an absolute scale using standard software supplied by the NCNR. Here, we report the full sets of fitting parameters of the cylinder, core-shell sphere, core-shell cylinder, ellipsoid, bimodal Schulz sphere and Schulz sphere fits for PgC_1Ho and PgC_3Ho. All modeling used resolution-smeared model functions that incorporate the finite resolution of the SANS instrument into the data analysis. Fitted values are one standard deviation of each fitted parameter. Comparing the quality of each different model fit with respect to the criteria enlisted below leads to the conclusion that the Schulz Sphere model best represents the structure of PgC_1Ho and PgC_3Ho in solution. It is the only model that fits well with respect to all of the criteria (see notes below each graph).

Selection Criterion:
The model fits shows some deviation between model and data at high q values but that is true for all types of model fits (cylinder, core shell sphere, core shell cylinder, bimodal Schulz sphere), and the data has large error bars at high q. The Schulz sphere fit is described as the best fit due to low error bars on its parameters (radius, volume fraction, polydispersity, SLDs, incoherent scattering) and its lowest overall chi-squared goodness of fit. All of the fits presented have converged to the optimal, non-zero chi-squared. The chi-squared value for the Schulz spheres is in fact the lowest, and chi-squared is only one of the critera for selecting the best structural model. Some of the model fits have uncertainties in the fitted parameters that are larger than the parameter values themselves and these models were immediately rejected. Other models gave lengths for parameters that were unphysical or impossible and these were also rejected. In these rejected cases there is clearly not enough information content in the data to support the parameters for the (incorrect) structural model. We provided a note below each fit in the SI explaining why this fit is incorrect or unphysical and thus rejected. The model fits in the SI are presented to show evidence that the sphere structure is indeed the most appropriate structural model. The wide variety of structural models was tested by fitting each to the data, and interpreting the results, indicating why certain structures were rejected. In this way, the *data* selected the best structure.

Synthesis of PgC_1Ho
$C_{32}H_{30}O_{12}$: Molar mass: 606 gms: 72 mgs or 11.31 millimols of PgC_1 is combined with 0.84 gms or 181.4 millimol of Ho(NO3)3.5H2O (molar mass:441 gms) in acetonitrile: water (20:1) mixture. i.e. 1 equivalent of PgC1 is added to 15.4 equivalents of homium nitrate. To this 5 drops of pyridine (88.65 mmoles) were added and more water was added to dissolve the material. So the combining ratios is: 1:16:8::PgC_1:$Ho(NO_3)_3$:Pyridine
Synthesis of PgC_3Ho
$C_{40}H_{48}O_{12}$: Molar mass: 720 gms: 86 mgs or 11.38 millimoles mols of PgC_3 is combined with 0.84 gms or 181.4 millimoles of Ho(NO3)3.6H2O (molar mass:441 gms) in acetonitrile: water (20:1) mixture. i.e. 1 equivalent of PgC3 is added to 15.4 equivalents of homium nitrate. To this 5 drops of pyridine (88.65 mmoles) were added and few more drops of water was added to dissolve the material. So the combining ratios is:

1:16:8::PgC_1:$Ho(NO_3)_3$:Pyridine

TABLE OF FIGURES

SCATTERING LENGTH DENISTIES (SLDs)			
PgC_1Ho		λ Å	d = 1.65
$Ho_{48}(C_{32}H_{30}O_{12})_{12}$: $Ho_{48}C_{384}H_{360}O_{144}$	dodecamer	6	$1.59x10^{-6}$
$Ho_{40}(C_{32}H_{30}O_{12})_{10}$: $Ho_{40}C_{320}H_{300}O_{120}$	decamer	6	$1.59x10^{-6}$
$Ho_{32}(C_{32}H_{30}O_{12})_{8}$: $Ho_{32}C_{256}H_{240}O_{96}$	octamer	6	$1.59x10^{-6}$
$Ho_{24}(C_{32}H_{30}O_{12})_{6}$: $Ho_{24}C_{192}H_{180}O_{72}$	hexamer	6	$1.59x10^{-6}$
PgC_3Ho			d = 1.65
$Ho_{48}(C_{40}H_{48}O_{12})_{12}$: $Ho_{48}C_{480}H_{576}O_{144}$	dodecamer	6	$1.35x10^{-6}$
$Ho_{40}(C_{40}H_{48}O_{12})_{10}$: $Ho_{40}C_{400}H_{480}O_{120}$	decamer	6	$1.35x10^{-6}$
$Ho_{32}(C_{40}H_{48}O_{12})_{8}$: $Ho_{32}C_{320}H_{384}O_{96}$	octamer	6	$1.35x10^{-6}$
$Ho_{24}(C_{40}H_{48}O_{12})_{6}$: $Ho_{144}C_{240}H_{288}O_{72}$	hexamer	6	$1.35x10^{-6}$
d6-DMSO		6	$5.28x10^{-6}$
$(PgC_1)_{12}Ho_{48}(ligand)_{48}$			
$Ho_{48}(C_{32}H_{30}O_{12})_{12}(NO_3)_{48}$:$Ho_{48}C_{384}H_{360}O_{144}N_{48}O_{144}$	dodecamer	6	$2.03x10^{-6}$
$Ho_{48}(C_{32}H_{30}O_{12})_{12}(C_5H_5N)_{48}$:$Ho_{48}C_{384}H_{360}O_{144}N_{48}C_{240}H_{240}$	dodecamer	6	$1.87x10^{-6}$
$Ho_{48}(C_{32}H_{30}O_{12})_{12}(C_2H_6SO)_{48}$:$Ho_{48}C_{384}H_{360}O_{144}S_{48}O_{48}C_{96}H_{288}$	dodecamer	6	$1.26x10^{-6}$
$Ho_{48}(C_{32}H_{30}O_{12})_{12}(C_2H_6SO)_{48}$:$Ho_{48}C_{384}H_{360}O_{144}S_{48}O_{48}C_{96}D_{288}$	dodecamer	6	$2.79x10^{-6}$
$(PgC_3)_{12}Ho_{48}(ligand)_{48}$			
$Ho_{48}(C_{40}H_{48}O_{12})_{12}(NO_3)_{48}$: $Ho_{48}C_{480}H_{576}O_{144}N_{48}O_{144}$	dodecamer	6	$1.8x10^{-6}$
$Ho_{48}(C_{40}H_{48}O_{12})_{12}(C_5H_5N)_{48}$:$Ho_{48}C_{480}H_{576}O_{144}N_{48}C_{240}H_{240}$	dodecamer	6	$1.66x10^{-6}$
$Ho_{48}(C_{40}H_{48}O_{12})_{12}(C_2H_6SO)_{48}$:$Ho_{48}C_{480}H_{576}O_{144}S_{48}O_{48}C_{96}H_{288}$	dodecamer	6	$1.09x10^{-6}$
$Ho_{48}(C_{40}H_{48}O_{12})_{12}(C_2H_6SO)_{48}$:$Ho_{48}C_{480}H_{576}O_{144}S_{48}O_{48}C_{96}D_{288}$	dodecamer	6	$2.52x10^{-6}$
$PgC_nHo_{2n}Ligand_{0\text{-}2n}$: n=1			
$Ho_{24}(C_{32}H_{30}O_{12})_{12}$: $Ho_{24}C_{384}H_{360}O_{144}$	dodecamer	6	$1.98x10^{-6}$
$Ho_{24}(C_{32}H_{30}O_{12})_{12}(NO_3)_{24}$:$Ho_{24}C_{384}H_{360}O_{144}N_{24}O_{72}$	dodecamer	6	$2.25x10^{-6}$
$Ho_{24}(C_{32}H_{30}O_{12})_{12}(C_5H_5N)_{24}$:$Ho_{24}C_{384}H_{360}O_{144}N_{24}C_{120}H_{120}$	dodecamer	6	$2.12x10^{-6}$
$Ho_{24}(C_{32}H_{30}O_{12})_{12}(C_2H_6SO)_{24}$:$Ho_{24}C_{384}H_{360}O_{144}S_{24}O_{24}C_{48}H_{144}$	dodecamer	6	$1.68x10^{-6}$
$Ho_{24}(C_{32}H_{30}O_{12})_{12}(C_2H_6SO)_{24}$:$Ho_{24}C_{384}H_{360}O_{144}S_{24}O_{24}C_{48}D_{144}$	dodecamer	6	$2.79x10^{-6}$
$PgC_nHo_{2n}Ligand_{0\text{-}2n}$: n=3			
$Ho_{24}(C_{40}H_{48}O_{12})_{12}$: $Ho_{24}C_{480}H_{576}O_{144}$	dodecamer	6	$1.63x10^{-6}$
$Ho_{24}(C_{40}H_{48}O_{12})_{12}(NO_3)_{24}$:$Ho_{24}C_{480}H_{576}O_{144}N_{24}O_{72}$	dodecamer	6	$1.91x10^{-6}$
$Ho_{24}(C_{40}H_{48}O_{12})_{12}(C_5H_5N)_{24}$:$Ho_{24}C_{480}H_{576}O_{144}N_{24}C_{120}H_{120}$	dodecamer	6	$1.81x10^{-6}$
$Ho_{24}(C_{40}H_{48}O_{12})_{12}(C_2H_6SO)_{24}$:$Ho_{24}C_{480}H_{576}O_{144}S_{24}O_{24}C_{48}H_{144}$	dodecamer	6	$1.41x10^{-6}$
$Ho_{24}(C_{40}H_{48}O_{12})_{12}(C_2H_6SO)_{24}$:$Ho_{24}C_{480}H_{576}O_{144}S_{24}O_{24}C_{48}D_{144}$	dodecamer	6	$2.41x10^{-6}$

Fit to Smeared Core Shell Sphere
Data file: PgC_3Ho

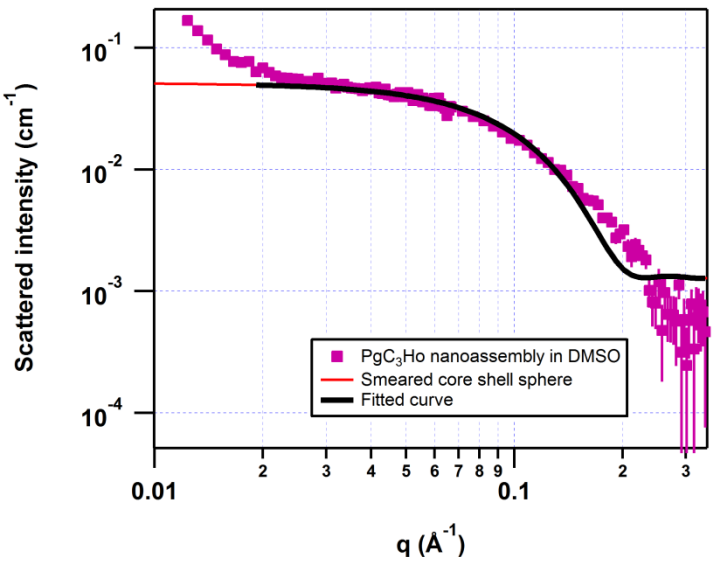

Figure 1 Core shell sphere fit (SANS) of PgC_3Ho nanoassembly in *d6*-DMSO

scale	0.000449673	±	0.000497995
core radius (Å)	-13.2562	±	12.9019
shell thickness (A)	36.2241	±	14.7905
Core SLD (Å-2)	5.28e-06	±	(held fixed)
Shell SLD (Å-2)	1.35e-06	±	(held fixed)
Solvent SLD (Å-2)	5.28e-06	±	(held fixed)
bkg (cm-1)	0.00127343	±	8.09698e-05

chisq = 764.859 Npnts = 115
Sqrt(χ^2/N) = 2.57894
Fitted range = [13,127] = 0.01915 < Q < 0.3396

NOTE: SANS data fitted as a core shell sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly does not fit well. The high chi-sqrt value of 2.6, high error bars on core and shell

parameters indicate poor statistics. Also the negative value of core radius curve gives an unphysical representation of a nanoassembly in solution.

Fit to Smeared Core Shell Cylinder
Data file: PgC_3Ho

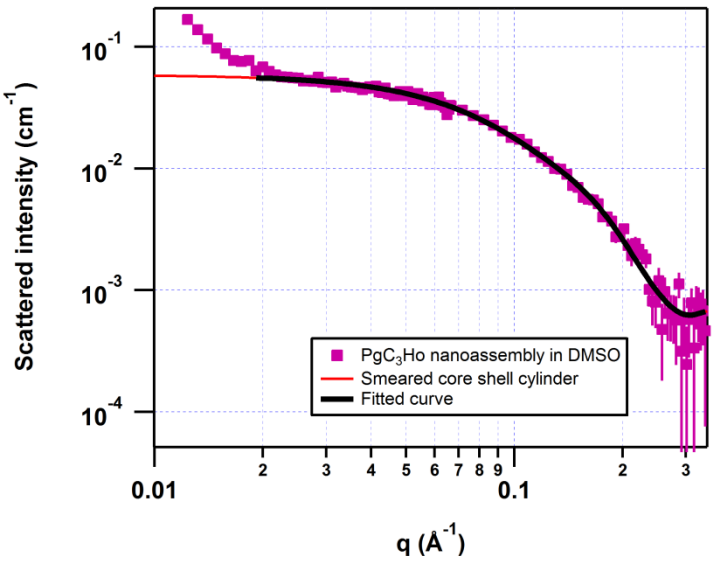

Figure 2 Core shell cylinder fit (SANS) of PgC_3Ho nanoassembly in *d6*-DMSO

scale	0.00108203	±	0.000373388
core radius (Å)	0.73473	±	17.528
shell radius (Å)	12.379	±	18.3543
length (Å)	40.1633	±	34.8413
SLD core (Å^{-2})	5.28e-06	±	(held fixed)
SLD shell (Å^{-2})	1.35e-06	±	(held fixed)
SLD solvent (Å^{-2})	5.28e-06	±	(held fixed)
incoh. bkg (cm^{-1})	0.000566621	±	8.57532e-05

chisq = 164.373 Npnts = 115
Sqrt(χ^2/N) = 1.19555
Fitted range = [13,127] = 0.01915 < Q < 0.3396

NOTE: SANS data fitted as a core shell cylinder for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly does not fit well. Albeit the chi-sqrt value is low, the high error bars on length, core and shell parameters indicate poor statistics.

Fit to Smeared Cylinder Form
Data file: PgC_3Ho

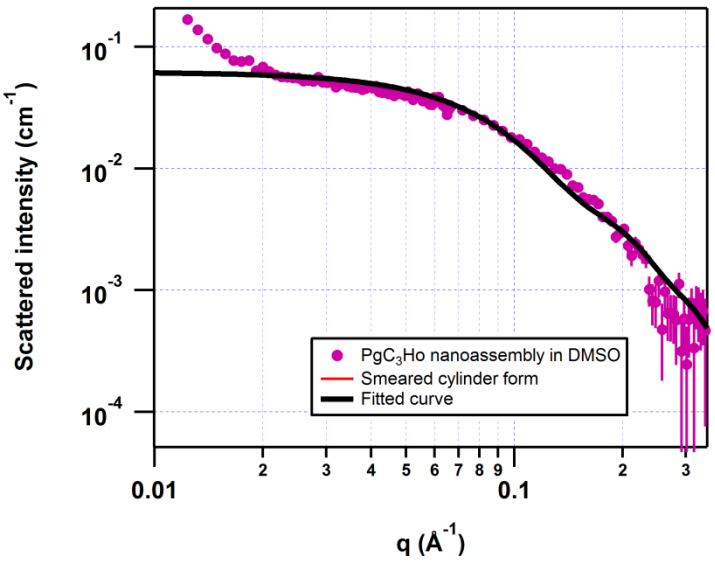

Figure 3 Cylinder fit (SANS) of PgC_3Ho nanoassembly in *d6*-DMSO

scale	0.00147008	±	8.73962e-05
radius (Å)	27.894 ±	0.182962	
length (Å)	11.1999±	0.711672	
SLD cylinder (Å^{-2})	1.35e-06	±	(held fixed)
SLD solvent (Å^{-2})	5.28e-06	±	(held fixed)
incoh. bkg (cm^{-1})	6.87101e-05	±	7.88896e-05

chisq = 6872.95 Npnts = 168
Sqrt(χ^2/N) = 6.39613
Fitted range = [0,167] = 0.008085 < Q < 0.514

NOTE: SANS data fitted as a uniform ellipsoid for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly does not fit well. The high chi-sqrt value of 6.4 indicates poor statistics. Also the Ra of 28 Å or diameter of 56 Å gives an unphysical representation of nanoassembly in solution.

Fit to Smeared Ellipsoid Form
Data file: PgC_3Ho

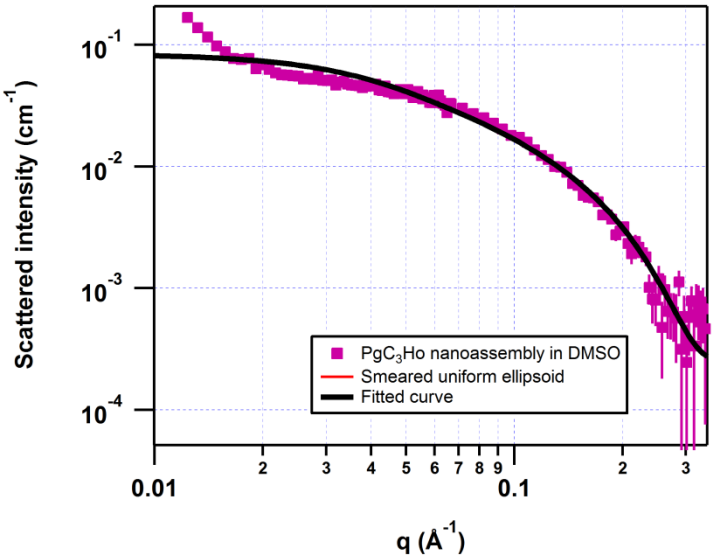

Figure 4 Uniform Ellipsoid fit (SANS) of PgC_3Ho nanoassembly in *d6*-DMSO

scale	0.00127104	±	2.36716e-05
R a (rotation axis) (Å)	71.7721	±	1.04621
R b (Å)	11.9864	±	0.127295
SLD ellipsoid (Å^{-2})	1.35e-06	±	(held fixed)
SLD solvent (Å^{-2})	5.28e-06	±	(held fixed)
incoh. bkg (cm^{-1})	0.000181762	±	4.69562e-05

chisq = 5959.99 Npnts = 168
Sqrt(χ^2/N) = 5.95619
Fitted range = [0,167] = 0.008085 < Q < 0.514

NOTE: SANS data fitted as a uniform ellipsoid for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly does not fit well. The high chi-sqrt value of 6 indicates poor statistics. Also the Ra of 71 Å or diameter of 142 Å gives an unphysical representation of nanoassembly in solution.

Fit to Smeared Triaxial Ellipsoid
Data file: PgC_3Ho

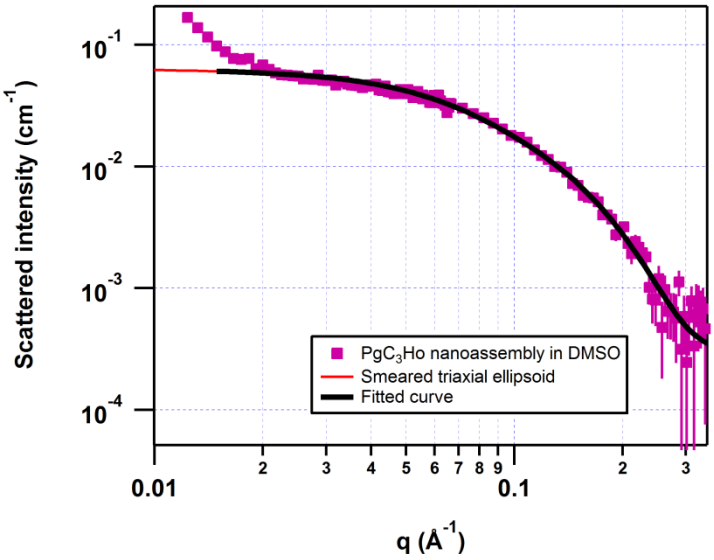

Figure 5 Triaxial ellipsoid fit (SANS) of PgC_3Ho nanoassembly in *d6*-DMSO

Scale Factor	0.00123971	±	3.57061e-05
Semi-axis A [smallest](Å)	10.5056	±	0.699147
Semi-axis B (Å)	15.8698	±	0.783635
Semi-axis C [largest](Å)	47.2744	±	0.843326
SLD ellipsoid (Å^-2)	1.35e-06	±	(held fixed)
SLD solvent (Å^-2)	5.28e-06	±	(held fixed)
Incoherent Bgd (cm-1)	0.00021946	±	5.2908e-05

chisq = 490.427 Npnts = 160
Sqrt(χ^2/N) = 1.75076
Fitted range = [8,167] = 0.01489 < Q < 0.514

NOTE: SANS data fitted as triaxial ellipsoid for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly does not fit well. Albiet the chi-sqrt value of 1.75 is low, semi-axis C of radius 47 Å or diameter 94Å gives an unphysical representation of nanoassembly in solution.

Fit to Smeared Bimodal Schulz Spheres
Data file: PgC_3Ho

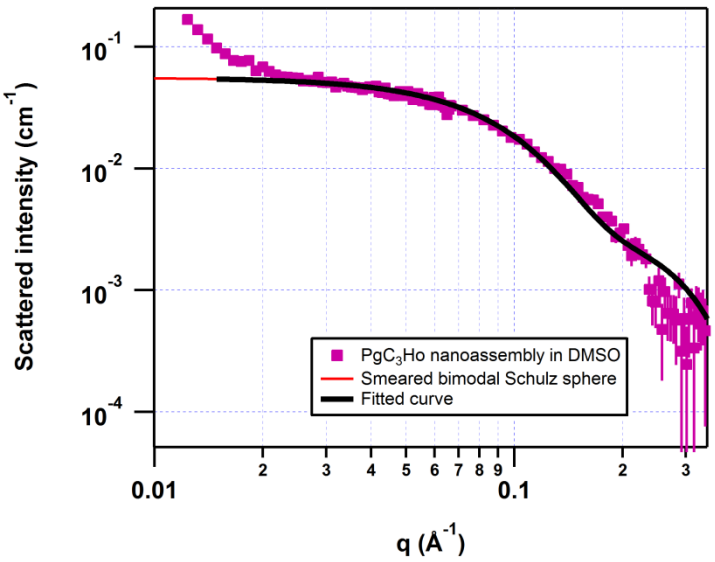

Figure 6 Bimodal Schulz sphere fit (SANS) of PgC_3Ho nanoassembly in *d6*-DMSO

volume fraction(1)	0.00160739	±	9.9919e-05
Radius (1) (Å)	6.37586	±	0.0145592
polydispersity(1)	0.182558	±	0.000297204
SLD(1) ($Å^{-2}$)	1.35e-06	±	(held fixed)
volume fraction(2)	0.000696281	±	1.94178e-05
Radius (2)	19.5493	±	0.593127
polydispersity(2)	0.2	±	0.017936
SLD(2)	1.35e-06	±	(held fixed)
SLD (solvent)	5.28e-06	±	(held fixed)
background (cm^{-1} sr^{-1})	-0.000394893	±	7.12749e-05

chisq = 841.908 Npnts = 160
Sqrt(χ^2/N) = 2.29389
Fitted range = [8,167] = 0.01489 < Q < 0.514

NOTE: SANS data fitted as a bimodal Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly does not fit well. The high chi-sqrt value of 2.3 indicates poor statistics. Also the radius value of 6.3 is even smaller than that of a dimer (7 Å) and hence gives an unphysical representation of nanoassembly in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

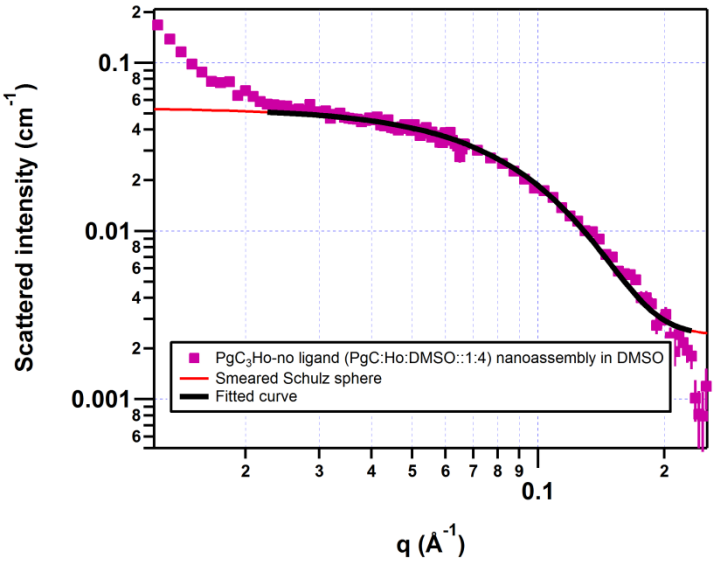

Figure 7. Schulz sphere fit (SANS) of PgC_3Ho-no ligand (PgC_3:Ho::1:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000780501	±	3.1307e-05
mean radius (Å)	18.2128	±	0.893493
polydisp (sig/avg)	0.22	±	0.0267319
SLD sphere ($Å^{-2}$)	1.35e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221596	±	0.000173089

chisq = 178.74
Npnts = 89 Sqrt(X^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly with pyrogallol[4]arene:metal ratio of 1:4 gives good statistics. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The presence or absence of ligand or the change in macrocylce to metal ratio does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

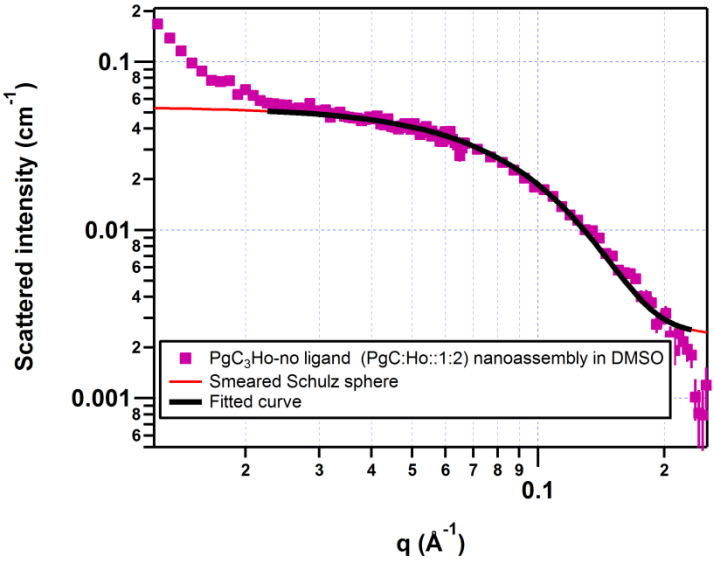

Figure 8. Schulz sphere fit (SANS) of PgC_3Ho-no ligand (PgC_3:Ho::1:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000904922	±	3.65842e-05
mean radius (Å)	18.2121	±	0.902841
polydisp (sig/avg)	0.22	±	0.0270515
SLD sphere (Å^{-2})	1.63e-06	±	0
SLD solvent (Å^{-2})	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221559	±	0.000174078

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3Ho) nanoassembly with pyrogallol[4]arene:metal ratio of 1:2gives good statistics. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The presence or absence of ligand or the change in macrocylce to metal ratio does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

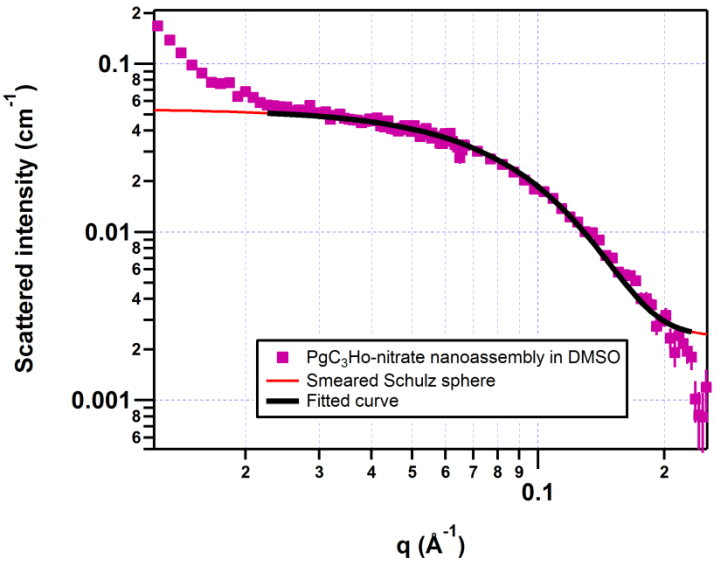

Figure 9.Schulz sphere fit (SANS) of PgC_3HoNO3 (PgC_3:Ho:NO_3::1:4:4) nanoassembly in d6-DMSO

Volume Fraction (scale)	0.000995452	±	4.02916e-05
mean radius (Å)	18.2124	±	0.905065
polydisp (sig/avg)	0.22	±	0.0271483
SLD sphere ($Å^{-2}$)	1.8e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221576	±	0.000174191

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3HoNO_3) nanoassembly gives good statistics. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DSMO (hydrogenated/deuterated) does not affect the overall radius of observed spherical species in solution (see subsequent fits).

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

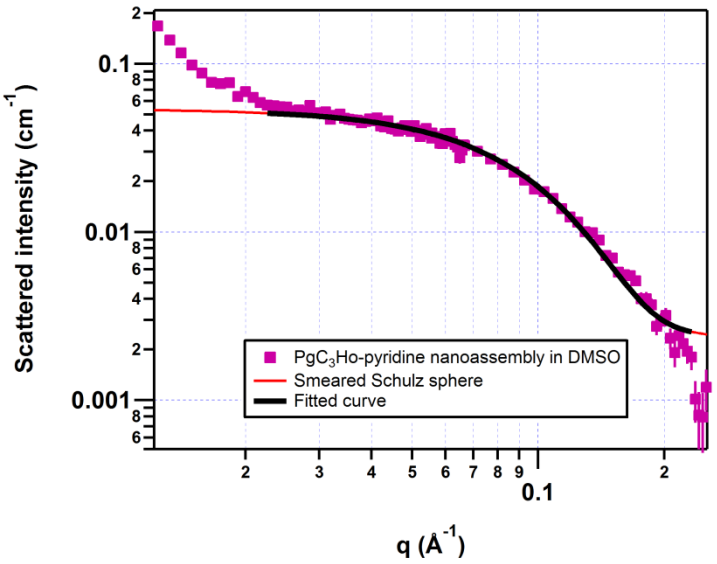

Figure 10. Schulz sphere fit (SANS) of PgC_3HoPy (PgC_3:Ho:Py::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000920119	±	3.72675e-05
mean radius (Å)	18.211	±	0.904338
polydisp (sig/avg)	0.22	±	0.027093
SLD sphere ($Å^{-2}$)	1.66e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221489	±	0.000174342

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3HoPy) nanoassembly gives good statistics. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DSMO (hydrogenated/deuterated) does not affect the overall radius of observed spherical species in solution (see subsequent fits).

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

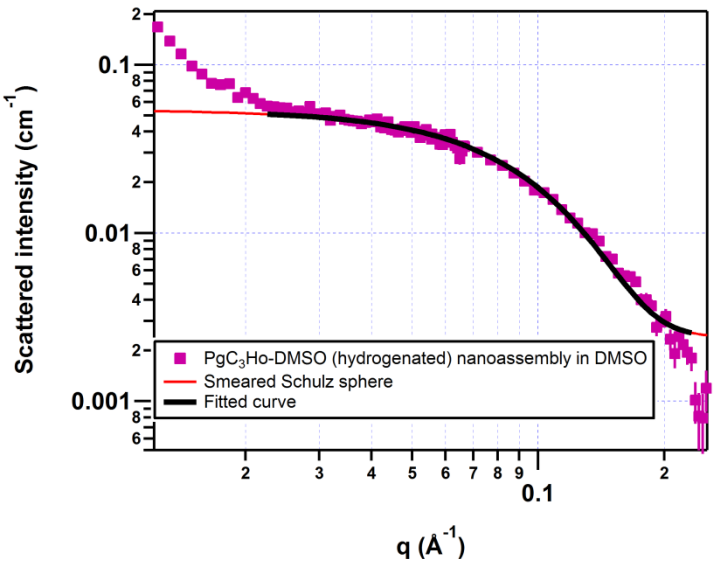

Figure 11. Schulz sphere fit (SANS) of $PgC_3HoDMSO$ (hydrogenated) (PgC_3:Ho:DMSO::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00068668	±	2.77699e-05
mean radius (Å)	18.2123	±	0.902951
polydisp (sig/avg)	0.22	±	0.0270519
SLD sphere ($Å^{-2}$)	1.09e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221571	±	0.000174111

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene ($PgC_3HoDMSO$) nanoassembly gives good statistics. The metal-coordinated DSMO ligand on the is modeled as hydrogenated ligands whereas the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DSMO (hydrogenated/deuterated) does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

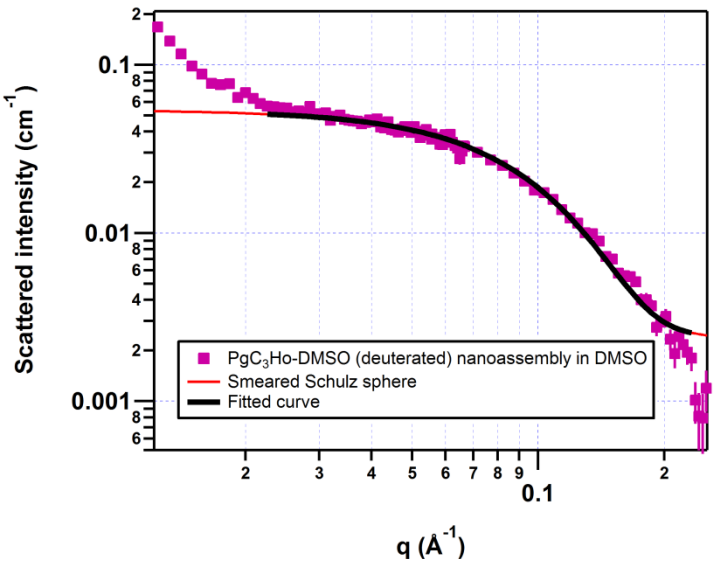

Figure 12. Schulz sphere fit (SANS) of $PgC_3HoDMSO$ (deuterated) (PgC_3:Ho:DMSO::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.0015829	±	6.40481e-05
mean radius (Å)	18.2107	±	0.903453
polydisp (sig/avg)	0.22	±	0.027069
SLD sphere ($Å^{-2}$)	2.52e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221478	±	0.000174215

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene ($PgC_3HoDMSO$-d6) nanoassembly gives good statistics. The metal-coordinated DSMO ligand is modeled as deuterated ligands as well as the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DSMO (hydrogenated/deuterated) does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

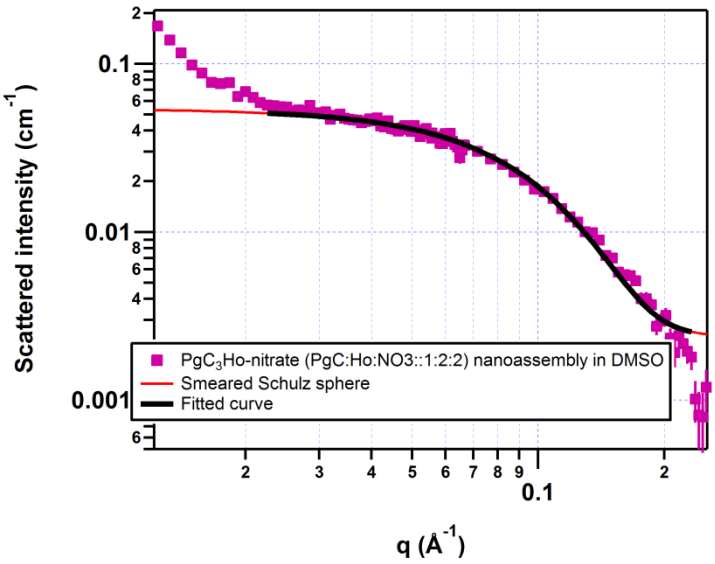

Figure 13. Schulz sphere fit (SANS) of PgC_3Ho-NO_3 (PgC_3:Ho:NO_3::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00106128	±	4.30709e-05
mean radius (Å)	18.214	±	0.907416
polydisp (sig/avg)	0.22	±	0.0271979
SLD sphere ($Å^{-2}$)	1.91e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221668	±	0.000174556

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3HoNO3) nanoassembly with pyrogallol[4]arene:metal:NO3 ratio of 1:2:2 gives good statistics. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DMSO or the change in macrocylce to metal to ligand ratio does not affect the overall radius of observed spherical species in solution (fits below).

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

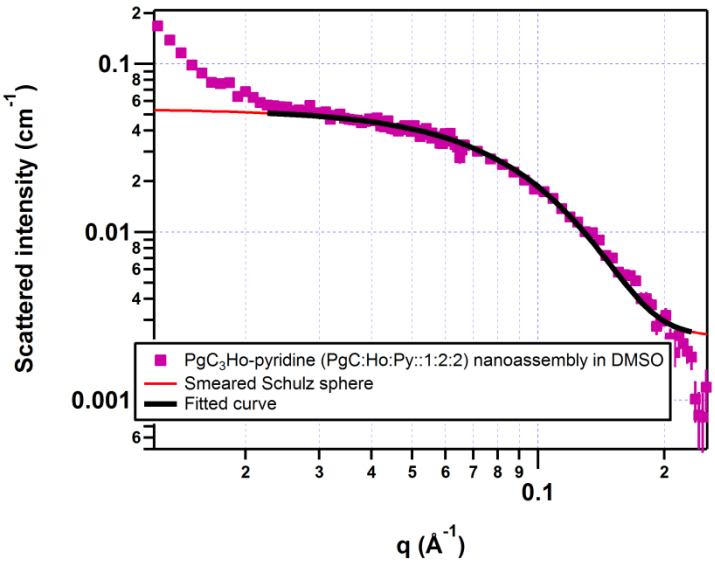

Figure 14. Schulz sphere fit (SANS) of PgC_3Ho-Py (PgC_3:Ho:Py::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00100111	±	4.02602e-05
mean radius (Å)	18.2131	±	0.896459
polydisp (sig/avg)	0.22	±	0.0268353
SLD sphere ($Å^{-2}$)	1.81e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221616	±	0.000173398

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene (PgC_3HoPy) nanoassembly with pyrogallol[4]arene:metal:DMSO ratio of 1:2:2 gives good statistics. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine or the change in macrocylce to metal to ligand ratio does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

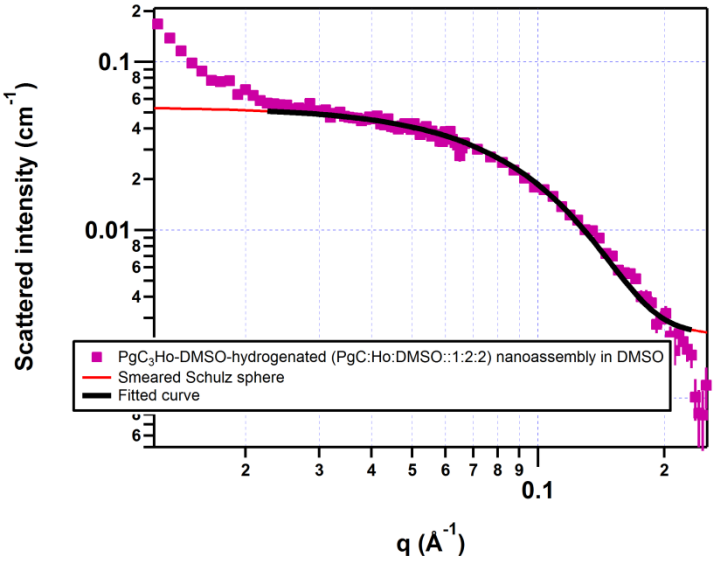

Figure 15. Schulz sphere fit (SANS) of PgC_3Ho-DMSO-hydrogenated (PgC_3:Ho:DMSO::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000805042	±	3.24516e-05
mean radius (Å)	18.2113	±	0.898547
polydisp (sig/avg)	0.22	±	0.0268881
SLD sphere ($Å^{-2}$)	1.41e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.0022151	±	0.000173741

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene ($PgC_3HoDMSO$) nanoassembly with pyrogallol[4]arene:metal:DMSO ratio of 1:2:2 gives good statistics. The external DSMO ligand is modeled as hydrogenated ligands whereas the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DMSO or the change in macrocylce to metal to ligand ratio does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_3Ho

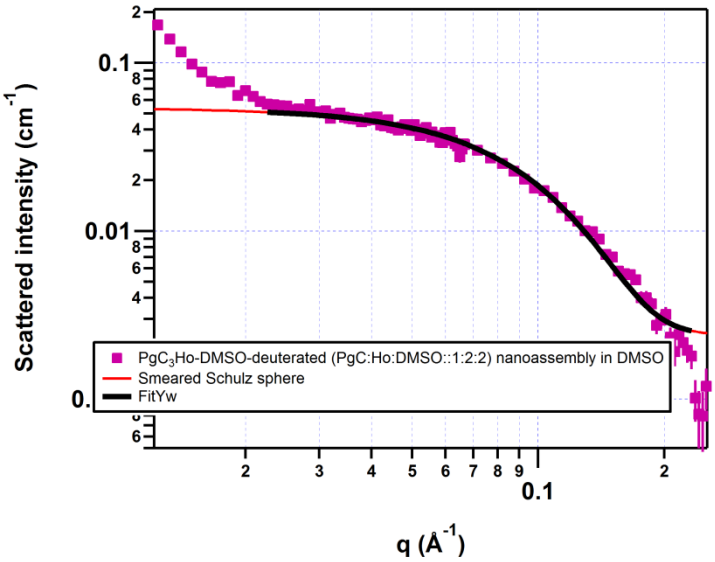

Figure 16. Schulz sphere fit (SANS) of PgC_3Ho-DMSO-deuterated (PgC_3:Ho:DMSO::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00146349	±	5.92196e-05
mean radius (Å)	18.2129	±	0.904128
polydisp (sig/avg)	0.22	±	0.0270938
SLD sphere ($Å^{-2}$)	2.41e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.00221602	±	0.000174196

chisq = 178.74
Npnts = 89 Sqrt(χ^2/N) = 1.41715
Fitted range = [17,105] = 0.02255 < Q < 0.2323

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-propylpyrogallol[4]arene ($PgC_3HoDMSO$) nanoassembly with pyrogallol[4]arene:metal:DMSO ratio of 1:2:2 gives good statistics. The external DSMO ligand is modeled as deuterated ligands and the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.4 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DMSO or the change in macrocylce to metal to ligand ratio does not affect the overall radius of observed spherical species in solution.

Fit to Smeared Core Shell Sphere
Data file: PgC_1Ho

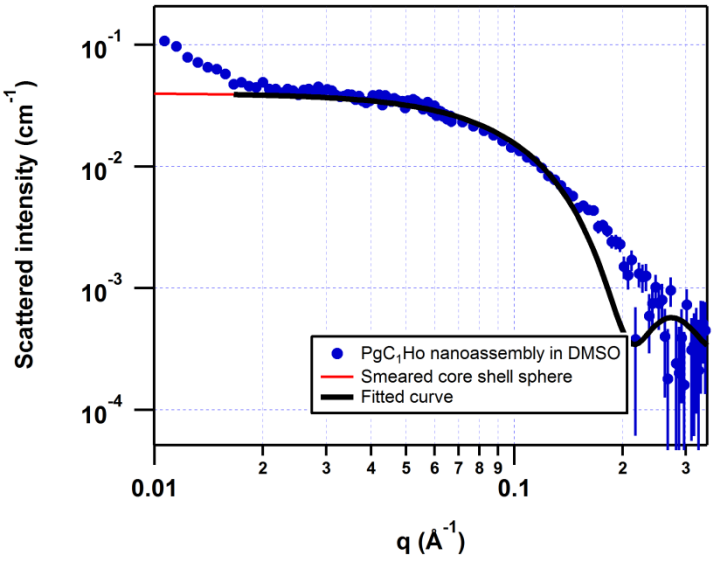

Figure 17. Core shell sphere fit (SANS) of PgC_1Ho nanoassembly in *d6*-DMSO

scale	0.000745498	±	3.40446e-05
core radius (Å)	1.64887	±	12.1295
shell thickness (Å)	19.4832	±	12.3012
Core SLD ($Å^{-2}$)	5.28e-06	±	(held fixed)
Shell SLD ($Å^{-2}$)	1.6e-06	±	(held fixed)
Solvent SLD ($Å^{-2}$)	5.28e-06	±	(held fixed)
bkg (cm^{-1})	0.000298118	±	4.16328e-05

chisq = 776.634
Npnts = 158
Sqrt(χ^2/N) = 2.21707

Fitted range = [10,167] = 0.01659 < Q < 0.514

NOTE: SANS data fitted as a core shell sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly does not fit well. The high chi-sqrt value of 2.2, large error bars on core and shell parameters and the poor visual fit indicate that the model function is a poor description of the solution structures.

Fit to Smeared Core Shell Cylinder,
Data file: PgC_1Ho

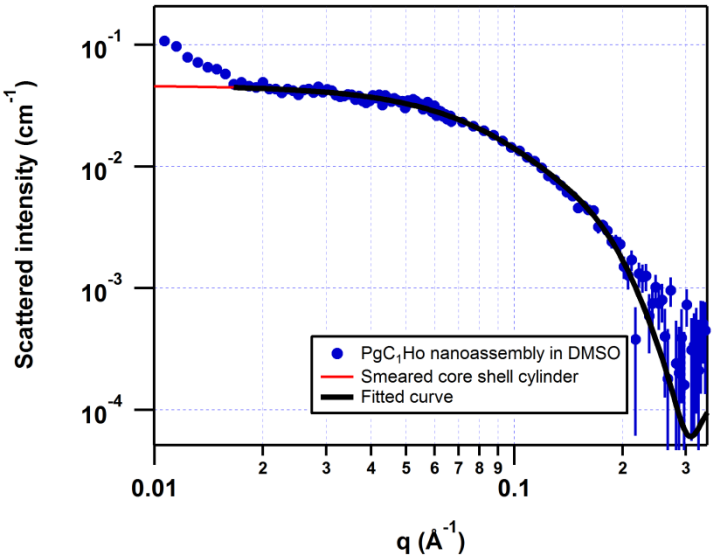

Figure 18 Core shell cylinder fit (SANS) of PgC_1Ho nanoassembly in *d6*-DMSO

scale	0.00100627	±	0.000241265
core radius (Å)	0.673498	±	12.9203
shell radius (Å)	12.4153	±	13.5327
length (Å)	38.8482	±	25.9597
SLD core (Å^{-2})	5.28e-06	±	(held fixed)
SLD shell (Å^{-2})	1.6e-06	±	(held fixed)
SLD solvent (Å^{-2})	5.28e-06	±	(held fixed)
incoh. bkg (cm^{-1})	1.09253e-05	±	7.63735e-05

chisq = 227.473 Npnts = 158
Sqrt(χ^2/N) = 1.19988
Fitted range = [10,167] = 0.01659 < Q < 0.514

NOTE: SANS data fitted as a core shell cylinder for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly does not fit well. The large error bars on length, core and shell parameters suggest that the model function is not an appropriate description of the structures in solution.

Fit to Smeared Cylinder Form
Data file: PgC_1Ho

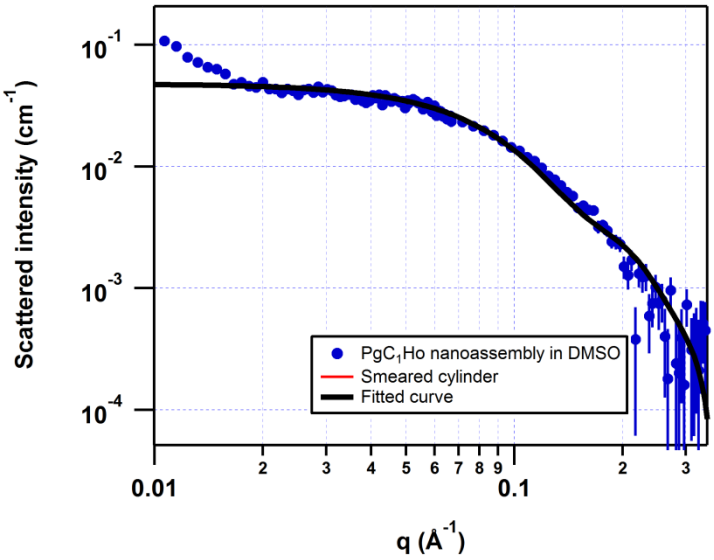

Figure 19 Cylinder fit (SANS) of PgC_1Ho nanoassembly in *d6*-DMSO

scale	0.00185289	±	0.000348218
radius (Å)	27.4845	±	0.212189
length (Å)	8.16355	±	1.56026
SLD cylinder ($Å^{-2}$)	1.6e-06	±	(held fixed)
SLD solvent ($Å^{-2}$)	5.28e-06	±	(held fixed)
incoh. bkg (cm^{-1})	-0.000521729	±	0.000146033

chisq = 1868.12 Npnts = 168
Sqrt(χ^2/N) = 3.33463
Fitted range = [0,167] = 0.008085 < Q < 0.514

NOTE: SANS data fitted as a cylinder for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly does not fit well. The high chi-sqrt value of 3.3 and large error bars on the fitted length indicate a poor fit. Also, the radius of 27 Å is an unphysically large structure.

Fit to Smeared Ellipsoid Form
Data file: PgC_1Ho

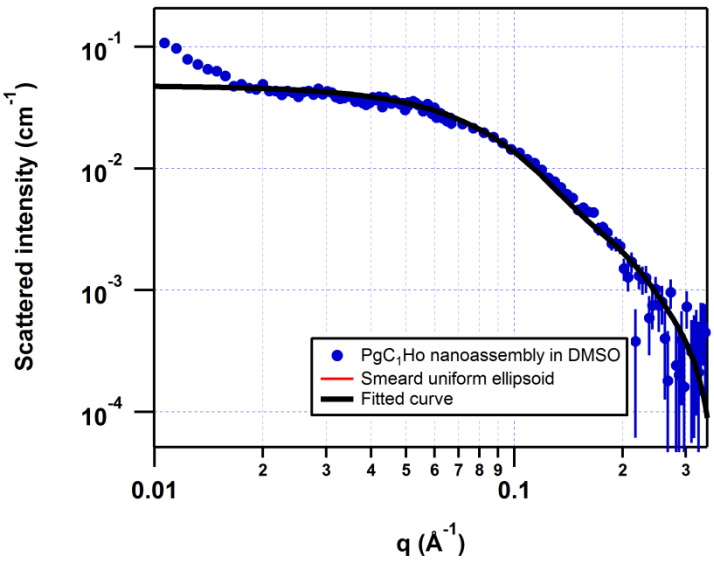

Figure 20 Uniform Ellipsoid fit (SANS) of PgC_1Ho nanoassembly in *d6*-DMSO

scale	0.00158737	±	0.000190387
R a (rotation axis) (Å)	5.61568±	0.698934	
R b (Å)	30.9802±	0.246887	
SLD ellipsoid (Å^{-2})	1.6e-06	±	(held fixed)
SLD solvent (Å^{-2})	5.28e-06	±	(held fixed)
incoh. bkg (cm^{-1})	-0.000351561	±	9.91991e-05

chisq = 1836.4 Npnts = 168

Sqrt(χ^2/N) = 3.3062
Fitted range = [0,167] = 0.008085 < Q < 0.514

NOTE: SANS data fitted as a uniform ellipsoid for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly does not fit well. The high chi-sqrt value of 3.3 indicates poor quality fit. Further, Ra is smaller than the bowl size of a single pyrogallol[4]arene unit and hence is an unphysical representation of nanoassembly in solution.

Fit to Smeared Triaxial Ellipsoid
Data file: PgC_1Ho

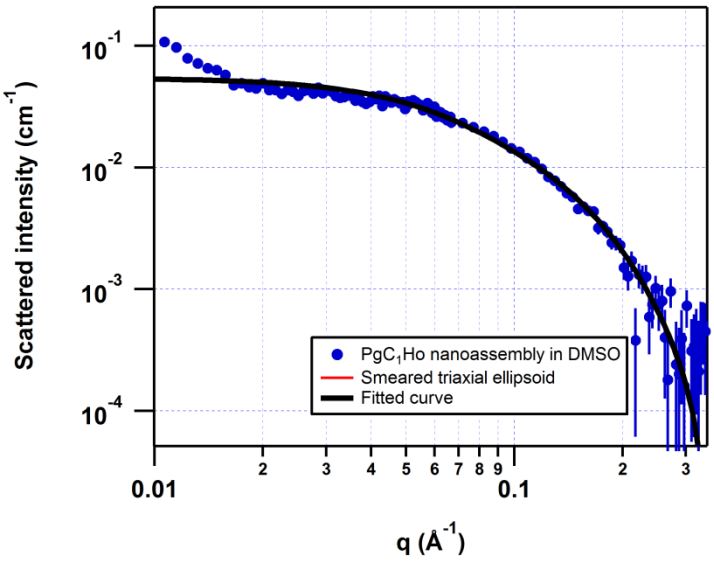

Figure 21 Triaxial ellipsoid fit (SANS) of PgC_1Ho nanoassembly in *d6*-DMSO

Scale Factor	0.00121444	±	5.35927e-05
Semi-axis A [smallest] (Å)	9.10131	±	0.665282
Semi-axis B (Å)	16.7588	±	0.718958
Semi-axis C [largest] (Å)	52.2814	±	1.03132
SLD ellipsoid (Å^{-2})	1.6e-06	±	(held fixed)
SLD solvent (Å^{-2})	5.28e-06	±	(held fixed)
Incoherent Bgd (cm^{-1})	-0.000172957	±	5.79477e-05

chisq = 1635.95 Npnts = 168
Sqrt(χ^2/N) = 3.12054
Fitted range = [0,167] = 0.008085 < Q < 0.514

NOTE: SANS data fitted as a triaxial ellipsoid for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly does not fit well. The high chi-sqrt value of 3.3 indicate a poor quality fit.

Fit to SmearedBimodalSchulzSpheres
Data file: PgC_1Ho

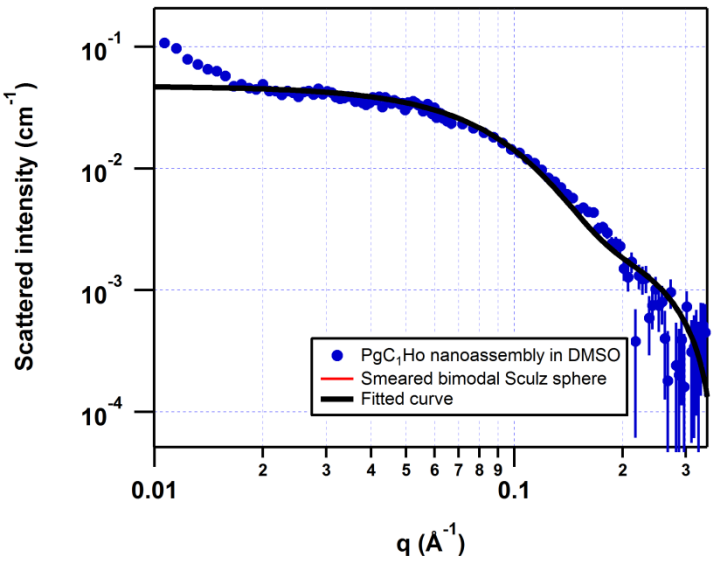

Figure 22 Bimodal Schulz sphere fit (SANS) of PgC_1Ho nanoassembly in *d6*-DMSO

volume fraction(1)	0.00139499	±	7.67148e-05
Radius (1) (Å)	6.86627	±	0.0158154
polydispersity(1)	0.174856	±	0.000297927
SLD(1) (Å^{-2})	1.6e-06	±	(held fixed)
volume fraction(2)	0.000591734	±	1.15867e-05
Radius (2) (Å)	20.4393	±	0.189617
polydispersity(2)	0.2	±	0.00301689
SLD(2) (Å^{-2})	1.6e-06	±	(held fixed)

SLD (solvent) (Å^{-2})	5.28e-06	±	(held fixed)
background (cm-1 sr-1)	-0.000600753	±	6.20329e-05

chisq = 1915.26 Npnts = 168
Sqrt(χ^2/N) = 3.37644
Fitted range = [0,167] = 0.008085 < Q < 0.514

NOTE: SANS data fitted as a bimodal Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly does not fit well. The high chi-sqrt value of 3.4 indicates that this is not the best model description of PgC_1Ho nanoassembly.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

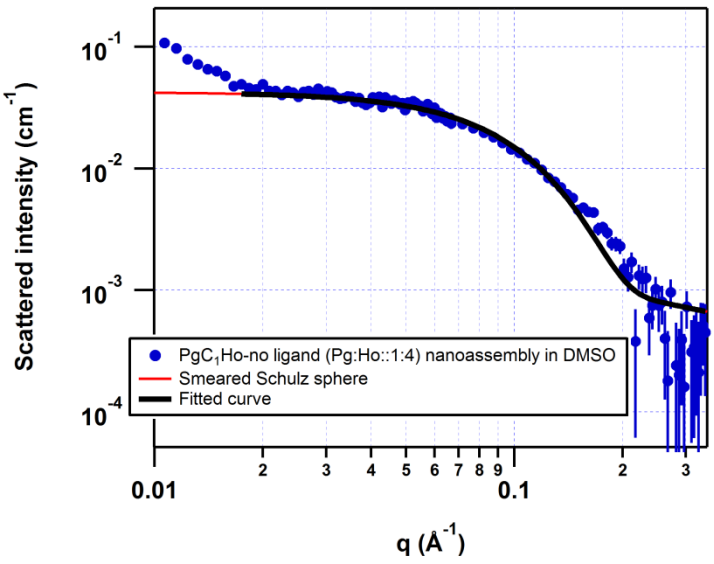

Figure 23. Schulz sphere fit (SANS) of PgC_1Ho (no ligand) (PgC_1:Ho::1:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000782746	±	1.81346e-05
mean radius (A)	18.1972	±	0.565971
polydisp (sig/avg)	0.2	±	0.0190903
SLD sphere (A-2)	1.59e-06	±	0
SLD solvent (A-2)	5.28e-06	±	0

bkg (cm-1 sr-1)	0.00058722	±	5.95925e-05

chisq = 305.419
Npnts = 117 Sqrt(X^2/N) = 1.61568
Fitted range = [11,127] = 0.01744 < Q < 0.3396

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly with pyrogallol[4]arene to metal ratio of 1:4 gives good statistics. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The presence or absence of ligand does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

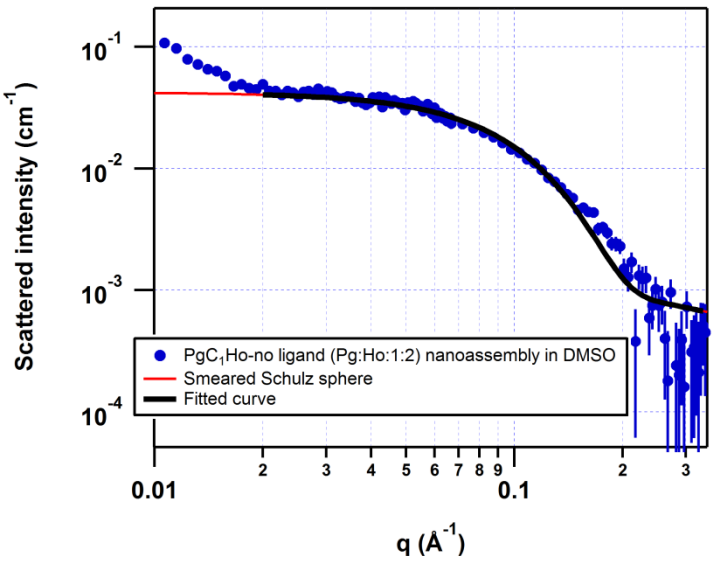

Figure 24. Schulz sphere fit (SANS) of PgC_1Ho-no ligand (PgC_1:Ho::1:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000982848	±	2.35449e-05
mean radius (Å)	18.1395±	0.588992	
polydisp (sig/avg)	0.2 ±	0.01999	
SLD sphere ($Å^{-2}$)	1.98e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000584461	±	6.12215e-05
	291.711		

Npnts = 113 Sqrt(χ^2/N) = 1.60671

Fitted range = [14,126] = 0.02 < Q < 0.3349

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1Ho) nanoassembly with pyrogallol[4]arene to metal ratio of 1:2 gives good statistics. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The presence or absence of ligand does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

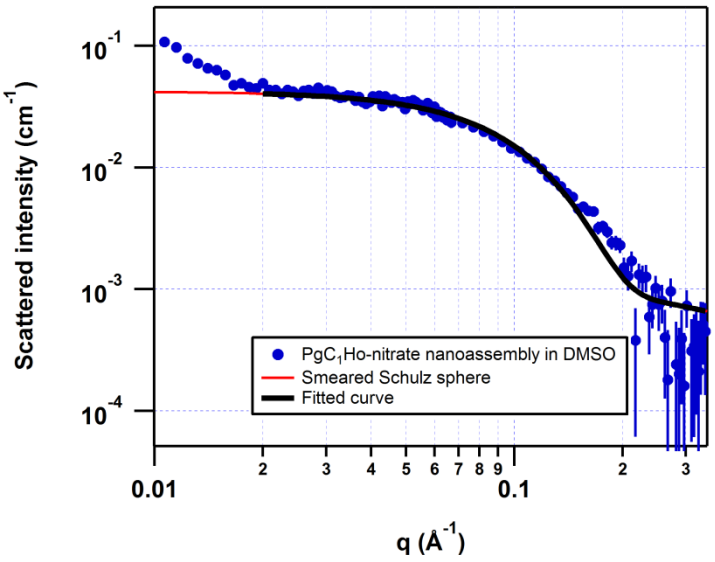

Figure 25. Schulz sphere fit (SANS) of PgC_1HoNO_3 (PgC_1:Ho:NO_3::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00101364	±	2.38813e-05
mean radius (Å)	18.1389	±	0.579579
polydisp (sig/avg)	0.2	±	0.0197359
SLD sphere (Å^{-2})	2.03e-06	±	0
SLD solvent (Å^{-2})	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000579353	±	5.99716e-05

chisq = 292.18
Npnts = 114 Sqrt(χ^2/N) = 1.60093
Fitted range = [14,127] = 0.02 < Q < 0.3396

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1HoNO_3) nanoassembly gives good statistics. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

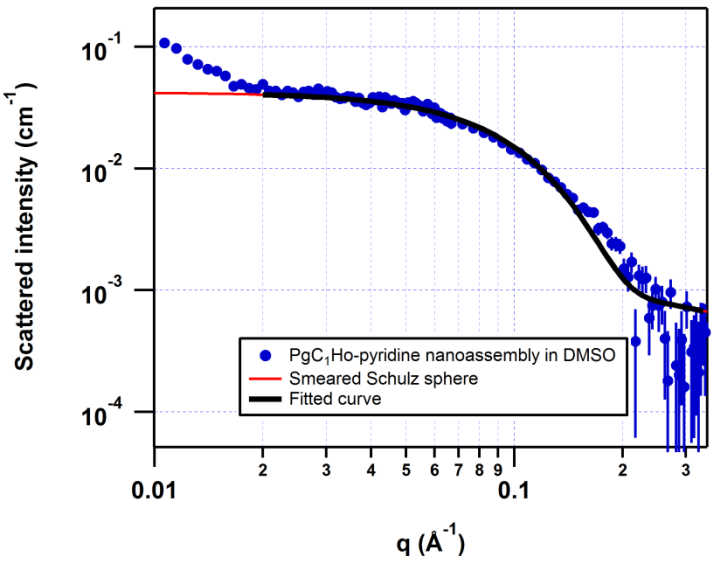

Figure 26. Schulz sphere fit (SANS) of PgC_1HoPy (PgC_1:Ho:Py::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000919807	±	2.18857e-05
mean radius (Å)	18.1455	±	0.586517
polydisp (sig/avg)	0.2	±	0.0199982
SLD sphere ($Å^{-2}$)	1.87e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000585782	±	6.10389e-05

chisq = 291.711
Npnts = 113 Sqrt(χ^2/N) = 1.60671
Fitted range = [14,126] = 0.02 < Q < 0.3349

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1HoPy) nanoassembly gives good statistics. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

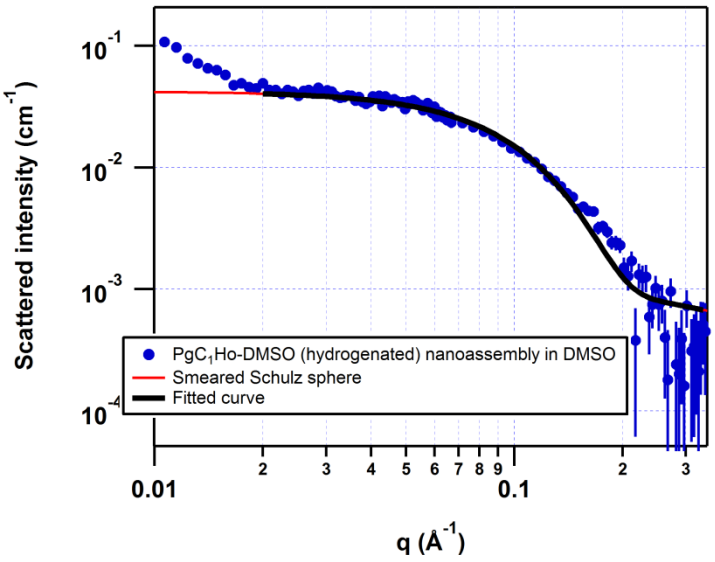

Figure 27. Schulz sphere fit (SANS) of $PgC_1HoDMSO$ (hydrogenated) (PgC_1:Ho:DMSO::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000662117	±	1.54747e-05
mean radius (Å)	18.142	±	0.566351
polydisp (sig/avg)	0.2	±	0.0191278
SLD sphere (Å^{-2})	1.26e-06	±	0
SLD solvent (Å^{-2})	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000585012	±	6.07215e-05

chisq = 291.711
Npnts = 113 Sqrt(χ^2/N) = 1.60671
Fitted range = [14,126] = 0.02 < Q < 0.3349

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene ($PgC_1HoDMSO$) nanoassembly gives good statistics. The metal-coordinated DSMO ligand is modeled as hydrogenated ligands whereas the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DMSO does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

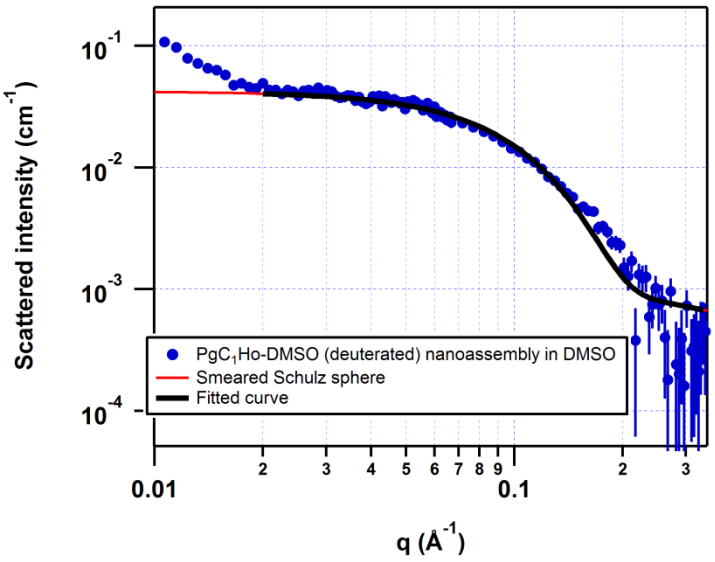

Figure 28. Schulz sphere fit (SANS) of $PgC_1HoDMSO$ (deuterated) (PgC_1:Ho:DMSO::1:4:4) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00172571	±	4.09626e-05
mean radius (Å)	18.1426	±	0.580717
polydisp (sig/avg)	0.2	±	0.0196678
SLD sphere ($Å^{-2}$)	2.79e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000585056	±	6.10305e-05

chisq = 291.711

Npnts = 113 Sqrt(χ^2/N) = 1.60671
Fitted range = [14,126] = 0.02 < Q < 0.3349

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene ($PgC_1HoDMSO$) nanoassembly gives good statistics. The metal-coordinated DSMO ligand is modeled as deuterated ligands and the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to (hydrogenated / deuterated) DMSO does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

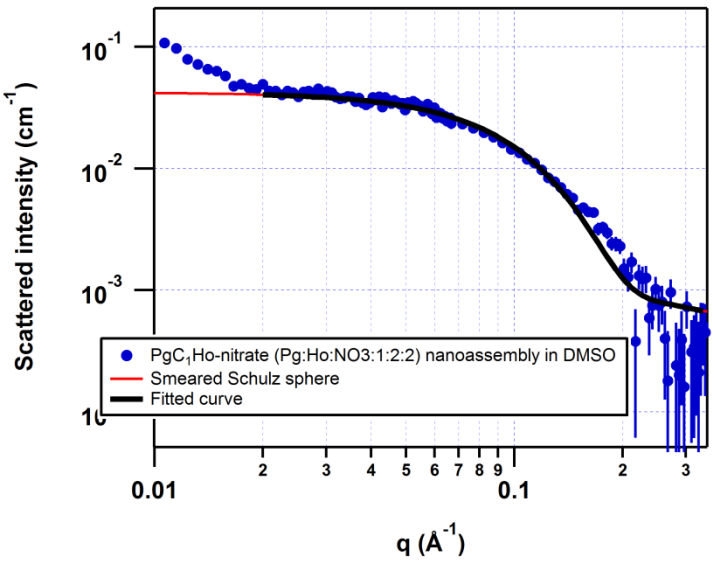

Figure 29. Schulz sphere fit (SANS) of PgC_1Ho-NO3 (PgC_1:Ho:NO_3::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00116523	±	2.79777e-05
mean radius (Å)	18.1438	±	0.594467
polydisp (sig/avg)	0.2	±	0.020288
SLD sphere ($Å^{-2}$)	2.25e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000585395	±	6.12309e-05

chisq = 291.711

Npnts = 113 Sqrt(χ^2/N) = 1.60671
Fitted range = [14,126] = 0.02 < Q < 0.3349

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1HoNO3) nanoassembly with pyrogallol[4]arene:metal:nitrate ratio of 1:2:2 gives good statistics. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

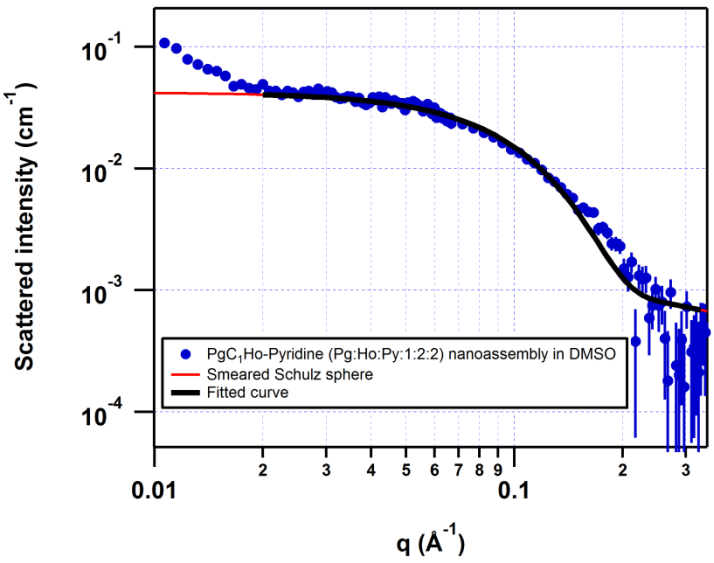

Figure 30. Schulz sphere fit (SANS) of PgC_1Ho-Py (PgC_1:Ho:Py::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00107011	±	2.57986e-05
mean radius (Å)	18.1512	±	0.589767
polydisp (sig/avg)	0.2	±	0.0199667
SLD sphere ($Å^{-2}$)	2.12e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000592469	±	6.26307e-05

chisq = 291.325
Npnts = 112 Sqrt(χ^2/N) = 1.6128
Fitted range = [14,125] = 0.02 < Q < 0.3302

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene (PgC_1HoPy) nanoassembly with pyrogallol[4]arene:metal:pyridine ratio of 1:2:2 gives good statistics. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

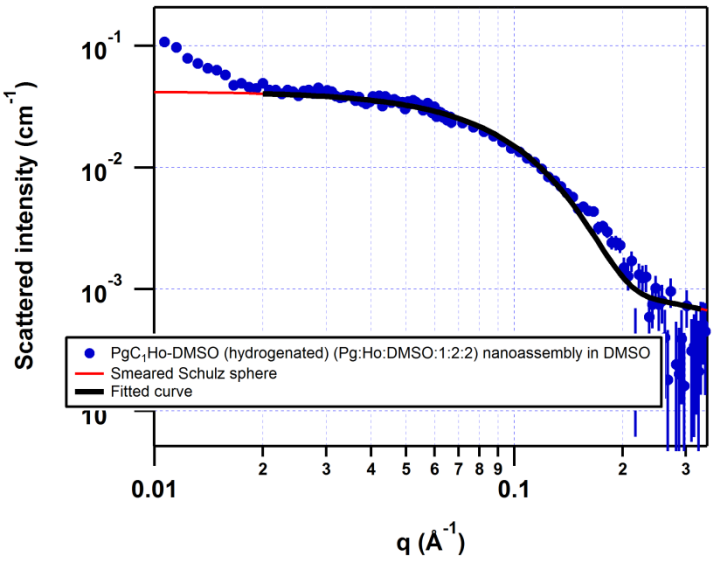

Figure 31. Schulz sphere fit (SANS) of PgC_1Ho-DMSO-hydrogenated (PgC_1:Ho:DMSO::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.000824628	±	1.98492e-05
mean radius (Å)	18.15	±	0.589588
polydisp (sig/avg)	0.2	±	0.0200058
SLD sphere ($Å^{-2}$)	1.68e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000592199	±	6.25925e-05

chisq = 291.325
Npnts = 112 Sqrt(χ ^2/N) = 1.6128
Fitted range = [14,125] = 0.02 < Q < 0.3302

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene ($PgC_1HoDMSO$) nanoassembly with pyrogallol[4]arene:metal:DMSO ratio of 1:2:2 gives good statistics. The metal-coordinated DSMO ligand is modeled as hydrogenated ligands whereas the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to DMSO does not affect the overall radius of observed spherical species in solution.

Fit to SmearedSchulzSpheres,
Data file: PgC_1Ho

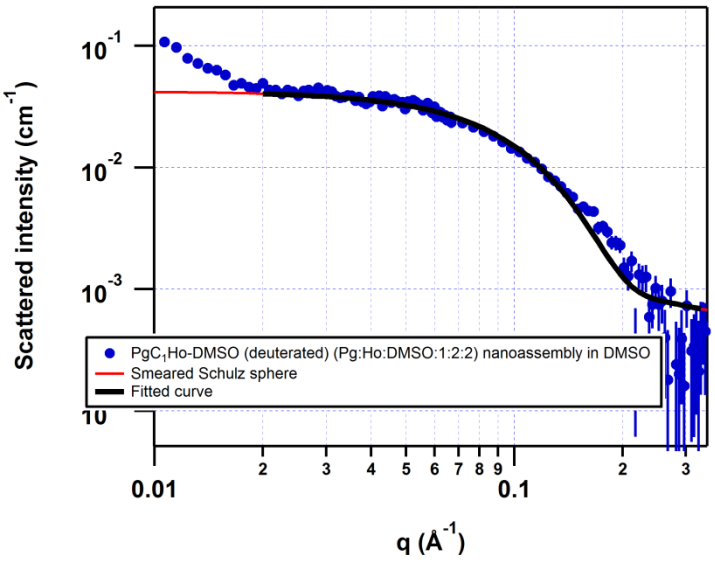

Figure 32. Schulz sphere fit (SANS) of PgC1Ho-DMSO-deuterated (PgC_1:Ho:DMSO::1:2:2) nanoassembly in *d6*-DMSO

Volume Fraction (scale)	0.00172241	±	4.17201e-05
mean radius (Å)	18.1563	±	0.595255
polydisp (sig/avg)	0.2	±	0.0201983
SLD sphere ($Å^{-2}$)	2.79e-06	±	0
SLD solvent ($Å^{-2}$)	5.28e-06	±	0
bkg (cm^{-1} sr^{-1})	0.000593633	±	6.27133e-05

chisq = 291.328
Npnts = 112 Sqrt(χ^2/N) = 1.61281

Fitted range = [14,125] = 0.02 < Q < 0.3302

NOTE: SANS data fitted as a Schulz sphere for holmium-containing *C*-methylpyrogallol[4]arene ($PgC_1HoDMSO$) nanoassembly with pyrogallol[4]arene:metal:DMSO ratio of 1:2:2 gives good statistics. The metal-coordinated DSMO ligand is modeled as deuterated ligands as well as the bulk solvent is modeled as deuterated DMSO. The small chi-sqrt value of 1.6 and small error bars on all parameters indicate good statistics. The change in ligand from nitrate to pyridine to hydrogenated DMSO to deuterated DMSO does not affect the overall radius of observed spherical species in solution.