Electronic Supporting Information

Microstructured Objects Produced by the Supramolecular Hierarchical Assembly of an Organic Free Radical gathering Hydrophobic-Amphiphilic Characteristics

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S1.- Elemental Analysis of Compound 1.

Molecular formula: $C_{87}H_{127}Cl_{14}O_3^{\cdot}$

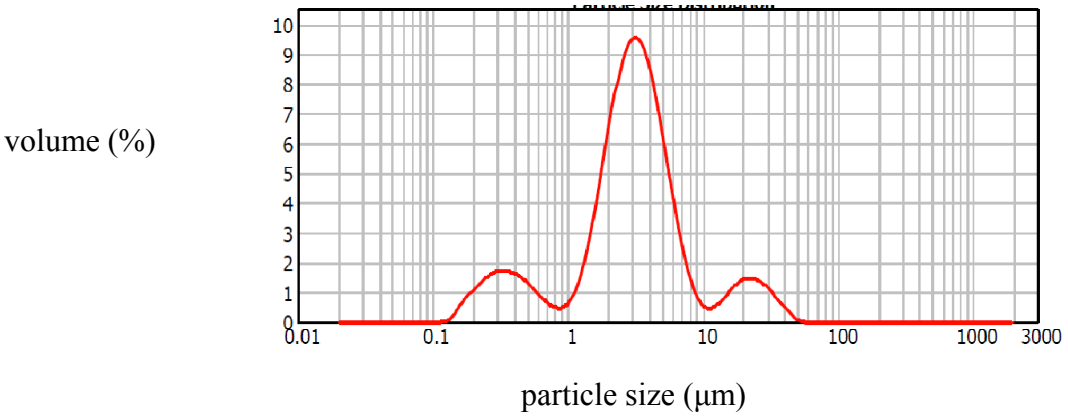
EA calculated: C (60.85%); H (7.45%); Cl (28.90%)

EA found: C (60.91%); H (4.470%); Cl (28.99%)

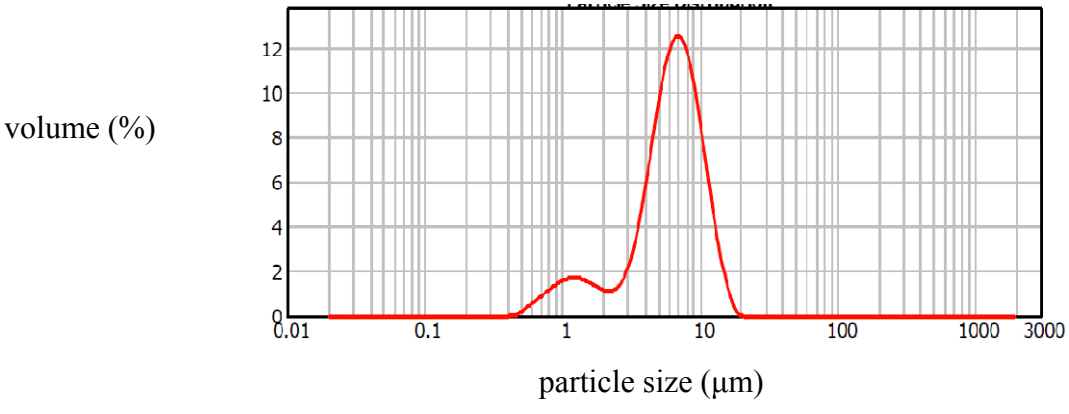
S1.- Particle Size (Dynamic light scattering)

Particle Size measurements were made with a Mastersizer 2000 instrument (Malver Instruments) with a Hydro2000µP accessory. All the samples were dispersed in iProH and sonicated 20 min at approx. 10ºC. The accessory was replenished until get obscuration around 5%.

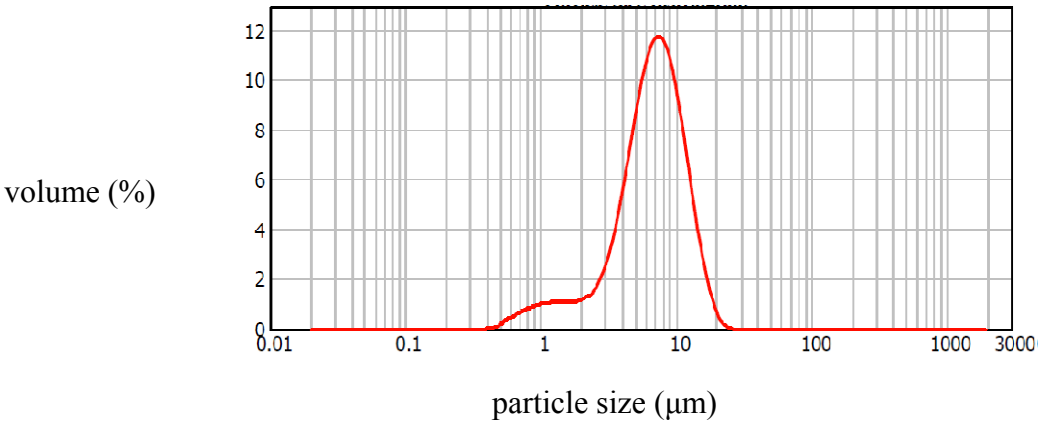
Precipitate from 1-hexanol



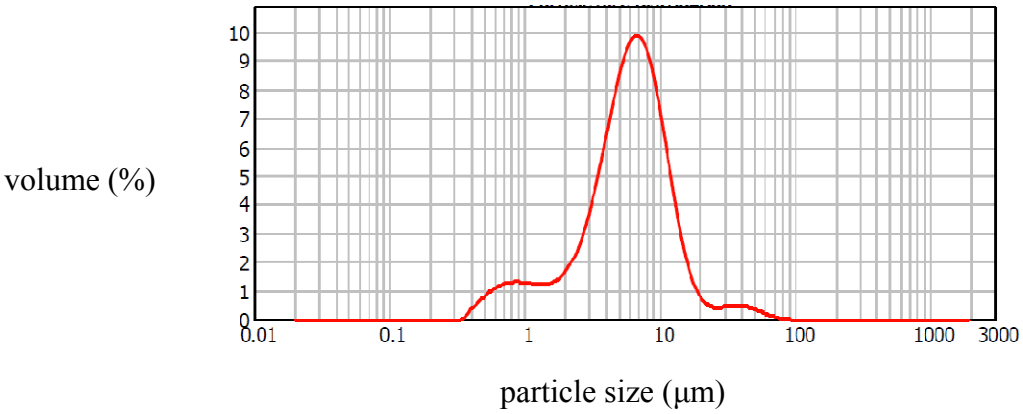
Precipitate from 1,4-dioxane



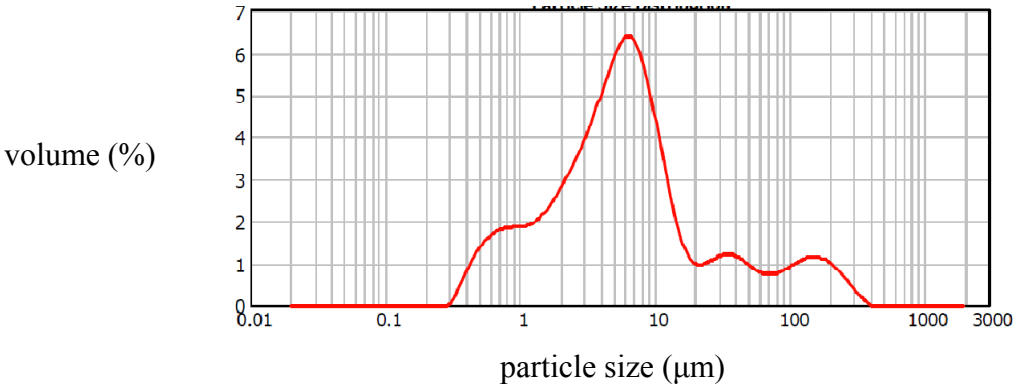
Precipitate from 1,4-dioxane/acetonitrile (3/1)



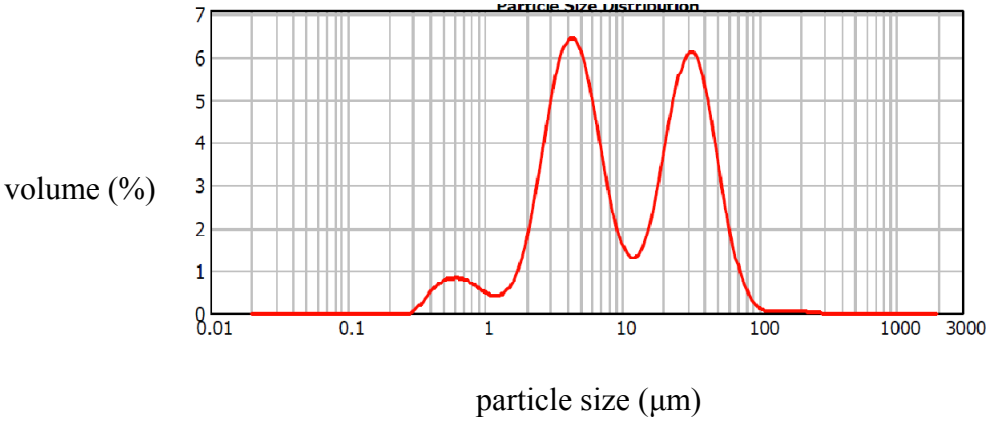
Precipitate from dioxane/methanol (1/1)



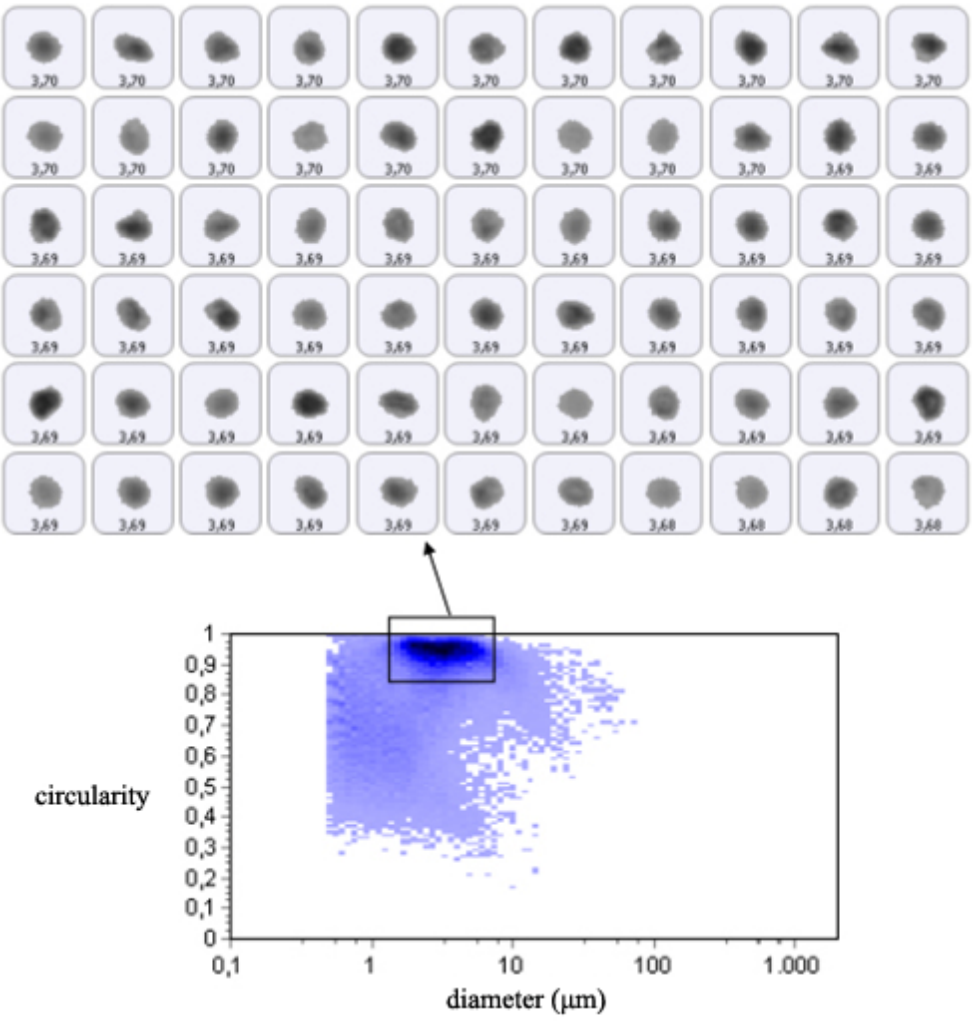
Precipitate from isopropanol



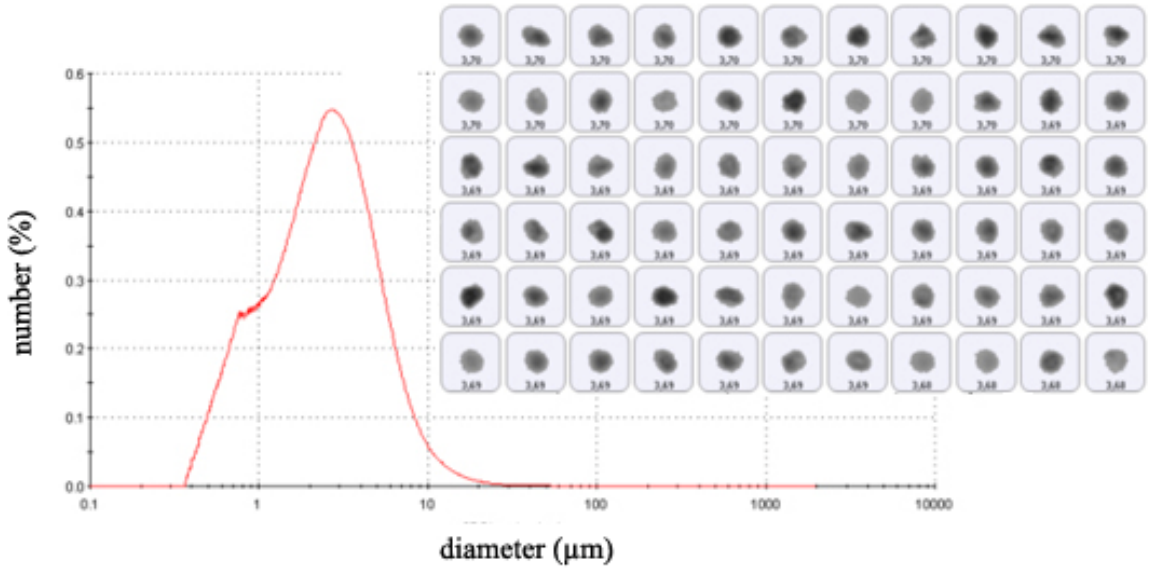
Precipitate from THF/water (2/1)



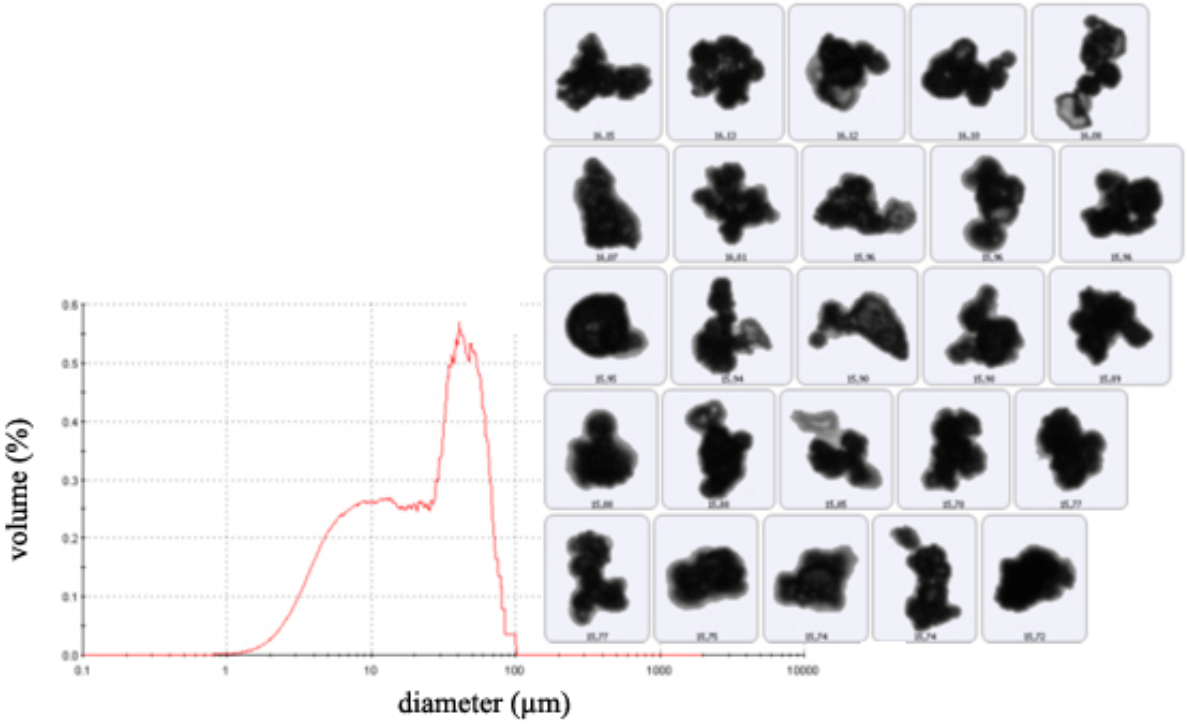
The shape and size of these assemblies were also studied by a statistic analysis with a Morphologi® G3S instrument (Malvern). Suspensions of the samples were drop cast on a glass substrate. The x20 objective counted 66.126 different objects. Most of them exhibited a diameter size in the range 1,5-7 µm and had circular shapes. It was observed that the second peak observed in the light scattering measurements that corresponds to larger particle diameters can be clearly attributed to the formation of particle aggregates.



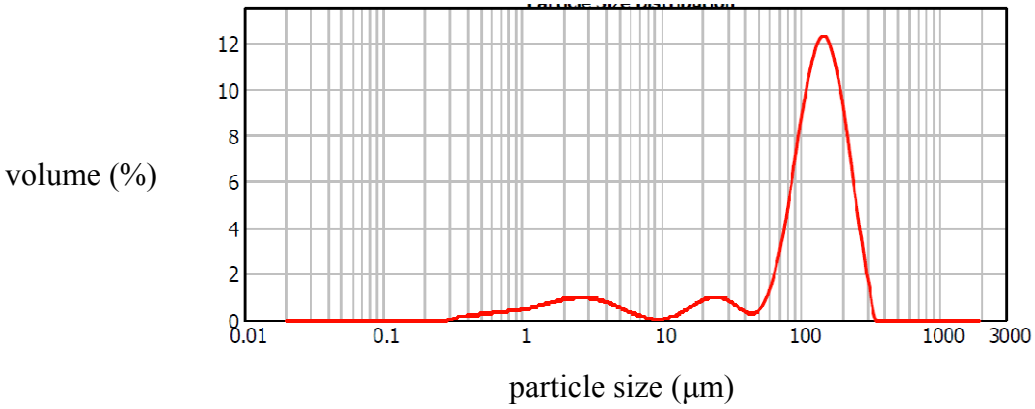
This graphic shows some of the microobjects detected in the region under the Gaussian curve:



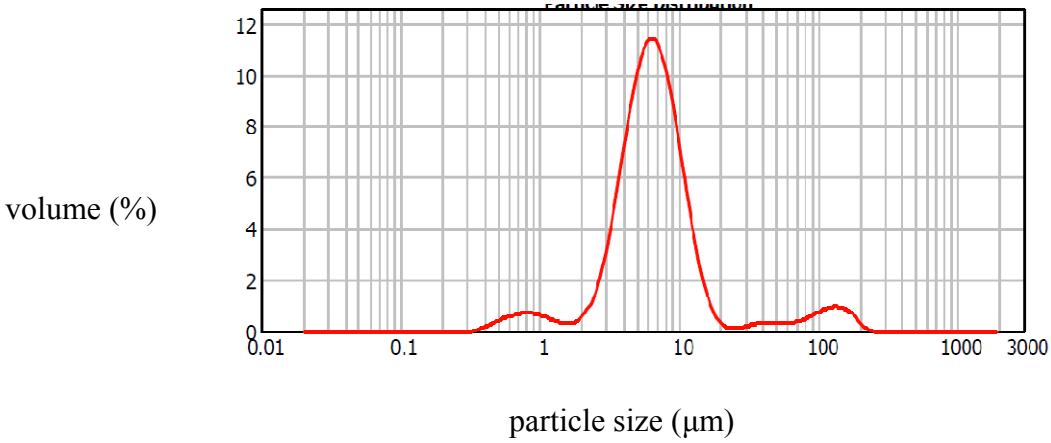
The next graphic shows some of the aggregates that correspond to the maximum (in volume) detected, due to the agreggation of several microobjects:



Precipitate from toluene/ethanol (1/1)



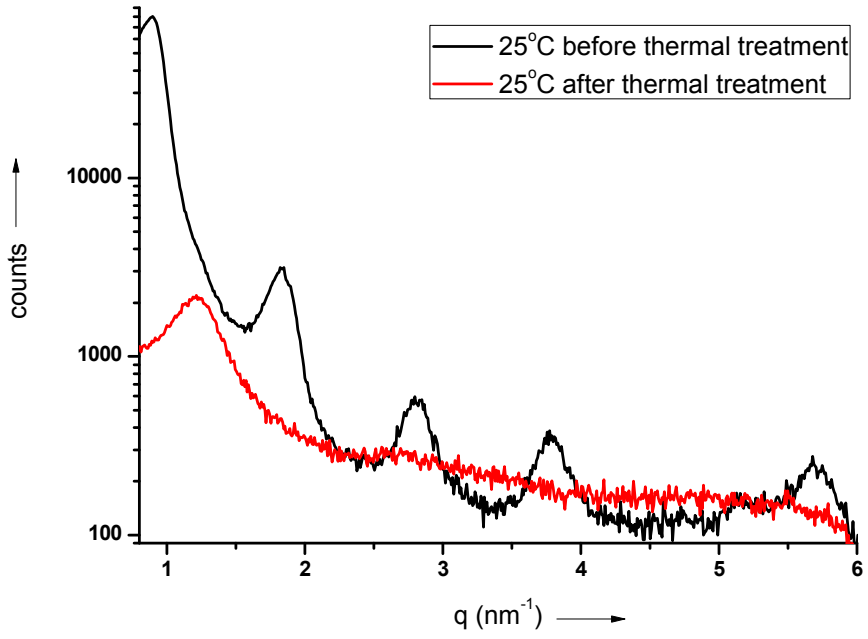
Precipitate from toluene/methanol (1/1)

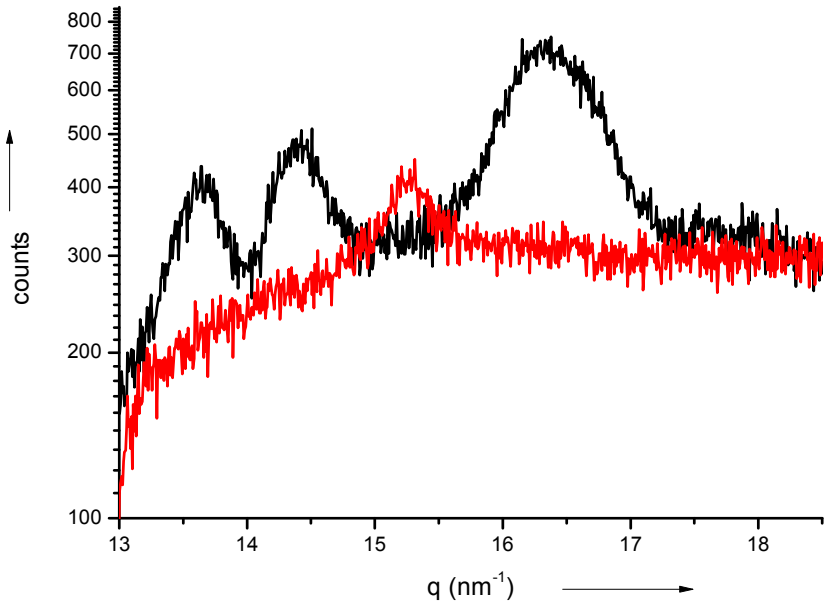


S2.- X-Ray Difraction (SAXS and WAXS)

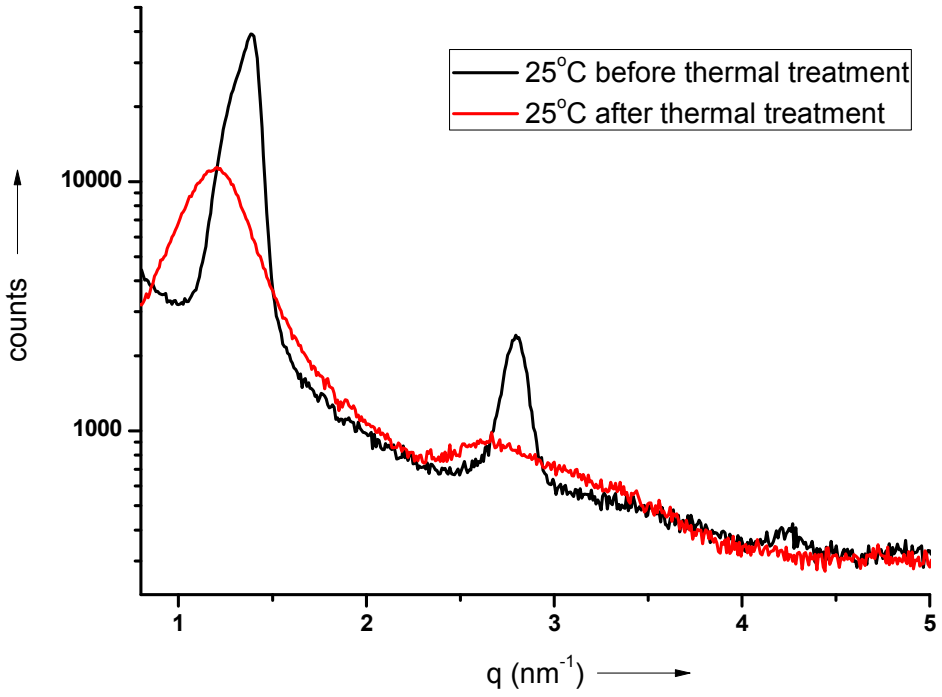
X-ray diffraction measurements were carried out in 1mm-diameter capillaries. Samples were measured during its first heating-cooling cycle, first measuring at 25ºC, than at 85ºC, cooling down to 40ºC and finally measuring again at 25ºC. Time of irradiation was 2 hours, in which temperature was kept constant. The equilibration time between determinations was 30 min in every case. Titles correspond to the solvents used to obtain the microobjects.

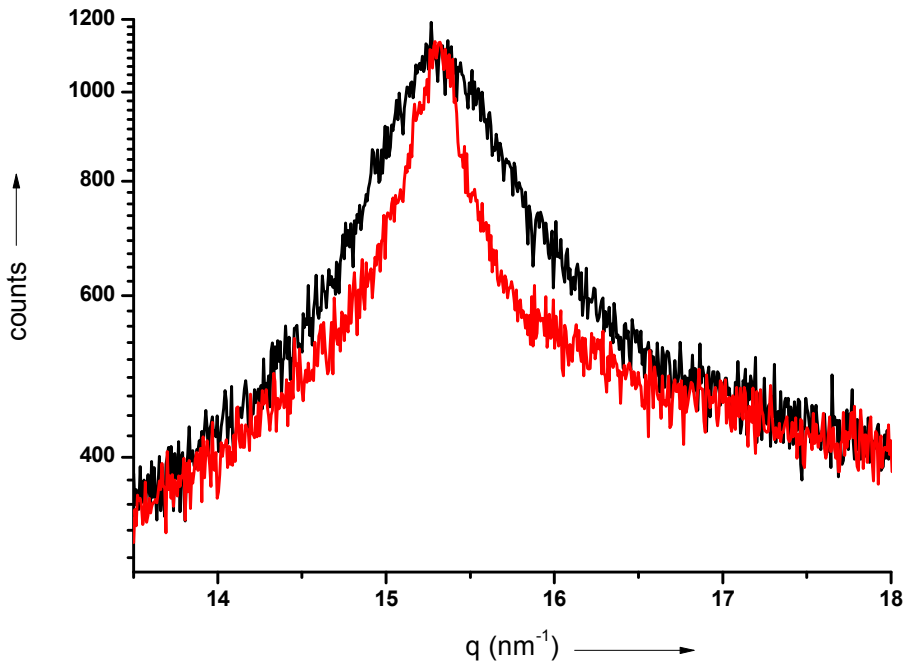
SAXS and WAXS difractograms of precipitates form 1-hexanol:



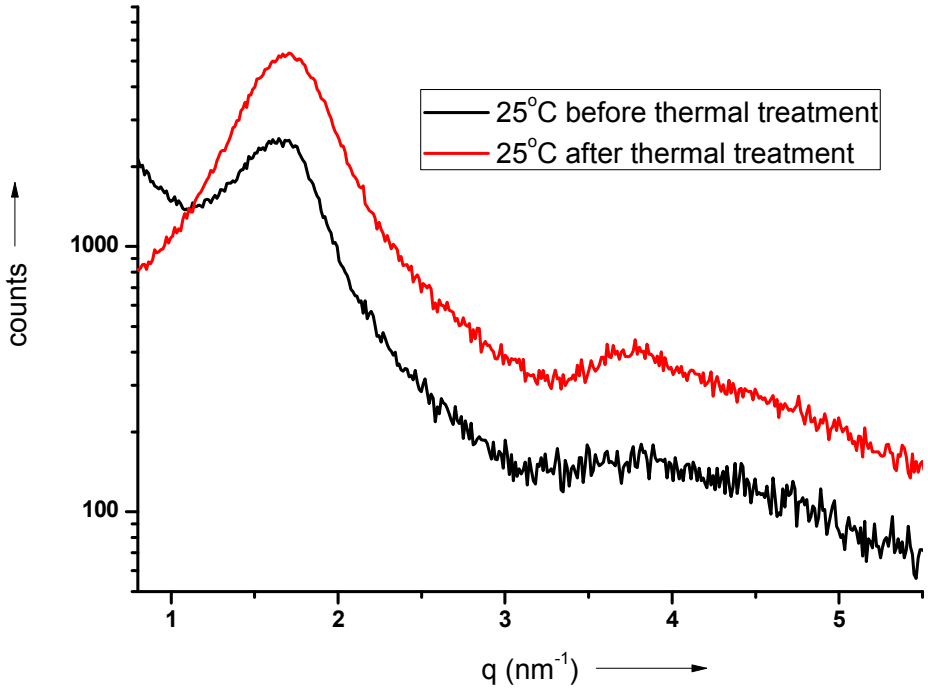


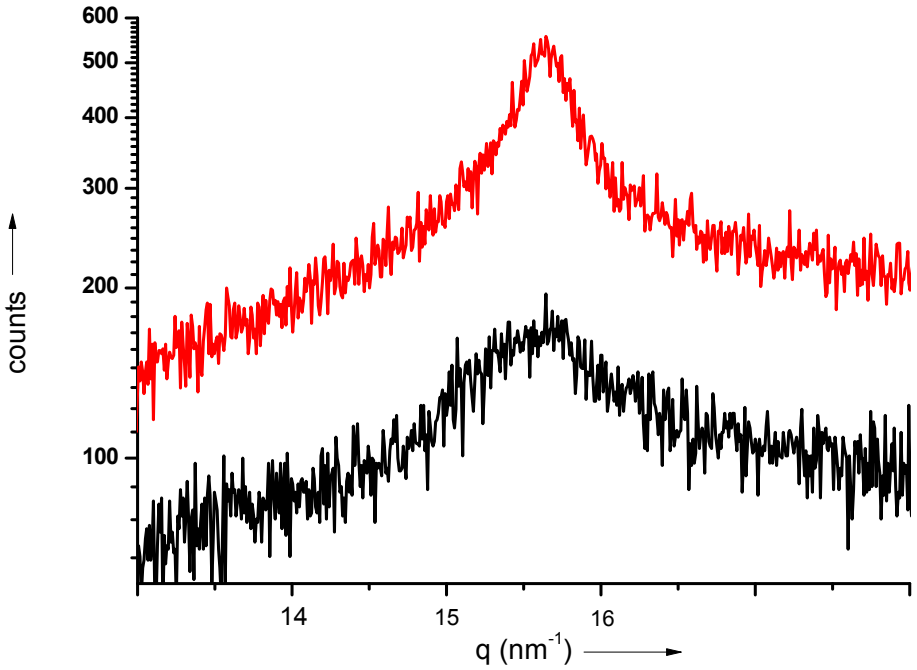
SAXS and WAXS difractograms of precipitates form 1,4-dioxane:



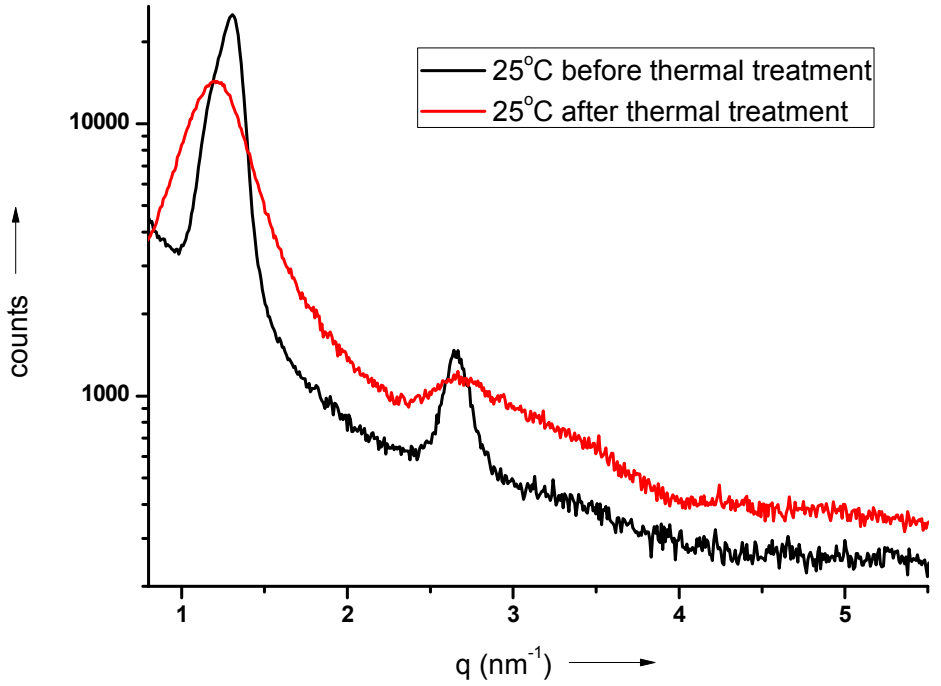


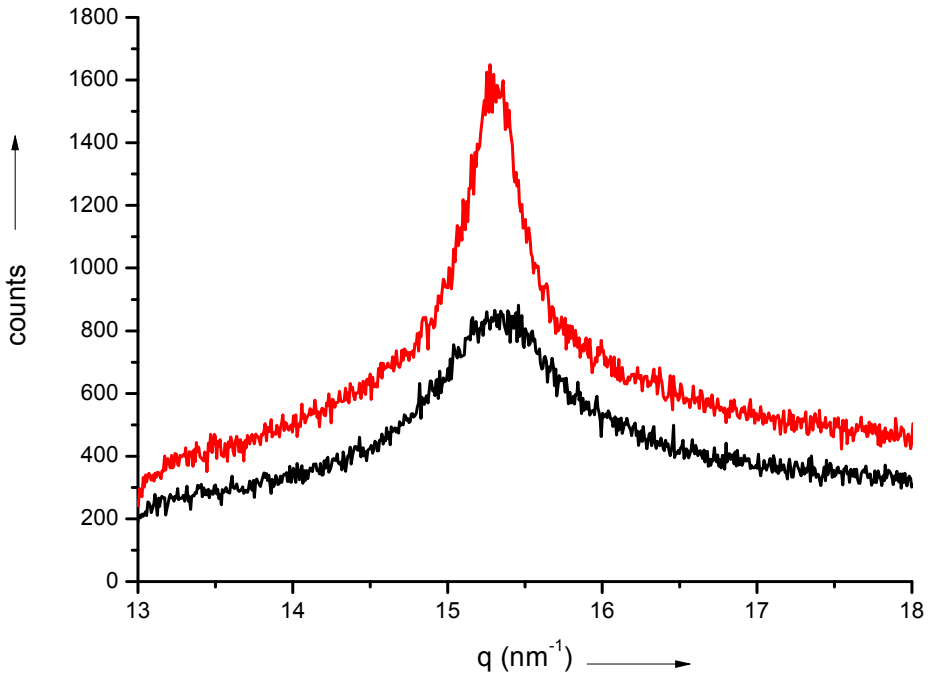
SAXS and WAXS difractograms of precipitates form isopropanol (SAXS):



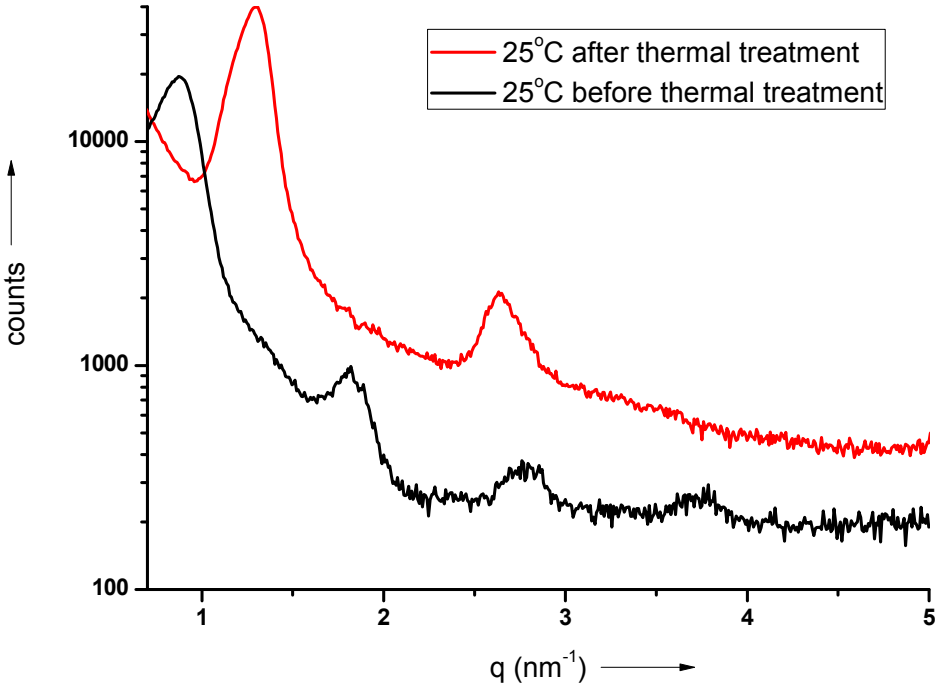


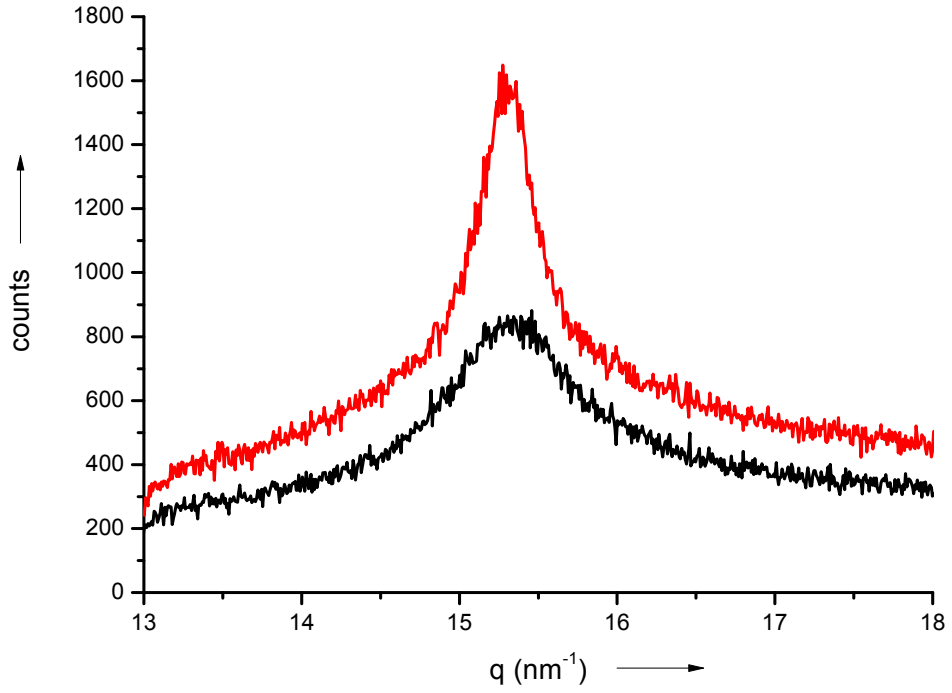
SAXS and WAXS difractograms of precipitates form 1,4-dioxane/acetonitrile (3/1):



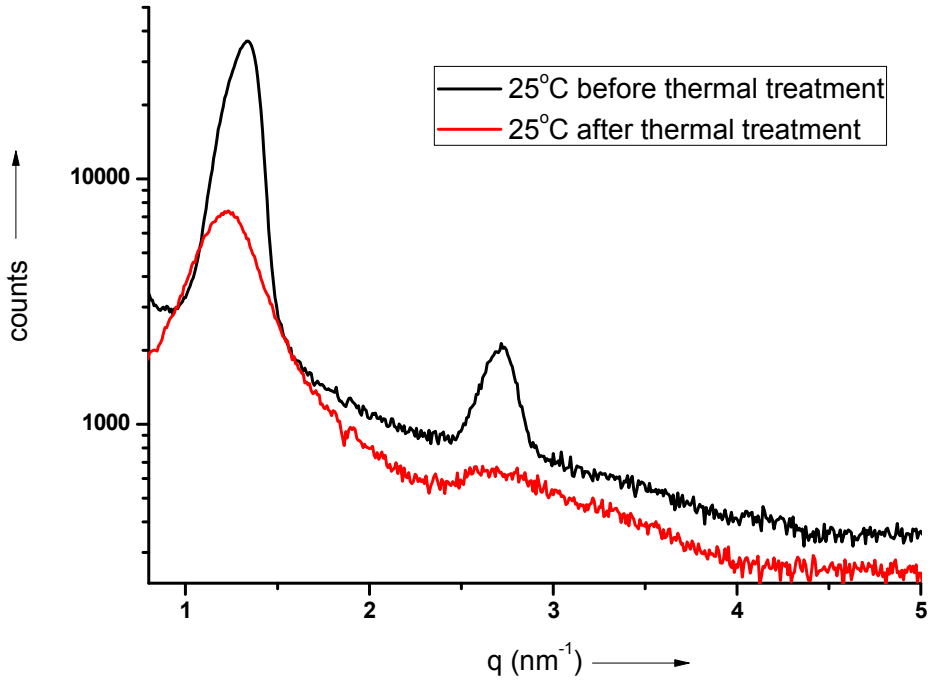


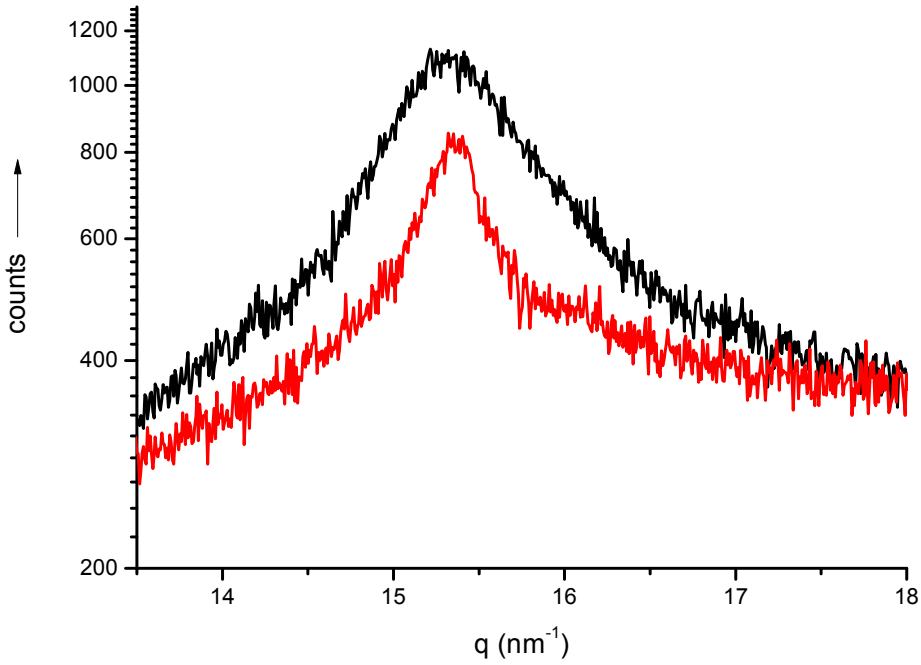
SAXS and WAXS difractograms of precipitates form 1,4-dioxane/methanol 2/1:



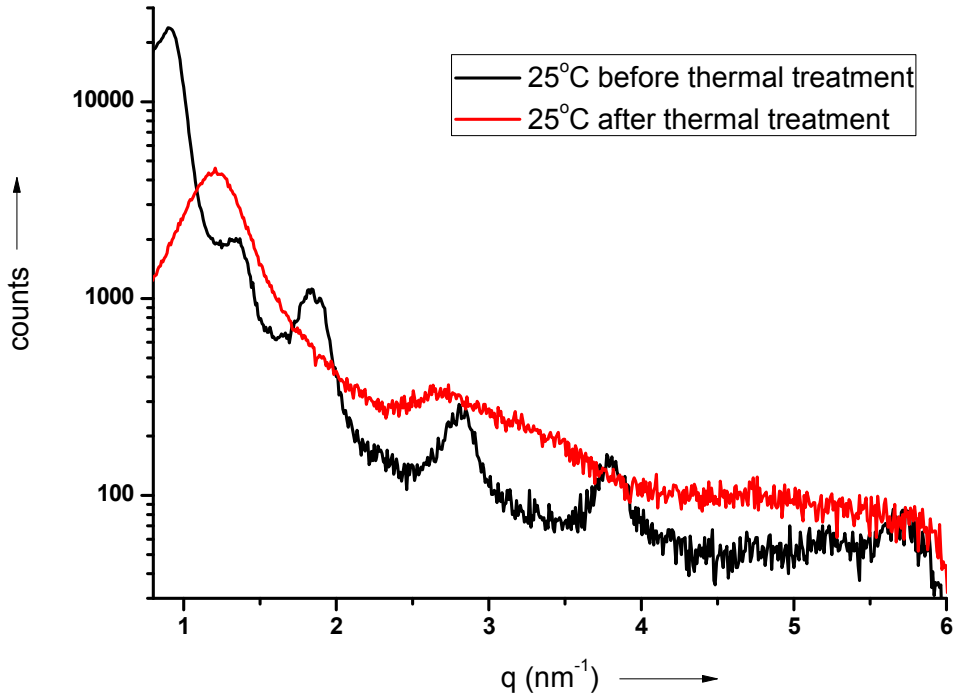


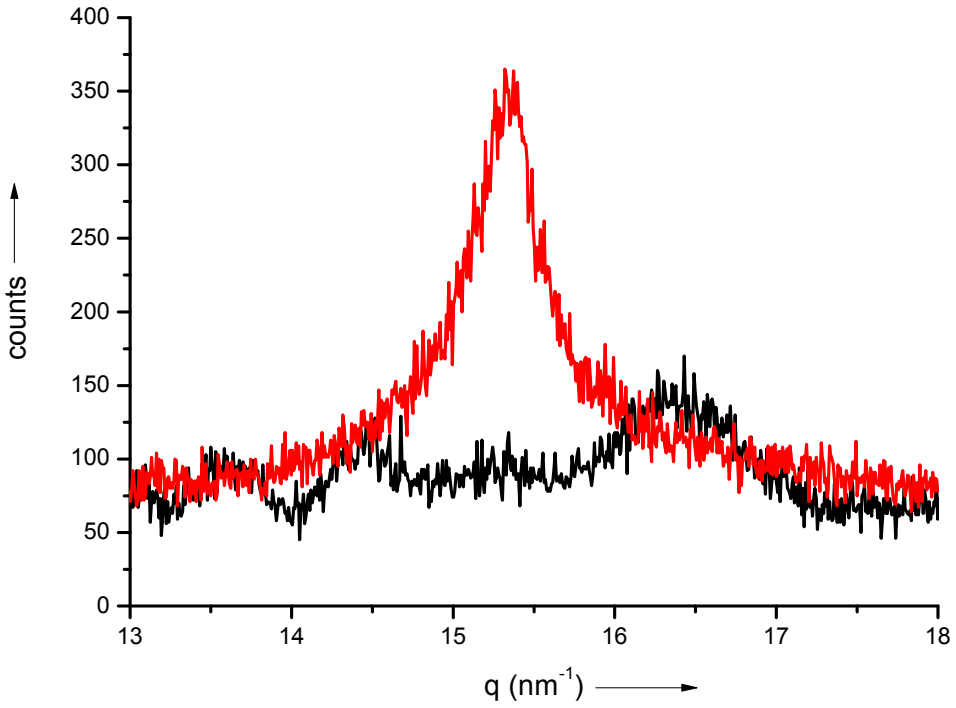
SAXS and WAXS difractograms of precipitates form THF/water (2/1):





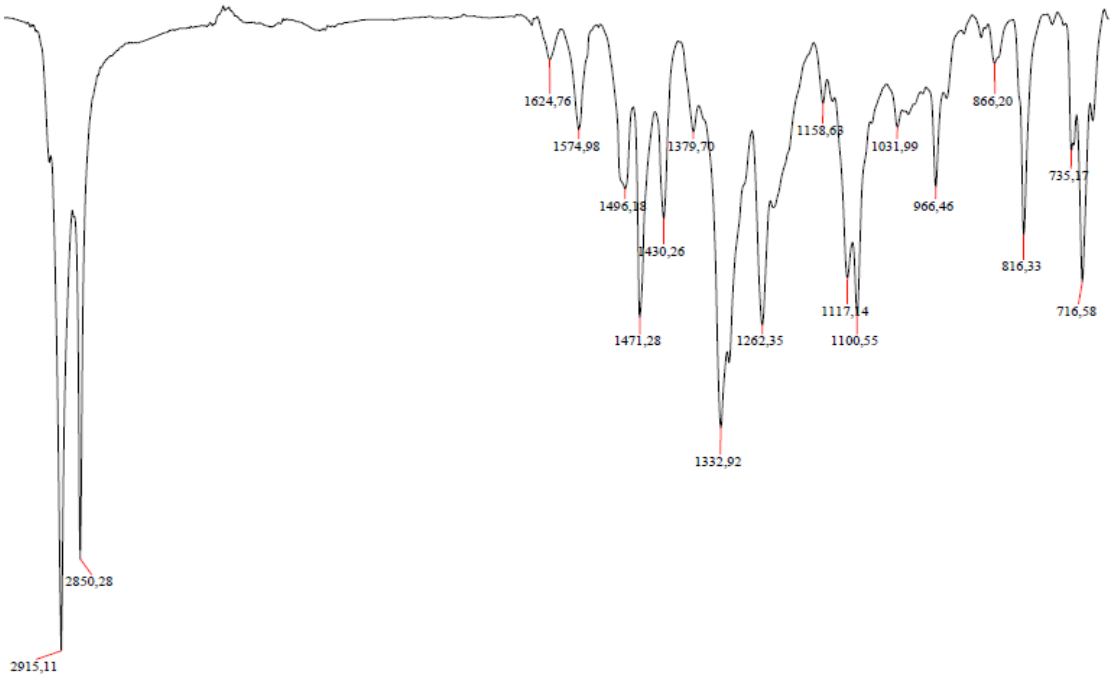
SAXS and WAXS difractograms of precipitates form toluene/methanol (1/1):



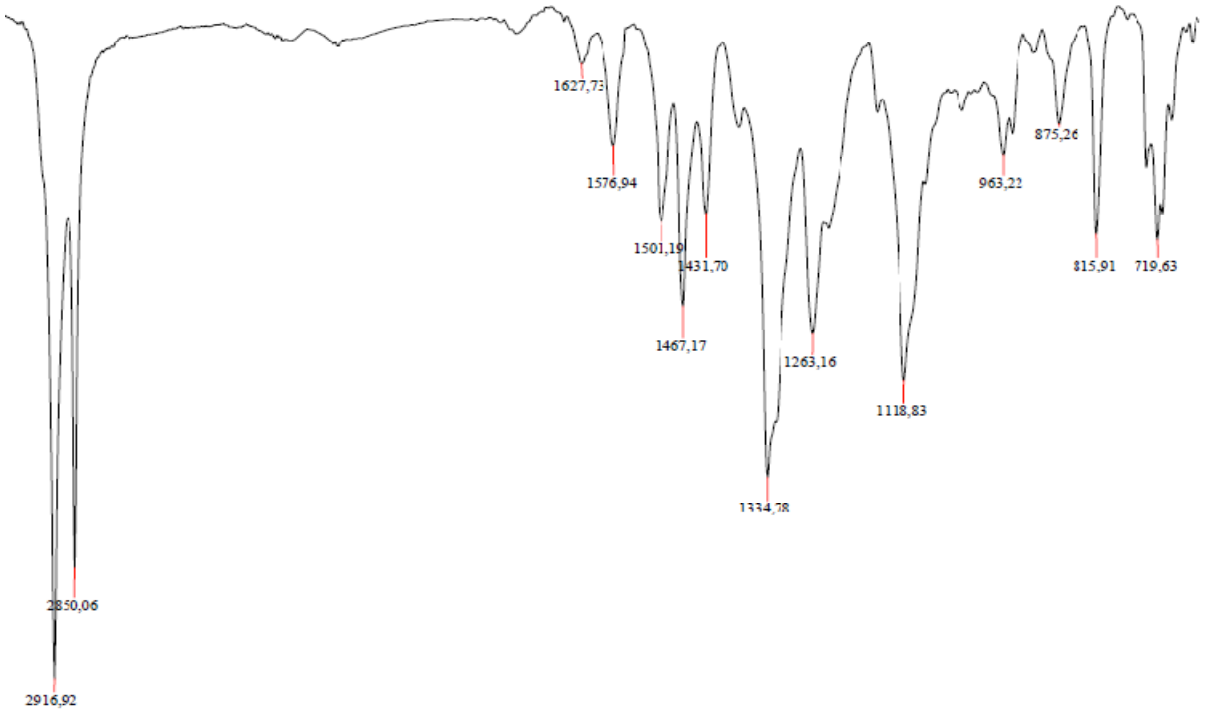


S3.- FT-IR spectra

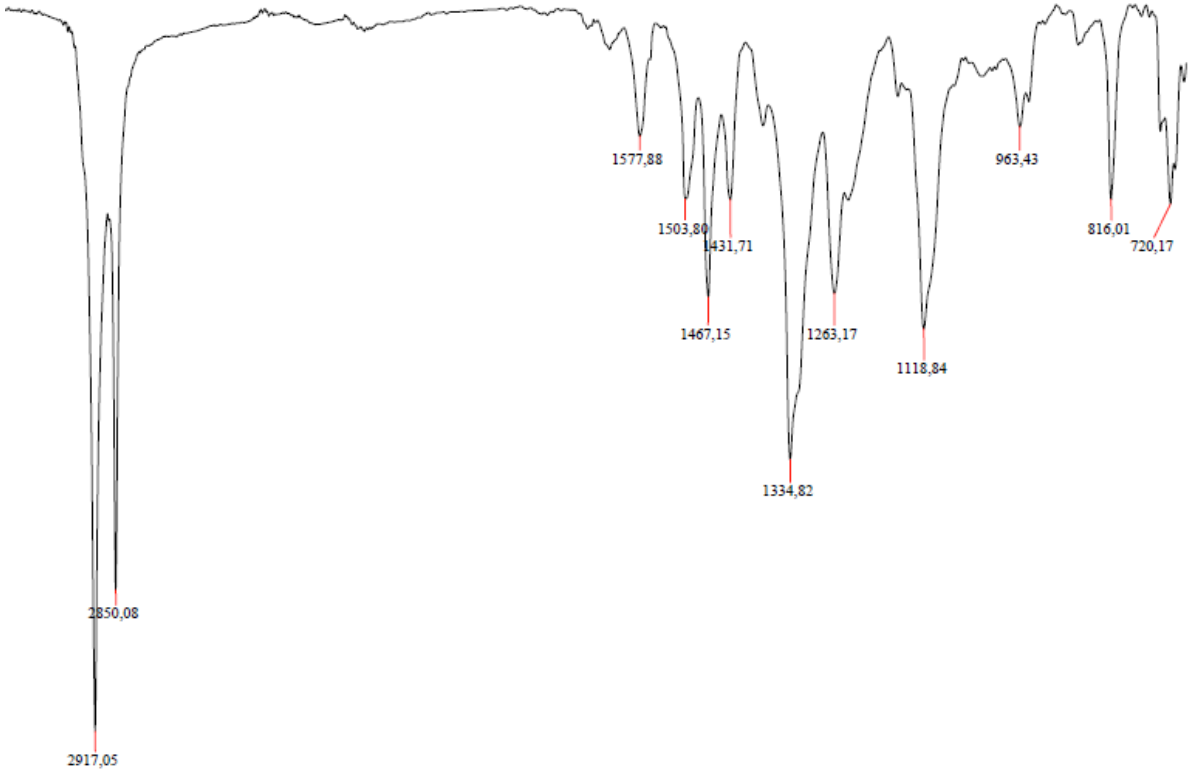
Precipitate from 1-hexanol:



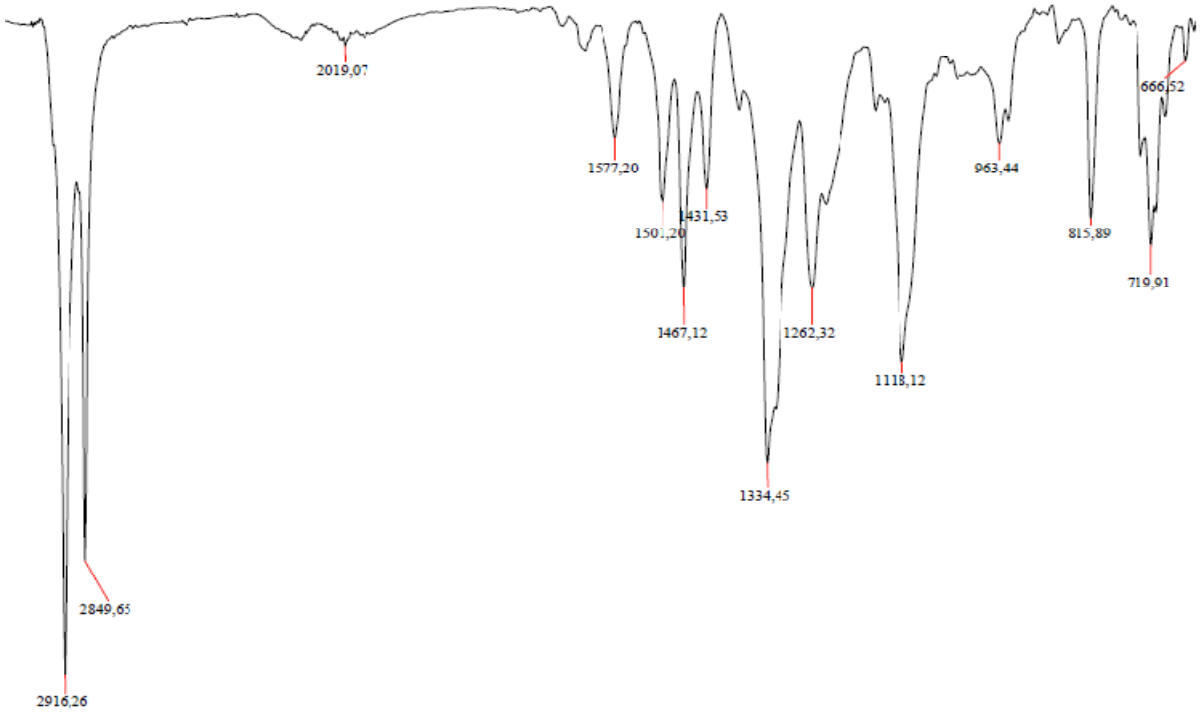
Precipitate from 1,4-dioxane:



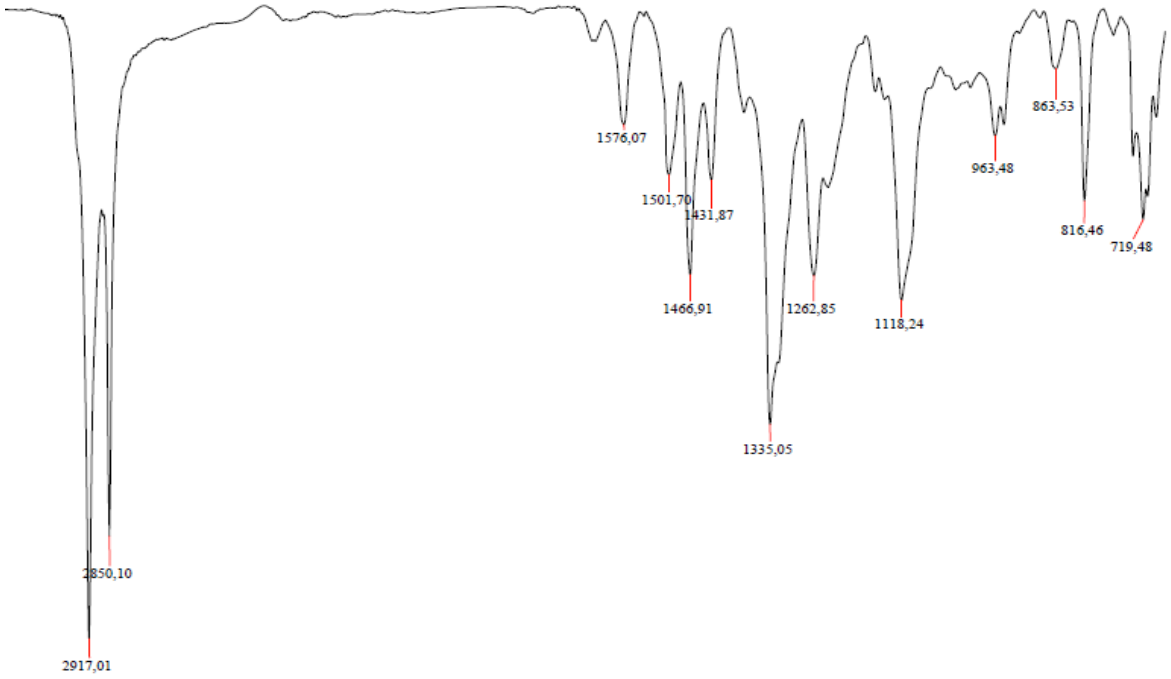
Precipitate from 1,4-dioxane/acetonitril (3/1):



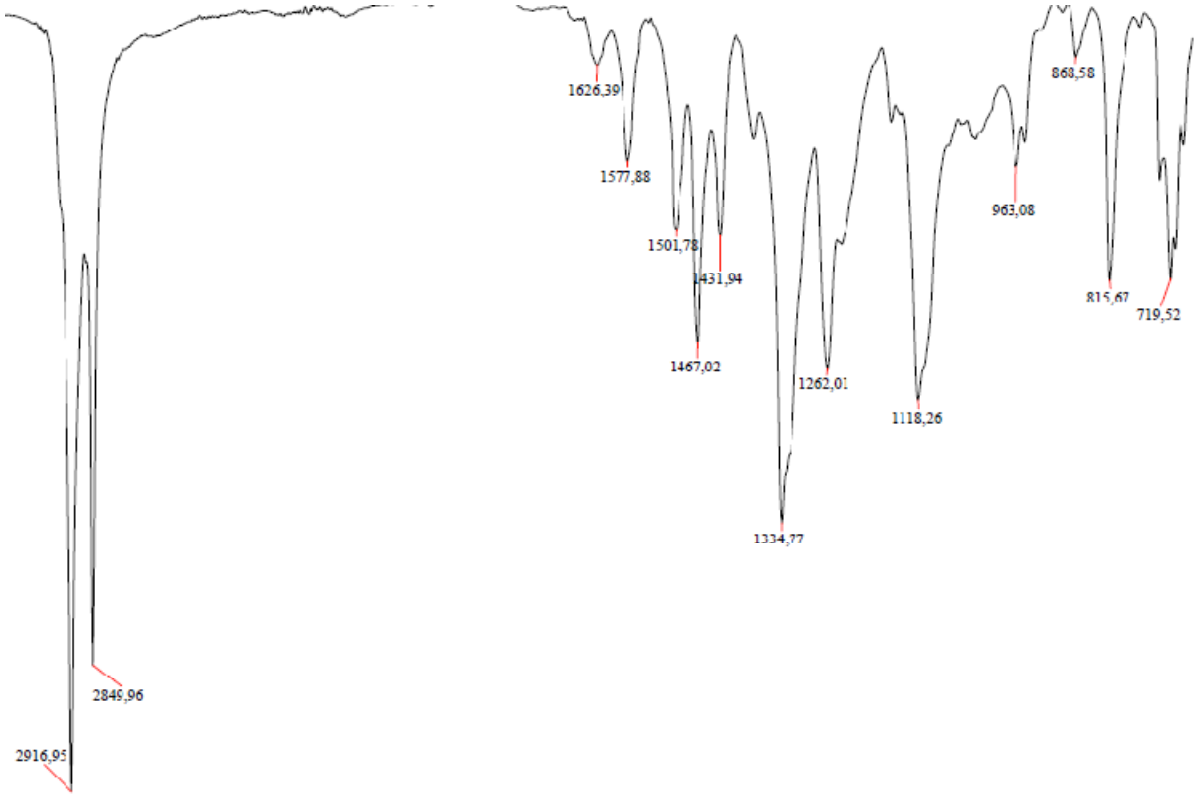
Precipitate from dioxane/methanol (3/1):



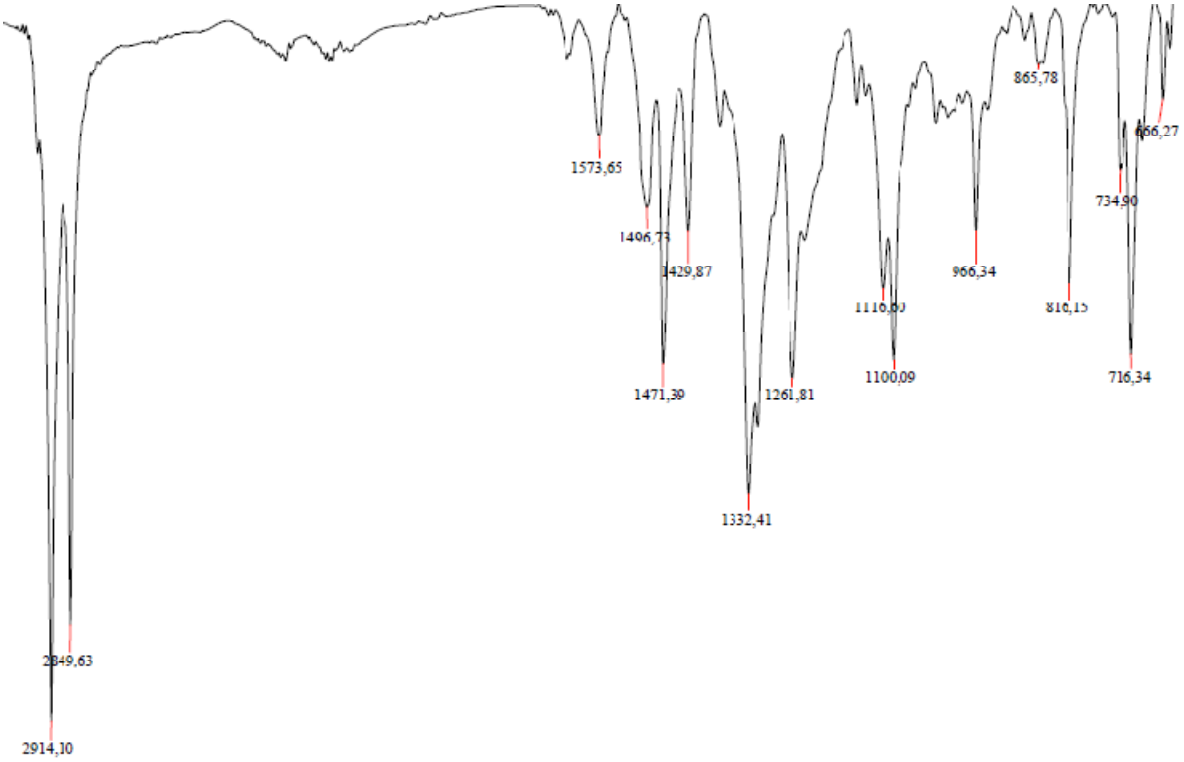
Precipitate from isopropanol:



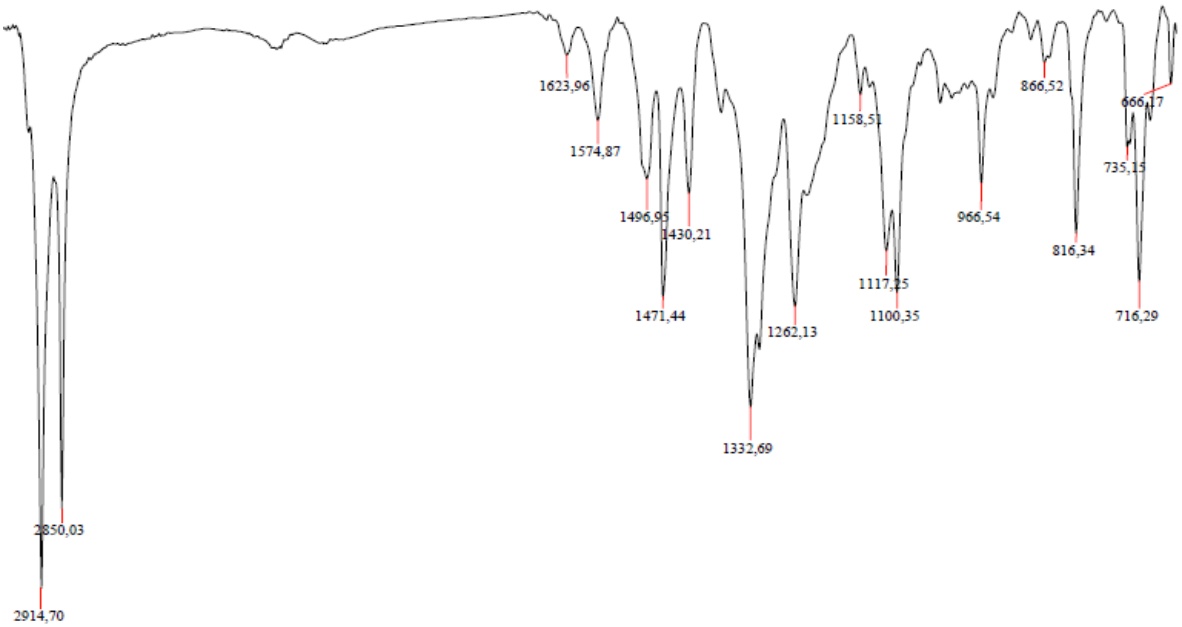
Precipitate from THF/water (2/1):



Precipitate from toluene/ethanol (1/1):



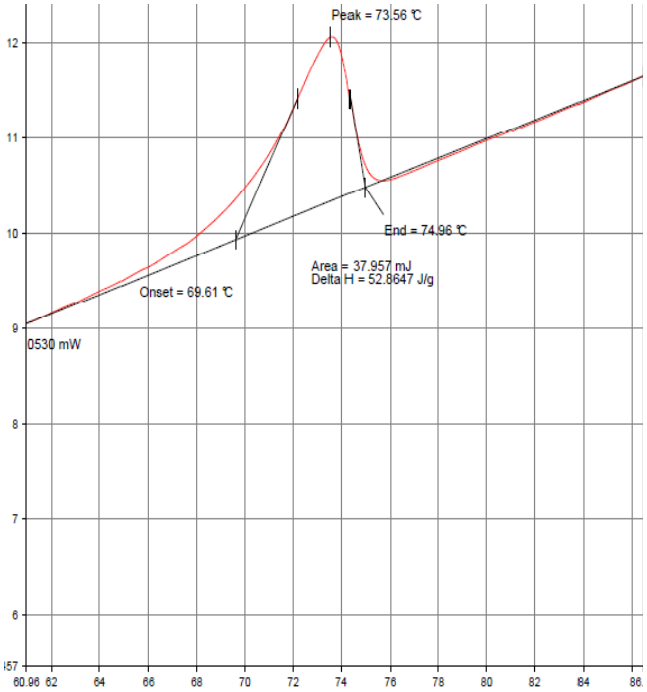
Precipitate from toluene/ethanol (1/1):



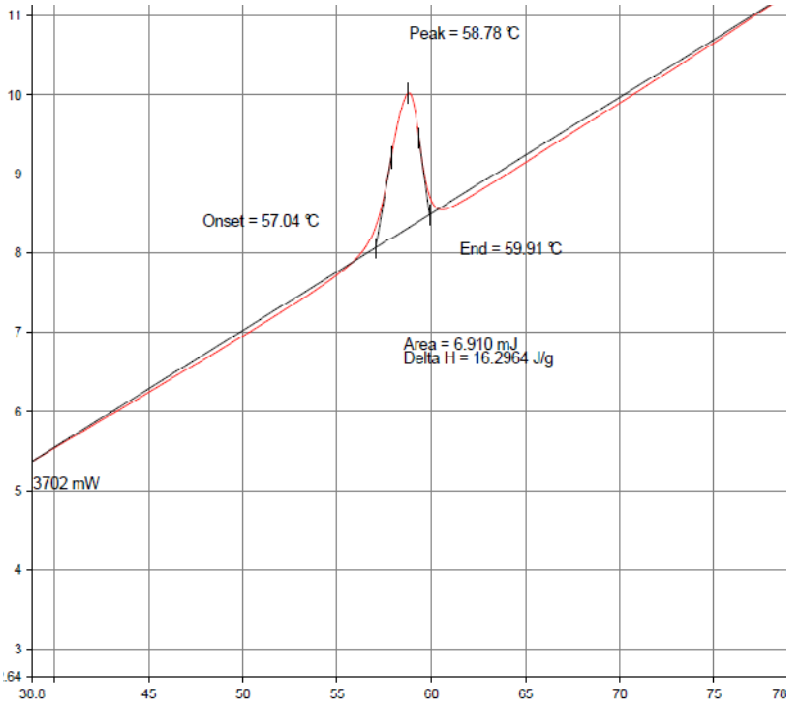
S4.- DSC thermograms

DSC thermograms were recorded using a temperature ramp of 10ºC/min starting from 25ºC. In diagrams are represented the peak observed in the first heating cycle, corresponding to the nanostructured microobjects.

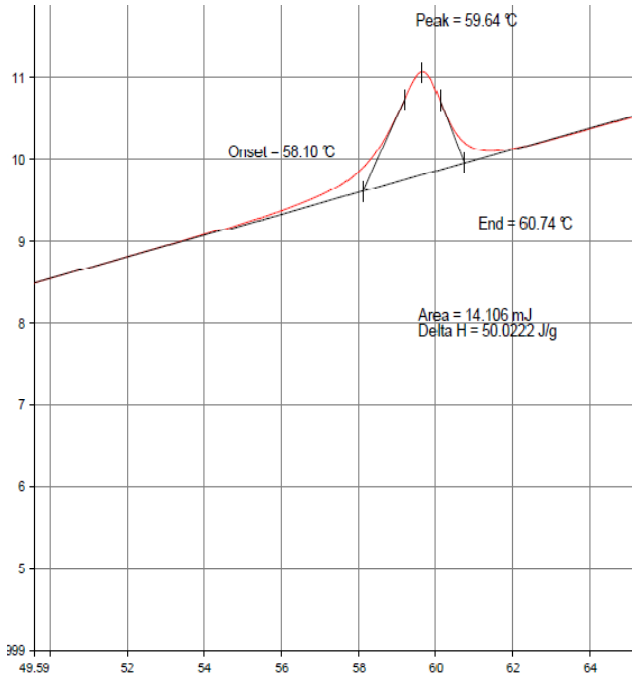
From 1-hexanol:



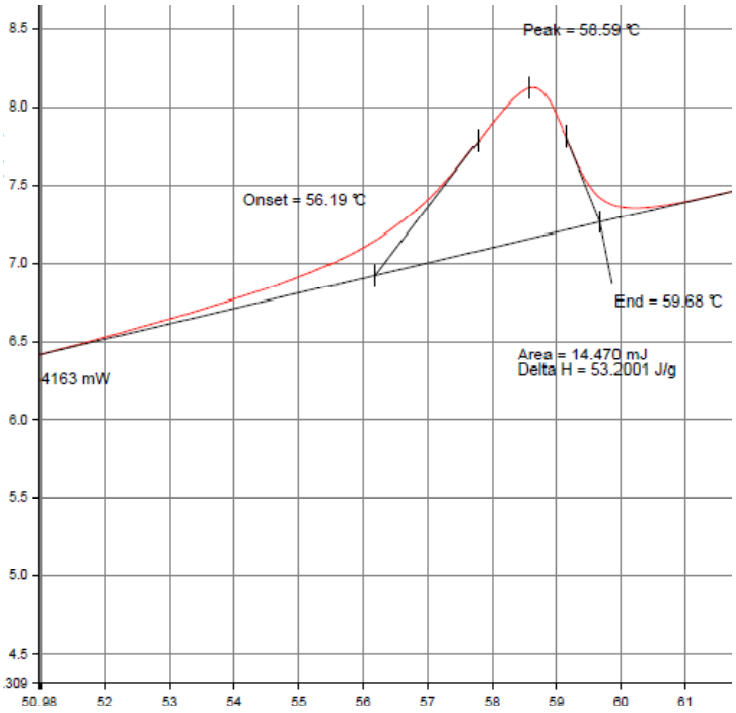
From 1,4-dioxane:



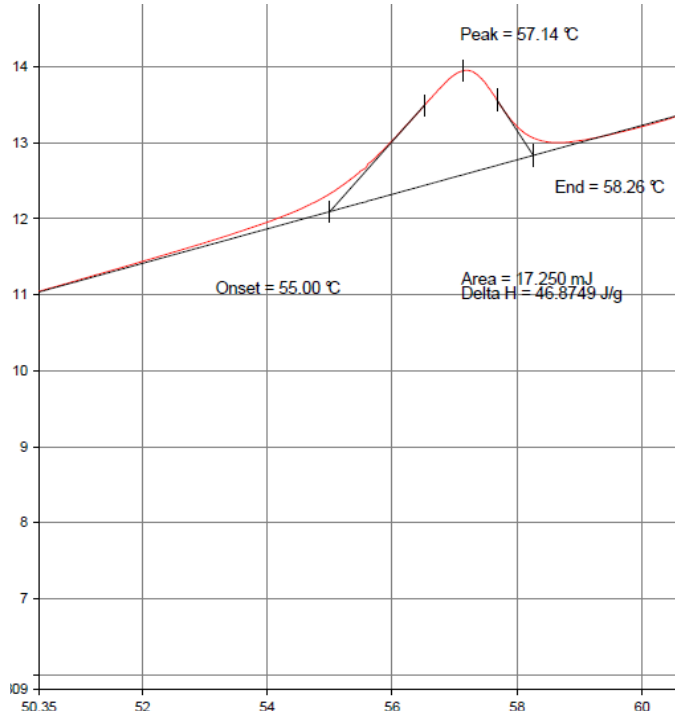
From dioxane/acetonitrile (3/1):



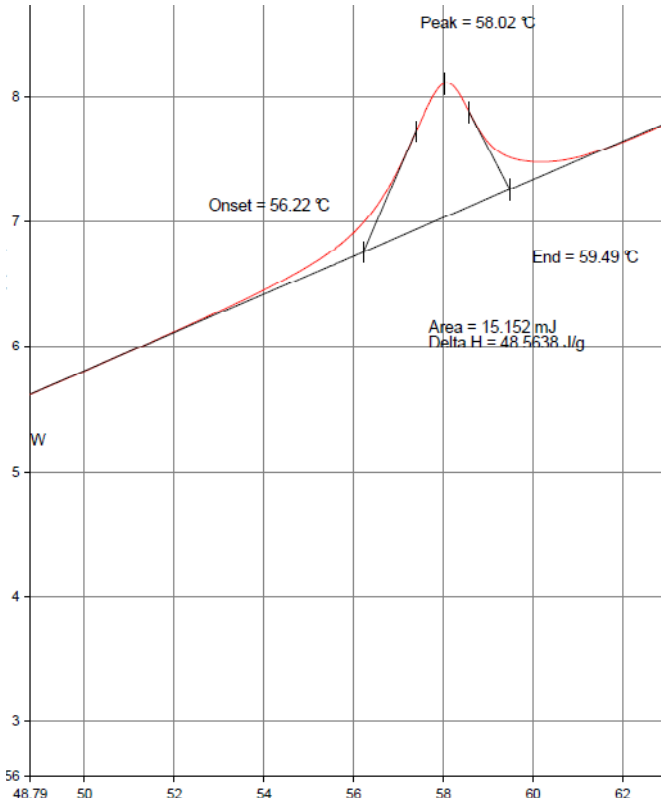
From dioxane/methanol (2/1):



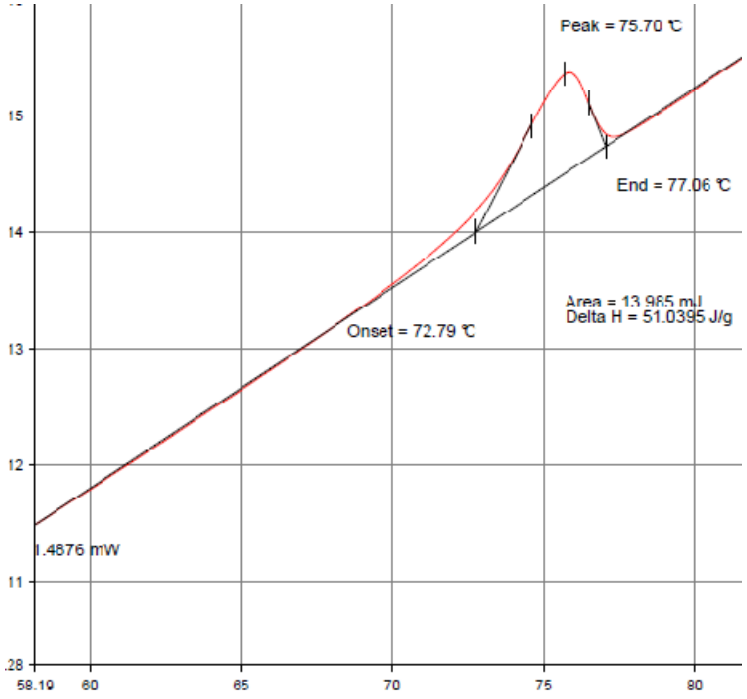
From isopropanol:



From THF/water (2/1):



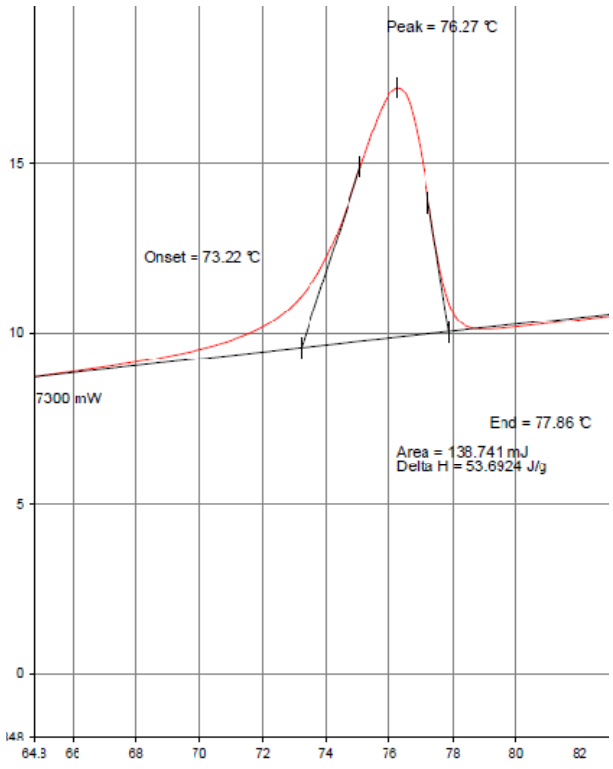
From toluene/ethanol (1/1):



heat flow (exo down, mW)

temperature (ºC)

From toluene/methanol (1/1):

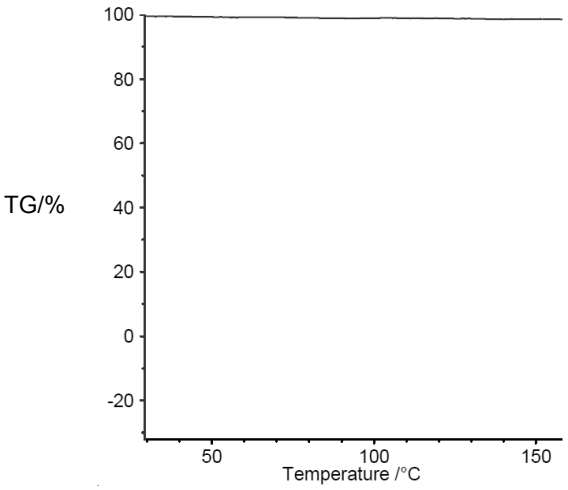


heat flow (exo down, mW)

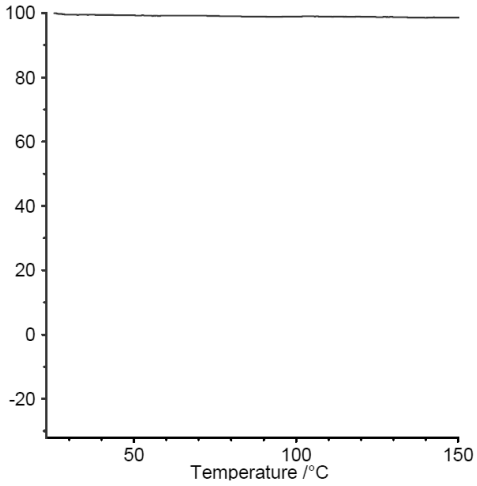
temperature (ºC)

S5.- Thermogravimetry (TG)

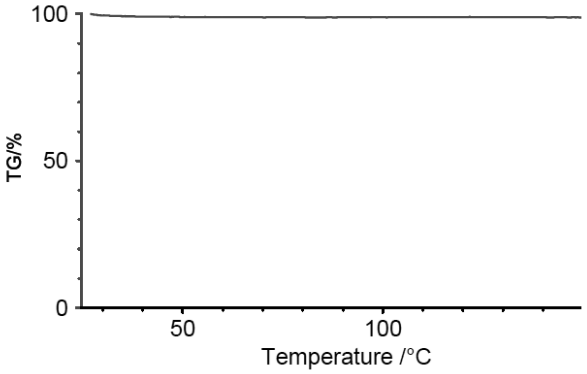
From 1-hexanol:



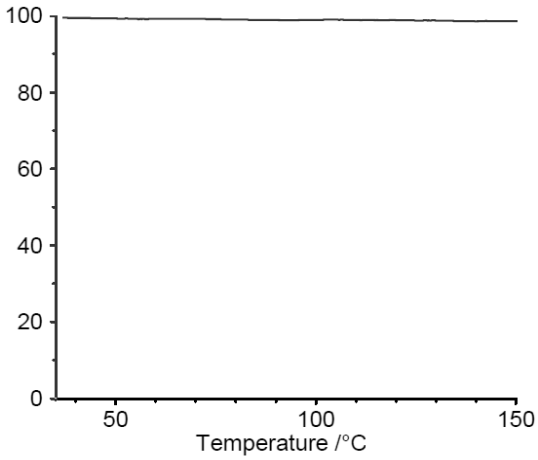
From 1,4-dioxane:



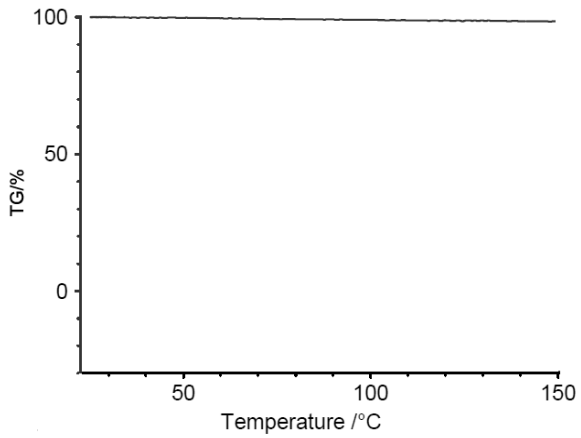
From 1,4-dioxane/acetonitrile (3/1):



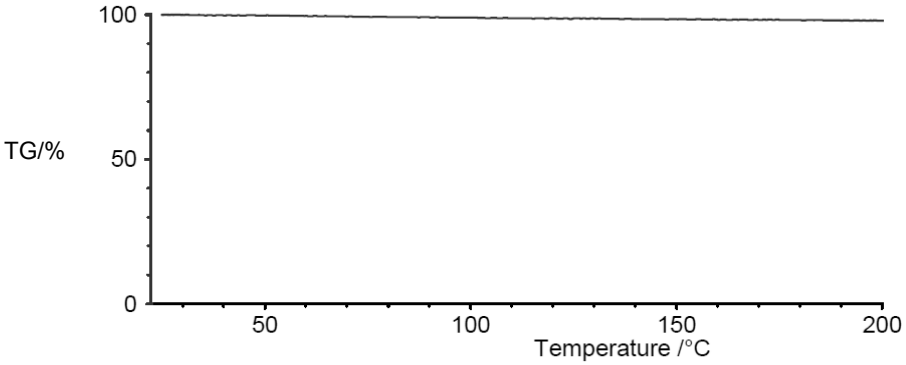
From 1,4-dioxane/methanol (2/1):



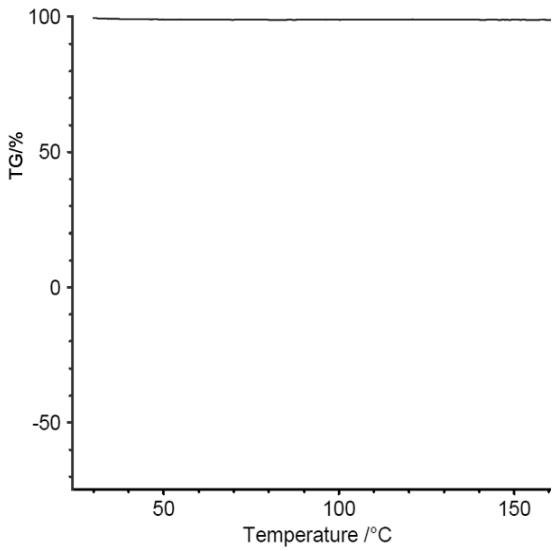
From isopropanol:



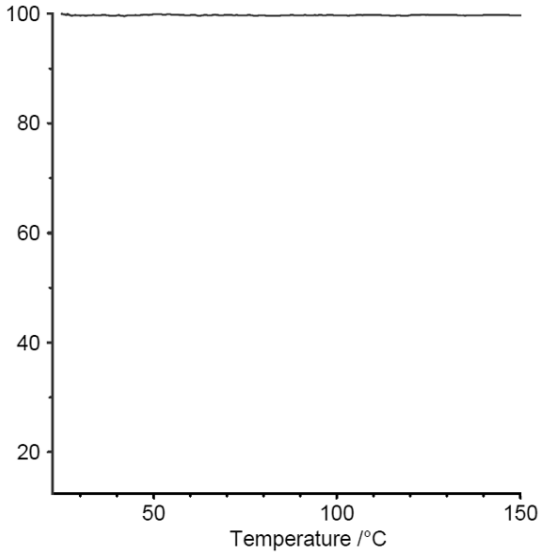
From THF/water (2/1):



From toluene/methanol:

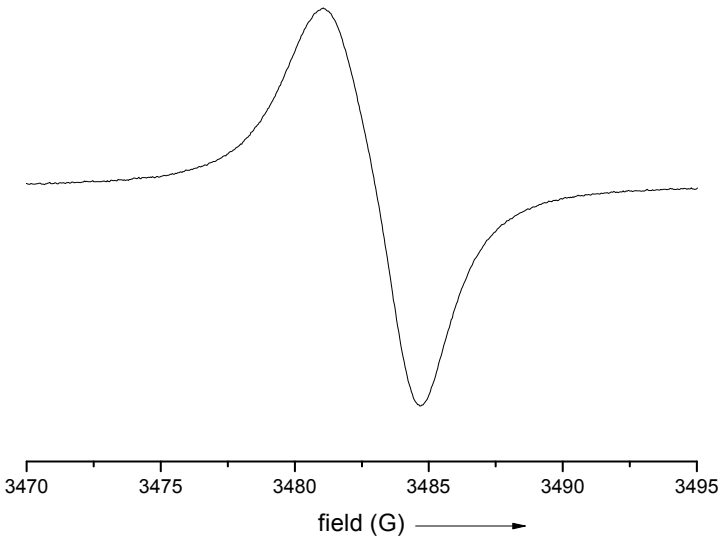


From toluene/ethanol:

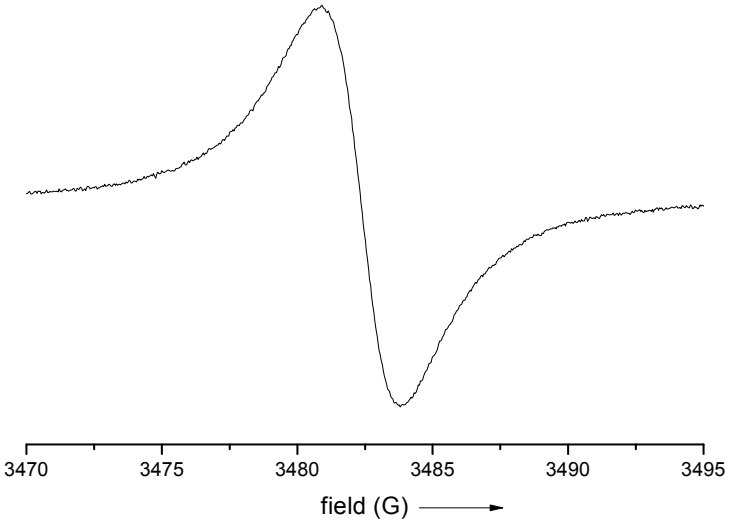


S6.- EPR spectra

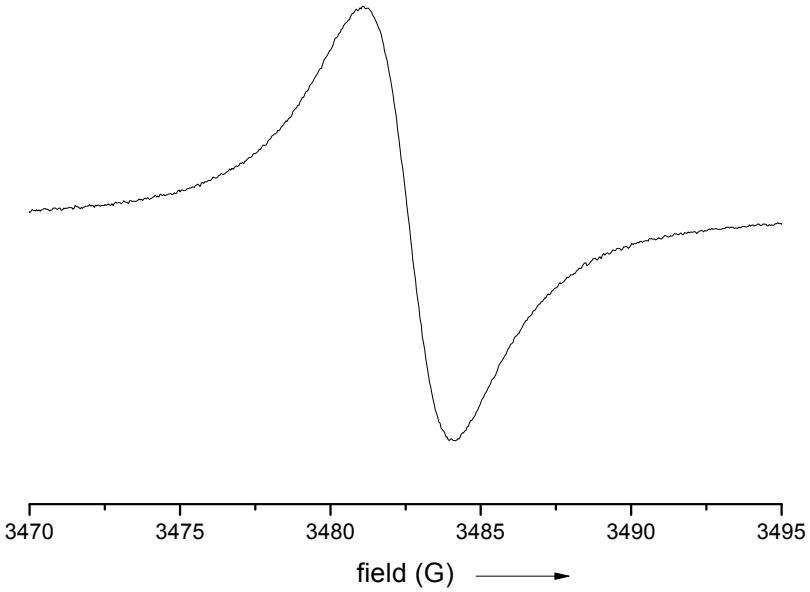
From 1-hexanol:



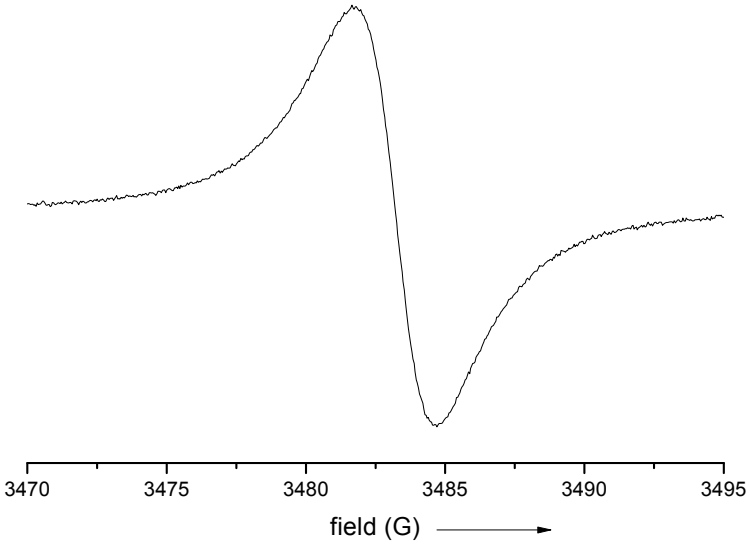
From 1,4-dioxane:



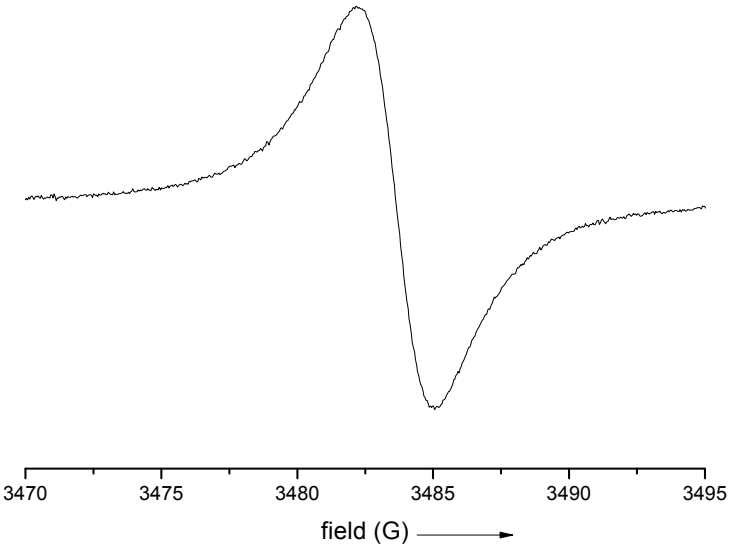
From 1,4-dioxane/acetonitrile (3/1):



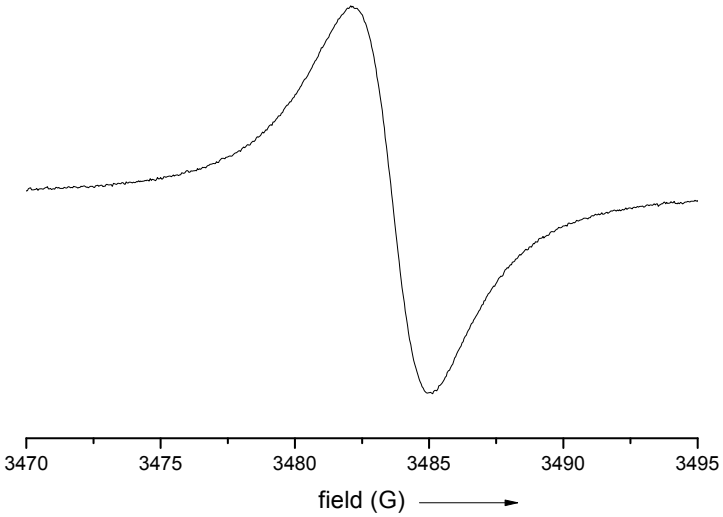
From 1,4-dioxane/methanol (2/1)



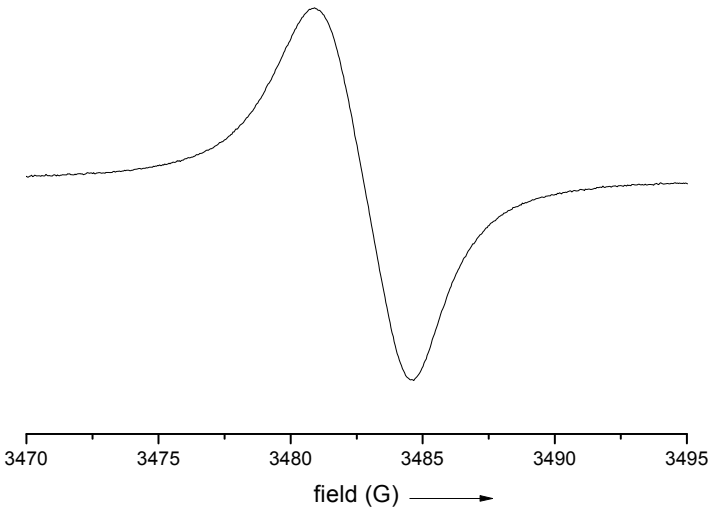
From isopropanol:



From THF/water (2/1):



From toluene/methanol:



From toluene/ethanol: