Electronic Supplementary Material (ESI) for Dalton Transactions

Supporting information

A new air-stable zinc complex based on a 1,2-phenylene-dinitrilo-2-cyanoacrylate ligand as efficient catalyst of the epoxide-CO_2 coupling

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References:

1 Bruker, Bruker AXS Inc., Madison, Wisconsin, USA, 2007; G. M. Sheldrick, *Acta Crystallogr., Sect. A: Fundam. Crystallogr. A,* 2008, **64**, 112; L. Zsolnai, XPMA, University of Heidelberg, Germany, 1996.
2 C. R. Groom and F. H. Allen, *Wiley Interdiscip. Rev.: Comput. Mol. Sci.*, 2011, **1**, 368.
3 MERCURY 3.0 (Build RC5); http://www.ccdc.cam.ac.uk/Mercury
4 H. Kristen and O. Hamann, *Pharmazie*, 1976, **31**, 222; E.-G. Jäger, *Z. anorg. allg. Chem.*, 1967, **349**, 139; E.-G. Jäger, E. Häussler, M. Rudolph and A. Schneider, *Z. anorg. Allg. Chem*., 1985, **525**, 67.
5 M. A. Fuchs, T. A. Zevaco, E. Ember, O. Walter, I. Held, E. Dinjus and M. Döring, *Dalton Trans.*, 2013,**42**, 5322.
6 M. A. Fuchs, C. Altesleben, T. A. Zevaco and E. Dinjus, *Eur. J. Inorg. Chem.,* DOI: 10.1002/ejic.201300634.
7 A. L. Singer, D. A. Atwood, *Inorg. Chim. Acta*, 1998, **277**, 157; E. C. Escudero-Adan, J. Benet-Buchholz and A. W. Kleij, *Inorg.Chem*., 2008, **47**, 256;
8 N. E. Eltayeb, S. G. Teoh, S. Chantrapromma, H.-K. Fun and R. Adnan, *Acta Crystallogr., Sect.E: Struct. Rep. Online*, 2008, **64**, m738.
9 E. C. Escudero-Adan, J. Benet-Buchholz and A.W. Kleij, *Chem.-Eur. J.*, 2009, **15,** 4233.
10 A. Decortes, M.M. Belmonte, J. Benet-Buchholz and A.W. Kleij, *Chem. Commun.*, 2010, **46**,4580.
11 E. S. Aazam, S. W. Ng and E. R. T. Tiekink, *Acta Crystallogr., Sect.E: +uct. Rep. Online*, 2011, **67**, m314.
12 R. M. Haak, A. M. Castilla, M. M. Belmonte, E. C. Escudero-Adan, J. Benet-Buchholz and A. W. Kleij, *Dalton Trans*., 2011,**40**, 3352.

Experimental Section

General: The ^{1}H- and ^{13}C-NMR were recorded operating at 399.91 and 100.56 MHz respectively, by means of a Varian Inova Unity 400 spectrometer (software VNMRJ 3.2) equipped with an Oxford Magnet (9,4T). IR spectra were obtained with a Varian 660-IR FT-IR Spectrometer. Mass spectra were measured with a Bruker ApexQe hybrid 9.4 T FT-ICR. **Gel Permeation Chromatography (GPC) was performed at a PSS SDV 5µ 1000 Å (8x300mm) and a PSS SDV 5µ 100 Å (8x300mm) column (The system is equipped with a pre-column SDV 5µ 8x50mm), all columns from the company Polymer Standard Service (PSS). The columns are temperated using a column oven T-6300 from Merck (VWR, Darmstadt). The HPLC pump used was a Lichgraph L-6200 gradient pump from Merck. The molecules were detected with a RI-Detector L-7490 (Merck). Dry tetrahydrofuran was used as eluent and toluene as an internal standard. Data processing was done with the software PSS WINGPC 6 of the company PSS (Polymer Standards Service).. The concentration of the probes was 1 g.ml-1 (10mg probe in 10ml THF+traces of toluene). The columns were operated with a constant flow rate of 1 ml.min-1 at a temperature of 35 °C. The calibration was done using polystyrene standards from PSS (MW: 54000-24000-18100-8390-4700-3420-1920-1100-376-162).** For the determination of the glass transition temperature of the polycarbonates Differential Scanning Calorimetry was applied using a Mettler Toledo DSC822^e. For Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES) a 720/725-ES emission spectrometer with CCD detector from Agilent Technologies was used. The plasma was generated with a 40 MHz quartz-controlled generator where argon is the carrier gas. Elementary analysis was performed with a CHNS-Analyser from the company Elementar. UV-Vis spectra were measured with an MCS 501 UV-NIR (Zeiss). The X-ray analyses were performed on a Bruker Apex II Quazar diffractometer. 2124 frames were collected with an irradiation time of 1 s per frame. Integration of the data proceeded with SAINT, the data were corrected for Lorentz- and polarisation effects, and an experimental absorption correction with sadabs was performed. [1] For searches relating to single-crystal X-ray diffraction data, the Cambridge Structural Database was used. [2] Crystallographic data have been deposited at the Cambridge Crystallographic Database Centre (CCDC), supplementary publications No. CCDC 954796. Figures were prepared with the appropriate software of the CCDC, MERCURY 3.0 (Build RC5) for Windows. [3] For a description of the structure see below.

The starting materials (*o*-phenylene diamine, ethyl (ethoxymethylene)cyanoacetate, methanol, dry tetrahydrofuran, epoxides, diethyl zinc solution (1M in hexane) and the cocatalysts) were purchased from Sigma Aldrich. Except for the epoxides all of them were used without further purification. The epoxides were distilled from CaH_2 and stored under an argon atmosphere before using them in the catalytic tests. The synthesis of ligand 1 is performed according to the procedure reported firstly by H. Kristen and optimised by the group of E.G. Jäger. [4] In the synthesis and the further utilization of the zinc complex **2** standard Schlenk techniques were applied; complex **2** was stored under inert atmosphere.

Synthesis of 2: Under an argon atmosphere ligand **1** (5.00 g, 14.1 mmol) is suspended in THF (75 ml). Then the diethyl zinc solution (1M in hexane) (14.1 ml, 14.1 mmol) is added under stirring. After a few minutes the white suspension changes to a deep yellow solution. The reaction mixture is stirred overnight and then the solvent is removed under reduced pressure leaving **2** as a deep yellow solid (5.88 g, 14.1mmol, 99.8%).

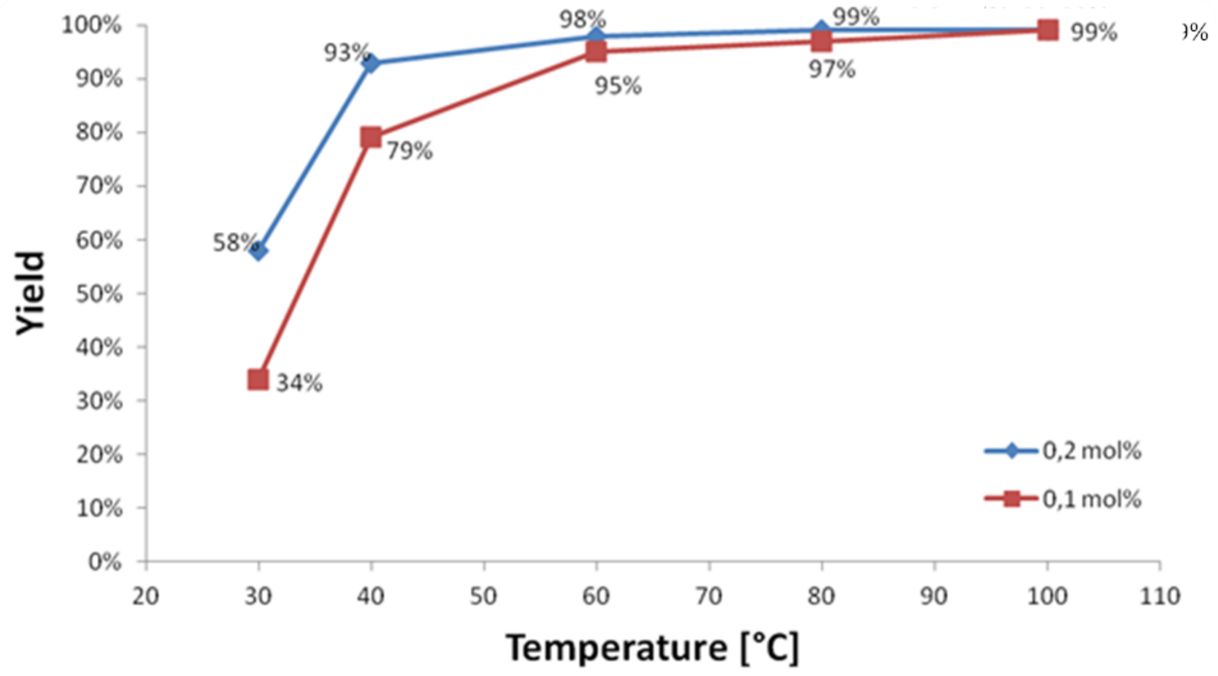
^{1}H NMR (400 MHz, DMSO-d$_6$): 1.29 (tr, $^3J_{HH}$=7.1, 6H, CH_3), 4.25 (q, $^3J_{HH}$=7.0, 4H, CH_2), 7.05 (dd, $^3J_{HH}$=3.4, $^4J_{HH}$=6.1, 2H, CH_{aromat}), 7.56 (dd, $^3J_{HH}$=3.5, $^4J_{HH}$=6.1, 2H, CH_{aromat}), 8.35 (s, 6H, CH); ^{13}C NMR (100 MHz, DMSO-d$_6$): 14.13 (**C**H_3), 60.24 (**C**H_2), 68.05 (=**C**(CN)(C=O)), 114.89 (**C**H_{aromat}), 121.19 (-**C**≡N), 124.39 (**C**H_{aromat}), 138.04 (**C**$_{aromat}$), 156.27 (NH-**C**H=), 170.77 (**C**=O). MS: (ESI+) m/z (%): 417 (100) [M], 418 (23) $[M+H]^+$, 835 (15) $[2M+H]^+$. IR (KBr): 2203 cm^{-1}, ν(C≡N); 1625 cm^{-1}, $\nu_{as}(C=O)_{free}$ and ν(C=N); 1023cm^{-1}, ν(C-O); 748 cm^{-1}, δ (C-H). Anal. calcd. for C18H16N4O4Zn: C, 51.75; H, 3,86; N, 13.41, found: C, 51.60; H, 4.05; N, 13.47. ICP-OES: Anal. calcd. for C18H16N4O4Zn: Zn, 15.65, found: Zn, 15,62. UV/VIS (CH_2Cl_2): λmax (ε) = 241 (16522), 307 (27550), 360 nm (23931 mol^{-1} dm^3 cm^{-1}).

Catalytic Screening tests: The tests were performed in a screening test-bench consisting of 70 ml autoclaves (mat. 1.4571 (SS 316Ti), 70 ml, p_{max} 200 bar, T_{max} 250 °C) equipped with magnetic stirrers and p,T-Acquisition via multi-meters (Agilent 34970A Data Acquisition/Data Logger Switch Unit + 34901A 20 Channel Multiplexer) and PCs. The heating was simply performed with an aluminium heating block controlled via the electronic contact thermometer of the magnetic stirrers (temp. difference between outside inside ± 2°C). The autoclaves were filled with the catalyst/cocatalyst and dried under vacuum at 100°C for 1 h. Afterwards the epoxide was added under argon via one of the autoclave's ports and the reaction mixture was pressurized with CO_2. The autoclave was cooled to 0 °C during the CO_2 loading ensuring that enough CO_2 was available for the reaction (CO_2/epoxide molar ratio around 2). Once the autoclave was heated, pressure and temperature were recorded every 30 seconds during the whole reaction time. After the reaction the autoclaves were cooled down, slowly vented in a fume hood, opened and the products collected. The analysis was done via ^{1}H-NMR and FT-IR spectroscopy. The reproducibility of a typical reaction was assessed via repeating tests and a good reproducibility was found.

Low pressure (2 bar) experiments were carried out in a 70 ml autoclave connected during the course of the reaction to the gas cylinder via Swagelock fittings and a backstop valve. The autoclave is operated the same way as for the high pressure experiments: after filling the autoclave with the catalyst/cocatalyst mixture the autoclave is dried at 100°C under vacuum. After adding propylene oxide under an argon atmosphere, the autoclave is pressurized with 2 bar CO_2 and heated for the same reaction time. Afterwards the products were analyzed by ^{1}H-NMR spectroscopy and IR.

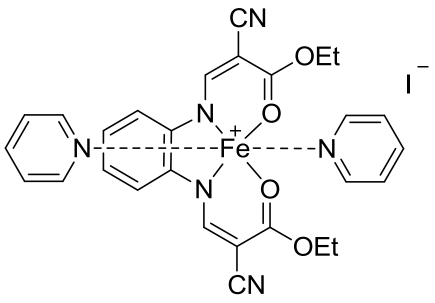
Comparison of the yield in the temperature screening with two different catalyst concentrations:

Reaction conditions: 50 bar CO_2, c (**2** = Bu_4NI) = 0.2 mol%, 20 h.

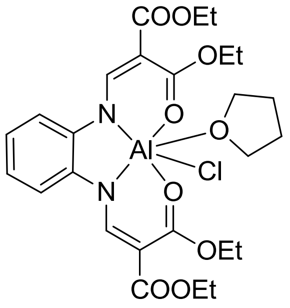


Other N_2O_2-metal complexes for the comparison with the zinc system:

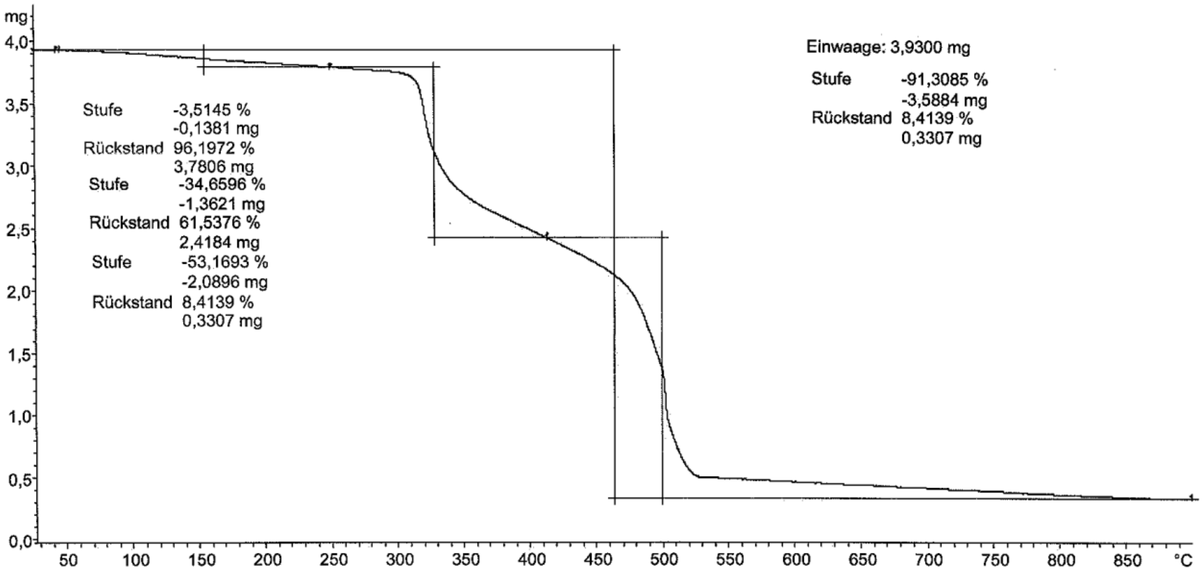
Fe-N_2O_2[5]:



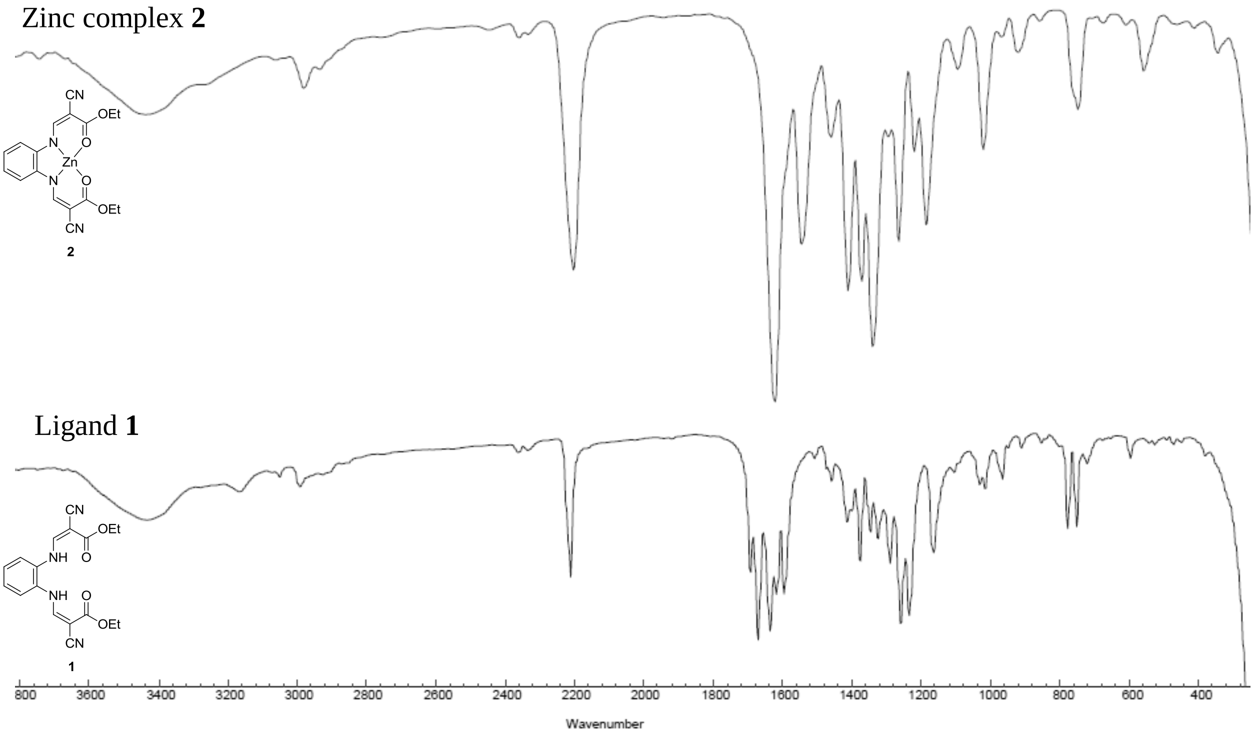
Al-N_2O_2[6]:



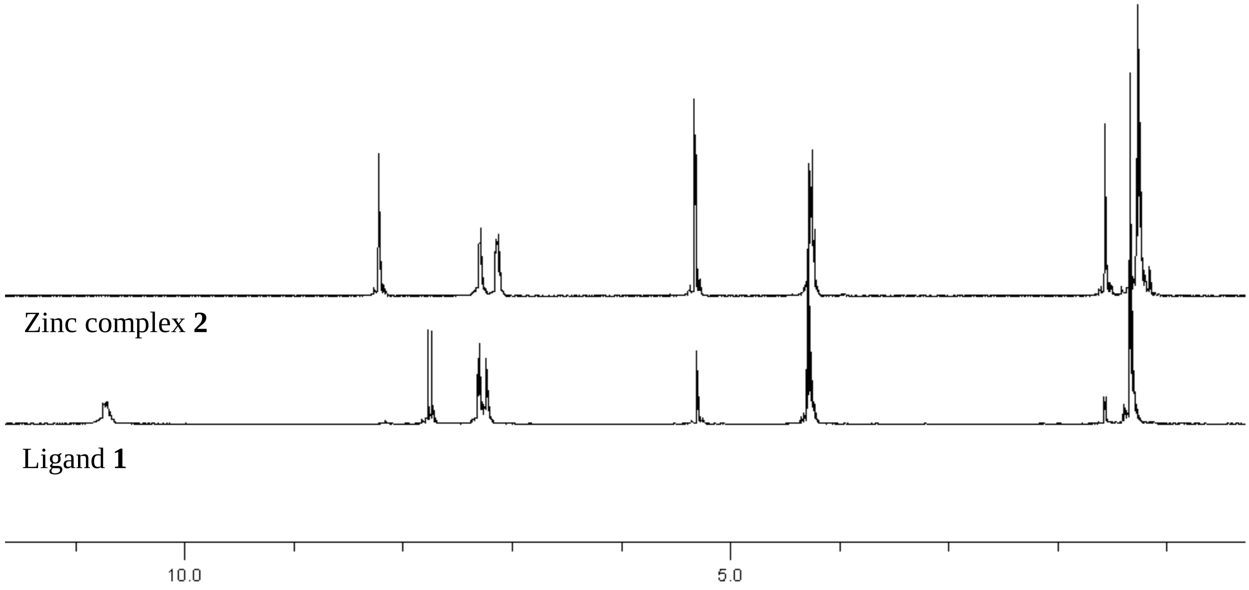
TGA of zinc complex 2:



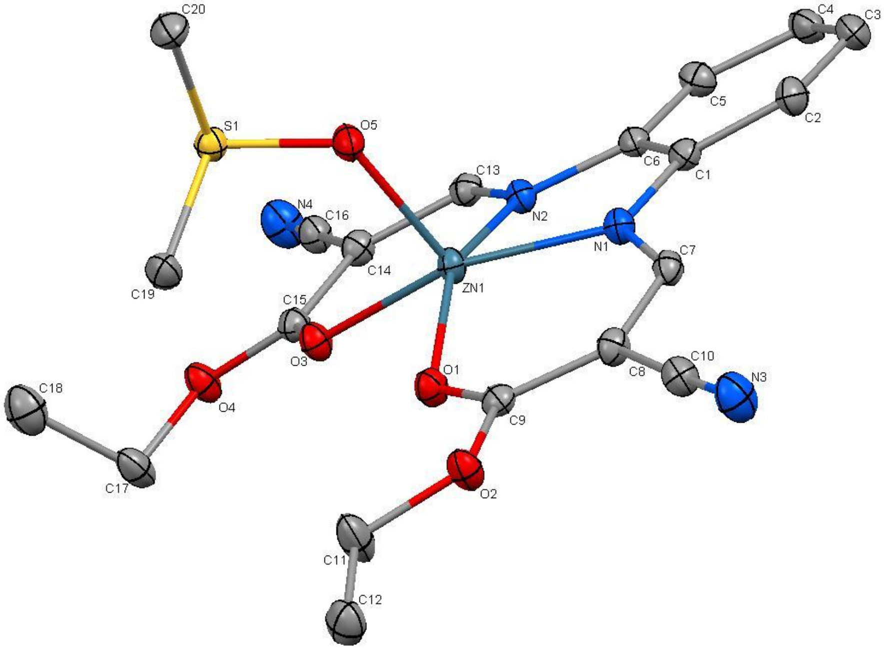
Comparison of the FT-IR spectra of ligand 1 and zinc complex 2:

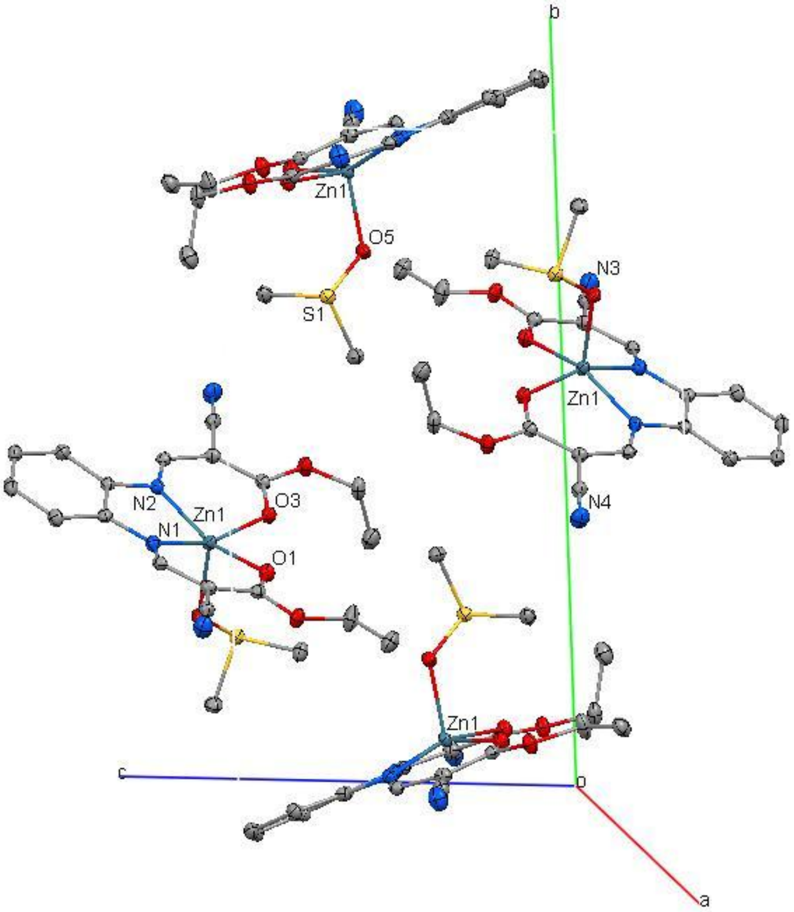


Comparison of the ^{1}H-NMR spectra (in CD_2Cl_2) of ligand 1 and zinc complex 2:



crystal structure of zinc complex 2: Figures





Crystal structure of zinc complex 2: Description

Single crystals suitable for a crystal structure analysis could be isolated from a concentrated DMSO solution after a couple of days at room temperature (crystallographic data of the structure have been deposited at the Cambridge Crystallographic Database Centre –CCDC-; supplementary publication No CCDC 954796). The structural data shows that complex **2** displays a tetragonal pyramidal coordination geometry around the metal centre, with one oxygen-bound dimethyl sulfoxide molecule in the axial position and the N_2O_2–ligand occupying the equatorial plane. This structure is the first one involving zinc and a N_2O_2 ligand with amine and ester functions, while the bulk of the other documented Zn/N_2O_2+L structures involve the more common salen/salophen- ligands (with phenol and aldimine functionalities). [7]
The overall tetragonal pyramidal structure of 2 is however comparable to the structure patterns found in the symmetrical zinc salophen/acetone [8], or in the more distorted zinc salophen/methanol [9] and zinc salophen/cyclohexene oxide [10] complexes recently reported in the literature.
The structure is slightly distorted from the ideal geometry (maximal distortion from the mean N_2O_2-plane: 0.04 Å, distance of Zn from this mean plane 0.367 Å), the N_2O_2-ligand being almost planar as found in the iron N_2O_2-complex [5] and in the zinc salophen/acetone reported by H.K. Fun and co-workers. [8]

The bond lengths found within the N_2O_2 coordination sphere are within a small range, the Zn–O bonds being a tad shorter than the Zn–N ones: Zn(1)–N(1) and Zn (1)–N(2) with 2.034(11) Å and 2.037(11) Å while Zn(1)–O(1) and Zn(1)–O(3) amount to 2.046(10) Å and 2.060(10) Å respectively. The Zn–O_{DMSO} bond is, with Zn(1)–O(5) at 2.025(10) Å, shorter than the equatorial ones and, interestingly, also shorter than those found in related DMSO-tetragonal pyramidal zinc-“salen” complexes displaying e.g. a salen with dicyano-ethylene diamino linker (Zn–O_{DMSO}: 2.047 Å [11]) and one with a 1,2-diamino cyclohexyl linker (Zn–O_{DMSO}: 2.057 Å [12]) complexes. The four (N,O)–Zn angles measured within the plane showed well balanced features, the angle O(3)– Zn–O(1) being the widest with 92.06(4)°. The angles involving zinc and DMSO range from 92.76(4) to 110.40(4)° with smaller Zn-O angles as a consequence of the slightly distorted tetragonal pyramidal geometry.

References:

1 Bruker, Bruker AXS Inc., Madison, Wisconsin, USA, 2007; G. M. Sheldrick, *Acta Crystallogr., Sect. A: Fundam. Crystallogr. A,* 2008, **64**, 112; L. Zsolnai, XPMA, University of Heidelberg, Germany, 1996.
2 C. R. Groom and F. H. Allen, *Wiley Interdiscip. Rev.: Comput. Mol. Sci.,* 2011, **1**, 368.
3 MERCURY 3.0 (Build RC5); http://www.ccdc.cam.ac.uk/Mercury
4 H. Kristen and O. Hamann, *Pharmazie*, 1976, **31**, 222; E.-G. Jäger, *Z. anorg. allg. Chem.*, 1967, **349**, 139; E.-G. Jäger, E. Häussler, M. Rudolph and A. Schneider, *Z. anorg. Allg. Chem*., 1985, **525**, 67.
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10 A. Decortes, M.M. Belmonte, J. Benet-Buchholz and A.W. Kleij, *Chem. Commun.*, 2010, **46**,4580.
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12 R. M. Haak, A. M. Castilla, M. M. Belmonte, E. C. Escudero-Adan, J. Benet-Buchholz and A. W. Kleij, *Dalton Trans*., 2011,**40**, 3352.

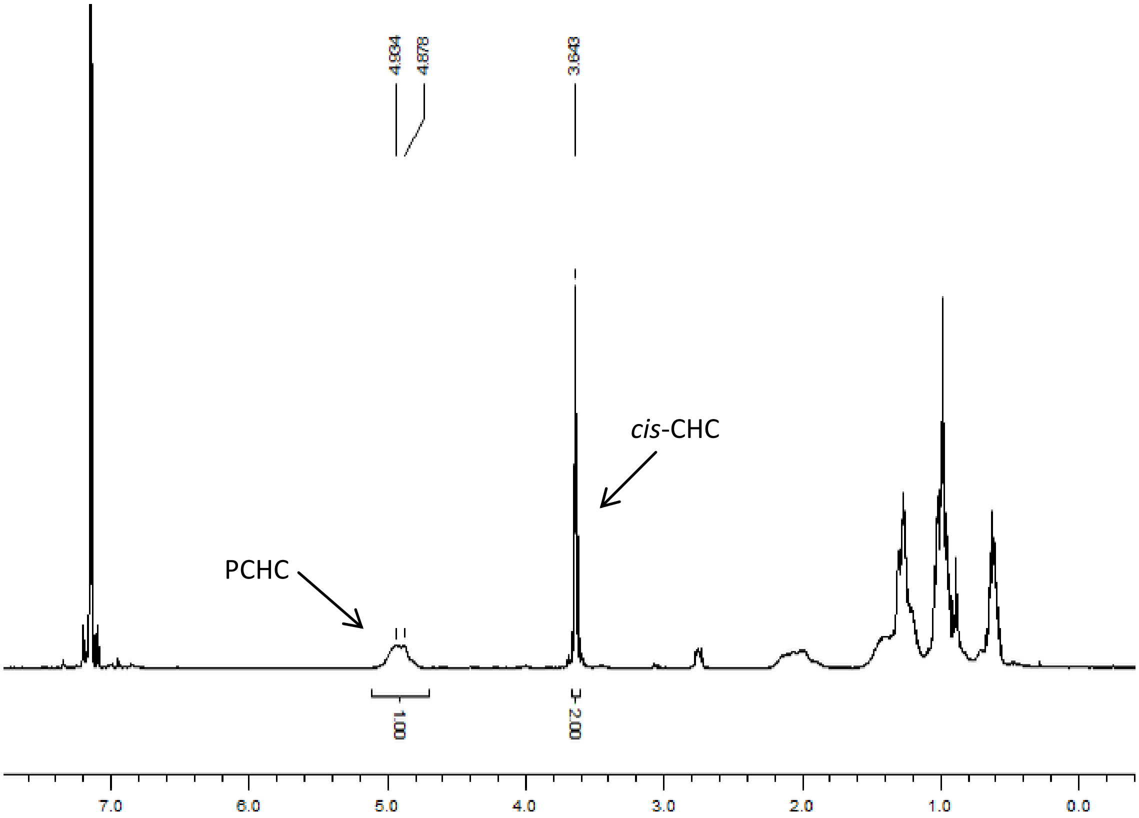
Table 1. Crystal data and structure refinement for complex 2.

Empirical formula	$C_{20} H_{22} N_4 O_5 S Zn$	
Formula weight	495.85	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	monoclinic, P2(1)/c (no. 14)	
Unit cell dimensions	a = 10.5413(6) Å	alpha = 90 deg.
	b = 19.7935(10) Å	beta = 116.6470(10) deg.
	c = 11.4038(6) Å	gamma = 90 deg.
Volume	2126.7(2) $Å^3$	
Z, Calculated density	4, 1.549 Mg/m^3	
Absorption coefficient	1.293 mm^{-1}	
F(000)	1024	
Crystal size	0.13 x 0.11 x 0.05 mm	
Theta range for data collection	2.06 to 28.57 deg.	
Limiting indices	-13<=h<=13, -26<=k<=26, -14<=l<=14	
Reflections collected / unique	38754 / 5093 [R(int) = 0.0293]	
Completeness to theta = 25.00	99.90%	
Max. and min. transmission	0.9323 and 0.8488	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5093 / 0 / 289	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0234, wR2 = 0.0593	
R indices (all data)	R1 = 0.0275, wR2 = 0.0612	
Largest diff. peak and hole	0.364 and -0.244 e.$Å^{-3}$	

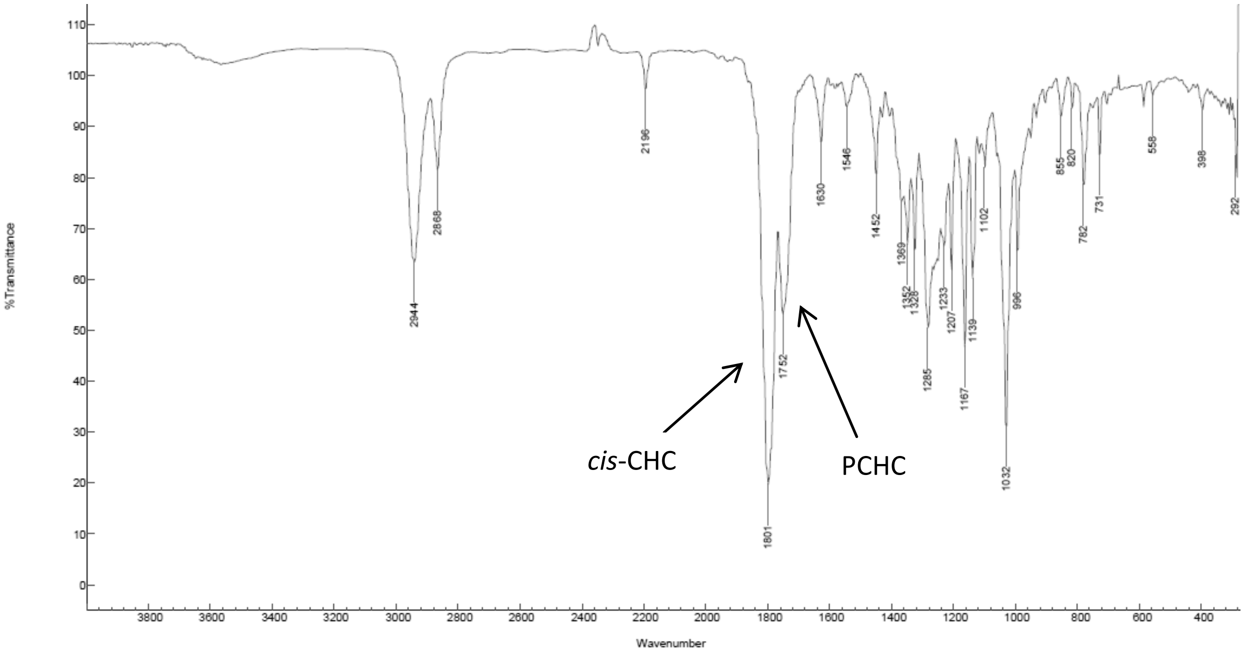
Product analysis from the reaction of CHO with CO_2:

a) Mixture of *cis*-CHC and PCHC (80°C, 50 bar, c(**2**=Bu_4NI)=1.0 mol%, 20h, Table 2, Entry 2):

^{1}H NMR (benzene-d_6):

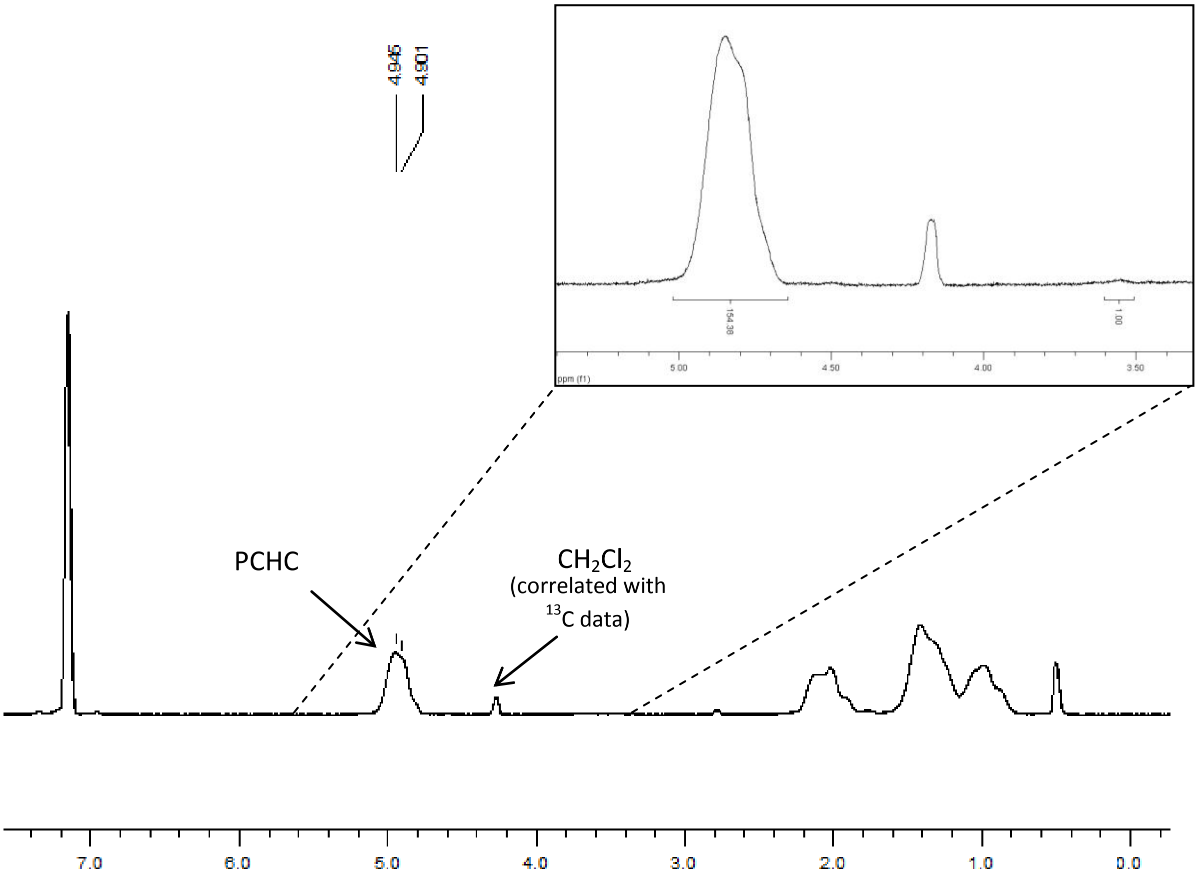


IR (KBr):

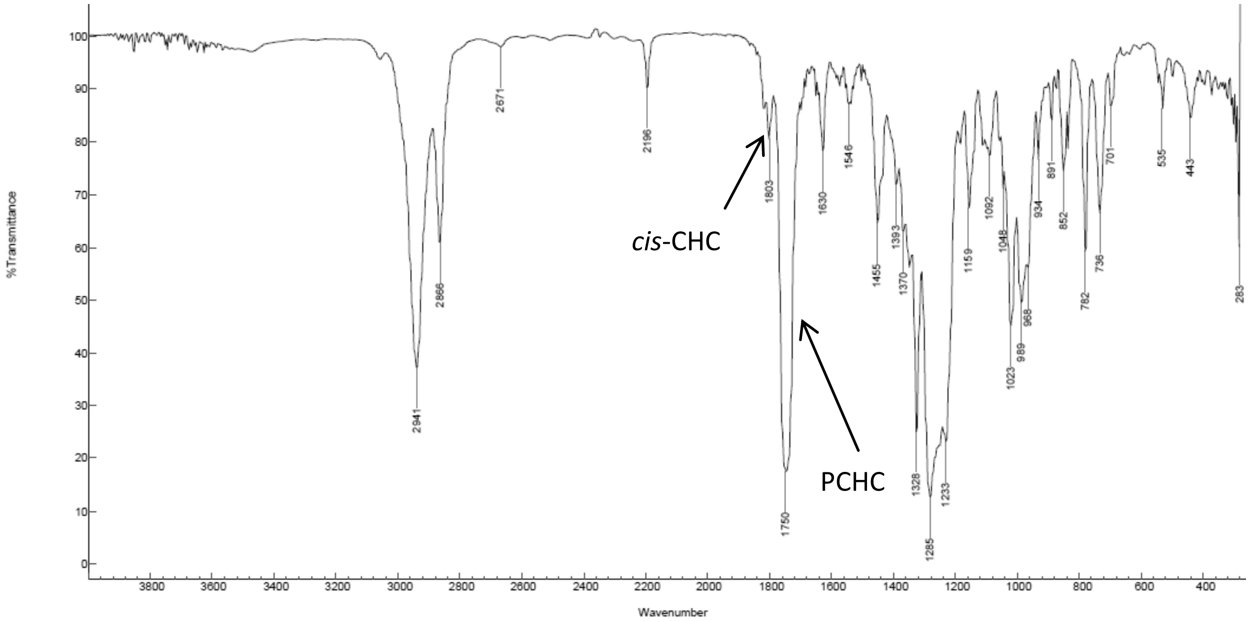


b) PCHC (80°C, 50 bar, c(**2**=PPNCl)=1.0 mol%, 20h, Table 2, Entry 5):

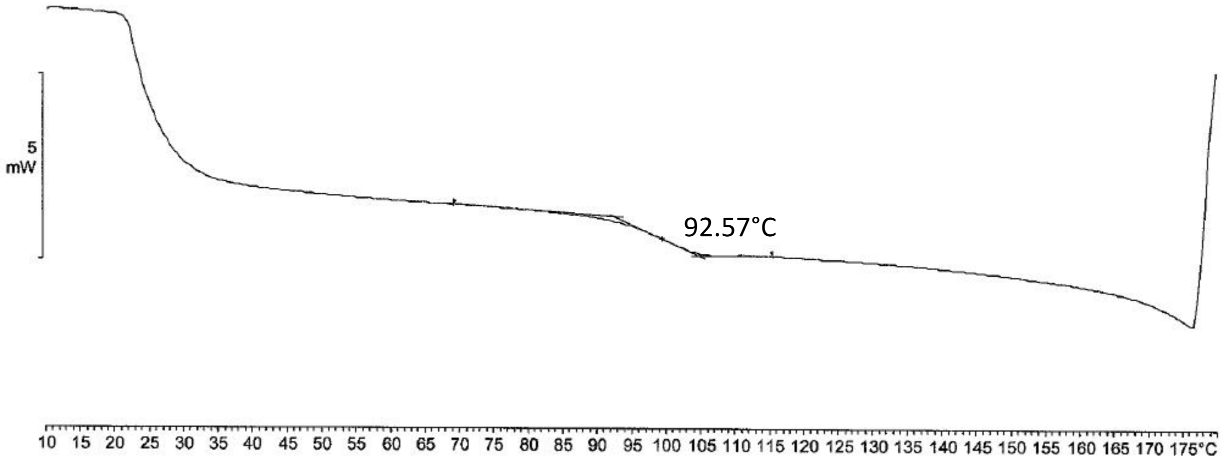
^{1}H NMR (benzene-d$_6$):



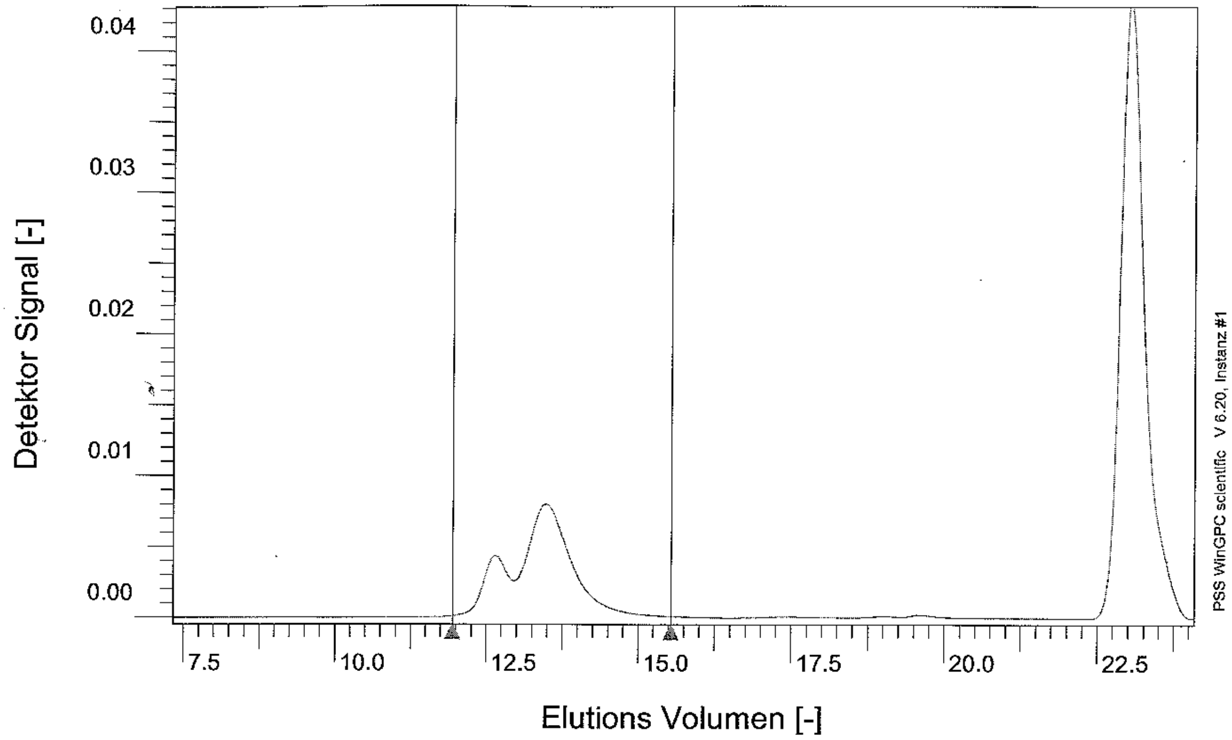
IR (KBr):

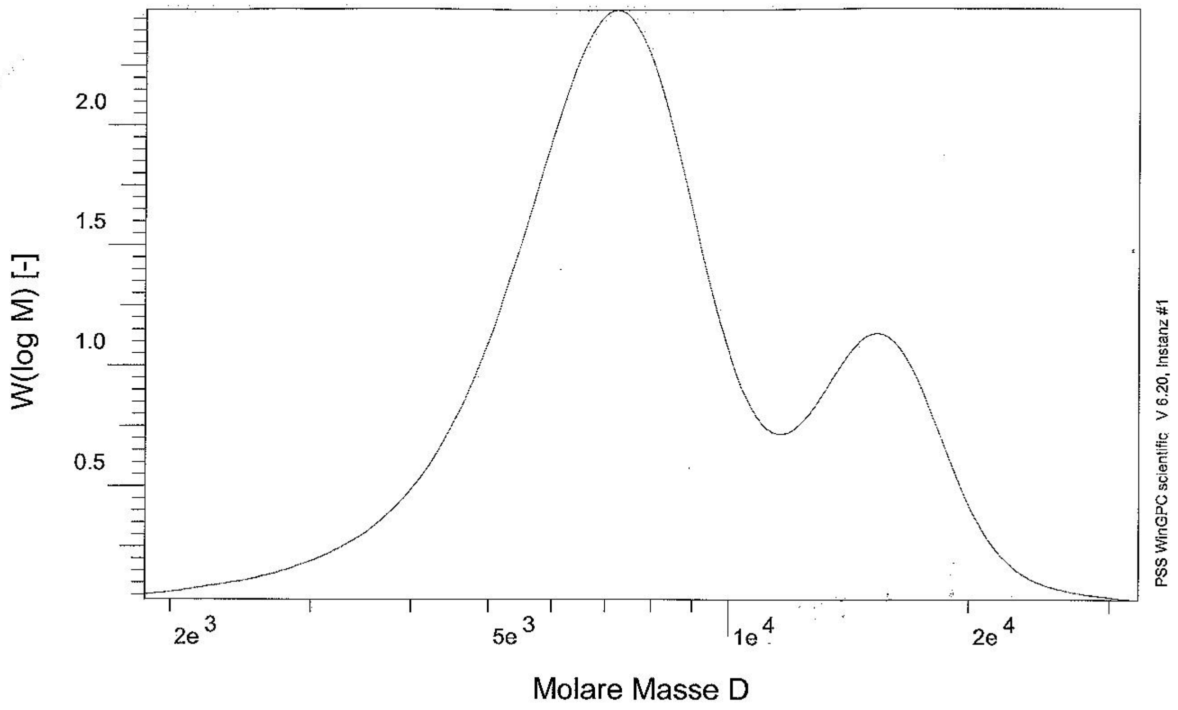


Differential Scanning Calorimetry (DSC):



Gel Permeation Chromatography (GPC): Best fit (according to PSS Software) for the elution volumes window



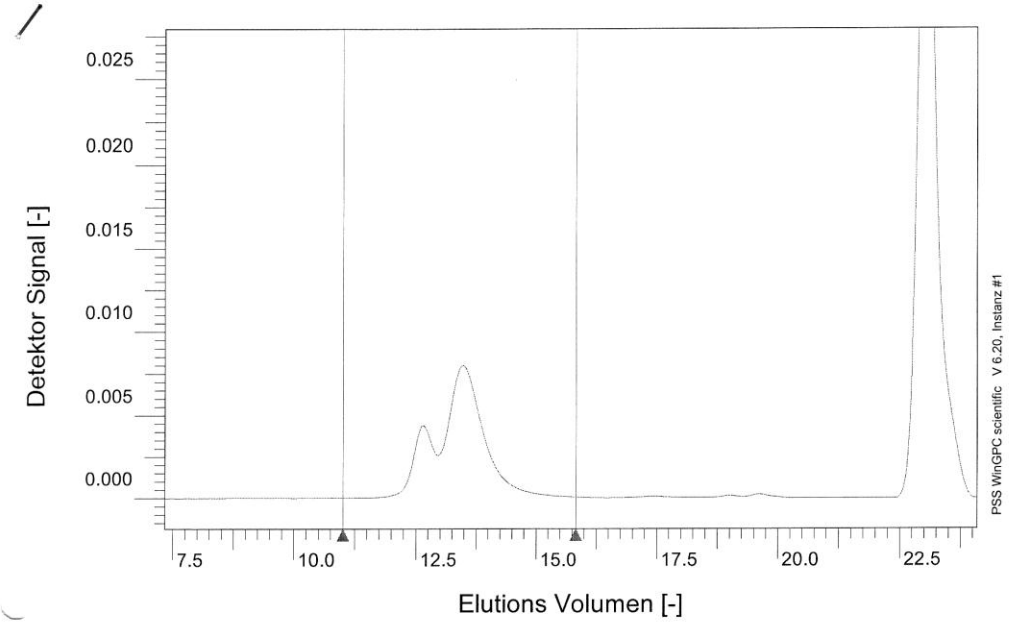


Probe :	MF445A_LK		
Integration von :	Mittwoch 31.07.13 13:35:14		11.944 ml
Integration bis :	Mittwoch 31.07.13 13:38:50		15.532 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------ ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec

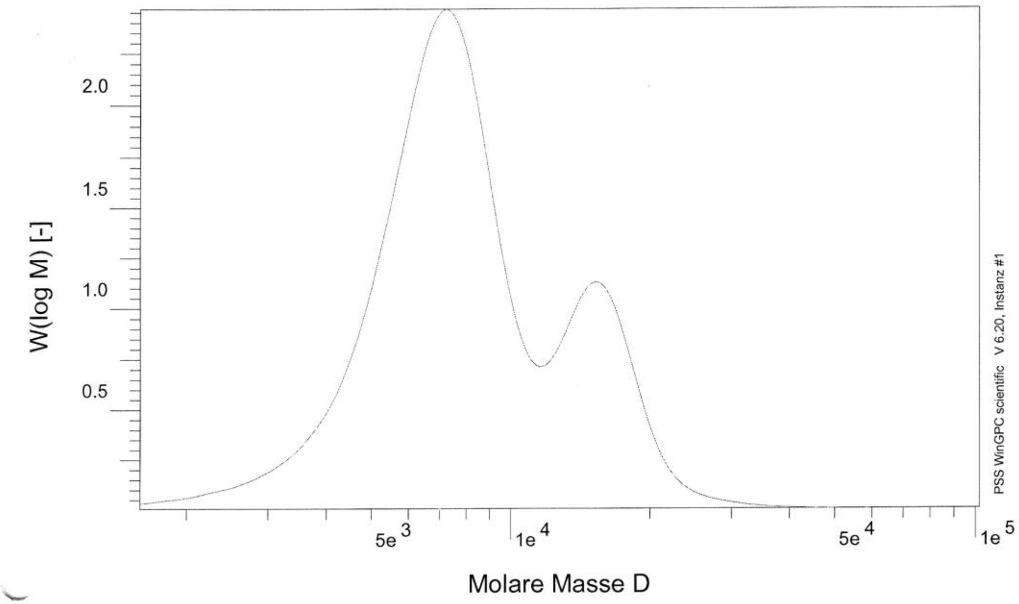
Merck RI

Mn :	7.2016e3	g/mol
Mw :	9.0662e3	g/mol
Mz :	1.1409e4	g/mol
Mv :	0.000000	g/mol
D :	1.2589e0	
[n]:	0.000000	ml/g
Vp :	1.3474e1	ml
Mp :	7.3396e3	g/mol
FI :	9.038e-3	ml*V
10%	4.5980e3	g/mol
30%	6.3116e3	g/mol
50%	7.6456e3	g/mol
80%	1.2947e4	g/mol
90%	1.6107e4	g/mol

Gel Permeation Chromatography (GPC): Broader elution volumes window



Probe :	MF445A_LK	**Methode :**	C:\Dokumente und Einstellungen\donat
Basislinie von :	7.329 ml	**bis :**	24.095 ml
Integration von :	11.008 ml	**bis :**	15.827 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------- ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec

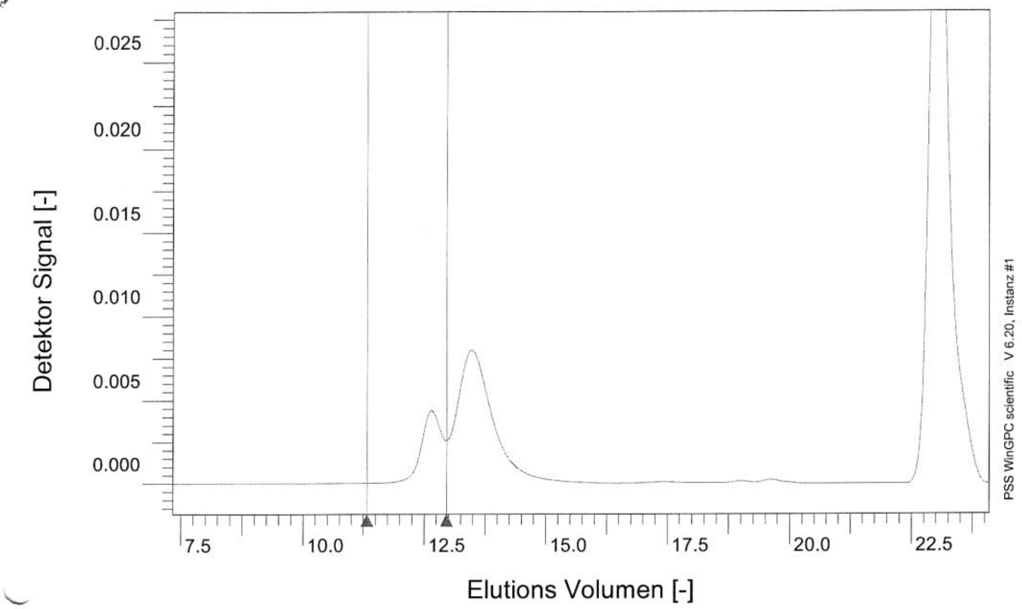


Probe :	MF445A_LK		
Integration von :	Mittwoch 31.07.13 13:34:18		11.008 ml
Integration bis :	Mittwoch 31.07.13 13:39:07		15.827 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------- ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec

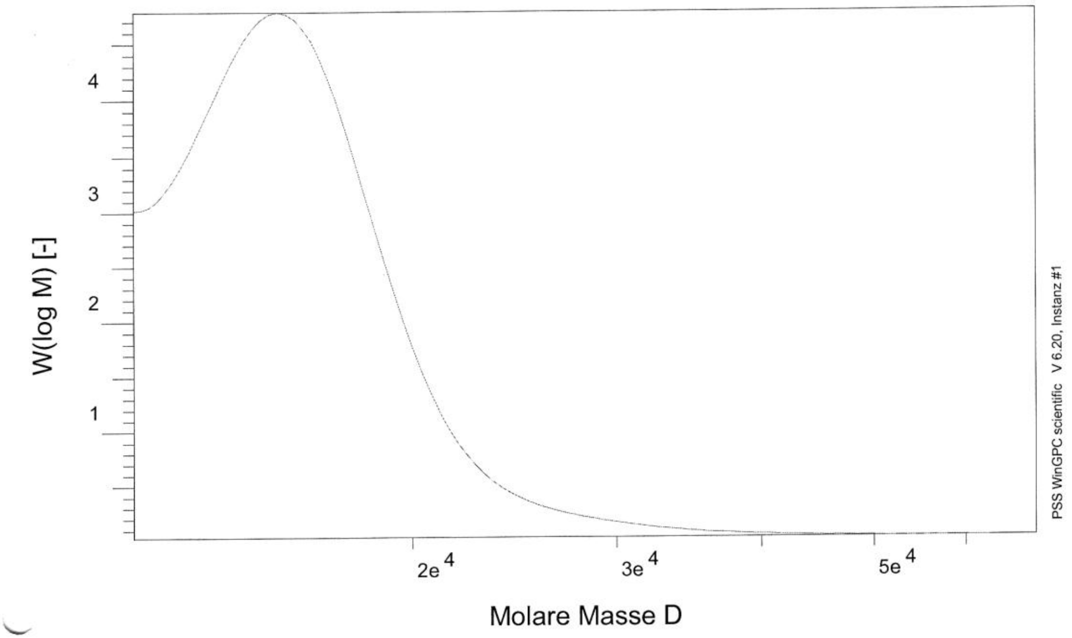
Merck RI

Mn :	7.1656e3	g/mol
Mw :	9.2707e3	g/mol
Mz :	1.2847e4	g/mol
Mv :	0.000000	g/mol
D :	1.2938e0	
[n]:	0.000000	ml/g
Vp :	1.3474e1	ml
Mp :	7.3396e3	g/mol
FI :	9.110e-3	ml*V
10%	4.5700e3	g/mol
30%	6.3079e3	g/mol
50%	7.6533e3	g/mol
80%	1.3067e4	g/mol
90%	1.6254e4	g/mol

Gel Permeation Chromatography (GPC). First signal: long-chained copolymers



Probe :	MF445A_LK	**Methode :**	C:\Dokumente und Einstellungen\donat
Basislinie von :	7.329 ml	**bis :**	24.095 ml
Integration von :	11.324 ml	**bis :**	12.957 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------- ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec

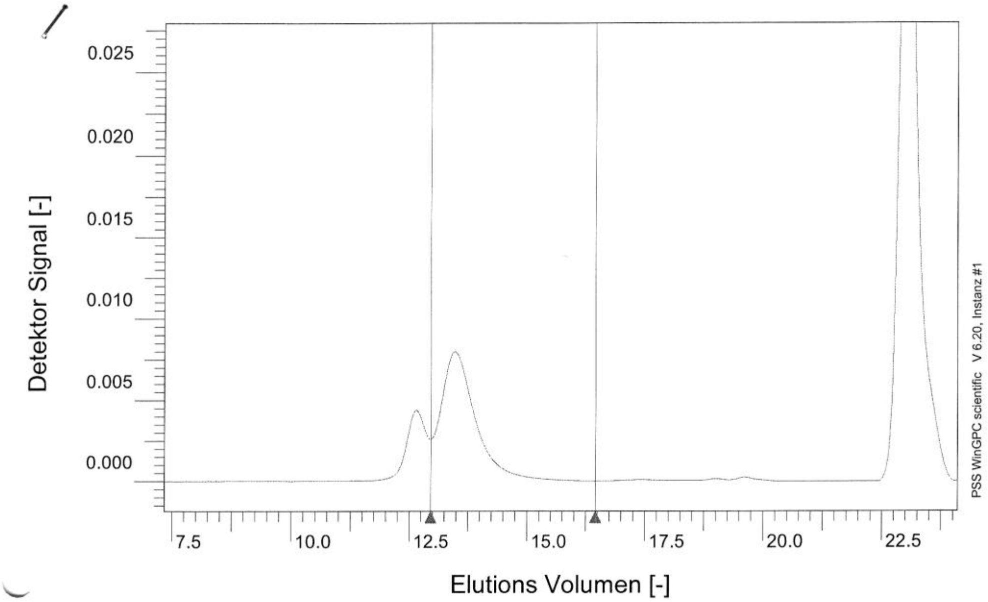


Probe :	MF445A_LK		
Integration von :	Mittwoch 31.07.13 13:34:37		11.324 ml
Integration bis :	Mittwoch 31.07.13 13:36:15		12.957 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------- ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec

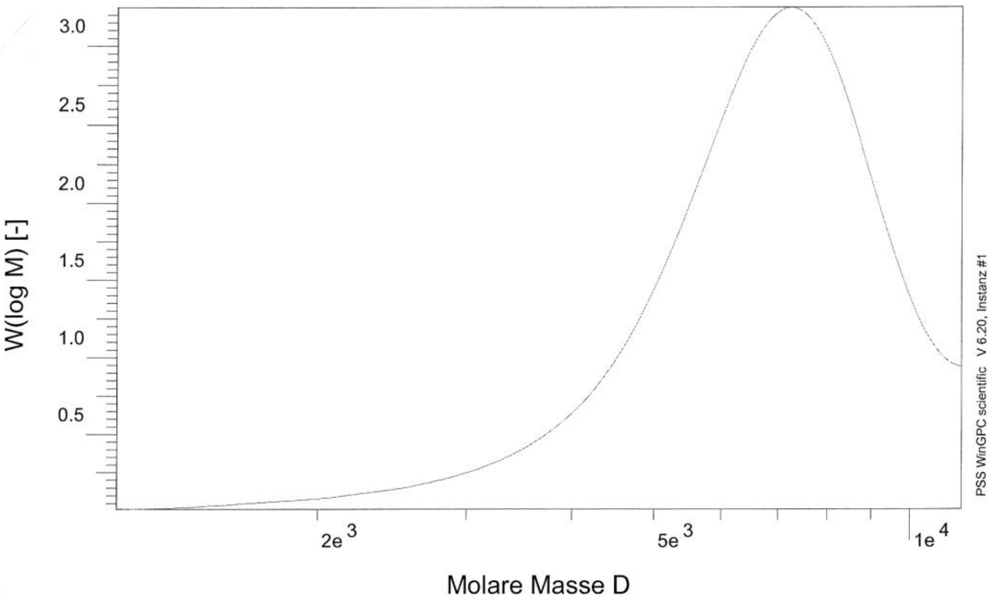
Merck RI

Mn :	1.5644e4	g/mol
Mw :	1.6541e4	g/mol
Mz :	1.8116e4	g/mol
Mv :	0.000000	g/mol
D :	1.0573e0	
[n]:	0.000000	ml/g
Vp :	1.2641e1	ml
Mp :	1.5557e4	g/mol
FI :	2.198e-3	ml*V
10%	1.2415e4	g/mol
30%	1.4044e4	g/mol
50%	1.5534e4	g/mol
80%	1.8499e4	g/mol
90%	2.0722e4	g/mol

Gel Permeation Chromatography (GPC). Second signal: short-chained copolymers



Probe :	MF445A_LK	**Methode :**	C:\Dokumente und Einstellungen\donat
Basislinie von :	7.329 ml	**bis :**	24.095 ml
Integration von :	12.958 ml	**bis :**	16.457 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------- ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec



Probe :	MF445A_LK		
Integration von :	Mittwoch 31.07.13 13:36:15		12.958 ml
Integration bis :	Mittwoch 31.07.13 13:39:45		16.457 ml
Kalibration :	23.03.12_CA1_THF.CAL	**Eluent :**	n.a.
MHK - A (Kal.):	0.000E+0	**MHK - K (Kal.):**	1.000E+0 ml/g
Int.Stand.-K :	22.821 ml	**Int.Stand.-M :**	------- ml
Pumpe :	Merck L-62000	**Flußrate :**	1.000 ml/min
Konzentration :	1.000 g/l	**Injektvolumen :**	50.000 ul
Säule 1 :	PSS SDV 5µ VS	**Temperatur :**	35.000 C
Säule 2 :	PSS SDV5µ 1000Å	**Temperatur :**	35.000 C
Säule 3 :	PSS SDV 5µ 100Å	**Temperatur :**	35.000 C
Detektor 1 :	Merck RI	**Versatz :**	0.000 ml
Operateur :	donath	**Messintervall :**	1.000 sec

Merck RI

Mn :	6.0340e3	g/mol
Mw :	6.8191e3	g/mol
Mz :	7.4251e3	g/mol
Mv :	0.000000	g/mol
D :	1.1301e0	
[n]:	0.000000	ml/g
Vp :	1.3474e1	ml
Mp :	7.3396e3	g/mol
FI :	6.924e-3	ml*V
10%	4.1739e3	g/mol
30%	5.7779e3	g/mol
50%	6.8191e3	g/mol
80%	8.5438e3	g/mol
90%	9.5148e3	g/mol