Order-disorder phase transition induced by proton transfer in a co-crystal of 2,4-dichlorobenzoic Acid and Trimethylamine N- oxide

R. F. D'Vries,[a]* R. Moreno-Fuquen,[b] I. Camps,[c] A. P. Ayala,[d] A. R. Kennedy,[e] E. W. Reinheimer[f] and J. Ellena.[g]

[a]*Universidad Santiago de Cali, Calle 5 # 62-00, Cali, Colombia.*
[b]*Departamento de Química - Facultad de Ciencias, Universidad del Valle, Apartado 25360, Santiago de Cali, Colombia.*
[c]*Laboratório de Modelagem Computacional—LaModel, Instituto de Ciências Exatas, Universidade Federal de Alfenas—Unifal-MG, CEP 37130-000 Alfenas, Minas Gerais, Brazil.*
[d] *Departamento de Física, Universidade Federal do Ceará, CP 6030, 60440-900, Fortaleza, CE, Brazil.*
[e]*WestCHEM, Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland.*
[f] *Rigaku Oxford Diffraction • 9009 New Trails Drive • The Woodlands, TX 77381 USA.*
[g]*Instituto de Física de São Carlos, Universidade de São Paulo, USP, São Carlos, SP, Brasil.*

Supporting Information.

Section S1. Ortep diagram with 50% of probability ellipsoid and crystallographic data and refinement parameters for TMANO-2,4-DCB molecular complex in P212121 space group.

Section S2. Heating and cooling X-ray powder diffraction patterns for the TMANO-2,4-DCB molecular complex and cell parameters changes.

Section S3. Simulated powder diffraction patterns for the TMANO-2,4-DCB molecular complex measured to 100, 150, 200, 250 and 298K.

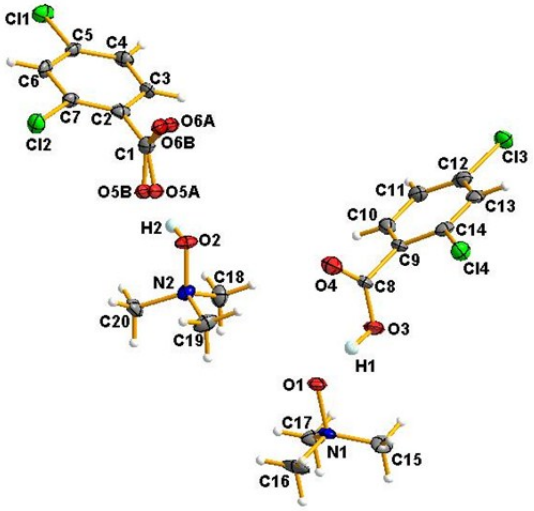
Section S4. Hot Stage microscopy for the TMANO-2,4-DCB molecular complex.

Section S5. TG, DSC analysis and hot stage microscopy for the TMANO-2,4-DCB molecular complex.

Section S6. FT-IR vs T spectrum for the TMANO-2,4-DCB molecular complex and FT-IR for the 2,4-dichlorobenzoic acid.

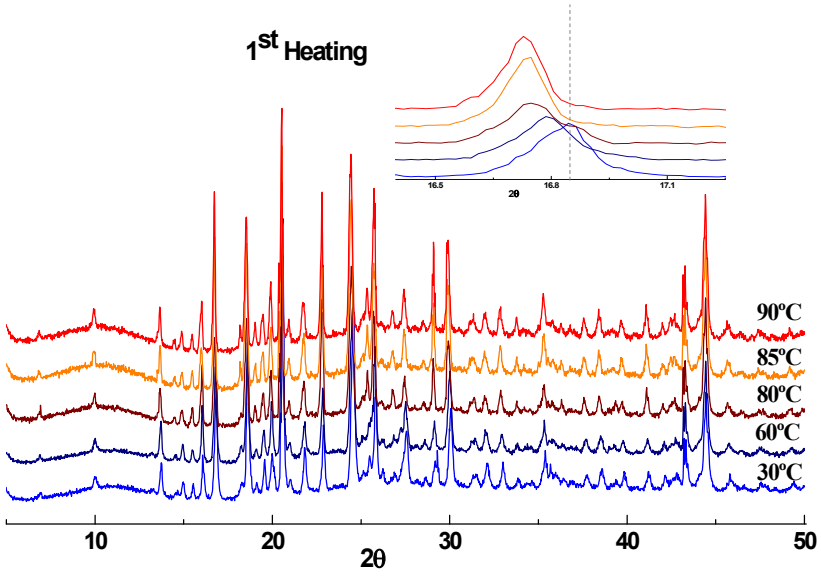
Section S7. PASCal output obtained for cell parameters data, showing the principal components of the expansivity tensor and the expansivity indicatrix representation.

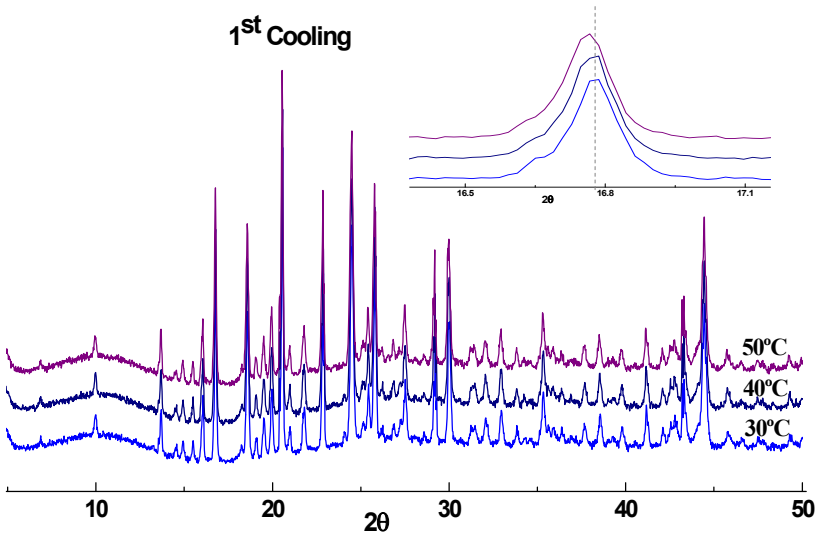
Section S1. Ortep diagram with 50% of probability ellipsoid and crystallographic data and refinement parameters for TMANO-2,4-DCB molecular complex in P212121 space group.

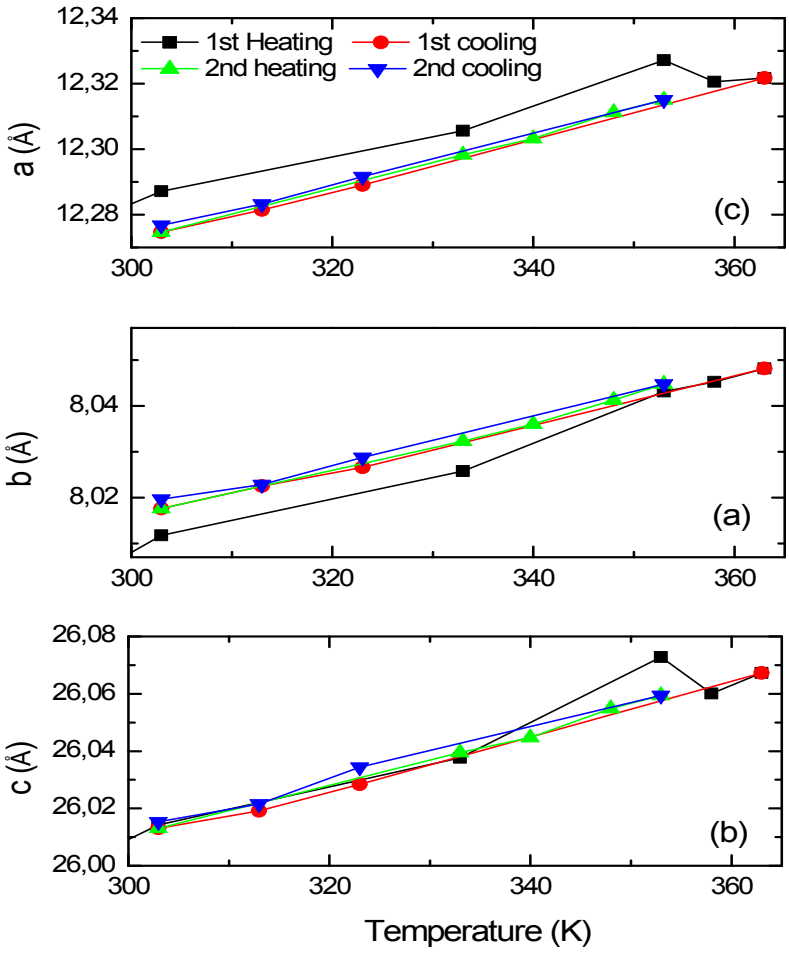


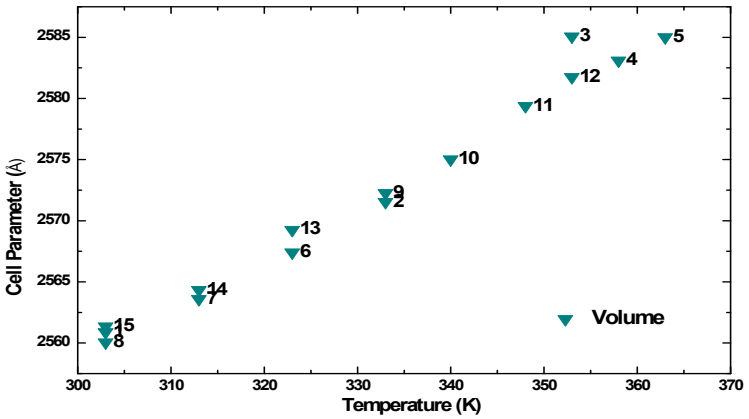
	1
Emp. Formula	$(C_7H_4O_2Cl_2 \cdot C_3H_9NO)_2$
FW (g/mol)	532.23
Temp. (K)	100
λ (Å)	1.54184
Crystal system	orthorhombic
Space Group	P212121
Unit cell	
a (Å)	12.1285(1)
b (Å)	7.92362(7)
c (Å)	25.8171(2)
Volume (Å^3)	2481.07(3)
Z	4
ρ calcd (mg/m^3)	1.425
Abs.Coeff (mm^{-1})	4.667
F(000)	1104
θ range (°)	4.0 to 74.1
Reflections collected / Unique [R(int)]	47327/4994 [0.036]
Flack x	0.488(16)
Completeness (%)	99.92
Data / restraints / parameters	4994/0/317
Gof on F^2	1.06
R1 [I>2σ(I)]	0.0295
wR2[I>2σ(I)]	0.0803

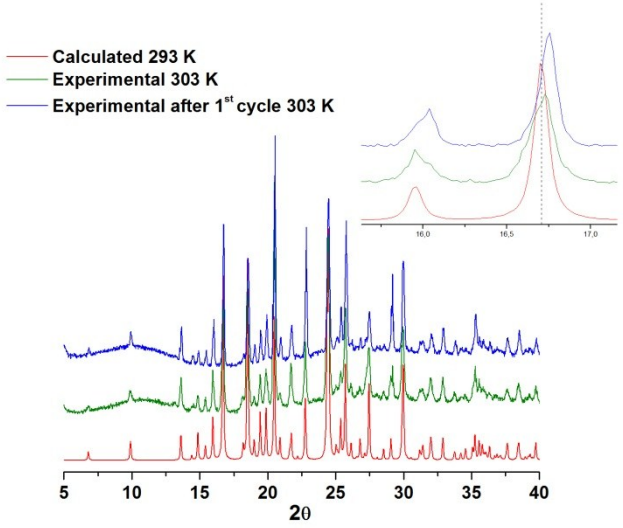
Section S2. Heating and cooling X-ray powder diffraction patterns for the TMANO-2,4-DCB molecular complex and cell parameters changes.



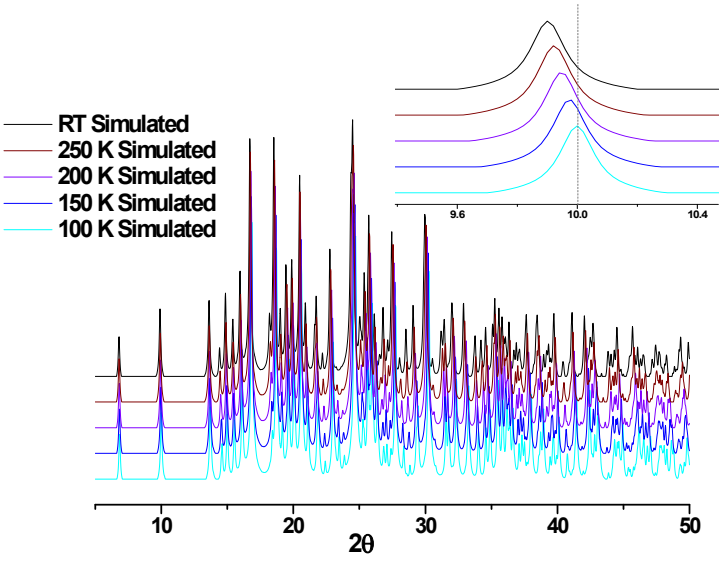



1st Heating
1st cooling
2nd heating
2nd cooling
a (Å)
b (Å)
c (Å)
(c)
(a)
(b)
Temperature (K)

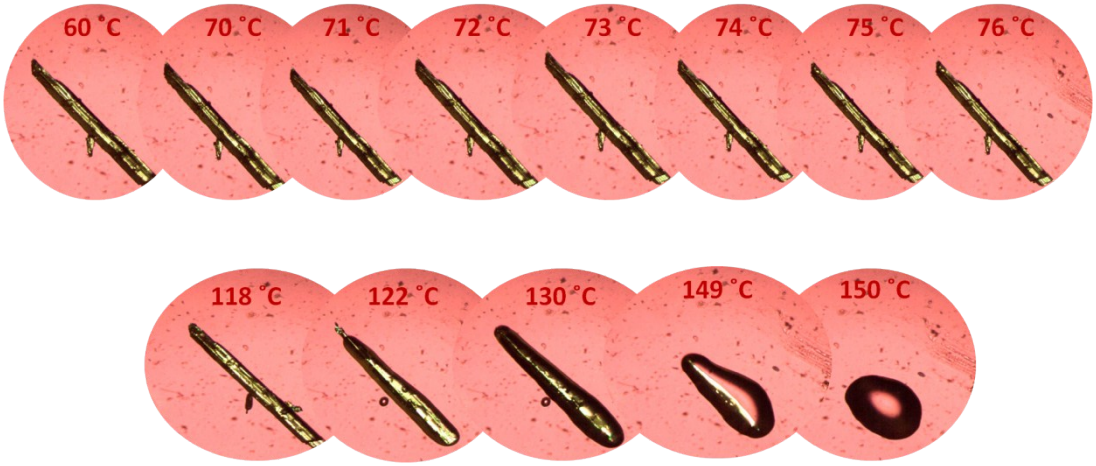

Cell Parameter (Å)
Temperature (K)
Volume



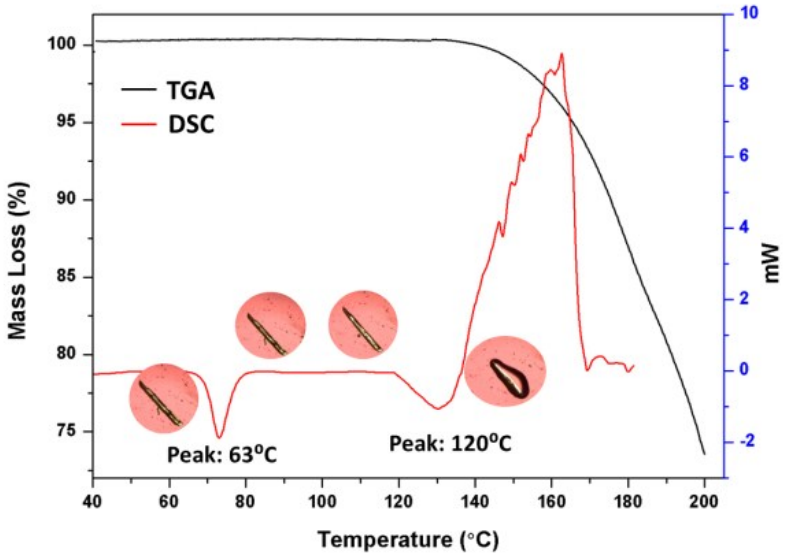
Section S3. Simulated powder diffraction patterns for the TMANO-2,4-DCB molecular complex measured to 100, 150, 200, 250 and 298K.



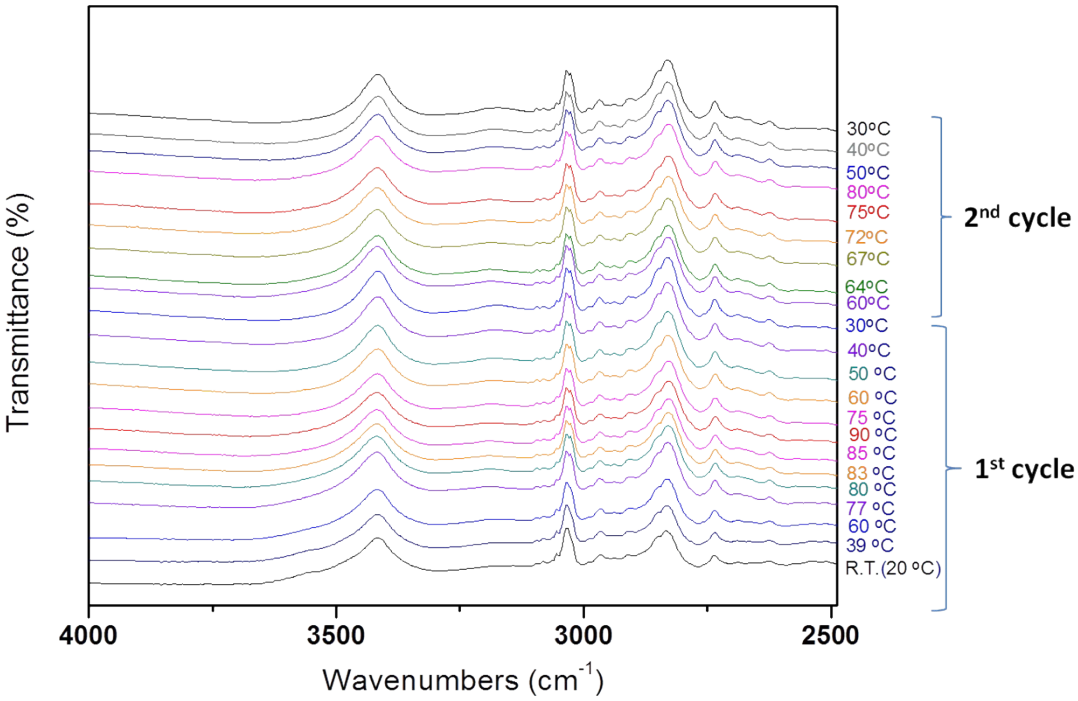
Section S4. Hot Stage microscopy for the TMANO-2,4-DCB molecular complex.

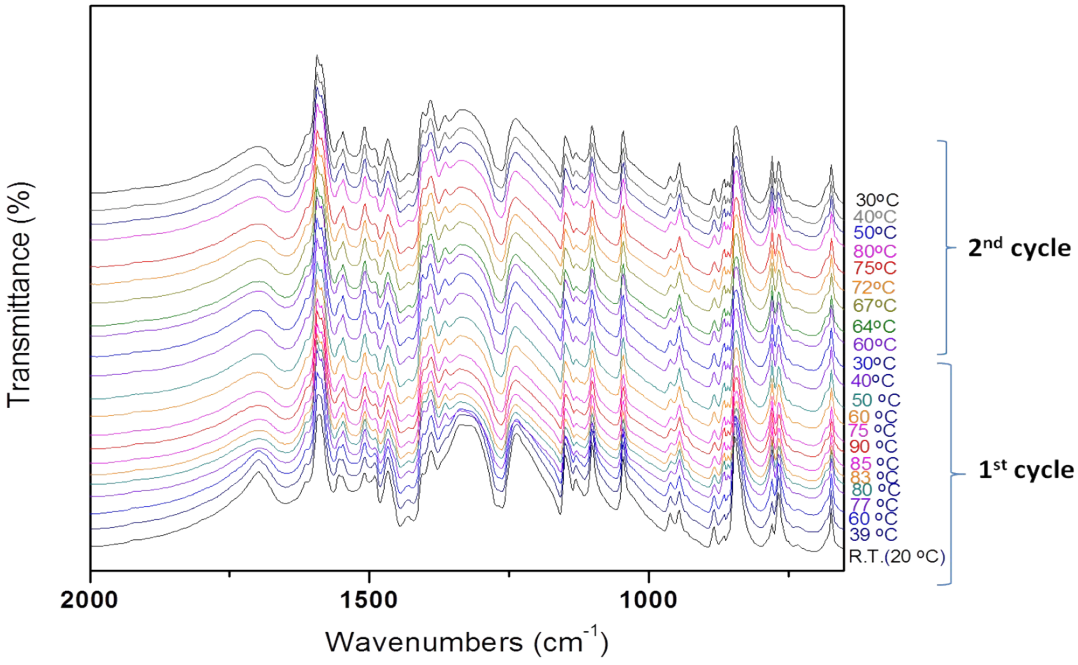


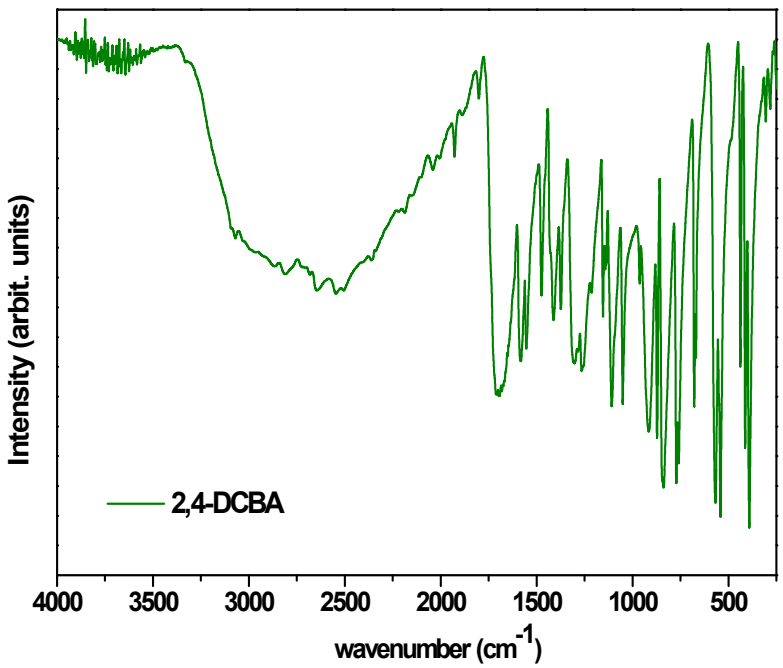
Section S5. TG, DSC analysis and hot stage microscopy for the TMANO-2,4-DCB molecular complex.



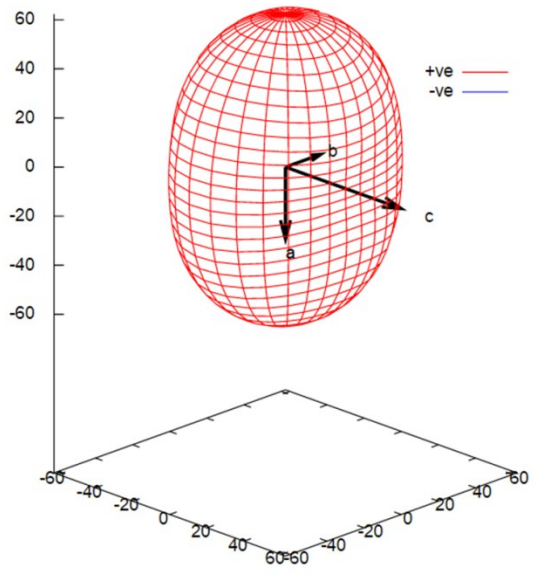
Section S6. FT-IR vs T spectrum for the TMANO-2,4-DCB molecular complex and FT-IR for the 2,4-dichlorobenzoic acid.




Transmittance (%)
2000
1500
1000
Wavenumbers (cm-1)
30ºC
40ºC
50ºC
80ºC
75ºC
72ºC
67ºC
64ºC
60ºC
30ºC
40ºC
50 ºC
60 ºC
75 ºC
90 ºC
85 ºC
83 ºC
80 ºC
77 ºC
60 ºC
39 ºC
R.T.(20 ºC)
2nd cycle
1st cycle


Intensity (arbit. units)
2,4-DCBA
4000
3500
3000
2500
2000
1500
1000
500
wavenumber (cm-1)

Section S7. PASCal output obtained for cell parameters data, showing the principal components of the expansivity tensor and the expansivity indicatrix representation.



			Direction		
Axes	$\alpha(MK^{-1})$	$\sigma\alpha\ (MK^{-1})$	a	b	c
X_1	36.3519	1.7228	0.0000	0.0000	1.0000
X_2	49.3726	1.5904	0.0000	1.0000	-0.0000
X_3	62.3441	0.7792	-1.0000	0.0000	0.0000
V	149.5019	0.8547			