

Application of in Situ FTIR for the preparation of 17- α -estradiol via Mitsunobu reaction

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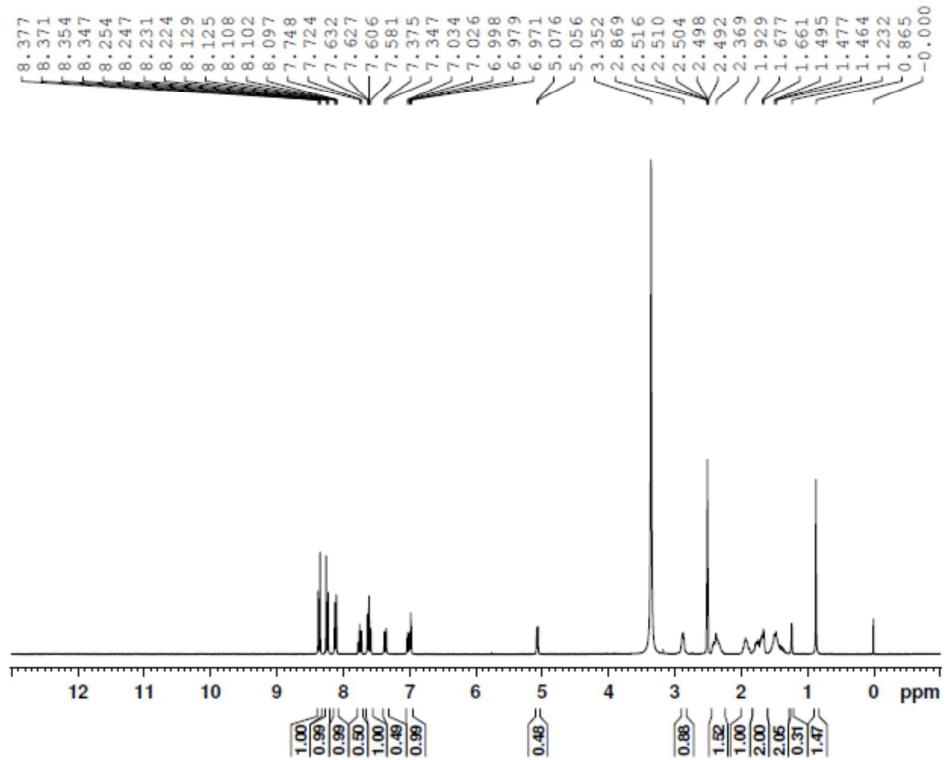
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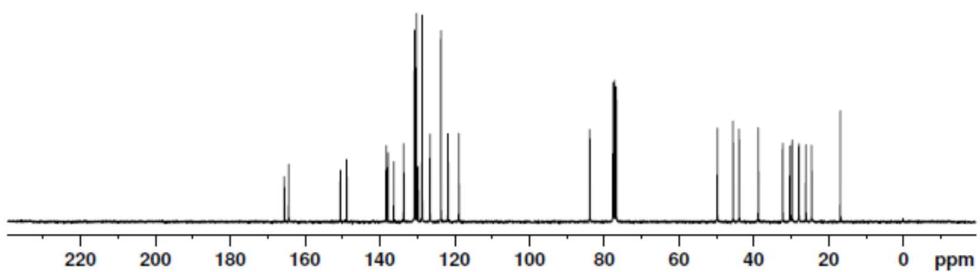
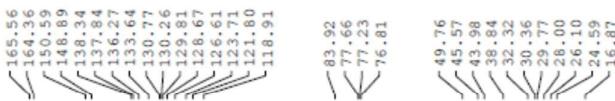
SUPPORTING INFORMATION

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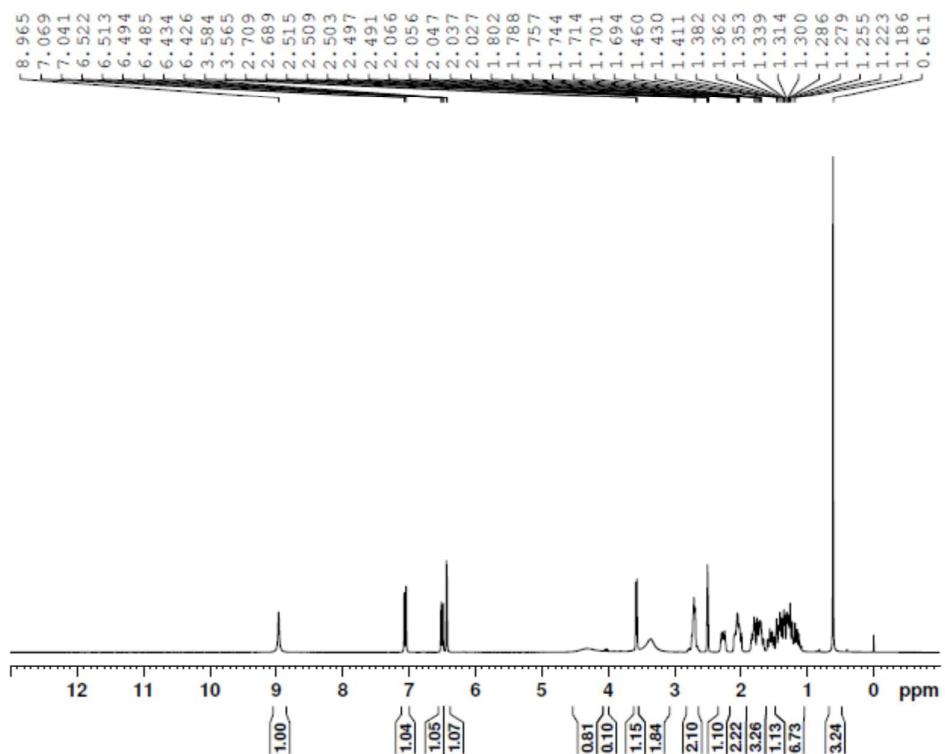
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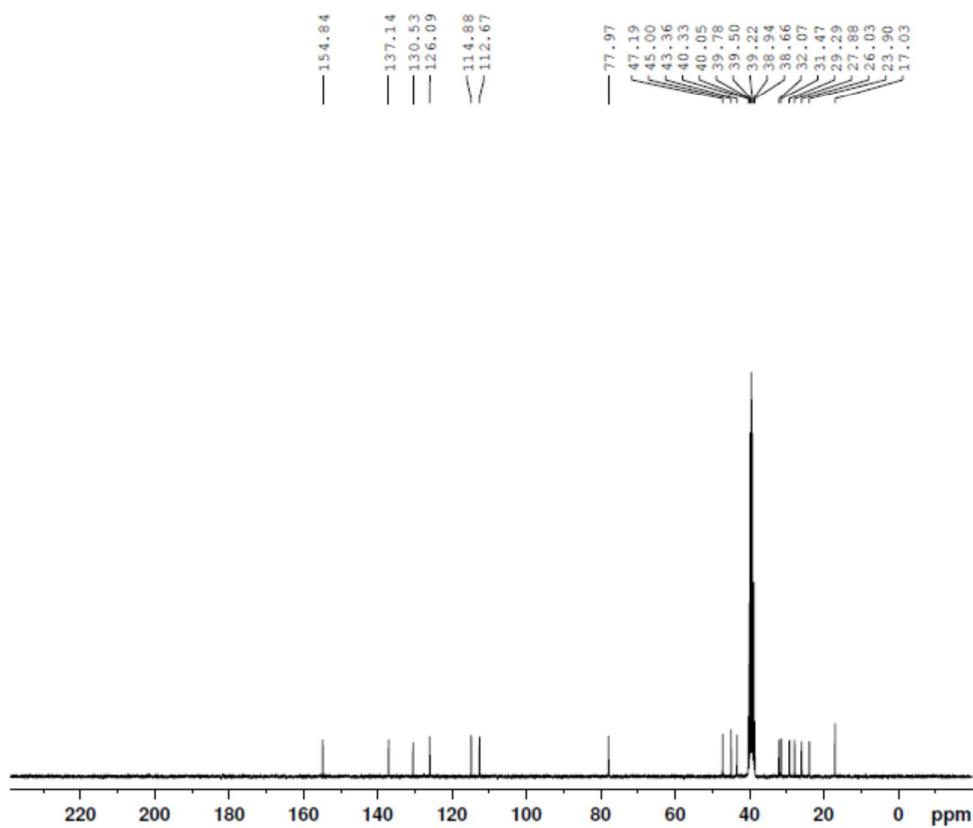
^1H NMR (300 MHz, DMSO-d₆) of compound **5**



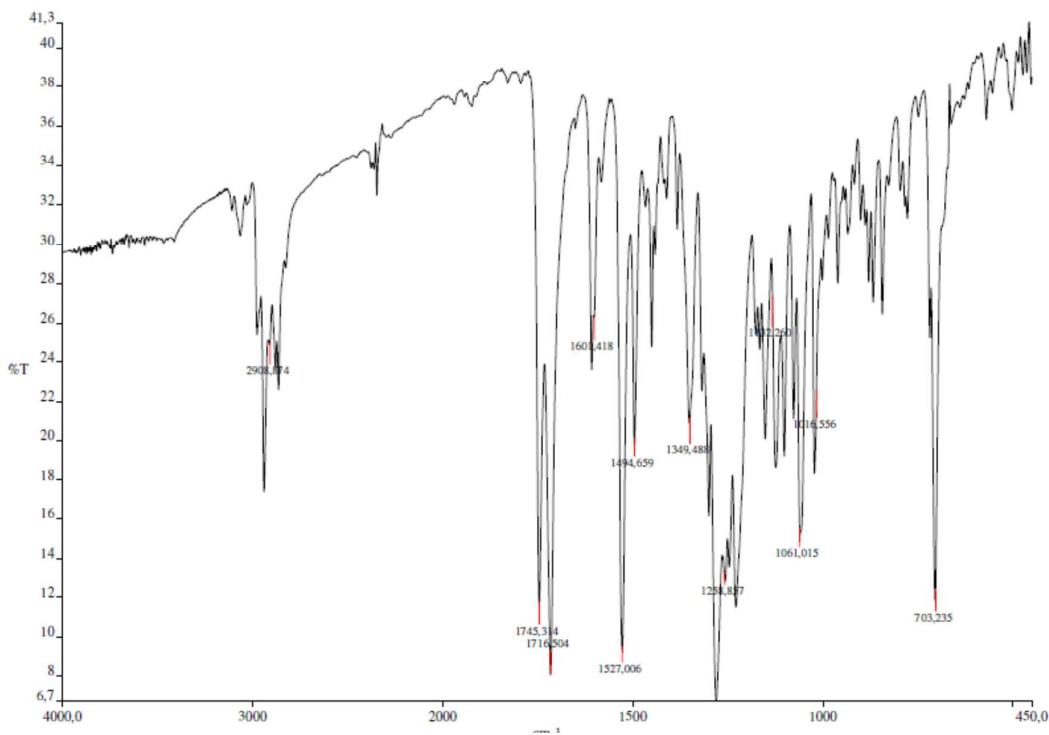
^{13}C NMR (75 MHz, CDCl₃) of compound **5**



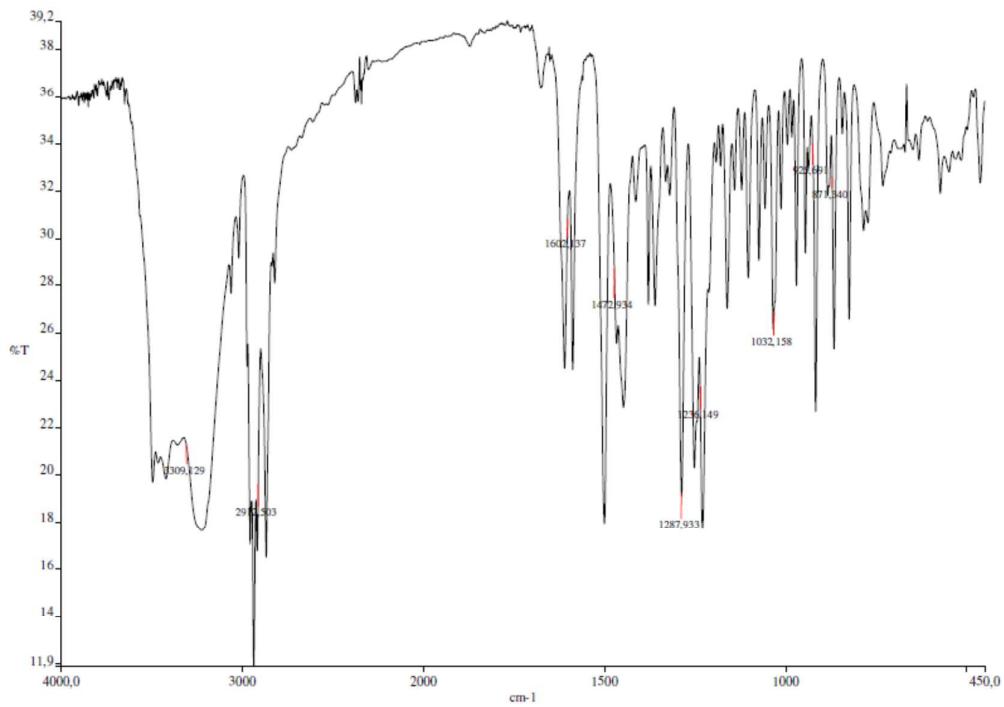
^1H NMR (300 MHz, DMSO-d₆) of 17- α -estradiol **1**



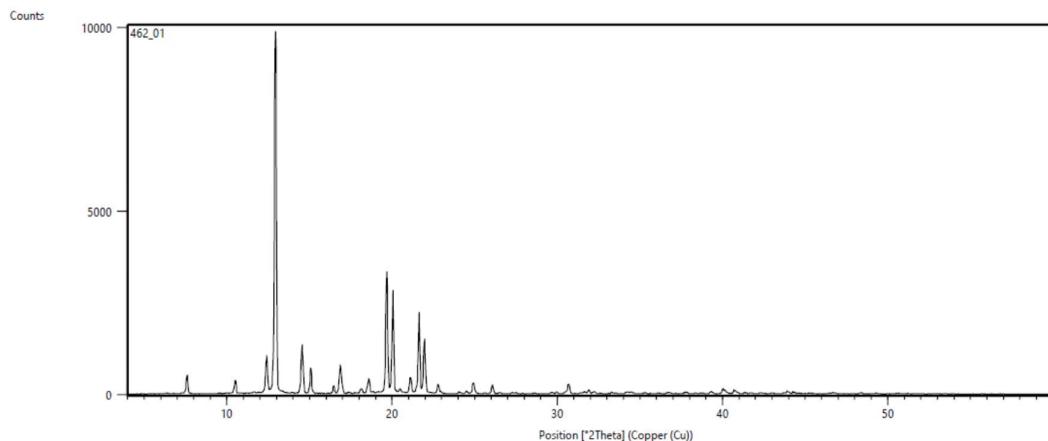
^{13}C NMR (75 MHz, DMSO-d₆) of 17- α -estradiol **1**



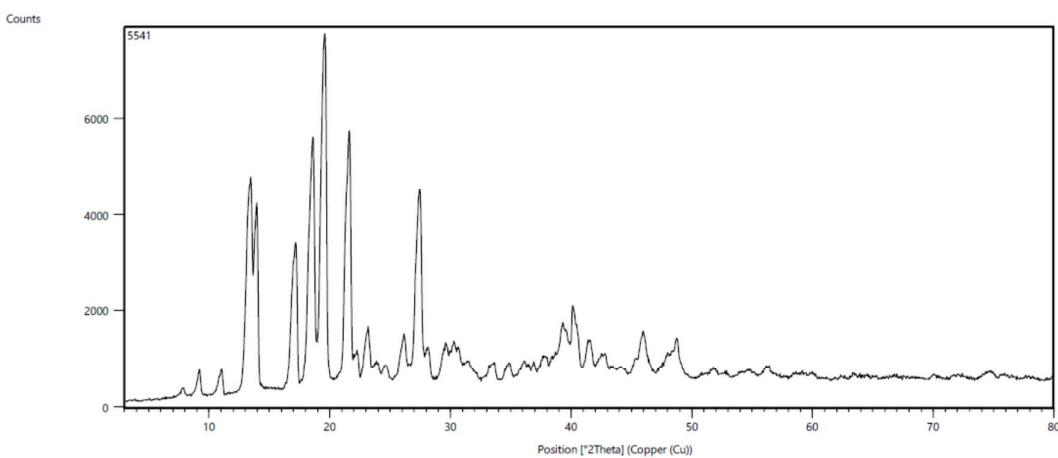
IR of compound 5



IR of 17- α -estradiol 1



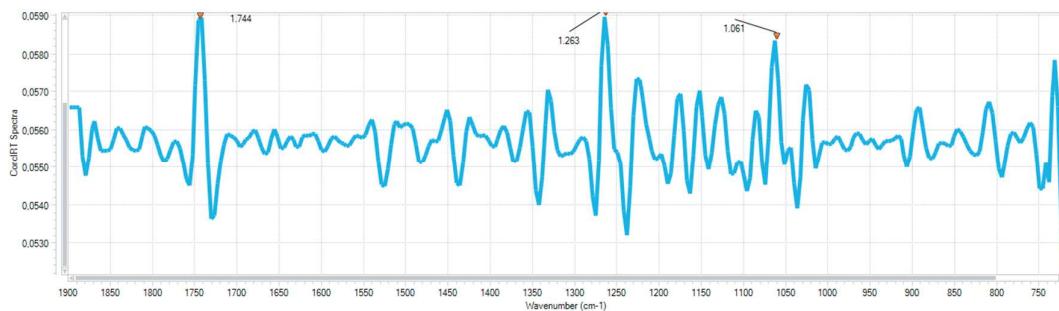
XRD of anhydrous 17- α -estradiol **1**



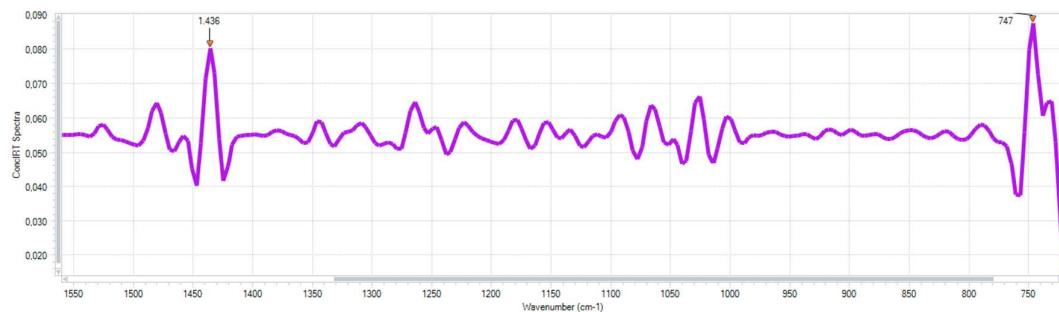
XRD of 17- α -estradiol hemi-hydrate

Solubility of compound **5** and triphenylphosphine oxide in different solvents at reflux temperature

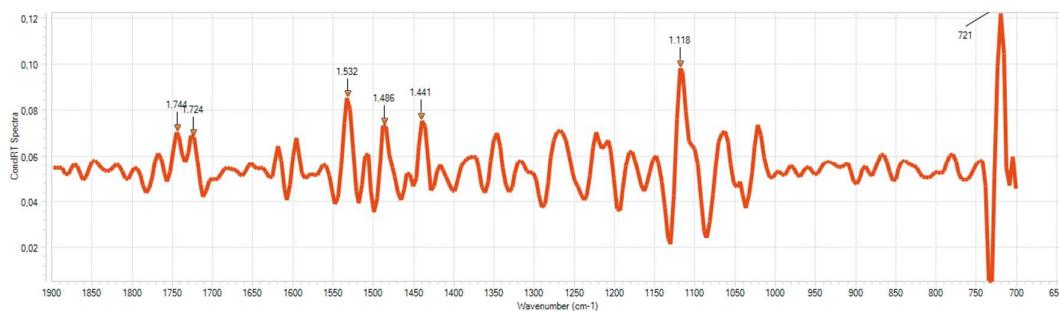
Entry	Solvent	Compound 5		Triphenylphosphine oxide	
		T (°C)	Solubility (mg/mL)	T (°C)	Solubility (mg/mL)
1	Methyl <i>t</i> -butyl ether	54	6.1	53	13.4
2	Ethyl acetate	73	20.5	74	33.6
3	Acetone	54	28.9	53	40.1
4	Tetrahydrofuran	62	200.6	63	50.5
5	Methanol	62	1.2	62	200.1
6	Ethanol	76	2.2	74	201.8
7	Isopropanol	81	2.5	79	201.8
8	Toluene	107	25.2	108	133.5
9	<i>n</i> -heptane	95	2.5	95	8.0
10	Cyclohexane	77	4.0	78	5.7
11	Acetonitrile	77	18.2	79	100.7



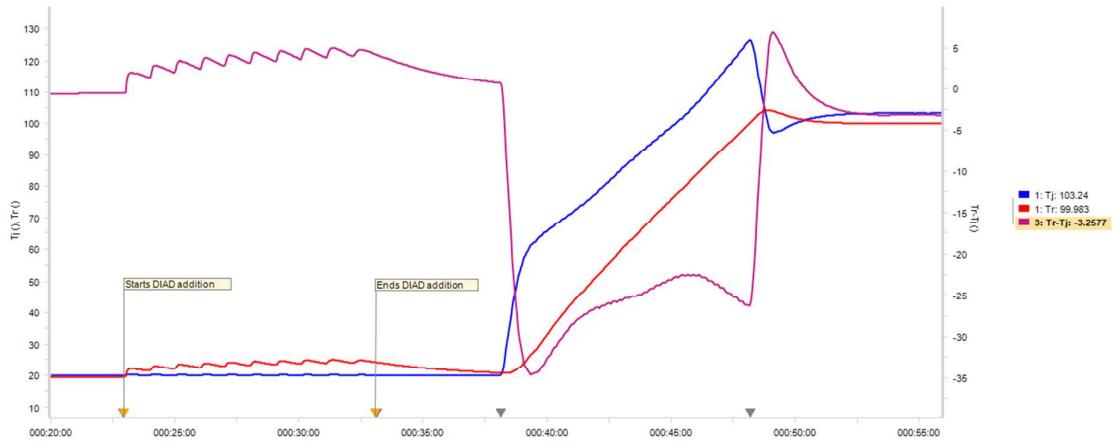
ConcIRT spectrum profile of Compound 3 (estradiol 3-benzoate)



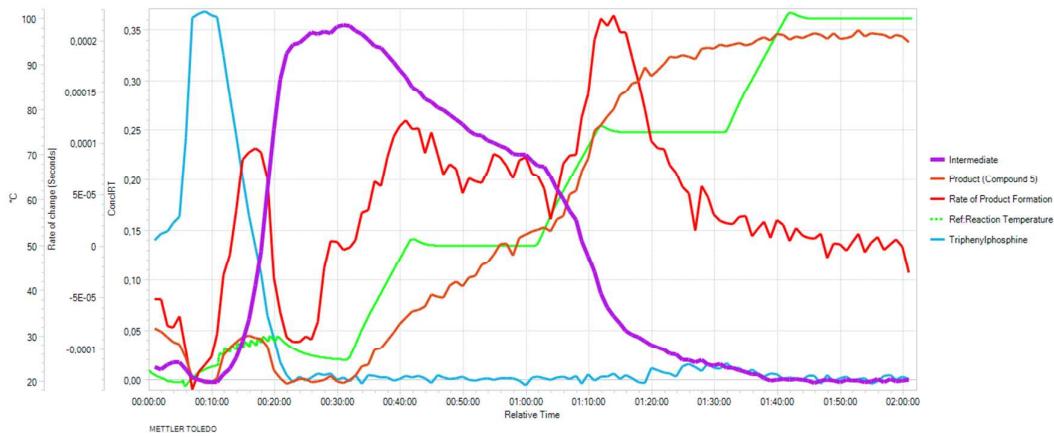
ConcIRT spectrum profile of triphenylphosphine



ConcIRT spectrum profile of Compound 5 (Mitsunobu Reaction Product)



Jacket vs Reaction Temperatures during the Mitsunobu Reaction presented in Figure 2



ConcIRT trends profile for Mitsunobu Reaction under different temperatures